

Application Note: Water in dimethylformamide (DMF)

The ability to monitor low levels of water in dimethylformamide (DMF) is a key consideration in the production of 1,3 butadiene. The quality of the final product (purity) can be compromised when water in excess of 400ppm is present in the DMF solvent stream. NIR spectroscopy provides the ideal solution for the problem. In-situ water measurement is made possible by using optical probes coupled to a spectrometer with fiber optic cables. This real-time method provides the information necessary to take action when the water concentration reaches a critical level.

Background

The NIR region of the electromagnetic spectrum allows the use of the overtone and combination bands of the C-H, O-H, and N-H fundamentals. By measuring the NIR spectra of a series of DMF samples of known water concentration, a quantitative model can be developed which will allow the measurement of future samples based only on their NIR spectrum. Guided Wave analyzer systems use fiber optics to allow the sample probe to be located in remote locations away from the spectrometer itself.

Experimental

The spectrum of dry DMF is compared with that of 1% water in DMF as shown in Figure 1. This comparison shows the region from 1900 to 1980 nm which is ideal for making the water measurement. Should a contaminant be present in the DMF stream it would also be possible to detect that using a full spectrum measurement.

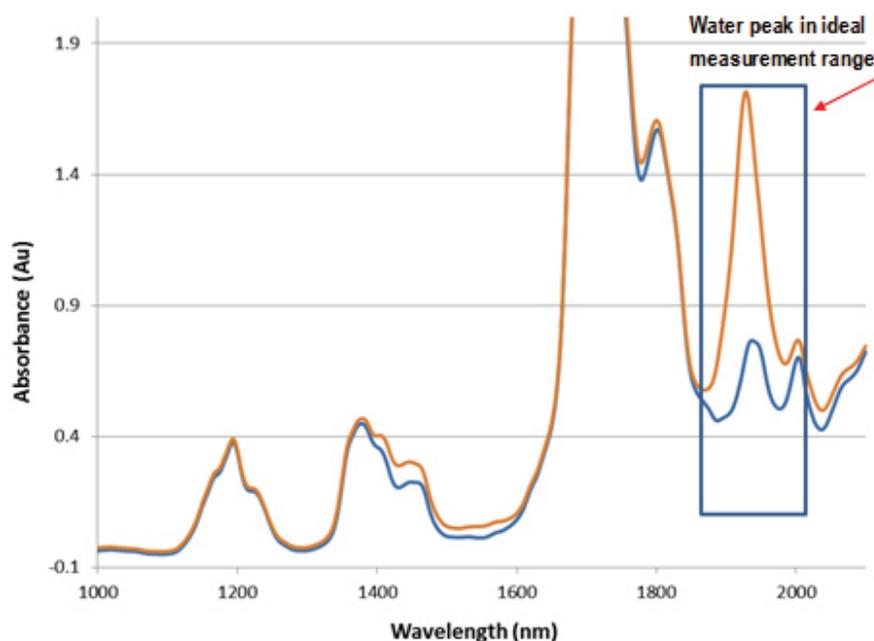


Figure 1: Water in DMF

A sample of DMF was spiked with water at different levels in the range of 58 to 604 ppm. The sample spectra were measured using both a Guided Wave full scanning, dual-beam spectrometer and a dual-beam photometer. Both systems were coupled to a 10 mm pathlength sample cell via two 20 m lengths of 500 μ m diameter fiber optic cable.

Analysis

Spectra of the spiked DMF samples demonstrate a high degree of linearity when comparing spectral data to water concentration. The results for both systems are shown in Figures 2 and 3 (respectively). In each case the standard error is less than 20ppm water.



Guided Wave Model412 Spectrometer
NIR Process Analyzer

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Discussion

Near-infrared spectroscopy coupled with fiber optic probes provides an ideal online measurement solution for low levels of water in DMF in real time. For one or two sample points a Guided Wave dual-beam photometer can provide the measurement of water concentrations from 0 to 1000ppm water. The Guided Wave full scanning, dual-beam spectrometer can provide a more comprehensive set of measurements for multiple sample points. Both systems use Guided Wave's fiber optic sample cells for optimal performance. NIR methods minimize the need for laboratory sample collection. Results are available in real-time (seconds). For more detailed information regarding system selection or specifications please contact a Guided Wave sales or technical specialist.

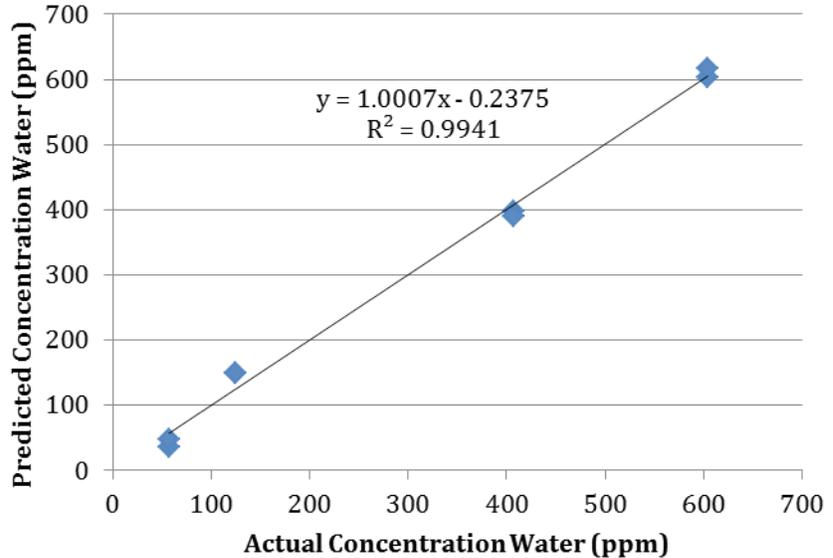


Figure 2: Water Results -- Model 412



Guided Wave ClearView® db Photometer
Zpurge Unit Class I, Division 2

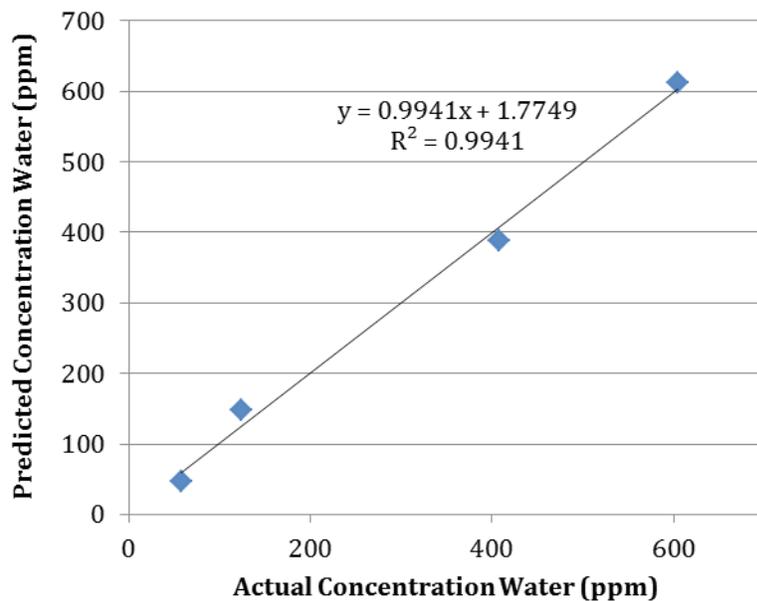


Figure 3: Water Results – ClearView db