INTERNATIONAL STANDARD

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Cereals and milled cereal products — Determination of total ash

Céréales et produits de mouture des céréales — Dosage des cendres totales

Reference number
ISO 2171:1993(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 2171 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 4, *Cereals and pulses*.

This third edition cancels and replaces the second edition (ISO 2171:1980), which has been technically revised in that method B has been deleted and annexes A and B have been added.

This International Standard is based on Standard ICC 104/1 of the International Association for Cereal Science and Technology.

Annexes A and B of this International Standard are for information only.

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Cereals and milled cereal products — Determination of total ash

1 Scope

This International Standard specifies a method for the determination of ash yielded by cereals and milled cereal products intended for human consumption.

It is not applicable to starch and starch derivatives, or to cereals and derived cereal products intended for animal feeding stuffs (for which methods are described in ISO 3593 and ISO 5984 respectively), nor is it applicable to seeds of cereals and pulses for sowing.

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 712:1985, *Cereals and cereal products — Determination of moisture content (Routine reference method).*

3 Definition

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

3.1 ash: Incombustible residue obtained after incineration according to the method given in this International Standard.

4 Principle

Incineration of a test portion in an oxidizing atmosphere at a temperature of 900 °C ± 10 °C until combustion of organic matter is complete, then weighing the residue obtained.

5 Reagent

5.1 Ethanol, 95 % (*V/V*) min.

6 Apparatus

6.1 Grinding mill, having the following characteristics:

a) easy to clean and having as little dead space as possible;

b) allowing grinding to be carried out rapidly and uniformly without appreciable development of heat and, as far as possible, restricting contact with the outside air;

c) capable of providing ground material which meets the following particle size requirements:

> 1,7 mm	0 %
> 1,0 mm	< 10 %
> 0,5 mm	< 50 %

6.2 Ashing dish, preferably of platinum or any other material which is unaffected under the experimental conditions; of at least 20 ml capacity, flat-bottomed, and with at least 15 cm^2 of surface area (for instance, diameter 40 mm to 60 mm and maximum height 30 mm).

6.3 Electrically heated muffle furnace, with adequate ventilation, provided with temperature control and a refractory coating which is not liable to lose particles at the ashing temperature, and capable of being maintained at a temperature of 900 °C ± 10 °C.

6.4 Desiccator, (internal diameter about 18 cm) with tube, with perforated porcelain or aluminium plate, and with calcium chloride, phosphorus pentoxide or blue silica gel as drying agent.

6.5 Analytical balance, with an accuracy of 0,1 mg.

6.6 Heat-resistant plate

7 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.

Sampling is not part of the method specified in this International Standard. Recommended sampling methods are given in ISO 950 and ISO 2170.

8 Preparation of test sample

8.1 Whole cereals or coarsely ground products

Run the grinding mill (6.1) with a few grams of the product and throw away the grindings.

Then grind about 25 g of the product. In the case of whole cereals, it is recommended to take all the grindings of the test sample to avoid separation of the different components of the kernel.

8.2 Finely ground products

Ground products having particles of sizes less than 1,7 mm [less than 10 % (*m/m*) being larger than 1 mm and more than 50 % (*m/m*) being smaller than 0,5 mm] do not require further grinding.

9 Procedure

9.1 Preparation of ashing dishes

Clean the ashing dishes (6.2), whether new or previously used, for instance with boiling dilute hydrochloric acid, then rinse with plenty of tap water and subsequently with distilled water.

Immediately before use, heat the ashing dishes in the muffle furnace (6.3) at 900 °C until they are of constant weight; normally 15 min is sufficient. Cool to room temperature in the desiccator (6.4) for at least 1 h and weigh to a precision of 0,1 mg.

9.2 Determination of moisture content

Determination of the moisture content of the test sample shall be carried out immediately and in accordance with ISO 712.

9.3 Preparation of test portion

From the test sample, prepared according to clause 8, take the following:

a) (5 g to 6 g) ± 0,01 g for flour whose ash yield is expected to be less than 1 % on a dry basis;

b) (2 g to 3 g) ± 0,01 g for those products whose ash yield is expected to be greater than 1 % on a dry basis.

Place the test portion [a) or b) according to the ash yield expected] in an ashing dish, prepared and weighed as described in 9.1. Spread the material, without pressing it, to form a uniform layer and weigh rapidly to the nearest 0,001 g.

9.4 Ashing

To promote uniform ashing, moisten the test portion in the ashing dish, immediately before ashing, with 1 ml or 2 ml of the ethanol (5.1).

Place the ashing dish at the entrance of the furnace with the door open and, after the sample has burnt off, push the ashing dish further into the furnace at a temperature of 900 °C. With the door of the furnace closed, sufficient ventilation has to be provided but the draught shall not be so strong that it removes substance from the ashing dish.

Carry out ashing until the whole substance, including the carbon particles in the residue, is ashed completely.

Ashing is completed when the cooled residue is white or nearly white, which normally takes about 2 h.

9.5 Weighing the residue

When ashing is completed and a constant colour is obtained, remove the dish from the furnace and allow it to cool on the heat-resistant plate (6.6) for 1 min, then transfer it to the desiccator (6.4) (see note 1). Because of the hygroscopic character of the ash, as soon as it has reached ambient temperature, rapidly weigh it to the nearest 0,1 mg.

The mass of the clean empty dish (9.1) shall be subtracted from the total mass to obtain the mass of the residue.

NOTE 1 In the case of testing in series, not more than four dishes at a time should be put in the desiccator (6.4).

10 Expression of results

The ash yield, expressed as a percentage by mass on a dry basis, is equal to

$$m_1 \times \frac{100}{m_0} \times \frac{100}{100 - H}$$

where

m_0 is the mass, in grams, of the test portion (9.3);

m_1 is the mass, in grams, of the residue (9.5);

H is the moisture content, expressed as a percentage by mass, of the test sample (see 9.2).

Express the result to the nearest 0,01 % (*m/m*).

11 Precision

11.1 Repeatability

The absolute difference between two independent single 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

a) 0,02 g if the ash yield is less than 1 % (*m/m*) or

b) 2 % of the arithmetic mean of the two results, if the ash yield is 1 % (*m/m*) or more.

Reject both results if the difference exceeds the indicated repeatability value and carry out two new single determinations.

11.2 Reproducibility

The results of an interlaboratory test are given in annex A.

12 Test report

The test report shall specify

— the method in accordance with which sampling was carried out (if known),

— the method used,

— the result(s) obtained, and

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

It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, may have influenced the result(s).

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

Annex A
(informative)

Results of an interlaboratory test

An interlaboratory test carried out by the International Association for Cereal Science and Technology (ICC) in 1989, in which sixteen laboratories participated, each of which carried out five determinations on each sample, gave the statistical results (evaluated in accordance with ISO 5725) shown in table A.1.

Table A.1 — Determination of ash

Sample	Whole wheat meal	Wheat flour I	Wheat flour II	Wheat/barley flour	Oatbran
Number of laboratories retained after eliminating outliers	14	13	7	12	12
Mean ash yield, % (*m/m*), dry basis	1,817	0,725	0,516	0,946	2,530
Standard deviation of repeatability, s_r, % (*m/m*), dry basis	0,0134	0,0104	0,0017	0,0071	0,0150
Coefficient of variation of repeatability, %	0,740	1,44	0,336	0,749	0,588
Repeatability, $2{,}83s_r$, % (*m/m*), dry basis	0,0376	0,0292	0,0049	0,0198	0,0420
Standard deviation of reproducibility, s_R, % (*m/m*), dry basis	0,0283	0,0142	0,0118	0,0185	0,0210
Coefficient of variation of reproducibility, %	1,56	1,95	2,28	1,95	0,837
Reproducibility, $2{,}83s_R$, % (*m/m*), dry basis	0,0790	0,0397	0,0330	0,0517	0,0590