Monitoring Tall Oil distillation using a Vortex™ process flow cell

In this note: NIR measurement of crude tall oil

The distillation of crude tall oil (CTO) occurs in four fractional distillation columns: the first separates pitch from the CTO; the second removes the rosin soaps from the de-pitched CTO; the third distills crude fatty acids, and the final column (Figure 1) separates these into tall oil fatty acids and distilled tall oil (DTO) [1]. Traditional process analytics require samples to be removed from the process line for testing by chromatographic or wet chemistry techniques. The length of time between sampling and analysis delays the taking of corrective action if the process has drifted out of specified bounds. This can cause costly production delays and result in wasted material and effort. With on-line near-infrared sampling, instantaneous feedback is possible.

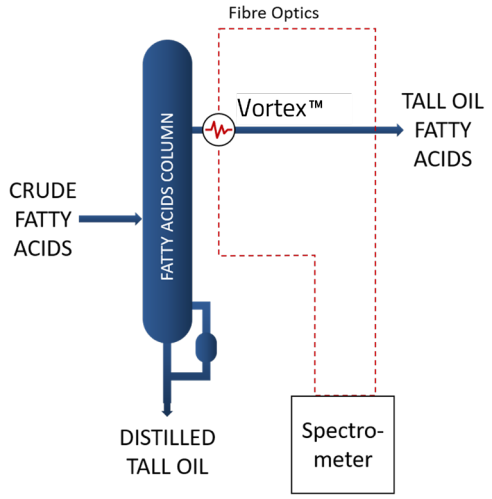

Figure 1: Final distillation column showing a possible placement of an NIR cell to monitor tall oil fatty acids.

Introduction

The near-infrared region of the spectrum contains overtones of the fundamental molecular vibrations seen in mid-infrared spectroscopy, as shown in Figure 2. These spectral features fluctuate and shift position as changes in composition occur and can be used to monitor deviation from the expected content of the process line at a given place.

Experimental

For this application note, a sample of tall oil fatty acids was obtained and the NIR spectrum collected on a commercially available spectrometer using a Specac Pearl fitted with a CaF_2 1000 µm pathlength Oyster cell. Such laboratory-based measurements are complementary to process monitoring testing with an in-line fibre optic process cell.

The resulting spectrum is seen in Figure 2. The bands at 1700-1800 nm and 2250-2400 nm are indicative of the first overtone of the C-H stretching vibration and a combination band (stretch + bend), respectively.

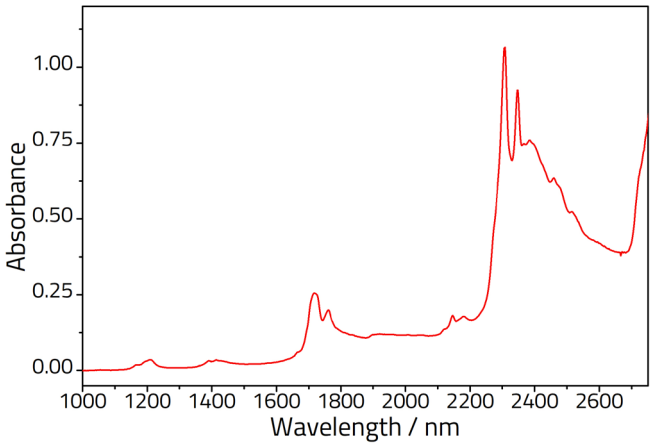

Figure 2: NIR spectrum showing an example spectrum of a sample of tall oil. Vortex process cell.

Results & Discussion

In practice, sampling of the process is achieved by positioning a measurement cell either directly into the process line or into a by-pass. The cell is connected by fiber optic to a spectrometer located away from the process environment. Process temperatures can range from ambient level to 200 °C and the production requires the use of strongly basic and acidic solutions. In order to sample the process stream directly a measurement cell is needed which is robust, free of leakage, and well-protected against the effects of temperature and chemical attack.

The Specac Vortex™ range is ideal for the conditions encountered in tall oil refineries, as shown in the schematic. These are designed for direct insertion into the process stream via standard DIN or ANSI flanges. The cells can be constructed in stainless-steel or Hastelloy, depending on the chemistry of the fluid sampled, and interface optically with industry standard SMA fiber optic connections. Design features throughout ensure maximum reliability and performance during service: protective hoods shield the fiber connections, and since there may be a build-up of particulate and solid matter inside the process lines, a cleaning port can be provided to allow flushing of the cell without removing it from the process line.

Measurement cells such as the Vortex™ can remain within the process line for many years without removal for servicing. Nonetheless, Specac offer a continuous maintenance plan for servicing of all cells.

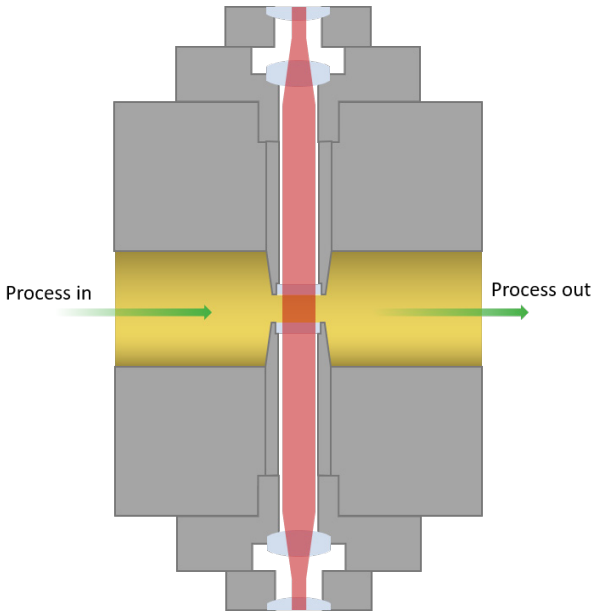

Figure 3: Schematic diagram of the NIR Vortex process cell.

References

[1] https://www.harima.co.jp/en/pine_chemicals/

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