TC 34

INTERNATIONAL STANDARD 711

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Cereals and cereal products — Determination of moisture content (Basic reference method)

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

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 711 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in November 1977.

It has been approved by the member bodies of the following countries :

Australia	India	South Africa, Rep. of
Austria	Iran	Spain
Bulgaria	Israel	Thailand
Canada	Kenya	Turkey
Chile	Mexico	United Kingdom
Czechoslovakia	New Zealand	U.S.S.R.
Egypt, Arab Rep. of	Portugal	Yugoslavia
France	Romania	

The member body of the following country expressed disapproval of the document on technical grounds :

Ireland

This International Standard cancels and replaces ISO Recommendation R 711-1968, of which it constitutes a technical revision.

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Cereals and cereal products — Determination of moisture content (Basic reference method)

0 INTRODUCTION

The basic reference method specified in this International Standard ensures complete removal of moisture from the product, as has been demonstrated by tests of reversibility and addition of moisture, while avoiding any alteration in its chemical composition, particularly oxidation and loss of volatile organic substances.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the basic reference method for the determination of the moisture content of cereals and cereal products.[1)]

The method is not applicable to maize.

This basic reference 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 routine methods for the determination of moisture content (see particularly ISO 712). It is not intended to be used for settling commercial disputes.

2 REFERENCES

ISO 712, *Cereals and cereal products — Determination of moisture content (Routine reference method).*[2)]

ISO/R 950, *Cereals — Sampling (as grain).*

3 DEFINITION

moisture content : The loss in mass, expressed as a percentage, undergone by the product under the conditions specified in this International Standard.

4 PRINCIPLE

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

5 APPARATUS

5.1 Analytical balance.

5.2 Apparatus for reducing pressure to 1,3 to 2,6 kPa*, for example a water pump.

5.3 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;

c) enabling grinding to be carried out rapidly and uniformly, without appreciable development of heat and, as far as possible, without contact with the outside air;

d) adjustable so as to obtain particles of the dimensions indicated in 7.1.1.

5.4 Metal dish,[3)] non-corrodible under the test conditions, with a sufficiently tight-fitting lid and having an effective surface area such 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 .

5.5 Glass or porcelain boat.

5.6 Drying tube,[4)] of glass, in two parts, one of which, intended to receive the dish (5.4), is closed at one end, while the other, intended to receive the boat (5.5), 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 (5.9) being then unnecessary for this operation.

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

2) At present at the stage of draft. (Revision of ISO/R 712.)

3) See clause A.1 of the annex for a diagram (for guidance only) of a suitable metal dish.

4) See clause A.2 of the annex for a diagram (for guidance only) of a suitable drying tube.

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

5.7 Constant temperature oven, electrically heated, enabling the part of the drying tube (5.6) containing the dish (5.4) to be maintained at a temperature between 45 and 50 °C.

5.8 Air-drying train : gas-washing bottle containing pure analytical grade sulphuric acid ($\rho_{20} \geqslant 1{,}83$ g/ml), connected to a tube containing pure analytical grade phosphorus(V) oxide spread on glass wool.

5.9 Desiccator, containing an efficient desiccant.

6 SAMPLING

See ISO/R 950.

7 PROCEDURE

7.1 Preparation of the test sample

7.1.1 *Products not requiring to be ground*

Products having particles of sizes less than or equal to 1,7 mm, less than 10 % (*m/m*) being over 1 mm and more than 50 % (*m/m*) being less than 0,5 mm, do not need to be ground before the determination.

Well mix the laboratory sample before taking the test portion (7.2.1).

7.1.2 *Products requiring to be ground*

If the sample does not have the particle size characteristics mentioned in 7.1.1, it shall be ground either without pre-conditioning (7.1.2.1) or with pre-conditioning (7.1.2.2).

7.1.2.1 Grinding without pre-conditioning

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 % (*m/m*)[1] (see 9.1)], carry out grinding without pre-conditioning.

Adjust the grinding mill (5.3) to obtain particles of the dimensions indicated in 7.1.1, grind a small quantity of the laboratory sample and discard this quantity.

Then quickly grind about 3,5 g of the laboratory sample, and immediately proceed in accordance with 7.2.2.

7.1.2.2 Grinding with pre-conditioning

Products which are likely to undergo changes in moisture content in the course of grinding [in general, products with a moisture content more than 17 % (*m/m*)[1] or less than 7 % (*m/m*)] shall be pre-conditioned to bring their moisture content to between 7 and 17 % (*m/m*)[1] [if possible 9 to 15 % (*m/m*)] (see 9.1) before grinding.

If the moisture content is more than 17 % (*m/m*)[1] (the more frequent case), weigh, to the nearest 0,2 mg, about 3,5 g of the laboratory sample and pre-dry it in accordance with 7.3, except that the drying time shall be 1,5 to 2 h (see 9.2.) and it is unnecessary to renew the phosphorus(V) oxide.

If the moisture content is less than 7 % (*m/m*), place about 3,5 g of the laboratory sample, weighed to the nearest 0,2 mg, in a suitable atmosphere (usually that of the laboratory) and leave it to acquire a moisture content within the limits specified above.

Weigh the pre-conditioned sample to the nearest 0,2 mg, grind it immediately in the grinding mill (5.3), adjusted to obtain particles of the dimensions indicated in 7.1.1, and immediately proceed in accordance with 7.2.2.

7.2 Test portion

7.2.1 For products not requiring to be ground, rapidly weigh, to the nearest 0,2 mg, about 3 g of the test sample (7.1.1) into the dish (5.4), previously dried and weighed, together with its lid, to the nearest 0,2 mg.

7.2.2 In the case of products which have had to be ground, rapidly weigh all the grindings obtained (7.1.2.1 or 7.1.2.2) into the dish (5.4), previously dried and weighed, together with its lid, to the nearest 0,2 mg.

7.3 Drying

Place the open dish (leaving its lid in the desiccator) containing the test portion (7.2) at the closed end of the drying tube (5.6); introduce, near to it, the boat (5.5) containing a layer of phosphorus(V) oxide about 1 cm 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 to 2,6 kPa, using the vacuum apparatus (5.2); this should be done gradually (for example, by the use of a semi-capillary tube) 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 (5.7), controlled at 45 to 50 °C (see 9.4).

When the phosphorus(V) oxide agglomerates at the surface, renew it after restoring atmospheric pressure inside the drying tube by causing air, which has passed through the drying train (5.8), 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.

1) 15 % (*m/m*) in the case of oats.

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 9.3).

7.4 Number of determinations

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

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formulae

The moisture content, expressed as a percentage by mass of the product as received, is given by the following formulae :

a) *without pre-conditioning* :

$$(m_0 - m_1)\frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (7.2.1 or 7.2.2);

m_1 is the mass, in grams, of the test portion after drying (7.3).

b) *with pre-conditioning* :

$$\left[(m_0 - m_1)\frac{m_3}{m_0} + m_2 - m_3\right]\frac{100}{m_2}$$

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

where

m_0 is the mass, in grams, of the test portion (7.2.2);

m_1 is the mass, in grams, of the test portion after drying (7.3);

m_2 is the mass, in grams, of sample taken before pre-conditioning (7.1.2.2);

m_3 is the mass, in grams, of the pre-conditioned sample (7.1.2.2).

Take as the result the arithmetic mean of the two values obtained, if the requirement for repeatability (see 8.2) is satisfied.

8.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,10 g of moisture per 100 g of sample. If it does so, the determinations shall be repeated.

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

9 NOTES ON PROCEDURE

9.1 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 different atmospheric conditions.

9.2 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 cereals used.

9.3 The drying period is of the order of 150 h at least.

9.4 A coloration at the surface of the phosphorus(V) oxide 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.

10 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention all operating details not specified in this International Standard, or regarded as optional, as well as any incidents which may have influenced the result.

The report shall include all details required for complete identification of the sample, and in particular the date on which the analysis was carried out.

ANNEX

SUITABLE DISH AND DRYING TUBE

A.1 DISH (5.4)

The dish shown in the diagram below has a flat bottom of effective surface 16 cm^2 and an internal height of 14 mm. It may be used with the drying tube shown in clause A.2.

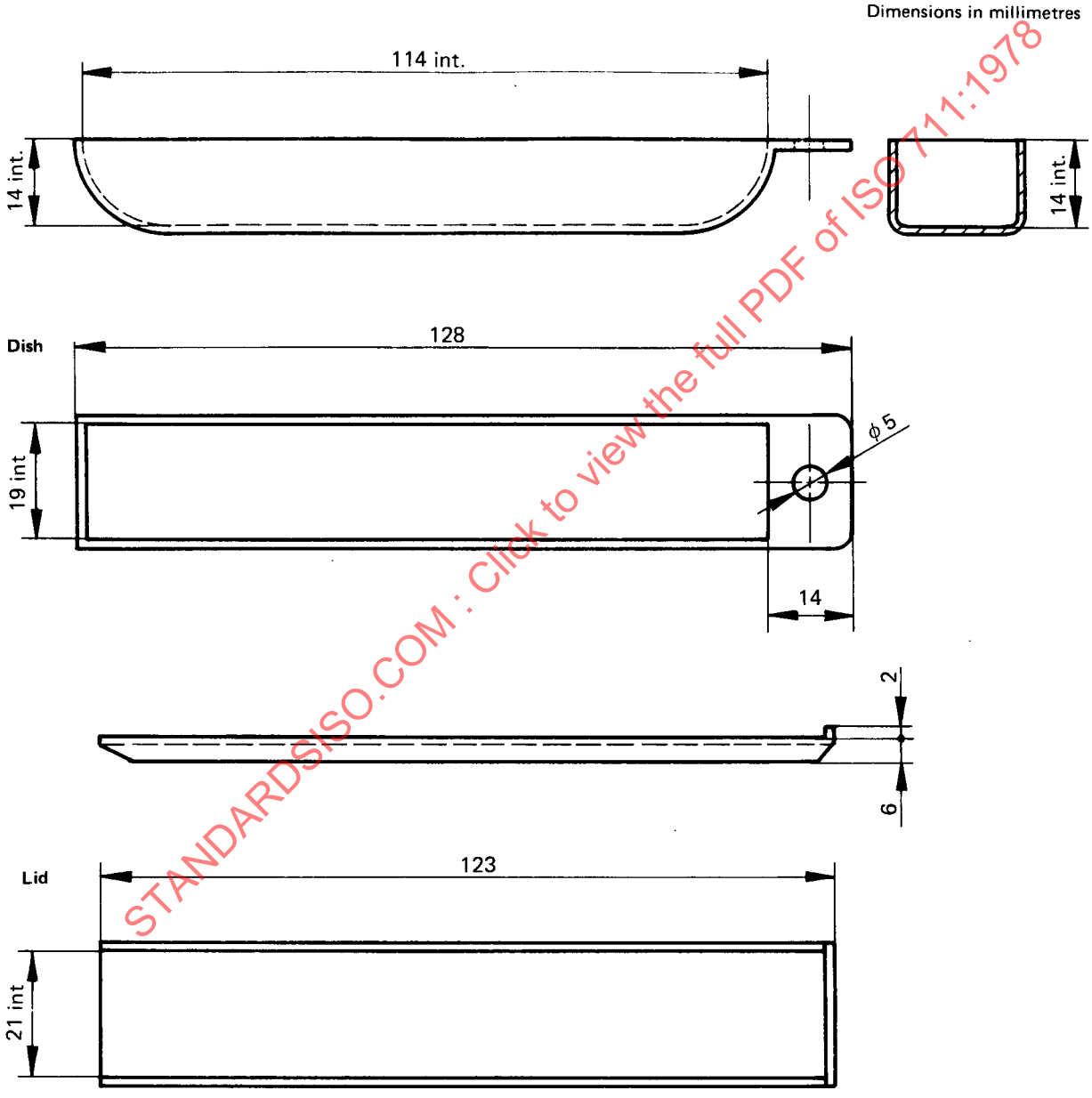
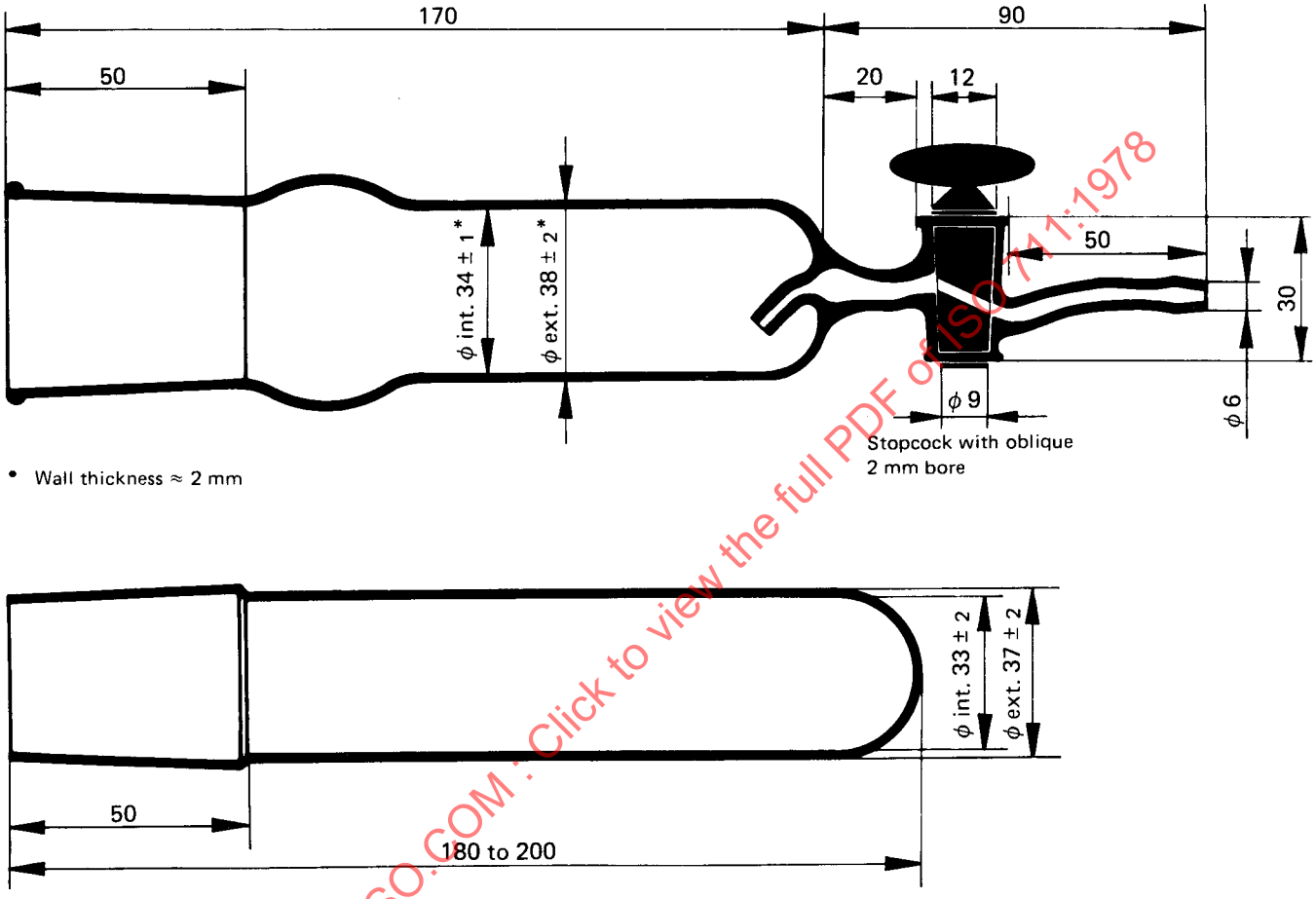


FIGURE 1 — **Diagram of suitable metal dish and lid (for guidance only)**

A.2 DRYING TUBE (5.6)

The drying tube shown in the diagram below has a 40/50 ground joint (40 mm large end diameter, 50 mm length of ground portion). It is suitable for use with the dish described in clause A.1. The olive ending to the stopcock side arm may be replaced by a ground glass joint.

Dimensions in millimetres



• Wall thickness ≈ 2 mm

FIGURE 2 — **Diagram of suitable drying tube (for guidance only)**