APPLICATION NOTE

Hydroxyl Number in Polyols (OH)
Using a NIR-O™ Spectrometer or ClearView® db Photometer

The use of polymeric polyols is commonplace in the manufacturing of polyurethanes and other speciality polymers. The hydroxyl number (OH#) is a measure of the concentration of the hydroxyl groups on the polyol. The number of reactive hydroxyl groups (OH) on the polyol directly impacts the quantity of urethane linkages, which greatly influences the physical properties of the final polyurethane product. This is an important parameter to monitor and control during polyol production. The laboratory method that is commonplace for hydroxyl number determination is both time consuming and involves the use of hazardous materials. This note will discuss the use of our GUIDED WAVE™ hardware and software tools for the measurement of hydroxyl number in polyols using fiber optic-based, Near-Infrared (NIR) spectroscopy. NIR can be applied in real time directly in process or as a laboratory procedure. In either case NIR is a time and money saving alternative to traditional methods. NIR also offers the benefit of increased safety over traditional methods.

Measurement 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 polyol samples of known hydroxyl number, a quantitative model can be developed which will allow the measurement of hydroxyl number in future samples based only on their NIR spectrum. Our analyzer systems use fiber optics to allow the sample probe to be located in remote locations away from the spectrometer itself.

Experimental

The NIR spectra of a group of different process polyether polyol samples were measured between 1000 and 1600 nm using a NIR-O Spectrometer. Figure 1 shows the absorbance spectra of some representative samples using an on-line process probe with a 1 cm pathlength. The hydroxyl number for these samples ranges from 9 to 44. For this application, data preprocessing consisted of a simple 2-point baseline correction to remove any offset. The spectra and concentration data were submitted to a third party chemometric software and a calibration model was developed using PLS regression methodology. For a discussion of PLS and other multivariate calibration techniques please see Martens & Naes[1] and ASTM E1655[2].
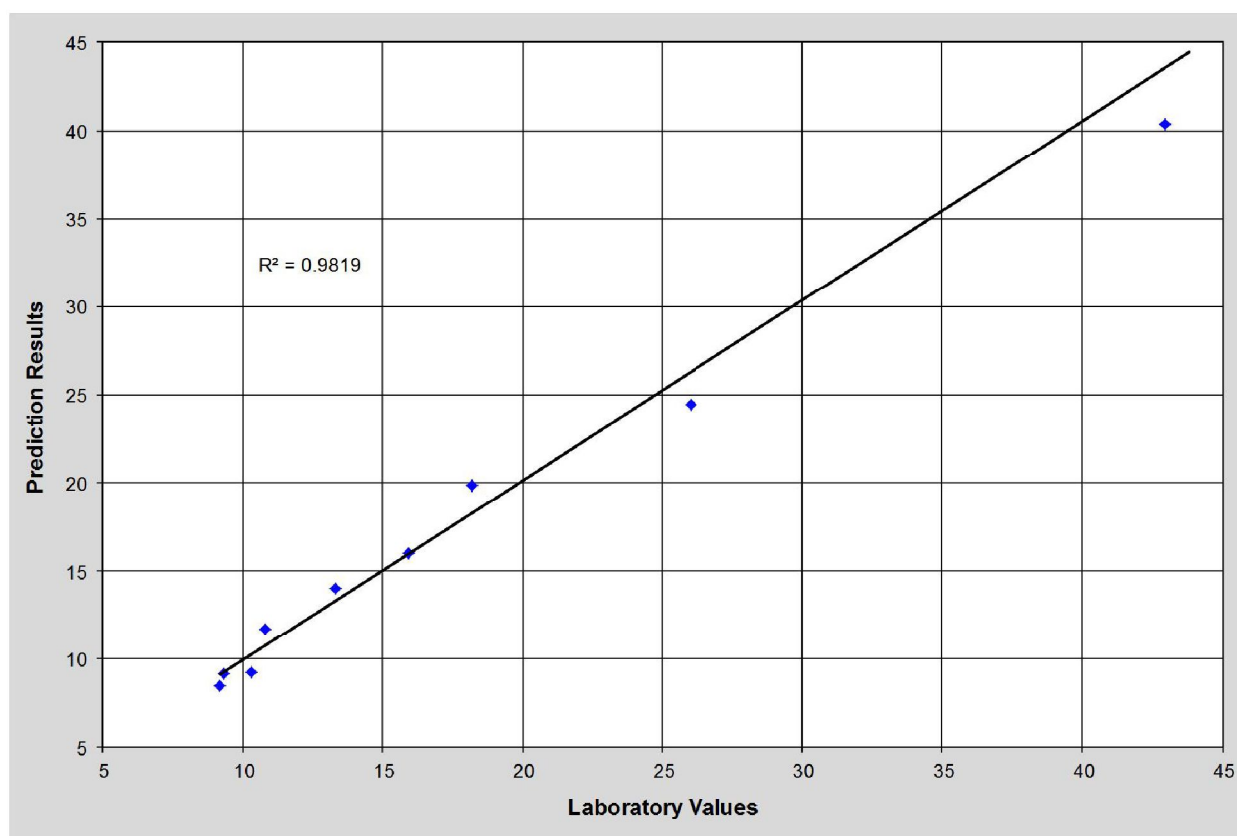
Figure 1: NIR Spectra of Polyester Polyol Samples

Analyzer Selection

For well established process measurements, a ClearView db multi-wavelength photometer can be used to achieve similar results. The photometer systems offer robust measurement capability for a more focused set of applications. Visit the Process Insights website for guidance on the choice between measurement systems.

Results

The model was used to predict hydroxyl number values in a laboratory setting. The results are shown in Figure 2 as a scatter plot. The model produced a RMSEP (root mean square error of prediction) of 1.8. This is in good agreement with the accuracy of the standard laboratory method.
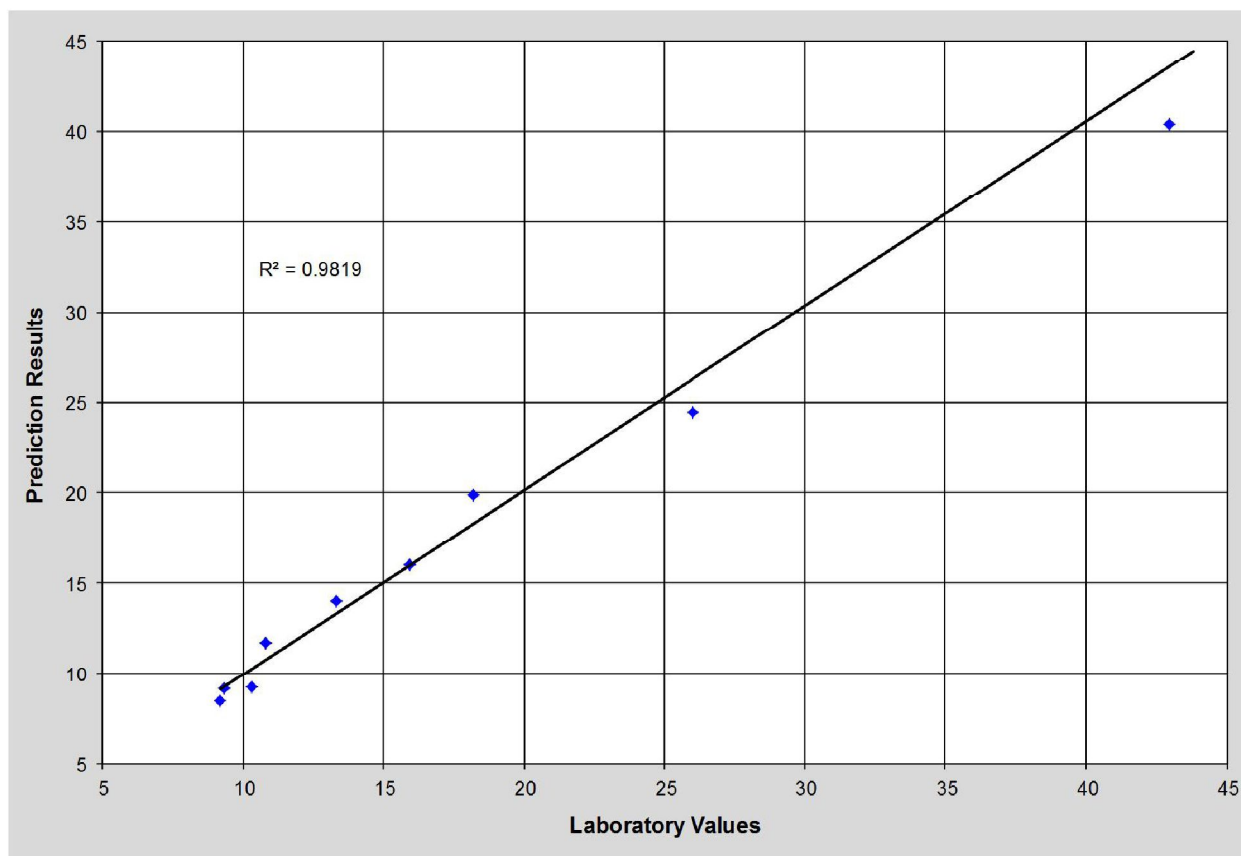
Figure 2: OH Number Laboratory vs. Measured

Conclusion

The measurement of OH number of polyols using NIR spectroscopy is both fast and reliable utilizing our hardware and software tools as described here. This method minimizes the need for performing the previous laboratory method and hence the results are available in real-time (seconds). This method can be applied in a batch reactor system for control of this important property during mixing and to determine the endpoint of the reaction thus resulting in significant savings of time and meterial costs. For more detailed information regarding system specifications and the NIR-O process analyzer 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.

GAIN REAL-TIME INSIGHT INTO YOUR PROCESS

Process Insights manufactures and delivers premium sensors, monitors, detectors, analyzers, instrumentation, and software that are mission-critical to keep your operations, personnel, and the environment safe – every day across the globe.

Get the most reliable, precision analytical technologies available on the market today. We will work to match your needs and budget, and provide the optimal, and most stable process analysis solution for your application.

CENTERS OF EXCELLENCE | PROVIDING PROVEN SOLUTIONS

Process Insights is committed to solving our customers' most complex analytical, process, and measurement challenges everyday.

Process Insights – The Americas

4140 World Houston Parkway Suite 180, Houston, TX 77032, USA +1 713 947 9591

Process Insights – EMEA

ATRICOM, Lyoner Strasse 15, 60528 Frankfurt, Germany +49 69 20436910

Process Insights – APAC

Wujiang Economic and Technology, Development Zone, No. 258 Yi He Road, 215200 Suzhou, Jiangsu Province, China +86 400 086 0106

For a complete range of products, applications, systems, and service options, please contact us at:
info@process-insights.com

For a complete list of sales & manufacturing sites, please visit:
https://www.process-insights.com/about-us/locations/

COSA Xentaur, Tiger Optics, Extrel, Alpha Omega Instruments, ATOM Instrument, MBW Calibration, MGA, Guided Wave, ANALECT and LAR TOC Leader are trademarks of Process Insights, Inc.

REVOLUTIONIZING MEASUREMENT

EVERYWHERE