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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)

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

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:

$$w_{H20} = (m_0 - m_1)\frac{100}{m_0} \quad (1)$$

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:

$$w_{H20} = \left[(m_0 - m_1)\frac{m_3}{m_0} + m_2 - m_3\right]\frac{100}{m_2} \quad (2)$$

$$w_{H20} = 100\left(1 - \frac{m_1 m_3}{m_0 m_2}\right)$$

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):

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

where

S_r is the standard deviation of repeatability;

S_R is the standard deviation of reproducibility;

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

4.7.6 Uncertainty

It is possible to evaluate measurement uncertainties using data obtained from studies carried out in accordance with ISO 5725-2. The reproducibility standard deviation obtained during an interlaboratory test is a valid basis to assess measurement uncertainty because, by definition, uncertainty characterizes the dispersion of values that can be reasonably attributed to the parameter.

The calculated expanded standard uncertainty should be ≤ ±2 reproducibility standard deviations (see Annex B).

4.7.7 Comparison with the absolute method

Compared to the absolute method (see Annex A), the results generally differ from less than 0,15 g of water per 100 g of samples.

4.8 Notes on procedure

4.8.1 The range of moisture contents for which the conditioning of the products before grinding is to be carried out corresponds, in the laboratory, to a temperature of (20 ± 2) °C and a relative humidity of 45 % to 75 %. It should be modified for different atmospheric conditions.

4.8.2 The conditioning and grinding carried out on 100 g and 30 g, respectively, for a test portion of 8 g are intended to provide a more representative sample. A direct sampling for grinding of only 8 g would correspond to an insufficient quantity of initial product to be representative and would lead to too great a dispersion of the results.

4.9 Test report

The test report shall contain at least the following information:

a) all the information necessary for the identification of the sample (type of sample, origin and designation of the sample);

b) a reference to this document, i.e. ISO 6540;

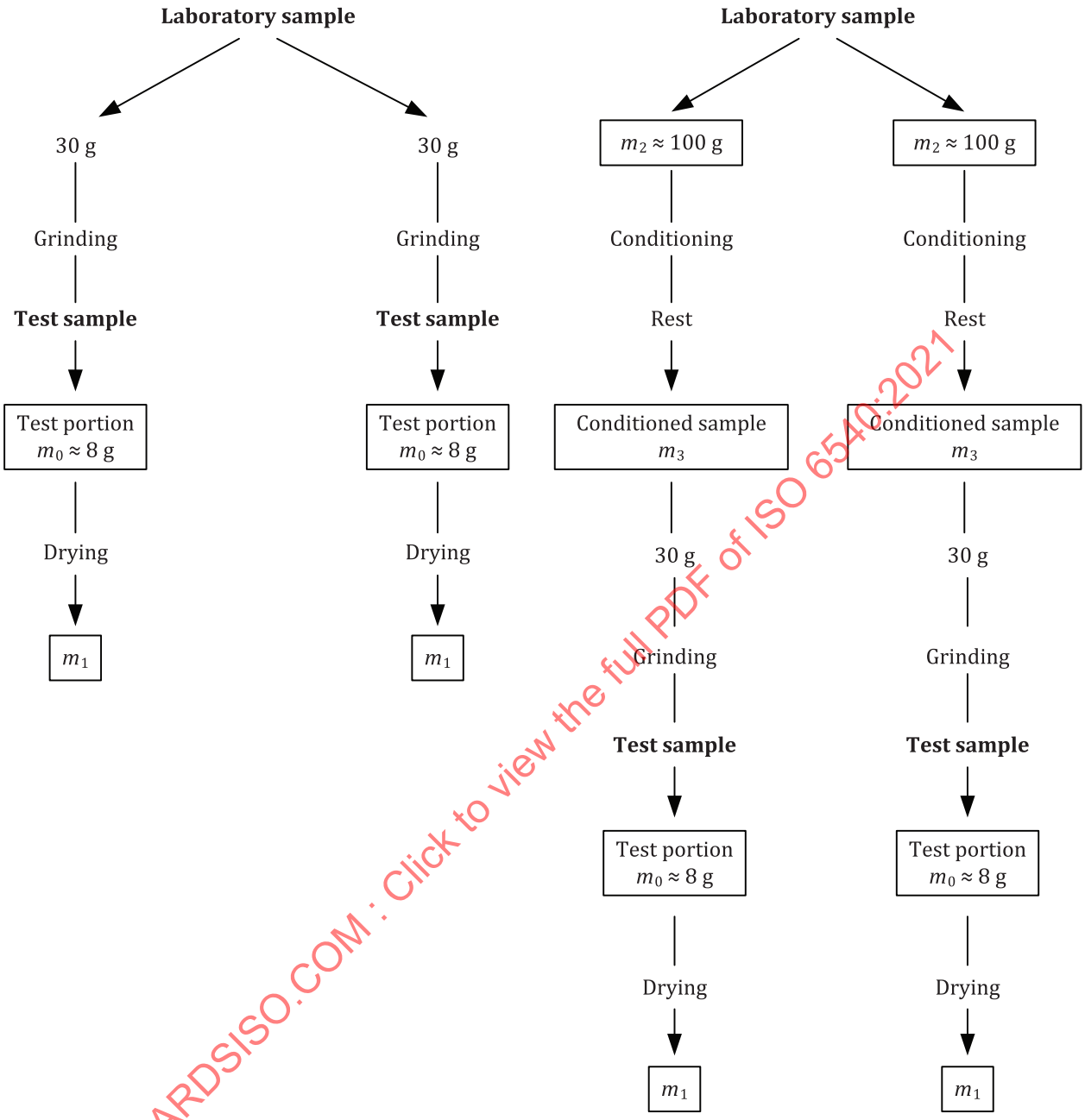
c) the date and type of sampling procedure (if known);

d) the date of receipt;

e) the date of the test;

f) the test results and the units in which they have been expressed;

g) any operation not specified in the method or regarded as optional which might have affected the result.



a) Without conditioning **b) With conditioning**

NOTE The weighing operations are boxed.

Figure 1 — Diagram of the two possible procedures for products requiring to be ground

5 Routine method on whole grains

5.1 Principle

Drying of whole grains for 38 h at a temperature between 130 °C and 133 °C.

5.2 Apparatus

5.2.1 Metal dish, non-corrodible under the test conditions, with a sufficiently tight-fitting lid, a diameter of 50 mm to 60 mm and a minimum height of 25 mm.

5.2.2 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 130 °C to 133 °C, in the neighbourhood of the test portions, in normal working condition.

5.2.3 Desiccator, containing an efficient desiccant.

5.2.4 Analytical balance, capable of weighing with an accuracy of ±0,001 g and therefore having a display accuracy of 0,000 1 g.

5.3 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 or modified during transport and storage.

5.4 Procedure

5.4.1 Test portion

Weigh, to the nearest 0,001 g, the metal dish (5.2.1) and its lid (m_0).

Introduce rapidly, according to the diameter of the dish, from 25 g to 40 g of whole grains.

Immediately, close the dish and weigh to the nearest 0,001 g (m_1).

5.4.2 Drying

Place the open dish containing the test portion, with the lid by its side, in the oven (5.2.2) controlled at 130 °C to 133 °C and leave it for (38 ± 2) h.

NOTE In practice, leave it for two nights and one day.

Never place moist products in an oven containing test portions at the end of dehydration, as this will result in partial rehydration of the latter.

Following this period, proceeding rapidly, take the dish out of the oven, cover it and place it in the desiccator (5.2.3). When several tests are being carried out simultaneously, never place dishes on top of one another in the desiccator.

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 (m_2).

5.4.3 Number of determinations

Carry out at least two determinations on test portions taken from the same laboratory sample.

5.5 Expression of results

5.5.1 Method of calculation and formulae

The moisture content, expressed as a percentage by mass of the product as received, is given by the Formula (5):

$$(m_1 - m_2) \times \frac{100}{m_1 - m_0} \tag{5}$$

where

m_0 is the mass, in grams, of the empty dish and its lid;

m_1 is the mass, in grams, of the dish, its lid and the test portion before drying;

m_2 is the mass, in grams, of the dish, its lid and the test portion after drying.

Take as the result the average of the values obtained, provided that the requirement for repeatability (see 5.5.2) is satisfied. If it is not, repeat the determinations.

Express the result to one decimal place.

5.5.2 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 exceed the repeatability limit r in maximum 5 % of cases.

$$r = 2{,}8\, S_r$$

$$r = 2{,}8 \times (0{,}002\ 6\ w + 0{,}026\ 6)$$

where

S_r is the standard deviation of repeatability;

w is the water content of the sample, in mass fraction expressed as a percentage.

5.5.3 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 5.5.5.

5.5.4 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 5.5.1), 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 (6):

$$D_r = 2{,}8 S_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8 S_r \sqrt{\frac{1}{2}} = 1{,}98 S_r \quad (6)$$

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.

5.5.5 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 (7):

$$D_R = 2{,}8 \sqrt{S_R^2 - S_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}8 \sqrt{S_R^2 - 0{,}5 S_r^2} \quad (7)$$

where

S_r is the standard deviation of repeatability;

S_R is the standard deviation of reproducibility;

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

5.5.6 Application of fidelity limits

In order to facilitate the application of repeatability limits and critical differences to compare results, a table of these different values for several moisture content levels is given in Table C.1.

5.6 Remark

The results compared with those obtained by the absolute method (see Annex A) generally differ by less than 0,5 g of moisture per 100 g of sample. A positive bias of approximately a mass fraction of 0,30 % compared to the reference method described in Clause 4 was demonstrated by an interlaboratories test (see Annex B).

5.7 Test report

The test report shall contain at least the following information:

a) all the information necessary for the identification of the sample (type of sample, origin and designation of the sample);

b) a reference to this document, i.e. ISO 6540;

c) the date and type of sampling procedure (if known);

d) the date of receipt;

e) the date of the test;

f) the test results and the units in which they have been expressed;

g) any operations not specified in the method or regarded as optional, which could have affected the result.

Annex A
(informative)

Absolute method

A.1 General

This annex specifies the absolute method for the determination of the moisture content of maize grains and ground whole maize.

This method is intended to serve as a standard for checking and perfecting routine methods for the determination of moisture content, in particular the methods specified in sections one and two. It is not intended to be used for settling commercial disputes.

The absolute method specified in this annex 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.

A.2 Principle

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

A.3 Apparatus

A.3.1 Analytical balance, able to weigh with an accuracy of ±0,000 1 g and therefore having a display accuracy of 0,000 01 g.

A.3.2 Apparatus for reducing pressure to 1,3 kPa and 2,6 kPa, for example a water pump.

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

A.3.3 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 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 A.5.1.1.

A.3.4 Metal boat, without lid, with an effective surface area enabling 100 g of maize grains to be distributed in a single layer.

A.3.5 Metal dish, non-corrodible under the test conditions, 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.

NOTE A suitable metal dish is shown, for information only, in Figure A.1.

A.3.6 Apparatus for drying at a reduced pressure, with a volume such that the metal boats (A.3.4) can be placed inside.

A.3.7 Cup, made from glass or porcelain.

A.3.8 Drying tube, of glass, in two parts, one of which, closed at one end, is intended to receive the dish (A.3.5) and the other, intended to contain the cup (A.3.7), carries a semi-capillary tube, with a stopcock, for connection to the vacuum source (A.3.2). The two parts are connected by a ground glass joint.

The test portion may be cooled in this apparatus after drying, the desiccator (A.3.11) being then unnecessary for this operation.

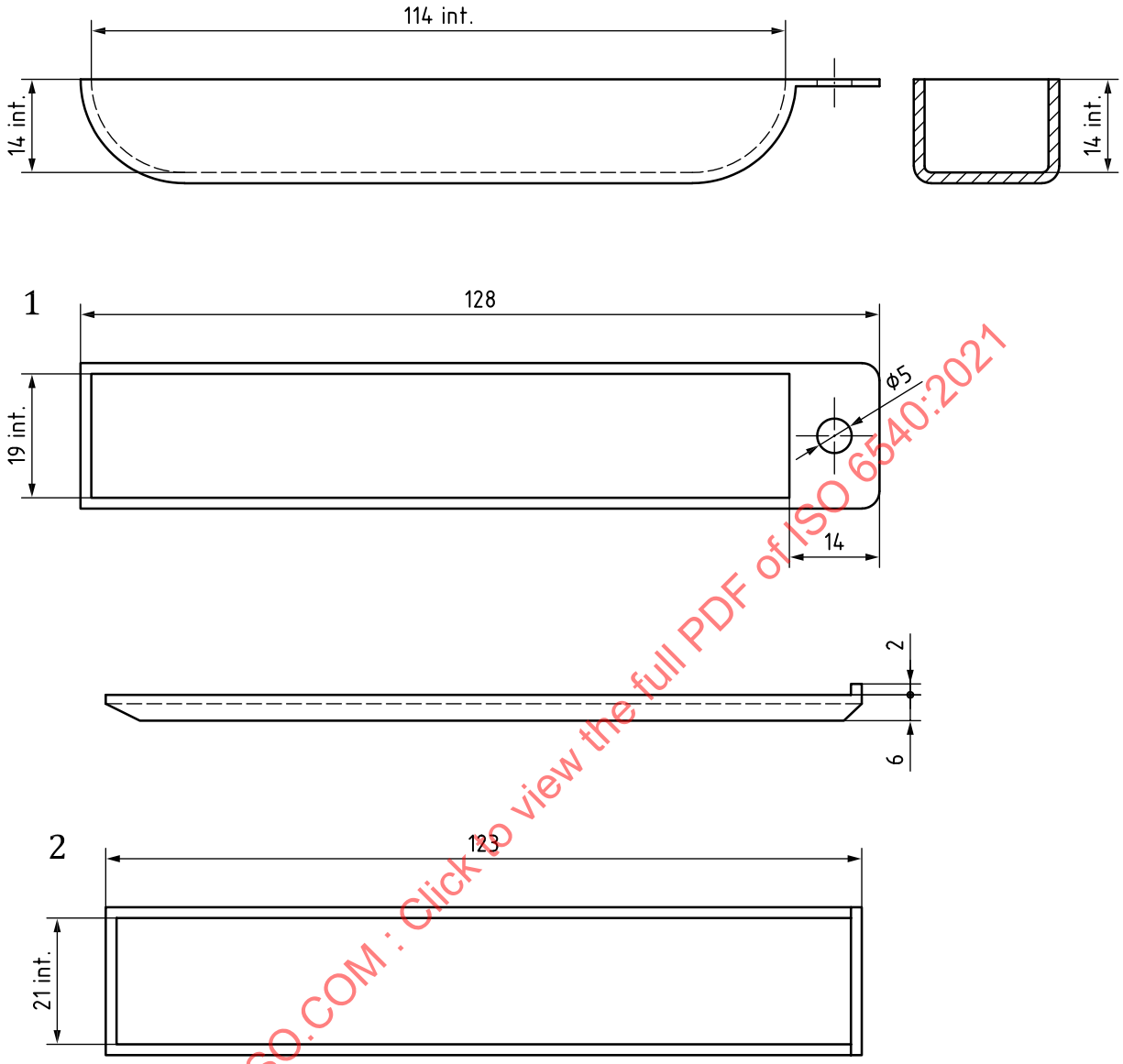
NOTE A suitable drying tube is shown, for information only, in Figure A.2.

A.3.9 Constant-temperature oven, electrically heated, enabling the part of the drying tube (A.3.8) containing the dish (A.3.5) to be maintained at a temperature between 45 °C and 50 °C.

A.3.10 Air-drying train, gas-washing bottle containing pure analytical grade sulphuric acid (ρ_{20} > 1,83 g/ml), connected to a tube containing pure analytical grade phosphorus(V) oxide spread on glass wool.

A.3.11 Desiccator, containing an efficient desiccant.

Dimensions in millimetres



Key

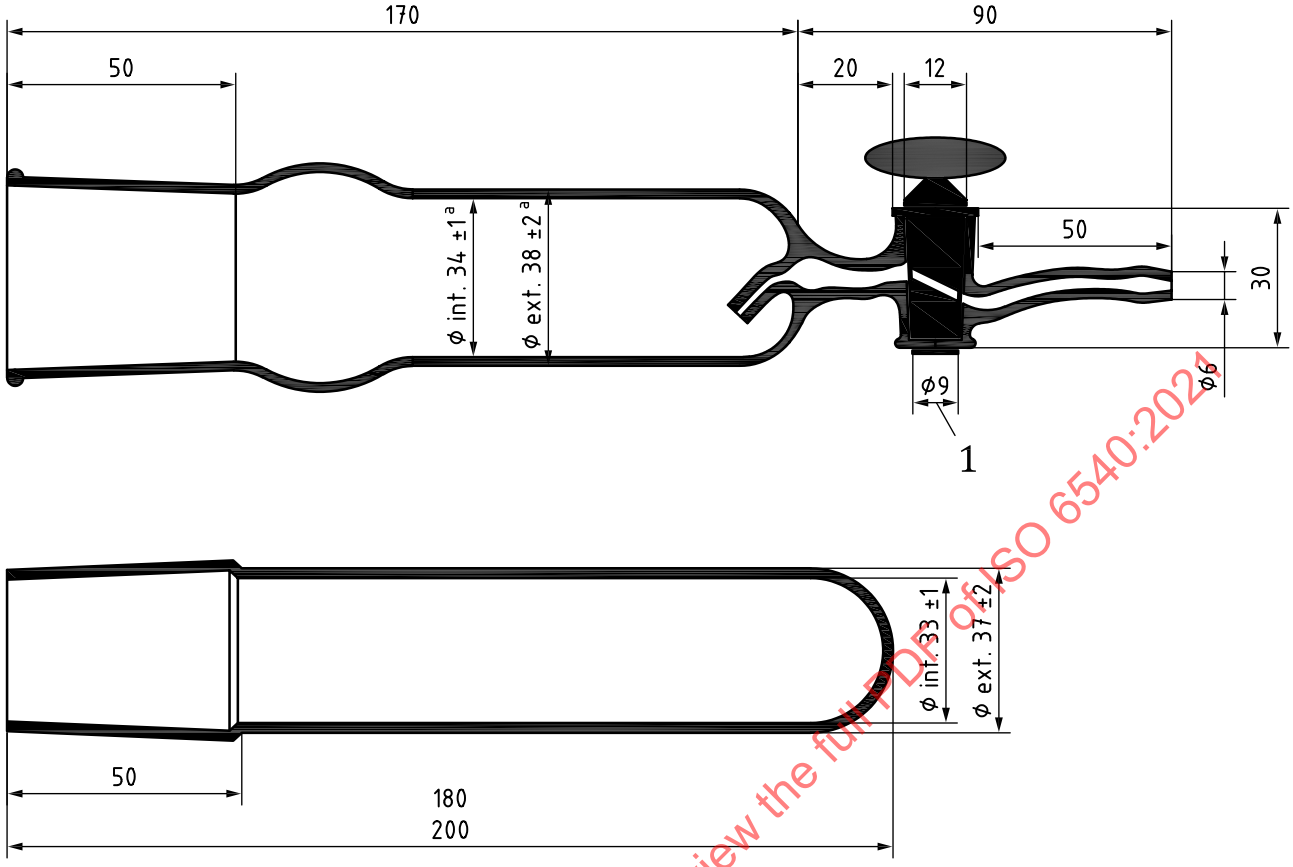
1 dish

2 lid

NOTE The dish shown in the diagram 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 A.2.

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

Dimensions in millimetres



Key

1 stopcock with inclined 2 mm bore

[a] Wall thickness ≈ 2 mm.

NOTE 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 A.1. The olive ending to the stopcock side arm may be replaced by a ground glass joint.

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

A.4 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 or modified during transport and storage.

A.5 Procedure

A.5.1 Preparation of the test sample

A.5.1.1 Products not requiring to be ground

Products that have particles of sizes less than or equal to 1,7 mm, less than a mass of fraction of 10 % being over 1 mm and more than a mass of 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 A.5.2).

A.5.1.2 Products requiring to be ground

A.5.1.2.1 General

If the laboratory sample does not have the particle size characteristics mentioned in A.5.1.1, it shall be ground either without pre-conditioning (A.5.1.2.2) or with pre-conditioning (A.5.1.2.3) as required.

A.5.1.2.2 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 A.7.1), carry out grinding without pre-conditioning.

Adjust the grinding mill (A.3.3) to obtain particles of the dimensions indicated in A.5.1.2.

Then quickly grind about 30 g of the laboratory sample, mix with a spatula and proceed immediately as specified in A.5.2.

A.5.1.2.3 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 A.7.1) before grinding.

If the moisture content is more than a mass fraction of 15,00 % (which is the more frequent case), weigh, to the nearest 0,01 g, about 100 g of the laboratory sample in the metal boat (A.3.4), and place this in the drying apparatus (A.3.6) in which have been placed Petri dishes containing a layer of phosphorus(V) oxide about 1 cm thick. Reduce the pressure to a value of the order of 1,3 kPa to 2,6 kPa, using the vacuum apparatus (A.3.2); this should be done gradually in order to avoid material being sucked out of the boat. Close the connection to the vacuum apparatus (A.3.2), and leave the sample at laboratory temperature for the time needed to bring its moisture content to between a mass fraction of 9 % and 15 % (usually from 2 to 4 days, see A.7.2). Restore atmospheric pressure in the drying apparatus by causing air, which has passed through the drying train (A.3.10), to enter slowly.

Then keep the pre-dried sample for at least 24 h in the laboratory atmosphere (see A.7.4).

After conditioning, weigh the sample, to the nearest 0,01 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 10 mg, in a suitable atmosphere (usually that of the laboratory) and leave it until a moisture content within the limits specified above is obtained.

A.5.2 Test portion

Rapidly weigh, to the nearest 0,000 2 g, about 3 g of the test sample (see A.5.1.1, A.5.1.2.2 or A.5.1.2.3, as appropriate) in the metal dish (A.3.5), which has been previously weighed, together with its lid, to the nearest 0,000 2 g.

A.5.3 Drying

Place the open dish (leaving its lid in the desiccator) containing the test portion (A.5.2) at the closed end of the drying tube (A.3.8). Introduce, near to it, the cup (A.3.7) 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 kPa to 2,6 kPa, using the vacuum apparatus (A.3.2); this should be done gradually in order to avoid material being sucked 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 (A.3.9), controlled at 45 °C to 50 °C.

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 (A.3.10), 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 0,000 2 g.

Repeat the operations specified above until the mass is practically constant (i.e. until the difference between two successive weightings at an interval of 240 h is less than 0,000 6 g).

A.5.4 Number of determinations

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

A.6 Expression of results

A.6.1 Method of calculation and formulae

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

a) without pre-conditioning:

$$w_{H2O} = (m_0 - m_1)\frac{100}{m_0} \tag{A.1}$$

where

m_0 is the mass, in grams, of the test portion (see A.5.2);

m_1 is the mass, in grams, of the test portion after drying (see A.5.3).

b) with pre-conditioning:

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

$$w_{H2O} = 100\left(1 - \frac{m_1 m_3}{m_0 m_2}\right) \tag{A.2}$$

where

m_0 is the mass, in grams, of the test portion (see A.5.2);

m_1 is the mass, in grams, of the test portion after drying (see A.5.3);

m_2 is the mass, in grams, of the sample before conditioning (see A.5.1.2.3);

m_3 is the mass, in grams, of the sample after conditioning (see A.5.1.2.3).

Take as the result the arithmetic mean of the two values obtained, provided that the requirement for repeatability (see A.6.2) is satisfied. If it is not, repeat the determinations.

Express the result to the second decimal place.

A.6.2 Repeatability

The difference between the values obtained from the 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.

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

A.7 Notes on procedure

A.7.1 The range of moisture contents for which the conditioning of the products before grinding is to be carried out corresponds, in the laboratory, to a temperature of (20 ± 2) °C and a relative humidity of 45 % to 75 %. It should be modified for different atmospheric conditions.

A.7.2 The duration of pre-drying is given for guidance only. Check that it enables the desired conditioning to be obtained with the apparatus and the products used.

A.7.3 The conditioning and grinding carried out on 100 g and 30 g, respectively, for a test portion of 3 g are intended to provide a more representative sample. A sample of 3 g would correspond to an insufficient quantity of ground product to be representative and would lead to too great a dispersion of the results.

A.7.4 The period of rest of 24 h that follows the pre-drying is necessary to obtain uniform distribution of the moisture.

A.7.5 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.

A.8 Test report

The test report shall contain at least the following information:

a) all the information necessary for the identification of the sample (type of sample, origin and designation of the sample);

b) a reference to this document, i.e. ISO 6540;

c) the date and type of sampling procedure (if known);

d) the date of receipt;

e) the date of the test;

f) the test results and the unit in which they have been expressed;

g) any operations not specified in the method or regarded as optional which could have affected the result.

Annex B
(informative)

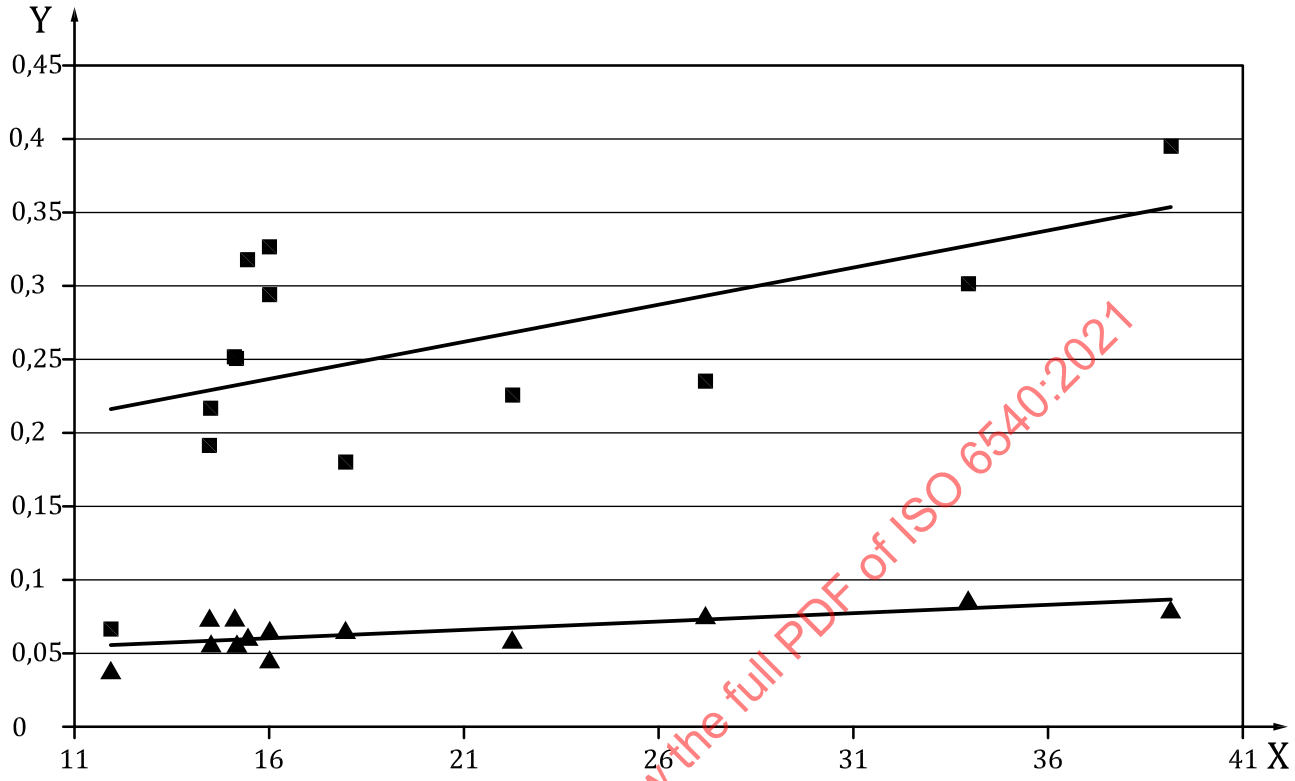
Interlaboratory test results

An interlaboratory test organized internationally with the participation of 15 laboratories for the protocol of Clause 4 and 14 laboratories for the protocol of Clause 5 gave the statistical results (determined in accordance with ISO 5725-2) indicated in Table B.1 for the protocol of Clause 4 and Table B.2 for the protocol of Clause 5.

Table B.1 — Statistical results of the interlaboratory test for ground grains determinations (procedure in Clause 4)

Sample	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Pre-drying	No	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Number of selected laboratories	14	15	15	14	14	14	14	14	14	13	13	13	13	12
Average (M)	11,91	14,46	14,48	15,08	15,14	15,44	15,44	16,00	16,00	17,95	22,23	27,19	33,94	39,16
Standard deviation of repeatability, s_r	0,04	0,07	0,06	0,07	0,06	0,06	0,06	0,07	0,05	0,07	0,06	0,08	0,09	0,08
Coefficient of variation, $C_{V,r}$ (s_r /M, in %)	0,3	0,5	0,4	0,5	0,4	0,4	0,4	0,4	0,3	0,4	0,3	0,3	0,3	0,2
Repeatability limit, r	0,11	0,21	0,16	0,21	0,16	0,17	0,17	0,19	0,13	0,19	0,17	0,22	0,25	0,23
Standard deviation of reproducibility, s_R	0,07	0,19	0,22	0,25	0,25	0,32	0,32	0,29	0,33	0,18	0,23	0,24	0,30	0,40
Coefficient of variation, $C_{V,R}$ (s_R /M, in %)	0,6	1,3	1,5	1,7	1,7	2,1	2,1	1,8	2,0	1,0	1,0	0,9	0,9	1,0
Reproducibility limit, R	0,19	0,54	0,61	0,71	0,71	0,90	0,90	0,83	0,93	0,51	0,64	0,66	0,85	1,12

The repeatability and reproducibility standard deviation do not vary (see Figure B.1) with the measured water content and are considered constant for moisture contents between 12,00 % and 40,00 %.



Key

X water content

Y fidelity standard deviation

▲ repeatability standard deviation (S_r)

■ reproducibility standard deviation (S_R)

Formula of the regression line for S_r: Y = 0,001 1 X + 0,042 4

Coefficient of determination R^2 for S_r = 0,481 7

Formula of the regression line for S_R: Y = 0,005 0 X + 0,156 4

Coefficient of determination R^2 for S_R = 0,261 9

Figure B.1 — Evolution of the fidelity standard deviations as a function of the water content for the determination on ground grains (see Clause 4)