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Nuclear technology — Nuclear fuels — Procedures for the measurement of elemental impurities in uranium- and plutonium-based materials by inductively coupled plasma mass spectrometry

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee NCE/9, Nuclear fuel cycle technology.

A list of organizations represented on this committee can be obtained on request to its secretary.

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**Nuclear technology — Nuclear fuels —
Procedures for the measurement of
elemental impurities in uranium- and
plutonium-based materials by inductively
coupled plasma mass spectrometry**

*Technologie nucléaire — Combustibles nucléaires — Modes
opératoires pour le mesurage des impuretés élémentaires des
matériaux à base d'uranium et de plutonium par spectrométrie de
masse avec plasma à couplage inductif*



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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 26062 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel cycle*.

Introduction

The technique presented in this International Standard is capable of being used to perform quantitative measurements of elements with the specific exceptions of H, He, C, N, O, F, Cl, Br, and the noble gases. The measurement of other elements such as Si, P, S, K and I will require specialized sample introduction options, specialized solutions, mass analysers or in-line systems for the measurement.

This International Standard is presented in general terms for actinide samples because of the complexity and variation of the ICP-MS technique and its sample introduction accessories. Sufficient and appropriate method development should be undertaken to ensure that the procedure used for the determination of impurities in uranium and plutonium matrices is validated.

It is assumed that the user of this International Standard has a basic understanding of standard sample control, chemical extraction techniques and operation of ICP-MS instrumentation. Blank control is critical when undertaking ICP-MS analysis. Assessment of reagents and standards may be required when undertaking ultra trace analysis.

Nuclear technology — Nuclear fuels — Procedures for the measurement of elemental impurities in uranium- and plutonium-based materials by inductively coupled plasma mass spectrometry

1 Scope

This International Standard specifies a procedure for the determination of trace impurities in uranium- or plutonium-based, or mixed uranium- and plutonium-based, materials by inductively coupled plasma mass spectrometry (ICP-MS). It provides both guidelines and specific options for the determination of an element or group of elements.

It is applicable to solutions such as uranyl or plutonium nitrate, solids such as the oxides and to mixed actinide materials such as unirradiated mixed oxide material in either solid or dissolved forms. It is not directly suitable for the analysis of uranium or plutonium matrices containing significant quantities of other elements such as uranium–gadolinium mixtures. It may nevertheless form the basis of a process for analysing this type of matrix, provided that the impact of the gadolinium component is ascertained.

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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

The method is based upon the dissolution of solid samples, the use of an optional chemical treatment to remove the actinide matrix, followed by the spectroscopic determination of the analytes using inductively coupled plasma mass spectrometry (ICP-MS).

In principle, the ICP-MS technique relies on the introduction of the sample in an aerosol form into the inductively coupled plasma. In the plasma, the sample atoms are ionized then extracted from the plasma through a series of differential pumping chambers into the analyser region. The ionized species are mass separated using a mass analyser and then measured using an appropriate detection system. Quantitative measurements can be performed using a number of techniques, including standard addition and isotope dilution analysis; however, the generation of calibration graphs from measured standards is the conventional technique.

To provide an indication of the application of the technique, Table A.1 lists the elements commonly measured in uranium, plutonium and mixed actinide materials. The table includes the optimum isotope to be measured, the mass analyser options and comments regarding the measurement of that element.

The process is presented in a series of stages, as given in the following list. For each stage a series of criteria or principles that require consideration is presented. Annex B gives summaries of specific procedures for the dissolution and optional chemical extraction stages.

a) Dissolution to dissolve solid samples:

general details provided for hot plate and microwave oven based dissolution;

b) Optional chemical extraction procedure to remove the sample matrix:

general details provided for solvent extraction process and chromatography process;

c) Direct analysis of samples in the actinide matrix:

general principles to be considered if using this option;

d) Analysis of the extracted sample:

general principles of calibration, quality control and sample measurement are provided.

No details are provided for the specific instrumental set up, owing to the significant range of options available from the variety of instruments currently available commercially, for example,

- quadrupole mass analyser vs. magnetic sector mass analyser vs. time-of-flight mass analyser,
- conventional nebulisation sample introduction vs. specialised applications e.g. flow injection or ultra sonic nebuliser,
- the use of “collision/reaction cell” or “cool plasma” systems,
- quantitative techniques.

A brief description of the relative merits of these options is provided in Annex C. However, detailed information should be obtained from textbooks, journals or from manufacturers' manuals.

4 Reagents and materials

Use only reagents of recognized analytical grade, unless otherwise specified.

4.1 Water

Normal industry practice is to use high purity water designated 18 M Ω -cm, judged as being suitable for this guideline. Water complying with better than grade 2 as defined in ISO 3696:1987 is the recommended water quality.

4.2 Acids and other reagents

High purity reagents shall be used for all ICP-MS analysis. The quality of the reagents used in the process shall be validated by undertaking suitable blank measurements before use. Procedural blanks should be used at the sample preparation, sample extraction (where applicable) and instrument measurement stages.

4.2.1 Nitric acid, 16 M

4.2.2 Hydrofluoric acid, 40 %