



**SLOVENSKI STANDARD**  
**oSIST prEN ISO 2171:2020**  
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**Žita, stročnice in stranski proizvodi - Določanje izkoristka pepela po sežiganju  
(ISO/DIS 2171:2020)**

Cereals, pulses and by-products - Determination of ash yield by incineration (ISO/DIS 2171:2020)

Getreide, Hülsenfrüchte und Nebenprodukte - Bestimmung des Aschegehalts durch Verbrennung (ISO/DIS 2171:2020)

Céréales, légumineuses et produits dérivés - Dosage du taux de cendres par incinération (ISO/DIS 2171:2020)

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**ICS:**

67.060	Žita, stročnice in proizvodi iz njih	Cereals, pulses and derived products
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## Cereals, pulses and by-products — Determination of ash yield by incineration

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

ICS: 67.060

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## ISO/DIS 2171:2020(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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

The committee responsible for this document is ISO/ *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This fifth edition cancels and replaces the fourth edition (ISO 2171:2007), which has been technically revised. The main changes in the ISO 2171, determination of ash yield by incineration are, the use of platinum dishes and a temperature of incineration of 900 °C for the flour analysis instead of a choice between 900 °C and 550 °C (cf. [tableau 1](#)) and all the normative references were updated.

# 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 and products covered are:

- a) grains of cereals;
- b) flours and semolinas;
- c) milled products (bran and high bran content products, shorts);
- d) mixed cereal flours (mixes);
- e) cereal by-products other than milled products;
- 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 cited edition 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 — Reference method*

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

ISO 24557, *Pulses — Determination of moisture content — Air-oven method*

ISO 12099, *Animal feeding stuffs, cereals and milled cereal products — Guidelines for the application of near infrared spectrometry*

## 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

## 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  $(550 \text{ °C} \pm 10 \text{ °C})$  and vitrified after incineration at  $(900 \text{ °C} \pm 25 \text{ °C})$ .

In general, products containing salts (e.g. sodium chloride, pyrophosphate) shall be incinerated at  $(550 \pm 10) \text{ °C}$ .

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### 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 or ready-to-use 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<sup>2</sup>. Suitable materials for the ashing dish which do not deteriorate under test conditions at the temperature of operation are:

- a) at 900 °C — platinum 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 10 h in aqueous hydrochloric acid (5.1), then rinsed with running water and then with distilled water.

After rinsing, the quartz or silica 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**, provided with a refractory coating, with a control system, capable of reaching and maintaining the temperatures of  $(900 \pm 25)$  °C or at  $(550 \pm 10)$  °C.

It is recommended to check the thermal geography of the oven by filling it with capsules containing a control sample in order to determine its degree of possible filling.

When the combustion is done inside the furnace, case of the ashing at 550°C by rise of the temperature, it is recommended to carry out a sweeping of the chimney of the oven, which clogs more quickly.

**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, capable of being maintained at a temperature  $\geq 100^\circ\text{C}$

### 7 Sampling

Sampling is not part of the method specified in this International Standard.



However, it is important that the laboratory receive a truly representative sample, in accordance with the standard ISO 24333.

## 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

**Table 1 — Incineration temperatures and product type**

Product type	Ashing temperature	Ashing time	Type of dish	Test portion
Flour	(900 ± 25) °C	1:00	Platinum rhodium	Between 3.9 and 4.1 g
Semolina / milled cereal grains and milled products	(900 ± 25) °C	1:00 to 1:30	Platinum rhodium	Between 3.9 and 4.1 g
	Or (550 ± 10) °C	4:00 minimum	Quartz or silica	Between 4.9 and 5.1 g
Mixed cereal products (mixes)	(550 ± 10) °C	4:00 minimum	Quartz or silica	Between 4.9 and 5.1 g
Cereal by-products other than milled products	(550 ± 10) °C	4:00 minimum	Quartz or silica	Between 4.9 and 5.1 g
Pulses and their by-products	(550 ± 10) °C	4:00 minimum	Quartz or silica	Between 4.9 and 5.1 g

### 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.

The moisture content can also be determined with an apparatus using near-infrared spectrometry, the performance of which has been demonstrated in accordance with ISO 12099 and reaching at least a standard prediction error (SEP) of ≤ 0.15 mass fraction determined on the whole scope of this standard.

### 9.2 Preparation of the ashing dishes

For ashing dishes suitable for use at 900 °C (6.2a), 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 (6.2b), 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.

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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

At 900 °C, place the ashing dish and its contents at the entrance of the furnace brought up to the ashing temperature. The products burst into flame spontaneously. If several dishes are placed at the entrance of the oven, contact between them will be avoided.

At 550 °C, it is necessary to ignite them with ethanol (5.3). But it is better to introduce the dishes in a cold oven and then let rise the temperature.

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 between 1 h to 1 h30 at 900 °C without exceeding 1 h for flour , and 4 h minimum at 550 °C.

NOTE in the case of an ashing at 550 °C, it is possible to melt the flaky residues by raising the temperature of the oven to 900 °C for 1h. Under these conditions, one shall let the oven cool down before removing the dishes (6.2b), considering their fragility.

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 platine 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).

NOTE: if carbon particules are still present after a one-hour ashing in the oven at 900 °C, the analysis should be done again.

### 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