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

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

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Foreword

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products,* Subcommittee SC 4, *Cereals and pulses,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, *Cereal and cereal products,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 2171:2007), which has been technically revised. The main changes are as follows:

— the Scope has been updated;

— silica gel has been added as a desiccant (5.2);

— the use of platinum dishes and a temperature of incineration of 900 °C for the flour analysis has replaced a choice of a temperature between 900 °C and 550 °C (see Table 1);

— the interlaboratory critical difference ($C_{D,R}$) has been updated in 11.4.2.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

1 Scope

This document specifies a method for determining the ash yield 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) other milling products (bran and high bran content products, shorts);

d) mixed cereal flours (mixes);

e) cereal by-products other than c) (such as wheat gluten, maize grits, broken rice kernels);

f) pulses and their by-products (flour).

This document does not apply 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 — 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*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
ash
incombustible residue obtained after incineration

Note 1 to entry: This is according to the method given in this document.

3.2
critical difference
difference between two averaged values obtained from two test results under repeatability conditions

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 ± 10) °C and vitrified after incineration at (900 ± 25) °C.

In general, products containing salts (e.g. sodium chloride, pyrophosphate) shall be incinerated at (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 or ready-to-use solution of one part by volume of HCl (35 % mass fraction) and one part by volume of water.

5.2 Desiccant, purified diphosphorus pentoxide (P_4O_{10}) or **silica gel**.

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 rhodium is recommended or, if unavailable, quartz can be used;

b) at 550 °C: quartz or silica.

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

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

As quartz ashing dishes produce relatively lower ash yield and poorer precision compared with those obtained with platinum dishes[8], the use of platinum-rhodium ashing dishes is recommended when incineration is carried out at 900 °C. The precision data for 900 °C in Clause 11 was derived from the results of interlaboratory tests carried out with platinum-rhodium ashing dishes. The users of quartz ashing dishes for incineration at 900 °C are advised to check the precision data. In addition, the crucible material is more prone to failure and it is possible the analysis will need to be repeated due to crucible breakage.

6.3 Electrically heated muffle furnace, provided with a refractory coating, with a control system, capable of reaching and maintaining the temperatures of (900 ± 25) °C or (550 ± 10) °C.

It is recommended to check the thermal geography of the furnace 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, in the case of the ashing at 550 °C, it is recommended to carry out a sweeping of the chimney of the oven, as this can clog quickly.

6.4 Vacuum desiccator, equipped with a perforated aluminium or porcelain plate, and desiccant (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 ≥ 100 °C

7 Sampling

Sampling is not part of the method specified in this document.

The laboratory should receive a truly representative sample in accordance with 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

9.1 Incineration temperatures

Table 1 summarizes incineration temperatures according to product type.

Table 1 — Incineration temperatures and product type

Product type	Ashing temperature	Ashing time	Type of dish	Test portion
Flour	(900 ± 25) °C	1 h	Platinum rhodium or, if unavailable, quartz	3,9 g to 4,1 g
	(550 ± 10) °C	4 h minimum	Quartz or silicia	4,9 to 5,1 g
Semolina/cereal grains and other milling products	(900 ± 25) °C	1 h to 1,5 h	Platinum rhodium or, if unavailable, quartz	3,9 g to 4,1 g
	(550 ± 10) °C	4 h minimum	Quartz or silicia	4,9 g to 5,1 g
Mixed cereal products (mixes)	(550 ± 10) °C	4 h minimum	Quartz or silicia	4,9 g to 5,1 g
Cereal by-products other than milled products	(550 ± 10) °C	4 h minimum	Quartz or silicia	4,9 g to 5,1 g
Pulses and their by-products	(550 ± 10) °C	4 h minimum	Quartz or silicia	4,9 g to 5,1 g

9.2 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 which reaches at

least a standard prediction error (SEP) of ≤ 0,15 mass fraction determined on the whole scope of this document.

9.3 Preparation of the ashing dishes

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

9.4 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 9.3, spread out the product, without packing it, to form a uniform layer.

9.5 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 will burst into flame spontaneously. If several dishes are placed at the entrance of the furnace, contact between them should be avoided.

At 550 °C, it is necessary to ignite the products with ethanol (5.3). It is recommended to put the dishes in the cold furnace and to let the temperature of the furnace rise to the target temperature.

9.6 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 1,5 h at 900 °C (without exceeding 1 h for flour) and 4 h minimum at 550 °C.

In the case of ashing at 550 °C, the flaky residues can be vitrified by raising the temperature of the furnace to 900 °C for 1 h. Under these conditions, the furnace shall be allowed to cool down before removing the dishes [6.2 b)], 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).

If carbon particles are still present after ashing in the furnace for 1 h at 900 °C, the analysis should be done again.

9.7 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 Formula (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 (9.4);

m_1 is the mass, in grams, of the ashing dish (9.3);

m_2 is the mass, in grams, of the ashing dish (9.3) and the incinerated residue (9.6);

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

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

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

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, *r*

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 as shown by Formula (3):

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

$r = 2{,}8 \times 0{,}009 = 0{,}025$ for $0{,}49\ \% < w_{a,d} \leq 1{,}00\ \%$

r = 2,8 × 0,012 = 0,034 for 1,00 % < $w_{a,d}$ ≤ 2,53 %

where s_r is the standard deviation of repeatability.

11.3 Reproducibility, *R*

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. Practically, the comparison between individual results is only realistic for single-determination tests and not for tests conducted under repeatability conditions as specified in this document.

The possible comparison is the one of the averages, as defined in 11.4.

NOTE The reproducibility standard deviations (s_R = 0,023 for an ash yield between 0,49 % and 1,00 %, and 0,027 for an ash yield between 1,00 % and 2,53 %) estimated by the interlaboratory tests are elements for the assessment of the uncertainty.

11.4 Critical difference

11.4.1 Comparison of two groups of measurements in one laboratory

The difference between two averaged values obtained from two test results in the same laboratory under repeatability conditions is the intralaboratory critical difference, $C_{D,r}$, as shown by Formula (4):

$$C_{D,r} = 2{,}8 s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}8 s_r \sqrt{\frac{1}{2}} = 1{,}98 s_r \quad (4)$$

$C_{D,r}$ = 0,018 for 0,49 % < $w_{a,d}$ ≤ 1,00 %

$C_{D,r}$ = 0,024 for 1,00 % < $w_{a,d}$ ≤2,53 %

where n_1 and n_2 are the number of test results corresponding to each of the averaged values; here $n_1 = n_2 = 2$.

11.4.2 Comparison of two groups of measurements in two laboratories

The difference between two averaged values obtained in two different laboratories from two test results under repeatability conditions is the interlaboratory critical difference, $C_{D,R}$, as shown by Formula (5):

$$C_{D,R} = 2{,}8 \sqrt{s_R^2 - s_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}8 \sqrt{s_R^2 - 0{,}5 s_r^2} \quad (5)$$

$C_{D,R}$ = 0,062 for 0,49 % < $w_{a,d}$ ≤ 1,00 %

$C_{D,R}$ = 0,072 for 1,00 % < $w_{a,d}$ ≤ 2,53 %

An application table is given in Annex B.

11.5 Uncertainty, *U*

Uncertainty can be estimated from data obtained from studies conducted in accordance with ISO 5725-2. The reproducibility standard deviation obtained in the collaborative study is a valid basis for assessing measurement uncertainty since, by definition, uncertainty characterizes the dispersion of