

---

---

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

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



**PDF disclaimer**

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

This document is a preview generated by EVS

© ISO 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.org](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

|                                                                     |    |
|---------------------------------------------------------------------|----|
| Foreword.....                                                       | iv |
| 1 Scope.....                                                        | 1  |
| 2 Normative references .....                                        | 1  |
| 3 Principle.....                                                    | 1  |
| 4 Reagents.....                                                     | 2  |
| 5 Apparatus.....                                                    | 2  |
| 6 Procedure.....                                                    | 7  |
| 6.1 Plutonium determination.....                                    | 7  |
| 6.2 Analysis of subsequent samples .....                            | 12 |
| 7 Expression of results.....                                        | 12 |
| 7.1 Calculation of the electrical calibration factor.....           | 12 |
| 7.2 Calculation of the blank.....                                   | 12 |
| 7.3 Fraction of electrolysed plutonium.....                         | 13 |
| 7.4 Plutonium content.....                                          | 14 |
| 7.5 Quality control.....                                            | 14 |
| 8 Characteristics of the method .....                               | 14 |
| 8.1 Repeatability.....                                              | 14 |
| 8.2 Confidence interval.....                                        | 14 |
| 8.3 Analysis time .....                                             | 15 |
| 9 Interferences.....                                                | 15 |
| 10 Procedure variations and optimisation .....                      | 17 |
| 10.1 Accountability measurements.....                               | 17 |
| 10.2 Process control measurements .....                             | 18 |
| 10.3 Measurement cell design .....                                  | 18 |
| 10.4 Electrolyte and electrode options .....                        | 18 |
| 10.5 Test sample size.....                                          | 19 |
| 10.6 Background current corrections .....                           | 19 |
| 10.7 Correction for iron .....                                      | 19 |
| 10.8 Control-potential adjustment.....                              | 20 |
| Annex A (normative) Purification by anion-exchange separation ..... | 21 |
| Annex B (normative) Determination of Formal Potential $E_0$ .....   | 23 |
| Bibliography .....                                                  | 24 |

## 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.

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 12183 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

This second edition cancels and replaces the first edition (ISO 12183:1995), which has been technically revised.

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

## 1 Scope

This International Standard specifies an analytical method for the electrochemical assay of pure plutonium nitrate solutions of nuclear grade, with a total uncertainty of 0,1 % to 0,2 % at the confidence level of 0,95 for a single determination. 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, must be demonstrated by the user as having adequate reliability for their specific application.

Preliminary purification by anion exchange is required to remove interfering substances when present in significant amounts. Purification is also appropriate in situations where the purity of the sample is unknown or when it may unpredictably fluctuate in the manufacturing process. Refer to Clause 9 for a discussion of interferences and iron corrections.

Clause 10 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 International Standard.

## 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 10980:1995, *Validation of the strength of reference solutions used for measuring concentrations*

## 3 Principle

The method consists of the following steps:

- test samples are prepared by weight and fumed with sulfuric acid to achieve a consistent and stable chemical form that is free from chloride, fluoride, nitrate, nitrite, hydroxylamine, and volatile organic compounds;
- when appropriate, preliminary purification by anion exchange with fuming in sulfuric acid to restore the dry plutonium sulfate chemical form in preparation for measurement;
- measuring a blank of the nitric-acid supporting electrolyte and performing an appropriate calculation to determine the background current correction applicable to the electrolysis of the test sample from charging, faradic, and residual current; [1]
- dissolution of the residue in the previously measured supporting electrolyte (the blank);