



SLOVENSKI STANDARD
SIST EN ISO 12183:2019

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Tehnologija jedrskih goriv - Kulometrična analiza plutonija z nadzorovanim potencialom (ISO 12183:2016)

Nuclear fuel technology - Controlled-potential coulometric assay of plutonium (ISO 12183:2016)

Kernbrennstofftechnologie - Coulometrische Bestimmung von Plutonium mit kontrolliertem Potential (ISO 12183:2016)

Technologie du combustible nucléaire - Dosage du plutonium par coulométrie à potentiel imposé (ISO 12183:2016)

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EUROPEAN STANDARD

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Nuclear fuel technology - Controlled-potential coulometric assay of plutonium (ISO 12183:2016)

Technologie du combustible nucléaire - Dosage du plutonium par coulométrie à potentiel imposé (ISO 12183:2016)

Kernbrennstofftechnologie - Coulometrische Bestimmung von Plutonium mit kontrolliertem Potential (ISO 12183:2016)

This European Standard was approved by CEN on 8 March 2019.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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European foreword

The text of ISO 12183:2016 has been prepared by Technical Committee ISO/TC 85 "Nuclear energy, nuclear technologies, and radiological protection" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 12183:2019 by Technical Committee CEN/TC 430 "Nuclear energy, nuclear technologies, and radiological protection" the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2019, and conflicting national standards shall be withdrawn at the latest by December 2019.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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INTERNATIONAL
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Third edition
2016-08-15

**Nuclear fuel technology — Controlled-
potential coulometric assay of
plutonium**

*Technologie du combustible nucléaire — Dosage du plutonium par
coulométrie à potentiel imposé*

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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 (see 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 (see 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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 12183:2005), which has been technically revised.

Nuclear fuel technology — Controlled-potential coulometric assay of plutonium

1 Scope

This document describes an analytical method for the electrochemical assay of pure plutonium nitrate solutions of nuclear grade, with a total uncertainty not exceeding $\pm 0,2$ % at the confidence level of 0,95 for a single determination (coverage factor, $K = 2$). The method is suitable for aqueous solutions containing more than 0,5 g/L plutonium and test samples containing between 4 mg and 15 mg of plutonium. Application of this technique to solutions containing less than 0,5 g/L and test samples containing less than 4 mg of plutonium requires experimental demonstration by the user that applicable data quality objectives will be met.

For some applications, purification of test samples by anion exchange is required before measurement to remove interfering substances when present in significant amounts. Refer to [Clause 10](#) for a discussion of interferences and corrective actions. Purification is also appropriate in situations where the purity of the test sample is unknown or when it may fluctuate unpredictably in a manufacturing process.

[Clause 11](#) discusses the changes in application of the method and methodology that can be applied and important considerations when selecting measurement parameters, while still remaining within the intended scope of this document.

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 terminological 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

The key steps and their purposes are outlined below:

- test samples are prepared by weighing and then fuming to dryness with sulphuric acid to achieve a consistent and stable anhydrous plutonium sulphate salt that is free from chloride, fluoride, nitrate, nitrite, hydroxylamine, and volatile organic compounds;
- if needed to remove interferences, dissolve test samples and purify by anion exchange, then fume the eluted plutonium solution in the presence of sulphuric acid to obtain the dry plutonium sulphate chemical form;
- measure a blank of the nitric acid supporting electrolyte and calculate the background current correction applicable to the electrolysis of the test sample from charging, faradaic, and residual current^[1];
- dissolve the dried test sample in the previously measured supporting electrolyte (the blank);

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- reduce the test sample at a controlled potential that electrolyses the plutonium to greater than 99,8 % Pu³⁺ and measure the equilibrium solution potential at the end of this step by control-potential adjustment[2];
- oxidize the test sample at a controlled potential that electrolyses the plutonium to greater than 99,8 % Pu⁴⁺ and measure the equilibrium solution potential at the end of this electrolysis by control-potential adjustment;
- correct the result for the background current and the fraction of plutonium not electrolysed;
- calibrate the coulometer using traceable electrical standards and Ohm's Law;
- use the measured value of the coulometer calibration factor and the Faraday constant to convert the coulombs of integrated current from the electrolyses to moles of plutonium;
- use traceable quality-control plutonium standards to demonstrate independently the performance of the measurement system;
- periodically measure the formal potential of the plutonium couple, E_0 , which is user-specific based on the cell design, connections, reference electrode type, and the acid-type and molarity of the supporting electrolyte.

These steps ensure that representative, reproducible, and stable test samples are prepared for measurement. The test samples are measured using a protocol that is based upon first principles and is consistent with a traceable, electrical calibration of the coulometer. Additional details are provided in [Clauses 10](#) and [11](#).

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5 Reagents

Use only analytical grade reagents.

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All aqueous solutions shall be prepared with double-distilled or distilled, demineralized water with a resistivity greater than 10 M Ω ·cm, i.e. ISO 3696 Grade 1 purified water.

5.1 Nitric acid solution, $c(\text{HNO}_3) = 0,9 \text{ mol/L}$.

NOTE Refer to [11.4](#) for other electrolyte options.

5.2 Amidosulphuric acid solution, $c(\text{NH}_2\text{HSO}_3) = 1,5 \text{ mol/L}$.

5.3 Sulphuric acid solution, $c(\text{H}_2\text{SO}_4) = 3 \text{ mol/L}$.

NOTE Molarity is not a critical parameter for sulphuric acid used to fume plutonium test samples, provided the concentration of the reagent is well above the level where colloidal plutonium complexes form.

5.4 Pure argon or nitrogen, (O_2 content lower than 10 ppm).

5.5 Pure air, free of organic contaminants.

6 Apparatus

Usual laboratory equipment found in a medium-activity radiochemical laboratory suitable for work with plutonium shall be used.

6.1 Analytical balance, installed in radiological containment unit and must be capable of weighing 1 g mass, with an uncertainty of $\pm 0,1 \text{ mg}$ (coverage factor, $K = 1$). This represents a relative uncertainty of 0,01 %.