INTERNATIONAL STANDARD

ISO 2171

Fourth edition
2007-11-01

Cereals, pulses and by-products — Determination of ash yield by incineration

Céréales, légumineuses et produits dérivés — Dosage du taux de cendres par incinération

Reference number
ISO 2171:2007(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

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 2171 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This fourth edition cancels and replaces the third edition (ISO 2171:1993), which has been technically revised.

Cereals, pulses and by-products — Determination of ash yield by incineration

1 Scope

This International Standard specifies a method for determining the ash yielded by cereals, pulses and their milled products intended for human consumption. The source materials covered are:

a) grains of cereals;

b) flours and semolinas;

c) milled products (bran and high bran content products, sharps);

d) mixed cereal flours (mixes);

e) cereal by-products other than milled products; and

f) pulses and their by-products.

This International Standard is not applicable to starches and starch derivatives (see ISO 3593), to products intended for animal feeding stuffs (see ISO 5984), or to seeds.

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 712, *Cereals and cereal products — Determination of moisture content — Routine reference method*

ISO 6540, *Maize — Determination of moisture content (on milled grains and on whole grains)*

ISO 24557:—[1)], *Pulses — Determination of moisture content — Air-oven method*

3 Terms and definitions

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

3.1
ash
incombustible residue obtained after incineration according to the method given in this International Standard

1) To be published.

4 Principle

A test portion is incinerated until combustion of organic matter is complete, then the residue obtained is weighed. The residue obtained is flaky after incineration at 550 °C and vitrified after incineration at 900 °C.

In general, products containing salts (e.g. sodium chloride, pyrophosphate) shall be incinerated at (550 ± 10) °C.

Table 1 summarizes incineration temperatures according to product type.

Table 1 — Incineration temperatures and product type

Product type	Incineration temperature	
Flours	(550 ± 10) °C	(900 ± 25) °C
Semolinas	(550 ± 10) °C	(900 ± 25) °C
Cereal grains	(550 ± 10) °C	(900 ± 25) °C
Other milled products (e.g. bran, high bran content products, sharps)	(550 ± 10) °C	—
Mixed cereal products (mixes)	(550 ± 10) °C	—
Cereal by-products other than milled products	(550 ± 10) °C	—
Pulses and their by-products	(550 ± 10) °C	—

5 Reagents

Unless stated otherwise, use only reagents of recognized analytical quality and distilled or demineralized water or water of equivalent purity.

5.1 Hydrochloric acid, aqueous solution of one part by volume of HCl (35 % volume fraction) and one part by volume of water.

5.2 Purified diphosphorus pentoxide (P_4O_{10}).

5.3 Ethanol.

6 Apparatus

6.1 Grinding mill, easy to clean and having as little dead space as possible, and ensuring rapid, uniform grinding.

6.2 Ashing dish, of capacity not less than 20 ml, rectangular or round shape, flat-bottomed and having a surface area of not less than 12 cm^2. Suitable materials for the ashing dish which do not deteriorate under test conditions at the temperature of operation are:

a) at 900 °C — platinum or rhodium;

b) at 550 °C — quartz or silica.

In both cases, the material used shall allow compliance with the precision values.

The dishes shall be cleaned by complete immersion for at least 1 h in hydrochloric acid (5.1), then rinsed with running water and then with distilled water.

After rinsing, the dishes shall be dried in an oven (6.7) at a temperature and for a period sufficient to eliminate water.

6.3 Electrically heated muffle furnace, with adequate ventilation, provided with temperature control system and a refractory coating which is not liable to lose particles at the ashing temperature, and capable of being maintained at (900 ± 25) °C or at (550 ± 10) °C.

6.4 Vacuum desiccator, equipped with a perforated aluminium or porcelain plate, and diphosphorus pentoxide (5.2) as drying agent.

6.5 Analytical balance, with an accuracy of 0,01 mg.

6.6 Riffle splitter or **cone-shaped divider**.

6.7 Oven for drying the ashing dishes.

7 Sampling

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. Recommended sampling methods are given in ISO 6644 and ISO 13690.

8 Preparation of the test sample

For grains or products containing whole grains, mix and divide the sample in order to obtain a representative quantity compatible with the type of grinding mill (6.1) being used.

Grind the sample thus obtained.

The other products do not require grinding.

9 Procedure

9.1 Determination of the moisture content

Determine beforehand the moisture content of the test sample in accordance with ISO 712 for cereals other than maize or ISO 6540 in the case of maize or ISO 24557 in the case of pulses.

Pulses and their derived products should be processed according to ISO 712 with a 90 min drying time and preconditioning if moisture mass fraction is below 7 % or above 13 %.

9.2 Preparation of the ashing dishes

For ashing dishes suitable for use at 900 °C (6.2), bring the previously cleaned dishes up to the incineration temperature being employed by putting them in the muffle furnace (6.3) for 5 min, leave them to cool in the desiccator (6.4), then weigh (6.5) them to within 0,1 mg.

For ashing dishes suitable for use at 550 °C, place the cleaned dishes in an oven (6.7) for the time required for drying (e.g. 90 min at 130 °C). Immediately before use, remove the dishes from the oven and leave them to cool in a desiccator (6.4), then weigh (6.5) them to within 0,1 mg.

9.3 Preparation of the test portion

From the test sample prepared according to Clause 8 and carefully mixed, rapidly weigh (6.5) to within 0,1 mg a test portion between 3,9 g and 4,1 g in the case of incineration at 900 °C and between 4,9 g and 5,1 g in the case of incineration at 550 °C.

In the case of low density products, the test portion can be between (2 ± 0,1) g and (3 ± 0,1) g.

In the ashing dish, prepared and weighed as described in 9.2, spread out the product, without packing it, to form a uniform layer.

9.4 Pre-ashing

Place the ashing dish and its contents at the entrance of the furnace brought up to the ashing temperature.

At 900 °C, the products burst into flame spontaneously. At 550 °C, it is necessary to ignite them with ethanol (5.3).

For pre-ashing at 550 °C, it is permissible to put the dishes in the cold furnace and to let the temperature of the furnace rise to the target temperature.

9.5 Ashing

Wait until the product has finished burning, then place the dish inside the furnace.

Close the furnace door.

Continue the ashing until combustion of the entire product, including the carbon particles contained in the residue, is complete, namely 1 h minimum at 900 °C, and 4 h minimum at 550 °C.

Once the ashing is completed, remove the dish from the furnace and place it in the desiccator (6.4) to cool. In order to maintain the efficiency of the desiccator, do not stack dishes.

Due to the hygroscopic nature of the ash, as soon as the dish has reached ambient temperature (namely 15 min to 20 min for metallic dishes and 60 min to 90 min minimum for quartz or silica dishes), weigh rapidly to within 0,1 mg.

For test portions incinerated at 550 °C, special precautions shall be taken to avoid flaky residues being swept away with the influx of air on opening the desiccator.

The validity of the results obtained on this sample shall be checked with respect to the laboratory's self-inspection criteria (e.g. control chart).

9.6 Number of determinations

Conduct at least two determinations on the same test sample.

10 Expression of results

The ash yield, as a mass fraction on the dry matter basis expressed as a percentage, $w_{a,d}$, is given by Equation (1):

$$w_{a,d} = (m_2 - m_1) \times \frac{100}{m_0} \times \frac{100}{100 - w_m} \quad (1)$$

where

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

m_1 is the mass, in grams, of the ashing dish (9.2);

m_2 is the mass, in grams, of the ashing dish (9.2) and the incinerated residue (9.5);

w_m is the moisture content, as a percentage by mass, of the sample (see 9.1).

Take as a result the arithmetic mean of the two determinations if the repeatability conditions (see 11.1) are fulfilled.

Express the result to the nearest 0,01 % by mass.

If needed, the ash yield, as mass fraction on the wet matter basis expressed as a percentage, $w_{a,w}$, is given by Equation (2):

$$w_{a,w} = (m_2 - m_1) \times \frac{100}{m_0} \qquad (2)$$

11 Precision

11.1 Interlaboratory tests

The values for repeatability and reproducibility limits and for critical differences were derived from results of interlaboratory tests carried out in accordance with ISO 5725-1, ISO 5725-2, and ISO 5725-6. Details are summarized in Annex A.

The values deriving from these interlaboratory tests may not be applicable to content ranges and matrices other than those given.

11.2 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, will in not more than 5 % of cases be greater than:

$$r = 2{,}8 \times s_r \qquad (3)$$

$$r = 2{,}8 \times 0{,}009 = 0{,}025 \quad \text{for} \quad 0{,}49\ \% < w_{a,d} \leqslant 1{,}00\ \% \qquad (4)$$

$$r = 2{,}8 \times 0{,}012 = 0{,}034 \quad \text{for} \quad 1{,}00\ \% < w_{a,d} \leqslant 2{,}53\ \% \qquad (5)$$

where s_r is the standard deviation of repeatability.

11.3 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than:

$$R = 2{,}8 \times s_R \qquad (6)$$

$$R = 2{,}8 \times 0{,}023 = 0{,}064 \quad \text{for} \quad 0{,}49\ \% < w_{a,d} \leqslant 1{,}00\ \% \qquad (7)$$