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NIRS Standard ISO 12099 – setting new performance standards for feed calibrations?

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ANALYTICS BEYOND MEASURE

NIRS Standard ISO 12099 – setting new performance standards for feed calibrations?

Today, some 50 million analyses are performed annually using indirect spectroscopic methods like NIRs, but until a few years ago, no applicable international standards existed to form a basis for the communication of NIRs results, and to allow users of NIR spectrometry to gain accreditation. This is why the ISO 12099 project, "Guidelines for the application of near infrared spectroscopy", was launched. The standard was developed during 2007 – 2010 within the European Standardisation Organisation CEN, Technical Committee TC 327 (Animal Feed) in liaison with CEN TC 338 (Cereals and Cereal Products) and ISO TC 34 SC 10 (Animal Feed), and the final standard was published in 2010 (1).

The project involved more than 20 NIRs specialists and users. Stakeholders came from science & research; control laboratories (users of NIR); industry (including main suppliers of NIR solutions), and standardisation bodies.

This International Standard provides guidelines for determination by near infrared spectroscopy of constituents such as moisture, fat, protein, starch and crude fibre, as well as parameters such as the digestibility in animal feeding stuffs, cereals and milled cereal products. It should be noted that NIR methods can be developed and validated for parameters and sample types other than those listed above, provided that the procedure from this standard is observed. The measuring units of the parameters determined must follow the units utilised in the reference methods.

The standard allows any kind of instruments based on diffuse reflectance or transmittance measurement covering the near infrared wavelength region of 770–2500 nm or segments of this. 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).

ISO12099 is a general standard that focuses on the validation of calibration models with independent test sets. This standard forms the basis for quality systems or accreditation schemes when using NIR. Regarding the development of calibration, reference is made to a textbook (3).

Regarding the validation, it is important to have enough 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)

The standard states that at least 20 samples are required for a solid validation, and that a calibration is only valid for the variations, i.e. sample types, ranges and temperatures, used in the validation.

To form a visual impression of calibration performance, results obtained via the independent test set are plotted and referenced against NIR, while residuals are checked against reference results. The standard error of prediction (SEP)) is calculated, and the residual plot of data corrected for mean systematic error (bias) is examined for outliers, i.e. samples with a residual exceeding three times the standard deviation of the SEP.

1 – 45° line (ideal line with bias = 0 and slope = 1),
2 – (45° – bias) line,
3 – linear regression line
4 – outliers (x and y)

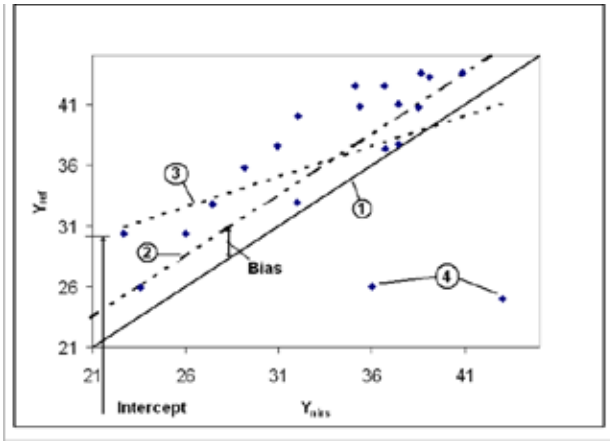

Figure 1: Plot of reference results versus predicted results, indicating bias and intercept.

If the validation process reveals that the model cannot produce acceptable statistics, it should not be used. Instead, corrections and adjustments - including a new calibration with an expanded calibration set - should be made.

The standard also has a revised and updated section on statistics for performance measurement, defining bias and slope/intercept, root mean square error of prediction (RMSEP), and standard error of prediction (SEP), including expressions for different confidence limits. Further outliers are explained together with methods of handling them.

Of interest for the routine user is that the standard also covers the standardisation of instruments in a network and the validation of a calibration on local samples before use. Instrument stability should be checked using a control sample, and instrument diagnostics performed on regular basis. A performance check should also be made of calibrations during use, using control charts. The standard provides detailed information on this.

Finally, the standard includes a comprehensive glossary with an explanation of terms and definitions. This has proved to be very valuable, as terms are sometimes applied differently within the same company.

Acknowledging the necessity for specific NIR standards - i.e. precise calibrations for the determination of specific constituents and parameters in animal feeding stuff, cereals and milled cereal products - the guideline developers have covered these standards in an informative Appendix.

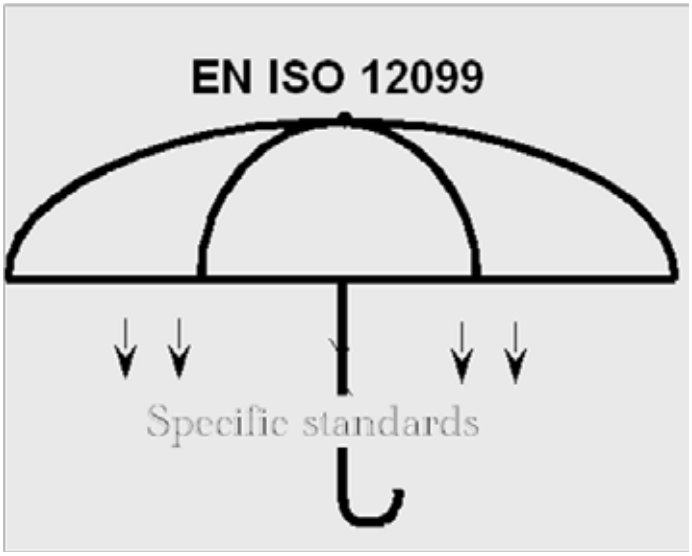

Figure2: ISO 12099 as a general standard covering several specific standards

Conclusions
The 12099 standard is an important cornerstone for the development and application of NIR solutions.

A community of experts and users has removed inconsistencies, agreed on how on to interpret the various terms, and provided guidelines for the validation of NIR solutions.

These guidelines are expected to lead to new performance benchmarks for feed calibrations, particularly when used in combination with a proficiency testing scheme like the one from the Association of American Feed Control Officials (2).

References:

1. ISO 12099:2010 – Animal feeding stuffs, cereals and milled cereal products Guidelines for the application of near infrared spectrometry, http://www.iso.org

2. AAFCO, Association of American Feed Control Officials, Check Sample Program, www.aafco.org

3. Næs, T., Isaksson, T., Fearn, T., Davies, T. : A user-friendly guide to multivariate calibration and classification. NIR Publications, Chichester (2002).