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ISO/FDIS 712-1

Cereals and cereal products — **Determination of moisture** content —

Part 1: Reference method

Document Preview

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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 document 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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This document was prepared by Technical Committee ISO/TC 34, Food products, Subcommittee SC 4, Cereals and pulses, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, Cereal and cereal products, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 712-1 cancels and replaces ISO 712:2009, of which it constitutes a minor revision. The changes are as follows:

— numeration and change in the title to align with the new ISO 712-2.

A list of all parts in the ISO 712 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 and cereal products — Determination of moisture content —

Part 1:

Reference method

1 Scope

This document specifies a routine reference method for the determination of the moisture content of cereals and cereal products.

This document applies to: wheat, rice (paddy, husked and milled), barley, millet (*Panicum miliaceum*), rye, oats, triticale, sorghum in the form of grains, milled grains, semolina or flour.

The method is not applicable to maize and pulses.

NOTE For moisture content determination in maize, see ISO 6540^[5]; and for pulses, see ISO 24557^[7].

2 Normative references iTeh Standards

There are no normative references in this document.

3 Terms and definitions Document Preview

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 https://www.electropedia.org/

3.1

moisture content true moisture content

mass loss undergone by a product

Note 1 to entry: Moisture content is expressed as a percentage.

4 Principle

If necessary, a laboratory sample is ground after conditioning. A test portion is dried at a temperature between 130 °C and 133 °C, under conditions which enable a result to be obtained which corresponds to that obtained by the absolute method described in <u>Annex B</u>.

5 Apparatus

5.1 Analytical balance, capable of weighing to an accuracy of $\pm 0,001$ g.

- **5.2 Grinding mill**, having the following characteristics:
- a) made of material which does not absorb moisture;
- b) easy to clean and having as little dead space as possible;
- c) enables grinding to be carried out rapidly and uniformly, without appreciable development of heat (difference of temperatures before and after grinding smaller than or equal to 5 °C);
 - NOTE A grinding mill fitted with a cooling device can conform with this requirement.
- d) air tight to avoid water exchange between sample and external air;
- e) adjustable to obtain particles of the dimensions indicated in <u>Table 1</u>.
- **5.3 Metal dish**, non-corrodible under the test conditions, or **glass dish**, with a lid and having an effective surface area enabling the test portion to be distributed, to give a mass per unit area of not more than 0,3 g/cm².
- **5.4 Constant-temperature oven**, electrically heated, controlled in such a way that, during normal working, the temperature of the air and of the shelves carrying the test portions is maintained within the range 130 °C to 133 °C in the vicinity of the test portions.

The oven shall have a heat capacity such that, when initially adjusted to a temperature of $131\,^{\circ}$ C, it can regain this temperature in less than 30 min after insertion of the maximum number of test portions that can be dried simultaneously.

The effectiveness of the ventilation shall be determined using durum wheat semolina, of maximum particle size of 1 mm, as the test material. The ventilation shall be such that, after insertion of the maximum number of test portions that the oven can accommodate and drying at a temperature of 130 $^{\circ}$ C to 133 $^{\circ}$ C, the results, after heating the same test portions for 2 h and then for a further 1 h, do not differ by more than 0,15 g of moisture per 100 g of sample.

5.5 Desiccator, containing an effective desiccant.

6 Sampling

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Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 24333[6].

A representative sample, in an airtight packaging, should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

7 Preparation of the test sample

7.1 Products not requiring grinding

Products having particle size characteristics indicated in <u>Table 1</u> do not need to be ground before the determination.

Mix the laboratory sample thoroughly before taking the test portion (8.2).

Table 1 — Particle size characteristics of products not requiring grinding

Particle size characteristics	Proportion	
mm	%	
u 1,7 (1,8) ^a	100	
> 1,0 (1,0)b	u 10	
< 0,5 (0,56)a	W 50	
Nominal size of openings, ISO 3310-1,[1] that does not retain this particle size.		
Naminal size of analyze ISO 2210.1 [1] that retains this partials size		

b Nominal size of openings, ISO 3310-1, $^{[\![1\!]}$ that retains this particle size.

7.2 Products requiring grinding

7.2.1 General

If the products do not have the particle size characteristics mentioned in <u>Table 1</u>, they shall be ground either without preconditioning (7.2.2) or with preconditioning (7.2.3), as required.

7.2.2 Grinding without preconditioning

For products that are not likely to undergo variations in moisture content during grinding (in general, products with a moisture content between 9 % and 15 %), grind without preconditioning.

NOTE The range of moisture contents given for conditioning products before grinding corresponds approximately in the laboratory to a temperature of 20 $^{\circ}$ C and a relative humidity of 40 $^{\circ}$ 6 to 70 $^{\circ}$ 6.

Adjust the grinding mill (5.2) to obtain particles of the dimensions indicated in <u>Table 1</u>.

Then, quickly grind a quantity of the laboratory sample according to the apparatus used and at least greater than that required for the test portion, and immediately proceed in accordance with 8.2.

7.2.3 Grinding with preconditioning

Products which are likely to undergo changes in moisture content during the course of grinding (in general, products with a moisture content greater than 15% or less than 9%) shall be preconditioned so as to bring their moisture content to between 9% and 15% before grinding.

If the moisture content is more than 15 % (the most frequent case), weigh, to the nearest 0,001 g, a sufficient quantity of the laboratory sample to provide a test portion greater than 5 g (see 7.2.2). Record the mass of the test portion before preconditioning and dish as m'_2 . Pre-dry in accordance with 8.3, except that the time of heating in the oven (5.4) shall be 7 min to 10 min and the product shall be cooled to laboratory temperature with the dish (5.3) uncovered and without a desiccator, for at least 2 h.

NOTE It is possible that these times are not be suitable for all products, e.g. paddy rice.

For products having moisture contents of less than 9 %, weigh, to the nearest 0,001 g, a sufficient quantity of the laboratory sample to provide a test portion greater than 5 g (see 7.2.2). Record the mass of the test portion before preconditioning and dish as m'_2 . Place in a suitable atmosphere (generally that of the laboratory) and leave until a moisture content within the limits indicated above is obtained.

After conditioning, weigh the sample to the nearest 0,001 g. Record the mass of the test portion after preconditioning and dish as m'_3 . Grind immediately, adjusting the grinder so as to obtain particles of the dimensions indicated in <u>Table 1</u>, and immediately proceed in accordance with <u>7.2.2</u>.

8 Procedure

8.1 Number of determinations

Carry out separate determinations on two test portions taken from the laboratory sample in accordance with <u>8.2</u> and <u>8.3</u>. If the absolute difference between the two values obtained is greater than the repeatability limit given in <u>Clause 10</u>, repeat the determination until requirements are satisfied.

8.2 Test portion

Rapidly weigh, to the nearest 0,001 g, a quantity of 5 g \pm 1 g of the laboratory sample (7.2.2 or 7.2.3) in the dish (5.3). Record the mass of the undried test portion and dish as m'_0 . Previously dry and tare the dish, together with its lid, and record the mass, m_d , to the nearest 0,001 g.

8.3 Drying

Place the open dish containing the test portion (8.2), together with the lid, in the oven (5.4) and leave for 120 min \pm 5 min (90 min for flours).

In certain cases, and particularly in hot and dry countries, the drying period may be reduced to $60 \text{ min} \pm 5 \text{ min}$, which is sufficient time for the test portions to attain constant mass. Review these times regularly.

Do not open the door of the oven during drying and do not place moist products in the oven before removing the dry test portions, as this will result in partial rehydration of the latter.

After drying, quickly remove the dish from the oven, cover, and place in the desiccator (5.5). When several tests are being carried out, never place dishes on top of one another in the desiccator, but place them side by side.

8.4 Weighing

When the dish has cooled to laboratory temperature (generally between 30 min and 45 min after it has been placed in the desiccator), weigh to the nearest 0.001 g. Record the mass of the dried test portion and dish as m'_1 .

9 Expression of results

9.1 Without preconditioning

The moisture content, $w_{\rm H_2O}$, expressed in grams per 100 g of the product as received, is given by Formula (1):

$$w_{\rm H_2O} = \left(1 - \frac{m_1}{m_0}\right) \times 100 \tag{1}$$

where

 $m_0 = m'_0 - m_d$ is the mass, in grams, of the test portion (8.2);

 $m_1 = m'_1 - m_d$ is the mass, in grams, of the test portion after drying (8.4).

Calculate the arithmetic mean of two results satisfying the repeatability conditions (see $\underline{10.2}$). Round the result to two places of decimals.

9.2 With preconditioning

The moisture content, $w_{\rm H_2O}$, expressed in grams per 100 g of the product as received, is given by Formula (2):

$$w_{\text{H}_2\text{O}} = \left[\left(m_0 - m_1 \right) \frac{m_3}{m_0} + m_2 - m_3 \right] \times \frac{100}{m_2} = \left(1 - \frac{m_1 m_3}{m_0 m_2} \right) \times 100$$
 (2)

where

 $m_2 = m'_2 - m_d$ is the mass, in grams, of the sample taken before preconditioning (7.2.3);

 $m_3 = m'_3 - m_d$ is the mass, in grams, of the preconditioned sample (7.2.3).

Calculate the arithmetic mean of two results satisfying the repeatability conditions (see $\underline{10.2}$). Round the result to two places of decimals.

10 Precision

10.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in $\underline{\text{Annex A}}$. The values derived from this interlaboratory test can only be applied to other moisture contents in the range 10 % to 18 % and matrices given therein.

10.2 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 that 5 % of cases be greater than the repeatability limit

$$r = 2,77s_{\rm r}$$

$$\underline{\rm ISO/FDIS~712-1}$$

$$r = 2,77 \times 0,043 = 0,12$$

$$\underline{\rm ISO/FDIS~712-1}$$

$$r = 2,77 \times 0,043 = 0,12$$

for products whose moisture content is between 10,00 % and 18,00 % (see Table A.1 and Figure A.1).

10.3 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, shall in not more that 5% of cases be greater than the reproducibility limit

$$R = 2,77s_{R}$$

 $R = 2,77 \times 0,161 \ 4 = 0,45$

for products whose moisture content is between 10,00 % and 18,00 % (see Table A.1 and Figure A.1).

10.4 Comparison of two groups of measurements in one laboratory

Critical difference (CD) 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 7.1), the comparison of two moisture contents shall be made with the CD.

The CD between two averaged values obtained from two test results under repeatability conditions is given by Formula (3):

$$2.8 \, s_{\rm r} \, \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2.8 \, s_{\rm r} \, \sqrt{\frac{1}{2}} = 1.98 \, s_{\rm r} = 0.09 \, \text{w} \, 0.1 \tag{3}$$

where

 s_r is the standard deviation of repeatability;

 n_1 , n_2 are the numbers of test results corresponding to each of the averaged values.

10.5 Comparison of two groups of measurements in two laboratories

The CD between two averaged values obtained in two different laboratories from two test results under repeatability conditions is given by <u>Formula (4)</u>:

$$2.8\sqrt{s_{\rm R}^2 - s_{\rm r}^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2.8\sqrt{s_{\rm R}^2 - 0.5 s_{\rm r}^2} = 0.51 \times 0.5$$
 (4)

where

 s_r is the standard deviation of repeatability;

 s_R is the standard deviation of reproducibility;

 n_1 , n_2 are the numbers of test results corresponding to each of the averaged values.

10.6 Uncertainty

Uncertainty, $U_{\rm e}$, is a parameter characterizing the dispersion of values that can reasonably be attributed to the result. This uncertainty is established through the statistical distribution of results given by the interlaboratory test and characterized by the experimental standard deviation

$$U_{\rm e} = \pm 2 \, s_{\rm R} = \pm 0.30$$

 $u_{\rm e} = \pm 2 \, s_{\rm R} = \pm 0.30$
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 $u_{\rm e} = 0.30$

where $s_{\rm R}$ is the standard deviation of reproducibility.

11 Test report

The test report shall contain at least the following information:

- a) all information necessary for the complete identification of the sample;
- b) the sampling method used, if known;
- c) the method used, with reference to this document, i.e. ISO 712-1;
- d) the test result(s) obtained;
- e) if repeatability has been checked, the final result obtained;
- f) all operating details not specified in this document, or regarded as optional, as well as any incidents that may have influenced the test result(s);
- g) the date of the test.