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

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

Reference number
ISO 20938:2008(E)

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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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20938 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 15, *Coffee*.

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

1 Scope

This International Standard specifies a method for the determination of moisture content in instant coffee by the Karl Fischer titration method, suitable for use as a reference method.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
moisture content
content of water, determined according to the procedure specified in this International Standard

NOTE The moisture content is expressed as a percentage mass fraction.

4 Principle

4.1 General

In a Karl Fischer apparatus, a test portion of instant coffee is dissolved in a water-free solution of formamide, methanol, and salicylic acid (FMS). Subsequent titration with a Karl Fischer reagent is applied under continuous stirring until the endpoint of the titration is reached and electrometrically detected. The volume of Karl Fischer reagent consumed is used to calculate the moisture content of the test portion.

4.2 Reaction

During the Karl Fischer titration, water present in the sample reacts with iodine and sulfur dioxide in the presence of an amine and an alcohol (e.g. methanol):

$$\mathrm{H_2O + I_2 + SO_2 + ROH + 3R}_n\mathrm{NH}_{3-n} \rightarrow \mathrm{2R}_n\mathrm{NH}_{3-n} \cdot \mathrm{HI + R}_n\mathrm{NH}_{3-n} \cdot \mathrm{HSO_4R}$$

where R is an alkyl or alkoxyl group.

The endpoint of the reaction is detectable by a small, surplus amount of iodine and quantified electrometrically (either amperometrically or voltametrically). The procedure described in this International Standard is performed only with pyridine-free Karl Fischer (one-component) reagent (KFR).

5 Reagents and materials

Unless otherwise specified, use only reagents of recognized analytical grade, and only distilled or deionized water or water of equivalent purity.

5.1 Karl Fischer reagent (KFR, one-component system)[1], titre approximately 5 mg/ml, water mass concentration, pyridine-free.

5.2 Methanol, with low water mass concentration, preferably $< 0,5$ mg/ml.

5.3 Formamide, with low water mass concentration.

WARNING — The use of formamide is of outstanding importance with regard to the time of titration and the clear determination of the endpoint. Operators should be carefully instructed and adequately trained so as to avoid direct contact with the reagent (use splash guards and protective clothing), and to wash immediately and thoroughly both in cases of exposure to formamide, as well as at the end of the workshift.

5.4 Salicylic (2-hydroxybenzoic) acid.

NOTE Salicylic acid is applied for reasons of stabilizing the pH of the solution. Under these conditions, uncontrolled side reactions do not take place.

5.5 Disodium tartrate (disodium 2,3-dihydroxybutanedioate) dihydrate.

6 Apparatus

Usual laboratory equipment and in particular, the following. All devices used shall be as dry as possible.

6.1 Analytical balance, capable of weighing to an accuracy of 0,1 mg.

6.2 Titration apparatus, for the determination of the moisture content according to the Karl Fischer method. Complete sets of titration apparatus are commercially available, consisting of the individual components given in 6.2.1 to 6.2.5.

6.2.1 Karl Fischer titrator, for titrations according to the amperometric or the voltametric procedure.

6.2.2 Platinum double electrode.

6.2.3 Magnetic stirrer equipment, fitted with a polytetrafluoroethylene-coated stirring bar.

6.2.4 Titration vessel, of approximately 100 ml capacity, with at least four basic glass sockets. One of these is connected to the burette (6.2.5), the second to the platinum double electrode (6.2.2). The third serves for adding the FMS solution (8.1.1) and the fourth for the introduction of the sample. The latter shall be tightly closed during titration by a drying tube or receiver (6.2.5).

NOTE Attaching thermostatic mantling to the titration vessel enables an option of slightly raising the temperature, resulting in reaction acceleration.

6.2.5 Burettes, reservoirs and drying tubes.

A piston burette is used for accurately measuring the volume of KFR (5.1). The reagent bottle, burette and connecting tubes for transferring KFR should be protected from light. All ventilation sockets of the system shall be closed by either drying tubes that contain effective drying agents (e.g. silica gel with moisture indicator or molecular sieves), or with drying receivers filled with KFR in order to exclude any ambient moisture.

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1) Suitable products are commercially available.

6.3 Syringe or **weighing pipette**, suitable for measuring 25 mg to 30 mg water in the determination of the KFR titre according to 8.2.2.

If determining water content of liquid coffee extract, a syringe can also be used to measure the liquid coffee sample and to transfer it into the titration vessel.

6.4 Weighing spoon, made of glass with a protective cover, purchased from the titration apparatus supplier.

6.5 Graduated measuring cylinder, in accordance with ISO 4788, class A, of capacity 500 ml.

7 Sampling

7.1 General

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. The sampling procedure shall be subject to agreement by the interested parties.

7.2 Sample preparation

The sample should be truly representative and well homogenized, without damage or change during transportation and storage. To protect the laboratory sample from ambient moisture, it shall be transferred to an airtight glass jar immediately after opening the original packaging.

NOTE Instant coffee is highly hygroscopic.

8 Preparative steps

8.1 Preparation of solvent mixture and receiver for titration

8.1.1 Preparation of the formamide-methanol-salicylic acid-mixture (FMS)

For better solubility, first dissolve salicylic acid (5.4) in methanol (5.2), after which add formamide (5.3). In the FMS mixture, the ratio of formamide to methanol shall be 1 + 2 (parts by volume).

EXAMPLE 1 A mass of 60 g salicylic acid (5.4) is dissolved in 200 ml methanol (5.2) in a stoppered cylindrical flask under continuous stirring. A volume of 100 ml formamide (5.3) is subsequently added, the flask is then closed and the FMS completely homogenized by further stirring.

Direct preparation of the mixture in the titration vessel is also feasible.

EXAMPLE 2 A volume of 10 ml formamide (5.3) is placed in the titration vessel, then mixed with 20 ml methanol (5.2) in which 6 g salicylic acid (5.4) have been previously dissolved.

Prepare fresh solution daily.

NOTE FMS stabilizes the pH value during the Karl Fischer titration.

8.1.2 Conditioning of the titration system

Transfer 30 ml FMS into the titration vessel, optionally using a second piston burette (6.2.5). Allow the system to condition in a so-called stand-by titration with KFR for 20 min, during which time adherent, residual water is completely removed.

The electrometric default parameters are the polarization current, I_{pol}, of 50 µA, the titration stop at about 60 % to 80 %, with a waiting time of 30 s. Manufacturers' instructions for use, both for the titration apparatus and reagents, should be adhered to.

The buffer capacity of the Karl Fischer one-component reagent, in combination with the FMS receiver, varies according to the reagent used, and the type of coffee extract analysed. It is feasible that — depending on the water content — more than one titration may be performed in the same receiver. The course of titration can be used for reaction control (see Figure B.1). The titration time may prove a suitable indicator.

8.2 Determination of KFR titre

8.2.1 Determination with disodium tartrate dihydrate

Weigh approximately 0,12 g disodium tartrate dihydrate (5.5) (Karl Fischer primary titre substance) to the nearest 0,1 mg in a closed weighing spoon (6.4) to determine the gross mass, and transfer it via the appropriate socket into the titration vessel containing the conditioned, water-free FMS.

Close the titration vessel again and immediately initiate the titration under continuous stirring. Weigh the weighing spoon back to the nearest 0,1 mg to give the tare.

At the end of titration, note the volume of KFR used to the nearest 0,01 ml.

8.2.2 Determination with water (alternative method)

Fill the syringe (6.3) with water, drain to about 0,2 ml, and, after the surface of the injection needle has been dried, weigh the syringe on the analytical balance (6.1) to the nearest 0,1 mg (gross mass). Insert the injection needle of the syringe through the appropriate socket into the titration vessel. Once the needle has almost reached the solution surface, inject approximately 25 mg to 30 mg water into the FMS receiver.

The water shall be added quickly, trying to reduce the influence of ambient air humidity; in order to accelerate water addition, the number of water drops equivalent to the mass of water needed should be determined in a pre-trial with the syringe.

Close the titration vessel again and immediately start titration under continuous stirring. Weigh the syringe back to the nearest 0,1 mg to give the tare.

At the end of titration (same default values applied), note the volume of KFR consumed to the nearest 0,01 ml.

Using a weighing pipette (6.3), the introduction of water may be performed in an analogous manner.

NOTE Other water standards can be used alternatively.

8.2.3 Calculation of titre

Calculate the titre, ρ, according to Equation (1); see also Clause 10:

$$\rho = \frac{m_{\text{H}_2\text{O}}}{V} \tag{1}$$

where

$m_{\text{H}_2\text{O}}$ is the mass, in milligrams, of water used as titre (gross mass minus tare);

V is the volume, in millilitres, of KFR used.

When determining the titre with disodium tartrate dihydrate, the mass of the tartrate used corresponds to the mass of water as follows:

$$m_{\mathrm{H_2O}} = 0{,}156\ 6\ m_{\mathrm{T}} \tag{2}$$

where m_{T} is the mass, in milligrams, of tartrate used as titre (gross mass minus tare). A mass of 1 g disodium tartrate dihydrate corresponds to 0,156 6 g water.

Carry out the determination in duplicate, and use the mean as the resulting titre.

Although pyridine-free one-component reagents generally show high stability, check the titre from time to time.

9 Procedure with instant coffee

9.1 Instant coffee

Place 30 ml FMS (8.1.1) in the titration vessel and condition via a pre-titration (8.1.2).

Weigh a test portion of instant coffee (between 300 mg and 500 mg) to the nearest 0,1 mg and transfer it into the titration vessel (8.2.1).

Close the titration vessel and start the titration immediately, without changing any titration parameters up to the endpoint.

In order to exclude ambient air humidity, the protective cover of the weighing spoon shall be used in all cases during the weighing procedure.

9.2 Liquid coffee extract

Perform the moisture content determination in liquid coffee extract concentrates according to the procedure given in 8.2.2. Use a microlitre syringe (6.3) to measure the volume of the extract sample.

10 Expression of results

Calculate the moisture content in the coffee extract, $w_{\mathrm{H_2O}}$, expressed as a percentage mass fraction, according to Equation (3)

$$w_{\mathrm{H_2O}} = \frac{\rho_{\mathrm{KFR}}\ V_1}{10\ m_{\mathrm{E}}} \tag{3}$$

where

ρ_{KFR} is the KFR titre, in milligrams of water per millilitre KFR, as determined in 8.2;

V_1 is the volume, in millilitres, of KFR consumed in the sample titration;

m_{E} is the mass, in grams, of the test portion.

Take as the result the arithmetic mean of two determinations which satisfy the repeatability limits given in Table 1.

11 Precision

11.1 Interlaboratory test

Detailed results of an interlaboratory test are summarized in Annex A, the values of interest are given in Table 1.

The interlaboratory test was carried out using three types of coffee extract, spray dried, agglomerate (often called granules) and freeze dried.

Table 1 — Means, repeatability limits and reproducibility limits

Sample	Mean $\bar{w}_{H_2O}$ g/100 g	Repeatability limit r g/100 g	Reproducibility limit R g/100 g
Instant coffee, spray dried (sample A)	4,442	0,234	0,336
Instant coffee, agglomerate (sample B)	4,598	0,193	0,329
Instant coffee, freeze dried (sample C)	1,722	0,129	0,289

Precision data derived from this interlaboratory test may not apply to other types of products or to other ranges of moisture content than those reported here.

11.2 Repeatability

The absolute difference between two independent test results, obtained using the same method with identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, may not exceed the repeatability limits, r, given in Table 1 in more than 5 % of cases (i.e. repeatability at a 95 % probability).

The overall repeatability limit narrows to 0,2 % (mass fraction).

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method with identical test material in different laboratories with different operators using different equipment, may not exceed the reproducibility limits, R, given in Table 1 in more than 5 % of cases (i.e. reproducibility at a 95 % probability).

The overall reproducibility limit narrows to 0,3 % (mass fraction).

12 Test report

The test report shall include at least the following information:

a) all information required for complete identification of the sample;

b) the method used, with reference to this International Standard;

c) the test results obtained and their units;

d) date when the analysis was completed;

e) whether the repeatability requirements are met;

f) any operating details not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

Annex A
(informative)

Results of interlaboratory test

The precision of the method was evaluated by DIN in an interlaboratory test with the co-operation of other laboratories using different reagent suppliers.

Three different samples were analysed. The 11 laboratories participating carried out at least two determinations on each sample, some of them ran two series.

The statistical results, evaluated in accordance with ISO 5725-1[1] and ISO 5725-2[2], are given in Table A.1.

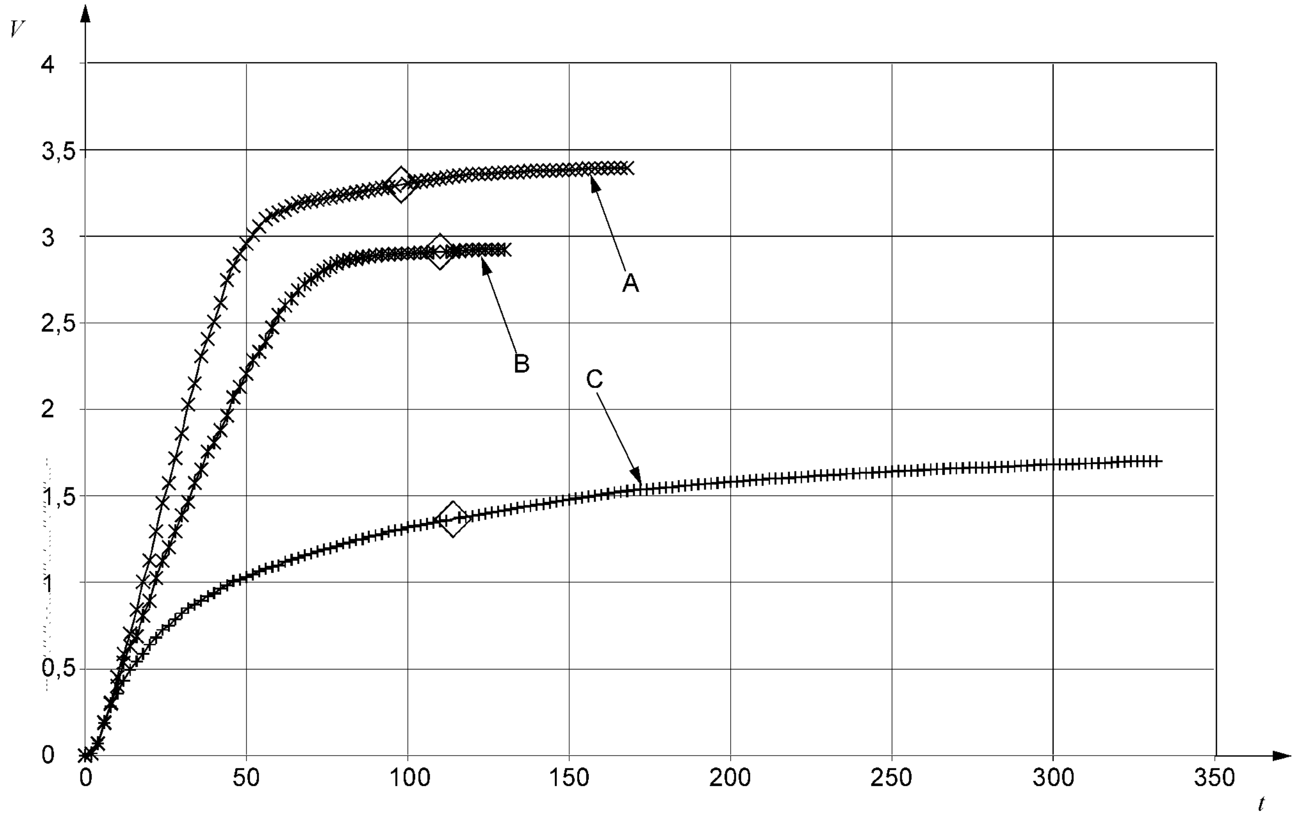
Table A.1 — Moisture content of instant coffee

Parameter	**Sample A** spray dried	**Sample B** agglomerate (granules)	**Sample C** freeze dried
Year of test	2002	2002	2002
No. laboratories participating	11	11	11
No. test series submitted by the laboratories participating	13	13	13
No. test series retained after elimination of outliers	13	13	10
Mean moisture content, $\overline{w}_{H_2O}$, % mass fraction	4,442	4,598	1,722
Standard deviation of repeatability, s_r	0,083	0,069	0,046
Coefficient of variation of repeatability, CV(r), %	1,9	1,5	2,7
Repeatability limit, r (2,8s_r)	0,234	0,193	0,129
Standard deviation of reproducibility, s_R	0,120	0,117	0,103
Coefficient of variation of reproducibility, CV(R), %	2,7	2,6	6,0
Reproducibility limit, R (2,8s_R)	0,336	0,329	0,289

Annex B
(informative)

Titration curve diagram

The follow-up of the Karl Fischer titration for the three types of instant coffee analysed was recorded by one participant, see Figure B.1.



Key

A	sample A — spray dried	t	time, s
B	sample B — agglomerate	V	KFR volume, ml
C	sample C — freeze dried		

Figure B.1 — Moisture determination in instant coffees according to the Karl Fischer method (Titration points equivalent to mean values are indicated by diamonds)

Bibliography

[1] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[2] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[3] ISO 5725-6:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

[4] ISO Guide 30:1992, *Terms and definitions used in connection with reference materials*

[5] THOMPSON, M., ELLISON, S. L. R., WOOD, R. The International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories [IUPAC Technical Report]. *Pure Appl. Chem.* 2006, **78**, pp. 145-196

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