

Nuclear fuel technology - Determination of the isotopic and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions by thermal-ionization mass spectrometry (ISO 8299:2019)

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

See Eesti standard EVS-EN ISO 8299:2021 sisaldab Euroopa standardi EN ISO 8299:2021 ingliskeelset teksti.	This Estonian standard EVS-EN ISO 8299:2021 consists of the English text of the European standard EN ISO 8299:2021.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation and Accreditation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 10.02.2021.	Date of Availability of the European standard is 10.02.2021.
Standard on kättesaadav Eesti Standardimis-ja Akrediteerimiskeskusest.	The standard is available from the Estonian Centre for Standardisation and Accreditation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile standardiosakond@evs.ee.

ICS 27.120.30

Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardimis- ja Akrediteerimiskeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonsesse süsteemi või edastamine ükskõik millises vormis või millisel teel ilma Eesti Standardimis- ja Akrediteerimiskeskuse kirjaliku loata on keelatud.

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardimis- ja Akrediteerimiskeskusega: Koduleht www.evs.ee; telefon 605 5050; e-post info@evs.ee

The right to reproduce and distribute standards belongs to the Estonian Centre for Standardisation and Accreditation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without a written permission from the Estonian Centre for Standardisation and Accreditation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation and Accreditation:

Homepage www.evs.ee; phone +372 605 5050; e-mail info@evs.ee

ICS 27.120.30

English Version

Nuclear fuel technology - Determination of the isotopic and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions by thermal-ionization mass spectrometry (ISO 8299:2019)

Technologie du combustible nucléaire - Détermination de la teneur isotopique et des concentrations en matériaux nucléaires de l'uranium et du plutonium dans une solution d'acide nitrique par spectrométrie de masse à thermoionisation (ISO 8299:2019)

This European Standard was approved by CEN on 18 January 2021.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

European foreword

The text of ISO 8299:2019 has been prepared by Technical Committee ISO/TC 85 "Nuclear energy, nuclear technologies, and radiological protection" of the International Organization for Standardization (ISO) and has been taken over as EN ISO 8299:2021 by 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 August 2021, and conflicting national standards shall be withdrawn at the latest by August 2021.

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.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 8299:2019 has been approved by CEN as EN ISO 8299:2021 without any modification.

Contents

	Page
Foreword.....	v
1 Scope.....	1
2 Normative references.....	1
3 Terms and definitions.....	1
4 Principle.....	1
5 Reference materials and reagents.....	2
5.1 Spikes and reference materials.....	2
5.2 Other chemical reagents.....	3
5.3 Resin, applicable for separation/purification of Pu and U.....	4
5.3.1 General.....	4
5.3.2 Preparation of resin.....	4
6 Apparatus.....	5
7 Apparatus for mass spectrometry.....	6
8 Sample preparation.....	6
8.1 Subsampling and spiking.....	6
8.1.1 Pellet or powder samples.....	7
8.1.2 Concentrated nuclear fuel solution samples (such as reprocessing solution).....	7
8.1.3 Plutonium nitrate solution samples (such as product solution from a reprocessing plant).....	7
8.1.4 Dried nitrate samples.....	8
8.2 Chemical valency adjustment.....	8
8.2.1 Valence adjustment with ferrous solution.....	8
8.2.2 Valence adjustment with hydrogen peroxide.....	8
8.3 Sample separation/purification.....	9
8.3.1 Ion exchange with anion-exchange resin.....	9
8.3.2 Purification with extraction separation resins (see 5.3.1.2).....	10
8.4 Replicate treatments.....	10
9 Filaments preparation.....	10
9.1 Degassing of filaments.....	10
9.2 Sample loading.....	10
9.2.1 Normal sample loading.....	10
9.2.2 Graphite loading technique.....	10
9.2.3 Resin-bead loading on single filaments for Pu samples.....	11
9.3 Filament mounting (filament assemblies preparation).....	11
10 Instrument calibration.....	11
10.1 Mass calibration.....	11
10.2 Gain calibration for Faraday multi-detectors.....	11
10.3 Faraday detector calibration.....	11
10.4 Mass discrimination calibration.....	12
11 Isotopic mass spectrometric measurements.....	12
11.1 Total evaporation measurements using a single or double filament assembly and a multi-Faraday collector system.....	12
11.2 Bias correction method using a double filament assembly and a multi-Faraday collector system.....	13
12 Calculation of the results.....	13
12.1 Calculation of ion current intensities.....	13
12.2 Calculation of mean, weighted mean and standard deviation on a set of ratios x_i ($i = 1 \dots N$).....	14
12.3 Mass discrimination correction.....	14
12.4 Calculation of the atomic percent abundance A_i	14

12.5	Calculation of the isotopic mass percent W_j	15
12.6	Calculation of concentration	15
12.7	Isotope decay correction	16
13	Blanks	16
14	Quality control	16
15	Measurement uncertainty	17
15.1	Elemental assay	17
15.2	Isotopic analysis	17
16	Interferences	18
Annex A (normative) Preparation and standardization of spike solutions		19
Bibliography		25

This document is a preview generated by EVS

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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

This third edition cancels and replaces the second edition (ISO 8299:2005), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the procedure for the preparation of resin used for separation and purification of the samples has been added in [5.3](#);
- sample preparation procedure from pellet, powder and other material forms to the solution has been added in [8.1](#);
- uncertainty of the measurement is considered in [Clause 15](#) instead of repeatability and accuracy.

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 — Determination of the isotopic and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions by thermal-ionization mass spectrometry

1 Scope

This document specifies a method for the determination of the isotopic and elemental uranium and plutonium concentrations of nuclear materials in nitric acid solutions by thermal-ionization mass spectrometry.

The method applies to uranium and plutonium isotope composition and concentration measurement of irradiated Magnox and light water reactor fuels (boiling water reactor or pressurized water reactor), in final products at spent-fuel reprocessing plants, and in feed and products of MOX and uranium fuel fabrication. The method is applicable to other fuels, but the chemical separation and spike solution are, if necessary, adapted to suit each type of fuel.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Validation of the strength of reference solutions used for measuring concentrations*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

The described method is based on isotope ratio measurements by thermal ionization mass spectrometry (TIMS). TIMS analysis requires isotope separation of different elements that have the same or similar masses as an isotope of the element being measured, such as ^{238}U and ^{241}Am influences ^{238}Pu and ^{241}Pu . Separation method for Pu and U using columns purifications are described in [Clause 8](#). Other separation methods may be used provided that they lead to a separation of similar quality. Column extraction chromatography described in ISO 15366 (all parts) is an example of a suitable alternative.

The described method consists of two separate TIMS measurements:

a) Isotopic measurement

One measurement is made to determine the isotopic composition of the element in the sample. The ^{238}Pu isotope abundance is determined by combining mass spectrometry following the present method and alpha spectrometry as described in ISO 11483, if the interference of the isobar ^{238}U is not eliminated by chemical separation.