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Uranium dioxide pellets — Determination of density and amount of open and closed porosity — Boiling water method and penetration immersion method

Pastilles de dioxyde d'uranium — Détermination de la masse volumique et de la quantité de pores ouverts et fermés — Méthode à eau bouillante et méthode de pénétration par immersion

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Foreword

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Uranium dioxide pellets — Determination of density and amount of open and closed porosity — Boiling water method and penetration immersion method

1 Scope

This International Standard describes two methods for determining the bulk density and the amount of open and closed porosity of sintered UO_2 pellets. The methods can be applied to other bodies, for example green pellets, and UO_2—PuO_2 or UO_2—Gd_2O_3 pellets.

2 Principle

The methods are 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 that 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 Pan, in which the specimens are boiled in distilled water, when the boiling water method (4.1) is used.

3.6 Vacuum impregnation apparatus, which may consist of glass components, when the *m*-xylene-type impregnation method (4.2) is used (see table 1, step 2).

3.7 Test ball, made of any hard alloy or metal, e.g. carbide metal. The radius (r) should be between 5 mm and 10 mm, known with an accuracy of ± 0,5 µm, for the determination of the density of the penetration immersion liquid (see 4.2.4).

4 Procedure

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

4.1 Boiling water 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 dessicator, 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 immersed mass and the saturated mass (4.1.3 and 4.1.4), dry the pellets again and weigh. Take the average (m_D) of the two measurements.

4.1.2 Saturation

Place the pellets in a pan of distilled water and boil for 5 h, taking care that the specimens are covered

with water at all times. Isolate the specimens from the heated bottom and the sides of the pan and from each other.

After the 5 h boiling period, cool the pellets to room temperature while they are still completely covered with water. Before weighing, keep the pellets immersed in water for a minimum of 12 h.

4.1.3 Determination of the immersed mass (m_I)

After impregnation of the pellets, determine the mass (m_I) of each specimen while immersed in water. Weigh the specimen in water by placing it 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 water to the same depth as is used when the specimen is in place.

4.1.4 Determination of the saturated mass (m_S)

After the determination of the immersed mass, blot each specimen lightly with a moistened lint-free linen or cotton cloth or a moistened paper tissue to remove all excess water from the surface. Determine the saturated mass (m_S) by weighing in air. Perform the blotting operation by rolling the pellet lightly on the wet cloth, which has previously been saturated with water and then pressed just enough to remove any water that may drip from the cloth. Excessive blotting will introduce error by withdrawing water from the pores of the pellet. Place the pellet on the device in air and weigh it immediately after blotting. The whole operation should be completed as quickly as possible to minimize errors due to evaporation of water from the specimen.

4.2 *m*-Xylene impregnation method

Use *m*-xylene (analytical grade) (for possible modifications, see 4.3).

4.2.1 Determination of the dry mass (m_D)

Determine the dry mass (m_D) according to 4.1.1 (see table 1, steps 1 and 8).

4.2.2 Impregnation

For the impregnation of the UO_2 pellets with *m*-xylene, the vacuum impregnation apparatus (3.6) should be used (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 *m*-xylene.

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

4.2.3 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 *m*-xylene under atmospheric pressure.

b) The temperature of the liquid is brought up to room temperature.

4.2.4 Determination of the density of *m*-xylene

The density of *m*-xylene is determined by the use of a test ball 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 *m*-xylene.

NOTE 1 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 *m*-xylene, the density of which was determined as a function of temperature.

4.2.5 Determination of the saturated mass (m_S)

After the adjustment of weight controlling factors (see 4.2.3), blot the pellet and weigh it in a manner similar to that described in 4.1.4 (see table 1, step 6).

A representative value of the saturated mass (m_S) can be obtained by the following procedure.

Blot the pellet in such a way that a thin film of *m*-xylene remains on its surface.

Place the pellet on the device in air as shown in table 1, step 6.

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

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

4.2.6 Determination of immersed mass (m_l)

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_l) as described in 4.1.3.

4.3 Modifications

Both the boiling method and the vacuum impregnation method can also be performed with other penetration immersion liquids, e.g. cyclo-hexane, ethanol, toluene. The following conditions should be fulfilled.

a) There should be no chemical reaction with UO_2.

b) Any contamination should be easily removable.

c) The liquid shall be easily removeable 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 Expression of results

5.1 Method of calculation

5.1.1 Calculation of the density of the penetration immersion liquid

When using the method specified in 4.2.4, the density of the penetration immersion liquid (ϱ_1), in grams per cubic centimetre, is given by the equation

$$\varrho_1 = \frac{m_1 - m_2}{4\pi r^3/3} \quad \ldots (1)$$

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 reading on the balance, in grams, when the ball is immersed in the penetration immersion liquid.

5.1.2 Calculation of density and amount of open and closed porosity

When using the methods specified in 4.1 and 4.2, the following calculation procedure applies.

The geometric pellet volume (V), in cubic centimetres, is given by the equation

$$V = \frac{m_S - m_l}{\varrho_1} \quad \ldots (2)$$

where

m_S is the saturated mass, in grams;

m_l is the immersed mass, in grams;

ϱ_1 in the method described in 4.1, is the density of water at the relevant temperature, or in the method described in 4.2, is the density of the penetration immersion liquid, determined according to equation (1).

Calculate the volumes of the open pores (V_{op}) and closed pores (V_{cp}), in square centimetres, using the equations

$$V_{op} = \frac{m_S - m_D}{\varrho_1} \quad \ldots (3)$$

$$V_{cp} = \frac{m_D - m_l}{\varrho_1} - \frac{m_D}{\varrho_{th}} \quad \ldots (4)$$

where

m_D is the dry mass, in grams;

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

ϱ_1, m_S and m_l 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 amounts of total porosity (P_{tot}), open porosity (P_{op}) and closed porosity (P_{cp}), in percentage by volume, using the equations

$$\varrho_{UO_2} = \frac{m_D \times \varrho_1}{m_S - m_l} \quad \ldots (5)$$

$$P_{tot} = \left(1 - \frac{\varrho_{UO_2}}{\varrho_{th}}\right) \times 100 \quad \ldots (6)$$

$$P_{op} = \frac{m_S - m_D}{m_S - m_l} \times 100 \quad \ldots (7)$$

$$P_{cp} = \left(\frac{m_D - m_l}{m_S - m_l} - \frac{m_D}{m_S - m_l} \times \frac{\varrho_1}{\varrho_{th}}\right) \times 100 \quad \ldots (8)$$

The relative percentage of open porosity of the total porosity is given by the equation

$$\frac{P_{op}}{P_{tot}} = \frac{\frac{m_S - m_D}{m_S - m_l}}{1 - \frac{\varrho_{UO_2}}{\varrho_{th}}} \times 100 \qquad \ldots (9)$$

6 Precision

The relative standard deviation of the boiling water method is $\pm$ 0,1 %. In the *m*-xylene impregnation method, the relative standard deviation for the bulk density is about $\pm$ 0,05 % and the absolute standard deviation for P_{op} and P_{cp} is $\pm$ 0,03 % (V/V).

7 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) method of drying the test sample;

e) penetration conditions;

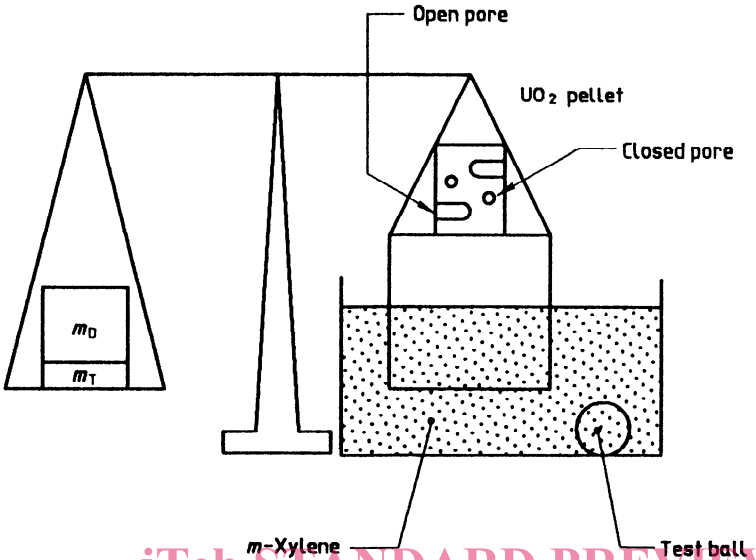
f) penetration immersion liquid used;

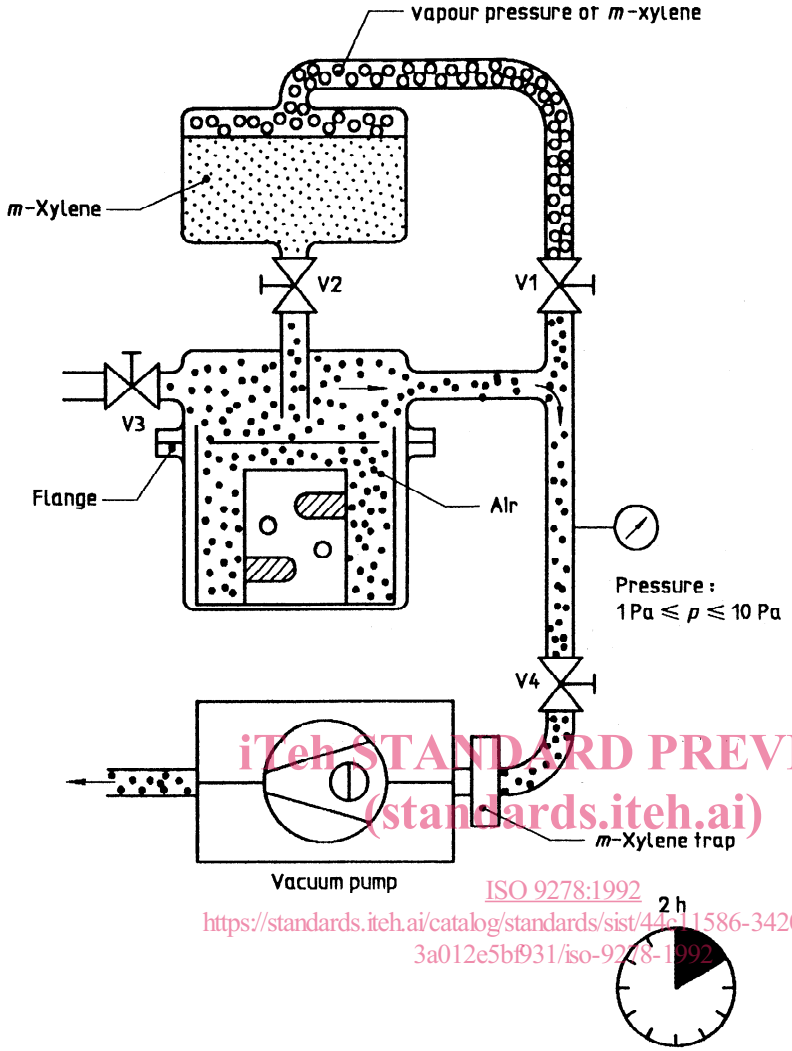
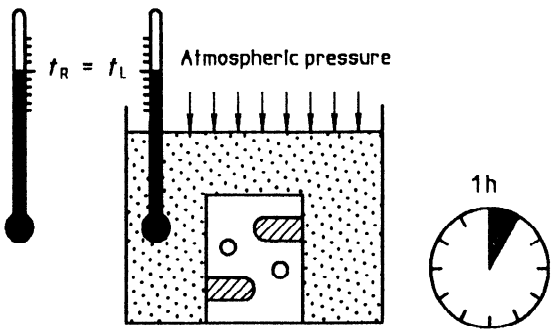
g) the results obtained;

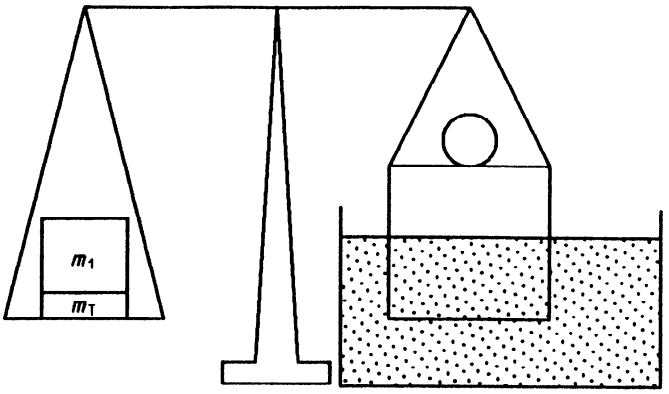
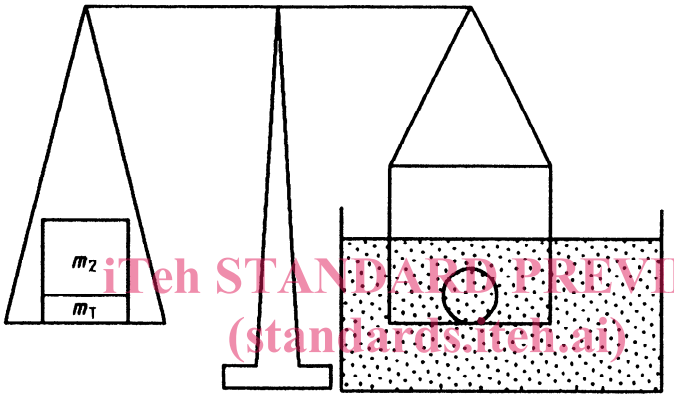
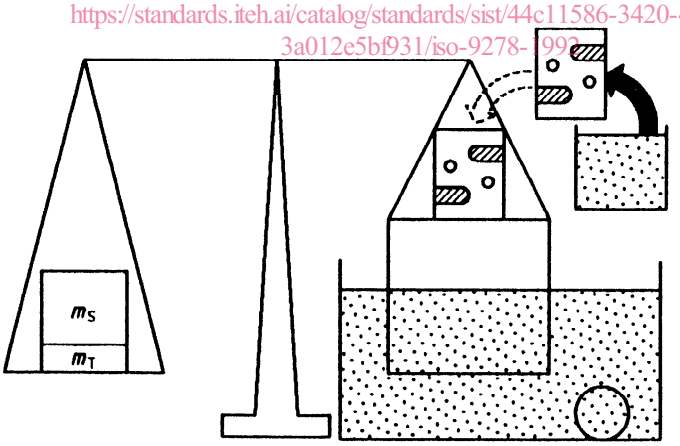
h) all operations not specified in this International Standard;

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

Table 1 — *m*-Xylene impregnation method

Step 1		Mass in air m_T = Tare mass m_D = Mass of dry pellets in air

Step 2		Impregnation a) Turn on the pump with valves V1, V2, V3 and V4 closed. Open V4. Pump until a pressure of < 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 *m*-xylene. f) Turn off the pump. Open V3 to allow air into the apparatus.
Step 3		Adjustment of weight controlling factors a) The open pores are totally filled with *m*-xylene. b) The temperature (t_L)) of the liquid is brought up to room temperature (t_R).

Step 4		Determination of the density of *m*-xylene m_1 = Mass of test ball m_2 = Reading on the balance when the test ball is immersed in *m*-xylene
Step 5		
Step 6		Determination of saturated mass (m_S) m_S = Mass of pellet and *m*-xylene impregnated pores