INTERNATIONAL STANDARD

ISO 11817

First edition
1994-10-01

Roasted ground coffee — Determination of moisture content — Karl Fischer method (Reference method)

Café torréfié moulu — Détermination de la teneur en eau — Méthode de Karl Fischer (Méthode de référence)

Reference number
ISO 11817:1994(E)

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.

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.

International Standard ISO 11817 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 15, *Coffee*.

Annex A of this International Standard is for information only.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Roasted ground coffee — Determination of moisture content — Karl Fischer method (Reference method)

1 Scope

This International Standard specifies a method for the determination of moisture content of roasted ground coffee by the Karl Fischer titration method. Since it is precise, it is suitable as a reference method.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 760:1978, *Determination of water — Karl Fischer method (General method).*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 moisture content: Content of water, extracted with dried methanol in accordance with the procedure specified in this International Standard.

Moisture content is expressed as a percentage by mass.

4 Principle

Extraction of the test portion with dried methanol at 65 °C (boiling temperature) under reflux with the exclusion of moisture. After cooling, titration of an aliquot part of the retained extract in a Karl Fischer apparatus until the end-point of the titration is reached according to the biamperometric method.

5 Reaction

During the determination of moisture content according to the Karl Fischer method, the water present in the sample reacts in the presence of an amine and an alcohol with iodine and sulfure dioxide:

$$H_2O + I_2 + SO_2 + ROH + 3R_nNH_{3-n} \rightarrow$$
$$2R_nNH_{3-n}\cdot HI + R_nNH_{3-n}\cdot HSO_4R$$

where R is an alkyl or alkoxyl group.

The end-point of the reaction is obtained electrometrically by a surplus of iodine.

6 Reagents and materials

Use only reagents of recognized analytical quality and distilled or demineralized water or water of equivalent purity.

6.1 Pyridine-free Karl Fischer reagent[1], one- or two-component system.

6.2 Methanol, containing not more than 0,01 % (*m/m*) of water.

6.3 Molecular sieve, 0,3 nm, pearl shaped; diameter approx. 2 mm; bulk density (loose) approx. 75 g per 100 ml.

1) Suitable products are commercially available.

7 Apparatus

Usual laboratory apparatus and, in particular, the following.

7.1 Drying oven, capable of being operated at 40 °C ± 5 °C, for constant storage of glassware.

7.2 Analytical balances.

7.2.1 Ultra-microbalance, capable of weighing to an accuracy of 0,000 1 g, for the determination of the titre of the Karl Fischer reagent.

7.2.2 Microbalance, capable of weighing to 0,001 g, for weighing the test portion.

7.3 Reflux apparatus.

7.3.1 Heating device, suitable for a 100 ml round-bottomed flask; heating rate to be maintained by means of a thyristor regulator.

7.3.2 Round-bottomed flask, of 100 ml capacity, with conical sleeve.

NOTE 1 At least three flasks are required.

7.3.3 Reflux condenser, 25 cm to 30 cm in length, with conical ground joint.

7.3.4 Drying tubes, filled with approx. 40 g of molecular sieve (6.3), changed daily.

7.4 Titration apparatus.

NOTE 2 For the determination of the moisture content according to the Karl Fischer method, complete titration apparatuses which consist of the single parts given in 7.4.1 to 7.4.5 are commercially available.

7.4.1 End-point indicator, for titration according to the biamperometric method.

7.4.2 Platinium double electrode.

7.4.3 Magnetic stirrer, fitted with a stirring rod covered with polytetrafluoroethylene.

7.4.4 Titration vessel, of approx. 100 ml capacity, with at least three ground glass sockets.

Connect one of the ground glass sockets to the burette, place the platinium double electrode in the second one, and use the third one to add the reagents and the sample. A discharge tap at the bottom of the vessel is an advantage. Connect it to the vessel with a capillary tube having a volume of not more than 0,5 ml. Titration vessels without a discharge tap should have four ground glass sockets. Empty them by means of a glass tube drawn out up to a capillary reaching the bottom of the vessel.

7.4.5 Burette, graduated in intervals of 0,05 ml, **reagent bottle** and **drying tubes**.

When using automatic titration devices, the minimum interval depends on data from the manufacturer (e.g. 0,02 ml).

Protect the reagent bottle, the burette and the Karl Fischer reagent (6.1) from light. Close all the ventilation sockets in the titration vessel either with drying tubes which have been charged with an effective drying agent or molecular sieve, or with drying receivers filled with the reagent (6.1) in order to exclude the influence of moisture from the air.

7.5 Syringe, for measuring 30 µl to 40 µl of water for the determination of the titre, e.g. a 0,05 ml Hamilton microsyringe.

7.6 Desiccator, containing an effective drying agent, e.g. silica gel with moisture indicator, or molecular sieve.

7.7 Measuring cylinder, of capacity 100 ml, graduated in 1 ml.

7.8 Pipettes, of capacities 5 ml, 10 ml and 50 ml.

8 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

9 Preparation of test sample

9.1 Roasted ground coffee

Mix thoroughly the laboratory sample.

9.2 Vacuum-packed roasted ground coffee

One original packing unit shall be considered as the test sample.

10 Procedure

10.1 Determination of titre of the Karl Fischer reagent

Determine the titre of the reagent (6.1) in accordance with ISO 760.

For an exact dosage of smaller amounts of water, use the syringe (7.5), the discharge of which is obtained by difference weighing with the ultra-microbalance (7.2.1).

Determine the volume required by several titrations (10.6) of 30 mg to 40 mg of water and calculate the titre, T, according to the following formula:

$$T = \frac{m}{V}$$

where

m is the mass, in milligrams, of water used;

V is the volume, in millilitres, of the reagent (6.1) used.

10.2 Preparation of glassware

Keep all required glassware in the oven (7.1) set at 40 °C. Prior to use, allow the glassware to cool to room temperature in the desiccator (7.6) and store it there.

10.3 Preparation of reflux apparatus

Place 75 ml of methanol (6.2) in a 100 ml round-bottomed flask (7.3.2). Adjust the heating rate of the heating device (7.3.1), with the cooling water supply closed, by means of the thyristor regulator so that the methanol condenses at the upper end of the reflux condenser (7.3.3). Note the adjustment. Then close the apparatus with the drying tube (7.3.4) and boil the methanol at an unchanged heating rate for 30 min under reflux. Open the cooling water supply and allow the apparatus to cool. Do not remove the flask until the apparatus has cooled to room temperature.

After this preparation, remove any adhering water from the inner surface of the reflux condenser. Immediately after having removed the flasks, close the reflux apparatus thus prepared with a round-bottomed flask containing an effective drying agent (molecular sieve or silica gel with humidity indicator).

10.4 Determination of blank value

Pipette 50,0 ml of the dried methanol into a dried 100 ml round-bottomed flask (7.3.2) and boil with running cooling water for 30 min. Then allow the round-bottomed flask to cool, remove it from the apparatus and close the flask immediately.

Take an aliquot part of 10,0 ml for titration of the blank value and titrate it as described in 10.5 and 10.6. Record the volume of Karl Fischer reagent used.

10.5 Extraction of samples

Weigh, to the nearest 0,001 g, approx. 3 g of the test sample (clause 9) into a 100 ml round-bottomed flask. Add 50,0 ml of dried methanol by means of a pipette and close the flask immediately.

Join the round-bottomed flask to the reflux condenser and heat the contents at the same heating rate as described in 10.3 (noted thyristor regulation) with running cooling water up to the start of reflux. Boil for a further 30 min at reflux. Then allow the round-bottomed flask to cool to room temperature, remove it from the reflux condenser and close the flask immediately.

10.6 Preparation of the titration apparatus

Prepare the titration apparatus (7.4) in accordance with the manufacturer's instructions. The air-tightness of the titration vessel is of special importance.

NOTE 3 A measure of lack of air-tightness is the leakage rate (µg H_2O per min) of the system. This can be between 10 µg/min and 60 µg/min depending on the ambient air humidity. The determination of the leakage rate is made in accordance with the instructions of the manufacturer of the Karl Fischer titrator used and should not exceed 60 µg/min for the determination.

Place 35 ml of the dried methanol (receiver) in the titration vessel. Take care that the metallic parts of the platinum double electrode (7.4.2) are immersed completely.

Before the first daily use, condition the system in a so-called stand-by titration for 20 min when the adhered water is removed completely.

10.7 Determination

Depending on the expected water content of the roasted ground coffee samples, take an aliquot part of 10,0 ml [water content < 3 % (*m/m*)] or 5,0 ml [water content > 3 % (*m/m*)] from the sample extracted in accordance with 10.5 and pipette it rap-

idly into the titration vessel prepared in accordance with 10.6, opening the vessel for as short a time as possible.

Start the automatic titration while stirring with the magnetic stirrer (7.4.3). The dosage of the reagent and the recognition of the end-point is controlled automatically by the apparatus.

Adjust the apparatus so that the electronic titration ends if no potential change of more than 15 mV occurs within 20 s.

Carry out three titrations on each extract (or for the determination of the blank value). Do not change the receiver (see 10.6). Use the arithmetic mean of the results of these three titrations as one value for the calculation (11.1).

11 Expression of results

11.1 Calculation

The moisture content of roasted ground coffee, w_w, expressed as a percentage by mass of the sample, is calculated according to the following formula:

$$w_w = \left(V_4 - \frac{V_3 \times V_2}{V_5} \right) \times T \times \frac{V_1}{V_2} \times \frac{100}{m_E}$$

where

T is the titre (10.1) of the Karl Fischer reagent, in milligrams of water per millilitre;

m_E is the mass of the test portion, in milligrams;

V_1 is the volume of methanol, in millilitres, used for the extraction (10.5);

V_2 is the aliquot volume of the extract, in millilitres, used for the titration;

V_3 is the volume, in millilitres, of the Karl Fischer reagent (6.1) used for the titration of the blank value (10.4);

V_4 is the volume, in millilitres, of the Karl Fischer reagent (6.1) used for the titration of the sample extract (10.7);

V_5 is the aliquot volume of methanol, in millilitres, used for the determination of the blank value (10.4).

Round the result to one decimal place.

11.2 Precision

Results of an interlaboratory test are given in annex A. The interlaboratory test was carried out using vacuum-packed samples exclusively.

11.2.1 Repeatability

The absolute difference between two independent test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, should not be greater than 0,14 %.

11.2.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, should not be greater than 0,25 %.

12 Test report

The test report shall specify

— the method used,

— the test result obtained,

— if the repeatability has been checked, the final quoted result obtained.

It shall also mention any operating details not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all information required for the complete identification of the sample.

Annex A
(informative)

Results of interlaboratory test

An interlaboratory test carried out in 1988, by the Deutsches Institut für Normung, in which nine laboratories participated, each of which carried out two determinations on the sample, gave the statistical results (evaluated in accordance with ISO 5725[2]) shown in table A.1.

Table A.1 — Moisture content of roasted coffee

Number of laboratories retained after eliminating outliers	9
Mean moisture content, % (*m/m*)	4,21
Standard deviation of repeatability, s_r	0,050 6
Coefficient of variation of repeatability, %	1,2
Repeatability 2,83s_r	0,14
Standard deviation of reproducibility, s_R	0,086 6
Coefficient of variation of reproducibility, %	2,5
Reproducibility 2,83s_R	0,24

2) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ICS 67.140.20

Descriptors: agricultural products, plant products, coffee, tests, determination of content, water, Karl Fischer method.

Price based on 5 pages