SLOVENSKI STANDARD
oSIST prEN 15948:2019

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Žito - Določevanje vlage in beljakovin - Metoda z uporabo bližnje infrardeče spektroskopije v celih zrnih

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

Getreide - Bestimmung der Feuchte und des Proteins - Verfahren der Nahinfrarot-Spektroskopie bei ganzen Körnern

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

Ta slovenski standard je istoveten z: **prEN 15948**

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-Spectroscopy in whole kernels

Getreide - Bestimmung der Feuchte und des Proteins - Verfahren der Nahinfrarot-Spektroskopie bei ganzen Körnern

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 338.

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

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European foreword

This document (prEN 15948:2018) has been prepared by Technical Committee CEN/TC 338 “Cereals and cereal products”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN ENQUIRY.

This document will supersede EN 15948:2015.

In comparison with the previous edition, the following technical modifications have been made:

— updating normative references,

— change in the number of validation samples according to the new version of EN ISO 12099-2017,

— precision of the expression of the protein content,

— repeatability and reproducibility determined from 3 interlaboratory tests and addition of uncertainty according to EN ISO 12099,

— removal of informative annexes concerning 3 interlaboratory tests.

1 Scope

This document defines a routine method for the determination of moisture and protein in whole kernels of barley and wheat using a near-infrared spectroscopy in the constituent ranges:

for wheat:

— moisture content minimum range from 8 % to 22 %;

— protein content minimum range from 7 % to 20 % DM.

for barley:

— moisture content minimum range from 8 % to 22 %;

— protein content minimum range from 7 % to 16 % DM.

This document describes the modalities to be implemented by the supplier (5.3 and 5.4) and the user of the method.

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.

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN ISO 12099 apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

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

5 Method of analysis

5.1 General

According to this document, the method of analysis is defined as the association between a NIR instrument and a model of prediction.

5.2 Near Infrared Instrument

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

5.3 Prediction models

Each model for the prediction of protein and moisture contents in whole grain of wheat and barley is amongst others characterized by:

— the number of samples used for the calibration development;

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

— the factor for calculation of the crude protein content from the total nitrogen content: 5,7 or 6,25 taken in account in reference values;

— the expression of the protein result (%DM)

— the temperature range of the samples;

— the number and performance of involved reference laboratories;

— the stability of the model i.e. by number of harvests covered;

— the calibration file defined by its name and its IT name (for example CHECKSUM) insuring its integrity;

— the seasonal, geographic and genetic variations covered.

5.4 Initial validation of the model

5.4.1 General

Since NIR analysis is an indirect, correlative technique, the results shall be validated against chemical analysis reference methods. It is important that the reference methods used are officially approved such as the methods described in the EN ISO standards [4], [5], [6] and [7]. The purpose of validation is to determine the standard error of prediction which expresses the accuracy of routine NIR results corrected for the mean difference (bias) between routine NIR and reference method.The standard error of prediction between chemical analysis methods and predictions shall be compared to calibration performance specifications and/or historical performance.

5.4.2 Initial validation sample set

The initial validation of a calibration model shall be done in accordance with EN ISO 12099 using independent test sets of wheat and barley samples, originating from different countries and analysed by the reference methods as [4], [5], [6] or [7].

Requirements for the validation sample set are:

— For each crop species, at least 200 representative samples coming from countries where the model will be used (20 representative samples min/country) distributed homogeneously over the entire constituent range;

— the part of the range without any reference sample shall not exceed 0,3 %;

— the same sample analysed on several instruments or several times on a same device counts for one sample;

— seasonal effects over at least a three year period, temperature effects, instrument variation and the variability of reference data shall be included in the set.

All this information shall be given in the report of the calibration model.

5.4.3 Initial validation performances

The results of the initial validation shall at least fulfil the specifications given in Table 1.

Table 1 — NIR performances for the determination of moisture and protein

		Moisture Wheat and barley	Protein Wheat	Protein Barley
Overall accuracy expressed as SEP as constituent % w/w		0,24 %	0,25 %	0,27 %
Constituent concentration in the independent validation data set	Min	8,0 %	7,0 %DM	7,0 %DM
	Max	22,0 %	20,0 %DM	16,0 %DM

NOTE The minimum performance given in Table 1 includes the variation of reference data as documented by the number of reference laboratories involved, regional and genetic variations, the number of countries and crop species involved.

5.5 Update of calibration model and validation of new model

The prediction model in accordance with this standard shall 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 according to the initial validation (5.4) and include at least 20 new samples of the species concerned by the update.

6 Sampling

Sampling is not part of the method specified in this European Standard. A recommended sampling procedure is given in EN ISO 24333 [2].

It is important that the sample analysed in routine is truly representative for the batch and has not been damaged or modified.

7 Procedure

7.1 Preparation of the best sample

No specific sample preparation is required.

7.2 Measurement

Follow the instructions of the instrument manufacturer.

7.3 Local validation of the method

Before use, the method shall be validated on an independent test set that is representative of the sample population to be analysed. At least 20 samples are needed for the determination of the bias, the Standard Error of Prediction (SEP, see EN ISO 12099; Clause Statistics for performance measurement) and the slope. Validation shall be carried out for each sample type (wheat, barley), constituent/ parameter (moisture, protein) and temperature (see EN ISO 12099; Clause Validation of calibration models).

Bias or inherent systematic error, as described in EN ISO 12099(Clause Statistics for performance measurement) 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 the limit for bias is exceeded, a correction is implemented in the instrument software and the validation process repeated.

When the limit for SEP or for slope is exceeded, contact the supplier of the prediction model. The validation will not be accepted.

7.4 Periodical adjustement of the instrument

As mentioned in EN ISO 12099 (Clause Checking instrument stability), if several instruments are used in a network, special attention shall be given to standardization of the instruments according to the manufacturer's recommendations.

7.5 Checking instrument stability

See Clause Checking instrument stability of EN ISO 12099.

7.6 Follow up of method performance

Performance of the method shall be checked at least annually, against reference methods to secure the constant adequacy of the model with the requirements of this standard (see 5.4.3).

This performance test shall be made on samples selected from the pool of analysed samples. It may be necessary to resort to some sampling strategy to ensure a balanced sample distribution over the entire calibration range and to ensure that samples with a commercially important range are covered. At least 20 samples are needed (to expect a normal distribution of variance).

For instruments operated in a network and adjusted against a master instrument, it is sufficient to run the performance check of the method on the master instrument.

The adjustment (7.4) respecting the requirements of this clause may be used for the follow-up of the method performance.

It is recommended to participate in an internationally accepted proficiency testing scheme (PTS) that includes NIRS predicted results and results generated by following the standards specified in Bibliography. That participation alone cannot be considered sufficient for the follow up of method performance given the reduced number of samples analysed per year.

8 Calculation and expression of results

The software of the instrument calculates the results for moisture and protein and displays them with two decimal places.

If two measurements are made on the same sample, calculate the arithmetic mean if the repeatability conditions are met (see 9.2.2).

In any case, express final result to one decimal place with the indicated mode given by the prediction model: % for moisture; % DM for protein.

9 Accuracy and precision of the method

9.1 Accuracy

The accuracy of the prediction model is determined by validation in accordance with EN ISO 12099and expressed by the Standard Error of Prediction (see Table 1). The Standard Error of Prediction (SEP), i.e. standard deviation of the residuals, expresses the average difference observed between the predicted values by the model corrected for a systematic bias and the reference values for 68 % of the samples of a set not included in the development of the model.

The predicted results will not in more than 5 % of cases deviate more than 1,96 x SEP (as determined in the above paragraph) 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 16634-2) = 0,21-0,26 %.

9.2 Precision

9.2.1 General

The repeatability and reproducibility limits of the method have been derived from the compilation of the results of three interlaboratory tests carried out in accordance with ISO 5725-1 and ISO 5725-2.

The details of these interlaboratory tests organized by three manufacturers are included in other standards.

The values obtained may not be applicable to concentration ranges and matrices other than those given:

Wheat protein: 10,0 – 18,56 % DM,

Barley protein: 9,2 – 15,4 %DM,

Wheat moisture: 9,5 – 15,7 %,