INTERNATIONAL STANDARD

ISO 7097-2

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Nuclear fuel technology — Determination of uranium in solutions, uranium hexafluoride and solids —

Part 2: Iron(II) reduction/cerium(IV) oxidation titrimetric method

Technologie du combustible nucléaire — Dosage de l'uranium dans des solutions, l'hexafluorure d'uranium et des solides —

Partie 2: Méthode titrimétrique par réduction au fer(II) et oxydation au cérium(IV)

Reference number
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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
Web www.iso.org

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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.

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 7097-2 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

This first edition of ISO 7097-2, together with ISO 7097-1:2004, cancels and replaces ISO 7097:1983, which has been technically revised, and ISO 9989:1996.

ISO 7097 consists of the following parts, under the general title: *Nuclear fuel technology — Determination of uranium in solutions, uranium hexafluoride and solids*:

Part 1: Iron(II) reduction/potassium dichromate oxidation titrimetric method

Part 2: Iron(II) reduction/cerium(IV) oxidation titrimetric method

Introduction

This part of ISO 7097 describes procedures for determination of uranium in solutions, uranium hexafluoride and solids. The procedures described in the two independent parts of this International Standard are similar: this part uses a titration with cerium(IV) and ISO 7097-1 uses a titration with potassium dichromate.

Nuclear fuel technology — Determination of uranium in solutions, uranium hexafluoride and solids —

Part 2: Iron(II) reduction/cerium(IV) oxidation titrimetric method

1 Scope

This part of ISO 7097 describes an analytical method for the determination of uranium in pure product material samples such as U metal, UO_2, UO_3, uranyl nitrate hexahydrate, uranium hexafluoride and U_3O_8 from the nuclear fuel cycle. This procedure is sufficiently accurate and precise to be used for nuclear materials accountability.

This method does not generate a toxic mixed waste as does the potassium dichromate titration. The method may not be applied to scrap or waste samples until such time as it is qualified by obtaining results statistically equivalent to those obtained by the potassium dichromate method on the same sample types.

The method recommends that an aliquot of sample is weighed and that a mass titration is used, in order to obtain improved precision and accuracy. This does not preclude the use of any alternative techniques which could give equivalent performance. As the performance of some steps of the method is critical, the use of some automatic device has some advantages, mainly in the case of routine analysis.

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 9894, *Subsampling of uranium hexafluoride in the liquid phase*

ISO 10980, *Validation of the strength of reference solutions used for measuring concentrations*

3 Principle

An aliquot of the sample containing about 40 mg to 60 mg of uranium in sulfuric acid solution is taken for the titration. Amidosulfuric acid is added to eliminate nitrites or nitrogen oxides (NO_x) formed in subsequent stages. An excess of iron(II) sulfate solution is then added to reduce all the uranium to the quadrivalent state in concentrated phosphoric acid solution. The excess of iron(II) is oxidized by nitric acid, catalyzed by molybdenum, in a time- and temperature-controlled operation. The uranium(IV) is determined by mass titration with standardized cerium(IV) sulfate solution to a potentiometric end point; see References[1, 2, 3, 4]. To improve precision, the titration is performed in the presence of vanadium(IV), which increases the kinetic reaction. The addition of the V(IV) solution also acts to dilute the sample solution and shift the redox potential so as to allow the titration to proceed.

The ceric sulfate solution is calibrated with an internationally recognized uranium reference material, as described in 5.15.