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Uranium dioxide pellets — Determination of density and total porosity — Mercury displacement method

Pastilles de dioxyde d'uranium — Détermination de la masse volumique et de la porosité totale — Méthode de déplacement du mercure

Reference number
ISO 9279:1992(E)

Foreword

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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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Uranium dioxide pellets — Determination of density and total porosity — Mercury displacement method

1 Scope

This International Standard describes a method for determining the density and total porosity of sintered UO_2 pellets. The method can be applied to other bodies, for example green pellets, UO_2–PuO_2 and UO_2–Gd_2O_3 pellets, and also to irradiated material in hot cells. Fractured pieces of a pellet can also be tested. The mass of the specimen should not be less than about 1 g.

2 Principle

The method is based on the determination of the pellet volume by displacement of mercury which does not penetrate the open pores due to its surface tension. The density and the total porosity are determined by this volume and the mass of the pellet.

3 Apparatus

3.1 Mercury pycnometer, consisting of two chambers and a mercury collection flask. The flanges of these components of the glass apparatus are sealed with vacuum grease. (See figure 1.)

The sample should occupy at least 10 % of the volume of chamber II (see figure 1).

The purity of the mercury shall be at least 99,99 %.

NOTE 1 Excessive use of grease should be avoided to prevent errors when the joint is separated for weighing.

3.2 Vacuum system, capable of reaching a vacuum of at least 1 Pa.

3.3 Balance, with an accuracy of ± 0,1 mg.

3.4 Thermometer, for measuring the temperature of mercury to the nearest 0,1 K.

4 Procedure

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

4.1 Calibration

The calibration of the balance shall be checked periodically according to the control plan which defines frequency and acceptable range.

4.2 Sample preparation and determination of its mass

4.2.1 Wash the pellet or the pieces of a pellet in acetone followed by ethanol.

4.2.2 Dry the sample for 1 h in a vacuum of approximately 10 Pa.

4.2.3 Determine the mass (m) of the sample to the nearest 0,1 mg.

4.3 Determination of the void volume of chamber II (see figure 1)

4.3.1 Fill chamber I with sufficient mercury to exceed the volume of chamber II. Determine the mass (m'_1) of chamber I filled with mercury, to the nearest 0,1 mg.

4.3.2 Assemble the mercury pycnometer with the sealing flanges.

4.3.3 Keep tap I closed and evacuate the pycnometer through tap II down to approximately 1 Pa, after connecting chambers I and II.

4.3.4 Close tap II and open tap I in order to fill up the void volume of chamber II with mercury from chamber I.

4.3.5 After equilibrium pressure and temperature is reached, close tap I and release the mercury to the collection flask below chamber II.

4.3.6 Determine again the mass (m'_2) of chamber I filled with the rest of mercury, to the nearest 0,1 mg.

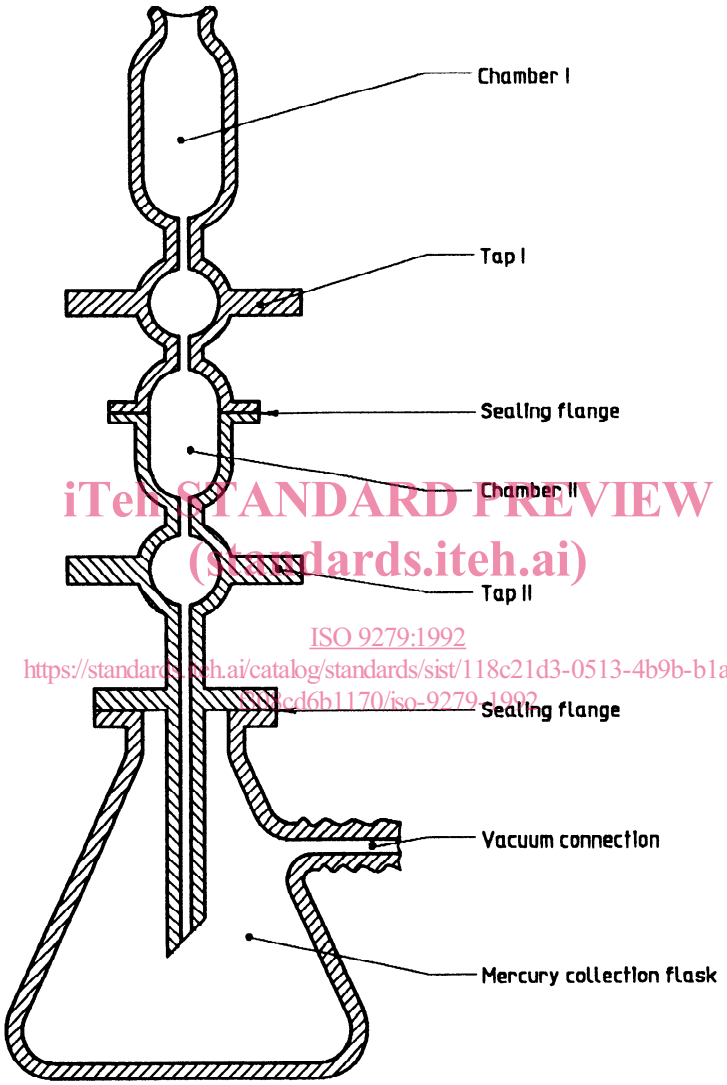

Figure 1 — Apparatus for density measurement by the mercury displacement method

4.3.7 Measure the temperature of the mercury to the nearest 0,1 K and determine its density (ϱ_{Hg}) using table 1 and by interpolation. The temperature of the mercury should not change by more than ± 0,5 K during the measurement procedure.

The determination of the void volume (V_{II}) of chamber II is performed before each sample measurement.

Table 1 — Density of mercury as a function of temperature at atmospheric pressure

Temperature °C	Density g/cm³
10	13,570 5
11	13,568 0
12	13,565 5
13	13,563 1
14	13,560 6
15	13,558 2
16	13,555 7
17	13,553 3
18	13,550 8
19	13,548 3
20	13,545 9
21	13,543 5
22	13,541 0
23	13,538 5
24	13,536 1
25	13,533 6
26	13,531 2
27	13,528 7
28	13,526 3
29	13,523 8
30	13,521 4
31	13,518 9
32	13,516 5
33	13,514 1
34	13,511 6
35	13,509 2
36	13,506 7
37	13,504 3
38	13,501 8
39	13,499 4
40	13,497 0
41	13,494 5
42	13,492 1
43	13,489 6
44	13,487 2
45	13,484 8

4.4 Determination of density and total porosity

4.4.1 At the end of the procedure described in 4.3, put the sample prepared in 4.2 into chamber II of the apparatus.

4.4.2 Repeat the procedure described in 4.3. Determine the mass (m_1) of chamber I before filling up chamber II and the mass (m_2) of chamber I after filling up chamber II as described in 4.3.1 to 4.3.7.

Perform the measurement on each sample at least three times. The result is given by the average values of these three determinations of density and total porosity.

5 Expression of results

5.1 Method of calculation

5.1.1 Calculation of the void volume of chamber II

Calculate the void volume (V_{II}) of chamber II, in cubic centimetres, using the equation

$$V_{II} = \frac{m'_1 - m'_2}{\varrho_{Hg}} \quad \ldots (1)$$

where

m'_1 is the mass (4.3.1), in grams, of chamber I partially filled with mercury before filling up the void volume of chamber II;

m'_2 is the mass (4.3.6), in grams, of chamber I partially filled with mercury after filling up the void volume of chamber II;

ϱ_{Hg} is the density of mercury, in grams per cubic centimetre, at the temperature of measurement.

5.1.2 Calculation of density and total porosity

Calculate the density (ϱ), in grams per cubic centimetre, of the pellet using the equation

$$\varrho = \frac{m}{V_{II} - \frac{m_1 - m_2}{\varrho_{Hg}}} \quad \ldots (2)$$

Calculate the total porosity (P_{tot}), in percentage by volume, of the pellet according to the equation

$$P_{tot} = \left(1 - \frac{\varrho}{\varrho_{th}}\right) \times 100 \quad \ldots (3)$$

where

m is the mass of the pellet (4.2), in grams;

m_1 is the mass (4.4.2), in grams, of chamber I containing the mercury before filling up chamber II which contains the pellet;

m_2 is the mass (4.4.2), in grams, of chamber I containing the mercury after filling up chamber II which contains the pellet;

ϱ_{th} is the theoretical density of the material (10,96 g/cm^3 for UO_2).

5.2 Precision

Provided that the sample mass exceeds 5 g and the sample occupies at least 10 % of the volume of chamber II, the following statements are valid.

a) The relative standard deviation for the mercury displacement method is $\pm$ 0,5 % for the density determination.

b) The absolute standard deviation for the total porosity determination is $\pm$ 0,3 % (V/V), in the density range between 90 % and 98 % of the theoretical density of UO_2.

6 Test report

The test report shall include the following information:

a) a reference to this International Standard;

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

c) the test method used;

d) the results obtained;

e) all operations not specified in this International Standard;

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

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