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Cereals — Determination of bulk density, called mass per hectolitre —

Part 3: Routine method

Céréales — Détermination de la masse volumique, dite masse à l'hectolitre —
Partie 3: Méthode pratique

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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).

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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 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

The second edition cancels and replaces the first edition (ISO 7971-3:2009), which has been technically revised. The main changes compared with the previous edition are as follows:

— a formulae has been introduced to express the results obtained with NILEMA LITRE for wheat and barley.

A list of all parts in the ISO 7971 series can be found on the ISO website.

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.

Cereals — Determination of bulk density, called mass per hectolitre —

Part 3: Routine method

1 Scope

This document specifies a routine method for the determination of bulk density, called "mass per hectolitre", of cereals as grain using manual or automatic, mechanical, electric or electronic mass per hectolitre measuring instruments.

NOTE Further details of the measuring instruments are specified in ISO 7971-2:2019, 6.4.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7971-2:2019, *Cereals — Determination of bulk density, called mass per hectolitre — Part 2: Method of traceability for measuring instruments through reference to the international standard instrument*

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
mass per hectolitre
bulk density
test weight
<cereals> ratio of the mass of a cereal to the volume it occupies after being poured into a container under defined manufacturer's conditions

Note 1 to entry: Mass per hectolitre is expressed in kilograms per hectolitre of grains as received.

Note 2 to entry: Mass per hectolitre, as defined in this document, is different from "packing density" or "intrinsic density" of cereals.

[SOURCE: ISO 7971-1:2009, 2.1, modified — In the definition, "defined manufacturer's conditions" has replaced "well-defined conditions".]

4 Principle

The mass per hectolitre of a cereal is obtained from the mass of a volume of cereal determined under controlled sample filling and flow conditions.

The mass per hectolitre can be affected by

a) space between the grains, which depends on the grain size and shape, and

b) density of the grains.

5 Apparatus

5.1 General requirement for mass per hectolitre apparatus. Any apparatus (5.2 and 5.3) shall be verified in accordance with ISO 7971-2 and shall fulfil the performance demands specified therein.

5.2 Hand-operated measuring instrument. Apparatus consisting of a filling hopper, a measuring container and the accessories necessary for their use.

The manner in which the grain is poured into the measuring container and the way in which it packs into the container can cause the measurements taken by the various instruments to vary and lead to measurement errors.

To minimize such variations, special attention should be given to ensuring that the design of the instruments and their size, material and shape are appropriate.

NOTE Annexes A and B contain examples of technical specifications of two hand-operated instruments with a capacity of 1 l.

5.3 Automatic measuring instrument. This category includes various types of devices, some of which can be used on their own or combined with an infrared analyser.

The measurement is based on the application of formulae to allow the correcting of the bias and/or the drifts monitored. It does not include manual weighing. The numeric value of the hectolitre mass is directly displayed.

5.4 Analytical balance, capable of being read to the nearest 0,1 g or 0,01 g depending on the volume of the container (see 6.2).

5.5 Spirit level.

6 Procedure

6.1 General

The measurements shall be taken using grain from which large impurities (straw, stones, large amounts of loose husks, etc.) have been discarded, taking environmental conditions into consideration to ensure that there is no difference in temperature between the grain and the room in which the test is performed.

Determine the mass per hectolitre in duplicate. For all the devices and for every sample, it is advisable to perform the two measurements on two different grain test portions, when the sample size enables it.

NOTE Repeating the measurement on the same grain test portion changes the friction coefficient which therefore makes it easier for the grains to slide; they are then more tightly packed, which increases the value of the mass per hectolitre.

6.2 Hand-operated instruments

Check that the various components of the instrument are clean and that they are working properly.

Make sure that equipment is placed on a firm, flat base, after using a spirit level to check that the base is horizontal.

Take great care to avoid any impact during filling. If the apparatus is jolted, cancel the test and start again.

Each type of apparatus is different; use each according to the manufacturer's instructions.

When using the analytical balance (5.4), weigh to the nearest 1 g for a 1 l container or the nearest 0,1 g for apparatus with a container of smaller volume.

6.3 Automatic instruments

As the operations to be performed prior to the actual measurement differ according to the type of equipment used, reference to the manufacturer's instructions is recommended.

Ensure that the instrument is placed on a horizontal surface in a room protected from extreme temperatures, humidity, dust and vibrations.

Take particular care to

a) select the correct cereal to be measured to ensure that the right calibration is used,

b) use the volume of cereals recommended for the device in question, and

c) empty the collector drawer between samples.

6.4 Expression of results

Take the arithmetic mean of the two determinations as the result if the repeatability conditions are met.

Express the result to the nearest 0,1 kg/hl.

If they are not met, take as a final result the mean of the four measurements.

Specify in the analysis report the conditions for obtaining the final result that can be attributed to sample variability.

7 Precision

7.1 Interlaboratory trial

Details of an interlaboratory test on the precision of the method are summarized in Annex C. The values derived from this interlaboratory test cannot be applied to other mass per hectolitre ranges and matrices than those given.

7.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, shall in not more than 5% of cases be greater than the repeatability limit

$$r = 0{,}4$$

for products where the mass per hectolitre is between 67,5 kg/hl and 84,5 kg/hl (see Tables C.1 and C.2, and Figure C.1).

7.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment.

In practice, it is not appropriate to compare the results from two laboratories if the test concerned imposes repeatability conditions.

The appropriate comparison tool is the critical difference as described in 7.5

7.4 Comparison of two groups of measurements in one laboratory

Critical difference, CD_r, is the difference between two averaged values obtained from two test results under repeatability conditions. As the result is a mean of two values (see 6.1), the comparison of two bulk densities shall be made with critical difference.

CD_r is given by Formula (1):

$$CD_r = 2{,}8 s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8 s_r \sqrt{\frac{1}{2}} = 1{,}98 s_r = 0{,}23 \quad (1)$$

i.e. 0,2 kg/hl, after rounding, where

s_r is the standard deviation of repeatability;

n_1 and n_2 are the number of test results corresponding to each averaged value (here, $n_1 = n_2 = 2$).

7.5 Comparison of two groups of measurements in two laboratories

The critical difference, CD_R, between two averaged values obtained in two different laboratories from two test results under repeatability conditions is given by Formula (2):

$$CD_R = 2{,}8 \sqrt{s_R^2 + s_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}8 \sqrt{s_R^2 - 0{,}5 s_r^2} = 1{,}18 \quad (2)$$

i.e. 1,2 kg/hl, after rounding, where

s_r is the standard deviation of repeatability;

s_R is the standard deviation of reproducibility;

n_1 and n_2 are the number of test results corresponding to each averaged value (here, $n_1 = n_2 = 2$).

7.6 Uncertainty

Uncertainty, U, is a parameter representing the distribution of the values which may reasonably be attributed to the result.

It is possible to evaluate the measurement uncertainties using data obtained from studies conducted in accordance with ISO 5725-2.

The reproducibility standard deviation obtained in a collaborative study is a valid basis for the evaluation of measurement uncertainty since, per definition, uncertainty characterizes the dispersion of the values which may be reasonably attributed to the parameter. The calculated expanded uncertainty should be ≤ +/−2 reproducibility standard deviation.

8 Test report

The test report shall contain at least the following information:

a) an indication of the method used, including a reference to this document, i.e. ISO 7971-3;

b) the result obtained;

c) all the operating details not specified in this document or those regarded as optional, in addition to any possible incidents that might have affected the result;

d) all the information necessary for a full identification of the sample.

Annex A
(informative)

Description of dimensions and use of KERN[1)]

A.1 Dimensions of the apparatus

A.1.1 General

The dimensions of the various components of the apparatus should be as specified in A.1.2 to A.1.7. See Figure A.1 for a depiction of the components.

A.1.2 Pre-filling measure

Volume to level mark:	1 350 ml ± 10 ml
Internal diameter:	86,0 mm ± 0,2 mm

A.1.3 Filling hopper

Internal diameter:	79,0 mm ± 0,1 mm
Wall thickness:	1,0 mm ± 0,2 mm
Height above piston:	280 mm ± 2 mm

A.1.4 Piston

Diameter:	87,5 mm ± 0,1 mm
Height:	40,0 mm ± 0,2 mm
Mass:	450 g ± 2 g

A.1.5 Measuring container

Internal diameter:	88,2 mm ± 0,1 mm
Internal height above piston:	163,7 mm ± 0,1 mm
Wall thickness:	1,2 mm ± 0,5 mm
External reinforcement of upper edge:	
— thickness:	2,5 mm ± 0,5 mm
— height:	6,0 mm ± 1,0 mm
Base thickness:	4,5 mm ± 0,1 mm

1) This is an example of a suitable product available commercially. This information is given for the convenience of users of this document 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.