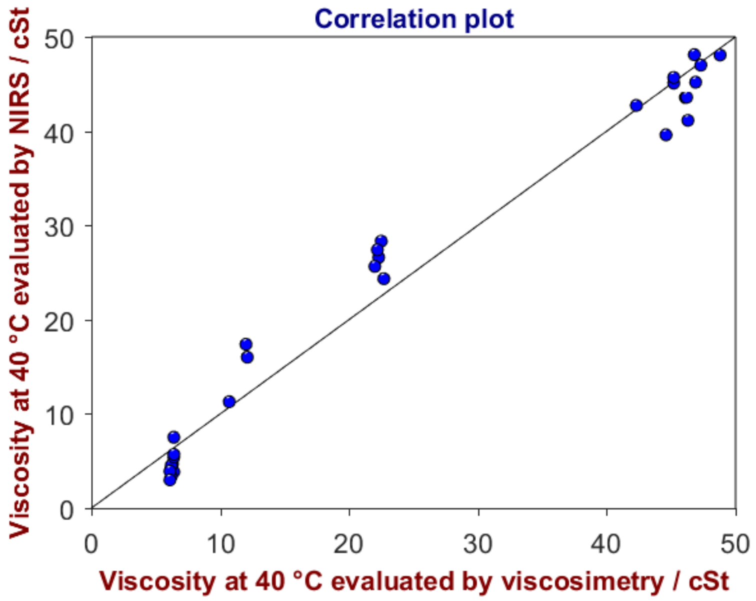
Near-Infrared Spectroscopy Application Note NIR–43

Analytical data transfer between a Fourier transform and a dispersive NIR instrument



This Application Note demonstrates the data transfer from a Fourier transform to a dispersive NIR instrument, using quality control of lubricating oils as an example application. It is shown that FT-NIR instruments can be replaced by dispersive ones without time-consuming sample remeasurement and subsequent method development.

Metrohm NIRSystems

Method description

Introduction

When replacing an old NIR spectrometer with a new one, the simplest case is a replacement with an instrument from the same supplier, typically having similar hardware characteristics. However NIR users may decide to switch to an instrument with completely different hardware from a different manufacturer. Reasons for such a change range from insufficient performance of the old hardware to dissatisfaction with the support and service of the former supplier. Quite often, this instrument replacement is accompanied by the customer’s wish to transfer the data from the old analyzer to the new one to avoid the cumbersome and time-consuming method development from scratch.

This wish is in conflict with the hypothesis from the last millennium that data transfer is too complex. As a result, users believe that they are forced to stay with the same NIR supplier and instrument type, despite their dissatisfaction. However, due to technological developments, data transfer between completely different types of analyzers can nowadays be accomplished. In such cases, data and method transfer are slightly more complex than between similar instruments, yet the local Metrohm agency can provide support. The additional steps required are described in this application note, which showcases successful data transfer of multiple quality parameters of lubricating oils from an FT-NIR instrument to a dispersive NIR instrument.

Configuration

The original calibration samples were measured on an FT-NIR analyzer (hereinafter referred as source instrument) with a spectral range of 12500–4000 cm^{-1} (800–2500 nm), a spectral resolution of 16 cm^{-1}, and 32 scans per spectrum. The calibration set consists of 100 samples analyzed according to different ASTM norms (D664 for acid number, D445 for kinematic viscosity at 40 °C, and D6304 for moisture analysis). FT-NIR spectra of the calibration samples are shown in Fig. 1.

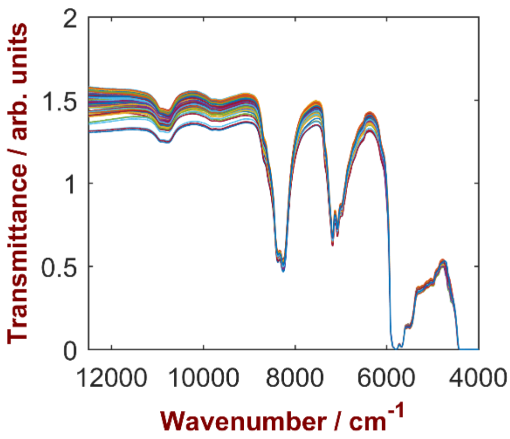


Fig. 1: FT-NIR spectra of the calibration samples

The customer’s request consisted of data transfer from this FT-NIR analyzer to a Metrohm NIRS XDS RapidLiquid Analyzer (Fig. 2, in following target instrument). The used Metrohm equipment is summarized in Tab. 1. For the measurements with the Metrohm NIRS XDS RapidLiquid Analyzer, the samples were placed in 8 mm disposable glass vials and measured in transmission mode over the full Vis-NIR wavelength range of 400–2500 nm. The temperature was kept constant at 40 °C to ensure identical measurement conditions as used in the viscosity reference analysis. The software package Vision Air 2.0 Complete was used for data acquisition, data management, and development of the quantification methods.

Tab. 1: Equipment and software used

Equipment	Metrohm code
NIRS XDS RapidLiquid Analyzer	2.921.1410
NIRS 8 mm disposable glass vials	6.7402.000
Vision Air 2.0 Complete	6.6072.208

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Fig. 2: A NIRS XDS RapidLiquid Analyzer was used to collect the spectral data of samples in transmission mode covering the full Vis-NIR wavelength range of 400–2500 nm.

Experimental

The data transfer was realized by the Metrohm representative in the following steps:

Step 1: Because FT-NIR data is often stored in wavenumber scale, it was necessary to convert the x-axis from the wavenumber to wavelength scale and the data from transmittance to absorbance.

Step 2: After the conversion, the data was interpolated to fit the resolution of the Metrohm NIRS XDS RapidLiquid Analyzer.

Step 3: Because the source and target analyzers were different in their instrumental setup, it was necessary to calculate the transfer model. This model minimizes the difference in the absorbance between both analyzers caused, e.g., by the differences in the detector response or light throughput. Such a transfer model is usually calculated using the spectra of 20–30 samples, which constitute the so-called transfer set. These samples need to be measured on both analyzers and ideally cover the whole calibration range of the application in question. In the present case, the transfer was realized using 30 samples. For the calculation of the transfer function, different mathematical algorithms were tested in order to minimize the difference between two analyzers and reduce the error of the data transfer. Piecewise direct standardization (PDS) was found to be the optimal algorithm.

The transfer model was calculated only for the spectral region of interest between 1120 and 2100 nm (i.e., 8930 and 4760 cm^{-1}) for the following reasons:

- The spectral region between 4000 and 4760 cm^{-1} (i.e., 2500 and 2100 nm) was not used for the method development as a consequence of the observed saturation.

- The spectral region between 12500 and 8930 cm^{-1} (i.e., 1120 and 800 nm) was not used for the method development because of the low sensitivity of FT-NIR in this spectral range.

- The spectral region between 400 and 800 nm was not used. This region is not accessible by FT-NIR spectroscopy as a result of the technical limitations of this technology.

Step 4: Subsequently, the transferred calibration data were imported into Vision Air Complete software and used for the method development. The developed methods were validated using an independent set of samples (validation set) and adjusted using a simple slope/bias correction in Vision software to minimize residual difference.

Results

One of the indicators of a successful data transfer is a small difference between the transferred FT-NIR spectra of the transfer sample set and the spectra of the same samples measured on the dispersive instrument. The average difference between the transferred FT-NIR data and spectra of the Metrohm NIRS XDS RapidLiquid Analyzer are shown in Fig. 3. It is clearly visible that this difference is minimal, which indicates the success of the data transfer.

Method description

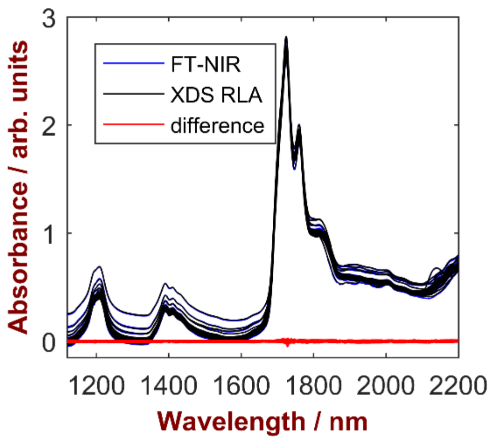

Fig. 3: Transferred spectra of the FT-NIR analyzer (blue lines), spectra of the same samples measured with the Metrohm NIRS XDS RapidLiquid Analyzer (black lines), and the difference between both data sets (red lines).

In the next step, new calibrations were developed in the Vision Air Complete software using the transferred data. Finally, the developed methods were adjusted via slope/bias correction and used for the analysis of the validation set. The analytical figures of merit of the methods built with the transferred data are summarized in Tab. 2. SECV is the standard error of cross-validation for the optimized calibrations with the transferred data. SEP is the standard error of prediction calculated using validation set.

Tab. 2: Analytical figures of merit of the transferred method

Parameter	SECV	SEP
Acid number in mg KOH / g sample	0.80	0.84
Moisture in %	0.012	0.018
Viscosity at 40 °C in cSt	2.1	2.9

The results of the analysis of the validation set are shown in Fig. 4–6. These plots show good correlation between the parameters determined by the reference analytical method (x-axis) and the values predicted by NIR (y-axis) using transferred FT-NIR spectra.

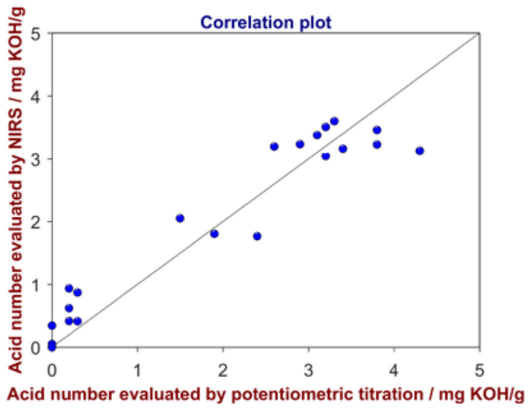

Fig. 4: Correlation plot for the determination of the acid value in the validation set. NIR values were predicted on a Metrohm XDS RapidLiquid Analyzer using transferred calibration data.

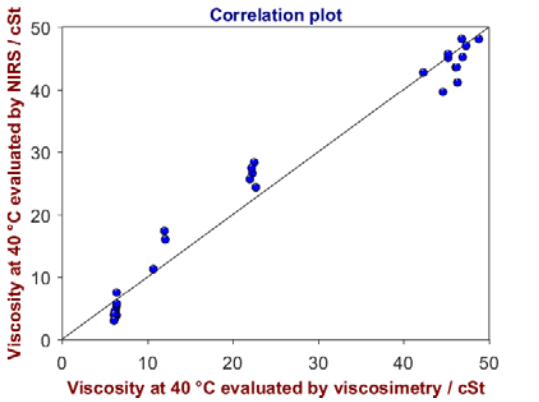

Fig. 5: Correlation plot for the determination of kinematic viscosity at 40 °C in the validation set. NIR values were predicted on a Metrohm XDS RapidLiquid Analyzer using transferred calibration data.

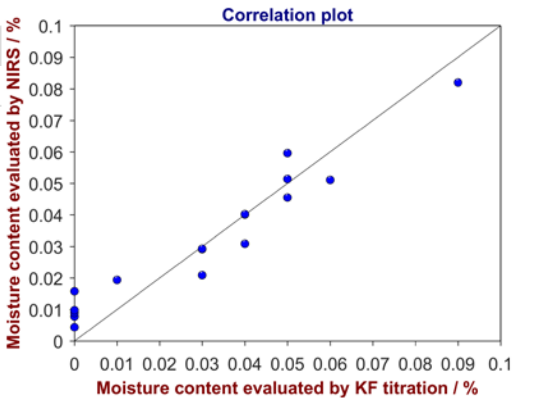

Fig. 6: Correlation plot for the determination of moisture content in the validation set. NIR values were predicted on a Metrohm XDS RapidLiquid Analyzer using transferred calibration data.

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Conclusion

This Application Note demonstrates that data from FT-NIR instruments can be successfully transferred to dispersive NIR instruments. With the approach presented herein, FT-NIR instruments can be exchanged with dispersive instruments, requiring neither tedious sample remeasurement nor time-consuming calibration model redevelopment. The transferred calibration models provide results with acceptable standard errors and accuracy. Furthermore, the whole procedure, including the measurement of transfer and validation sets, can be realized within a few hours.