International
Standard

ISO 16795

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

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Foreword

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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).

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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.

Sintering of the standard pellet is performed under the same conditions as during production of the fuel pellet. Then Gd_2O_3 content of the standard pellet should be evaluated as $Gd_2O_3/(U, Gd)\ O_2$.

NOTE Specific procedure for preparation of standards differ between laboratories. Recommended to prepare by similar condition with fuel pellet preparation.

NOTE Fabrication operations, identification and traceability of standards can be documented.

8 Polishing

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

9 Equipment calibration

Verification and setting of the measurement channels shall be performed.

9.1 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 calibrated peak angles should be shown both graphically and numerically.

See Annex A.

9.2 Pulse-height distribution

A standard is exposed to the X-ray beam at the peak angle 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.

9.3 Background correction

Before measuring the sample pellet, background should be measured using standard pellet and used to correct results from fuel pellet.

Generally, background correction can be performed automatically using the instrument software.

9.4 Stability (sensitivity) check

Stability (sensitivity) of the spectrometer shall be checked and compensated for the drift.

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

10 Calibration curve

Samples for analysis are compared with samples of a known Gd_2O_3 mass fraction (standard pellet).

The linear calibration curve is obtained by using the standard pellets prepared as in Clause 5 to cover the range of Gd_2O_3 from 2 % to 10 %.

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 instrument software.

11 Measurements

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

If measurement results exceed the range of calibration curve, it is highly recommended to renew the calibration curve to cover the results.

12 Precision and accuracy

Typical results are the following.

The root mean square error (RMS) of the calibration curve prepared using standard pellets 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 0,98 %, and the minimum was 0,29 %. The maximum relative deviation was 0,44 %.

See Annex B.

Table 1 — Typical parameters

Voltage	40 kV
Gd Lα_1 line	6,056 keV
Current	60 mA
Filter	Out
Collimator	Fine
X-Crystal	LiF 200
Order	1
Angle (2θ)	61,115
Background correction	2θ ± 1,20
Detector	Flow
Window lower level energy	15 %
Window upper level energy	70 %
Counting time of the Gd peak	200 s
Spin	Yes

13 Test report

The test report shall include the following information:

a) identification of the sample;

b) a reference to this document, i.e. ISO 16795:2024;

c) reference to the method used;

d) results and their units;

e) any unusual features noted during the test;

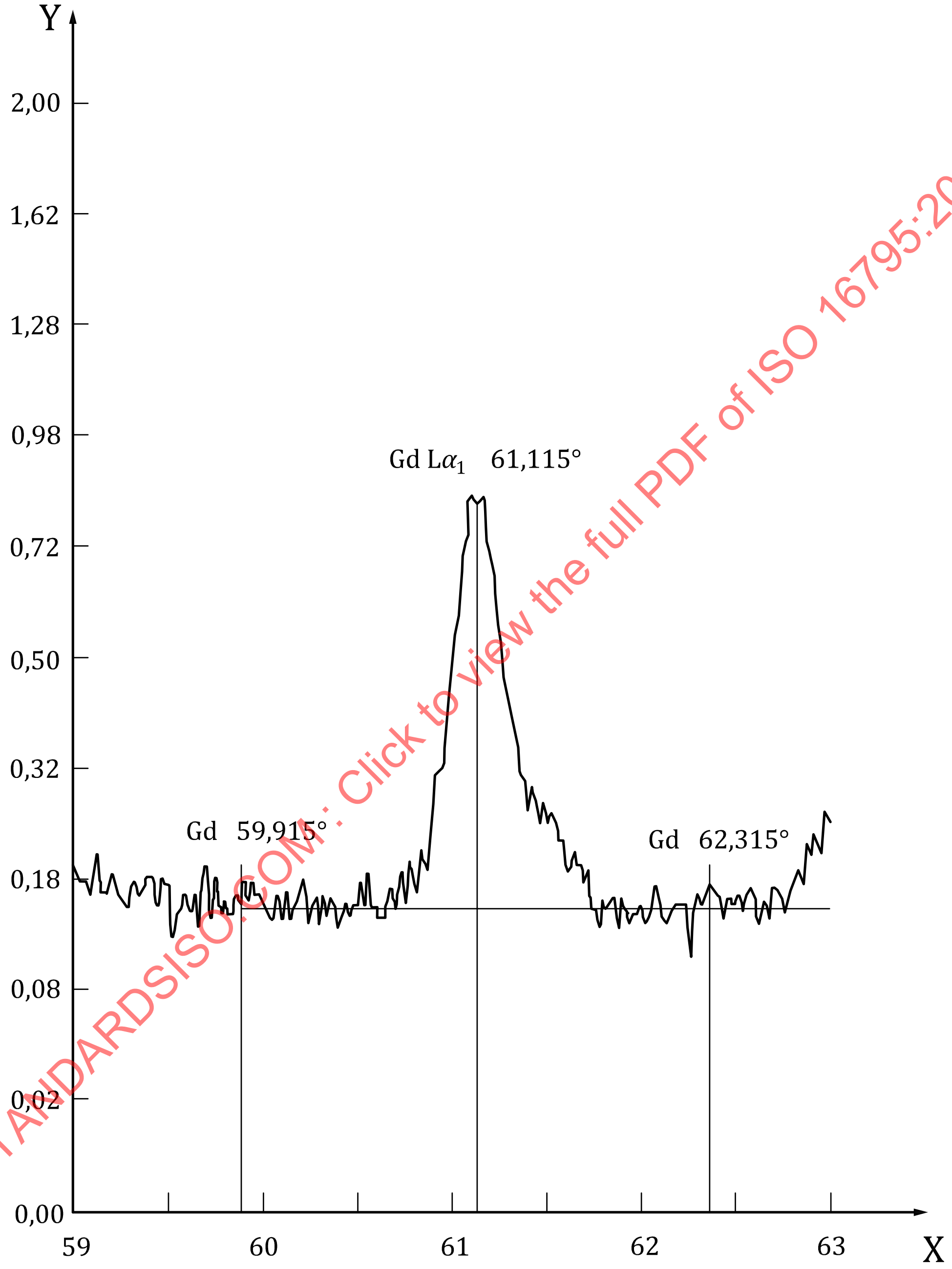
f) any operations not included in this document;

g) indication of whether or not buoyancy corrections have been applied;

h) reference of the standard used for calibration.

Annex A
(informative)

Angle calibration and background correction



Key

X 2θ, in degrees

Y intensity of X-ray [kCPS]

Figure A.1 — Angle calibration and background correction

Annex B
(informative)

Precision and accuracy

Table B.1 — Precision and accuracy

Gd_2O_3 mass fraction in standard, w, in g/g	2,021	3,927	5,884	6,826	9,981
Number of data	20	20	20	20	20
Average of measured values, $\bar{v}$	2,021	3,937	5,878	6,849	9,937
Standard deviation, σ	0,020	0,020	0,026	0,020	0,030
Coefficient of variation, C_V, in %	0,984	0,509	0,440	0,295	0,306
Relative deviation, in %	0,002	0,261	0,105	0,343	0,443

The coefficient of variation, expressed in percentage, has been calculated as follows by Formula (B.1):

$$C_v = \frac{\sigma}{\bar{v}} \times 100\,\% \qquad \text{(B.1)}$$

The relative deviation, expressed in percentage, has been calculated as follows by Formula (B.2):

$$U_{\text{rel}} = \frac{\bar{v} - w_{Gd_2O_3}}{\bar{v}} \times 100\,\% \qquad \text{(B.2)}$$