SLOVENSKI STANDARD
SIST EN ISO 12183:2024

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Tehnologija jedrskih goriv - Kulometrična meritev plutonija z nadzorovanim potencialom (ISO 12183:2024)

Nuclear fuel technology - Controlled-potential coulometric measurement of plutonium (ISO 12183:2024)

Kernbrennstofftechnologie - Coulometrische Bestimmung von Plutonium mit kontrolliertem Potential (ISO 12183:2024)

Technologie du combustible nucléaire - Dosage du plutonium par coulométrie à potentiel imposé (ISO 12183:2024)

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Nuclear fuel technology - Controlled-potential coulometric measurement of plutonium (ISO 12183:2024)

Technologie du combustible nucléaire - Dosage du plutonium par coulométrie à potentiel imposé (ISO 12183:2024)

Kernbrennstofftechnologie - Coulometrische Bestimmung von Plutonium mit kontrolliertem Potential (ISO 12183:2024)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN ISO 12183:2024) has been prepared by Technical Committee ISO/TC 85 "Nuclear energy, nuclear technologies, and radiological protection" in collaboration with 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of an identical text or by endorsement, at the latest by November 2024, and conflicting national standards shall be withdrawn at the latest by November 2024.

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.

This document supersedes EN ISO 12183:2019.

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Endorsement notice

The text of ISO 12183:2024 has been approved by CEN as EN ISO 12183:2024 without any modification.

International Standard

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Nuclear fuel technology — Controlled-potential coulometric measurement of plutonium

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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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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection,* Subcommittee SC 5, *Nuclear fuel cycle,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 430, *Nuclear energy,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 12183:2016), which has been technically revised.

The main changes are as follows:

- Figures 1 and 2 have been revised to resolve errors introduced in the third edition of this document;
- quantity values and uncertainties values have been reformatted to comply with requirements for properly stating these values with SI units;
- editorial changes were made throughout the document to ensure clarity of the instructions;
- words with optional spellings were corrected to match ISO/IEC guidance;
- an additional key step was added to Clause 4 to indicated that the moles of plutonium obtained by controlled-potential coulometry is multiplied by the molar mass of plutonium obtained by other means, such as mass spectrometry or process knowledge;
- a formula has been added to 8.4 to calculate the amount of substance of plutonium in millimoles in addition to the mass of plutonium in milligrams;
- Clause 12 has been added to discuss traceability to SI units.

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 — Controlled-potential coulometric measurement of plutonium

1 Scope

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

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:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The key steps and their purposes are outlined below:

— test samples are prepared from homogenous solutions by weighing and then fuming to dryness with sulfuric acid to achieve a stable anhydrous plutonium sulfate 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 sulfuric acid to obtain the anhydrous plutonium sulfate salt;

— measure the supporting electrolyte blank and calculate the background current correction applicable to the electrolysis of the test sample from charging, faradaic, and residual currents[1];

— dissolve the dried test sample in the previously measured supporting electrolyte (the blank);

— reduce the test sample at a controlled potential that electrolyses the plutonium to a Pu^{3+} amount of substance fraction greater than 99,8 % 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 a Pu^{4+} amount fraction greater than 99,8 % and measure the equilibrium solution potential at the end of this electrolysis by control-potential adjustment;

— correct the integrated current (integrator output from the test sample) for the background current, including the residual current corrections, and for the amount fraction of plutonium not electrolysed;

— calibrate the coulometer using traceable electrical standards and Ohm's law;

— use the measured value of the electrical calibration factor and the Faraday constant to convert the integrator output to coulombs and then to moles of plutonium measured by the coulometer;

— calculate the mass of plutonium by multiplying the moles of plutonium determined by controlled-potential coulometry times a molar mass of plutonium determined by other means, such as thermal ionization mass spectrometry, magnetic sector inductively coupled plasma mass spectrometry, or process knowledge.

— use quality-control standards with traceable plutonium quantity values 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, acid-type and molarity of the supporting electrolyte, and the presence of any complexing agents in the electrolyte.

These steps ensure that test samples are taken from reproducible and stable sample solutions and prepared for measurement. The test samples are measured using a protocol based upon first principles and a traceable, electrical calibration of the coulometer. Further details are provided in Clauses 10 and 11.

5 Reagents

Use only analytical grade reagents.

All aqueous solutions shall be prepared with double-distilled or distilled, demineralized water with a resistivity greater than 10 MΩ·cm, i.e. ISO 3696[3] Grade 1 purified water.

5.1 Nitric acid solution, $c(HNO_3) = 0{,}9$ mol/l.

NOTE Refer to 11.4 for alternative electrolyte options.

5.2 Amidosulfuric acid solution, $c(NH_2HSO_3) = 1{,}5$ mol/l.

5.3 Sulfuric acid solution, $c(H_2SO_4) = 3$ mol/l.

NOTE The concentration of the sulfuric acid solution used to fume the plutonium test samples is not a critical parameter, provided the sulfate ion concentration remains in large excess (above 50) compared to the plutonium ion in order to avoid the formation of colloidal Pu complexes.

5.4 Pure argon or nitrogen, (O_2 amount of substance fraction less than 10 µmol/mol).

5.5 Pure air (optional reagent), free of organic contaminants.

6 Apparatus

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

6.1 Analytical balance, installed in radiological containment unit and shall be capable of weighing a mass of 1 g, with a standard uncertainty of ±0,1 mg, $k = 1$. This represents a relative standard uncertainty of 0,01 %.

— Weighing less than 1 g will increase the relative uncertainty to >0,01 %, in an inversely proportional manner.

— If the uncertainty of the balance, as installed, does not meet the criterion of ±0,1 mg, then test samples greater than 1 g should be used.