International Standard

ISO 16795

Second edition

Nuclear Energy — Determination of Gd_2O_3 content in pellets containing uranium oxide by X-ray fluorescence spectrometry

Énergie nucléaire — Détermination de la teneur de Gd_2O_3 par spectrométrie à fluorescence X dans des pastilles combustibles contenant de l'oxyde d'uranium

PROOF/ÉPREUVE

Reference number
ISO 16795:2024(en)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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

This second edition cancels and replaces the first edition (ISO 16795:2004), which has been technically revised.

The main changes are as follows:

— the title of this document has been modified;

— requirements for the standard pellet has been added in Clause 7;

— range of Gd_2O_3 content covered by calibration curve has been added in Clause 10;

A list of all parts in the ISO 16795 series can be found on the ISO website.

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 Energy — Determination of Gd_2O_3 content in pellets containing uranium oxide by X-ray fluorescence spectrometry

1 Scope

This document specifies a method which covers the determination of Gd_2O_3 content in UO_2 fuel pellets, by X-ray fluorescence spectrometry.

Either wave dispersion X-ray fluorescence (WD-XRF) or energy dispersion X-ray fluorescence (ED-XRF) is applicable, however, this document states a method by using WD-XRF using Gd Lα-line.

This method has been tested for mass fractions of from 2 % to 10 % Gd_2O_3.

2 Normative references

ISO 17034, *General requirements for the competence of reference material producers*

ASTM C1128, *Standard Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials*

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 fuel pellets are polished before X-ray examination.

After excitation by the primary X-ray beam, the sample emits characteristic radiation from all of its components.

The appropriate 2θ angle (2-theta) for gadolinium is selected, for example 61,115 (2θ in degrees).

The digitized signal intensity (in terms of counts) of the selected L X-ray line is proportional to the concentration of gadolinium in the sample.

The process is fully automatic.

5 Apparatus

5.1 Sequential X-ray spectrometer, including the following:

5.1.1 Compact microprocessor-controlled spectrometer.

5.1.2 Precision-engineered goniometer.

5.1.3 Sample holder.

5.1.4 High-efficiency X-ray generator available to generate approximately 3 kW.

5.1.5 Accurate internal temperature control.

5.1.6 Analyser crystal (LiF 200).

5.1.7 Scintillation and flow detectors.

5.1.8 Multi-channel analyser.

5.2 Pellet press.

5.3 Analytical balance, sensitivity ±0,1 mg.

5.4 Powder blender and/or shaker.

5.5 Sintering furnace, able to reach temperatures of about 1 800 °C**.**

6 Reagents

6.1 Uranium dioxide, nuclear grade as specified in ASTM C1128.

6.2 Gadolinium oxide Gd_2O_3, with a purity of 99,99 % in mass fraction.

7 Preparation of standards

Standard pellets are required to obtain calibration curve (see Clause 5), required for data evaluation in the program. Standards shall be prepared using specifically designated equipment.

Standards are prepared as sintered pellets of (U, Gd) O_2 with mass fractions of Gd_2O_3 from 2 % to 10 %.

The standards shall be fabricated under laboratory-controlled conditions by blending UO_2 standard powder (6.1) with Gd_2O_3 standard powder (6.2) – both powders dried at 110 °C for 2 h in desired proportions before blending.

Standard powders of UO_2 (6.1) and Gd_2O_3 (6.2) are available commercially, or working standard materials, prepared and characterized its purity at each laboratory are also applicable. Reference value of standard powder and pellet can be calculated from the certificate or characterized value. Standard materials shall be metrologically traceable in accordance with ISO 17034 (if obtained commercially) or with ASTM C1128 (in the case of working standard materials). The acceptable maximum uncertainty of the standard powder and pellets depends on the specification required for the fuels to be fabricated.

The powders shall be weighed on an analytical balance to the nearest 0,1 mg. The blending will be accomplished by combining the Gd_2O_3 and UO_2 powders, shaking the contents for at least 4 h (or the time necessary to ensure the homogeneity of the blend).

After blending, the powders are pressed into pellets. Extra care shall be taken to clean up the press before pressing the standard pellets. The press is operated in the manual mode, and the first set of pressed pellets for each Gd_2O_3 weight per cent is discarded. The size of the standard pellet should be the same as the fuel pellet to be measured for Gd_2O_3 content.