SLOVENSKI STANDARD
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Žito - Določanje vlage in beljakovin - Metoda z uporabo bližnje infrardeče transmisije v metodi bližnje infrardeče spektroskopije v celih zrnih

Cereals - Determination of moisture and protein - Method using Near-Infrared-Transmittance in Method using Near-Infrared-Spectroscopy in whole kernels

Getreide - Bestimmung der Feuchte und des Proteins - Verfahren der Ganzkorn-Nahinfrarot-Transmission bei einer Nahinfrarot-Spektroskopie-Methode

Céréales - Détermination de la teneur en eau et en protéines - Méthode utilisant la transmission dans le proche infrarouge issue de la méthode utilisant la spectroscopie dans le proche infrarouge sur des grains entiers

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ICS:

67.060	Žita, stročnice in proizvodi iz njih	Cereals, pulses and derived products

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Cereals - Determination of moisture and protein - Method using Near-Infrared-Transmittance in Method using Near-Infrared-Spectroscopy in whole kernels

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Contents

Page

Foreword

This document (prEN 15948:2011) 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 second CEN Enquiry.

1 Scope

This standard specifies a routine method for the determination of moisture and protein in whole kernels of barley and wheat in the constituent ranges of 8 -28% moisture and 7-22% protein using a near-infrared spectrophotometer.

2 Normative references

The following documents are indispensable for the application 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.

EN ISO 712 *Cereals and cereal products -- Determination of moisture content -- Routine reference method*

EN ISO 5983-2 *Animal feeding stuffs -- Determination of nitrogen content and calculation of crude protein content -- Part 2: Block digestion/steam distillation method*

EN ISO 12099 *Animal feeding stuff, cereals and milled cereal products — Guidance for the application of near infrared spectrometry*

EN ISO TS 16634-2 *Food products - Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content -- Part 2: Cereals, pulses and milled cereal products*

EN ISO 20483 *Cereals and pulses -- Determination of the nitrogen content and calculation of the crude protein content -- Kjeldahl method*

ISO 5725-2 *Application of statistics- Accuracy (trueness and precision) of measurement methods and results –Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

See terms and definitions (Annex C) in EN ISO 12099

4 Principle

The method is based on Near-Infrared (NIR) spectroscopy, a secondary, correlative technique to predict the concentration of various constituents in organic samples. Linear and nonlinear regression modeling is used to relate NIR spectra to moisture or protein concentrations determined by officially approved standard methods (e.g. artificial neural network-ANN, Partial Least Square Regression –PLS,…)

5 Principle

5.1 Near Infrared Instrument

Based on diffuse reflectance or transmittance measurement covering the wavelength region of 700–2500 nm or segments of this or at selected wavelengths.

5.2 Prediction model

The model for the prediction of protein and moisture contents in whole grain of wheat and barley is defined by:

— the number of samples used for the calibration development

— the constituent ranges covered in the model for moisture and protein

— the calibration file name associated to each parameter

— the seasonal, geographic and genetic varieties covered.

6 Sampling

Sampling is not part of the method specified in this standard. A recommended sampling procedure is given in EN ISO 24333.

It is important that the sample analyzed is truly representative for the lot and has not been damaged or changed.

7 Procedure

7.1 Preparation of the test sample

No specific sample preparation is required.

7.2 Determination

Follow the instructions of the instrument manufacturer.

Check the instrument stability as described in EN ISO 12099

7.3 Performance check of calibration

Regularly run a performance check of the calibration model according to EN ISO 12099.

In situations where several instruments have been standardized against each other and are operated in a network it is sufficient to run the performance check of the calibration on just one instrument.

8 Calculation and expression of results

The software of the instrument calculates the results for moisture and protein and displays them in e.g. % w/w (g/100g) to 2 decimal places.

If multiple measurements are made on the same sample, calculate the arithmetic mean.

Express final results to one decimal place.

9 Validation

Since NIR analysis is a secondary, correlative technique, the results must be validated against chemical analysis reference methods. It is important that the reference methods used are officially approved such as the EN ISO standards previously cited (clause 2). The purpose of validation is two-fold; determine the overall correlation of predicted results to the chemical analysis and to evaluate any bias.

The linear correlation (R^2) between chemical analysis methods and predictions can be compared to calibration performance specifications and/or historical performance.

9.1 Initial validation

9.1.1 Validation sample set

The initial validation of a calibration model must be done in accordance with EN ISO 12099 using independent test sets of wheat and barley samples, originating from different countries and analyzed by the reference methods given in clause 2.

Requirements for the validation sample set are:

— at least 300 samples coming from 15 countries (20 representative samples min/country) distributed homogeneously over the entire range,

— the part of the range without any reference sample cannot exceed 0.3 % ,

— different scans from one sample cannot be considered as different samples,

— seasonal and temperature effects, instrument variation must be included in the set

9.1.2 Initial validation performances

The results of the initial validation must fulfill the specifications given in table 1.

Table 1 — NIR performances for the determination of moisture and protein (see Annex B)

		Moisture	Protein
Overall accuracy expressed as SEP as constituent % w/w		0,24	0,27
Constituent concentration in the independent validation data set	Min	8,0 %	7 % d.m.
	Max	28 %	22 % d.m.

9.2 Local validation

Before use, the calibration model shall be validated on an independent test set that is representative of the sample population to be analyzed. For the determination of bias, at least 10 samples are needed; for the determination of Standard Error of Prediction (SEP, see EN ISO 12099, Clause 6.5) at least 20 samples are needed. Validation must be carried out for each sample type, constituent/ parameter and temperature (see EN ISO 12099, Clause 5.4).

Bias or inherent systematic error, as described in EN ISO 12099 (Clause 6), is exhibited when the predicted results of a specific sample group or product show a mean offset value when compared to their reference values. This may occur with unique sample types.

The bias (i.e. mean difference between the chemical analysis results and the predicted results) may or may not be statistically significant. Based on the procedure described in EN ISO 12099, a bias confidence limit can be calculated.

When this limit is exceeded, a bias (or intercept) is implemented in the instrument software and the validation process repeated. Refer to the manufacturer instructions and to EN ISO 12099 for procedure.

9.3 Validation during use

See clause 10 in EN ISO 12099.

10 Update of calibration model and validation of new model

The prediction model in accordance with this standard should be updated by the one issuing the calibration model to ensure inclusion of new climatic crop conditions and new varieties introduced on the market. These updates shall be made by keeping the original database with addition of the new samples as needed.

The new prediction model shall be updated according to EN ISO 12099. Validation shall be made with the original validation set including a representative number of samples covering the newly added sample properties. The new independent test set should include 30% of the new samples, but at least 20 samples.

Annual validation shall be made with samples from an internationally accepted proficieny testing scheme (PTS) that includes NIRS predicted results and results generated by the reference methods specified in clause 2.

11 Accuracy and precision

11.1 Accuracy

The accuracy of the prediction model is determined by validation in accordance with EN ISO 12099 and expressed by the Standard Error of Prediction (see table 1). The Standard Error of Prediction (SEP) is an expression of the bias corrected average difference between predicted and reference values predicted by the model when applied to a set of samples not included in the derivation of the model. The values also include the uncertainty of reference results.

The predicted results will not in more that 5 % of cases deviate more than 1,96 x the above values from the best estimate of the true value.

NOTE As NIR is an indirect method, the typical standard deviation of reproducibility for the used reference methods are given here for comparison

- Moisture (EN ISO 712) = 0,16%
- Protein (EN ISO 20483) = 0,20%
- Protein (EN ISO 5983-2) = 0,20%
- Protein (EN ISO/TS 16634-2)= 0,21-0,26%

11.2 Precision

The precision of the prediction model shall be determined from an interlaboratory test organized according to ISO 5725-2 and at least fulfil the performances of repeatability and reproducibility given below.

Details of an example of interlaboratory test are summarized in Annex A, that is informative.

The figures in Annex A show that the repeatability and the reproducibility are independent of the concentration. The figures in Annex B show that the dispersion is identical over the validated range. The figures B.1 and B.2

can therefore be used in the whole validated range, even though the interlaboratory trial covered a smaller range.

11.3 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 ($r=s_r*2,8$) with:

r (protein) = 0,42

r (moisture) = 0,15

11.4 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 that 5 % of cases be greater than the reproducibility limit R ($R=s_R*2,8$) with:

R (protein) = 0,45

R (moisture) = 0,25

11.5 Critical difference

When the difference between two averaged values obtained from two test results under repeatability or reproducibility conditions is to be assessed, the repeatability or reproducibility limit cannot be used, one shall use the Critical Difference (CD)

11.5.1 Comparison of two groups of measurements in one laboratory

The critical difference (CD) between two averaged values obtained from two test results under repeatability conditions is equal to:

$$CD = 2,8\ s_r \sqrt{\frac{1}{2n1} + \frac{1}{2n2}} = 2,77\ s_r \sqrt{\frac{1}{2}} = 1,98\ S_r$$

where

Sr is the standard deviation of repeatability;

*n*1 and *n*2 are the number of test results corresponding to each of the averaged values

CDr (protein) = 0,30

CDr (moisture) = 0,11

11.5.2 Comparison of two groups of measurements in two laboratories

The critical difference (CD) between two averaged values obtained in two different laboratories from two test results under repeatability conditions is equal to:

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

where

Sr is the standard deviation of repeatability;

SR is the standard deviation of reproducibility;

*n*1 and *n*2 are the number of test results corresponding to each of the averaged values

CD_R (protein) = 0,32

CD_R (moisture) = 0,23

12 Test Report

The test report shall specify:

— all information necessary for the complete identification of the sample

— the sampling method used (if known)

— the application model and instrument used with reference to this standard

— all operating details not specified in this standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s)

— the test result(s) obtained