INTERNATIONAL STANDARD

ISO/CD 9161

Second edition

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Uranium dioxide powder — Determination of apparent density and tap density

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Poudre de dioxyde d'uranium — Détermination de la masse volumique apparente et de la masse volumique après tassement

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Reference number
ISO 9161:2004(E)

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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 9161 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel cycle*.

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This second edition cancels and replaces the first edition (ISO 9161:2004), which has been technically revised. The major changes in the second edition are as follows:

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- An Introduction has been added.
- Definitions have been updated.
- MORE

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Introduction

Uranium dioxide (UO_2) powder is produced for use in nuclear reactors by a variety of processes. For UO_2 powder to be used in the production of sintered pellets as a nuclear fuel, it must meet specifications in standards such as ASTM C753[5] or specifications supplied by the user. These specifications may include a requirement for apparent (or bulk) density, tap density, or both.

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ISO 9161 specifies a method for determination of the apparent density and tap density of free-flowing UO_2 powder, and can be used for a variety of powder types. The method can also be applied to other fuel powders, and to powder mixtures, to demonstrate compliance with appropriate specifications for those powders.

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It has been assumed in the preparation of ISO 9161 that the execution of its provisions and the interpretation of the results obtained are entrusted to appropriately qualified and experienced people.

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Uranium dioxide powder — Determination of apparent density and tap density

1 Scope

This International Standard specifies a method of determining the apparent density and tap density of free-flowing uranium dioxide (UO_2) powder which will be used for pelleting and sintering of UO_2 pellets as a nuclear fuel.

This method can be used for different UO_2 powder types including grains, granules, spheres or other kinds of particles. The method can also be applied to other fuel powders as PuO_2, ThO_2 and powder mixtures as UO_2-PuO_2 and UO_2-Gd_2O_3.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
apparent density
bulk density
density of a powder obtained by free pouring under specified conditions

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2.2
tap density
density of a powder in a container that has been tapped under specified conditions

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3 Principle

3.1 Apparent density

A portion of sample is allowed to fall through a funnel of standard dimensions into a tared density cup which is filled up to a mark which defines a distinct volume. The cup and contents are weighed and the apparent density is calculated from the mass and volume of the powder.

3.2 Tap density

A calibrated density cup containing a weighed portion of sample is tapped by means of a special apparatus. The tapping conditions are fixed. The tap density is determined from the weight and volume of the powder after the treatment.

4 Apparatus

4.1 Powder flowmeter funnel, having an orifice diameter, d, of 2,5 mm, 5,0 mm, 10 mm or 15 mm, depending on the powder type, having a conical angle of 60° and some means of closing the orifice (see Figure 1).

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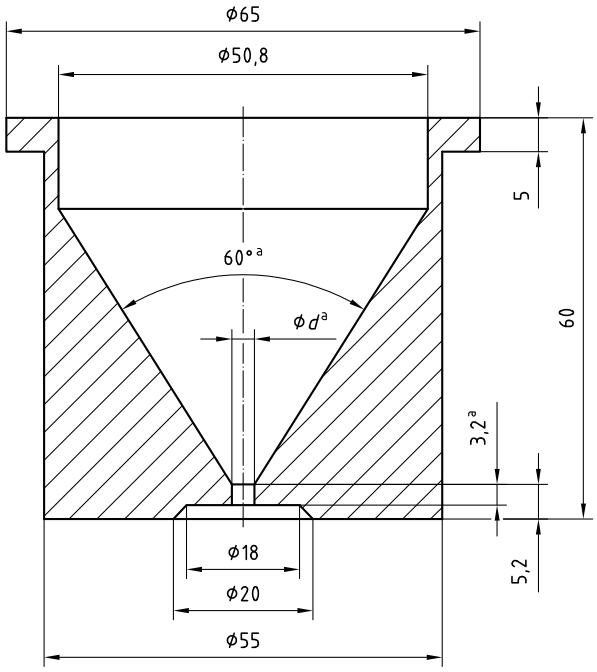
Dimensions in millimetres



Key

d [a] = $2,5^{+0,2}_{0}$, $5,0^{+0,2}_{0}$, $10,0^{+0,2}_{0}$ or $15,0^{+0,2}_{0}$

a These dimensions, in millimetres, are mandatory.

b The symbol Ø represents diameter.

Figure 1 — Powder flowmeter funnel (according to Reference [1])

4.2 Density cup, a graduated cylindrical cup having a capacity of 10 cm^3, 25 cm^3, 50 cm^3 or 100 cm^3, depending on the powder type.

4.3 Tapping device, consisting, for example, of a baseplate which is tapped by a motor with worm drive and a cam shaft speed of (250 ± 15) min^{-1} and enabling a tapping stroke travel of about 2 mm to 3 mm.

4.4 Adjustable counter, which can be preset to deliver a number of taps between 1 and 9 999.

4.5 Stand, to support the powder flowmeter concentric with the density cup so that the bottom of the powder flowmeter orifice is 50 mm above the mark of the density cup when the apparatus is assembled as shown in Figure 2.

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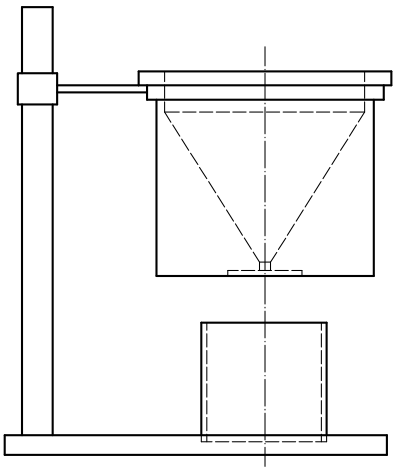

Figure 2 — Stand with powder flowmeter funnel and density cup (without tapping device)

4.6 Balance, having a capacity which is suitable for the chosen density cup and a sensitivity of 0,1 g or better.

NOTE 1 A flowmeter funnel, density cup and stand from the Alcan Ingot and Powders Div of Alcan Aluminium Corp, Box 290, Elisabeth, N. J. 07207, USA or an equivalent equipment (60°, diameter D) can be used. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

NOTE 2 A device with the necessary other equipment as Tap-Pak Volumeter model No. JEL ST2 from J. Engelsmann AG, Ludwigshafen a. Rh., Fed. Rep. of Germany or an equivalent equipment can be used. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5 Sampling and samples

A representative laboratory test portion has to be taken from the UO_2 powder production batch to be characterized.

Measures have to be undertaken, if necessary, to prevent separation of the powder particle fractions or any change of properties during the transport of the powder.

The test portion size has to be sufficient to exceed the volume of the graduated density cup (approximately twice the volume of the density cup).

6 Procedure

6.1 Safety precautions

Standard precautions, including appropriate containment and personal protective equipment (PPE), shall be observed when handling uranium dioxide samples.

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6.2 Calibration

A calibration check of the balance, in accordance with ISO/IEC 17025 [5], which defines the frequency and the acceptable deviation, shall be made periodically.

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6.3 Determination of the apparent density

6.3.1 Weigh the empty density cup (cylindrical container) and note its mass (m_1).

6.3.2 Adjust the funnel so that its axis coincides approximately with that of the cylinder, with the bottom of the funnel orifice at a height of about 25 mm above the brim of the density cup or of about 50 mm above the mark. Close the orifice of the funnel.

6.3.3 Fill the funnel with the test sample of UO_2 powder.

6.3.4 Open the orifice of the funnel so that the powder flows steadily into the density cup.

Fill the density cup up to the mark which defines the volume (V_1). Level the surface of the powder at this mark by carefully inclining the density cup and moving it back into vertical position. Precautions shall be taken to ensure that actions taken to level the surface of the powder do not cause packing of the powder, leading to an erroneous result.

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Alternatively, fill the density cup which volume is measured up to the upper brim of the density cup. Use a spatula to remove powder which is over the brim of the density cup, to make the powder surface level at the open end of the density cup.

6.3.5 Pour the rest of the powder back into the sampling flask.

6.3.6 Determine the mass of the filled density cup in connection with the measurement of the density (m_2).

6.4 Determination of tapped density

6.4.1 Put the filled density cup (from determination of the apparent density, 6.3) onto the tapping device.

6.4.2 Adjust the counter to a suitable number of taps to obtain a constant powder volume (200 to 2 400 taps). The amplitude shall be about 2 mm to 3 mm. Record the number of taps and the tapping time.

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For new processes, the appropriate number of taps required to obtain a constant powder volume shall be determined. An example of how to do this is found in ASTM C1770.[6]

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6.4.3 Determine the volume of the UO_2 powder in the cylindrical container after tapping by means of the graduations and record this value (V_2).

6.4.4 Weigh the filled density cup and note its mass (m_3).

6.5 Repeated determinations

Repeat 6.3 and 6.4 with new powder test portions so that a total of three single determinations are made from each powder.

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7 Expression of results

7.1 Method of calculation

The apparent density before tapping of the UO_2 powder (ρ_a), in grams per cubic centimetre, is given by the expression:

$$\rho_a = \frac{m_2 - m_1}{V_1}$$

where

m_1 is the mass, in grams, of the empty density cup (6.3.1);

m_2 is the mass, in grams, of the filled density cup (6.3.6);

V_1 is the volume, in cubic centimetres, of the powder before tapping.

The tap density of the UO_2 powder (ρ_v), in grams per cubic centimetre, is given by the expression:

$$\rho_v = \frac{m_2 - m_1}{V_2}$$

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where

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V_2 is the volume, in cubic centimetres, of the UO_2 after tapping.

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The final values for the apparent and the tap density are obtained by calculating the mean values of three determinations.

Deleted: m_3 is the mass, in grams, of the filled density cup after tapping.¶

7.2 Precision

The relative standard deviation for the determination depends mainly on the height of the density cup and can be under optimal conditions 0,6 % for the apparent density and 0,5 % for the tap density.

8 Test report

The test report shall include the following information:

a) the reference to this International Standard (e.g., ISO 9161:-year of publication -);

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

c) the orifice diameter of the powder flowmeter funnel and the volume of the density cup used;

d) the number of taps and tapping time;

e) the results obtained, including estimated uncertainty;

f) all operations not specified by this International Standard;

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

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Bibliography

[1] ISO 3923-1, *Metallic powders — Determination of apparent density — Part 1: Funnel method*

[2] ISO 3953, *Metallic powders — Determination of tap density*

[3] ISO 3252, *Powder metallurgy — Vocabulary*

[4] ISO/IEC 17025, *General Requirements for the Competence of Testing and Calibration Laboratories*

[5] ASTM C753, *Standard Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder*

[6] ASTM C1770, *Standard Test Method for Determination of Loose and Tapped Bulk Density of Plutonium Oxide*

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