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Cereals and cereal products — Determination of moisture content — Reference method

Céréales et produits céréaliers — Détermination de la teneur en eau — Méthode de référence

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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 712 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This fourth edition cancels and replaces the third edition (ISO 712:1998), which has been technically revised. (standards.iteh.ai)

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Cereals and cereal products — Determination of moisture content — Reference method

1 Scope

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

This International Standard 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 Terms and definitions STANDARD PREVIEW

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

2.1

moisture content

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mass loss undergone by a product under the conditions specified in this international Standard 4263d511993f/iso-712-2009

NOTE Moisture content is expressed as a percentage.

3 Principle

If necessary, a laboratory sample is ground, after conditioning, if required. 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 Annex B.

4 Apparatus

- **4.1** Analytical balance, capable of weighing to an accuracy of \pm 0,001 g.
- 4.2 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) enabling 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 comply with this requirement.

- d) tightness to air to avoid water exchange between sample and external air;
- e) adjustable so as to obtain particles of the dimensions indicated in Table 1.

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- **4.3 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 so as to give a mass per unit area of not more than 0,3 g/cm².
- **4.4 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 °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 °C to 133 °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.

4.5 Desiccator, containing an effective desiccant.

5 Sampling

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

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

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6 Preparation of the test/sample eh.ai/catalog/standards/sist/114e56aa-6bf8-460c-a7af-4263d511993f/iso-712-2009

6.1 Products not requiring grinding

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

Mix the laboratory sample thoroughly before taking the test portion (7.2).

Table 1 — Particle size characteristics of products not requiring grinding

Particle size characteristics	Proportion			
mm	%			
≤ 1,7 (1,8) ^a	100			
> 1,0 (1,0) ^b	≤ 10			
< 0,5 (0,56) ^a	≥ 50			

a Nominal size of openings, ISO 3310-1[1], that *does not retain* this particle size.

Nominal size of openings, ISO 3310-1[1], that *retains* this particle size.

6.2 Products requiring grinding

6.2.1 General

If the products do not have the particle size characteristics mentioned in Table 1, they shall be ground either without preconditioning (6.2.2) or with preconditioning (6.2.3), as required.

6.2.2 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 °C and a relative humidity of 40 % to 70 %.

Adjust the grinding mill (4.2) to obtain particles of the dimensions indicated in Table 1.

Then, quickly grind a quantity of the laboratory sample according to the apparatus used and at least slightly greater than that required for the test portion (about 5 g), and immediately proceed in accordance with 7.2.

6.2.3 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 slightly greater than 5 g (see 6.2.2). Record the mass of the test portion before preconditioning and dish as m'_2 . Pre-dry in accordance with 7.3, except that the time of heating in the oven (4.4) shall be 7 min to 10 min and the product shall be cooled to laboratory temperature with the dish (4.3) 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 slightly greater than 5 g (see 6.2.2). 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 Table 1, and immediately proceed in accordance with 6.2.2.

7 Procedure

7.1 Number of determinations

Carry out separate determinations on two test portions taken from the laboratory sample in accordance with 7.2 and 7.3. If the absolute difference between the two values obtained is greater than the repeatability limit given in Clause 9, repeat the determination until requirements are satisfied.

7.2 Test portion

Rapidly weigh, to the nearest 0,001 g, a quantity of 5 g \pm 1 g of the laboratory sample (6.2.2 or 6.2.3) in the dish (4.3). 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.

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7.3 Drying

Place the open dish containing the test portion (7.2), together with the lid, in the oven (4.4) 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 min \pm 5 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 (4.5). When several tests are being carried out, never place dishes on top of one another in the desiccator, but place them side by side.

7.4 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 .

8 Expression of results

8.1 Without preconditioning the STANDARD PREVIEW

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

$$w_{\text{H}_2\text{O}} = \left(1 - \frac{m_1}{m_0}\right) \times 100 \quad \text{https://standards.iteh.ai/catalog/standards/sist/114e56aa-6bf8-460c-a7af-4263d511993f/iso-712-2009}$$

where

 $m_0 = m'_0 - m_d$ is the mass, in grams, of the test portion (7.2);

 $m_1 = m'_1 - m_d$ is the mass, in grams, of the test portion after drying (7.4).

Calculate the arithmetic mean of two results satisfying the repeatability conditions (see 9.2). Round the result to two places of decimals.

8.2 With preconditioning

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

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

where

 $m_2 = m'_2 - m_d$ is the mass, in grams, of the sample taken before preconditioning (6.2.3);

 $m_3 = m'_3 - m_d$ is the mass, in grams, of the preconditioned sample (6.2.3);

Calculate the arithmetic mean of two results satisfying the repeatability conditions (see 9.2). Round the result to two places of decimals.

9 Precision

9.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in 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.

9.2 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

$$r = 2,77s_r$$

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

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

9.3 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

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$$R = 2.77s_R$$
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for products whose moisture content is between 10,00% and 18,00% (see Table A.1 and Figure A.1).

9.4 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 6.1), 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:

$$2.8 \ s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2.8 \ s_r \sqrt{\frac{1}{2}} = 1.98 \ s_r = 0.09 \approx 0.1$$

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.

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:

$$2.8 \sqrt{s_R^2 - s_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2.8 \sqrt{s_R^2 - 0.5 s_r^2} = 0.51 \approx 0.5$$

where

is the standard deviation of repeatability; S_r

is the standard deviation of reproducibility; S_R

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

9.6 Uncertainty

Uncertainty, U_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

$$U_e = \pm 2 s_R = \pm 0.30$$
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where s_R is the standard deviation of reproducibility dards.iteh.ai)

10 Test report

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The test report shall contain at least the following information:

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

Annex A (informative)

Results of interlaboratory test

The repeatability, reproducibility, and critical difference of the method were established in an interlaboratory test conducted in accordance with the requirements of ISO 5725-1 $^{[2]}$, ISO 5725-2 $^{[3]}$ and ISO 5725-6 $^{[4]}$.

In this test, 19 laboratories took part. Seven products were analysed.

The statistical results of the study are presented in Table A.1 and in Figure A.1.

Table A.1 — Statistical results of the interlaboratory test

	Products								
Parameters	Semolina	Common wheat 1	Barley	Rice	Durum wheat 1	Durum wheat 2	Common wheat 2	Overall mean	
No. of participating laboratories after eliminating outliers	17	18	18	17	18	14	14		
Mean value, \overline{w}_{H_2O} , g/100 g iTeh ST	A10,50	A11,91	12,67	14,17	14,59	16,92	17,87		
Repeatability standard deviation, s_r	angla	rd,95.it	eb,04a	0,04	0,03	0,06	0,05	0,04	
Coefficient of variation of repeatability, $C_{V,r}$ ($s_r / \overline{w}_{\rm H_2O}$), %	0,29 _{ISC}	0,42	0,32	0,28	0,21	0,35	0,28		
Repeatability limit, $r(2,77 \frac{https://standards.lieh.}{s_r}$	ai/catalog/st 0,08 4263d511	andards/sist 0,14)93†/iso-712	/114e56aa 2-2009	i-6b18-4 0,11	60c-a7af- 0,08	0,17	0,14		
Reproducibility standard deviation, s_R	0,13	0,13	0,17	0,16	0,21	0,19	0,14	0,16	
Coefficient of variation of reproducibility, $C_{V,R}$ ($s_R/\overline{w}_{\rm H_2O}$) %	1,24	1,09	1,34	1,13	1,44	1,12	0,78		
Reproducibility limit, R (2,77 s _R)	0,36	0,36	0,47	0,44	0,58	0,53	0,39		