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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

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Contents

Page

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*.

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 Lα-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 Lα_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.