# BHUTAN STANDARD

## CEREALS AND CEREAL PRODUCTS-
## DETERMINATION OF MOISTURE CONTENT-
## REFERENCE METHOD.

ICS 67.060



XXXX, 2019 Price Group C

# BTS 291: XXXX ISO 712: 2009

## NATIONAL FOREWORD

This Bhutan Standard which is identical with ISO 712: 2009 CEREALS AND CEREAL PRODUCTS-DETERMINATION OF MOISTURE CONTENT-REFERENCE METHOD Standard issued by the International Organization for Standardization was adopted by Bhutan Standards Bureau by Food and Agriculture Technical Committee (TC 02) and approved by the Bhutan Standards Bureau Board (BSB Board) on xxxx, 2019.

The text of the ISO Standard has been approved as suitable for publication as Bhutan Standard without deviation. Certain conventions are however, not identical to those used in Bhutan Standard.

Attention is particularly drawn to the following:

a) Where the words “ISO Standard” appear referring to this standard, they should be read as “Bhutan Standard”.

b) Wherever page numbers are quoted, they are “ISO Standard” page numbers.

# INTERNATIONAL STANDARD

# ISO 712

Fourth edition
2009-11-15



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

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

Reference number
ISO 712:2009(E)

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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

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

**2.1**
**moisture content**
mass loss undergone by a product under the conditions specified in this International Standard

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 ± 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.

**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/cm$^2$.

**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].

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.

## 6 Preparation of the test sample

### 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<br>mm | Proportion<br>% |
|---|---|
| $\leqslant$ 1,7 (1,8)[a] | 100 |
| $>$ 1,0 (1,0)[b] | $\leqslant$ 10 |
| $<$ 0,5 (0,56)[a] | $\geqslant$ 50 |

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

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

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

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

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

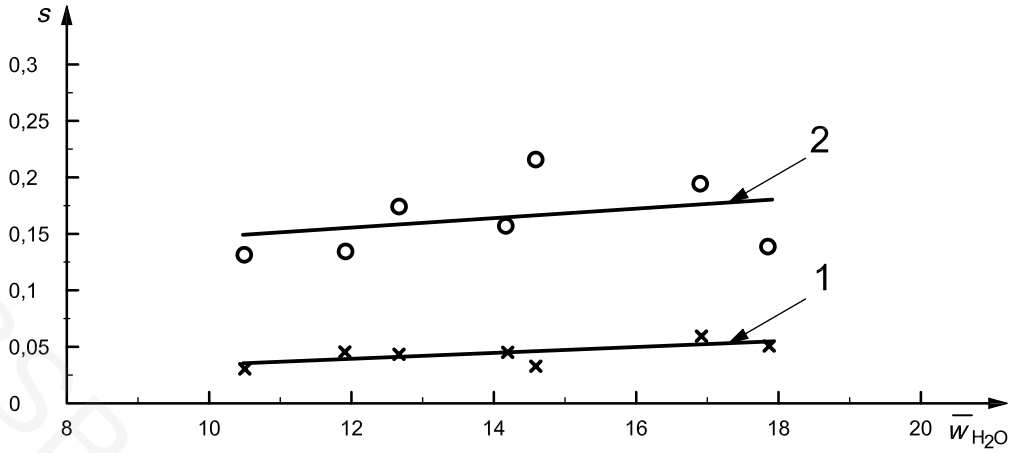
$$w_{H_2O} = \left[(m_0 - m_1)\frac{m_3}{m_0} + m_2 - m_3\right] \times \frac{100}{m_2} = \left(1 - \frac{m_1\, m_3}{m_0\, m_2}\right) \times 100$$

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.




**Key**

$s$ standard deviation

$\overline{w}_{H_2O}$ mean moisture content

1 Regression line for the standard deviation of repeatability, $s_r$

$s_r = 0{,}002\,4\ \overline{w}_{H_2O} + 0{,}008\,9$

$r^2_{\overline{w}_{H_2O}\, s_r} = 0{,}443\,5$

where $r_{\overline{w}_{H_2O}\, s_r}$ is the correlation coefficient

2 Regression line for the standard deviation of reproducibility, $s_R$

$s_R = 0{,}003\,9\ \overline{w}_{H_2O} + 0{,}106\,6$

$r^2_{\overline{w}_{H_2O}\, s_R} = 0{,}107\,2$

where $r_{\overline{w}_{H_2O}\, s_R}$ is the correlation coefficient

**Figure A.1 — Accuracy values versus mean values**

# Annex B
(informative)

# Cereals and cereal products — Determination of moisture content Absolute method

## B.1 Scope

This annex describes the absolute method for the determination of the actual moisture content of cereals and cereal products[1)] against which the routine reference method specified in this International Standard has been elaborated.

The method is not applicable to maize, for which an identical method, called the absolute method, is specified in ISO 6540:1980[5], Annex A.

This absolute method, which necessitates the employment of special equipment and experienced analysts, is therefore only suitable for use in specialized laboratories, and is intended to serve as a standard for checking and perfecting other methods for the determination of moisture content. It is not intended to be used for settling commercial disputes.

## B.2 Terms and definitions

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

**B.2.1**
**true moisture content**
mass loss undergone by a product under the conditions specified in this annex

NOTE True moisture content is expressed as a percentage.

## B.3 Principle

If necessary, a laboratory sample is ground, after any conditioning required. A test portion is dried under reduced pressure, at a temperature between 45 °C and 50 °C, in the presence of a desiccant, until constant mass is reached.

## B.4 Apparatus

**B.4.1 Analytical balance**.

**B.4.2 Apparatus for reducing pressure** to 1,3 kPa to 2,6 kPa[2)], e.g. a water pump.

**B.4.3 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;

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1) This method has been applied successfully to: wheat, rice (paddy, husked, and milled), barley, millet, rye and oats, in the form of grains, milled grains, semolina or flour.

2) 1,3 kPa to 2,6 kPa = 13 mbar to 26 mbar = 10 mmHg to 20 mmHg.

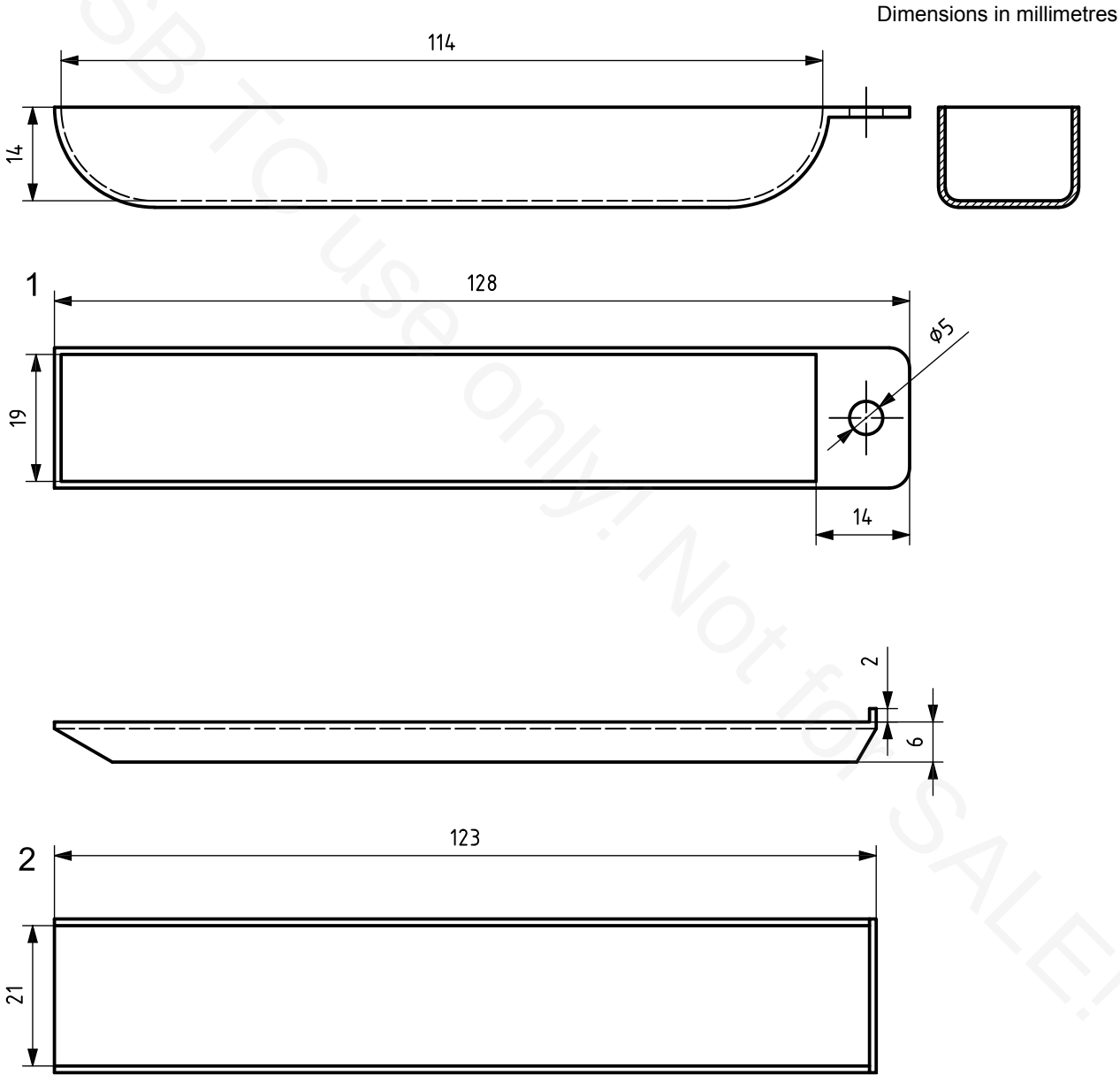
c) enabling grinding to be carried out rapidly and uniformly, without appreciable development of heat;

d) as far as possible, tightness to outside air;

e) adjustable so as to obtain particles of the dimensions indicated in B.6.1.1.

**B.4.4 Metal dish**, non-corrodible under the test conditions, with a sufficiently tight-fitting lid and having an effective surface area so as to allow the test portion to be distributed in a layer having a mass per unit area of not more than 0,3 g/cm$^2$.

See Figure B.1.




**Key**

1 metal dish

2 lid

NOTE The dish shown has a flat bottom of effective surface 16 cm$^2$ and an internal height of 14 mm. It can be used with the drying tube shown in Figure B.2.

**Figure B.1 — Diagram of suitable metal dish and lid (for guidance only)**

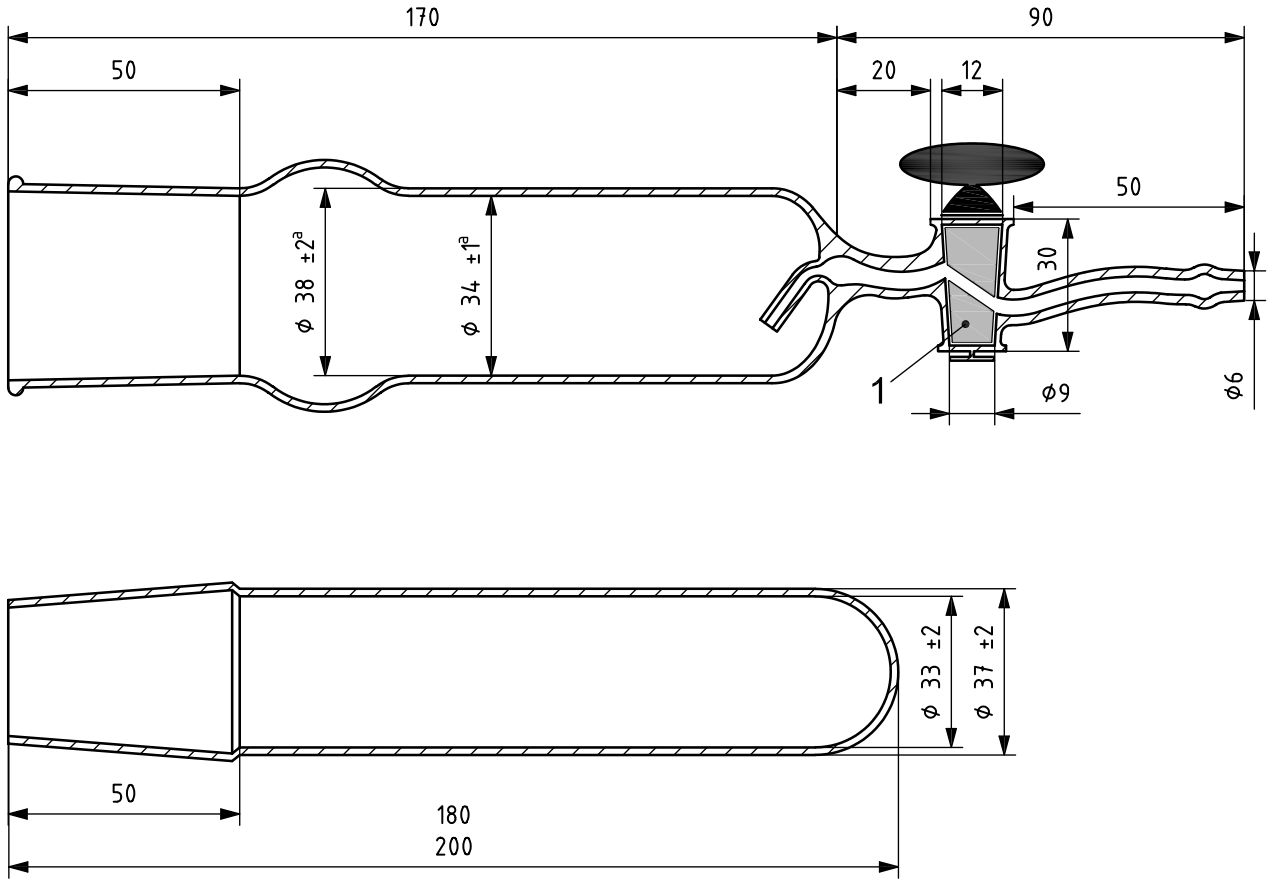
**B.4.5 Cup**, made from glass or porcelain.

**B.4.6 Drying tube**, of glass, in two parts, one of which, intended to receive the dish (B.4.4), is closed at one end, while the other, intended to receive the cup (B.4.5), carries a semi-capillary tube, with a stopcock, for evacuation purposes. The two parts are connected by a ground-glass joint.

The test portion may be cooled in this apparatus after drying, a desiccator (B.4.9) being then unnecessary for this operation.

See Figure B.2.

Dimensions in millimetres




**Key**

1 olive

[a] The drying tube shown in the diagram has a 40/50 ground-glass joint (40 mm in diameter at the large end, and having a length of the ground portion of 50 mm). It is suitable for use with the dish shown in Figure B.1. The olive ending to the stopcock side arm may be replaced by a ground-glass joint.

**Figure B.2 — Diagram of suitable drying tube (for guidance only)**

**B.4.7 Constant-temperature oven**, electrically heated, enabling the part of the drying tube (B.4.6) containing the dish (B.4.4) to be maintained at a temperature between 45 °C and 50 °C.

**B.4.8 Air-drying train**: gas-washing bottle containing pure analytical grade sulfuric acid (relative density, $d_{20} \geqslant 1{,}83$ g/ml), connected to a tube containing pure analytical grade phosphorus pentaoxide spread on glass wool.

**B.4.9 Desiccator**, containing an efficient desiccant.

## B.5 Sampling

See ISO 24333[6].

## B.6 Procedure

### B.6.1 Preparation of the test sample

#### B.6.1.1 Unground products

Products having particles of sizes less than or equal to 1,7 mm, where the mass fraction of particles with sizes over 1 mm is less than 10 % and where the mass fraction of particles with sizes less than 0,5 mm is more than 50 %, do not need to be ground before the determination.

Mix the laboratory sample thoroughly before taking the test portion (B.6.2.1).

#### B.6.1.2 Ground products

If the sample does not comply with the particle size characteristics mentioned in B.6.1.1, grind it either without preconditioning (B.6.1.2.1) or with preconditioning (B.6.1.2.2).

##### B.6.1.2.1 Grinding without preconditioning

For products which are not likely to undergo variations in moisture content in the course of grinding [in general, products with a moisture content between 7 % and 17 %[3)] (see B.8.1)], carry out grinding without preconditioning.

Adjust the grinding mill (B.4.3) to obtain particles of the dimensions indicated in B.6.1.1, grind a small quantity of the laboratory sample and discard it.

Then quickly grind about 3,5 g of the laboratory sample, and immediately proceed in accordance with B.6.2.2.

##### B.6.1.2.2 Grinding with preconditioning

Products which are likely to undergo changes in moisture content in the course of grinding (in general, products with a moisture content greater than 17 %[3)]) shall be preconditioned so as to bring their moisture content to between 7 % and 17 %[3)] [if possible between 9 % and 15 % (see B.8.1)], before grinding.

If the moisture content is more than 17 %[3)] (the more frequent case), weigh, to the nearest 0,2 mg, about 3,5 g of the laboratory sample. Record the mass as $m'_2$. Calculate the mass of the test portion before preconditioning, $m_2$, as the difference between $m'_2$ and the mass of the dish, $m_{\text{d.}}$

Then pre-dry the test portion in accordance with 7.3, except that the drying time shall be 1,5 to 2 h (see B.8.2) and it is unnecessary to renew the phosphorus pentaoxide.

If the moisture content is less than 7 %, prepare a test portion of about 3,5 g of the laboratory sample. Weigh it in the dish to the nearest 0,2 mg. Record the mass as $m'_2$. Calculate the mass of the test portion before preconditioning, $m_2$, as the difference between $m'_2$ and the mass of the dish, $m_{\text{d.}}$ Then place the test portion and dish in a suitable atmosphere (usually that of the laboratory) and leave it to acquire a moisture content within the limits specified above.

After conditioning, weigh the sample to the nearest 0,2 mg. Record the mass as $m'_3$. Calculate the mass of the test portion after preconditioning, $m_3$, as the difference between $m'_3$ and the mass of the dish, $m_{\text{d}}$. Grind the test portion immediately in the grinding mill (B.4.3), adjusted to obtain particles of the dimensions indicated in B.6.1.1, and immediately proceed in accordance with B.6.2.2.

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3) For oats and rice (paddy, husked and milled rice), 15 % mass fraction.

### B.6.2 Test portion

**B.6.2.1** For products not requiring grinding, rapidly weigh, to the nearest 0,2 mg, about 3 g of the test sample (B.6.1.1) in the dish (B.4.4), previously dried and weighed, together with its lid, to the nearest 0,2 mg. Record the mass as $m'_0$. Calculate the mass of the test portion, $m_0$, as the difference between $m'_0$ and the mass of the dish, $m_d$.

**B.6.2.2** For products which have had to be ground, rapidly weigh all the grindings obtained (6.2.2 or 6.2.3) in the dish (B.4.4), previously dried and weighed, together with its lid, to the nearest 0,2 mg. Record the mass as $m'_0$. Calculate the mass of the test portion, $m_0$, as the difference between $m'_0$ and the mass of the dish, $m_d$.

### B.6.3 Drying

Place the open dish (leaving its lid in the desiccator) containing the test portion (B.6.2) at the closed end of a drying tube (B.4.6); introduce, near to it, the cup (B.4.5) containing a layer of phosphorus pentaoxide about 10 mm thick. Fit the two parts of the drying tube together and reduce the pressure in the assembled tube to a value of the order of 1,3 kPa to 2,6 kPa, using the vacuum apparatus (B.4.2); this should be done gradually in order to avoid material being thrown out of the dish. Close the connection to the vacuum apparatus, and place the part of the tube containing the test portion in the oven (B.4.7), maintained at 45 °C to 50 °C (see B.8.4).

When phosphorus pentaoxide agglomerates at the surface, renew it after restoring atmospheric pressure inside the drying tube by causing air, which has passed through the drying train (B.4.8), to enter slowly through the semi-capillary tube. Reduce the pressure in the drying tube again and continue the drying as before.

After about 100 h, take the tube out of the oven, allow it to cool to laboratory temperature and restore atmospheric pressure inside it as described above. Disconnect the two parts of the tube, quickly remove the dish, cover, and weigh it to the nearest 0,2 mg. Record the mass as $m'_1$. Calculate the mass of the test portion after drying, $m_1$, as the difference between $m'_1$ and the mass of the dish, $m_d$.

Repeat the operations specified above until the mass is practically constant (i.e. until the difference between two successive weighings at an interval of 48 h is less than 0,6 mg) (see B.8.3).

### B.6.4 Number of determinations

Carry out two determinations on test portions taken from different test samples but from the same laboratory sample.

## B.7 Expression of results

### B.7.1 Method of calculation and formulae

#### B.7.1.1 Without preconditioning

The actual moisture content, $w_{H_2O}$, expressed as a percentage by mass of the product as received, is given by:

$$w_{H_2O} = \frac{m_0 - m_1}{m_0} \times 100$$

where

$m_0$ is the mass, in grams, of the test portion (B.6.2.1 or B.6.2.2);

$m_1$ is the mass, in grams, of the test portion after drying (B.6.3).

Calculate the arithmetic mean of two results satisfying the repeatability condition (see B.7.2). If the repeatability condition is not met, repeat the determination.

Express the result to two places of decimals.

#### B.7.1.2 With preconditioning

The actual moisture content, $w_{H_2O}$, expressed as a percentage by mass of the product as received, is given by:

$$w_{H_2O} = \frac{1}{m_2}\left[(m_0 - m_1)\frac{m_3}{m_0} + m_2 - m_3\right] \times 100$$

$$= 100 \times \left(1 - \frac{m_1\, m_3}{m_0\, m_2}\right)$$

where

$m_0$ is the mass, in grams, of the test portion (B.6.2.2);

$m_1$ is the mass, in grams, of the test portion after drying (B.6.3);

$m_2$ is the mass, in grams, of sample taken before preconditioning (B.6.1.2.2);

$m_3$ is the mass, in grams, of the preconditioned sample (B.6.1.2.2).

Calculate the arithmetic mean of two results satisfying the repeatability condition (see B.7.2). If the repeatability condition is not met, repeat the determination.

Express the result to two places of decimals.

### B.7.2 Repeatability

The difference between the values obtained from two determinations (see B.6.4) carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,10 g of moisture per 100 g of sample.

NOTE With a little practice, differences of less than 0,05 g of moisture per 100 g of sample can be obtained in the same laboratory.

## B.8 Notes on procedure

**B.8.1** The range of moisture contents given for conditioning products before grinding corresponds approximately to a laboratory atmosphere of temperature 20 °C and relative humidity 40 % to 70 %. It should be modified for other atmospheric conditions.

**B.8.2** The duration of pre-drying is given only for guidance. Check that it enables the desired conditioning to be obtained with the apparatus and the products used.

**B.8.3** The drying period is of the order of 150 h at least.

**B.8.4** A coloration at the surface of the phosphorus pentaoxide indicates the loss of traces of volatile organic substances from the test portion. With certain deteriorated products, if the coloration becomes sufficiently pronounced, it is expedient to reduce the temperature of heating.

## B.9 Test report

The test report shall contain at least the following information:

a) the method used, including a reference to this annex of this International Standard;

b) the result obtained;

c) all operating details not specified in this International Standard, or regarded as optional, as well as any incidents which may have influenced the result;

d) all details required for the complete identification of the sample, and in particular the date on which the analysis was carried out.

# Bibliography

[1] ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

[5] ISO 6540:1980, *Maize — Determination of moisture content (on milled grains and on whole grains)*

[6] ISO 24333, *Cereals and cereal products — Sampling*

[7] ISO 24557, *Pulses — Determination of moisture content — Air-oven method*

**ICS 67.060**

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