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Interested committees:

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

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UK Vote

Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
- All comments should be submitted online at <https://standardsdevelopment.bsigroup.com/>. You will need to register in order to comment.

Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



DRAFT International Standard

Oilseeds — Application of near infrared spectrometry

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ISO/TC 34/SC 2

Secretariat: **AFNOR**

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2024-09-05

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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 of 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 www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 34/SC 2.

Introduction

This International Standard has been drafted using as a basis of ISO 12099:2017, prepared by the Technical Committee, ISO/TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs* and ISO 21543:2020, prepared by the Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products* and other reference material found in the Bibliography.

Oilseeds — Application of near infrared spectrometry

1 Scope

This International Standard gives a procedure for the prediction by near infrared spectroscopy of constituents such as moisture, fat and protein and some minor parameters such as total glucosinolates in oilseeds and oilseed meals.

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 terminology databases for use in standardization at the following addresses:

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

3.1

Near infrared (NIR) instrument

apparatus which, when used under specified conditions, predicts *constituent contents* (3.2) in oilseeds and oilseeds meals through relationships to absorptions in the near infrared range

3.2

constituent content

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

Note 1 to entry: Examples of constituents determined include moisture, fat, protein, total glucosinolate etc. For appropriate methods, see reference list (Bibliography) as example. The measuring units of the determined parameters have to follow the units used in the reference methods.

Note 2 to entry: It is possible to develop and validate NIR methods for other parameters than the one listed, as long as the procedure from this International Standard is observed. The reference method should be a validated method of known accuracy, repeatability and reproducibility. The measuring units of the parameters determined have to follow the units used in the reference methods.

4 Principle

NIR Spectroscopy is an indirect analytical technique, it requires developing prediction models (also known as calibration models) using samples of known composition determined by standard/reference wet-chemical methods. These prediction models are based on the correlations of NIR spectra of samples with their wet chemistry data. Once a prediction model is developed, quantitative predictions for the composition of a sample can be attempted from the sample NIR spectrum alone. The accuracy of prediction models shall be validated by predicting the composition of "unknown" samples using the NIR spectrometer for which the

prediction models were developed and comparing the predicted NIR results with the results obtained by the standard/reference wet-chemistry methods used for the prediction model development.

NOTE The term calibration model is well known and well understood by advanced NIR spectroscopy users. To outline that NIR spectroscopy is a secondary form of measurement, the term prediction model will be used in the document instead of the term calibration model.

5 Apparatus

5.1 Near-infrared instruments

Instrument using diffuse reflectance or transmittance measurement covering the NIR wavelength region, 780 nm to 2 500 nm ($25\,000\text{ cm}^{-1}$ to $4\,000\text{ cm}^{-1}$), or segments of this region or selected wavelengths or wavenumbers. Some instruments span a wider range of wavelengths (400 to 2 500 nm) and are able to measure in the visible range (400 to 800 nm). 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 or wavenumber accuracy and wavelength or wavenumber precision (for scanning spectrophotometers).

For NIR instruments measuring in transmittance, the sample pathlength (sample thickness) measurements should be optimized according to the manufacturer's recommendation with respect to signal intensity for obtaining linearity and maximum signal/noise ratio.

For NIR instruments measuring in reflectance, a quartz window or other appropriate material to eliminate drying effects might be used to cover the interacting sample surface layer.

For both instruments, sampling effects can be reduced by scanning a sufficiently large sample volume or surface to eliminate any significant influence by the non-homogeneity of chemical composition or physical properties of the test sample. The sample size effect can be determined through validation of constituent predictions using a representative set of samples of interest.

NOTE Some compounds need instruments with the visible range to be analyzed. Chlorophyll is an important parameter for canola seed quality. Chlorophyll content can only be predicted only by including wavelengths in the 650-700 nm range (visible range).

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

NOTE Changes in grinding or milling conditions will likely affect the NIR measurements.

When a model has been developed for a constituent for a ground sample with a specific particle size, it is recommended to use the same particle size for all the samples analyzed with the model or ensure the prediction model was developed using representative samples spanning the range of particle sizes intended to be measured and the validation of prediction model is conducted using representative samples spanning all particle sizes.

6 Calibration and initial validation

6.1 General

Prior to any analyses with the NIR spectrometer, ensure to run the system diagnostic and the manufacturer's daily check according to the manufacturer guidelines.

NIR spectroscopy allows for the prediction of compounds of interest by the development of statistical models acquired via a process called instrument calibration. A number of different calibration systems can be applied to different NIR instruments and therefore no specific procedure can be given for calibration. The raw spectra of the samples can be subjected to some mathematical pre-treatments before model

development to reduce the effect of some samples physical variations (e.g. sample presentation) on the scattering of the light.

EXAMPLE As an example, raw spectra can be processed first with a standard normal variate (SNV) to remove multiplicative interferences such as baseline shift. A Savitzky-Golay (SG) second derivative transformation can then be applied to ensure that: (1) peak positions are maintained at the same place as in the original spectra, (2) scattering effects are removed from the background, and (3) to improve spectral resolution and therefore assist in resolving overlapping peaks. In that case, the prediction models will be developed using the second derivative of the scan and not the raw spectra.

The NIR prediction model accuracy and robustness are strongly correlated with the samples chosen to develop the calibration, the method used to determine the constituent concentration and the methods chosen to develop the statistical prediction model.

The state of the sample varies as a function of the types of sample (e.g. intact seeds, grounds seeds, ground meals) and the instrument. It is very important to standardize the sample presentation for all samples throughout the entire process (calibration, validation and routine analysis).

6.2 Reference methods

Internationally accepted reference methods or well validated methods of known should be used for determination of the constituents of interest (e.g. moisture, fat, protein, glucosinolate and other) by wet-chemistry. Oilseeds reference methods are given in the reference list as example.

The reference/validated methods 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 (e.g. accuracy, repeatability and reproducibility).

NOTE Precision, repeatability and reproducibility of the reference method will influence the NIR method robustness (see reference).

6.3 Calibration

To build robust prediction models it is recommended to:

- a) develop models with calibration samples that will be truly representative of the samples to be predicted (concentration and state). The distribution of the constituents to be predicted should be as uniform as possible over the concentration range to be predicted. If the reference data of the constituents of interest are known before starting the process, the range values should be included in the factorial design across all components;
- b) duplicate samples shall not be used to develop the calibration model. Average, standard deviation, median and range, plus the number of samples shall always be reported for the calibration sample set;
- c) develop models using the least possible principle components and wavelengths when developing the model as overfitting will diminish the robustness of the model;
- d) avoid the use of absorbance data out of the linear range of the detector;
- e) use processing methods to smooth, correct, enhance and scale raw spectra prior to model development;
- f) use calibration samples that present seasonal, geographic, genetic, storage and processing variations as the samples to be predicted will present the same variations

It is also known that sample temperature and instrument temperature might cause shifts in the NIR spectra. Ensure model is developed and validated spanning the intended sample and instrument temperature range.

It is also known that sample age might affect the NIR scan of the sample.

NOTE The problem of developing models with sample sets that have an uneven distribution of the constituent concentration is that the model will tend to fit closely to the majority of the samples in the calibration.