



SLOVENSKI STANDARD

SIST EN 15948:2020

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Nadomešča:
SIST EN 15948:2015

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

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

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EUROPEAN STANDARD

EN 15948

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

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

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

This European Standard was approved by CEN on 21 September 2020.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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

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European foreword

This document (EN 15948:2020) 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 April 2021, and conflicting national standards shall be withdrawn at the latest by April 2021.

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

This document supersedes 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,
- precision about practical use of precision,
- removal of informative annexes concerning 3 interlaboratory tests. All these data being included in technical report CEN/TR 17474¹ (Cereals (wheat and barley) Technical Report of the interlaboratory studies for the determination of moisture and protein in whole kernels by near infrared spectroscopy) [1].

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

¹ Under preparation. Stage at the time of publication: FprCEN/TR 17474.

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1 Scope

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

- for wheat:
 - moisture content minimum range from 8 % to 22 %;
 - protein content minimum range from 7 %DM to 20 %DM.
- for barley:
 - moisture content minimum range from 8 % to 22 %;
 - protein content minimum range from 7 %DM 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)*

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3 Terms and definitions

standards.iteh.ai/catalog/standards/sist/899d9bc3-4681-4162-bce1-14cbb84c79c1/sist-en-15948-2020

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:

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

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 Squares (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 or calibration.

5.2 Near Infrared Instrument

Instrument based on diffuse reflectance or transmittance measurement covering the wavelength region of 700 nm to 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 (prediction model) 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 for wheat or 6,25 for barley taken into account in reference values;
- the expression of the protein result (%DM or as is);
- 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 [2], [3], [4] and [5]. The purpose of validation is to determine the standard error of prediction (SEP or SEP(C)) 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 performances specifications and/or historical performances.

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5.4.2 Initial validation sample set

The initial validation of a calibration 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 [2], [3], [4] or [5].

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 % or %DM;
- 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.

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 contents

		Moisture Wheat and barley	Protein Wheat	Protein Barley
Overall accuracy expressed as SEP as constituent		0,24 %	0,25 %DM	0,27 %DM
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 performances 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 and validation of new model

The prediction model in accordance with this document shall be updated by the one issuing the calibration to ensure inclusion of new climatic crop conditions and new varieties introduced on the market. These updates shall integrate new samples to include their variability.

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 document. A recommended sampling procedure is given in EN ISO 24333 [6].

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 test 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 or 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 could occur with unique sample types.

The bias (i.e. mean difference between the chemical analysis results and the predicted results) might or might 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.

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 adjustment 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.