

ISO

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Revision

ISO RECOMMENDATION R 1446

GREEN COFFEE BEANS

DETERMINATION OF MOISTURE CONTENT
(BASIC REFERENCE METHOD)

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1446, *Green coffee beans – Determination of moisture content (Basic reference method)*, was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question led to the adoption of Draft ISO Recommendation No. 1446, which was circulated to all the ISO Member Bodies for enquiry in February 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Brazil	Iran	Romania
Chile	Israel	South Africa, Rep. of
Czechoslovakia	Netherlands	Spain
France	Norway	Turkey
Hungary	Poland	United Kingdom
India	Portugal	U.S.S.R.

The following Member Bodies opposed the approval of the Draft :

Colombia
U.S.A.

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

GREEN COFFEE BEANS

DETERMINATION OF MOISTURE CONTENT

(BASIC REFERENCE METHOD)

1. SCOPE

This ISO Recommendation describes the basic reference method for the determination of the moisture content of green coffee beans.

NOTE. This method is designed to serve as a standard for the checking and perfecting of methods particularly suitable for the routine determination of moisture in green coffee beans.

2. DEFINITION

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

In the present state of knowledge, it is considered that this loss in mass corresponds to the actual moisture in green coffee beans.

The moisture content is expressed as a percentage by mass.

3. PRINCIPLE

Determination of the loss in mass when the product, previously ground without alteration of its moisture content, is brought into equilibrium with an anhydrous atmosphere at a temperature of 48 ± 2 °C, at a pressure of 20 ± 7 mbar.*

Pre-drying of beans which are too moist.

4. APPARATUS

4.1 *Suction device* permitting a pressure of 20 ± 7 mbar to be obtained (for example, a water pump).

4.2 *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 grains have a diameter of less than 1 mm and more than 50 % have a diameter of less than 0.5 mm.

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

4.4 *Glass or porcelain boat* containing reagent-grade phosphorus pentoxide. The effective surface should if possible be at least equal to that of the metal dish (4.3).

* i.e. approximately 10 to 20 mmHg.

** See diagram of a metal dish, for information only, in Annex A.

- 4.5 *Drying tube*, of glass*, of which one part is closed at one end and the other part carries a semi-capillary tube with a stopcock for evacuation purposes, the two parts being connected by a conical ground joint.
- 4.6 *Electrically heated constant-temperature oven*, or any other system enabling the part of the drying tube containing the dish (4.3) to be brought to a temperature of 48 ± 2 °C.
- 4.7 *Wash bottle* with reagent-grade sulphuric acid of density $\rho_{20} \geq 1.84$ (g/ml).
- 4.8 *Analytical balance*.

5. PROCEDURE

5.1 Preparation of sample

Thoroughly mix the laboratory sample** without modifying its moisture content.

- 5.1.1 *Preliminary evaluation of the moisture content of the sample*. Make an approximate determination of the moisture content, using either the routine method***, or a suitable rapid method.

If this test indicates a moisture content greater than 11 %, dry the sample before grinding (see clause 5.1.3), since it is difficult to grind coffee which is too moist, and losses of moisture during grinding are to be expected.

If the preliminary test indicates a moisture content of less than 11 %, grind the sample without previous drying.

- 5.1.2 *Sample for analysis*. Rapidly draw a sample of 3 to 4 g of green coffee beans. If this test portion contains a heavy impurity (nails, stones, pieces of wood, etc.), discard it and draw a further quantity from the laboratory sample.

Each of the quantities taken from the same laboratory sample, which form the analysis samples, should be treated individually, including any pre-drying and the grinding.

- 5.1.3 *Pre-drying*. If the preliminary test has indicated a moisture content of more than 11 %, place the analysis sample (5.1.2) in the metal dish (4.3), previously dried and tared, and weigh to the nearest 0.0002 g.

Place the metal dish in that part of the drying tube (4.5) which does not include the tap. In the part including the tap, place the boat (4.4), filled with a layer of phosphorus pentoxide approximately 10 mm thick, and fit together the two parts of the tube, the ground joint having been previously coated with a suitable lubricant. Connect the tubing from the tap to the suction device (4.1) and reduce the pressure inside the apparatus to 20 ± 7 mbar (see clause 7.1). Close the tap, remove the suction device and place the part of the tube containing the analysis sample into one of the openings of the oven (4.6), the part containing the phosphorus pentoxide remaining outside the oven.

After a time which varies in accordance with the initial moisture content of the coffee (see clause 7.2), 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 joint from coming apart. Introduce into the tube (see clause 7.1) air previously dried by bubbling through the sulphuric acid contained in the wash bottle (4.7).

Open the tube, remove the metal dish, fit its lid and weigh immediately.

If the loss in mass is sufficient to bring the moisture content of the analysis sample below 11 %, immediately carry out the grinding operation (see clause 5.1.4).

If the moisture content is still too high, renew the phosphorus pentoxide contained in the boat and repeat the pre-drying operations described above until the moisture content of the analysis sample is approximately 8 to 10 % (see clause 7.3).

- 5.1.4 *Grinding*. Place in the grinder (4.2) 3 to 4 g of green coffee beans, or, if pre-drying has been necessary, the contents of the metal dish. Grind. *Immediately* afterwards take the test portion for the final drying.

* See diagram of a desiccating tube, for information only, in Annex B.

** See ISO Recommendation R . . . , *Green coffee beans -- Sampling* (in preparation). Pending the completion of that ISO Recommendation, the term "laboratory sample" is used in the English text, to denote the sample *as delivered* to the laboratory.

*** See ISO Recommendation R 1447, *Green coffee beans -- Determination of moisture content (Routine method)*.

5.2 Test portion

Place in the metal dish (4.3), previously dried and tared, virtually all the powder obtained by grinding (see clause 5.1.4), cover immediately and weigh to the nearest 0.0002 g.

5.3 Determination

Proceed as indicated in the second paragraph of clause 5.1.3; renew the phosphorus pentoxide as soon as it is no longer active (see clause 7.4).

After 80 to 100 hours, weigh (proceeding as indicated in the third paragraph of clause 5.1.3).

Continue dehydration to constant mass (less than 0.0005 g deviation between two weighing operations carried out at an interval of 48 hours) (see clause 7.5).

Carry out at least two determinations, each on a sample which has been treated separately, including any pre-drying and the grinding.

6. EXPRESSION OF RESULTS

6.1 Method of calculation and formulae

The moisture content of the sample, as a percentage by mass, is obtained from the following formulae :

(a) without pre-drying

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

(b) with pre-drying

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

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

where

m_0 is the initial mass, in grammes, of the analysis sample in the form of beans;

m_1 is the mass, in grammes, of the analysis sample after pre-drying;

m_2 is the mass, in grammes, of the test portion of the ground product (whether pre-dried or not);

m_3 is the mass, in grammes, of the dry material (see clauses 7.6 and 7.7).

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

6.2 Repeatability

The difference between the results of two determinations carried out simultaneously, or in rapid succession, by the same analyst should not be greater than 0.2 g of moisture per 100 g of sample.

7. NOTES

7.1 When low pressure is produced, and when pressure is restored in the tube, the passage of air should be very gradual so as to avoid the movement of particles of powder (for example, by the use of a semi-capillary tube).

7.2 The duration of pre-drying should be 2 to 3 hours.

- 7.3 The conditions of pre-drying 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 to 25 °C and a relative humidity of 50 to 80 %. Should the conditions be appreciably different from the above, it would be advisable to consider modifying the pre-drying
- 7.4 Observe the phosphorus pentoxide to make sure that it remains active, and if it does not (formation of a skin, frosted appearance, etc.), replace it with fresh phosphorus pentoxide.
- 7.5 Drying at 48 ± 2 °C generally lasts from 150 to 200 hours.
- 7.6 When the sample is pre-dried, the calculation takes into account the fact that the loss in mass during drying proper is expressed in grammes per 100 g of pre-dried and ground sample, and not of the initial sample.
- 7.7 The calculation may also be presented in the following manner :

7.7.1 *Pre-drying.* The loss in mass (P_1) due to the elimination of part of the water during pre-drying (see clause 5.1.3), expressed in grammes per 100 g of initial sample, is equal to

$$P_1 = (m_0 - m_1) \times \frac{100}{m_0}$$

where

m_0 is the initial mass, in grammes, of the analysis sample in the form of beans;

m_1 is the mass, in grammes, of the analysis sample after pre-drying.

7.7.2 *Final drying.* The moisture loss (P_2) during final drying (see clause 5.3), expressed in grammes per 100 g of coffee powder, is equal to

$$P_2 = (m_2 - m_3) \times \frac{100}{m_2}$$

where

m_2 is the mass, in grammes, of the test portion (see clause 5.2) of the ground product (whether pre-dried or not);

m_3 is the mass, in grammes, of the dry material.

7.7.3 *Moisture content without pre-drying.* The moisture content of the sample, as a percentage by mass, is equal to

$$P = P_2$$

7.7.4 *Moisture content with pre-drying.* The moisture content of the initial sample (P), as a percentage by mass, is equal to

$$P = P_1 + P_2 - \frac{P_1 P_2}{100}$$

8. TEST REPORT

The test report should show the method used and the result obtained. It should specify any intermediate results such as loss in mass during pre-drying and successive losses in mass during final drying. It should also mention any operating conditions not specified in this ISO Recommendation, or regarded as optional, and any circumstances that may have influenced the result.

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