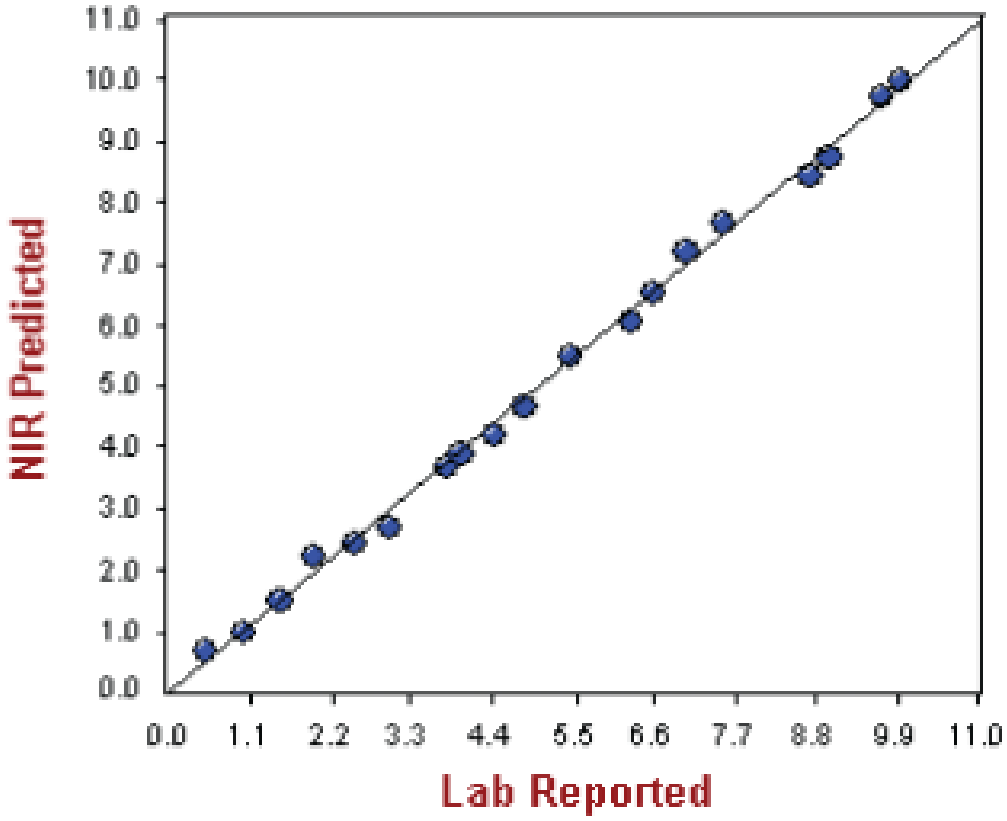
Near-Infrared Spectroscopy Application Note NIR–11

Calibration model transfer of caffeine on the NIRS XDS Rapid Content Analyzer



This Application Note demonstrates that a calibration model of a caffeine and microcrystalline cellulose mixture developed on one NIRS XDS Rapid Content Analyzer (RCA) is transferable to more other NIRS XDS RCAs without any adaptation. Due to the improved signal-to-noise ratio, reduced bandwidth and improved wavelength precision of the NIRS XDS, the transferability of the calibration model can be easily and efficiently performed.

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Method description

Introduction

Many manufacturers own more than one NIR instrument with the same monochromator/sample module configuration. It is often desirable to be able to transfer a calibration model developed on one instrument to one or more other instruments that are of like configuration.

The NIRS XDS Rapid Content Analyzer (RCA) with Vision Software™ makes this possible with ease and confidence in the results. The improved signal-to-noise ratio (low instrument noise), reduced bandwidth and improved wavelength precision of the XDS make model transfer routine.

In this Application Note the transferability of a simple two-component sample matrix of caffeine in microcrystalline cellulose, along with a simple calibration model was developed using multiple linear regression (MLR).

Experimental

Four units of the NIRS XDS RCA near infrared spectrophotometer were used in this experiment. Twenty samples of a mixture of caffeine and microcrystalline cellulose were packed into quartz cups for analysis and analyzed in the reflectance mode. The caffeine content of the samples ranged from 0.493% to 9.925%.

Results

Figure 1 shows the raw spectrum of pure caffeine overlaid with the raw spectrum of microcrystalline cellulose. Figure 2a shows the second derivative of pure caffeine overlaid with microcrystalline cellulose. Figure 2b shows the expanded view of the analytical region for caffeine. The second derivative math pretreatment corrects bias offset, enhances peaks and provides smoothing of the raw spectra. The absorbance peaks appear inverted in the second derivative. Figure 3 and Figure 4 are the raw and second derivative spectra, respectively, of the mixture of caffeine in microcrystalline cellulose.

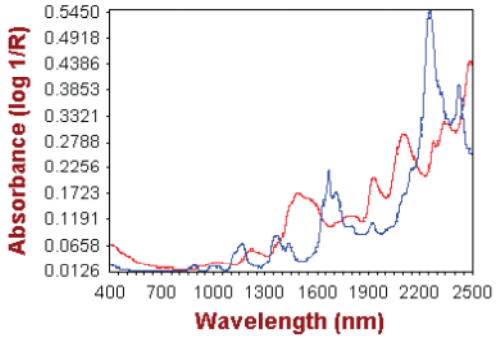

Fig. 1: Absorbance spectra of caffeine and microcrystalline cellulose.

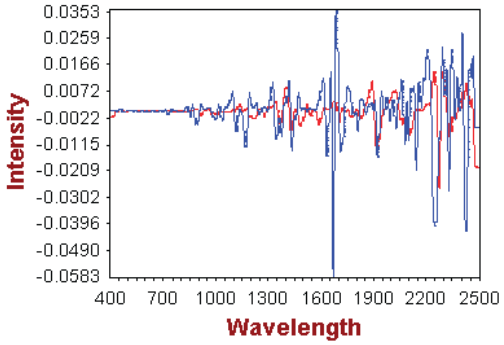

Fig. 2a: Second derivative spectra of caffeine and microcrystalline cellulose.

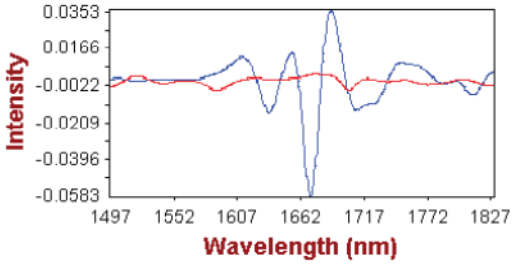

Fig. 2b: Expanded caffeine absorbance around 1670 nm.

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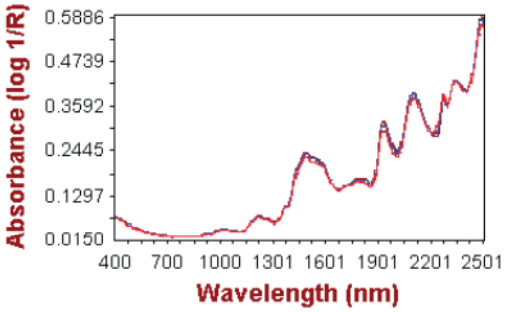

Fig. 3: Absorbance spectra of varying levels of caffeine in microcrystalline cellulose.

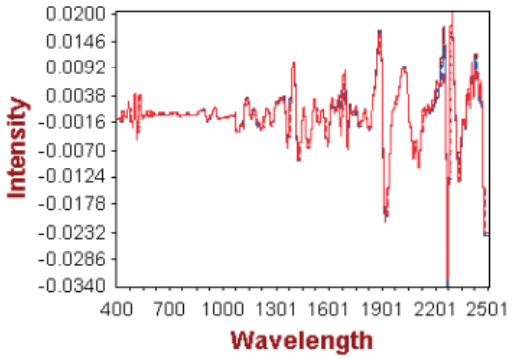

Fig. 4: Second derivative spectra of varying levels of caffeine in microcrystalline cellulose.

To test calibration transfer, a calibration model was developed for caffeine by scanning 20 samples with the range of concentrations noted above on the instrument labeled "A". A single wavelength multiple linear regression (MLR) model for caffeine concentration was developed using the absorbance at 1670 nm.

Figure 5 is an expanded view of the analytical region around 1670 nm for low, medium and high values. The standard error of calibration (SEC) from the calibration set was compared to the standard error of prediction (SEP) of the prediction sets from the other two instruments. For calibration transfer to be successful the SEC and SEP should be of comparable magnitude and there should be no bias offset or slope error in the prediction results. The coefficient of determination (R^2) of this model was 0.997 and the SEC was 0.17%. Figure 6 is a scatter plot of the NIR prediction of the calibration set versus the reported laboratory values for each sample. Figure 7 shows the scatter plot of the NIR prediction of the validation set versus the reported laboratory values. This model was then used to predict the caffeine concentration of sample spectra collected on the other three analyzers (labeled "B", "C" and "D").

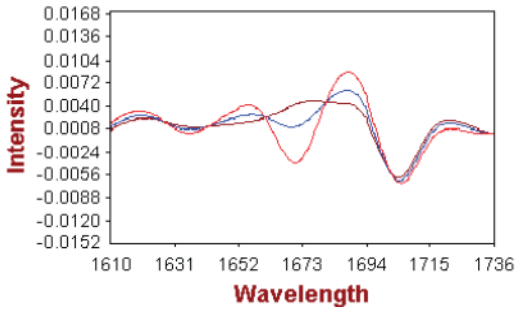

Fig. 5: Expanded view of analytical region for caffeine.

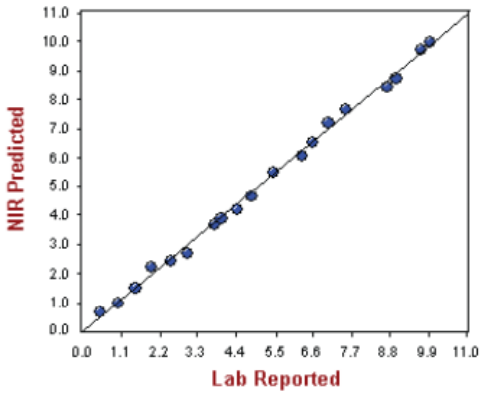

Fig. 6: Scatter plot of NIR calibration set vs. laboratory reported values (calibration unit "A", SEC: 0.17%).

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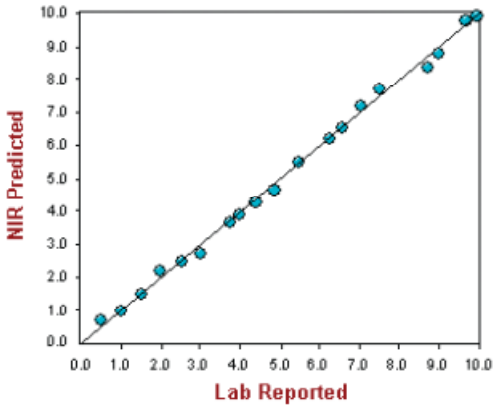

Fig. 7: Scatter plot of NIR validation set vs. laboratory reported values (RCA unit "B", SEC: 0.16%).

To test the effect of source lamp change on the model transfer, the source lamp was changed three times on unit "D" and validation samples were predicted after each change. The MLR calibration model developed on the first instrument "A" had an SEC of 0.17%. The SEP on the three instruments to which the prediction models were transferred were 0.16% on "B" and 0.17% on "C". Instrument "D" initially had an SEP of 0.17%. Source lamp changes had little or no effect on the SEP on subsequent predictions after lamp changes. The data are listed below in Table 1.

Table 1:

Calibration Unit "A"	SEC:017%	
XDS RCA Unit	SEP	Bias or Slope Error
B	0.16%	None
C	0.17%	None
D, lamp 1	0.17%	None
D, lamp 2	0.17%	None
D, lamp 3	0.17%	None
D, lamp 4	0.16%	None

Conclusions

Calibration model transfer of a mixture of caffeine in microcrystalline cellulose can be successfully accomplished on the NIRS XDS RCA. The model was developed on one instrument and transferred to three other instruments, one of which had the source lamp changed three times. There was no resultant bias or slope error from the transfer and the differences in SEP were negligible.

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