Transfer of calibration between on-farm whole grain analysers

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Introduction

On-farm near infrared (NIR) analysers are becoming more accepted around the world. The benefits offered to growers to measure the quality of their wheat, barley, canola, sorghum, soy bean, corn and other crops leads us to believe that on-farm NIR analysers will become a standard piece of farming equipment in the next 20 years.

With such a large potential number of instruments placed in remote locations, the problems of calibration and calibration transfer become very significant. To this end, NIR Technology Australia has been very conscious of the need to make NIR analysers with the ability to transfer calibrations from master to slave instruments.

Calibrations based on partial least squares (PLS) techniques may make this task even more complex since PLS calibrations tend to compensate for instrument and sampling noise. Since these two parameters are commonly instrument-dependent, then a PLS calibration developed on a master instrument may not transfer easily to all slave instruments. Nonetheless, PLS calibrations have proven very successful for the analysis of whole grains and, therefore, a transfer method had to be developed which would allow PLS calibrations to be transferred across a network of instruments.

Procedure

The first step in calibration transfer should always be the standardisation and normalisation of the slave instruments to the master instrument. There are two parameters to be considered, (a) wavelength alignment and (b) photometric response.

Wavelength alignment

The NIR Technology Australia Cropscan 2000G and 2000B Whole Grain Analysers are diode array-based spectrophotometers. They use a flat field spectrograph to project an image of the entrance slit onto a large silicon photodiode array detector. The flat field spectrograph is a concave holographic grating with a spectral dispersion from 720 to 1100 nm. To align the wavelengths from master to slave instruments requires the adjustment of the angle of the grating relative to the incident beam of light. By scanning a sample of wheat grains and using the moisture absorption peak at 967 nm, it is possible to align the gratings between instruments. However to correctly align the gratings, it has been found that a first derivative spectra of wheat grains provides several points to match the spectra and is far more sensitive than the absorbance spectra.

Photometric response

Even though the gratings are produced as identical masters, there is always a mechanical tolerance which effects the efficiency curve of the gratings. As well, the lamp, lenses, detector and sampling compartment have mechanical tolerances which result in differences between the photometric re-

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sponse from instrument to instrument. To correct for the total variance which exists between master and slave instruments, we scan samples on the master and the slave instruments and compare the spectral data. By scanning samples of wheat with absorbances covering the broadest range, i.e. two to four absorbance units, we can look at the presence of skew and shift at each wavelength. Simple linear regression techniques can then be used to estimate the required correction to each wavelength reading in order to make the slave instrument's response the same as the master instrument. We have found that either a slope and bias or simply a slope correction of the photometric output of the pixels on the diode array detector, is sufficient to match instruments.

The Cropscan 2000G and 2000B can use either of these methods. Within the instruments set up files, there is a look-up table which contains the S and B factors (slope and bias). When a sample of grain is scanned, the reading from each pixel is multiplied by the S factor and the B factor is added to give the corrected output. The PLS calibration is then applied to the corrected spectrum to compute the protein and moisture results. Other methods, such as multiplicative scatter correction (MSC), have been tried but showed poorer results.

Analysis

A study was undertaken to prove the viability of this procedure. The master calibration was developed over three years using 425 samples of Australian hard wheats. All samples were scanned on the one instrument, not at one time, but progressively. This calibration set includes as much variation in variety, growing conditions and region as was available. Five samples were scanned at 10°C, 25°C and 45°C and added to the set for sample temperature stabilisation. As well, samples were scanned when the instrument was at 10°C and 45°C and added to the calibration set for instrument temperature stabilisation. Five scans of each sample were collected and used in the PLS calibration. By using the un-averaged scans, we feel that it builds in a tolerance to the sample packing variation. Even though using the five scans increases the standard error of calibration, it reduces the standard error of prediction and provides a more robust calibration. It is also felt that the use of the five scans makes the calibration less instrument-dependent and, therefore, more easily transferred to another instrument. The calibration statistics for this calibration are listed below:

Number of scans:	2125			
Number of PC:	Protein:	12	Moisture:	9
SEC:		0.33%		0.26%

This calibration for hard wheat is used in all instruments sold in Australia and is provided in instruments sold overseas, although local calibrations have been developed in Europe and the USA. To match the instruments to a specific laboratory, each instrument has slope and bias adjustments for each calibration. Generally, five or six samples of wheat from 10% to 15% protein content are used to compute a simple linear slope and bias adjustment. This slope and bias adjustment corrects the final protein and moisture results but does not change the B coefficients of the master calibration nor the photometric response of the instruments.

Five Cropscan 2000G and two Cropscan 2000B instruments were set up using the above procedure for wavelength alignment and photometric response. A set of nine hard wheat samples were then scanned on each instrument. Table 1. shows the prediction data from the seven instruments.

Table 1. Comparison of five Cropscan 2000G and two Cropscan 2000B Whole Grain Analysers against the Master Cropscan 2000G. Wheat(2000) calibration was used on all instruments. Nine samples of Australian hard wheats were scanned on each instrument including the Master instrument.

ID	Prot%	Master Diff.		Cr104 Diff.		Cr105 Diff.		Cr106 Diff.		Cr107 Diff.		Cr 108 Diff.		Cr109BDiff.		CrIOB Diff.		Average St dev.		Ref – Av
1	13	13.5	–0.5	13.1	–0.1	13.6	–0.6	13.3	–0.3	13.4	–0.4	13.6	–0.6	13.5	–1	13.7	–0.7	13.5	0.19	–0.5
2	14	14,1	–0.1	13.8	0.2	13.9	0.1	14	0	14	0	14	0	14.2	0	14	0	14	0.12	0
3	12.2	12.4	–0.2	12.6	–0.4	12.2	0	12.4	–0.2	11.9	0.3	12.2	0	12.1	0.1	12	0.2	12.1	0.25	0.1
4	10.4	10,1	0.3	10	0.4	9.8	0.1	9.7	–0.2	9.6	0.3	9.7	0.2	9.8	0.1	9.8	0.1	9.9	0.21	0.5
5	13	12.8	0.2	12.7	0.3	12.8	0.2	12.9	0.1	12.7	0.3	12.8	0.2	12.9	0.1	12.4	0.6	12.8	0.16	0.2
6	11.2	11.7	–0.5	11.6	–0.4	11.3	–0.1	11.3	–0.1	11.6	–0.4	11.5	–0.3	11.3	0	11.4	–0.2	11.5	0.19	–0.3
7	11.9	11.7	0.2	11.6	0.3	12.1	–0.2	11.4	0.5	11,9	0	12.2	-0.3	12.1	0	11.8	0.1	11.8	0.27	0.1
8	12.7	119	–0.2	12.7	0	12.5	0.2	12.4	0.3	12.4	0.3	12.5	0.2	12.4	0.3	12.3	0.4	12.4	0.13	0.3
9	9.2	9.7	–0.5	9.4	–0.2	9.1	–0.1	9.7	–0.5	9.1	–0.1	9.2	0	9.3	0	9.6	-0.4	9.5	0.29	–0.3
SEP		0.34		0.31		0.25		0.32		0.28		0.28		0.2		0.41		0.32		

Conclusion

It can be seen that Sample 1 is consistently predicting higher than the reference value. However the remaining samples are predicted well. Nonetheless, all seven instruments predict to the level expected of whole grain analysis.

It should be noted that Cr109 and Cr110 are 2000B models which use a mechanically-driven sample cell with a pathlength of 18 mm. The other five instruments use a manually-loaded cell with a 20 mm pathlength. This illustrates the ability of the above procedure to correct for even 10% variation in effective pathlength.

There are several algorithms for transferring calibrations between instruments sighted in the literature, but the above study shows that a simple linear slope and bias correction can be effective in normalising instruments and thus allowing a single master calibration to be used on all instruments.