Inline process monitoring of the moisture content in propylene oxide

Summary

Propylene oxide (C_3H_6O, PO) is a major industrial product with a global production of more than 10 million tons per year [**1**]. PO is mostly produced to make polyether polyols, propylene glycol, propylene glycol ether solvents, and other products. There are several production processes available, however the majority of PO is still co-produced along with styrene monomer (approximately one-third of PO production worldwide). Other PO production routes include the chlorohydrin process, epoxidation of propylene with hydrogen peroxide, epoxidation of propylene with organic peroxides, and even epoxidation using molten salts.

This Process Application Note presents a method to closely monitor in «real-time» low levels of moisture in PO safely, reliably, and optimally. Due to the hazardous and hygroscopic nature of PO, a single explosion-proof inline analyzer is the preferred solution to reduce chemical treatment, improve product quality, and increase profits.

Configuration

A629280230C - NIRS XDS Process Analyzer – SingleFiber 9 Channels

The NIRS XDS Process Analyzer – SingleFiber provides the next generation of process analyzers for real-time analysis in the pharmaceutical and chemical industries. Non-destructive, accurate measurements of clear liquids are performed directly in the process line or in a reaction vessel. Up to 9 probes and/or flow-cells can be connected to the analyzer. All 9 channels can be configured independently from each other in the software. Single Fiber optics allows the 9 measuring points to be up to 100 meter away from the XDS NIR process analyzer.

Introduction

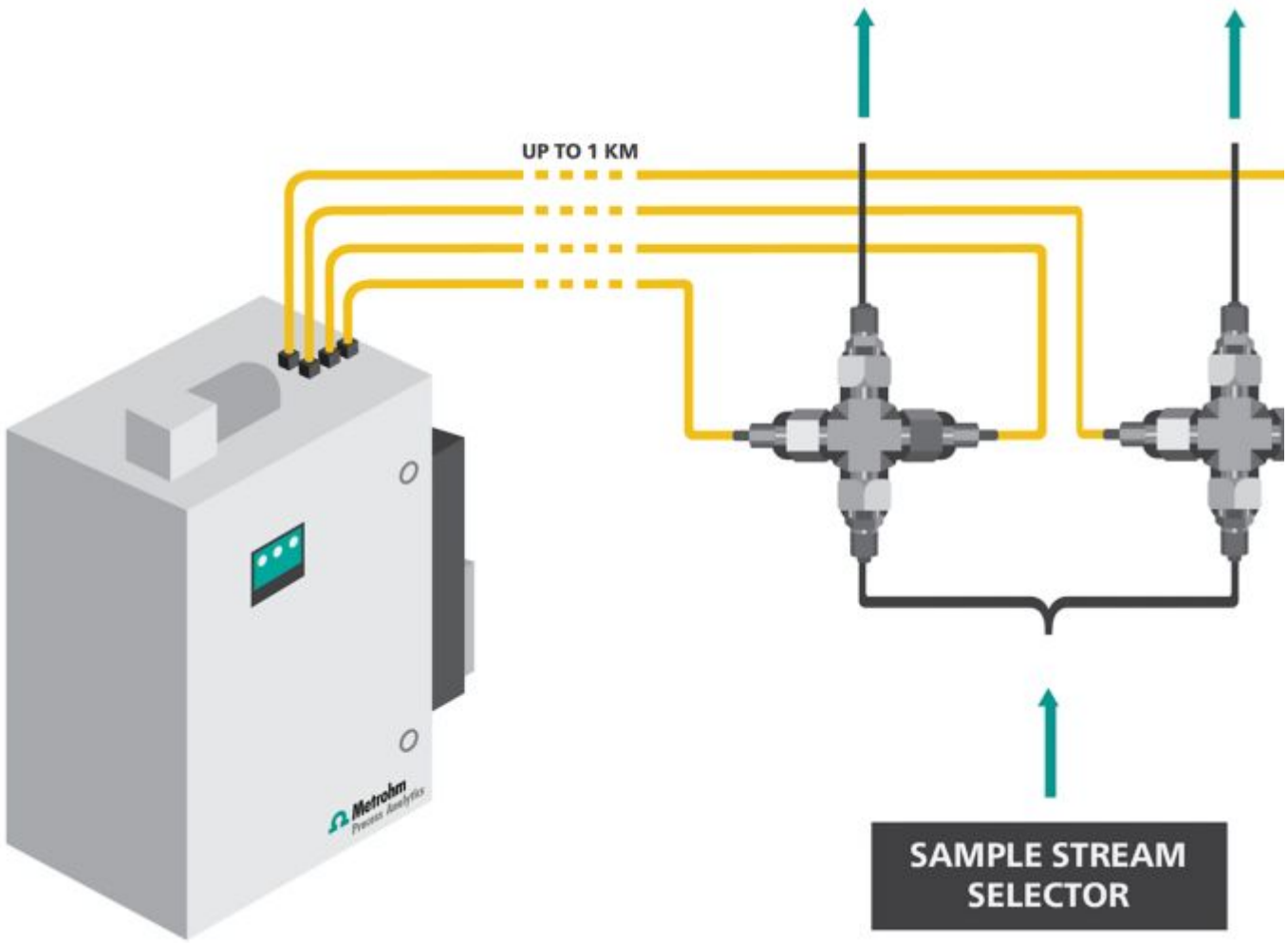

Figure 1. NIRS system configuration for online analysis of water content in PO streams. All figures were adapted from GIT Labor-Fachzeitschrift article [2].

PO is a highly hazardous, flammable, and hygroscopic substance, and therefore needs to be handled with extreme caution. Tight control over moisture and other impurities in the final product (as well as along the manufacturing process at critical points) is necessary to overcome unwanted side reactions or poor yields. Manual laboratory methods can be quite cumbersome and can introduce bias depending on the analyst. Therefore, the hygroscopic nature of PO necessitates inline or online analysis of water content for the most precise results. Additionally, «real-time» analysis is a requisite for high throughput PO production because this gives short response times in case of process changes or increased water content in the final product.

Safe analysis of low moisture content in PO is possible with reagent-free techniques such as near-infrared spectroscopy (NIRS). Suitable NIRS process analyzers are available for use in hazardous environments with robust stainless-steel flow cells (**Figure 1**). Metrohm NIRS process analyzers enable comparison of «real-time» spectral data from the process to the

primary method (titration) to create a simple, yet indispensable model for the PO production process. Gain more control over production with a Metrohm Process Analytics NIRS XDS system configured for applications in hazardous areas, capable of monitoring up to 9 process points with the multiplexer option.

Application

Wavelength range used: 1850–1950 nm. Stainless steel flow cells were used for online measurements. Explosion-proof process analyzers are recommended for hazardous areas such as these.

Typical Range

Table 1. Typical water concentration range in PO according to ASTM guidelines

Components	Range (mg/L)
Water	20-30

Remarks

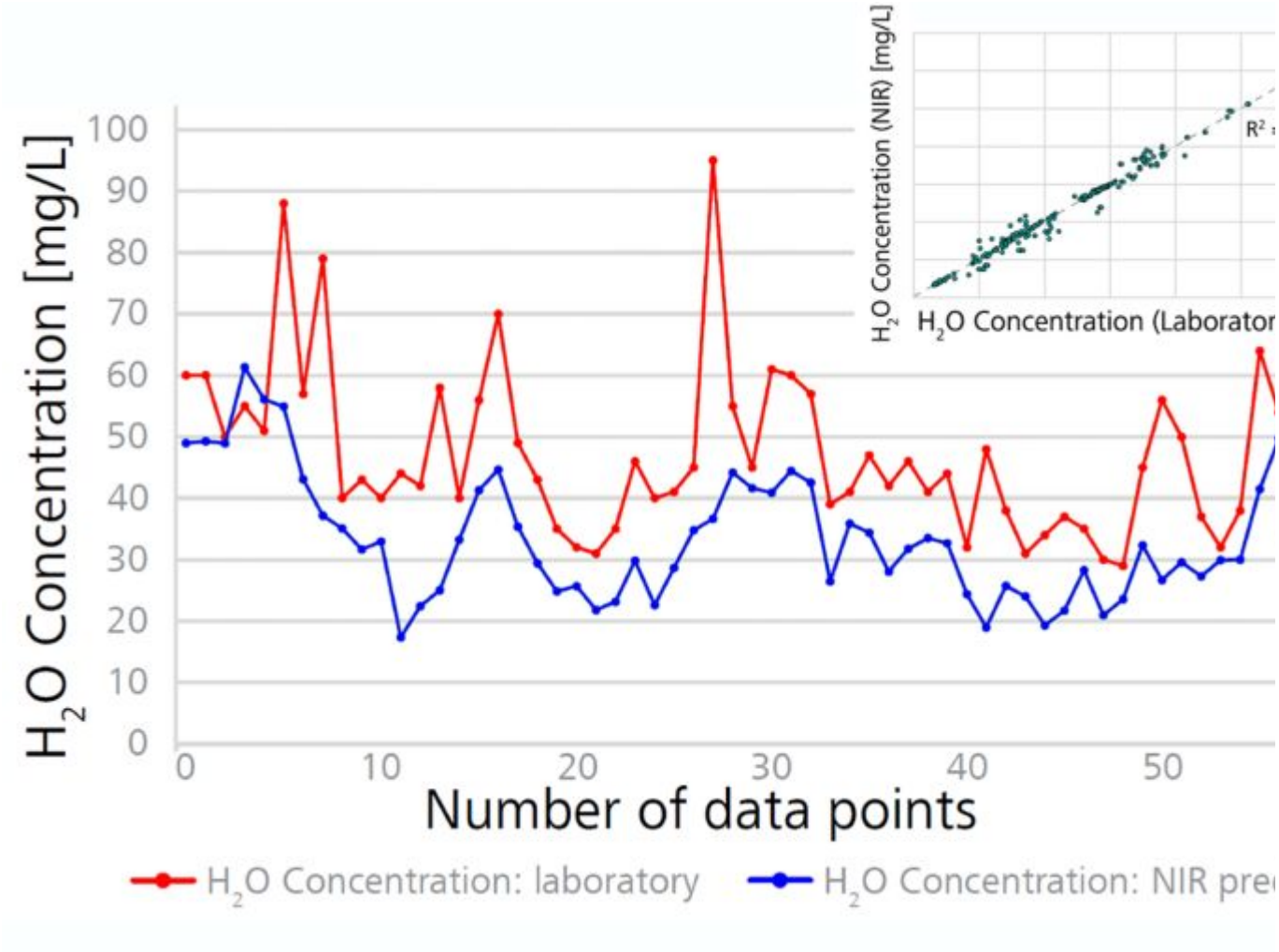

Figure 2. Validation of the NIR process data by laboratory Karl Fischer (KF) titration; Inset shows enclosed NIR calibration model for predicting water concentration in propylene oxide process streams. All figures were adapted from GIT Labor-Fachzeitschrift article [2].

A reference method (i.e. KF titration) (**Figure 2**) is mandatory to build the prediction models. Measurements performed in the laboratory showed higher water content values than those predicted via online NIRS. By the time the KF titration was performed, the samples had captured atmospheric moisture and were no longer completely representative of the actual process conditions. Therefore, online KF titration was used to build more accurate prediction models.

Table 2. Dedicated solutions offered by Metrohm Process Analytics for sampling needs

Sampling	Flow cells type	Measurement	Fiber type	Connection
Flow cell	Fixed pathlength 2 mm SS316	Transmission	Single	Swagelock
	Variable pathlength 0.5–12 mm	Transmission	Single	Swagelock
	PTFE Flow-through cell	Transmission	Single	Swagelock

Further reading

Related process application notes

AN-PAN-1007: Online analysis of peroxide in the HP-PO process

AN-PAN-1047: Inline monitoring of water content in naphtha fractions by NIRS

Other related documents

WP-023: Karl Fischer titration and near-infrared spectroscopy in perfect synergy

8.000.5325: Water Content Analysis. Karl Fischer titration and Near-Infrared Spectroscopy in perfect synergy

Benefits for NIRS in the PO process

- **Optimize product quality** and increase profit with faster response time to process deviations
- Greater and faster **return on investment (ROI)**
- **No manual sampling needed**, thus less exposure of personnel to dangerous chemicals

References

1. Kawabata, T.; Yamamoto, J.; Koike, H.; Yoshida, S. Trends and Views in the Development of Technologies for Propylene Oxide Production; Sumitomo Kagaku, 2019; pp 4–11.
2. Kleimeier. Nahinfrarotspektroskopie Produktionsprozesse Unter Der Lupe. GIT Labor-Fachzeitschrift **2018**, 36–38.

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