INTERNATIONAL STANDARD

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Green coffee — Determination of water content — Basic reference method

Café vert — Détermination de la teneur en eau — Méthode de référence fondamentale

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1446 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 15, *Coffee*.

This second edition cancels and replaces the first edition (ISO 1446:1978), which has been technically revised.

Annex A of this International Standard is for information only.

Green coffee — Determination of water content — Basic reference method

1 Scope

This international Standard specifies the basic reference method for the determination of the water content of green coffee.

This method is designed to serve as a standard for the checking and perfecting of methods suitable for the routine determination of the water content of green coffee.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 4072, *Green coffee in bags — Sampling*

ISO 6673, *Green coffee — Determination of loss in mass at 105 °C*

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
water content of green coffee
loss in mass undergone by the coffee when it is brought to true equilibrium with an atmosphere having zero water vapour pressure, under conditions such that interfering reactions are avoided

NOTE 1 In the present state at knowledge, it is considered that this loss in mass corresponds to the actual water in green coffee.

NOTE 2 The water content is expressed as a mass fraction in percent of the product as received [formerly expressed as % (m/m)].

4 Principle

The loss in mass is determined when the product (predried in the case of beans which are too moist), previously ground without alteration of its water content, is brought to equilibrium with an anhydrous atmosphere at a temperature of 48 °C ± 2 °C, at a pressure of 2,0 kPa ± 0,7 kPa [1)].

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Sulfuric acid, $\rho_{20} \geqslant 1{,}83$ g/ml.

5.2 Phosphorus(V) oxide (P_2O_5).

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Suction device, permitting pressure to be reduced to 2,0 kPa ± 0,7 kPa (e.g. a water pump).

6.2 Grinder, made of material which does not absorb moisture, and which:

— is easy to clean and has a minimum dead space;

— permits rapid and even grinding without producing appreciable heating and, as far as possible, without contact with outside air;

— can be regulated so as to obtain a ground product of which more than 90 % of the particules have a diameter of less than 1 mm and more than 50 % have a diameter of less than 0,5 mm.

6.3 Metal dish, non-corrodible, with a sufficiently tight-fitting lid, and with an effective surface area enabling the test portion to be distributed so as to give a mass per unit area of not more than 0,3 g/cm^2.

An example of a suitable dish tube is shown in annex A.

6.4 Glass or porcelain boat, containing phosphorus(V) oxide (5.2).

The effective surface area should, if possible, be at least equal to that of the metal dish (6.3).

6.5 Drying tube, of glass, in two parts, one of which, intended to receive the dish (6.3), is closed at one end, while the other, intended to receive the boat (6.4), carries a semi-capillary tube, with a stopcock, for evacuation purposes.

The two parts are connected by a ground glass joint. An example of a suitable drying tube is shown in annex A.

6.6 Electrically heated constant-temperature oven, or any other system enabling the part of the drying tube (6.5) containing the dish (6.3) to be brought to a temperature of 48 °C ± 2 °C.

6.7 Gas washing bottle, containing sulfuric acid (5.1).

6.8 Analytical balance, capable of weighing to the nearest 0,000 1 g.

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1) That is: 20 mbar ± 7 mbar or (approximately) 10 mmHg to 20 mmHg.

7 Preparation of test sample

7.1 Preliminary evaluation of the water content of the sample

Thoroughly mix the laboratory sample obtained as specified in ISO 4072, without modifying its water content.

Make an approximate determination of the water content, using either the routine method specified in ISO 6673 or a suitable rapid method.

7.2 Sampling

Rapidly take a sample of 3 g to 4 g of green coffee. If this quantity contains a heavy impurity (nail, stone, piece of wood, etc.), discard it and take a further quantity from the laboratory sample.

Each of the quantitites taken from the same laboratory sample, which form the test samples, shall be treated separately, including any predrying (7.3) and grinding (7.4).

7.3 Predrying

7.3.1 If the preliminary evaluation (7.1) indicates a water content greater than 11 % (mass fraction), dry the test sample as follows before grinding, since it is difficult to grind coffee which is too moist, and loss of water during grinding is to be expected.

7.3.2 Place the test sample (7.2) in the metal dish (6.3), previously dried and tared, and weigh to the nearest 0,000 2 g.

7.3.3 Place the metal dish in that part of the drying tube (6.5) which does not include the stopcock. In the part that includes the stopcock, place the boat (6.4), filled with a layer of phosphorus(V) oxide (5.2) approximately 10 mm thick, and fit together the two parts of the tube, having previously coated the ground glass joint with a suitable lubricant. Connect the tubing from the stopcock to the suction device (6.1) and reduce the pressure inside the apparatus to 2,0 kPa ± 0,7 kPa (see 6.1). Close the stopcock, remove the suction device and place the part of the tube containing the metal dish into one of the openings of the oven (6.6), the part containing the boat remaining outside the oven.

7.3.4 After drying for 2 h to 3 h, remove the tube from the oven and allow it to cool. Make sure that there is a sufficiently low pressure within the apparatus to prevent the ground glass joint from coming apart. Introduce into the tube air previously dried by bubbling through the sulfuric acid (5.1) contained in the gas washing bottle (6.7). Open the tube, remove the metal dish, fit its lid and weigh immediately to the nearest 0,000 2 g.

CAUTION When the pressure is being lowered or restored in the tube, the passage of air should be very gradual so as to avoid the movement of particles of powder (this may be achieved, for example, by the use of a semi-capillary tube).

7.3.5 If the loss in mass shows that the water content of the analysis sample has been reduced to below 11 %, immediately carry out the grinding operation (7.4).

7.3.6 If the water content is still high, renew the phosphorus(V) oxide contained in the boat and repeat the predrying operation described above until the water content of the analysis sample is approximately 8 % to 10 %.

The conditions of predrying are intended to bring the product more or less into hygrometric equilibrium with the atmosphere of a laboratory in which there is a temperature of 18 °C to 25 °C and a relative humidity of 50 % to 80 %. Should the conditions be appreciably different from the above, it is advisable to consider modifying the predrying.

7.4 Grinding

Place in the grinder (6.2) the test sample (7.2) or, if predrying has been necessary, the contents (see 7.3) of the metal dish. Grind. **Immediately** afterwards, take the test portion for the final drying.

8 Procedure

8.1 Test portion

Place in the metal dish (6.3), previously dried and tared, virtually all the powder obtained by grinding (7.4). Cover immediately and weigh to the nearest 0,000 2 g.

8.2 Determination

Proceed as indicated in 7.3.3. Renew the phosphorus(V) oxide as soon as it is no longer active.

CAUTION Observe the phosphorus(V) oxide to make sure that it remains active. If it does not (formation of skin, frosted appearance, etc.), replace it with fresh phosphorus(V) oxide.

After 80 h to 100 h, weigh, proceeding as indicated in 7.3.4.

Continue drying to constant mass (i.e. less than 0,000 5 g deviation between two weighings carried out at an interval of 48 h).

NOTE Drying at 48 °C ± 2 °C to constant mass generally requires from 150 h to 200 h.

8.3 Number of determinations

Carry out at least two determinations, each on a separate test sample (7.2).

9 Expression of results

9.1 Method of calculation

The water content of the sample as received, w, expressed as a mass fraction in percent, is given by the following formulae.

a) Without predrying

$$w = (m_2 - m_3) \times \frac{100}{m_2} \%$$

b) With predrying

$$w = \left[(m_2 - m_3) \frac{m_1}{m_2} + m_0 - m_1 \right] \times \frac{100}{m_0} \% = 100 \left(1 - \frac{m_1 \cdot m_3}{m_0 \cdot m_2} \right) \%$$

where

m_0 is the initial mass, in grams, of the test sample before predrying (7.3.2);

m_1 is the mass, in grams, of the test sample after predrying (7.3.4);

m_2 is the mass, in grams, of the test portion of the ground product (whether predried or not) (8.1);

m_3 is the mass, in grams, of the test portion after drying (8.2).

Take as the result the arithmetic mean of the two determinations, provided that the requirement concerning repeatability (see clause 10) is satisfied.

9.2 Alternative method of calculation

9.2.1 Predrying

The loss in mass, w_1, due to the elimination of part of the water during the predrying (7.3), expressed in grams per 100 g of the sample as received, is given by the formula:

$$w_1 = (m_0 - m_1) \times \frac{100}{m_0} \%$$

where

m_0 is the initial mass, in grams, of the test sample before predrying (7.3.2);

m_1 is the mass, in grams, of the test sample after predrying (7.3.4).

9.2.2 Final drying

The water loss, w_2, during the final drying (8.2), expressed in grams per 100 g of ground coffee, is given by the formula:

$$w_2 = (m_2 - m_3) \times \frac{100}{m_2} \%$$

where

m_2 is the mass, in grams, of the test portion of the ground product (whether predried or not) (8.1);

m_3 is the mass, in grams, of the test portion after drying (8.2).

9.2.3 Water content without predrying

The water content of the sample as received, w, expressed as a mass fraction in percent, is given by the formula:

$$w = w_2$$

9.2.4 Water content with predrying

The water content of the sample as received, w, expressed as a mass fraction in percent, is given by the formula:

$$w = w_1 + w_2 - \frac{w_1 w_2}{100} \%$$

10 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory with the same operator using the same equipment within a short interval of time, will not in more than 5 % of cases be greater than 0,2 g of water per 100 g of sample.