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Second edition
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Maize — Determination of moisture content (on milled grains and on whole grains)

Maïs — Détermination de la teneur en eau (sur grains broyés et sur grains entiers)

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Contents

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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).

This second edition cancels and replaces the first edition (ISO 6540:1980), which has been technically revised. The main changes compared with the previous edition are as follows:

— Clauses 7 to 10 and 17 to 20 (now 4.5 to 4.9 and 5.4 to 5.7) and the annexes have been revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The basic reference method and the routine reference method relating to cereals (see ISO 712) are only applicable to other cereals than maize and cereal products. Therefore, this document has been developed to specify the two methods for maize on the basis of research works published in 1979[4].

The basic reference method for maize, which is called the "absolute method", requires special equipment and experienced personnel, and can only be applied in specialized laboratories.

Due to the very high moisture content that can be present in samples of maize (sometimes greater than a mass fraction of 40 %) and because of the size and texture of the grains, the determination of the moisture in maize raises problems with regard to its grinding and pre-drying.

Consequently, to allow the pre-drying and grinding to be avoided, this document also describes a routine method for whole grains. which is easier to use and allows working in series. Its response time is longer but the workload is lower, because of the absence of grinding. However, this practical whole grain method has a positive bias of about a mass fraction of 0, 30 % compared to the reference method.

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Maize — Determination of moisture content (on milled grains and on whole grains)

1   Scope

This document specifies two methods:

— a reference method for the determination of the moisture content of maize grains and ground whole maize, groats, grits and maize flour, see Clause 4;

— a routine method for the evaluation of the moisture content of maize in whole grains, see Clause 5.

The latter is not suitable for use for experts' reports, or for calibration or checking of humidity meters, because of its significant bias to the reference method (see Table B.3).

2   Normative references

There are no normative references in this document.

3   Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
moisture content of maize
loss in mass undergone by a product under specified conditions

Note 1 to entry: It is expressed as a percentage.

4   Reference method

4.1   Principle

If necessary, grinding of a sample, after pre-conditioning, if required. Drying of a test portion at a temperature between 130 °C and 133 °C, under conditions that enable a result to be obtained in agreement with that obtained by the absolute method (see Annex A).

4.2   Apparatus

4.2.1   Analytical balance, able to weight with an accuracy of ±0,001 g and therefore having a display accuracy of 0,000 1 g.

4.2.2   Analytical balance, able to weight with an accuracy of ±0,1 g and therefore having a display accuracy of 0,01 g.

4.2.3   Grinding mill, having the following characteristics:

a)   made of material that does not absorb moisture;

b)   easy to clean and having as little dead space as possible;

c)   enabling grinding of 30 g of maize grains 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 4.4.1.

4.2.4   Metal boat, without lid, with an effective surface area enabling 100 g of maize grains to be distributed in a single layer.

4.2.5   Metal capsule, of suitable dimensions, non-corrodible under the test conditions, or, failing this, a **glass dish**, with a sufficiently tight-fitting lid, and having an effective surface area such as to allow distribution of the test portion with no more than 0,3 g per square centimetre.

4.2.6   Constant-temperature oven, electrically heated, adjustable between 60 °C and 80 °C, and with adequate ventilation.

4.2.7   Constant-temperature oven, electrically heated, capable of being controlled in such a way that the temperature of the air and of the shelves carrying the test portions is within the range of 130 °C to 133 °C in the neighbourhood of the test portions, in normal working condition.

The oven shall have a heat capacity such that, when initially adjusted to a temperature of 131 °C, it can again reach this temperature in less than 45 min (preferably 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, with a maximum particle size of 1 mm, as the test material. The ventilation shall be such that, after inserting all the test portions that the oven can hold and drying at a temperature of 130 °C to 133 °C, the results after a heating period of 2 h and then a further 1 h will not differ by more than 0,15 g of moisture per 100 g of sample.

4.2.8   Desiccator, containing an efficient desiccant.

4.3   Sampling

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

The laboratory should be provided with a truly representative sample, in a sealed package, that is undamaged and unmodified during transport and storage.

4.4   Preparation of the test sample

4.4.1   Products not requiring to be ground

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

Mix the laboratory sample thoroughly before taking the test portion (see 4.5.3).

4.4.2 Products requiring to be ground

4.4.2.1 General

If the laboratory sample does not have the particle size characteristics mentioned in 4.4.1, it shall be ground either without pre-conditioning (4.4.2.2) or with pre-conditioning (4.4.2.3) as required.

4.4.2.2 Grinding without pre-conditioning

For products that are not likely to undergo variations in moisture content in the course of grinding (in general, products with a moisture content between a mass fraction of 9,00 % and 15,00 %, see 4.8), carry out grinding without pre-conditioning.

Adjust the grinding mill (4.2.3) to obtain particles of the dimensions indicated in 4.4.1.

Then quickly grind about 30 g of the laboratory sample, mix with a spatula and proceed immediately as specified in 4.5.1.

4.4.2.3 Grinding with pre-conditioning

Products that are likely to undergo changes in moisture content in the course of grinding (in general, products with a moisture content more than a mass fraction of 15,00 % or less than a mass fraction of 9,00 %) shall be pre-conditioned to bring their moisture content to between a mass fraction of 9,00 % and 15,00 %, see 4.8) before grinding.

If the moisture content is greater than a mass fraction of 15,00 % (which is the more frequent case), weigh, to the nearest 0,1g, about 100 g of the laboratory sample in the metal boat (4.2.4), place this in the oven (4.2.7) controlled at between 60 °C and 80 °C, and leave it for the time necessary to bring the moisture content to between a mass fraction of 9,00 % and 15,00 %. Take the boat out of the oven and allow it to stand in the laboratory atmosphere for the time necessary (at least 2 h) for the pre-conditioned sample to return to the laboratory temperature and for the moisture distribution to be relatively uniform. During this rest, it shall be ensured that no addition or withdrawal of material is made to the contents of the boat. If necessary, cover it with a sheet of paper but not with a lid, since this could limit the exchange of moisture between the air and the grain.

After conditioning, weigh the sample to the nearest 0,1 g, then, proceeding rapidly, grind about 30 g of this product. Mix using a spatula.

If the moisture content is less than a mass fraction of 9,00 %, place about 100 g of the laboratory sample, weighed to the nearest 0,1 g, in a suitable atmosphere (usually that of the laboratory) and leave it until a moisture content within the limits specified above is obtained.

4.5 Procedure

4.5.1 Number of determinations

For each laboratory sample, carry out the determination in duplicate.

Carry out one determination on each of the two ground test portions taken from the laboratory sample, in accordance with 4.5.2 to 4.5.4. If the absolute difference between the two results is greater than the repeatability limit given in 4.7.2, repeat the determination until the requirements are met.

4.5.2 Test portion

For each sample, tare to the nearest 0,001 g two metal capsules (4.2.5) beforehand. For each capsule, note the tare t.

Weigh rapidly, to the nearest 0,001 g, approximately (8 ± 1) g of the test sample (see 4.4.1, 4.4.2.2 or 4.4.2.3, as appropriate) into the capsule. Note the mass m'_0.

4.5.3 Drying

Place the open capsule containing the test portion, and the lid, in the oven (4.2.7) controlled between 130 °C and 133 °C and leave it for 4 h ± 5 min.

Never place moist products in an oven containing test portions at the end of dehydration, nor open the oven door during drying, nor introduce new wet test samples before removing the dry test portions as this would rehydrate them.

At the end of the drying time and, proceeding rapidly, take the dish out of the oven, cover it and place it in the desiccator (4.2.8). When several tests are being carried out simultaneously, never place dishes on top of one another in the desiccator.

4.5.4 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 it to the nearest 0,001 g. Note the mass m'_1.

4.6 Expression of results

The moisture content, w_{H2O}, expressed as a percentage by mass of the product as received, is given by Formulae (1) and (2):

a) without pre-conditioning:

$$w_{H2O} = (m_0 - m_1)\frac{100}{m_0}$$

(1)

where

$m_0 = m'_0 - t$ is the mass, in grams, of the test portion (see 4.5.3);

$m_1 = m'_1 - t$ is the mass, in grams, of the test portion after drying (see 4.5.4);

t is the tare of the capsule, in grams (see 4.5.2).

b) with pre-conditioning:

$$w_{H2O} = \left[(m_0 - m_1)\frac{m_3}{m_0} + m_2 - m_3 \right]\frac{100}{m_2}$$

(2)

$$w_{H2O} = 100\left(1 - \frac{m_1 m_3}{m_0 m_2} \right)$$

where

m_0 and m_1 have the same signification as in a) above;

m_2 is the mass, in grams, of the sample before conditioning (see 4.4.2.3);

m_3 is the mass, in grams, of the sample after conditioning (see 4.4.2.3).

Take as the result the average of the two values obtained, provided that the requirement for repeatability (see 4.7.2) is satisfied. If it is not, repeat the determinations.

Express the result to the second decimal place.

4.7 Precision

4.7.1 Interlaboratory test

The details of an interlaboratory test relating to the precision of the method are summarized in Annex B. Values from this test can only be applied to water content ranges from 11,90 % to 39,20 % and the studied matrix (maize).

4.7.2 Repeatability

The absolute difference between two independent individual test results, obtained using the same method on identical material tested in the same laboratory by the same operator using the same apparatus and within a short time interval, shall be exceed in no more than 5 % of cases the repeatability limit r.

For maize with water content between 11,90 % and 39,20 %:

— $r = 2,8\,S_r$

— $r = 2,8 \times 0,07 = 0,19$

4.7.3 Reproducibility

Reproducibility is the absolute difference between two individual test results, obtained with the same method on identical material tested in different laboratories by different operators using different equipment.

In practice, it is not appropriate to compare the results of two laboratories if the related test imposes repeatability conditions.

The appropriate comparison tool is the critical difference as described in 4.7.5.

4.7.4 Comparison of two groups of measurements in a laboratory

The critical difference (D_r) is the difference between two averaged values obtained from two test results under repeatability conditions. Since each result is the average of two values (see 4.6), the comparison of the two water content results shall be done using the critical difference.

The D_r between two averaged values each obtained from two test results under repeatability conditions is shown by Formula (3):

$$D_r = 2,8 S_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2,8 S_r \sqrt{\frac{1}{2}} = 1,98 S_r = 0,14 \tag{3}$$

where

S_r is the standard deviation of repeatability;

n_1 and n_2 are the number of test results corresponding to each averaged values.

4.7.5 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 shown by Formula (4):

$$D_R = 2,8 \sqrt{S_R{}^2 - S_r{}^2 (1 - \frac{1}{2n_1} - \frac{1}{2n_2})} = 2,8 \sqrt{S_R{}^2 - 0,5 S_r{}^2} = 0,68 \tag{4}$$

where