INTERNATIONAL STANDARD

ISO 9278

Second edition
2008-12-01

Nuclear energy — Uranium dioxide pellets — Determination of density and volume fraction of open and closed porosity

Énergie nucléaire — Pastilles de dioxyde d'uranium — Détermination de la masse volumique et de la fraction volumique de pores ouverts et fermés

Reference number
ISO 9278:2008(E)

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Published in Switzerland

Foreword

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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 9278 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

This second edition cancels and replaces the first edition (ISO 9278:1992), which has been technically revised.

Nuclear energy — Uranium dioxide pellets — Determination of density and volume fraction of open and closed porosity

1 Scope

This International Standard specifies a method for determining the bulk density and the amount of open and closed porosity of sintered UO_2 pellets. The method can be applied to other materials, for example green pellets, and UO_2-PuO_2 or UO_2-Gd_2O_3 pellets.

2 Principle

The method is based on the determination of the pellet volume and the volume of open and closed pores by measurement of the dry mass, the saturated mass and the immersed mass of the samples. Alternative penetration immersion liquids and saturation conditions can be used, provided the samples can be completely impregnated during the procedure.

3 Apparatus

3.1 Balance, of adequate capacity, with an accuracy of 0,1 mg.

3.2 Oven, capable of maintaining a temperature of $(100\ ^{+5}_{0})$ °C. A vacuum drying oven is recommended for samples with a large amount of open pores.

3.3 Weighing device, to allow the test piece to be weighed in air and in the penetration immersion liquid, in order to make saturated mass and immersed mass measurements (see Table 1, step 1).

3.4 Container, a glass beaker or similar container of size and shape such that the sample, when suspended from the balance by the device, is completely immersed in the penetration immersion liquid, with the sample and the device for suspension being completely free from contact with any part of the container.

3.5 Vacuum impregnation apparatus, which may consist of glass components (see Table 1, step 2).

3.6 Test ball, made of any hard alloy or metal, e.g. carbide metal.

The radius, r, shall be between 5 mm and 10 mm, known with an accuracy of $\pm$ 0,5 μm, for the determination of the density of the penetration immersion liquid (see 4.1.4).

4 Procedure

SAFETY PRECAUTIONS — Standard precautions shall be observed when handling uranium dioxide and plutonium dioxide samples.

4.1 Ethanol impregnation method

Use ethanol of analytical grade (for possible modifications, see 4.2) for the impregnation method.

4.1.1 Determination of the dry mass (m_d)

Dry the pellets in the oven (3.2) at a temperature of 100 °C for at least 2 h. Cool to room temperature and weigh. The use of a desiccator, vacuum or a dry protective gas may be necessary during cooling, if the uptake of moisture from the environment does not allow a constant mass to be reached.

After the determination of the saturated mass and the immersed mass (4.1.5 and 4.1.6), dry the pellets again and weigh. Take the average, m_d, of the two measurements (see Table 1, steps 1 and 8).

4.1.2 Impregnation

For the impregnation of the UO_2 pellets with ethanol, use the vacuum impregnation apparatus (3.5) (see Table 1, step 2). Put the dry specimen in its container into the apparatus and proceed in the following way.

a) Turn on the pump with valves V1, V2, V3 and V4 closed. Open V4. Pump until a pressure of less than 10 Pa is achieved. Maintain pumping for 1 h.

b) Close V4 and carefully open V1. Allow 5 min for equilibration.

c) Close V1 and open V4. Pump until a pressure of 10 Pa is achieved.

d) Repeat sequence b).

e) Close V1, open V2 slowly to fill the container with liquid ethanol.

f) Turn off the pump. Open V3 to allow air into the apparatus.

4.1.3 Adjustment of weight controlling factors

Allow a period of 1 h to reach equilibrium (see Table 1, step 3).

a) The open pores are totally filled with ethanol under atmospheric pressure.

b) The liquid is brought up to room temperature.

4.1.4 Determination of the density of ethanol

The density of ethanol is determined by the use of a test ball (3.6) of known volume (see Table 1, steps 4 and 5).

Determine the mass of the test ball, m_1, in air.

Determine the reading on the balance, m_2, when the test ball is suspended from the balance by the device and is completely immersed in ethanol.

NOTE Density determination by means of a test ball is a useful working procedure because it is independent of the actual temperature and other factors influencing the density of the liquid, for example contamination. Alternative methods can also be used, for example recording the temperature of the ethanol, the density of which was determined as a function of temperature.

4.1.5 Determination of the saturated mass (m_s)

After the adjustment of weight controlling factors (see 4.1.3), blot the pellet and determine the saturated mass, m_s, by weighing in air (see Table 1, step 6).

A representative value of the saturated mass, m_s, can be obtained by the following procedure.

a) Blot the pellet by rolling it lightly on a lint-free linen or cotton cloth or paper tissue saturated with liquid ethanol in such a way that a thin film of ethanol remains on its surface.

b) Place the pellet on the device in air as shown in Table 1, step 6.

c) Read continuously or record the decrease in mass due to the evaporation of the surface film.

d) Determine the mass, where a sudden change of the evaporation rate can be observed.

4.1.6 Determination of immersed mass (m_i)

After determining the saturated mass, transfer the pellet immediately from the position in air to the position in the liquid on the suspension device (see Table 1, step 7).

Determine the immersed mass, m_i, by placing the pellet on the weighing device (3.3) that is suspended from one arm of the balance (3.1) (see Table 1, step 7).

Before actually weighing, counterbalance the scale with the device for suspension in place and immerse it in ethanol to the same depth as is used when the specimen is in place.

4.2 Modifications

The impregnation method can also be performed with other penetration immersion liquids, e.g. water, cyclohexane or toluene. The following conditions should be fulfilled.

a) There should be no chemical reaction with the test material.

b) Any contamination should be easily removable.

c) The liquid shall be easily removable from the open pores.

d) Surface tension forces on the device shall be negligible. If water is used as the penetration immersion liquid, the addition of an appropriate surfactant is recommended.

e) The liquid used shall be of analytical grade.

5 Calculation

5.1 Method of calculation

5.1.1 Calculation of the density of the penetration immersion liquid

The density of the penetration immersion liquid, ρ_1, in grams per cubic centimetre, is given by Equation (1):

$$\rho_1 = \frac{m_1 - m_2}{4\pi r^3 / 3} \tag{1}$$

where

r is the radius, in centimetres, of the test ball;

m_1 is the mass, in grams, of the test ball in air;

m_2 is the mass, in grams, of the test ball when it is immersed in the penetration immersion liquid.

5.1.2 Calculation of density and amount of open and closed porosity

The geometric pellet volume, V, in cubic centimetres, is given by Equation (2):

$$V = \frac{m_s - m_i}{\rho_1} \tag{2}$$

where

m_s is the saturated mass, in grams;

m_i is the immersed mass, in grams;

ρ_1 is the density of the penetration immersion liquid, determined in accordance with Equation (1).

Calculate the volumes of the open pores, V_{op}, and closed pores, V_{cp}, in cubic centimetres, using Equations (3) and (4):

$$V_{op} = \frac{m_s - m_d}{\rho_1} \tag{3}$$

$$V_{cp} = \frac{m_d - m_i}{\rho_1} - \frac{m_d}{\rho_{th}} \tag{4}$$

where

m_d is the dry mass, in grams;

ρ_{th} is the theoretical density, in grams per cubic centimetre, of the specimen (ρ_{th} = 10,96 for UO_2);

ρ_1,, m_s and m_i are defined in 5.1.1 and 5.1.2;

Calculate the bulk density of UO_2, ρ_{UO_2}, in grams per cubic centimetre, and the volume fractions of total pores, φ_{tot}, open pores, φ_{op}, and closed pores, φ_{cp}, in percentage, using Equations (5), (6), (7) and (8):

$$\rho_{UO_2} = \frac{m_d \times \rho_1}{m_d - m_i} \tag{5}$$

$$\varphi_{tot} = \left(1 - \frac{\rho_{UO_2}}{\rho_{th}}\right) \times 100 \tag{6}$$

$$\varphi_{op} = \left(\frac{m_s - m_d}{m_s - m_i}\right) \times 100 \tag{7}$$

$$\varphi_{cp} = \left(\frac{m_d - m_i}{m_s - m_i} - \frac{m_d}{m_s - m_i} \times \frac{\rho_1}{\rho_{th}}\right) \times 100 \tag{8}$$

The relative volume fraction of open pores, φ_{op}, of the total pores, φ_{tot}, in percentage, is given by Equation (9):

$$\frac{\varphi_{op}}{\varphi_{tot}} = \left(\frac{\dfrac{m_s - m_d}{m_s - m_i}}{1 - \dfrac{\rho_{UO_2}}{\rho_{th}}} \right) \times 100 \qquad (9)$$

6 Precision

The relative standard deviation of the bulk density is about ± 0,05 % and the absolute standard deviation of the volume fraction of open pores, φ_{op}, and the volume fraction of closed pores, φ_{cp}, is ± 0,03 %.

7 Test report

The test report shall include the following information:

a) reference to this International Standard (ISO 9278:2008);

b) all details necessary for identification of the test sample;

c) test method used;

d) method of drying the test sample;

e) penetration conditions;

f) penetration immersion liquid used;

g) results obtained;

h) all operations not specified in this International Standard;

i) details of any occurrence which may have affected the results.