Inline process monitoring of moisture content in propylene oxide

Propylene oxide ($C_{3}H_{6}O$, PO), is a major industrial product with a global production of more than 7 million tons per year (2017). It 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 methods for the production of propylene oxide include the chlorohydrin process, epoxidation of propylene with hydrogen peroxide, epoxidation of propylene with organic peroxides, and even epoxidation using molten salts.

PO is a highly hazardous, flammable, and hygroscopic substance and therefore needs to be treated with 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.



a) NIRS system configuration. b) NIR spectra of PO; Inset shows that the differences in the concentrations can be seen by the formation of the 2nd derivative and differentiated in the calibration model. c) 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.

Fast, inline analysis of low moisture content in PO is possible with reagentless techniques such as near-infrared spectroscopy (NIRS). Suitable NIRS process analyzers are available for use in hazardous environments with robust stainless steel flow cells. Our NIRS process analyzers enable comparison of real-time spectral data from the process to the primary method (KF titration) to create a simple, yet indispensable model for your process needs. Gain more control over your production with a Metrohm Process Analytics (MPA) NIRS XDS system configured for applications in explosion proof areas, capable of monitoring up to 9 process points with the multiplexer option.

Keywords: water content, NIR, spectroscopy, continuous processing, NIRS XDS, moisture, propylene oxide Visit our website **www.metrohm.com**



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

Typical Range: 20–30 mg/L (ppm) H₂O

*Remark: A reference method is mandatory to build the prediction models (i.e. KF). Measurements performed in the laboratory reported a higher level of water content than those predicted via online NIR. The hygroscopic nature of the samples caused them to absorb more ambient moisture prior to laboratory analysis. Therefore, online KF was used to build accurate prediciton models.

Benefits for NIR spectroscopy in process:

- Save analysis time, faster time to market
- Greater and faster return on investment
- Improved product quality and manufacturing efficiency
- Safe working environment, automated sampling, registration, and lab analysis

Related white papers and articles:

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

• Kleimeier, C. Nahinfrarotspektroskopie Produktionsprozesse unter der Lupe. GIT Labor-Fachzeitschrift, June, 2018, pp 36–38

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

Related ASTM methods:

- ASTM E1655: Standard Practices for Infrared Multivariate Quantitative Analysis
- ASTM D6122: Standard Practice for Validation of Multivariate Process Infrared Spectrophotometers

Related Process Application Notes:

- AN-PAN-1007: HPPO process for Propylene oxide (PO): Analysis of peroxide
- AN-PAN-1047: Inline monitoring of water content in naphtha fractions by NIRS



Dedicated solutions for your sampling needs

Sampling	Flow cells type	Measurement principle	Fiber type	Connection	Process measurement
Flow cell	• Fixed path length 2 mm SS316	 Transmission 	• Single	 Swagelock 	• Online
	• Variable path length 0.5–12 mm	 Transmission 	• Single	 Swagelock 	• Online
	PTFE Flow-through cell	 Transmission 	• Single	 Swagelock 	• Online

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NIR XDS Process Analyzer



