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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

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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.

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 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, 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.

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 ± 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.

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.

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

$$r = 2{,}77 s_r$$

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

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

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

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

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

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.

9.5 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:

$$2{,}8\sqrt{s_R^2 - s_r^2\left(1-\frac{1}{2n_1}-\frac{1}{2n_2}\right)} = 2{,}8\sqrt{s_R^2 - 0{,}5\,s_r^2} = 0{,}51 \approx 0{,}5$$

where

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.

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

$$U_e = \pm 2\, s_R = \pm 0{,}30$$

where s_R is the standard deviation of reproducibility.

10 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 International Standard;

d) the test result(s) obtained;

e) if repeatability has been checked, the final result obtained;

f) all operating details not specified in this International Standard, or regarded as optional, as well as any incidents that may have influenced the test result(s).

Annex A
(informative)

Results of interlaboratory test

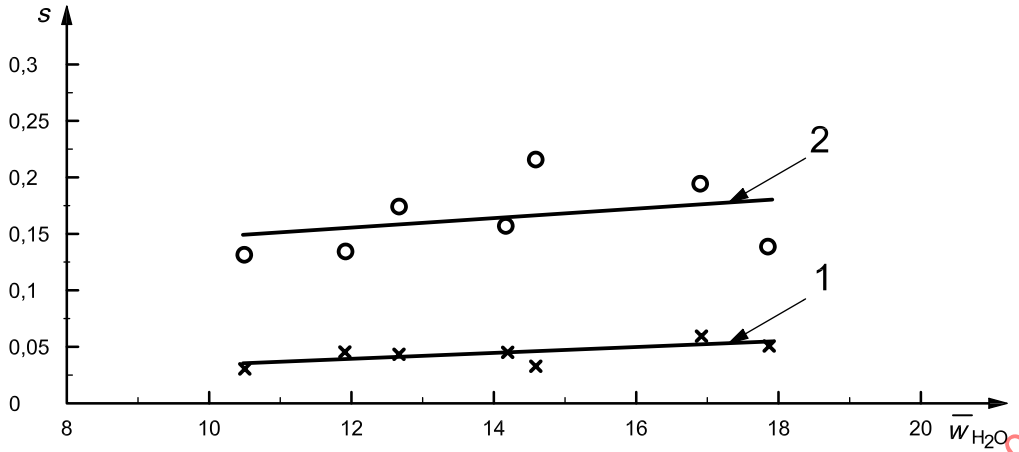
The repeatability, reproducibility, and critical difference of the method were established in an interlaboratory test conducted in accordance with the requirements of ISO 5725-1[2], ISO 5725-2[3] and ISO 5725-6[4].

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

Parameters	Products							
	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	10,50	11,91	12,67	14,17	14,59	16,92	17,87	
Repeatability standard deviation, s_r	0,03	0,05	0,04	0,04	0,03	0,06	0,05	0,04
Coefficient of variation of repeatability, $C_{V,r}$ $(s_r / \overline{w}_{H_2O})$, %	0,29	0,42	0,32	0,28	0,21	0,35	0,28	
Repeatability limit, r (2,77 s_r)	0,08	0,14	0,11	0,11	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, $C_{V,R}$ $(s_R / \overline{w}_{H_2O})$ %	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	



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;

---

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.

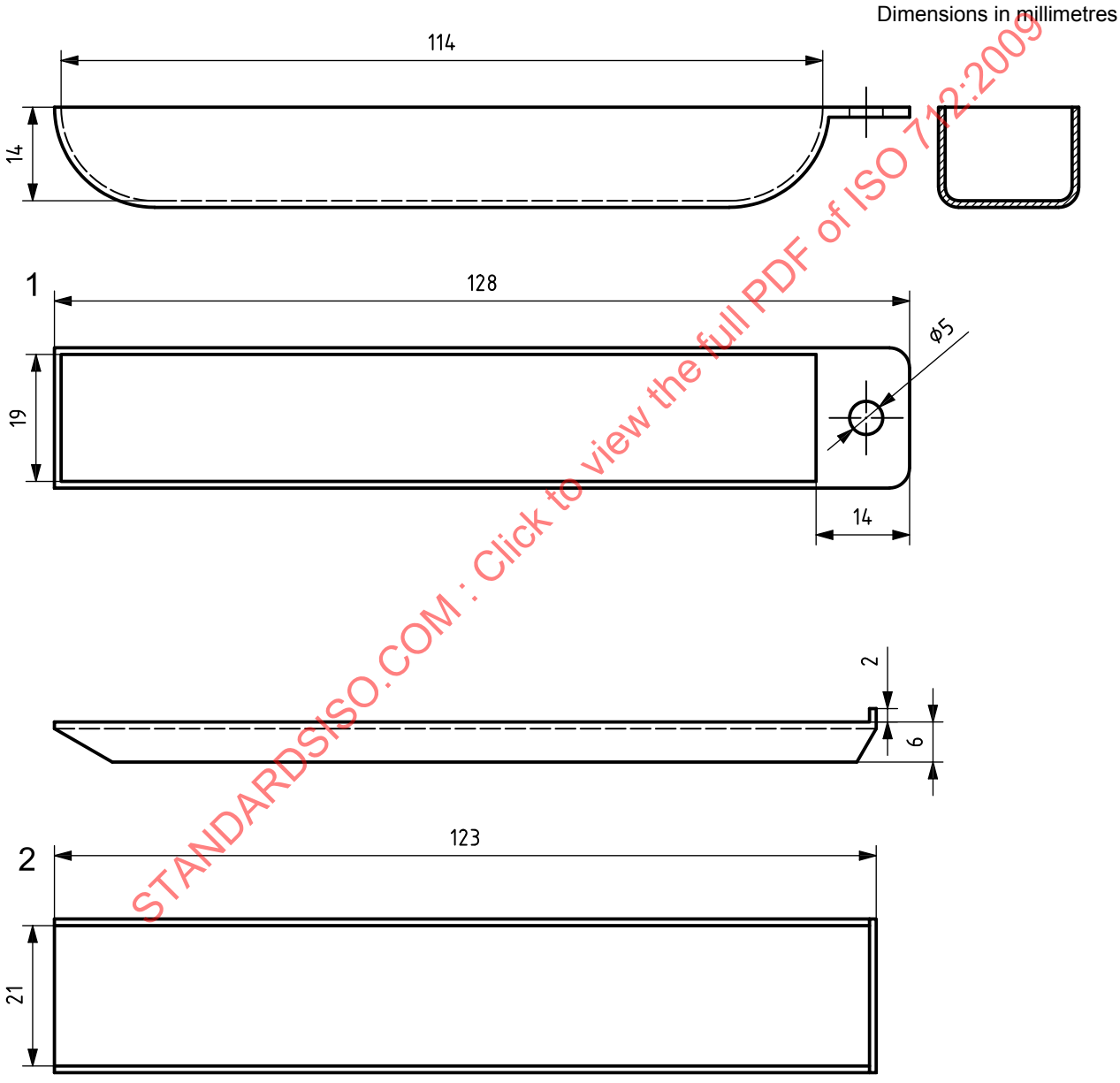
c) enabling grinding to be carried out rapidly and uniformly, without appreciable development of heat;

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)

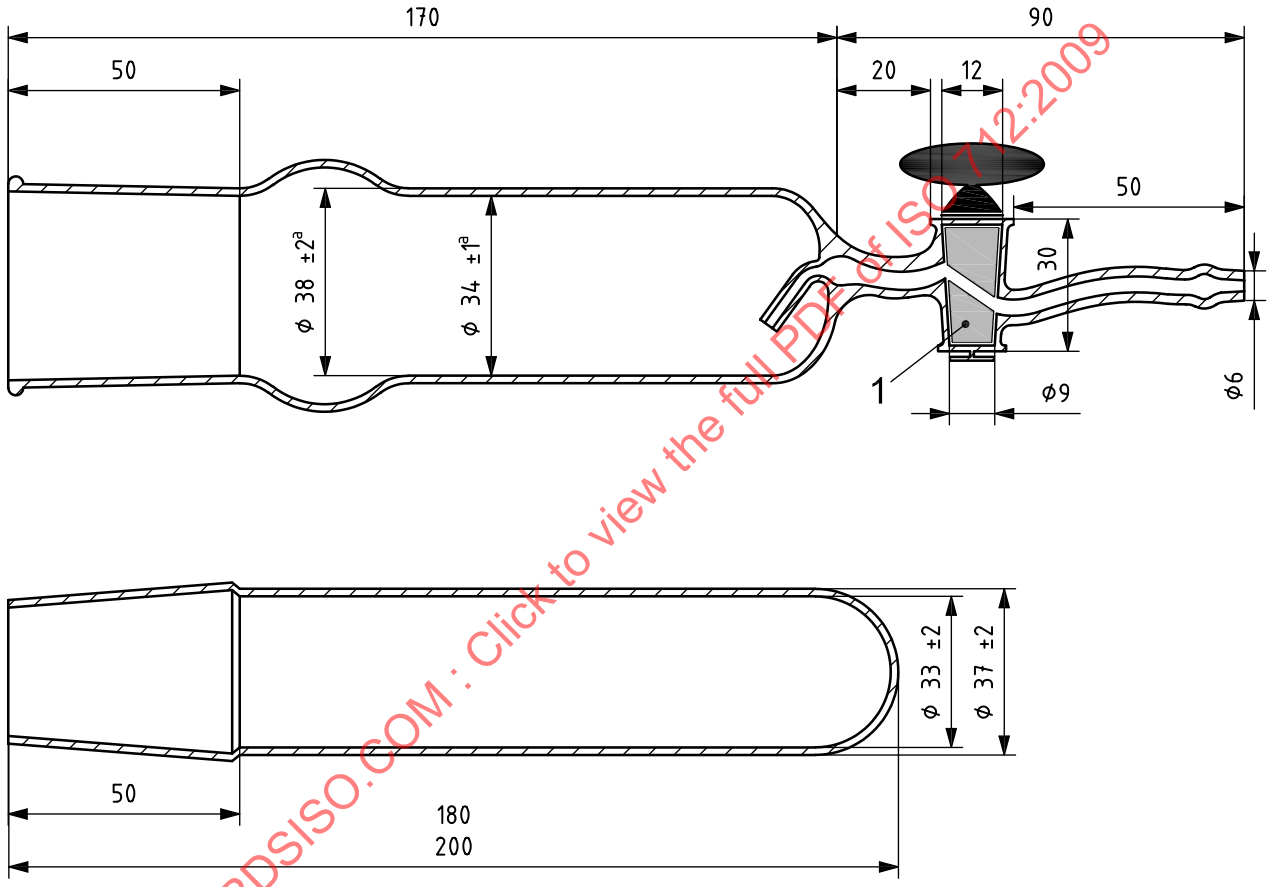
B.4.5 Cup, made from glass or porcelain.

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_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_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_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.

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).