
**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*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs*.

This second edition cancels and replaces the first edition (ISO 12099:2010), which has been technically revised.

Introduction

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

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

1 Scope

This document gives guidelines 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 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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>

3.1

near infrared instrument

NIR instrument

apparatus which, when used under the conditions defined in this document, predicts *constituent contents* (3.3) and *technological parameters* (3.4) in *animal feeding stuffs* (3.2), cereals and milled cereal products through relationships to absorptions in the near infrared range

3.2

animal feeding stuffs

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

EXAMPLE Raw materials, fodder, meat and bone meal, mixed feed and other end products, pet food, etc.

3.3

constituent content

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

Note 1 to entry: The mass fraction is often expressed as a percentage.

Note 2 to entry: For examples of appropriate methods, see References [1] to [12].

EXAMPLE Moisture, fat, protein, crude fibre, neutral detergent fibre and acid detergent fibre.

3.4 technological parameter

property or functionality of *animal feeding stuffs* (3.2), cereals and milled cereal products that can be determined using the appropriate, standardized or validated method(s)

Note 1 to entry: 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 document is observed. The measuring units of the parameters determined follow the units used in the reference methods.

EXAMPLE Digestibility.

4 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 products concerned.

5 Apparatus

5.1 Near infrared instruments.

Instruments based on diffuse reflectance or transmittance measurement covering the near infrared wavelength region of 770 nm to 2 500 nm ($12\,900\text{ cm}^{-1}$ to $4\,000\text{ cm}^{-1}$) 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.

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

NOTE Changes in grinding or milling conditions can influence NIR measurements due, for example, to heating which can drive off volatile components such as water.

6 Calibration and initial validation

6.1 General

The instrument shall be calibrated before use. Calibration involves the comparison with a reference and adjustment processes to the instrument. 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 Reference [16] and the respective manufacturer's manual. For the validation, it is important to have a sufficient number of representative samples, covering variations such as the following:

- 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 (i.e. differences between instruments).

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

6.2 Reference methods

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

The reference method used for calibration should be in statistical control. It is essential to know the precision of the reference method.

Where possible, references that provide measurement traceability to the SI (International system of units), such as certified reference materials, should be used.

6.3 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 [Figures B.1](#) to [B.5](#) for examples.

For the purpose of validation, samples are not to be regarded as outliers if they fulfil 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, for example, estimated by Mahalanobis distance;
- c) if the spectral residual is below a limit defined by the calibration process;
- d) 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, e.g. by repeated measurements. 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.

6.4 Validation of calibration models

6.4.1 General

Before use, calibration equations shall be validated locally on an independent test set that is representative of the sample population to be analysed. For the determination of bias, slope and for the determination of standard error of prediction (SEP, see [7.5](#)), at least 20 samples are needed. Validation shall be carried out for each sample type, constituent/parameter, temperature and other factors known to affect or expected to have an effect the measurement. The calibration is valid only for the variations, i.e. sample types, range and temperature, used in the validation.

NOTE 1 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 SEP is calculated (see [7.5](#)) and the residual plot of data corrected for mean systematic error (bias) is examined for outliers, i.e. samples with a residual exceeding $\pm 3 s_{SEP}$.