International Standard 6540

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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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Foreword

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

0 Introduction

The basic reference method and the routine reference method relating to cereals (ISO 711 and ISO 712) are only applicable to maize with a number of amendments. This is why it has been considered advisable to reproduce the whole of these two methods, amended for application to the case of maize.

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

Because of the very high moisture content which may be present in samples of maize [sometimes greater than 40 % (m/m)] and because of the size and texture of the grains, the determination of the moisture in maize raises problems with regard to its pre-drying and grinding.

Consequently, to allow the pre-drying and grinding to be avoided, this International Standard also describes a routine method for whole grain which is easier to use and allows working in series.

Section one : Reference method

1 Scope and field of application

This section specifies the reference method for the determination of the moisture content of maize grains and ground whole maize.

2 Reference

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

3 Definition

moisture content of maize : Conventionally, the loss in mass, expressed as a percentage, undergone by the product under the conditions specified in this section.

4 Principle

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

5 Apparatus

5.1 Analytical balance.

5.2 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 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 7.1.1.

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

5.4 Metal dish, 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.

5.5 Constant-temperature oven, electrically heated, capable of being maintained between 60 and 80 °C, and with adequate ventilation.

5.6 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 to 133 °C in the neighbourhood of the test portions, in normal working.

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 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.

5.7 Desiccator, containing an efficient desiccant.

6 Sampling

See ISO 950.

7 Procedure (See figure 1)

7.1 Preparation of the test sample

7.1.1 Products not requiring to be ground

Products which have 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.

Mix the laboratory sample thoroughly before taking the test portion (7.2).

7.1.2 Products requiring to be ground

If the laboratory 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) as required.

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 9 and 15 % (*m/m*) (see 9.1)], carry out grinding without pre-conditioning.

Adjust the grinding mill (5.2) to obtain particles of the dimensions indicated in 7.1.1, grind a small quantity of the laboratory sample and discard it.

Then quickly grind about 30 g of the laboratory sample, mix with a spatula and proceed immediately as specified in 7.2.

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 15 % (m/m) or less than 9 % (m/m)] shall be pre-conditioned to bring their moisture content to between 9 and 15 % (m/m) (see 9.1) before grinding.

If the moisture content is greater than 15 % (m/m) (the more frequent case), weigh, to the nearest 10 mg, about 100 g of the laboratory sample in the metal boat (5.3), place this in the oven (5.5) controlled at between 60 and 80 °C, and leave it for the time necessary to bring the moisture content to between 9 and 15 % (m/m). 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.

After conditioning, weigh the sample to the nearest 10 mg, then, proceeding rapidly, grind about 30 g of this product. Mix using a spatula.

NOTE – If the moisture content is less than 9 % (m/m), 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.

7.2 Test portion

Rapidly weigh, to the nearest 1 mg, about 8 g of the test sample (7.1.1, 7.1.2.1 or 7.1.2.2, as appropriate) in the dish (5.4), which has been previously dried and weighed, together with its lid, to the nearest 1 mg.

7.3 Drying

Place the open dish containing the test portion, and the lid, in the oven (5.6) controlled at 130 to 133 °C and leave it for 4 h, taken from the moment when the oven temperature is again between 130 to 133 °C.

Proceeding rapidly, take the dish out of the oven, cover it and place in the desiccator (5.7); 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 and 45 min after it has been placed in the desiccator), weigh it to the nearest 1 mg.

7.4 Number of determinations

Carry out two determinations on test portions taken from different test samples, but from the same laboratory sample (see figure 1).

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);

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);

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

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

m_3 is the mass, in grams, of the sample after conditioning (7.1.2.2).

Take as the result the arithmetic mean of the two values obtained, provided that the requirement for repeatability (see 8.2) is satisfied. If it is not, repeat the determinations.

Express the result to the second decimal place.

8.2 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 0,15 g of moisture per 100 g of sample.

8.3 Remark

The results compared with those obtained by the absolute method (see the annex) generally differ by less than 0,15 g of moisture per 100 g of sample.

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.

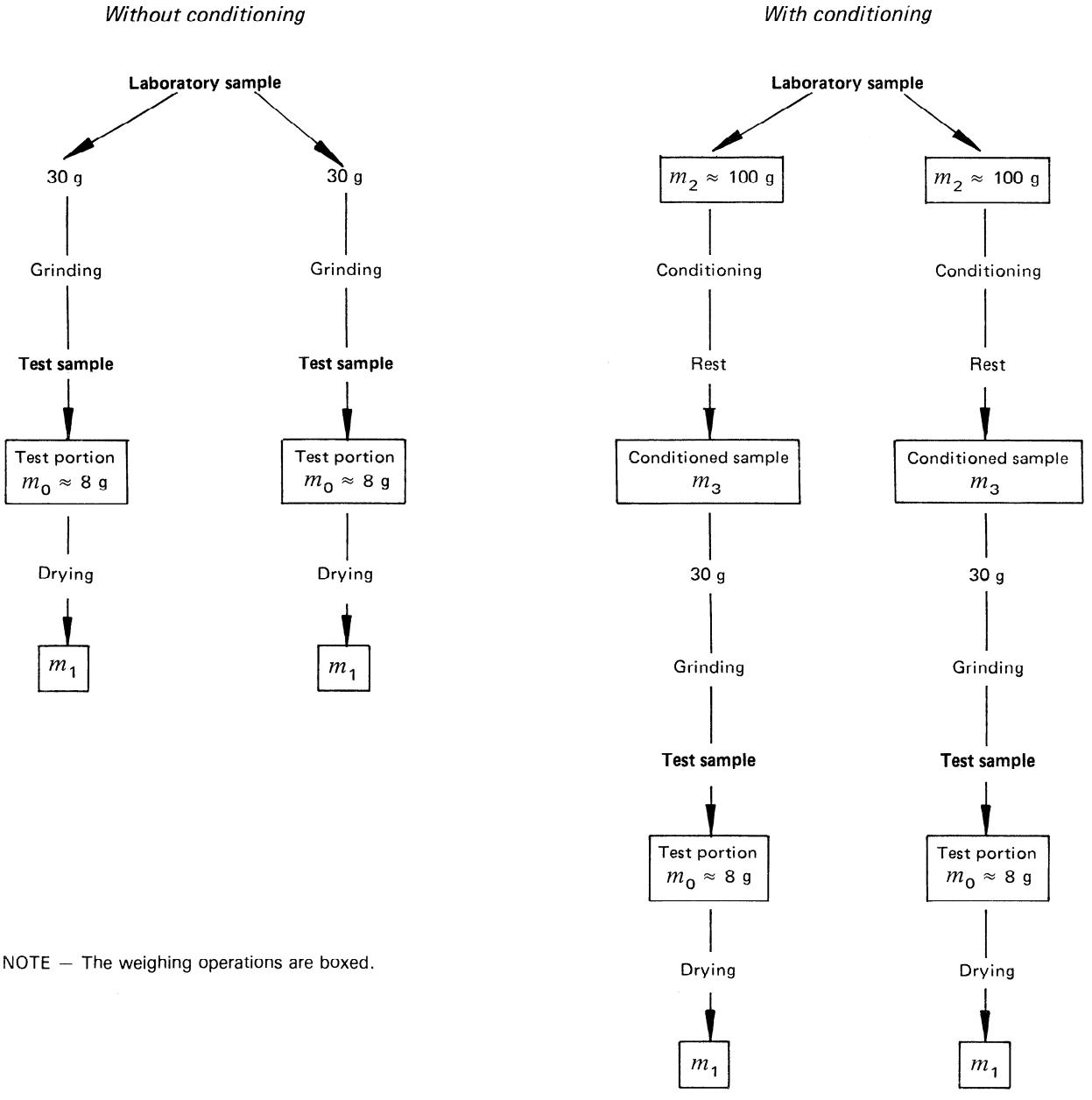
9.2 Never place moist products in an oven containing test portions at the end of dehydration, as this will result in partial rehydration of the latter.

9.3 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 sample of 8 g would correspond to an insufficient quantity of ground product to be representative and would lead to too great a dispersion of the results.

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 section, 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.



NOTE — The weighing operations are boxed.

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

Section two : Routine method on whole grains

11 Scope and field of application

This section specifies a method for the evaluation of the moisture content of maize in whole grains.

It is not suitable for use for experts' reports, or for calibration or checking of humidity meters.

12 Reference

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

13 Definition

moisture content of maize : Conventionally, the loss in mass, expressed as a percentage, undergone by the product under the conditions specified in this section.

14 Principle

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

15 Apparatus

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

15.2 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 to 133 °C, in the neighbourhood of the test portions, in normal working.

15.3 Desiccator, containing an efficient desiccant.

15.4 Balance.

16 Sampling

See ISO 950.

17 Procedure

17.1 Test portion

Weigh, to the nearest 0,01 g, the metal dish (15.1) and its lid, previously dried.

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

Immediately, close the dish and weigh to the nearest 0,01 g.

17.2 Drying

Place the open dish containing the test portion, with the lid by its side, in the oven (15.2) controlled at 130 to 133 °C and leave it for 38 ± 2 h.[1)]

Following this period, proceeding rapidly, take the dish out of the oven, cover it and place it in the desiccator (15.3); 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 and 45 min after it has been placed in the desiccator), weigh it to the nearest 0,01 g.

17.3 Number of determinations

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

18 Expression of results

18.1 Method of calculation and formulae

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

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

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 arithmetic mean of the values obtained, provided that the requirement for repeatability (see 18.2) is satisfied. If it is not, repeat the determinations.

Express the result to one decimal place.

1) In practice, two nights and one day.

18.2 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 0,2 g of moisture per 100 g of sample.

18.3 Remark

The results compared with those obtained by the absolute method (see the annex) generally differ by less than 0,5 g of moisture per 100 g of sample.

19 Note on procedure

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.

20 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 section, 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

Absolute method

A.0 Introduction

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.1 Scope and field of application

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.

A.2 Reference

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

A.3 Definition

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

A.4 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 and 50 °C and in the presence of a desiccant, until constant mass is reached.

A.5 Apparatus

A.5.1 Analytical balance.

A.5.2 Apparatus for reducing pressure to 1,3 and 2,6 kPa[1], for example a water pump.

A.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 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.7.1.1.

A.5.4 Metal boat, without lid, with an effective surface area enabling 100 g of maize grains to be distributed in a single layer.

A.5.5 Metal dish[2], 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.

A.5.6 Apparatus for drying at a reduced pressure, with a volume such that the metal boats (A.5.4) may be placed inside.

A.5.7 Cup, made from glass or porcelain.

A.5.8 Drying tube[3], of glass, in two parts, one of which, closed at one end, is intended to receive the dish (A.5.5) and the other, intended to contain the cup (A.5.7), carries a semi-capillary tube, with a stopcock, for connection to the vacuum source (A.5.2). The two parts are connected by a ground glass joint.

The test portion may be cooled in this apparatus after drying, the desiccator (A.5.11) being then unnecessary for this operation.

A.5.9 Constant-temperature oven, electrically heated, enabling the part of the drying tube (A.5.8) containing the dish (A.5.5) to be maintained at a temperature between 45 and 50 °C.

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

2) A suitable metal dish is shown, for information only, in figure 2.

3) A suitable drying tube is shown, for information only, in figure 3.

A.5.10 Air-drying train : gas-washing bottle containing pure analytical grade sulphuric acid ($\varrho_{20} \geqslant 1{,}83$ g/ml), connected to a tube containing pure analytical grade phosphorus(V) oxide spread on glass wool.

A.5.11 Desiccator, containing an efficient desiccant.

A.6 Sampling

See ISO 950.

A.7 Procedure

A.7.1 Preparation of the test sample

A.7.1.1 Products not requiring to be ground

Products which have 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.

Mix the laboratory sample thoroughly before taking the test portion (A.7.2).

A.7.1.2 Products requiring to be ground

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

A.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 9 and 15 % (m/m) (see A.9.1)], carry out grinding without pre-conditioning.

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

Then quickly grind about 30 g of the laboratory sample, mix with a spatula and proceed immediately as specified in A.7.2.

A.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 15 % (m/m) or less than 9 % (m/m)] shall be pre-conditioned to bring their moisture content to between 9 and 15 % (m/m) (see A.9.1) before grinding.

If the moisture content is more than 15 % (m/m) (the more frequent case), weigh, to the nearest 10 mg, about 100 g of the laboratory sample in the metal boat (A.5.4), and place this in the drying apparatus (A.5.6) 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 to 2,6 kPa, using the vacuum apparatus (A.5.2); this should be done gradually in order to avoid material being sucked out of the boat. Close the connection to the vacuum apparatus (A.5.2), and leave the sample at laboratory temperature for the time needed to bring its moisture content to between 9 and 15 % (m/m) (usually from 2 to 4 days — see A.9.2). Restore atmospheric pressure in the drying apparatus by causing air, which has passed through the drying train (A.5.10), to enter slowly.

Then keep the pre-dried sample for at least 24 h in the laboratory atmosphere (see A.9.4).

After conditioning, weigh the sample, to the nearest 10 mg, then, proceeding rapidly, grind about 30 g of this product. Mix using a spatula.

NOTE — If the moisture content is less than 9 % (m/m), 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.7.2 Test portion

Rapidly weigh, to the nearest 0,2 mg, about 3 g of the test sample (A.7.1.1, A.7.1.2.1 or A.7.1.2.2, as appropriate) in the metal dish (A.5.5), which has been previously dried and weighed, together with its lid, to the nearest 0,2 mg.

A.7.3 Drying

Place the open dish (leaving its lid in the desiccator) containing the test portion (A.7.2) at the closed end of the drying tube (A.5.8); introduce, near to it, the cup (A.5.7) 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 to 2,6 kPa, using the vacuum apparatus (A.5.2); 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.5.9), controlled at 45 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.5.10), 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 0,2 mg.

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

A.7.4 Number of determinations

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

A.8 Expression of results

A.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 (A.7.2);

m_1 is the mass, in grams, of the test portion after drying (A.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 (A.7.2);

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

m_2 is the mass, in grams, of the sample before conditioning (A.7.1.2.2);

m_3 is the mass, in grams, of the sample after conditioning (A.7.1.2.2).

Take as the result the arithmetic mean of the two values obtained, provided that the requirement for repeatability (see A.8.2) is satisfied. If it is not, repeat the determinations.

Express the result to the second decimal place.

A.8.2 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.9 Notes on procedure

A.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.

A.9.2 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.9.3 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.

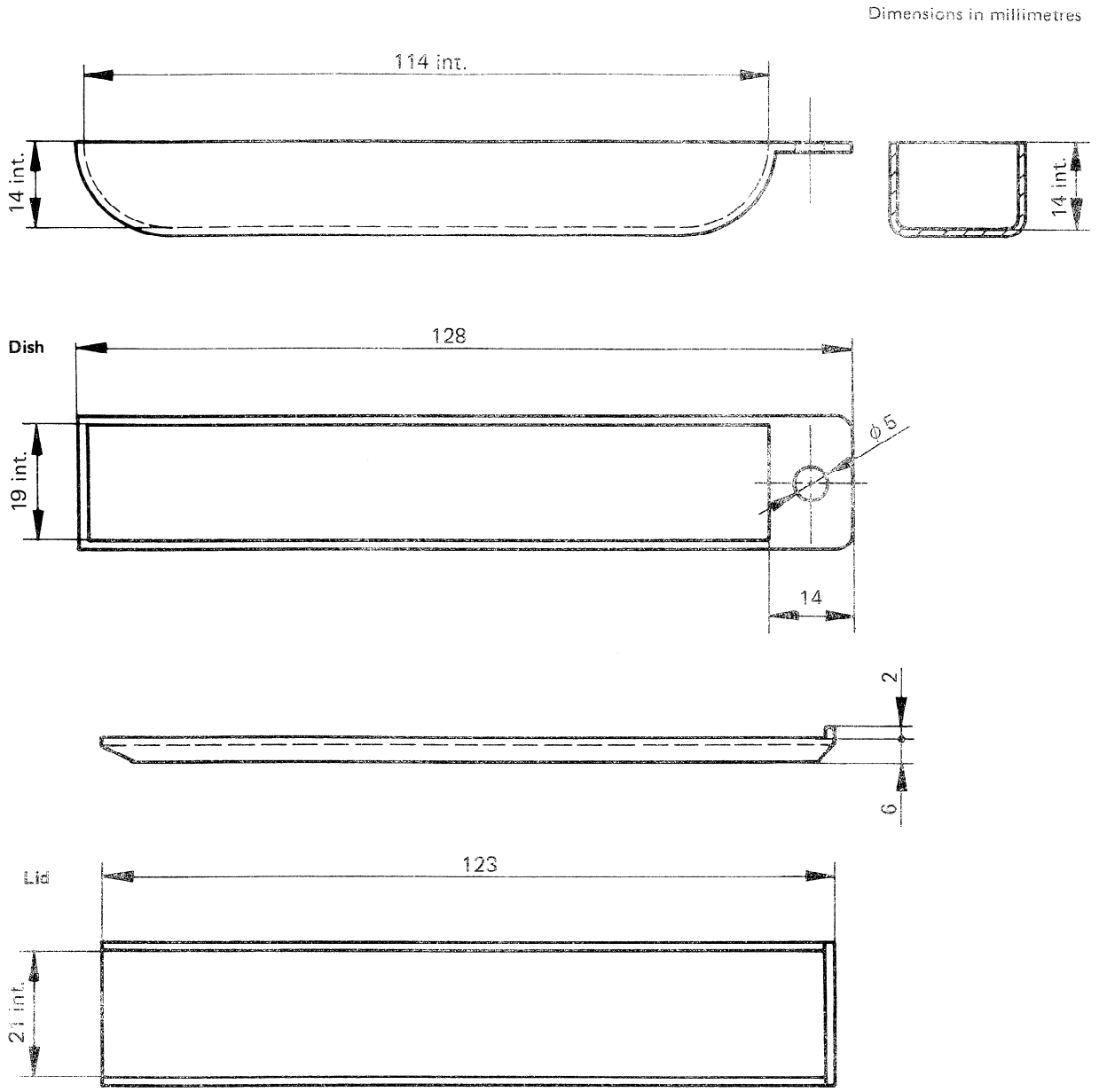
A.9.4 The period of rest of 24 h which follows the pre-drying is necessary to obtain uniform distribution of the moisture.

A.9.5 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.10 Test report

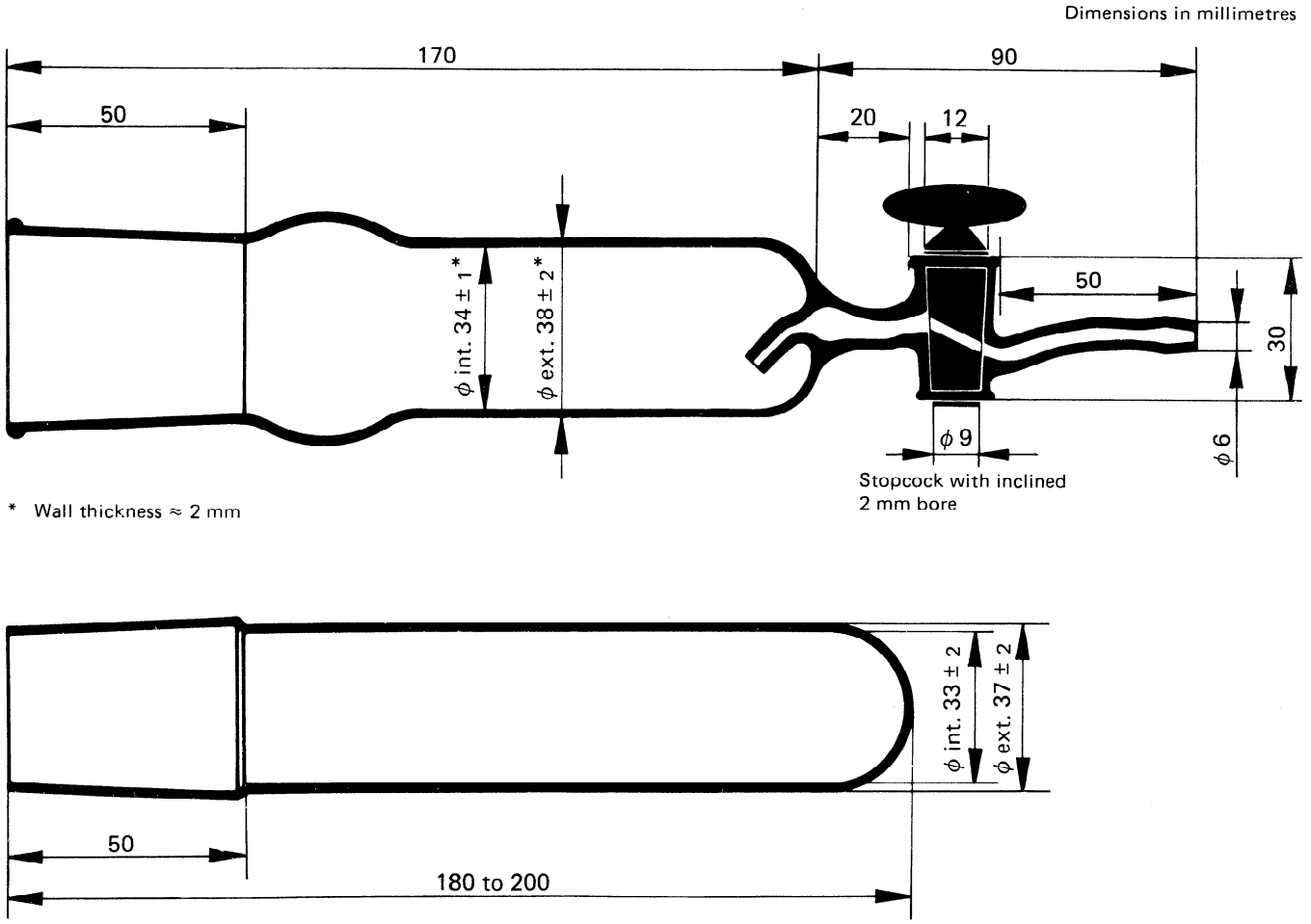
The test report shall show the method used and the result obtained. It shall also mention all operating details not specified in the annex of this International Standard, or regarded as optional, as well as any incidents which may have influenced the result.

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.



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 cube shown in figure 3.

Figure 2 — Diagram of suitable metal dish and lid (for guidance only)



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

Figure 3 — Diagram of suitable drying tube (for guidance only)

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