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R 711

CEREALS AND CEREAL PRODUCTS

DETERMINATION OF MOISTURE CONTENT

(Basic reference method)

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 711, *Cereals and cereal products – Determination of moisture content. (Basic reference method),* was drawn up by Technical Committee ISO/TC 34, *Agricultural food products,* the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question by the Technical Committee began in 1960 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In March 1966, this Draft ISO Recommendation (No. 908) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Bulgaria	Iran	U.A.R.
Colombia	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.S.R.
Finland	Norway	Yugoslavia
France	Poland	
Germany	Portugal	

One Member Body opposed the approval of the Draft :

Chile

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council which decided, in April 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 711 April 1968

CEREALS AND CEREAL PRODUCTS

DETERMINATION OF MOISTURE CONTENT

(Basic reference method)

INTRODUCTION

This ISO Recommendation has been established to serve as the basic reference method with which to compare routine or other test procedures for the determination of the moisture content of cereals and cereal products. The method is designed to avoid any chemical alteration in the substance, particularly oxidation and loss of volatile organic substances, while ensuring complete removal of all moisture from the product. Tests of reversibility and the addition of moisture indicate that, in the present state of knowledge, the method may be regarded as enabling the true moisture content of cereals and cereal products to be obtained.

1. SCOPE

1.1 This ISO Recommendation describes the basic reference method for the determination of water in cereals and cereal products.

1.2 **Field of application**

This method is intended to serve as a standard for checking and perfecting methods which are particularly suitable for the routine determination of moisture in cereals and cereal products. It is not intended to be used for settling commercial disputes.

NOTE. – For simplicity, in the following sections the term *product* is used to designate either a cereal or a cereal product.

2. DEFINITION

The *moisture content* is defined as the loss in mass, expressed as a percentage of the mass of the original sample, undergone by the product under the conditions specified in this ISO Recommendation.

3. PRINCIPLE

Determination of the loss in mass when the product, if necessary ground without change in moisture content, is brought into equilibrium with a dry atmosphere at a temperature between 45 and 50 °C, at a pressure of 13 to 26 mbar (10 to 20 mmHg).

4. APPARATUS

4.1 *Analytical balance*

4.2 *Apparatus for obtaining a pressure of 13 to 26 mbar (10 to 20 mmHg),* e.g. a water pump.

4.3 *Grinding mill*

- made of material which does not absorb moisture,
- easy to clean and having as little dead space as possible,
- 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,
- adjustable so as to obtain particles of the dimensions indicated in clause 5.1.1.

4.4 *Metal dish,* non-corrodible, with a sufficiently tight-fitting lid, the effective surface enabling the test portion to be distributed so as to give not more than 0.3 g/cm^2.*

4.5 *Glass or porcelain boat.*

4.6 *Drying tube***, of glass, of which one part is closed at one end and the other part carries a semi-capillary tube, with a stopcock, for evacuation purposes. The two parts are connected by a ground joint.

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

4.7 *Constant temperature oven,* electrically heated, or any other system enabling the part of the drying tube containing the dish (4.4) to be brought to a temperature between 45 and 50 °C.

4.8 *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 pentoxide spread on glass wool.

4.9 *Desiccator,* containing an efficient desiccant.

5. PROCEDURE

Carry out weighings to the nearest 0.0002 g.

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* See Annex A.1 for a diagram (for guidance only) of a metal dish.

** See Annex A.2 for a diagram (for guidance only) of a drying tube.

5.1 Preparation of sample

5.1.1 *Products not requiring to be ground*

Products having particles of sizes below or equal to 1.7 mm, less than 10 % by mass being over 1 mm and more than 50 % by mass being less than 0.5 mm, do not need to be ground before the determination.

5.1.2 *Products requiring to be ground*

If the sample does not comply with the particle size characteristics mentioned above, it requires to be ground either with or without pre-conditioning.

5.1.2.1 GRINDING WITHOUT PRE-CONDITIONING

This applies to products which are not likely to undergo variations in moisture content in the course of grinding; in general, grains with a moisture content between 7 and 17 %.

Adjust the grinding mill (4.3) to obtain particles of the dimensions indicated in clause 5.1.1, grind a small quantity of the product and reject this.

Then quickly grind about 3.5 g of the sample.

Transfer the grindings to the previously dried and tared dish (4.4); quickly close the latter, and weigh it. Then carry out the determination.

The time between taking the sample and weighing before drying should be less than 2 minutes, if a small mill of the classical cone or hammer type is used.

5.1.2.2 GRINDING WITH PRE-CONDITIONING

Products which are too dry (moisture content less than 7 %) or too moist (moisture content more than 17 %) need to be suitably humidified or pre-dried before they are ground.

For products with moisture content less than 7 %, re-humidify the sample by placing it in a suitable atmosphere so as to bring the final moisture content to between 7 and 17 % (if possible between 9 and 15 %).

More frequently, it is necessary to pre-dry the grain, this being generally done when the moisture content is above 17 %, in order to bring it within the range of 7 to 17 % (if possible between 9 and 15 %). Weigh about 3.5 g of the sample and carry out the drying operation according to the instructions in clause 5.3, except that the drying time is 1 1/2 to 2 hours at most and it is not then necessary to renew the phosphorus pentoxide.

Reweigh the sample after conditioning (M_1) and immediately grind it in the mill (4.3) previously adjusted. Transfer the grindings to the dish (4.4). Reweigh (M_2), ensuring that less than 2 minutes elapse between the two weighings M_1 and M_2. Then carry out the determination.

5.2 Test portion

5.2.1 For products not requiring to be ground (see clause 5.1.1), carry out the following operations rapidly

- introduce about 3 g of the substance into the metal dish (4.4), which has been tared after being heated for a time in the oven (4.7) and cooled to laboratory temperature in the desiccator (4.9).
- close the dish and weigh it.

5.2.2 For products requiring to be ground (see clause 5.1.2), use as the test portion the grindings in the dish (4.4) after the latter is closed and weighed (see clauses 5.1.2.1 or 5.1.2.2).

5.3 Determination

Put the open dish (4.4), containing the test portion, at the closed end of the drying tube (4.6); introduce, near to it, a boat (4.5) containing a layer of phosphorus pentoxide about 1 cm thick. Fit the two parts of the drying tube together; bring the pressure in the assembled tube to a value of the order of 13 to 26 mbar (10 to 20 mmHg). 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. Put the portion of the tube containing the dish in the oven (4.7) at 50 °C.

When the phosphorus pentoxide agglomerates, renew it after restoring atmospheric pressure inside the drying tube (4.6) by causing air, which has passed through the drying train (4.8), to enter slowly through the semi-capillary tube. Close the drying tube again and continue the drying under vacuum at 50 °C, as before.

After about 100 hours, take the tube out of the oven, allow it to cool to laboratory temperature and restore atmospheric pressure inside it as described above. Quickly take out the dish, cover it and weigh it.

Continue the dehydration to constant mass (less than 0.0006 g difference between weighings made 48 hours apart).

Carry out at least two determinations on the same sample.

6. EXPRESSION OF RESULTS

6.1 Method of calculation and formulae

The moisture content, as a percentage by mass of the product as received, is equal to

- without preliminary conditioning

$$(M_0 - M_3) \times \frac{100}{M_0}$$

- with preliminary conditioning

$$\left[(M_2 - M_3) \times \frac{M_1}{M_2} + M_0 - M_1\right] \times \frac{100}{M_0} = 100\left(1 - \frac{M_1 \times M_3}{M_0 \times M_2}\right)$$

where

M_0 is the initial mass, in grammes, of the test portion;

M_1 is the mass, in grammes, of the test portion after conditioning;

M_2 is the mass, in grammes, of the test portion after grinding;

M_3 is the mass, in grammes, of the dry test portion.

Take as the result the arithmetic mean of the determinations, if the requirement of clause 6.2 is satisfied.

6.2 Repeatability

The difference between two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed 0.1 g of moisture per 100 g of sample. If it does so, the determination should be repeated in duplicate.

With a little practice, differences less than 0.05 g of moisture per 100 g of sample are obtained in a single laboratory.

7. NOTES ON PROCEDURE

7.1 The range of moisture content given for conditioning cereal grains before grinding corresponds approximately to a laboratory atmosphere at 20 °C and 40 to 70 % relative humidity. It would be reasonable to modify it for different atmospheric conditions.

7.2 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. The drying period is of the order of 150 hours at least.

7.3 A coloration at the surface of the phosphorus pentoxide indicates the loss of traces of volatile organic substances. This indication serves as an "alarm bell". With certain deteriorated products, if the coloration becomes sufficiently pronounced, it is expedient to reduce the temperature of heating.

Renew the phosphorus pentoxide as soon as it agglomerates at the surface.

8. TEST REPORT

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

The report should include all details required for complete identification of the sample.

ANNEX A

A.1 Dish for test portion

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 Figure 2.

Dimensions in millimetres

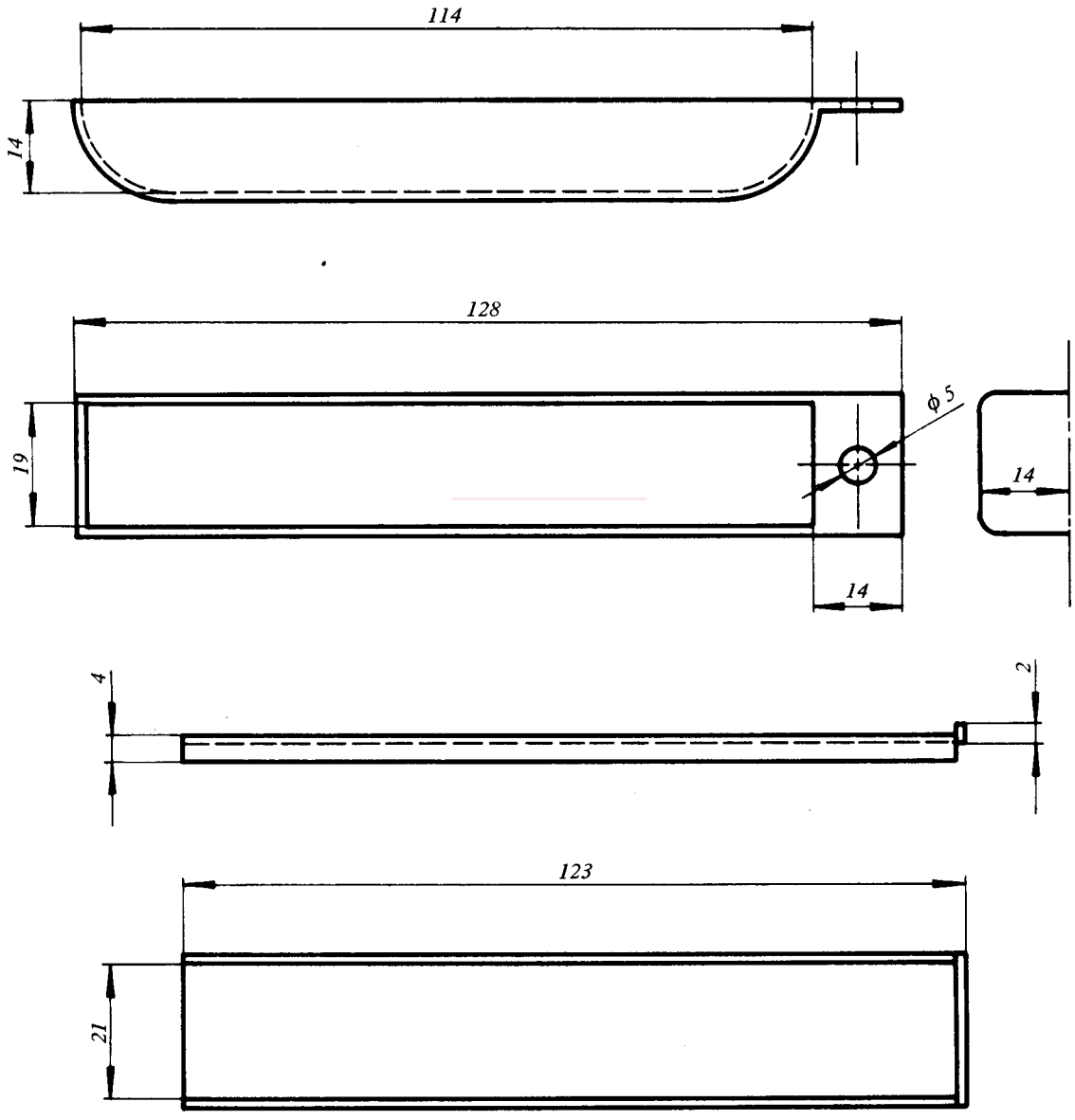

FIG. 1 – **Diagram of a metal dish (for guidance only)**

A.2 Drying tube

The drying tube shown in the diagram below has a 40/50 ground joint (40 mm in diameter, 50 mm length of ground portion). It is suitable for use with the dish described in Figure 1. The olive connection may be replaced by a ground joint.

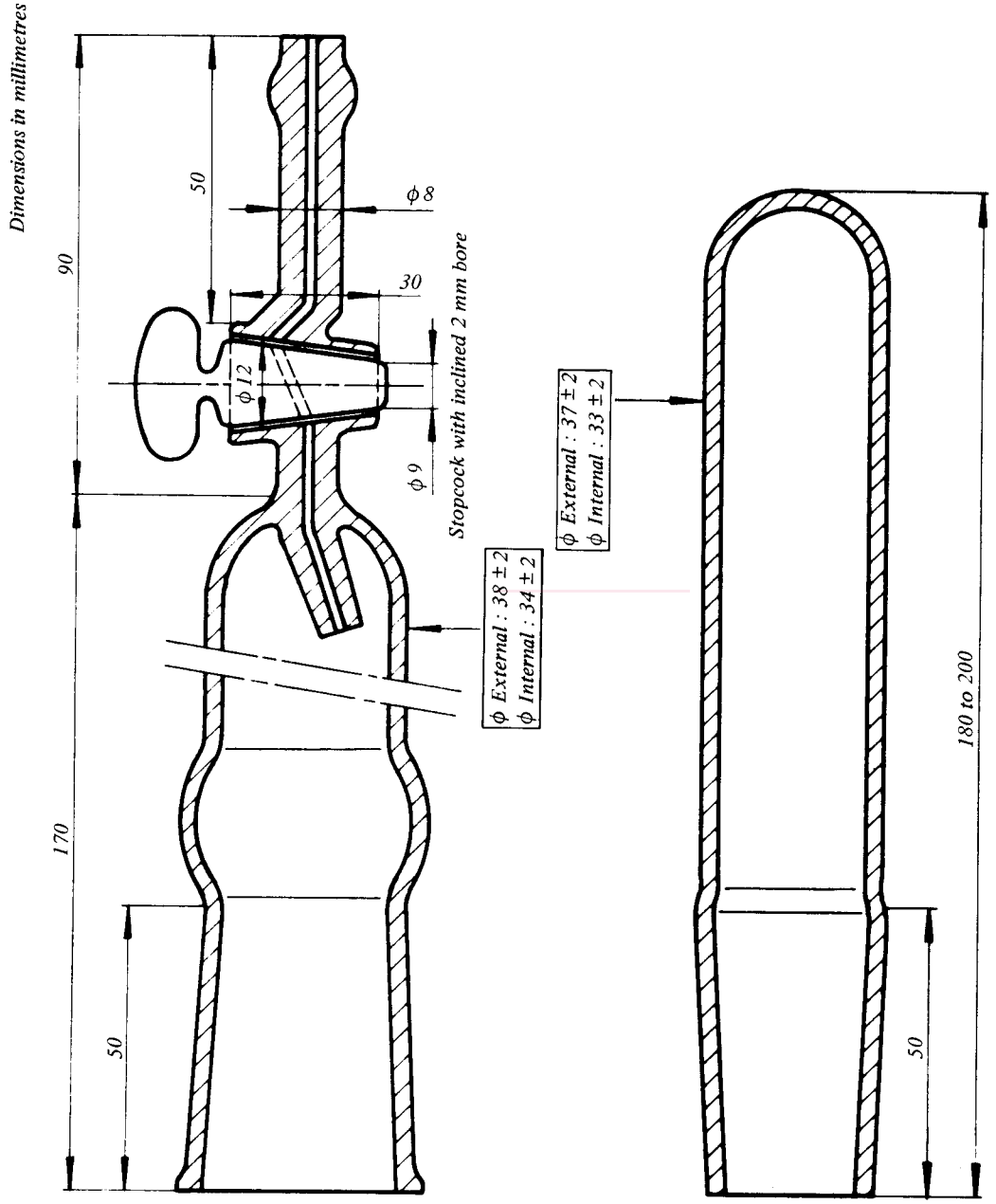

FIG. 2 – Diagram of a drying tube (for guidance only)