INTERNATIONAL STANDARD

ISO 16796

First edition
2004-08-15

Nuclear energy — Determination of Gd_2O_3 content in gadolinium fuel blends and gadolinium fuel pellets by atomic emission spectrometry using an inductively coupled plasma source (ICP-AES)

Énergie nucléaire — Dosage de Gd_2O_3 *dans des mélanges de poudres et dans des pastilles combustibles au gadolinium par spectrométrie par émission atomique à plasma à couplage inductif (ICP-AES)*

Reference number
ISO 16796:2004(E)

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.

© 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

Copyright International Organization for Standardization
Reproduced by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Contents

Page

Foreword iv
1 Scope 1
2 Principle 1
3 Apparatus 1
4 Reagents 2
5 Standard blends 2
6 Standard and sample preparation 3
7 Calibration and analysis of the samples 3
8 Precision and accuracy 4
9 Test report 4
Annex A (informative) Calibration curve 5
Annex B (informative) Standards data 6

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 16796 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

Nuclear energy — Determination of Gd_2O_3 content in gadolinium fuel blends and gadolinium fuel pellets by atomic emission spectrometry using an inductively coupled plasma source (ICP-AES)

1 Scope

This International Standard is applicable to the determination of Gd_2O_3 in powder blends and sintered pellets of $Gd_2O_3 + UO_2$ from 1 % to 10 %, by the ICP-AES method.

2 Principle

The sample is weighed and dissolved in nitric acid. The sample solutions are aspirated into an inductively coupled plasma using argon as a carrier. The emitted light from the sample in the plasma is dispersed, and the Gd line at 335,0 nm is measured by a spectrometer.

The intensity of the Gd line is proportional to the concentration of Gd present.

Impurity interferences have not been observed for the usual samples of the nuclear grade material.

3 Apparatus

3.1 High dispersion direct reading spectrometer with I.C.P. source

A typical value for resolution is 0,555 nm/mm in the first order.

3.2 Analytical balance; sensitivity ± 0,1 mg

3.3 Turbula mixer

3.4 Hot plate

3.5 Micropipettes

3.6 Volumetric flasks

3.7 Glass beakers

3.8 Percussion mortar

3.9 Muffle furnace

4 Reagents

4.1 Concentrated nitric acid (analytical reagent grade)

4.2 Deionized water

4.3 Nitric acid, 1:1

4.4 Uranium dioxide, nuclear grade

4.5 Gadolinium oxide, Gd_2O_3, minimum purity 99,99 %

5 Standard blends

Standard powder blends are prepared from the UO_2 and Gd_2O_3 high-purity materials in Clause 4. These standards will contain Gd_2O_3 in the range of 1 % to 10 %, depending on the required level.

5.1 Drying

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

5.2 Preparation

The appropriate amounts of Gd_2O_3 and UO_2 are weighed into different plastic vials to obtain the standard blends in the range from 1 % to 10 % Gd_2O_3. The mass of the UO_2 must be corrected for stoichiometry as follows:

$$gUO_{(2+x)} = gUO_2\left(1+\frac{xA_\mathrm{O}}{A_\mathrm{U}+2A_\mathrm{O}}\right)$$

where

$UO_{(2+x)}$ is the powder to be weighed;

UO_2 is the stoichiometric UO_2 powder in the blend;

A_U is the relative atomic mass of uranium;

A_O is the relative atomic mass of oxygen.

5.3 Blending

Each blend is mixed in the Turbula mixer for 4 h (or the time necessary to guarantee the homogeneity of the blend).

5.4 Identification

Each blend shall be identified and retained as an appropriate standard.

6 Standard and sample preparation

6.1 Standard preparation

6.1.1 Weigh 5,0 g of each standard into a beaker, weighed to nearest 0,000 1 g.

6.1.2 Add 25 ml of nitric acid (1:1).

6.1.3 Heat on the hot plate until the blend is completely dissolved and evaporate off the excess nitric acid (few minutes boiling).

6.1.4 Cool the solution and transfer quantitatively to a 100 ml volumetric flask.

6.1.5 Dilute to 100 ml with deionized water and mix the solution.

6.1.6 Pipette 1 ml of the prepared solution into a 100 ml volumetric flask.

6.1.7 Dilute to 100 ml with deionized water.

6.2 Preparation of the powder samples of Gd_2O_3 plus UO_2

6.2.1 Weigh 5,0 g of sample into a beaker, weighed to nearest 0,000 1 g.

6.2.2 Prepare the sample as described in 6.1.

6.3 Preparation of pellet samples

6.3.1 Crush the pellet.

6.3.2 Weight 5,0 g into a crucible, weighed to nearest 0,000 1 g.

6.3.3 Heat the crucible with the sample in a muffle furnace at 420 °C ± 25 °C minimum for 2 h to 3 h.

6.3.4 Quantitatively transfer the content from the crucible to a glass beaker. Then proceed as in 6.1.2.

7 Calibration and analysis of the samples

Standards as prepared in Clause 5 and 6.1 are used to calibrate the equipment.

7.1 Calibration

The I.C.P. spectrometer analytical conditions are developed by each laboratory.

The standard solutions are measured in duplicate and the calibration curve produced from the intensity-versus-concentration curve.

See example in Annex A.

7.2 Analysis of the samples

The samples prepared in 6.2 and 6.3 shall be analysed after making the calibration curve. To avoid daily correction and achieve the required precision, a calibration curve is carried out for each run of sample determinations. The background correction is not necessary.

Compare the sample emission intensity to the calibration curve and calculate the Gd_2O_3 concentration of the samples.

If the powder blend contents other products than UO_2 and Gd_2O_3, the calibration shall be done separately; the standards used for the calibration curve shall have the same matrix.

8 Precision and accuracy

Typical results are the following:

For 20 determinations of each of 6 standards from 2 % to 10 % Gd_2O_3, the maximum coefficient of variation was 0,991 % for 7 % Gd_2O_3, and the minimum was 0,323 % for 4 % Gd_2O_3.

Maximum relative error was 0,979 %.

See Annex B.

9 Test report

The test report shall include the following information:

a) identification of the sample;

b) the method used with reference to this International Standard;

c) the results and their units of measurement;

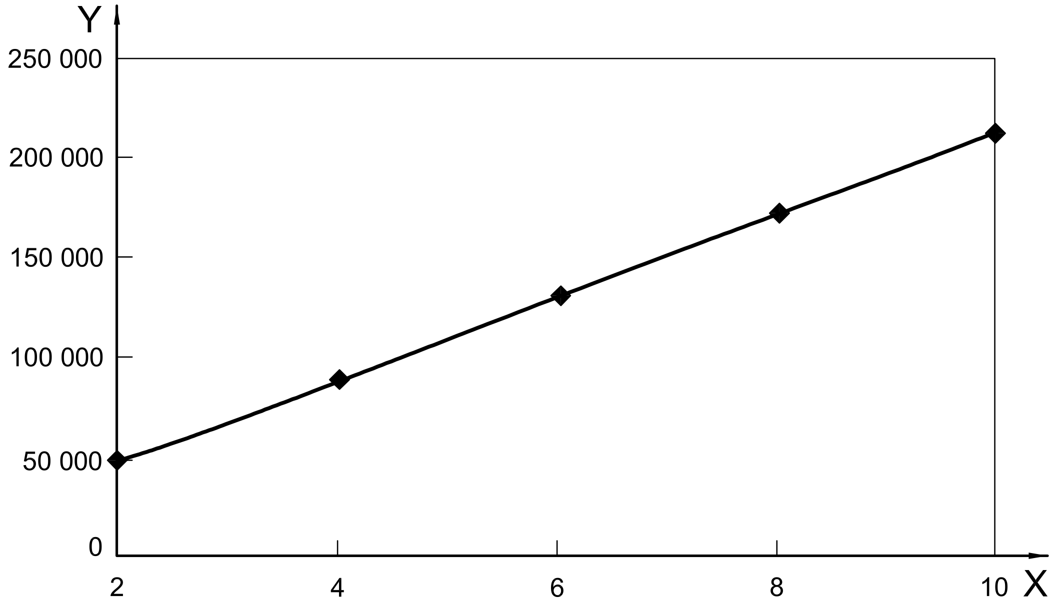
d) any unusual features noted during the test;

e) any operations not included in this International Standard;

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

Annex A
(informative)

Calibration curve



Key

X Gd_2O_3, % by mass

Y Intensity

NOTE To carry out the pattern curve, each standard is read twice.

Figure A.1

Table A.1

Data used to calculate the pattern curve		Regression results	
		Constant	–0,410 999 43
% Gd_2O_3	Intensity	Estimation error Y	0,057 089 23
2,007 5	48 780	R^2	0,999 757 19
4,014 6	89 691	Number of data	5
6,021 5	129 232	Freedom degrees	3
8,028 1	172 216	Slope	4,945 9E-05
10,034 3	210 333	Coefficient of error	4,450 1E-07

Annex B
(informative)

Standards data

Table B.1

Standard value % Gd_2O_3	2,007 5	4,014 6	6,021 5	7,024 8	8,028 1	10,034 3
Number of measurements	20	20	20	20	20	20
Average of measured value	2,002	4,025	5,981	7,044	8,107	9,992
Standard deviation	0,016	0,013	0,041	0,070	0,039	0,038
Coefficient of variation (%)	0,818	0,323	0,688	0,991	0,477	0,379
Relative error (%)	0,293	0,247	0,677	0,267	0,979	0,422

The coefficient of variation has been calculated as follows:

$$\text{Coefficient of variation (\%)} = \frac{\text{Std. Deviation} \times 100}{\text{Average}}$$

Relative error has been calculated as follows:

$$\text{Relative Error (\%)} = \frac{\left|\text{Std. Value-Average}\right| \times 100}{\text{Std.Value}}$$

ICS 27.120.30

Price based on 6 pages