

## APPLICATION NOTE

### Phosphoric Acid Concentration

The use of phosphoric acid solutions is common in the semi-conductor 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 our 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.

### 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. Our analyzer systems use fiber optics to allow the sample probe to be located in remote locations away from the spectrophotometer itself.

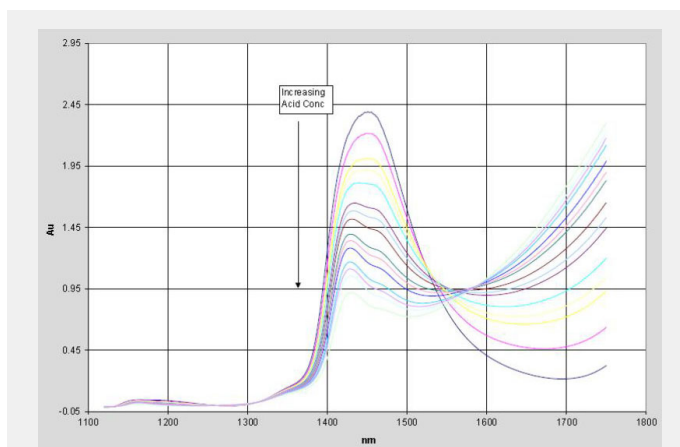


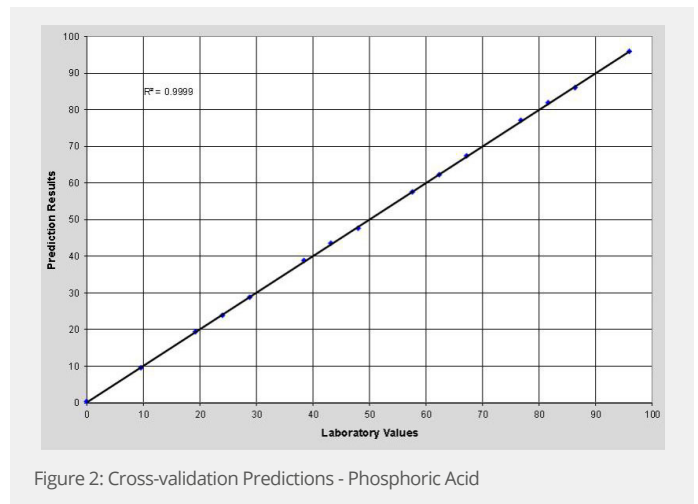
Figure 1: NIR Spectra of Phosphoric Acid Solutions



**NIR-O Spectrometer**

## Experimental

The NIR spectra of a series of prepared solutions of water and phosphoric acid were measured between 1050 and 1750 nm using a NIR-O™ process Spectrometer. 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 a third party 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<sup>1</sup> and ASTM E1655<sup>2</sup>.



---

## 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.

---

## Conclusion

The measurement of the phosphoric acid concentration in water using NIR spectroscopy is both fast and reliable utilizing the 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 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](mailto: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**