
**Nuclear energy — Determination of
Gd₂O₃ content of gadolinium fuel pellets
by X-ray fluorescence spectrometry**

*Énergie nucléaire — Dosage de Gd₂O₃ dans des pastilles combustibles
au gadolinium par spectrométrie à fluorescence X*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 16795:2004](https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004)

<https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004>



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.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 16795:2004

<https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004>

© ISO 2004

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
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope.....	1
2 Principle	1
3 Apparatus.....	1
4 Reagents	2
5 Standards.....	2
6 Polishing	2
7 Equipment calibration	2
8 Calibration curve.....	3
9 Measurements	3
10 Precision and accuracy	3
11 Test report.....	4
Annex A (informative) Angle calibration and background correction	5
Annex B (informative) Calibration curve	6
Annex C (informative) Precision and accuracy	7

<https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004>
 ISO 16795:2004

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 16795 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO 16795:2004](https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004)

<https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004>

Nuclear energy — Determination of Gd₂O₃ content of gadolinium fuel pellets by X-ray fluorescence spectrometry

1 Scope

This method covers the determination of Gd₂O₃ in sintered fuel pellets, by X-ray fluorescence spectrometry using the Gd L α -line.

The fuel pellets are polished before X-ray examination.

This method has been tested for mass fractions of from 2 % to 10 % Gd₂O₃.

2 Principle

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

The appropriate line for gadolinium is selected, for example L α ₁ = 6,056 keV.

The beam goes to the detector producing a quantity of counts proportional to the concentration of gadolinium in the sample.

The process is fully automatic.

3 Apparatus

[ISO 16795:2004](https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004)

<https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004>

3.1 Sequential X-Ray spectrometer, with the following:

3.1.1 Compact microprocessor-controlled spectrometer.

3.1.2 Precision-engineered goniometer.

3.1.3 High-efficiency 3 kW generator.

3.1.4 Accurate internal temperature control.

3.1.5 Analyser crystal (LiF 200).

3.1.6 Scintillation and flow detectors.

3.2 Press.

3.3 Analytical balance, sensitivity $\pm 0,1$ mg.

3.4 Powder blender and/or shaker.

3.5 Sintering furnace.

3.6 Muffle furnace.

4 Reagents

4.1 Uranium dioxide, nuclear grade.

4.2 Gadolinium oxide Gd_2O_3 , quality 99,99 % by mass.

5 Standards

All preparations shall be carried out using segregated equipment.

Standards are prepared as sintered pellets of $Gd_2O_3 + UO_2$ with mass fractions of Gd_2O_3 from 2 % to 10 %.

The standards shall be fabricated under laboratory-controlled conditions by blending Gd_2O_3 powder with UO_2 .

The UO_2 and Gd_2O_3 powders to be used in the standard blends are previously dried at 110 °C for 2 h.

The powders shall be weighed on an analytical balance. 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 guarantee the homogeneity of the blend).

After blending, the powders are pressed into pellets. Extra care must 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.

Sintering of the pellet standards is performed under the same conditions as during production.

6 Polishing

Before the X ray examination, the face of the sintered standard or sample pellet that will be exposed shall be polished using an 800-grit SiC paper or similar. The polished sample is cleaned with a tissue prior to analysis.

7 Equipment calibration

Verification and setting of the measurement channels.

7.1 Angle calibration

Before performing angular calibration on any channel, ensure that the goniometer is calibrated. In practice, this is only required at the installation stage, or if the goniometer has been altered in any way.

A standard is positioned in the counting chamber by entering the appropriate command. The operating range and the conditions are checked and set prior to the calibration.

The "measure" option is selected to perform the calibration measurement and produce a graph. This graph shows a 2θ angle against the count rate. The original and new peak angles to be shown both graphically and numerically.

See Annex A.

7.2 Pulse-height distribution

A standard is exposed to the X-ray beam using the appropriate command.

A pulse-height distribution curve is produced by entering the appropriate command. The equipment is fully automatic.

The software performs the measurements and displays a graph of the results.

7.3 Background correction

Expose a standard to the X-ray beam.

A background correction is produced automatically by entering the appropriate command.

The software performs the measurements and displays a graph of the results.

7.4 Stability check

Stability shall be checked and compensated for the drift.

A sample high in Gd_2O_3 (monitor) is measured at least three times. If the maximum difference among the three readings is less than 1 % (or the recommended target), the signal obtained is compared with the reference value for the same sample, and the drift is automatically compensated for all the samples to be measured. If the difference is greater than 1 %, the equipment is not considered stable.

8 Calibration curve

Samples for analysis are compared with samples of a known Gd_2O_3 mass fraction.

The calibration curve is obtained by using the standard pellets prepared as in Clause 5.

In the calibration graph, the y axis represents the net count rate and the x axis the Gd_2O_3 mass fraction.

The calibration curve is saved by the software.

See Annex B.

9 Measurements

The sample is placed in the sample holder in the same way as the standard. In both cases, the area to be exposed must be the same. Do the measurement using the appropriate parameters. Typical parameters are showed in Table 1.

10 Precision and accuracy

Typical results are the following.

The root mean square (RMS) for the calibration curve was 0,014 %.

Standard pellets having a different Gd_2O_3 mass fraction were analysed 20 times each using the same calibration curve. The maximum coefficient of variation obtained was 0,984 %, and the minimum was 0,295 %. The maximum relative error was 0,443 %.

See Annex C.

Table 1 — Typical parameters

kv	40
Gd $L\alpha_1$ line	6,056 keV
mA	60
Filter	Out
Collimator	Fine
X-Crystal	LiF 200
Order	1
Angle (2θ)	61,115
Background correction	$2\theta \pm 1,20$
Detector	Flow
Window lower level energy	15 %
Window upper level energy	70 %
Counting time of the Gd peak	200 s
Spin	Yes

11 Test report

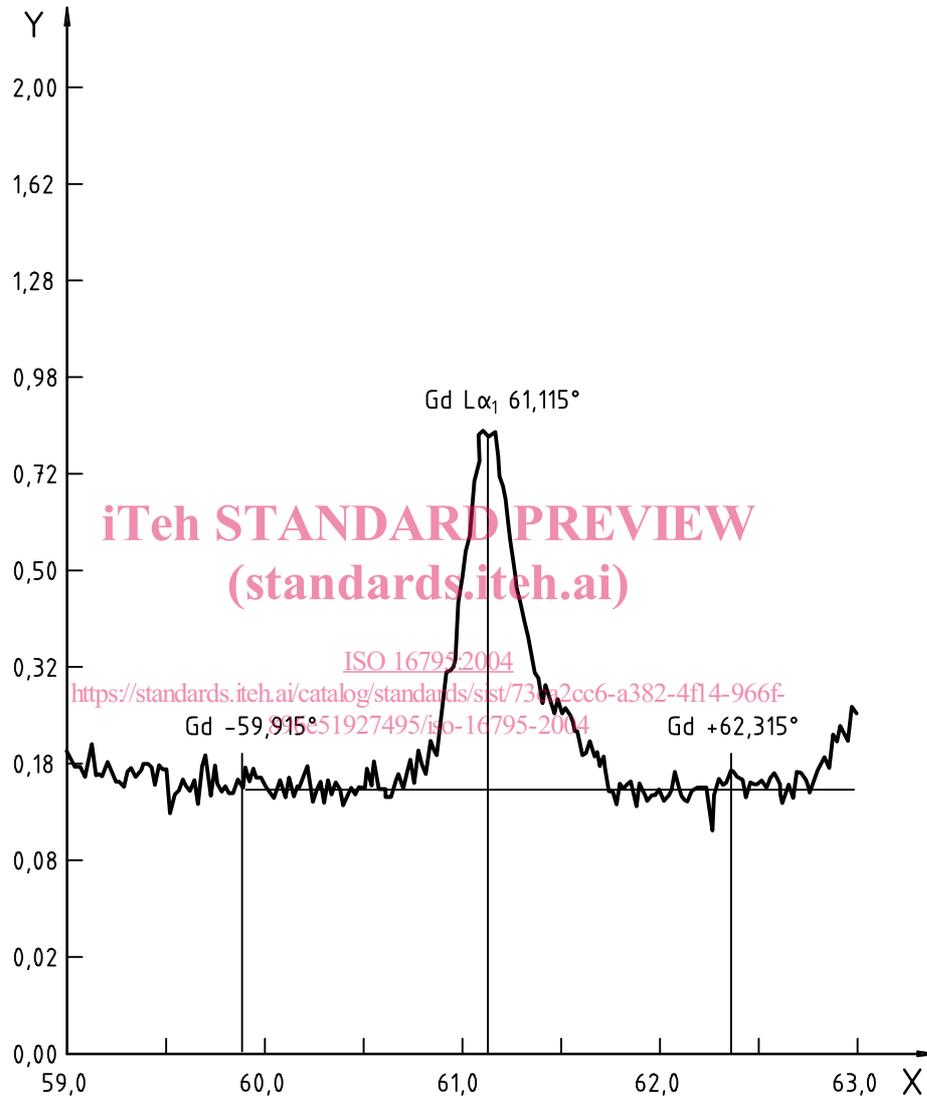
iTeh STANDARD PREVIEW
(standards.iteh.ai)

The test report shall include the following information:

- a) identification of the sample; [ISO 16795:2004](https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004)
- b) reference to the method used; <https://standards.iteh.ai/catalog/standards/sist/73ca2cc6-a382-4f14-966f-896e51927495/iso-16795-2004>
- c) results and their units;
- d) any unusual features noted during the test;
- e) any operations not included in this International Standard;
- f) indication of whether or not buoyancy corrections have been applied.

Annex A
(informative)

Angle calibration and background correction



Key

- X 2θ, in degrees
- Y kCSP