SLOVENSKI STANDARD
oSIST prEN ISO 12099:2016

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Krma, žito in mlevski proizvodi - Smernice za uporabo bližnje infrardeče spektrometrije (ISO/DIS 12099:2016)

Animal feeding stuffs, cereals and milled cereal products - Guidelines for the application of near infrared spectrometry (ISO/DIS 12099:2016)

Futtermittel, Getreide und gemahlene Getreideerzeugnisse - Anleitung für die Anwendung von Nahinfrarot-Spektrometrie (ISO/DIS 12099:2016)

Aliments des animaux, céréales et produits de mouture des céréales - Lignes directrices pour l'application de la spectrométrie dans le proche infrarouge (ISO/DIS 12099:2016)

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Animal feeding stuffs, cereals and milled cereal products — Guidelines for the application of near infrared spectrometry

Aliments des animaux, céréales et produits de mouture des céréales — Lignes directrices pour l'application de la spectrométrie dans le proche infrarouge

ICS: 65.120

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 12099 was prepared by Technical Committee ISO/TC 34, *Animal feeding stuffs*, Subcommittee SC 10, and by Technical Committee CEN/TC 327, *Animal feeding stuffs* in collaboration.

This second/third/... edition cancels and replaces the first/second/... edition (), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

Introduction

This document has been drafted using as a basis the ISO 21543 / IDF 201 standard [15], which was prepared by Technical Committee ISO/TC 34, Food products, Subcommittee SC 5, Milk and milk products, and the International Dairy Federation (IDF).

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

1 Scope

This International Standard gives guide lines for the determination by near infrared spectroscopy of constituents such as moisture, fat, protein, starch and crude fibre and parameters such as digestibility in animal feeding stuffs, cereals and milled cereal products.

The determinations are based on spectrometric measurement in the near infrared spectral region.

2 Terms and definitions

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

2.1 near infrared (NIR) instrument

proprietary apparatus which, when used under the conditions defined in this International Standard, predicts the mass fractions of constituents and technological parameters as described below in animal feeding stuffs, cereals and milled cereal products through relationships to absorptions in the near infrared range.

2.2 animal feeding stuffs

any substance or product, including additives, whether processed, partially processed or unprocessed, intended to be used for oral feeding to animals.

Examples: Raw materials, fodder, animal flour, mixed feed and other end products, pet food etc.

2.3 constituent content

mass fraction of substances determined using the appropriate, standardized or validated chemical method .

NOTE 1 The mass fraction is often expressed as a percentage.

NOTE 2 Examples of constituents determined include moisture, fat, protein, crude fibre, neutral detergent fibre, and acid detergent fibre. For appropriate methods see e.g. [1-12].

2.4 technological parameters

Property or functionality of animal feeding stuff, cereals and milled cereal products that can be determined using the appropriate, standardized or validated method(s).

Example of such a parameter is the digestibility.

NOTE It is possible to develop and validate NIR methods for other parameters and sample types than listed above, as long as the procedure from this standard is observed. The measuring units of the parameters determined have to follow the units used in the reference methods.

3 Principle

Spectral data in the near infrared region are collected and transformed to constituent or parameter concentrations by calibration models developed on representative samples of the concerned products.

4 Apparatus

4.1 Near-infrared instruments

Instruments based on diffuse reflectance or transmittance measurement covering the near infrared wavelength region of 770–2500 nm (12 900 cm^{-1} – 4 000 cm^{-1}) or segments of this or at selected wavelengths or wavenumbers. The optical principle may be dispersive (e.g. grating monochromators), interferometric or non-thermal (e.g. light emitting diodes, laser diodes and lasers). The instrument should be provided with a diagnostic test system for testing photometric noise and reproducibility, wavelength/wavenumber accuracy and wavelength/wavenumber precision (for scanning spectrophotometers).

The instrument should measure a sufficiently large sample volume or surface to eliminate any significant influence of inhomogeneity derived from chemical composition or physical properties of the test sample. The sample path length (sample thickness) in transmittance measurements should be optimized according to the manufacturer's recommendation with respect to signal intensity for obtaining linearity and maximum signal/noise ratio.

4.2 **Appropriate milling or grinding device**, for preparing the sample (if needed).

NOTE Changes in grinding or milling conditions may influence NIR measurements.

5 Calibration and initial validation

5.1 General

The instrument has to be calibrated before use. Because a number of different calibration systems can be applied with NIR instruments, no specific procedure can be given for calibration.

For an explanation of methods for calibration development see a current textbook [16] and respective manufacturers manuals. For the validation it is important to have a sufficient number of representative samples, covering variations such as

a) Combinations and composition ranges of major and minor sample components

b) Seasonal, geographic and genetic effects on forages, feed raw material and cereals

c) Processing techniques and conditions

d) Storage conditions

e) Sample and instrument temperature

f) Instrument variations (differences between instruments)

NOTE : For a solid validation at least 20 samples are needed.

5.2 Reference methods

Internationally accepted reference methods for determination of moisture, fat, protein and other constituents and parameters should be used. See bibliography [1-12] for examples.

The reference method used for calibration should be in statistical control, i.e. for any sample, the variability should consist of random variations of a reproducible system. It is essential to know the precision of the reference method.

5.3 Outliers

In many situations, statistical outliers are observed during calibration and validation. Outliers may be related to NIR data (spectral outliers, hereafter referred to as x-outliers) or errors in reference data or samples with a different relationship between reference data and NIR data (hereafter referred to as y-outliers) (see fig B1- B5).

For the purpose of validation, samples are not to be regarded as outliers if they fulfill the following conditions:

a) If they are within the working range of the constituents/parameters in the calibration(s).

b) If they are within the spectral variation of the calibration samples, as e.g. estimated by Mahalanobis distance

c) if the spectral residual is below a limit defined by the calibration process

d) and if the prediction residual is below a limit defined by the calibration process

If a sample appears as an outlier then it should be checked initially to see if it is an x-outlier. If it exceeds the x-outlier limits defined for the calibration it should be removed. If it is not an x-outlier, then both the reference value and the NIR predicted value should be checked. If these confirm the original values then the sample should not be deleted and the validation statistics should include this sample. If the repeat values show that either the original reference values or the NIR predicted ones were in error then the new values should be used.

5.4 Validation of calibration models

Before use, calibration equations must be validated locally on an independent test set that is representative of the sample population to be analyzed. For the determination of bias, slope and for the determination of Standard Error of Prediction (s_{SEP}, see clause 6.5), at least 20 samples are needed. Validation must be carried out for each sample type, constituent/ parameter and temperature. The calibration is valid only for the variations, i.e. sample types, range and temperature, used in the validation.

NOTE: Calibration models can only be used in the range they have been validated.

Results obtained on the independent test set are plotted, reference against NIR, and residuals against reference results, to give a visual impression of the performance of the calibration. The s_{SEP} is calculated (see clause 6.5) and the residual plot of data corrected for mean systematic error (bias) is examined for outliers, i.e. samples with a residual exceeding $\pm 3\ s_{SEP}$.

If the validation process shows that the model cannot produce acceptable statistics then it should not be used.

NOTE: What will be acceptable will depend i.e. on the performance of the reference method, the covered range, the purpose of the analysis etc. and is up to the parties involved to decide.

The next step is to fit NIR, y_{NIR} , and reference data, y_{ref} , by linear regression ($y_{ref} = by_{NIR} + a$) to produce statistics that describe the validation results.

5.4.1 Bias correction

The data are also examined for a bias between the methods. If the difference between means of the NIR predicted and reference values is significantly different from zero then this indicates that the calibration is biased. A bias may be removed by adjusting the constant term (see 6.3) in the calibration equation.

5.4.2 Slope adjustment

If the slope (*b*) is significantly different from 1, the calibration is skewed.

Adjusting the slope/intercept of the calibration is generally not recommended unless the calibration is applied to new types of samples or instruments. If a reinvestigation of the calibration does not detect outliers, especially outliers with high leverage, it is preferable to expand the calibration set to include more samples. However, if the slope is adjusted, the calibration should then be tested on a new independent test set.

5.4.3 Expansion of calibration set

If the accuracy of the calibration is not meeting the expectations the calibration set should be expanded to include more samples or a new calibration be made. In all cases when a new calibration is developed on an expanded calibration set, the validation process should be repeated on a new validation set. If necessary, expansion of the calibration set should be repeated until acceptable results are obtained on a validation set.

5.5 Changes in measuring and instrument conditions

Unless additional calibration is performed, a local validation of a NIR method stating the accuracy of the method can generally not be considered valid if the test conditions are changed.

For example, calibrations developed for a certain population of samples may not be valid for samples outside this population, although the analyte concentration range is unchanged. A calibration developed on grass silages from one area may not give the same accuracy on silages from another area if the genetic, growing and processing parameters are different.

Changes in the sample presentation technique or the measuring conditions (e.g. temperature) not included in the calibration set may also influence the analytical results.

Calibrations developed on a certain instrument cannot always be transferred directly to an identical instrument operating under the same principle. It may be necessary to perform bias or slope /intercept adjustments to calibration equations. In many cases it will be necessary to standardize the two instruments against each other before calibration equations can be transferred [16]. Standardization procedures can be used to transfer calibrations between instruments of different types provided that samples are measured in the same way (reflectance, transmittance) and that the spectral region is common.

If the conditions are changed, a supplementary validation should be performed.

The calibrations should be checked whenever any major part of the instrument (optical system, detector) has been changed or repaired.

6 Statistics for performance measurement

6.1 General

The performances of a prediction model must be determined by a set of validation samples. This set consists of samples which are independent of the calibration set. In a plant, it will be new batches; in agriculture, it will be a new crop or a new experiment location.

This set of samples must be carefully analyzed following the reference methods. The care to analyze validation samples must be emphasized and the precision of these results is more important for the validation set than for the samples used at the calibration phase.

The number of validation samples must be at least 20 to compute the statistics with some confidence.

The NIR protocol used for the determination of the performances of the prediction model must be the same as that used in routine (one measurement or two measurements).