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Second edition
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Uranium dioxide powder — Determination of apparent density and tap density

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

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

This second edition cancels and replaces the first edition (ISO 9161:2004), which has been technically revised.

The main changes compared to the previous edition are as follows:

— an introduction has been added;

— definitions in Clause 3 have been updated;

— safety precautions have been updated.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Uranium dioxide (UO_2) powder is the source material for the manufacture of nuclear fuel as pellets, and is produced for use in nuclear reactors by a variety of processes. Specifications for UO_2 powder used in the production of sintered pellets as a nuclear fuel are given in standards such as ASTM C753[5] or specifications supplied by the user. These specifications can include requirements for apparent (or bulk) density, tap density, or both.

This document 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.

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

Uranium dioxide powder — Determination of apparent density and tap density

1 Scope

This document 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.

This document is based on the principle of using a flowmeter funnel (see 4.1). Other measurement apparatus, such as a Scott volumeter, can also be used.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
apparent density
loose bulk density
dry mass per unit volume of a powder obtained by free pouring under specified conditions

3.2
tap density
dry mass per unit volume of a powder in a container that has been tapped under specified conditions

4 Principle

4.1 Apparent density

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

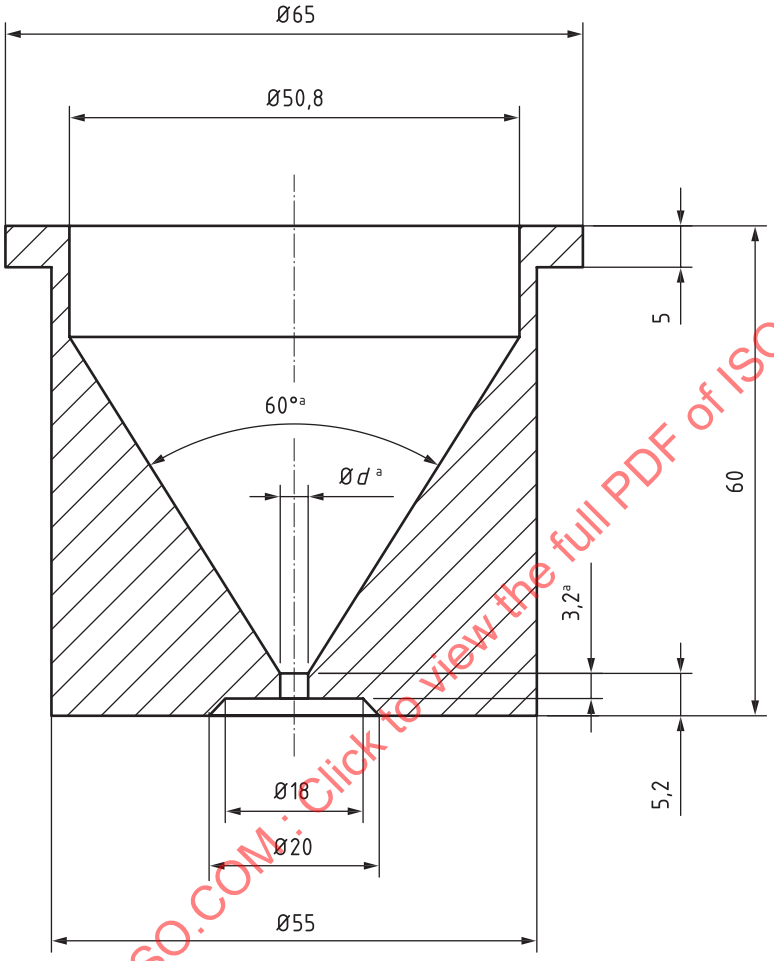
4.2 Tap density

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 mass and volume of the powder after the treatment.

5 Apparatus

5.1 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).

Dimensions in millimetres



Key

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

a These dimensions are mandatory.

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

5.2 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.

5.3 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.

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

5.5 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.

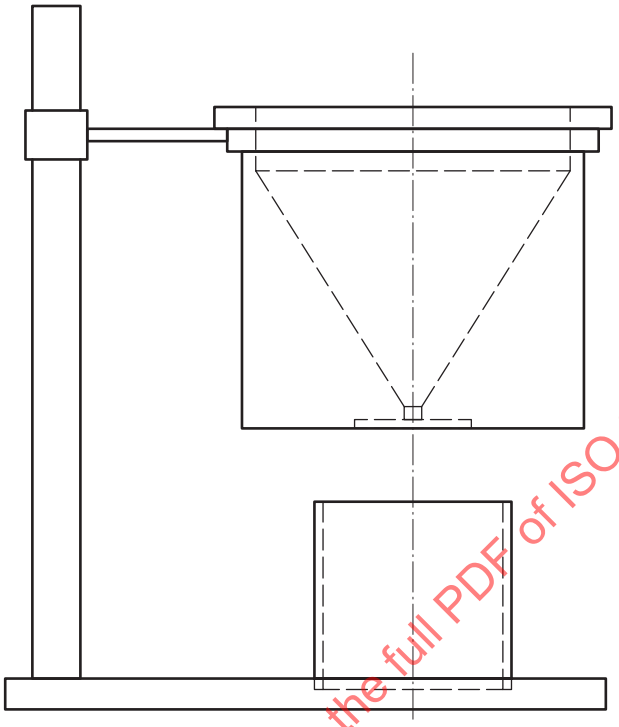


Figure 2 — Stand with powder flowmeter funnel and density cup (without tapping device)

5.6 Balance, having a capacity which is suitable for the chosen density cup and a sensitivity of 0,01 g or better.

6 Sampling and samples

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

Measures shall 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 shall be sufficient to exceed the volume of the graduated density cup (approximately twice the volume of the density cup).

Additional guidance on uranium dioxide sample collection can be found in ASTM C753[5].

7 Procedure

7.1 Safety precautions

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

7.2 Calibration

Periodic calibration checks of the balance shall be performed using an appropriate control plan, which requires the user to define the frequency of calibration checks and appropriate acceptance criteria. A calibration programme for laboratory equipment as described in ISO/IEC 17025 is recommended.

7.3 Determination of the apparent density

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

7.3.2 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.

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

7.3.4 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.

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.

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

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

7.4 Determination of tapped density

7.4.1 Put the filled density cup (from determination of the apparent density, see 7.3) onto the tapping device.

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

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].

7.4.3 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).

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

7.5 Number of determinations

Repeat 7.3 and 7.4 with new powder test portions so that a total of three single determinations are made from each powder.

7.6 Quality control

A measurement control program with appropriate quality control material(s) should be established to demonstrate satisfactory method performance. It is recommended to follow guidance in ISO/IEC 17025 for assuring the quality of results.

8 Expression of results

8.1 Method of calculation

The apparent density before tapping of the UO_2 powder, ρ_a, in grams per cubic centimetre, is given by Formula (1):

$$\rho_a = \frac{m_2 - m_1}{V_1} \quad (1)$$

where

m_1 is the mass, in grams, of the empty density cup (see 7.3.1);

m_2 is the mass, in grams, of the filled density cup (see 7.3.6);

V_1 is the volume, in cubic centimetres, of the UO_2 powder before tapping.

The tap density of the UO_2 powder, ρ_v, in grams per cubic centimetre, is given by Formula (2):

$$\rho_v = \frac{m_3 - m_1}{V_2} \quad (2)$$

where

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

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

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

8.2 Precision

The relative standard deviation for the determination depends mainly on the height of the density cup and can be under optimal conditions between 0,6 % to 1,5 % for the apparent density and 0,5 % to 2,0 % for the tap density.

9 Test report

The test report shall include the following information:

a) the reference to this document (i.e. ISO 9161:2019);

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 in this document;