Guided Wave
Process Analytical Systems

Application Note:

Phosphoric Acid Concentration

The use of phosphoric acid solutions is common in the semiconductor industry to both clean and etch metal surfaces. The concentration of the phosphoric acid is important to optimize these processes. Typically the laboratory method used for this determination involves one or more titrations. These methods can be both time and labor intensive, and may involve the use of hazardous material. Also, laboratory methods do not typically provide the ability for real time control of a process involving these solutions. This note will discuss the use of Guided Wave hardware and software tools for the measurement of phosphoric acid concentration in aqueous solutions using fiber optic-based, Near-Infrared (NIR) spectroscopy. NIR can be applied in real time directly in process monitoring or as a laboratory procedure. In either case NIR is a time and money saving alternative to traditional methods.

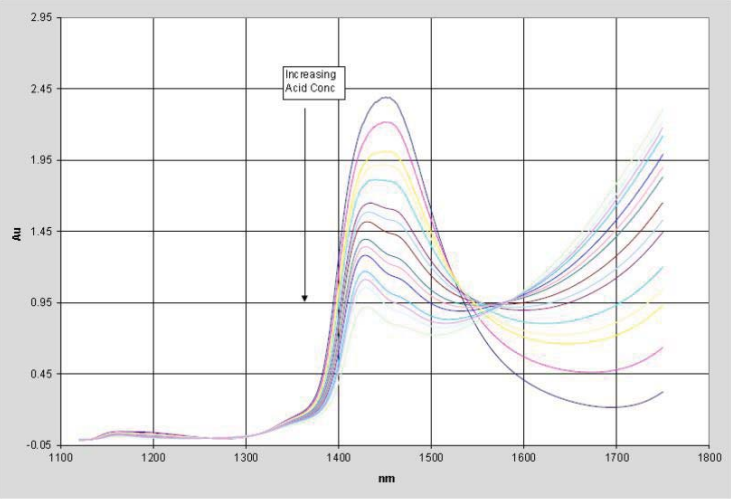

Figure 1:
NIR Spectra of Phosphoric Acid Solutions

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 NH fundamentals. By measuring the NIR spectra of a series of phosphoric acid samples of known concentration, a quantitative model can be developed which will allow the measurement of future samples based only on their NIR spectra. Guided Wave analyzer systems use fiber optics to allow the sample probe to be located in remote locations away from the spectrophotometer itself.

Experimental

The NIR spectra of a series of prepared solutions of water and phosphoric acid were measured between 1050 and 1750 nm using a Guided Wave Model 412 NIR Spectrophotometer. Figure 1 shows the absorbance spectra of these samples collected in a laboratory setting using a 2 mm pathlength. 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 the Unscrambler™ 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 ASTME1655[2].

Results

A procedure known as cross-validation was used to validate the model accuracy. This involves leaving out one or more samples from the model and then testing the predicted value(s) as a measure of model accuracy. This sequence is repeated several times until each sample has been left out of the model one time. The statistics calculated are then used as an estimate of prediction accuracy for future samples. Figure 2 shows the validation predictions for the phosphoric acid model. The resulting RMSEP is 0.25% phosphoric acid. This accuracy may be further improved on by narrowing the concentration range that is covered by the calibration model.

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Conclusion

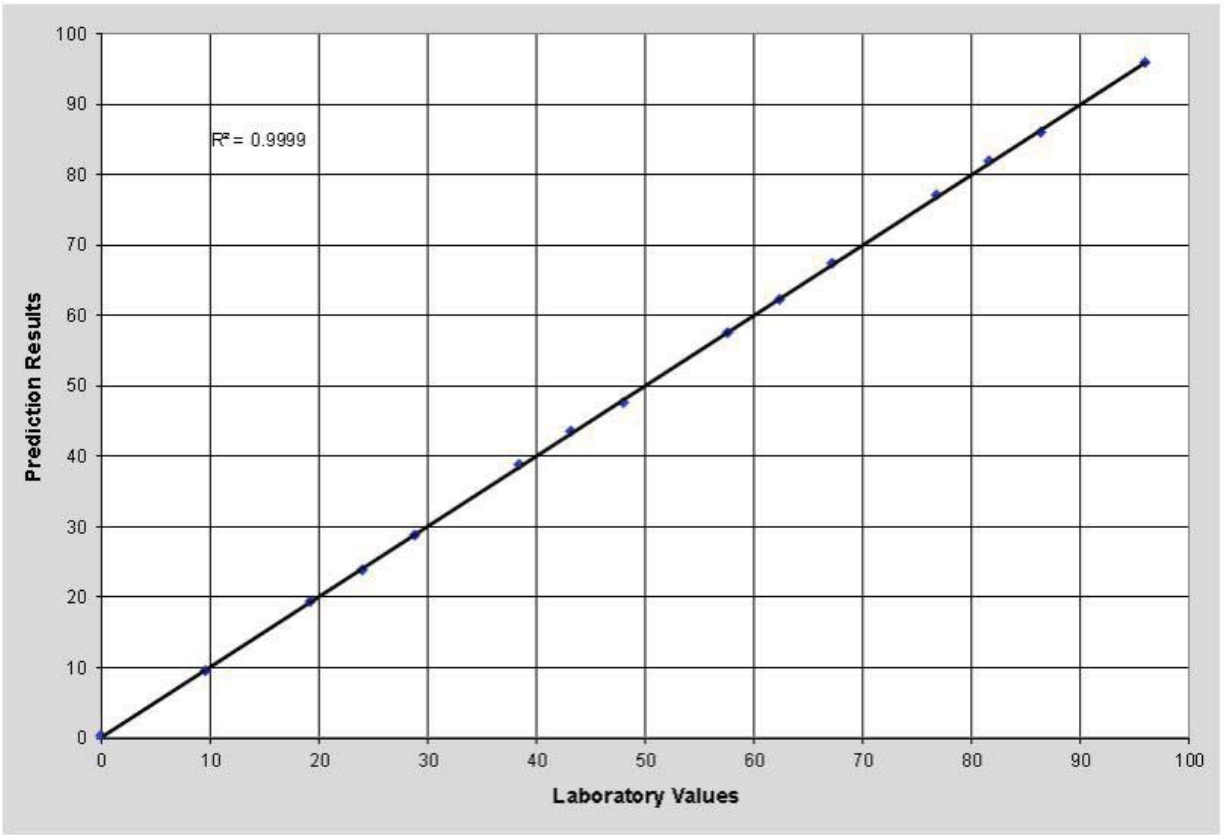
The measurement of the phosphoric acid concentration in water using NIR spectroscopy is both fast and reliable utilizing the Guided Wave hardware and software tools as described here.

This method minimizes the need for laboratory sample collection and reduces hazardous material handling by laboratory personnel. Results are available in real-time (seconds) for multiple parameters in both simple and complex streams. For more detailed information regarding system specifications please contact a Guided Wave sales or technical specialist.

References

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

Figure 2 - Cross-validation Predictions - Phosphoric Acid



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