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Žito in žitni proizvodi - Tehnično poročilo medlaboratorijske študije o določevanju nečistoč v koruzi (Zea mays, L.) in prosu (Sorghum bicolor, L.)

Cereal and cereal products - Technical report of the interlaboratory study for the determination of impurities content in maize (Zea mays, L.) and sorghum (Sorghum bicolor, L.)

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Cereal and cereal products - Technical report of the interlaboratory study for the determination of impurities content in maize (Zea mays, L.) and sorghum (Sorghum bicolor, L.)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (FprCEN/TR 16875:2015) has been prepared by Technical Committee CEN/TC 338 “Cereal and cereal products”, the secretariat of which is held by AFNOR.

This document is currently submitted to the Technical Committee Approval.

Introduction

The principle of the determination of impurities is to separate all the groups of impurities from the normal basic cereal grains of unimpaired quality by sieving and manual selection out of a subsample and to quantify them. The amount of impurities and its constituent groups is important for health, cleaning, milling and further processing aspects. For these reasons impurities content is a part of contracts in grain trade and also of the grain intervention system of the EU.

At present a European standard for the determination of impurities in maize and sorghum (EN 16378) has been developed.

An international interlaboratory trial for the determination of impurities in maize and sorghum was accomplished in order to get information on the intra- and interlaboratory variability of the determination of impurities content.

The technical report here describes the preparation and evaluation of the results of this interlaboratory test.

1 Scope

The term impurities applies to all components of a grain sample that differ from the normal basic cereal. It includes the following groups: broken grains, other cereals, grains damaged by pests, grains overheated during drying, sprouted grains, extraneous seeds, unsound grains, extraneous matter and impurities of animal origin.

The principle of the determination of impurities content is to separate all the groups of impurities from the normal basic cereal grains of unimpaired quality by sieving and manual selection out of a subsample and to quantify them. There are various problems in the determination of impurities:

Firstly, the identification of the different groups of impurities depends strongly on the experience and the knowledge of the investigator.

Also experienced investigators can differ in their characterization of grains.

Finally, one is faced with the fact that grain, even after mixing, is rarely homogenous. In other words, if a sample was divided by a sample divider into a number of portions, the amount of a specific group of impurities in each portion could be different, even if absolutely no human or machine error occurred in each determination.

These problems will result in variation of the results of the determination.

An international interlaboratory test for the determination of impurities, according to this standard and involving 14 laboratories in 4 countries, was carried out with 5 maize and 3 sorghum samples. It was asked to participants to make determination in duplicate.

Ten laboratories reported results for the complete sample set and two only for corns.

The test materials ranged between:

— 0,0 % and 2,7 % for broken grains;

— 0,2 % and 3,5 % for grain impurities;

— 0,0 % and 0,1 % for sprouted grains;

— 0,5 % and 3,3 % for miscellaneous impurities;

— 1,8 % and 8,7 % for total impurities.

The aim of the study is to determine the precision, repeatability and reproducibility of the method of determination of impurities content in maize and sorghum samples.

The analyses were realized in March - April 2011.

It occurs according to ISO 5725:1994.

2 Normative references

Not applicable.

3 Design of the study

3.1 Conception and organization

3.1.1 General

The interlaboratory comparisons of this test were designed according to the wishes of AFNOR and organized by BIPEA.

3.1.2 Method

The participants made a commitment to apply strictly the described method and to prepare the samples according to the method provided with the commitment letter for participating to the test: the standard EN 16378.

3.1.3 Participants

Fourteen laboratories made a commitment to participate to the test. Twelve did answer.

3.1.4 Design and schedule of the study

Each laboratory received eight samples: 5 samples of corn and 3 samples of sorghums, according to a blind distribution.

In order to take into account the sampling of the laboratory into the precision values, the laboratories realized two analytical series on two sub-samples from each sample, leading to 16 determinations of impurities.

Tests were conducted between March, 1st and April, 26th, 2011.

In order to be as close as possible to repeatability conditions, the two sub-samples analyses has been realized during a time as short as possible. The repeatability variance is an intra-laboratory variance. However, in order to be easier to read, it is called repeatability mean in the report.

3.2 Product

In order to cover the scope of the method, the products analysed are maize mixes and sorghum mixes.

3.3 Fabrication

The samples are produced according to the following procedure:

Each batch is homogenized and divided in samples serials. This operation is made with a carousel. The principle of turning spreading, that involves a progressive filling, ensures the homogeneity of the product between each sample.

The samples of maize 1, 2 and 4 and the samples of sorgho 1 and 3 were of 500 g in paper bags. The samples of maize 3 and 5 and the samples of sorgho 2 were of 1 kg in paper bags. The weights were increased for some samples in order to integer the division step inside the laboratory.

3.4 Homogeneity and stability

On this test, no control was planned. However, homogeneity and stability were considered as sufficient by the working group for the test, during its conception.

3.5 Form

In addition to final results, expressed in % of impurities, the weights values were requested to the participants. The form sheet template is in A.2.

4 Statistics

4.1 Methodology

The precision parameters were calculated on the two sub-samples of each impurity category according to the following plan.

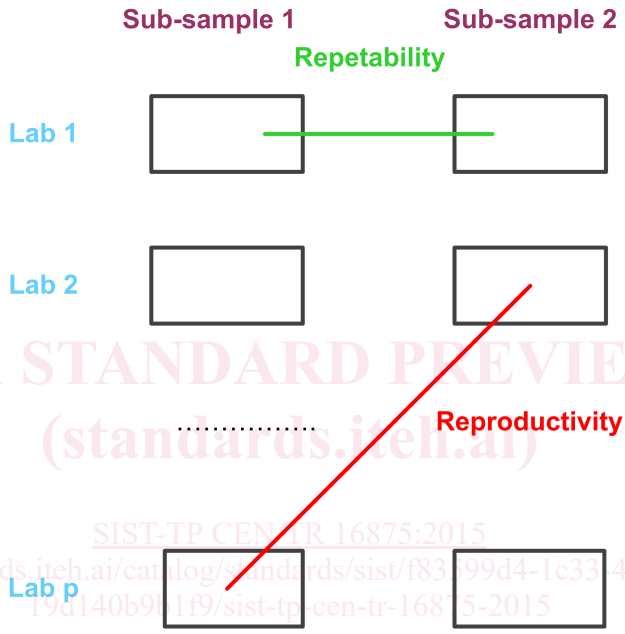

Figure 1

4.2 Statistical treatment

The statistical treatment was carried out according to ISO 5725-2. [1]

5 Results of the interlaboratory study

5.1 Validation of the raw results

The project leader realized the checks of the calculations of impurities contents from the weight values.

A laboratory seems not to have followed the method; it was excluded from the calculations of the precision parameters (laboratory 7568).

A laboratory did not write correctly his results, it has a lack of traceability of its samples (laboratory 2190). The results of the corn 3 have been reported on the corn 2 form sheets, those of corn 4 on the form sheet for corn 3 and those of corn 2 on the form sheet of corn 4.

The traceability and the results have been corrected.

5.2 Detection of stragglers and outliers

Stragglers and outliers detection have been realized with statistical tests on the provided results except for the excluded laboratory above:

— Cochran's test: outliers and stragglers for variances (at 5 % and 1 %);

— Grubbs' test: outliers and stragglers for means (at 5 % and 1 %).

The principles of the tests and the decision rules are described in ISO 5725-2.

The results of those tests are presented in Table A.1 to Table A.10. As abstract:

— For the corn 1:

  — the variance of the laboratory 8577 for miscellaneous impurities has been detected as outlier;

  — the variance of the laboratory 8577 for total impurities has been detected as straggler;

  — the mean of the laboratory 7489 for broken grains has been detected as outlier;

  — the mean of the laboratory 6723 for total impurities has been detected as straggler.

— For the corn 2:

  — the variance of the laboratory 5909 for broken grains has been detected as outlier;

  — the mean of the laboratory 7489 for broken grains has been detected as outlier;

  — the mean of the laboratories 6723 and 8577 for miscellaneous impurities has been detected as straggler;

— For the corn 3:

  — the variance of the laboratories 5909 for miscellaneous impurities and 6723 for total impurities has been detected as outlier;

  — the variance of the laboratory 6637 for sprouted grains has been detected as straggler;

  — the mean of the laboratory 8577 for miscellaneous impurities and for total impurities has been detected as straggler.

— For the corn 4:

  — the variance of the laboratory 8577 for sprouted grains and for grain impurities has been detected as outlier;

  — the variance of the laboratory 8577 for broken grains and for total impurities has been detected as straggler;

  — the mean of the laboratory 8577 for miscellaneous impurities has been detected as straggler.

— For the corn 5:

  — the variance of the laboratory 8577 for broken grains and for miscellaneous impurities has been detected as outlier;

- the variance of the laboratory 8577 for grain impurities and for total impurities has been detected as straggler;
- the mean of the laboratory 6723 for grain impurities, for miscellaneous impurities and for total impurities;
- the mean of laboratory 8577 for miscellaneous impurities has been detected as straggler.

— For the sorgho 1:

- the variance of the laboratory 5703 for grain impurities has been detected as outlier;
- the mean of the laboratory 6723 for grain impurities has been detected as straggler.

— For the sorgho 2:

- the variance of the laboratory 7489 for total impurities has been detected as outlie;
- the variance of the laboratory 7489 for miscellaneous impurities has been detected as straggle;
- the mean of the laboratory 6723 for grain impurities has been detected as straggler.

— For the sorgho 3:

- the variance of the laboratory 7489 for miscellaneous impurities and for total impurities has been detected as straggler.

The values for which the means are judged as stragglers have been excluded from the precision parameters calculation for the concerned corns and the sorghum.

The values for which the variances are judged as stragglers have been excluded from the precision parameters calculation for the concerned corns and the sorghum.

5.3 Stragglers and outliers detections

Table 1 — Cochran's test: stragglers (in bold) and outliers for variances

Lab numbers	Maize 1	Maize 2	Maize 3	Maize 4	Maize 5	Sorghum 1	Sorghum 2	Sorghum 3
Broken grains	-	5909	-	**8577**	8577	-	-	-
Grain impurities	-	-	-	8577	**8577**	5703	-	-
Sprouted grains	-	-	**6637**	8577	-	-	-	-
Miscellaneous impurities	**8577**	-	5909	-	8577	-	**7489**	**7489**
Total impurities	**8577**	-	-	**8577**	**8577**	-	7489	**7489**