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Cereals and cereal products — Determination of moisture content —

Part 2: Automatic drying oven method

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

Partie 2: Méthode par séchage en étuve automatique

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Foreword

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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).

A list of all parts in the ISO 712 series can be found on the ISO website.

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Cereals and cereal products — Determination of moisture content —

Part 2: Automatic drying oven method

1 Scope

This document specifies an automatic method for the reference method (see ISO 712-1) for the determination of moisture content of cereals and cereal products using an automatic drying oven.

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

The method does not apply to maize and pulses.

NOTE For moisture content determination in maize, see ISO 6540, and for pulses, see ISO 24557.

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 712-1, *Cereals and cereal products — Determination of moisture content — Part 1: Reference method*

3 Terms and definitions

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

ISO and IEC maintain terminology 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
loss of mass experienced by a product

Note 1 to entry: The moisture content is determined under the conditions specified in this document.

Note 2 to entry: Moisture content is expressed as a percentage.

4 Principle

A laboratory sample is milled, where necessary, once conditioned. A test portion is automatically dried and weighed by an automatic drying oven at a temperature between 130 °C and 133 °C. Due to the continuous air flow within the drying chamber of the automatic drying oven, the drying process takes considerably less time than in a conventional drying chamber without ventilation.

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

5.1 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 (the difference in temperature before and after grinding is ≤ 5 °C);

NOTE A grinding mill fitted with a cooling device can conform to 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.

5.2 Metal dish, non-corrodible under the test conditions, or **glass dish**, 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/cm^2. The dish shall be used without a lid, because the dish with portion is placed into the drying chamber directly after weighing.

5.3 Automatic drying oven, electrically heated and including an analytical balance, 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 of 130 °C to 133 °C in the vicinity of the test portions and is capable of weighing to an accuracy of ± 0,001 g.

The automatic drying oven shall automatically reweigh the sample dishes after the set drying time. The moisture content shall be calculated and saved by the software operating the oven.

The automatic drying oven shall be constructed in such a way that its short openings have no influence on the temperature in the drying chamber (130 °C to 133 °C) or that after the insertion of a sample, a temperature of 130 °C can be reached again in less than 5 min, so that test portions can be dried simultaneously.

6 Sampling

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

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 the particle size characteristics indicated in Table 1 may be used without grinding.

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

Table 1 — Particle size characteristics of products not requiring grinding

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

[a] Nominal size of openings (see ISO 3310-1) that does not retain this particle size.

[b] Nominal size of openings (see ISO 3310-1) 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 Table 1, they shall be ground without preconditioning (see 7.2.2).

7.2.2 Grinding

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. If the moisture content of the sample is outside this range, preconditioning in accordance with ISO 712-1 shall be applied.

NOTE The range of moisture content 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 (5.1) 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 10 g), and immediately proceed in accordance with 8.2.

8 Procedure

8.1 Number of determinations

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

8.2 Test portion

By using an automatic drying oven, a quantity of 10 g ± 1 g can be weighed in the dish (5.2) depending on the manufacturer's recommendations. The mass of the undried test portion m_0 and the dish are recorded automatically in the software. The tare of the dry dish and its mass m_d is recorded in the software.

8.3 Drying

Since an automatic drying oven is used, place the dish containing the test portion (8.2) in the automatic drying oven and proceed with drying until a constant mass is achieved.

Depending on the automatic drying oven and the material used, the drying period can be reduced to up to 60 min ± 5 min, which is sufficient time for the test portions to attain a constant mass. Review these times regularly by running a drying curve.

If the requirements of the automatic drying oven (5.3) are fulfilled (i.e. the time to heat up again after opening when bringing in a new sample), additional samples can be placed in the drying chamber during a running test.

8.4 Weighing

By using an automatic drying oven, the weighing starts immediately after the set drying time in the drying chamber using the integrated balance to the nearest 0,001 g. An additional cooling step within a desiccator is not necessary. The mass of the dried test portion m_1 is recorded automatically in the software.

9 Calculation of moisture content

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

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

where

m_0 is the mass, in grams, of the test portion (8.2);

m_1 is the mass, in grams, of the test portion after drying (8.4);

Since an automatic drying oven is used, the calculation is made automatically by software with the arithmetic mean of two results satisfying the repeatability conditions (see 10.2). The calculation result is automatically rounded 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 Annex A. The values derived from this interlaboratory test can only be applied to moisture contents in the range 8 % to 14 % 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 following repeatability limit for products whose moisture content is between 8,00 % and 14,00 % (see Table A.1 and Figure A.1):

— $r = 2{,}77 s_r$

— $r = 2{,}77 \times 0{,}026 = 0{,}07$

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 following reproducibility limit for products whose moisture content is between 8,00 % and 14,00 % (see Table A.1 and Figure A.1):

— $R = 2{,}77 s_R$

— $R = 2{,}77 \times 0{,}109 = 0{,}30$

10.4 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 shown in Formula (2):

$$U_e = \pm 2\, s_R = \pm 2 \times 0{,}109 = \pm 0{,}22 \tag{2}$$

where s_R is the standard deviation of reproducibility.

10.5 Comparison of two groups of measurements in one laboratory

The critical difference, D_r, between two averaged values each obtained in one laboratory from two test results under repeatability conditions is equal to Formula (3):

$$C_{Dr} = 1{,}98 * s_r = 1{,}98 * 0{,}026 = 0{,}05 \tag{3}$$

where s_r is the standard deviation of repeatability.

10.6 Comparison of two groups of measurements in two laboratories

The critical difference, D_R, between two averaged values each obtained in two different laboratories from two test results under repeatability conditions is equal to Formula (4):

$$D_R = 2{,}8\sqrt{{s_R}^2 - {s_r}^2\left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}8\sqrt{{s_R}^2 - 0{,}5 \times {s_r}^2} \tag{4}$$

$$D_R = 2{,}8\sqrt{0{,}109^2 - 0{,}5 \times 0{,}0255^2} = 0{,}30$$

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 712-2;

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 can have influenced the test result(s);

g) the date of the test.

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, ISO 5725-2 and ISO 5725-6.

In this test, 19 laboratories took part. Eight products with moisture contents between 8 % and 14 % were analysed. Prior to the actual test, a homogeneity test was conducted, and the performance of the balances was checked.

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

Table A.1 — Statistical results of the interlaboratory test

Parameter	Product								Overall mean
	Semolina	Common wheat 1	Common wheat 2	Wheat groats	Spelt groats	Oat groats	Spelt flour	Rye flour	
No. of participating laboratories after eliminating outliers	18	17	18	19	19	18	19	18	
Mean value, $\bar{w}_{H_2O}$, g/100 g	11,35	12,07	13,83	8,56	11,21	9,88	12,11	11,83	
Repeatability standard deviation, s_r	0,03	0,02	0,02	0,04	0,04	0,02	0,02	0,02	0,026
Coefficient of variation of repeatability, $C_{V,r}$ (s_r / $\bar{w}_{H_2O}$), %	0,71	0,36	0,40	1,34	1,05	0,52	0,52	0,37	
Repeatability limit, r (2,77 s_r)	0,08	0,04	0,05	0,11	0,12	0,05	0,06	0,04	
Reproducibility standard deviation, s_R	0,13	0,11	0,06	0,15	0,15	0,11	0,07	0,08	0,109
Coefficient of variation of reproducibility, $C_{V,R}$ (s_R / $\bar{w}_{H_2O}$), %	3,21	2,51	1,31	4,90	3,81	3,20	1,72	1,86	
Reproducibility limit, R (2,77 s_R)	0,36	0,30	0,18	0,42	0,43	0,32	0,21	0,22	

The regression line for the repeatability standard deviation, s_r is:

$$s_r = -0{,}010\,\bar{w}_{H_2O} + 0{,}187$$

$$R^2_{\bar{w}_{H_2O},\,s_r} = 0{,}283$$

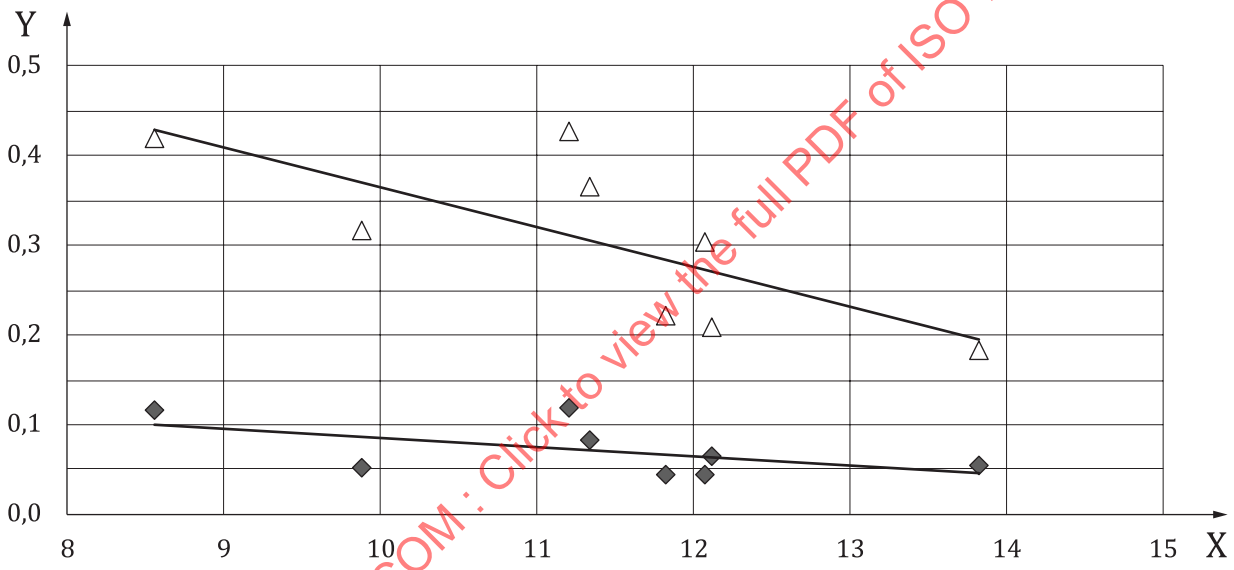
where $R_{\bar{w}_{H_2O},\,s_r}$ is the correlation coefficient.

The regression line for the reproducibility standard deviation, s_R is:

$$s_R = -0{,}044\,\bar{w}_{H_2O} + 0{,}810$$

$$R^2_{\bar{w}_{H_2O},\,s_R} = 0{,}543$$

where $R_{\bar{w}_{H_2O},\,s_R}$ is the correlation coefficient.



Key

X water content (%)

Y standard deviation

◆ repeatability

△ reproducibility

NOTE The repeatability (black) and reproducibility (grey) standard deviation do not vary with the mean moisture content and are considered constant to 0,03 and 0,11, respectively.

Figure A.1 — Accuracy values versus mean values