
Žito - Določanje nečistoč v koruzi (*Zea mays*, L.) in prosu (*Sorghum bicolor*, L.)

Cereals - Determination of impurities content in maize (*Zea mays*, L.) and sorghum (*Sorghum bicolor*, L.)

Getreide - Bestimmung von Besatz in Mais (*Zea mays*, L.) und Hirse (*Sorghum bicolor*, L.)

Céréales - Détermination de la teneur en impuretés dans le maïs (*Zea mays*, L.) et le sorgho (*Sorghum bicolor*, L.)

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Cereals - Determination of impurities content in maize (*Zea mays*, L.) and sorghum (*Sorghum bicolor*, L.)

Céréales - Détermination de la teneur en impuretés dans le maïs (*Zea mays*, L.) et le sorgho (*Sorghum bicolor*, L.)

Getreide - Bestimmung von Besatz in Mais (*Zea mays*, L.) und Hirse (*Sorghum bicolor*, L.)

This European Standard was approved by CEN on 19 July 2013.

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Foreword

This document (EN 16378:2013) has been prepared by Technical Committee CEN/TC 338 "Cereal and cereal products", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2014, and conflicting national standards shall be withdrawn at the latest by February 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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EN 16378:2013 (E)**1 Scope**

This European Standard specifies the term *Besatz* (impurities) and the method for the determination of its components. The term *Besatz* is used as a parameter for certain quality aspects in maize (*Zea mays* L.) and sorghum (*Sorghum bicolor* L.).

This method has been validated in an interlaboratory study via the analysis of samples containing natural amount of impurities, ranging from:

- 0,0 % to 2,7 % for broken grains;
- 0,2 % to 3,5 % for grains impurities;
- 0,5 % to 3,3 % for miscellaneous impurities;
- 1,8 % to 8,7 % for total impurities.

For further information on the validation, see Annex D.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5223, *Test sieves for cereals*

3 Terms and definitions

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

3.1**Besatz**

all matters of a sample of grain other than the basic cereal of unimpaired quality

Note 1 to entry: It comprises the four fractions: broken grains (3.2), grain impurities (3.3), sprouted grains (3.4) and miscellaneous impurities (3.5).

Note 2 to entry: The sum of the four fractions is also called Total *Besatz*.

Note 3 to entry: Live pests are not considered as *Besatz*. They are specified as a separate criterion.

Note 4 to entry: A schematic summary on *Besatz* is given in Annex A.

3.2**broken grains**

grains or pieces of grains which pass through a sieve with a circular mesh of:

- 4,5 mm in diameter for maize;
- 1,8 mm in diameter for sorghum

3.3**grain impurities**

elements consisting of other cereals, grains damaged by pests, grains overheated during drying

3.3.1**other cereals**

cereal grains and their impurities consisting of grains which do not belong to the species of grain sampled

3.3.2**grains damaged by pests**

grains which show visible damage owing to attack by insects, rodents, mites or other pests

3.3.3**grains overheated by drying**

grains which have been overheated and have a coloration of the cross section of the germ darker than the colour standard and in which the kernel is sound

Note 1 to entry: For determination of such grains, it is recommended to use the colour standard (5.8) to compare with the colour of the section of the cut germ.

3.4**sprouted grains**

grains in which the radical or plumule is clearly visible to the naked eye

3.5**miscellaneous impurities**

elements consisting of extraneous seeds, unsound grains, extraneous matter and impurities of animal origin

3.5.1**extraneous seeds**

seeds of plants, whether or not cultivated, other than cereals

Note 1 to entry: They comprise noxious and non-noxious seeds. The term "noxious seeds" means seeds which are toxic to humans and animals. This group also includes seeds hampering or complicating the cleaning and milling of cereals and seeds affecting the quality of products processed from cereals.

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Note 2 to entry: In some cases it may be necessary to distinguish between noxious seeds and non-noxious seeds. An indicative list of noxious seeds is given in ISO 7970:2000, Annex A [5].

3.5.2**unsound grains**

grains rendered unfit for human consumption and, as regards feed grain, for feed consumption, owing to putrefaction, mildew, grains affected with fusariosis, or bacterial or other causes

Note 1 to entry: Unsound grains also include grains damaged by spontaneous heat generation or too extreme heating during drying which are fully grown grains in which the tegument is coloured greyish-brown to black while the cross-section of the kernel is coloured-yellowish grey to brownish-black.

3.5.3**extraneous matter**

all matters in a sample of grains that passing through a sieve with apertures of 1,0 mm are considered extraneous

Note 1 to entry: Extraneous matter also includes stones, sand, fragments of straw, cob fragments and similar impurities in the samples which are retained by a sieve with apertures of 1,0 mm.

3.5.4**impurities of animal origin**

impurities originating from animals such as feathers, hairs, excrements, dead insects and fragment of insects

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4 Principle

The principle of this method is to separate all the groups of impurities defined in 3.1, from the normal basic grains by sieving and manual selection.

5 Apparatus and equipment

5.1 **Sample divider.**

5.2 **Balance**, with a reading accuracy of 0,001 g and capable of weighing to the nearest 0,01 g.

5.3 **Sieves**, with circular holes of 4,5 mm and 1,8 mm in diameter and slot-widths 1,0 mm × 20,0 mm, in accordance with the specifications of ISO 5223.

5.4 **Sieving machine**, having a rectilinear movement of go and come in the sense of the rectangular holes.

5.5 **Magnifying glass**, illuminated.

5.6 **Forceps or horn spatula.**

5.7 **Pots**, for retaining components.

5.8 **Colour standard**, as reference S 3030-Y30R in the Natural Colour System (NCS)¹.

6 Sampling

It is important that the laboratory receives a sample which is truly representative and has not been damaged or changed during transport and storage.

Sampling is not part of the method specified in this European Standard. For information, a recommended sampling procedure is given in EN ISO 24333 [1] for the sampling of cereals with constituents distributed uniformly or not uniformly.

7 Procedure

Prepare by division a representative sample of at least (but near) 500 g for maize and 250 g for sorghum. Weigh it to the nearest 0,1 g (a). Pass the sample through the slotted sieve with an aperture of 1,0 mm (5.3), for half a minute. For constant sieving, a sieving machine (5.4) is recommended. If sieving is performed by hand, it shall consist of horizontal movements parallel to the length of the slots.

The matter passed through the 1 mm sieve shall be regarded as extraneous matter. Stones, mud balls, straws, chaff, cob fragments and similar impurities from the over tail of the 1,0 mm slotted sieve have to be picked out. Both fractions are combined and are regarded as extraneous matter (3.5.3). Weigh them to the nearest 0,1 g (b). Impurities of animal origin should be counted (n), including those which passed through the sieve of 1,00 mm slot-width (5.3). If necessary, a magnifying glass should be used.

The count of impurities of animal origin (3.5.4) should be quoted separately in numbers per kilogram of maize or sorghum, as appropriate.

From the over tails of the 1,0 mm sieve (5.3), prepare, with the aid of a sample divider, a test sample, between 100 g and 200 g for maize and between 25 g and 50 g for sorghum. Weigh this test sample to the nearest 0,01 g (c).

1) The Natural Colour System is defined by the Scandinavian Colour Institute AB, Stockholm (SE). For further explanation see www.ncscolour.com.

Subsequently spread out this partial sample in a thin layer on a table, and pick out by means of forceps or a horn spatula the element constituting the groups of impurities: other cereals (3.3.1), grains damaged by pests (3.3.2), sprouted grains (3.4), extraneous seeds (3.5.1), unsound grains (3.5.2) and remained extraneous matters (3.5.3).

Grains whose tegument shows abnormal colour shall be cut longitudinally through the germ. If a major part or the totality of the section of the germ is identical or darker than the colour standard (5.8) then the two halves of the initial grain have to be accounted as grains overheated by drying (3.3.3).

In the case of multiple kinds of damages are observed, the damaged grain shall be added to the fraction with the highest importance for the overall quality.

Subsequently sieve the same partial sample through a sieve of 4,5 mm circular holes in diameter for maize or 1,8 mm circular holes in diameter for sorghum for half a minute. The through of this sieve belong to the group broken grains (3.2).

Weigh the clean sample material (d) and all the groups of Besatz to the nearest 0,01 g. If, for a partial sample, the sum of weights for broken grains (3.2), other cereals (3.3.1), grains damaged by pests (3.3.2), grains overheated during drying (3.3.3), sprouted grains (3.4), extraneous seeds (3.5.1), unsound grains (3.5.2) and extraneous matters (3.5.3) and (d) differs by more than 1 % from (c), the determination shall be invalid and a new partial sample shall be analysed.

8 Expression of results

The mass fraction in percent of the Besatz fractions (3.2, 3.3.1, 3.3.2, 3.3.3, 3.4, 3.5.1 and 3.5.2) are calculated as follows:

$$B = x \times \frac{a-b}{c} \times \frac{100}{a}$$

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where <https://standards.iteh.ai/catalog/standards/sist/e4321201-ed0f-4407-bff2-a18225fcf586/sist-en-16378-2013>

- B is the mass fraction of Besatz fractions (%);
- x is the weight of the Besatz group concerned (g);
- a is the weight of the average sample (g);
- b is the weight of the extraneous matter on average sample (g);
- c is the weight of subsample from which Besatz will be removed (g).

The mass fraction in percent of extraneous matter (3.5.3) is calculated as follows:

$$A = \left(\left(x_8 \times \frac{a-b}{c} \right) + b \right) \times \frac{100}{a}$$

where

- A is the percentage of extraneous matter (%);
- x_8 is the weight of extraneous matter of subsample from which Besatz was removed (g);
- a is the weight of the average sample (g);
- b is the weight of the extraneous matter of average sample (g);
- c is the weight of subsample from which Besatz will be removed (g).

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The percentage of grains impurities (3.3) is calculated by adding percentages of other cereals (3.3.1), grains damaged by pests (3.3.2) and grains overheated by drying (3.3.3).

The percentage of miscellaneous impurities (3.5) is calculated by adding percentages of extraneous seeds (3.5.1), unsound grains (3.5.2) and extraneous matters (3.5.3).

The percentage of total impurities (3.1) is calculated by adding percentages of broken grains (3.2), grains impurities (3.3), sprouted grains (3.4) and miscellaneous impurities (3.5).

The calculation should be carried out to the nearest 0,01 %.

In the investigation report, quote to a precision of 0,1 % for broken grains (3.2), grains impurities (3.3), sprouted grains (3.4) and miscellaneous impurities (3.5) and to a precision of 0,01 % for all sub components of these categories. Report the impurities of animal origin (3.5.4) in number per kilogram of grain.

An example of calculation is given in Annex C.

9 Precision**9.1 General**

Details of an international inter-laboratory test on the precision of the method are summarised in Annex D. The values derived from this test may not be applicable to concentration ranges and matrices other than those given:

- 0,0 % to 2,7 % for broken grains;
- 0,2 % to 3,5 % for grains impurities;
- 0,5 % to 3,3 % for miscellaneous impurities; [SIST EN 16378:2013](https://standards.iteh.ai/catalog/standards/sist/e4321201-ed0f-4407-bff2-a18225f586/sist-en-16378-2013)
- 1,8 % to 8,7 % for total impurities. <https://standards.iteh.ai/catalog/standards/sist/e4321201-ed0f-4407-bff2-a18225f586/sist-en-16378-2013>

The formulae in subclauses 9.2 to 9.4 have been elaborated using the data of the inter-laboratory test; see Tables D.1 to D.5.

Because of a too tight range for sprouted grains in the inter-laboratory test, it has not been possible to determine fidelity values for this class of impurities.

9.2 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 not in more that 5 % of cases be greater than the repeatability limit r :

- Broken grains: $r = 2,8 \times [(0,058 \times B_{\text{broken grains}}) + 0,036]$;
- Grain impurities: $r = 2,8 \times [(0,242 \times B_{\text{grain impurities}}) + 0,074]$;
- Miscellaneous impurities: $r = 2,8 \times [(0,107 \times B_{\text{miscellaneous impurities}}) + 0,119]$;
- Total impurities: $r = 2,8 \times [(0,087 \times B_{\text{total impurities}}) + 0,176]$.

9.3 Reproducibility

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

- Broken grains: $R = 2,8 \times [(0,252 \times B_{\text{broken grains}}) + 0,015]$;
- Grain impurities: $R = 2,8 \times [(0,618 \times B_{\text{grain impurities}}) + 0,228]$;
- Miscellaneous impurities: $R = 2,8 \times [(0,437 \times B_{\text{miscellaneous impurities}}) + 0,077]$;
- Total impurities: $R = 2,8 \times [(0,478 \times B_{\text{total impurities}}) - 0,058]$.

9.4 Uncertainty

Uncertainty (U_e) is a parameter representing the distribution of the values which may reasonably be attributed to the result. This uncertainty is given by a statistical distribution of the results from the interlaboratory test and is characterised by the experimental standard deviation.

For every parameter, the uncertainty is equal to plus or minus twice the reproducibility standard deviation given in Annex D of the document.

- Broken grains: $R = 2 \times [(0,252 \times B_{\text{broken grains}}) + 0,015]$;
 - Grain impurities: $R = 2 \times [(0,618 \times B_{\text{grain impurities}}) + 0,228]$;
 - Miscellaneous impurities: $R = 2 \times [(0,437 \times B_{\text{miscellaneous impurities}}) + 0,077]$;
 - Total impurities: $R = 2 \times [(0,478 \times B_{\text{total impurities}}) - 0,058]$.
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10 Test report

The test report shall specify:

- a) all information necessary for the complete identification of the sample;
- b) the sampling method used, if known;
- c) the test method used, together with the reference to this European Standard;
- d) all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents which may have influenced the test results;
- e) the test results obtained, and, if the repeatability has been checked, the final quoted result obtained.

EXAMPLE The test results obtained can be reported as follows:

1)	Broken grains (3.2)	X,X %
2)	Grain impurities (3.3)	X,X %
	i) Other cereals (3.3.1)	X,XX %
	ii) Grains damaged by pests (3.3.2)	X,XX %
	iii) Grains overheated during drying (3.3.3)	X,XX %