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Nuclear fuel technology — Controlled-potential coulometric measurement of plutonium

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

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Foreword

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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 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.

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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;

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