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# International Standard



# 2171

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## Cereals, pulses and derived products — Determination of ash

*Céréales, légumineuses et produits dérivés — Détermination des cendres*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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 2171 was developed by Technical Committee ISO/TC 34, *Agricultural food products*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 2171:1972), which had been approved by the member bodies of the following countries :

Australia	Iran	Romania
Austria	Ireland	South Africa, Rep. of
Chile	Israel	Spain
Czechoslovakia	Korea, Dem. P. Rep. of	Sri Lanka
Egypt, Arab Rep. of	Korea, Rep. of	Turkey
Germany, F. R.	Netherlands	United Kingdom
Hungary	New Zealand	USSR
India	Poland	

No member body had expressed disapproval of the document.

NOTE — Method A (incineration at 900 °C) in this International Standard is based on Standard No. 104 of the International Association of Cereal Chemistry (ICC).

# Cereals, pulses and derived products — Determination of ash

## 0 Introduction

Taking into consideration the latest investigations, two methods have been standardized for the determination of ash from cereals, pulses and their derived products. The principal differences between the methods are the temperatures of incineration ( $900 \pm 25$  °C or  $550 \pm 10$  °C), and, consequently, their fields of application.

The two methods have the same reproducibility but they do not always give the same results. It should be stressed that in milling and grinding technology, as well as in commercial transactions, incineration at  $900 \pm 25$  °C is usually preferable because of its greater speed. The method of incineration at  $550 \pm 10$  °C is slower but may be used more generally.

The description of two different methods in the same International Standard has been necessary because laboratories and analysts carrying out these tests need to have standard methods suitable for the determination of ash from cereals and their derived products and also from pulses and their derived products.

## 1 Scope and field of application

This International Standard specifies two methods for the determination of ash yielded by cereals, pulses and derived products :

- a) method A, by incineration at  $900$  °C, applicable to cereals and milled cereal products intended for human consumption;
- b) method B, by incineration at  $550$  °C, applicable to derived cereal products other than milled products intended for human consumption, to flours of composite cereals used for the manufacture of dietetic products, and to pulses and derived products.

Method B should not be used for cereals and their derived products intended for human consumption, method A being more rapid and generally used for the purpose of commercial contracts.

This International Standard 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 References

- ISO 712, *Cereals and cereal products — Determination of moisture content (Routine reference method)*.
- ISO 950, *Cereals — Sampling (as grain)*.
- ISO 951, *Pulses in bags — Sampling*.
- ISO 2170, *Cereals and pulses — Sampling of milled products*.
- ISO 3593, *Starch — Determination of ash*.
- ISO 5984, *Animal feeding stuffs — Determination of crude ash*.

## 3 Definition

**ash**: According to the method used :

- a) the residue obtained after incineration at  $900 \pm 25$  °C under the conditions described (method A);
- b) the residue obtained after incineration at  $550 \pm 10$  °C under the conditions described (method B).

## 4 Principle

### 4.1 Method A

Incineration of a test portion in an oxidizing atmosphere at a temperature of  $900 \pm 25$  °C until combustion of organic matter is complete, and weighing the residue obtained.

### 4.2 Method B

Incineration of a test portion in an oxidizing atmosphere at a temperature of  $550 \pm 10$  °C until combustion of organic matter is complete and constant mass is obtained.

## 5 Reagent

**5.1 Ethanol**, not less than 95 % (V/V) solution, or **vegetable oil** which leaves no residue on incineration.

## 6 Apparatus

**6.1 Grinding mill** (for whole grain or seeds or coarsely ground products, if necessary), 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.

**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<sup>2</sup> of surface (for instance, diameter 50 to 55 mm and height 15 to 20 mm).

Clean the ashing dish, 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 dish in the furnace (6.4), at the temperature corresponding to the incineration temperature, allow to cool to room temperature in the cooling device (6.5) and weigh to the nearest 0.000 1 g.

**6.3 Electric heater or gas burner.**

**6.4 Electric furnace**, with adequate air circulation, provided with temperature control and a refractory coating which is not liable to lose particles at the ashing temperature, and capable of being controlled at the temperature appropriate to the method used.

**6.5 Cooling device**, preventing moisture up-take, for example a desiccator provided with an efficient desiccant.

**6.6 Glass rods**, with one end flattened.

**6.7 Analytical balance.**

**6.8 Heat-resistant smooth plate.**

## 7 Sampling

Prepare a laboratory sample in accordance with ISO 950, ISO 951 or ISO 2170, as appropriate.

## 8 Procedure

### 8.1 Preparation of test sample

#### 8.1.1 Whole cereals or pulses or coarsely ground products

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

Then grind 2 to 6 g of the product, according to the yield of ash expected. In the case of whole cereals and pulses it is recommended to take all the grindings as the test portion to avoid separation of the different components of the kernel.

#### 8.1.2 Finely ground products

Ground products having particles of sizes less than or equal to 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.

### 8.2 Test portion

Place approximately 2 to 6 g of the test sample (8.1.1 or 8.1.2), according to the yield of ash expected, into the ashing dish prepared and weighed as described in 6.2. Spread the material, without pressing it, to form a uniform layer and weigh rapidly to the nearest 0.001 g.

Do not exceed 0,3 g of product per square centimetre of the surface of the dish.

### 8.3 Determination of moisture content

Immediately carry out the determination of moisture content, in accordance with ISO 712.

### 8.4 Preparation for ashing

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

### 8.5 Method A

#### 8.5.1 Pre-ashing

Open the door of the furnace (6.4) and place the ashing dish, with its contents, in the opening of the furnace, previously heated to  $900 \pm 25$  °C, until the material ignites. Take care to avoid too rapid combustion, so that solid particles of the material are not ejected.

#### 8.5.2 Ashing

As soon as the flames are extinguished, place the dish carefully into the furnace (6.4) controlled at  $900 \pm 25$  °C. Continue incineration until combustion of the material is complete, including any carbonaceous particles which may be contained in the residue.

NOTE – At this temperature, the incineration of a sample yielding less than 1 % (*m/m*) of ash should be completed within 2 h.

#### 8.5.3 Weighing the residue

When ashing is completed and a constant colour is obtained, remove the dish from the furnace and allow it to cool, for example on the heat-resistant plate (6.8), for 1 min, then transfer it to the cooling device (6.5) (see the note); as soon as it has

reached ambient temperature, rapidly weigh, because of the hygroscopic character of the ash, to the nearest 0,000 1 g.

NOTE – In the case of testing in series, do not put more than four dishes at a time in the cooling device (6.5).

## 8.6 Method B

### 8.6.1 Pre-ashing

Place the dish with its contents on the electric heater or the gas burner (6.3). Heat it carefully until the material ignites. Take care to avoid too rapid combustion, so that solid particles of the material are not ejected.

### 8.6.2 Ashing

As soon as the flames are extinguished, place the dish carefully into the furnace (6.4) previously heated to  $550 \pm 10$  °C. Continue incineration until combustion of the material is complete, including any carbonaceous particles which may be contained in the residue.

NOTE – In order to accelerate incineration, remove the ashing dish from the furnace, place it on the heat-resistant plate (6.8), moisten the contents of the dish with a few drops of distilled water and rub the carbonaceous particles with a glass rod (6.6).

Rinse the rod with distilled water, allowing the rinsings to fall into the dish, and let the water evaporate. Replace the dish in the furnace and heat it gradually, with care.

### 8.6.3 Weighing the residue

When ashing is completed, remove the dish from the furnace, and allow it to cool, for example on the heat-resistant plate (6.8), for 1 min, then transfer it to the cooling device (6.5) (see the note); as soon as it has reached ambient temperature, rapidly weigh, because of the hygroscopic character of the ash, to the nearest 0,000 1 g.

NOTE – In the case of testing in series, do not put more than four dishes at a time in the cooling device (6.5).

Repeat heating, cooling and weighing until constant mass is obtained, i.e. until the difference between two consecutive weighings, separated by 1 h of additional heating, does not exceed 0,000 2 g.

## 8.7 Number of determinations

Carry out at least two determinations on the same test sample.

## 9 Expression of results

### 9.1 Method of calculation and formula

The ash yield, expressed as a percentage by mass on the 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 (8.2);

$m_1$  is the mass, in grams, of the residue (8.5.3 or 8.6.3);

$H$  is the moisture content, expressed as a percentage by mass, of the test sample (see 8.3).

Take as the result the arithmetic mean of the two determinations, provided that the requirements for repeatability (see 9.2) are satisfied.

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

### 9.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst should not exceed

0,02 (absolute value) for ash yields of less than 1 % ( $m/m$ );

2 % of the mean for ash yields greater than 1 % ( $m/m$ ).

If the difference exceeds these limits, the determination shall be repeated in duplicate.

## 10 Test report

The test report shall indicate the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result. In particular the following shall be indicated :

- the ashing temperature used;
- any use of ethanol or vegetable oil;
- whether the determination had to be repeated.

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

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