Importance of reference analyses for the development and validation of NIR prediction models*

As NIR calibrations ´inherit the error' of the reference methods used, NIR predictions are totally dependent on the accuracy of reference methods

The accuracy and precision of the reference methods are essential for development, maintenance, update and validation of calibrations. They are crucial to the success of NIR calibrations and crucial for the cost of any NIR solution.

Furthermore, knowledge about the methods used for validation is unavoidable in discussions about the performance of instruments and applications. Let's take some examples. Figure 1 shows results for the determination of moisture in different mixed feed samples from the Association of American Feed Control Officials (AAFCO) proficiency testing scheme. There are obviously differences between the different methods applied, differences that could not be accounted for by simple bias adjustments. You must know what reference method the calibration is based on and, even more importantly, what method the prediction model has been validated against and what method you compare the predicted results with.

Neutral Detergent Fiber is an increasingly important parameter for the prediction of feed values. Figure 2 shows results from a ring test with two layer feed samples, in the form of a Youden diagram. Twelve official feed control laboratories participated, applying eight different method modifications. Ideally, results should distribute randomly in a circle defined by the measurement uncertainty. If results in a Youden diagram line up as in fig. 2, it is a strong indication of methodological differences.

The results also show that it is difficult to develop NIR/NIT calibrations for a poorly defined parameter. In addition, the figure illustrates the risks involved when basing a calibration only on results of a single laboratory.

Single laboratory calibrations do often result in good or at least reasonable performance data for a calibration. Figure 3 gives an example for the determination of amylose in rice[1]. A good performance in terms of regression coefficient Rsq, standard error of prediction SEP and low bias were obtained.

However, it must be noted that the reference data were results of one single laboratory. In practice, for example in international trade, several laboratories are often involved in the analysis of the samples/commodities. Figure 4 gives an example for the determination of amylose in rice performed by 44 rice research in-

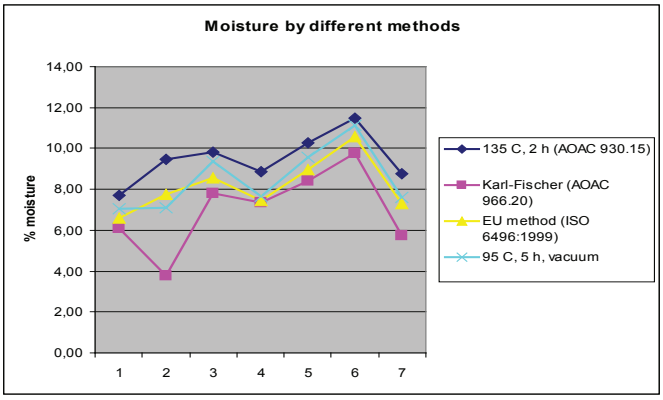

Figure 1: Moisture determination in mixed feed samples (AAFCO PTS)

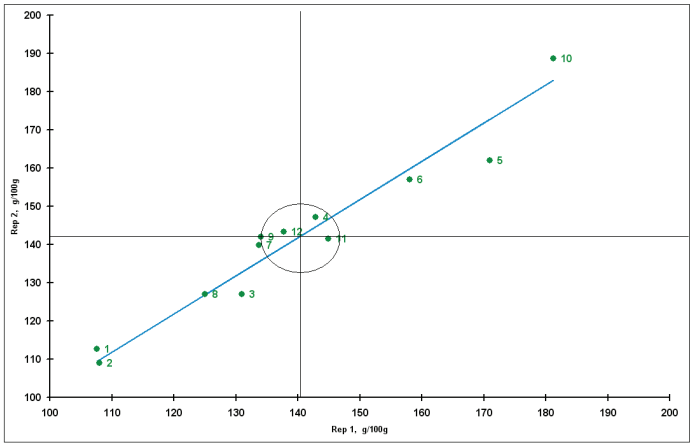

Figure 2: Results of a ring trial for the parameter Neutral Detergent Fiber (NDF)

stitutes and quality control laboratories in different parts of the world, analyzing 17 different varieties [2]. For sample 2 values between 16% and 42% were reported and for sample 17 (a waxy rice sample) -2% to 10%.

Laboratory clients often have to comply with specifications or have to know whether a certain food is suitable for consumption. They may, for example, demand an accuracy for oil content of less than 0.5%, meaning most probably that the deviation between the predicted value and the reference value shall not be larger than 0.5%. This implies that both client and laboratory are aware of the fact that the reproducibility of the method has to be less than 0.5% (otherwise the probability that predicted value and reference value will agree which each other will be lower) and that both know and agree on method and method performance data.

Conclusions

The reference method and its performance must be known and documented, not only when making/updating a calibration model, but also when comparing the results obtained between different laboratories or instruments.

Information about the measurement uncertainty, for example, by performance data of the method used (trueness/bias, repeatability/reproducibility), has to be available when making comparisons of results between methods, instruments and laboratories.

FOSS is focusing on global, reusable calibrations, involving data from several laboratories (up to several hundred), with tight control of reference methods used and quality control schemes applied. If needed, new global standard methods are also established.

As a result, dozens of new or improved standards for reference methods, often on basis of FOSS solutions for chemical analysis, have been established. In addition, new NIR standards have been published or are in the pipeline:

AOAC Official Method 2007.04 Simultaneous Determination of Fat, Moisture, and Protein in Meat and Meat Products using the FOSS FoodScan™ Near-Infrared (NIR) Spectrophotometer with FOSS Artificial Neural Network (ANN) Calibration Model.

EN ISO 12099:2009 Guidelines for the application of near infrared spectroscopy in agricultural analysis.

prEN 15948: ANN calibration for the determination of protein and moisture in wheat and barley by NIT.

** on basis of a presentation given at the ACHEMA 2009 congress*

References:

[1] Delwiche et al., Cereal Chem. 72(2):182-187, 1995

[2] Fitzgerald et al, Cereal Chem. 86(5):492-498, 2009

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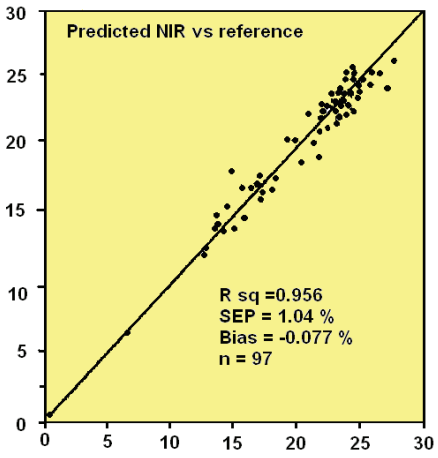

Figure 3: Predicted vs reference results for the determination of amylose in rice

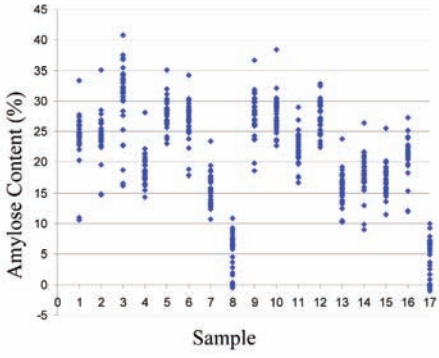

Figure 4: Results of a ring trial for the determination of the amylose content in rice