
International Standard



3726

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Instant coffee — Determination of loss in mass at 70 °C under reduced pressure

Café soluble — Détermination de la perte de masse à 70 °C sous pression réduite

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3726 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in April 1982.

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It has been approved by the member bodies of the following countries :

ISO 3726:1983

Australia	India	Romania
Brazil	Iran	South Africa, Rep. of
Canada	Iraq	Sri Lanka
Chile	Israel	Thailand
Czechoslovakia	Jamaica	Turkey
Egypt, Arab Rep. of	Malaysia	United Kingdom
Ethiopia	Netherlands	USA
France	New Zealand	USSR
Germany, F.R.	Poland	Venezuela
Hungary	Portugal	Yugoslavia

No member body expressed disapproval of the document.

Instant coffee — Determination of loss in mass at 70 °C under reduced pressure

0 Introduction

In the method described in this International Standard, a temperature of 70 °C and an absolute pressure of 5 000 Pa are used, since higher temperatures may cause decomposition of carbohydrates normally present in instant coffee, resulting in the formation of water as a reaction product.

The drying period of 16 h has been chosen because tests on instant coffees representative of those on the market demonstrated that no further loss in mass occurred when the drying period was extended.

1 Scope and field of application

This International Standard specifies a method for the determination of the loss in mass at 70 °C, under reduced pressure, of instant coffee.

It is applicable to all types of instant coffee, as defined in ISO 3509. It does not apply to liquid coffee extracts.

2 References

ISO 3509, *Coffee and its products — Vocabulary*.

ISO 6670, *Instant coffee in cases with liners — Sampling*.¹⁾

3 Definition

loss in mass at 70 °C under reduced pressure : Principally water and small quantities of volatile matter vaporized under the conditions specified in this International Standard, and expressed as a percentage by mass.

4 Principle

Heating a test portion at 70 °C for 16 h under reduced pressure (5 000 Pa).

5 Apparatus

Usual laboratory apparatus, and in particular :

5.1 Isothermal vacuum oven, electrically heated and adjustable so that the temperature of the shelves can be controlled at 70 ± 1 °C.

5.2 Oven, capable of being controlled at 103 ± 2 °C.

5.3 Vacuum pump, capable of reducing the pressure in the oven (5.1) to $5\,000 \pm 100$ Pa.²⁾

5.4 Dish, flat-bottomed, with a closely fitting lid, resistant to attack under the conditions of the test, made, for example, of stainless steel or glass, of diameter approximately 50 mm and of height 30 mm.

5.5 Air drying apparatus, consisting of two washing bottles made of glass, filled with glycerol, to form a bubble train, and two drying towers made of glass containing a desiccant.

The bubble train and the drying system are connected in series with the vacuum oven (5.1), the drying towers being between the oven and the bubble train.

5.6 Desiccator, containing an efficient desiccant, for example phosphorus(V) oxide or freshly dried silica gel.

5.7 Analytical balance.

1) At present at the stage of draft.

2) $5\,000\text{ Pa} = 5\,000\text{ N/m}^2 = 50\text{ mbar} = 37,5\text{ mmHg}$

6 Sampling

See ISO 6670.

It is important to proceed as rapidly as possible when samples are exposed to the atmosphere, in order to prevent any pick up or loss of moisture according to the humidity of the air.

7 Procedure

7.1 Preparation of the dish

Dry the dish (5.4) and its lid for 1 h in the oven (5.2), controlled at 103 ± 2 °C.

Remove the dish and its lid from the oven and allow them to cool to room temperature in the desiccator (5.6) for about 30 min.

Weigh the dish and lid to the nearest 0,1 mg.

7.2 Test portion

Place a test portion of approximately 3 g into the prepared dish (7.1) and spread it uniformly over the bottom of the dish.

Cover the dish with its lid and weigh the whole to the nearest 0,1 mg.

NOTE — If performing a series of tests, prepare dishes as described in 7.1, and place the covered and weighed dishes in the desiccator in order to avoid any pick up or loss of moisture.

7.3 Determination

Place the dish containing the test portion, with the lid removed but alongside the dish, in the oven (5.1).

Set the oven at 70 ± 1 °C and reduce the pressure slowly (during at least 2 min), using the vacuum pump (5.3), to $5\,000 \pm 100$ Pa.

Allow dry air to enter the oven slowly through the drying system (5.5) at such a rate that one bubble per second passes through the bubble train.

Dry for $16 \pm 0,5$ h, maintaining the current of dry air. At the end of the drying period, allow air to enter the oven slowly (for 2 to 3 min) in order to avoid any air turbulence that may carry away part of the product from the dish.

Fit the lid on the dish and place it in the desiccator (5.6). Allow to cool to room temperature for at least 25 min, and weigh to the nearest 0,1 mg.

7.4 Number of determinations

Carry out two determinations on the same test sample.

8 Expression of results

The loss in mass at 70 °C under reduced pressure, expressed as a percentage by mass, is equal to

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

where

m_0 is the mass, in grams, of the dish and lid (see 7.1);

m_1 is the mass, in grams, of the dish, test portion and lid before drying (see 7.2);

m_2 is the mass, in grams, of the dish, test portion, and lid after drying (see 7.3).

Take as the result the arithmetic mean of the two determinations (see 7.4).

Express the result to the second decimal place.

9 Precision

An inter-laboratory test, carried out at the international level, in which 14 laboratories, each performing two replicates, participated, gave the statistical information (evaluated in accordance with ISO 5725¹⁾) summarized in the following table.

Table 1
Results expressed as percentages by mass

Sample	A	B
Number of laboratories retained after eliminating outliers	10	10
Mean	3,35	4,68
Standard deviation of repeatability (s_r)	0,037	0,023
Coefficient of variation of repeatability	1,1 %	0,5 %
Repeatability ($2,83 \times s_r$)	0,10	0,07
Standard deviation of reproducibility (s_R)	0,15	0,16
Coefficient of variation of reproducibility	4,4 %	3,4 %
Reproducibility ($2,83 \times s_R$)	0,42	0,45

10 Test report

The test report shall show the method used and the 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 details required for the complete identification of the sample.

1) ISO 5725, Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.