

**FOSS**

# Best practice for calibration of NIR instruments with global models

May 2018  
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**ANALYTICS BEYOND MEASURE**

# FOSS best practice for calibration of your NIR instruments with global models

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When implementing Near Infrared (NIR) spectroscopy in your feed operations it is essential to have robust and comprehensive calibrations that can accommodate the natural variations in raw materials across seasons and suppliers. That said, the process of building these comprehensive calibrations yourself can take many years of development time to acquire the necessary reference data sets.

In contrast, ready-made calibrations provide a much faster alternative.

In some cases, you can simply install them and start using them directly. In others, it will be necessary to do some minor, so-called, bias adjustments according to your raw materials and sample-types. Even so, the calibration will get you a long way down the road and, using the latest in networking software, the bias adjustments can be implemented remotely, literally at the click of a mouse button.

In terms of the time factor at least, ready-made calibrations appear an attractive option. But is it still worth paying for them instead of doing your own reference tests? And, once you have them, how do you validate them and actually get them up and running on your instruments?

Let's take a closer look at the true value of ready-made calibrations and what it takes to use them.

# CONTENTS

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1. Teaming-up with ANN and network support – a typical scenario
2. A case story from the field
3. Reliable validation according to ISO standards
4. Six steps to an effective feed calibration implementation with online technical support

# Team-up with ANN and network support

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The FOSS global ANN feed calibrations are ready-made calibration for your feed NIR instrument. The calibrations cover a wide range of feed and feed ingredients. They are based on over 50.000 spectra data sets which are referenced against more than 300.000 wet chemistry values and thus offer a huge value in terms of readily available reference data.



Advantages include:

- Less sampling and reference analysis cost – now reference analysis is only required for checking the calibration
- Flexible sourcing – now you can switch supplier with ease because potential variations in raw material are already included in the comprehensive ANN data set.
- Faster operations - with a single calibration covering a number of different sample types, the operator does not need to keep changing the sample type

Once you have decided on the ANN calibrations you need, the next step is to use the services of a trained application specialist who has access to the instrument and data via secure networking software.

# ANN calibrations

- An Artificial Neural Network (ANN) is a calibration model which, in principle, is based on the neural structure of the human brain.
- FOSS has developed ANN calibration models since the early 1990's
- Calibrations with very large datasets can cover seasonal variations, geographical variation as well as different breeds/varieties. For example the FOSS vegetable by-product calibration includes data over 6700 samples
- The database used for the FOSS NIRS DS2500 analyser calibrations is based on over 50.000 NIR tests referenced against over 300.000 wet chemistry values.



## CHAPTER 2

# ANN plus network support – a typical scenario

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The following scenario illustrates the value of the readymade option combined with relevant support.

## The instrument only approach

A laboratory manager at a feed company has decided that an NIR analyser is required to help with her daily responsibilities. These include support analysis of animal feed (poultry, pig, cattle, rabbit) as well as raw materials used for production of these feeds such as wheat, triticale, maize, soybean meal, distillers grain and rapeseed meal.

She gladly unpacks her new NIR instrument, but before she can use it, she needs to install some calibrations.

She starts on some calibration development work, but quite soon, it is apparent that it is going to take a long time. The equipment vendor tries to help, but lacks knowledge about the feed application and the type of samples. The manager ends up doing a lot of explaining with little practical help in return.

A particular challenge is the number of wet chemistry tests involved in developing calibrations from scratch. It takes a lot of time to run sets of tests for fat or fibre and so on and because she is constantly trying to add data to the calibration, it becomes a battle against time to bring the calibration up to date. Meanwhile normal day-to-day operations continue relentlessly.

Eventually, she takes a time out and decides to try moving over to another NIR solution which is network-enabled and supplied with readymade calibrations.

## **The networked- enabled instrument and readymade calibrations approach**

The new solution is supplied with calibrations for testing plant based feed ingredients, compound feed, and wet silage (maize, lucern, and grass) and they prove an instant time saver. It is still necessary to make few minor adjustments to some of the ready-made calibrations, but these are within reach, specifically she needs to make a few tests to support a bias adjustment using triplicate wet chemistry tests run on the same sample with the average used to compare to the NIR. The bias is then set with the help of an application specialist working online from a remote location, thus avoiding travelling and waiting time.

The application support specialist then helps to validate the calibrations, once again, working from his desktop to access the required datasets.

Finally, the bias-adjusted and validated calibrations are uploaded to the instrument via the networking facilities. Once everything is in place, the specialist travels to the site to oversee the start-up of the instrument giving tips and on the spot training to users.

## Reliable calibrations constantly improved with network enabled updates

The application support specialist can keep an eye on how everything is performing via the network and make some ongoing adjustments that are occasionally required where ingredients can fluctuate a lot. This could be, for example, due to inconsistency across distiller's grains suppliers which might demand bias adjustments for each vendor. Any such adjustments are pushed out via the networking software. It takes a matter of minutes to synchronise with the instrument and then the operators can start using it.



## **Achieving the NIR advantage**

The laboratory manager can now make good on her original decision to invest in NIR. There is no more time spent on calibration development and the laboratory can support the feed mill better through more rapid control of basic parameters in products, and, in case of specification non-compliance, not to release them for shipment.

This scenario might not fit every case, but the details are drawn from real cases in the feed industry. Clearly there is a business case for making a good start with readymade calibrations and then taking advantage of network-enabled support for local adjustment and performance monitoring over time.

## CHAPTER 3

# Validate performance according to ISO before you start

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Before you start using readymade calibrations, you need to ensure that the calibrations match your specific selection of raw materials and finished product. Your technical support contact can do this for you, see next section.

You will need to validate the calibrations on samples from your own production.

Validation ensures:

1. That you can feel confident about results - An independent review of the validation confirming the work done by the supplier of the calibration for your specific samples.
2. Documentation for the validity of the calibrations to document performance to your suppliers and customers

## **Best practice in NIR management defined by ISO**

As part of the global initiative to improve the use of NIR in Feed production. The ISO Technical Committee ISO/TC 34, Food products, subcommittee SC 10, animal feeding stuffs has prepared a guideline on how to use NIR in Feed. The guideline was first prepared in 2010 and was updated in August 2017.

The guideline offers the global best practice on validating calibrations for feed. What does ISO 12099 stipulate Validation of calibration models?

*Calibration and initial validation:*

*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), 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 on the measurement. The calibration is valid on for the variations, i.e. sample types, range and temperature used in the validation.*

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 materials and cereals
- c) Processing techniques and conditions
- d) Storage conditions
- e) Sample and instrument temperature
- f) Instrument variations (i.e. differences between instruments)

Based on the requirements in the ISO standard, the initial validation seems like a major endeavor. To get a better perspective on what is actually involved, we have turned to an expert on the subject.

## CHAPTER 4

# Six steps to an effective feed calibration implementation with online technical support

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Maciej Socjusz is an application specialist at FOSS and has worked with calibration of NIR instruments for feed applications for many years.

Here are his six steps to a fast and successful implementation.

- 1) Start by ensuring online access to measuring results and reference data

Ensure that the instrument can be connected to networking facilities, for example FossConnect (link). This provides access to and from a support person who can work quickly and without disruption to daily operations.

- 2) Make the validation before starting to use the NIR instrument

It may sound obvious, but if overlooked, it will delay the implementation. Being able to see the full dataset acquired during the validation allows the specialist to make relevant statistical analysis to decide, for example, what requires a bias adjustment and what are outliers and mistakes.

- 3) Get off to a good start with timely advice

It is important that the application specialist conducts user-training during or immediately after installation. Well-trained users can follow approved standards and procedures from the start and will not do other tests and comparisons which might be very hard to evaluate or refer to. This provides a solid foundation for effective collaboration with FOSS service.

- 4) Focus your sampling on the critical raw materials and parameters

According to ISO, a minimum of 20 samples must be used to validate a calibration. If the calibration includes multiple sample types (like all the vegetal ingredients) and up to six parameters, 20 samples will only cover a few sample types. It is therefore recommended to start with the most critical raw materials and parameters. Equally, it is important that the samples cover the variation of the parameters.

- 5) Use the instrument feedback to select samples for reference analysis

When using the calibrations, the FOSS instrument will warn you of outliers when there is an uncertainty about the validity of the results. Being an outlier means that the sample type is not represented in the calibration or it is simply a wrong sample. If it is not represented in the calibration. The relevant samples should be measured by reference chemistry to test if the NIR results are correct. If there is a sufficient number of outliers, FOSS will create hotfix of the calibration. If the validation shows issues for individual samples or parameters, the global calibrations may need to be adjusted based on the samples.

- 6) Continue to do random sample checks during steady-state operations using network facilities

After the initial adjustment, the Calibration will be stable and it only needs to be validated by a random sample set once a year. Once again, remote access is a fantastic tool to help the application specialist to differentiate between instrument, operator or calibration issues. Likewise, uploading and reformatting data is performed quickly as a matter of routine without interrupting the work of operators. The regular check ensures that the instrument is always up to date, so should an actual problem occur, it is then easier and quicker to identify and fix it instead of starting with a retrospective analysis of performance.

