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Nuclear energy — Determination of Gd_2O_3 content of gadolinium fuel pellets by X-ray fluorescence spectrometry

Énergie nucléaire — Dosage de Gd_2O_3 *dans des pastilles combustibles au gadolinium par spectrométrie à fluorescence X*

Reference number
ISO 16795:2004(E)

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Contents

Foreword

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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.

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

INTERNATIONAL STANDARD **ISO 16795:2004(E)**

Nuclear energy — Determination of Gd_2O_3 content of gadolinium fuel pellets by X-ray fluorescence spectrometry

1 Scope

This method covers the determination of Gd_2O_3 in sintered fuel pellets, by X-ray fluorescence spectrometry using the Gd $L\alpha$-line.

The fuel pellets are polished before X-ray examination.

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

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 $L\alpha_1 = 6{,}056$ 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

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 ± 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 Lα_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

The test report shall include the following information:

a) identification of the sample;

b) reference to the method used;

c) results and their units;

d) any unusual features noted during the test;

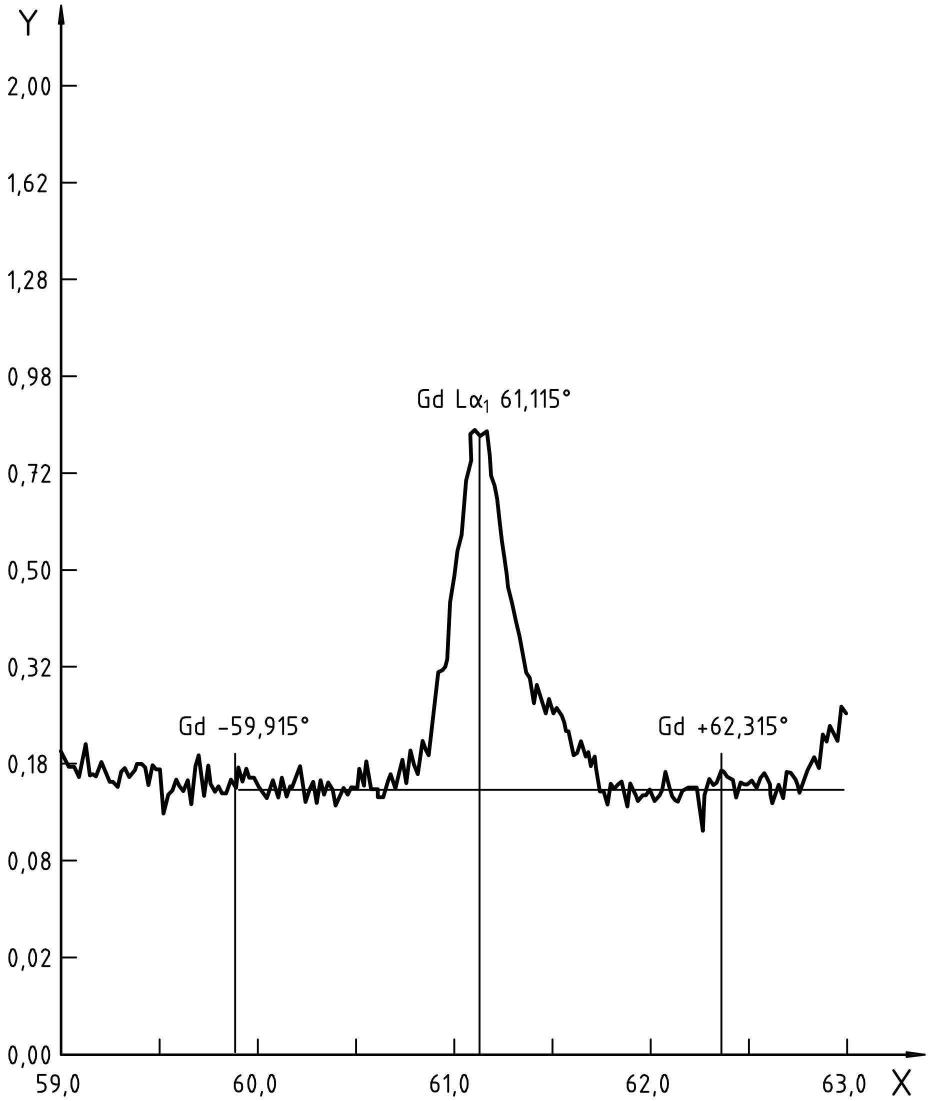
e) any operations not included in this International Standard;

f) indication of whether or not buoyancy corrections have been applied.

Annex A

(informative)

Angle calibration and background correction



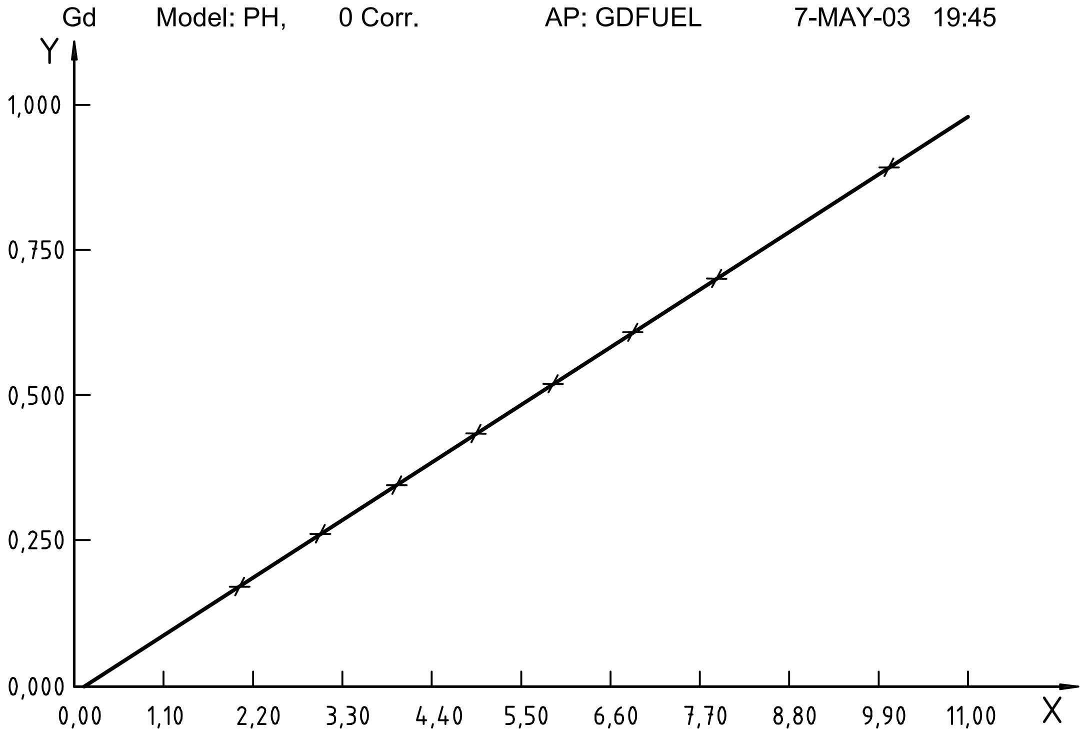
Key

X 2θ, in degrees

Y kCSP

Annex B
(informative)

Calibration curve



NOTE For the following conditions:

E = 11,065 75
D = 0,106 17
RMS = 0,01447
K_f = 0,006 95

Key

X ^{203} Gd mass fraction, in percent
Y R_{corr}

Annex C

(informative)

Precision and accuracy

Gd_2O_3 mass fraction in standard, %	2,021	3,927	5,884	6,826	9,981
Number of data	20	20	20	20	20
Average of measured values	2,021	3,937	5,878	6,849	9,937
Standard deviation	0,020	0,020	0,026	0,020	0,030
Coefficient of variation (%)	0,984	0,509	0,440	0,295	0,306
Relative error (%)	0,002	0,261	0,105	0,343	0,443

The coefficient of variation has been calculated as follows:

$$\text{Coefficient of variation} = \frac{\text{standard deviation} \times 100}{\text{average}}, \text{ expressed in percent.}$$

Relative error has been calculated as follows:

$$\text{Relative error} = \frac{\text{standard value} - \text{average} \times 100}{\text{standard value}}, \text{ expressed in percent.}$$

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