APPLICATION NOTE

Water in Solvents Using a NIR-O™ Spectrometer

The measurement of low levels of water in solvents is critical to many chemical processes. This note discusses the measurement of the water concentration in solvents using fiber optic based, Near-Infrared (NIR) spectroscopy. NIR can be applied in real time mode for continuous process monitoring, or as a laboratory procedure. In either case the NIR technique saves time and money through rapid non-destructive analysis. NIR also offers the benefit of increased safety over traditional methods through the use of remote sampling via fiber optics.

Measurement Background

The NIR region of the electromagnetic spectrum contains information from the overtone and combination bands of the C-H, O-H, and NH fundamentals. This information is related to the chemical composition and can be used for both quantitative and qualitative analysis. Water has two strong absorption bands in the NIR making it an excellent candidate for making low level measurements. The bands related to water occur at 1400 nm and 1900 nm, the latter being the most intense. The choice of which band to use is determined by the background solvent and water concentration. By measuring the NIR spectra of a series of samples of known water concentration, a quantitative model can be developed which will allow the measurement of water in future samples based only on their NIR spectrum.

Experimental - Water in Methanol

The NIR spectra of a group of 22 samples of different levels of water in methanol were measured between 1000 and 2100 nm using our GUIDED WAVE™ NIR-O™ process analyzer spectrometer and a 1 cm path-length cell at constant temperature. Figure 1 shows the absorbance spectra of the samples with water concentrations ranging from 0 to 2%. The data from 1400 to 1800 nm has very high absorbance levels due to the methanol. This region of the spectrum is not needed for water measurement and will be omitted from the analysis. The spectra and concentration data were used to create quantitative Partial Least Squares (PLS) calibration models using a third party chemometric analysis softeare. For a discussion of PLS and other multivariate calibration techniques please see Martens & Naes[1] and ASTM E1655[2].

Results - Water in Methanol

The results of the data analysis are shown in Figure 2. The model RMSEPcv (root mean square error of prediction for cross validation) was 507 ppm over the entire range of values. This equates to about 1% relative error on average. This experiment was run at constant temperature. Changes in spectral data are expected with temperature variation, but this can be compensated for by simply including samples measured at different temperatures in the model.
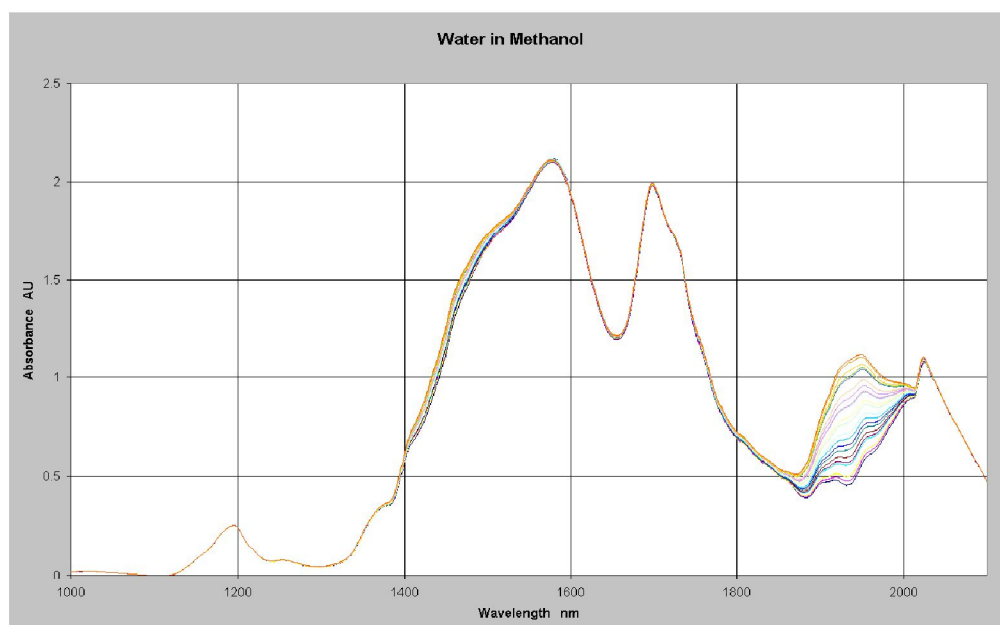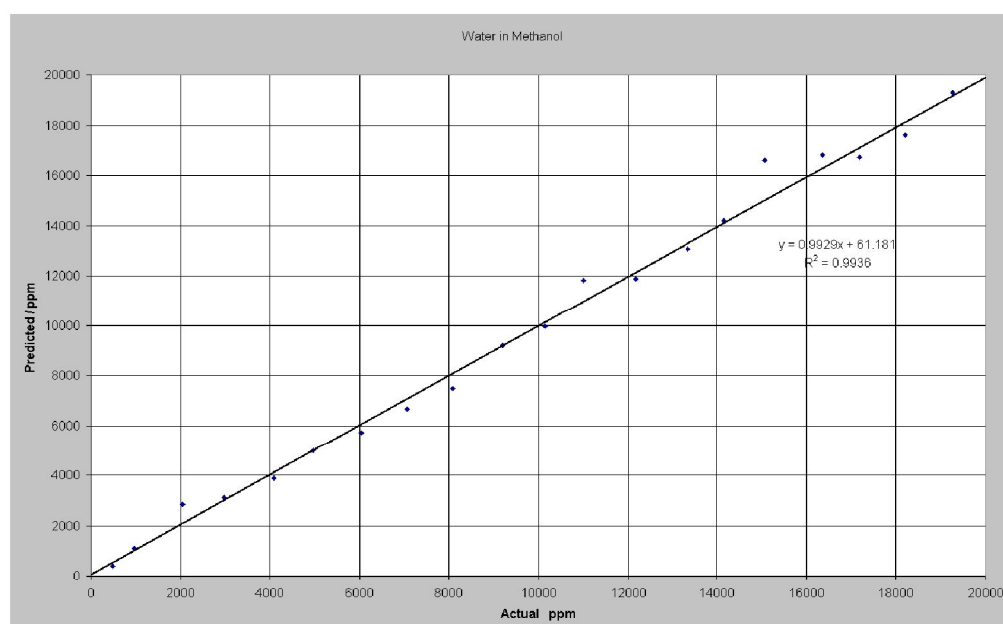
Figure 1:
NIR Spectra of Water in
Methanol 0-2%

Figure 2:
Predicted vs. Actual
Water in Methanol

Results - Low Levels of Water in Hydrocarbon

To demonstrate lower levels of water measurement, the NIR spectra of a group of 17 samples containing different levels of water in a hydrocarbon background were measured between 1100 and 1600 nm using a a NIR-O Process NIR Spectrometer and a 3 cm pathlength cell. The longer pathlength is used to allow the use of the 1400 nm peak. Figure 3 shows the absorbance spectra of the samples with water concentrations ranging from 0 to 1500 ppm. The water band is observed in the 1400 nm area. This region of the spectrum was selected for the regression analysis. A first derivative was applied to the spectral data to correct for baseline offsets and the resulting data used with the concentration data to create a quantitative model using PLS regression.

Results - Low Levels of Water in Hydrocarbon

The results of the low level water measurement are shown in figure 4. The model RMSEPcv (root mean square error of prediction for cross validation) was 16 ppm water. Water can be detected at much lower levels in non-OH containing solvents since there is less interaction between the base solvent and the water.
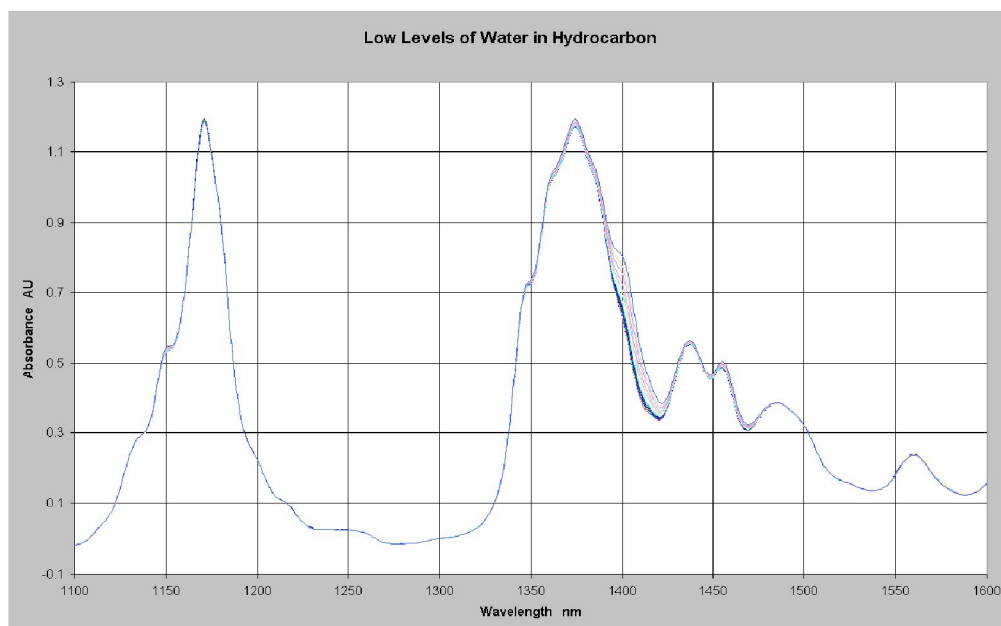
Figure 3:
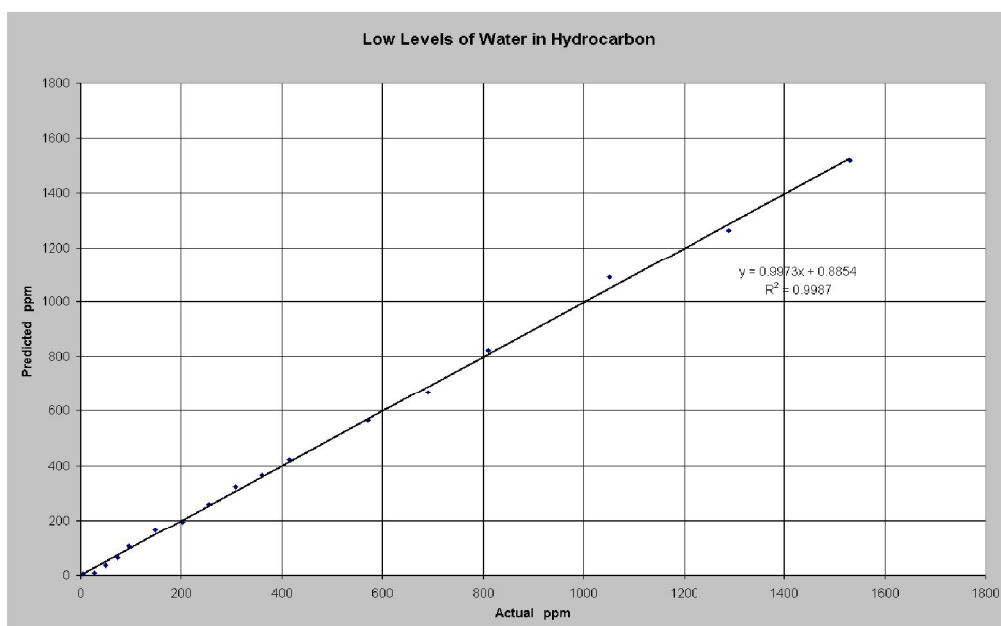Absorbance Spectra - Low Levels
of Water in Hydrocarbon

Figure 4:
Predicted vs. Actual
Water in Hydrocarbon

Conclusion

The measurement of the water levels in solvents with NIR spectroscopy is both fast and reliable utilizing the hardware and software tools described here. This method produces results that are available in real-time (seconds) thus making it a valuable tool for process measurement. This method can be applied in either a process or a laboratory environment. For more detailed information regarding system specifications please contact a Process Insights™ sales or technical specialist.

References

1. H. Martens, T. Naes, Multivariate Calibration, John Wiley & Sons, 1989.
2. ASTM E1655 Standard Practices for Infrared, Multivariate, Quantitative Analysis.

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