

Specac

Application Note

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NIR Process Monitoring of Continuous Industrial Acetic Acid Production Using the ProCell™ Vortex



Vortex ProCell

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Inside: Find how Specac can aid your industrial process adding continuous monitoring to your flow reaction.

Introduction

Acetic acid is an important intermediate chemical for the manufacturing of solvents like ethyl acetate and for key ingredients in paints like vinyl acetate monomer. It can also be used for the production of acyl chloride, which is widely used in the synthesis of pharmaceuticals and pesticides and acetic anhydride via the ketene route.

The annual production of acetic acid is about 1.2 Mtons [1]. If the throughput of the plants can be increased by just 1%, then at an average price of about \$500 per ton of acetic acid, extra product worth more than \$6m can be produced each year if the plant runs continuously. As a result, any installation to monitor the process in real time would pay itself back within months rather than years.

ProCell™ Vortex

NIR Process flow cells custom designed to your specific requirements ensuring quality and reliability from inside to out:

- ▶ Durable sapphire optics
- ▶ Double-sealed optical assemblies
- ▶ Fibre-supporting gland and shielding for environmental protection.
- ▶ Cleaning ports as standard
- ▶ Optional seal bypass tell-tales
- ▶ Fibre optic

Online Analysis of the Acetic Acid Production Line

The manufacturing of acetic acid involves high temperatures, pressures and toxic chemicals including methyl iodide, carbon monoxide and methanol. Historically samples of the reactor had to be taken manually to measure the key components of the carbonylation reaction off-line. The sample was then analysed in a laboratory for the concentration of key components. Not only did this require operators adhere to stringent safety control to allow safe removal of this sample, but also resulted in a significant delay between the time the sample was taken and the results being available to the reactor control system.

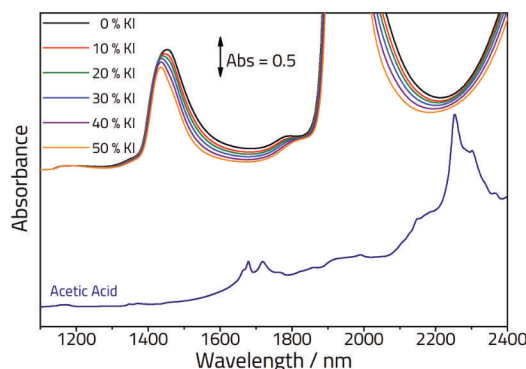


Figure 1: NIR spectra of KI mixtures with water and pure acetic acid (offset).

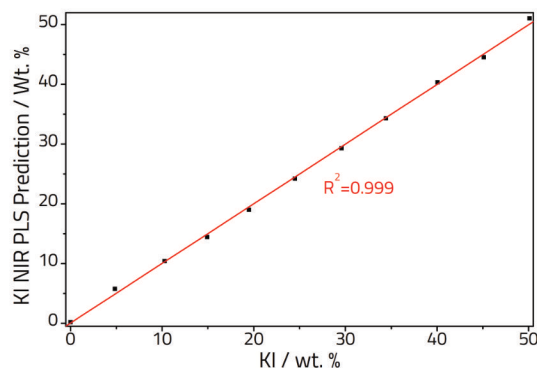


Figure 2: PLS-1 model for the prediction of KI in water

NIR spectroscopy can be used to monitor the key components of an acetic acid production process on-line [1], including: methyl acetate (MeOAc); acetic acid (AcOH); methyl iodide (MeI); water (H₂O); and potassium iodide (KI). Concentrations used in the study by Chung *et al.* were those typically encountered in an

acetic acid production process, even though they were not actual process samples. Standard errors of prediction of around 0.2 wt. % coupled with the excellent precision and accuracy of NIR spectroscopy mean that process parameters can be fine-tuned to optimize production.

At Specac similar measurements have been carried out to assess the suitability of our Vortex Pro cells for the measurement of key components in the acetic acid production process. The concentration of KI in distilled water ranged from 0 to 50 % and a 1 mm pathlength cell was used due to the strong absorbance of water. Resulting spectra are shown in Figure 1. As expected, a clear shift in the peak absorbance wavelength can be observed with increasing concentrations of KI.

A chemometric PLS-1 model was built to demonstrate how varying concentrations of KI can be measured with NIR spectroscopy, shown in Figure 2. The spectra (1100-1900 nm) were pre-processed by calculating the first derivative, followed by an area normalisation. An excellent regression coefficient could be achieved with only 1 factor, demonstrating again the feasibility of NIR for the measurement of key components.

A typical acetic acid process involves the following main parts, depending whether it is the original Monsanto™ or a later generation process [1]:

- ▶ One main and potentially a secondary reactor
- ▶ A flash tank
- ▶ A distillation train containing 2 or 3 columns

Sampling of the acetic acid production process can be achieved by positioning a measurement cell either directly into the process line (as shown in Figure 3) or into a by-pass loop. The cell is connected remotely by fiber optics to a spectrometer located away from the process in a safe environment.

The on-line measurement allows plant control to react much more quickly to any process variation and operate the plant more safely and closer to the original process design specification.

The importance of keeping the key components of the acetic acid production in concentrations ranges best suited for



optimum performance of the process was highlighted by Jones *et al.*, [1] regardless of the technology.

Specac have previously specified cells from our ProCell™ Vortex range for the production of acetic acid. These featured both zirconium or Hastelloy C276 metallurgies depending on the part of the process to be monitored and seals suitable for the conditions encountered. Because of the wide range of metallurgy and rating available Specac process cells can be directly located on an exit line from an acetic acid producing reactor to monitor the composition of key components of the reactor.

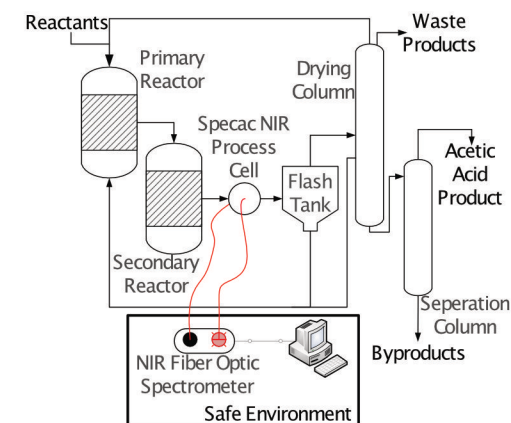


Figure 3: Schematic diagram showing one potential location for placement of the Specac Procell Vortex to monitor product stream out of the secondary reactor

References

- [1] J. Jones, *Platinum Metals Rev.*, **44**, (2000), 94
- [2] H. Chung & M.-S. Ku, *Vibrational Spectroscopy*, **31**, (2003), 125-131