

NIR on-line analysis in a chemical distillation plant

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Introduction

In recent years near-infrared (NIR) technology has become a very useful technique for quality control in the chemical industry. NIR technology is a rapid, non-destructive analysing method, which in most cases needs no sample preparation. Therefore for the process control in production plants other conventional, time consuming methods (for example, HPLC, titration...) are frequently replaced by NIR on-line technology. However, increasing productivity demands and high quality control requirements—especially in the basic chemical industry as a supplier for the pharmaceutical industry—call for sophisticated, high performance NIR on-line systems:

Fast

- Optimisation of the production process
- Availability of quality data in “real time”
- Online detection of deviations between actual and set values of the product parameters

Economical

- Low operating costs
- Easy to maintain

Secure

- Reliable operation
- Error and warning handling
- Automatic documentation of the measuring results

Easy

- Easy to handle during routine operation

Robust

- Equipment for harsh process environment
- Long-term stability of the installed instrumentation

Instrumentation

The introduced NIR on-line system is based on the robust Büchi NIRFlex N-419 process spectrometer, the flexible and easy-to-handle Büchi NIRLine N-420 Process Control Module as well as several accessory components like optical multiplexers, process probes and fibre optics.

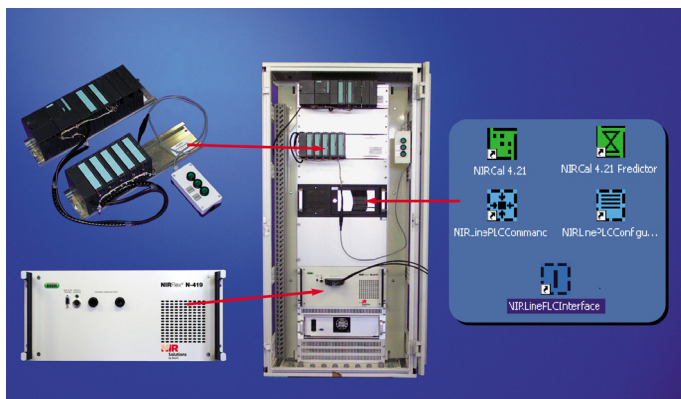


Figure 1. Büchi NIR on-line instrumentation.

Büchi NIRFlex N-419, FT-NIR process spectrometer

The BÜCHI NIRFlex N-419 is a Fourier (FT)-NIR spectrometer which is integrated in a robust 19" housing and equipped with the chemometric software NIRCAl 4.21.

Main features:

- The quartz wedge polarisation interferometer guarantees high robustness and reliability for the use in process environments.
- Results are obtained within seconds
- The robust 19" housing can be mounted directly into the rack of an industry cabinet
- Easy changeover from the lab to the production process because of identical FT-NIR technology in both, the laboratory as well as in the process spectrometer
- The "NIRCAl Calibration Wizard" leads quickly and completely through a calibration set-up routine and a "Best of" selection for the calculated calibrations is produced.

Büchi NIRLine N-420 process control module

The BÜCHI NIRLine N-420 Process Control Module consists of the NIRLine Process Control Software and the NIRLine programmable logic controller (PLC) which controls the Büchi NIRFlex N-419 process spectrometer as well as the optical multiplexers.

Main features

- Easy toggling between the manual mode (spectra recording, calibration development) and the automatic mode (time controlled routine operation)
 - Flexible, easy-to-handle configuration of the PLC control software enables to define measuring cycles for routine operation according to the particular production application in a fast and easy way
- Set-up for operating as a standalone unit, fully network integrated or linked to a superior process control system (PCS):
 - Analysing of up to 64 product parameters
 - 14 analogue outputs for measuring results
 - 7 digital outputs for defined error messages, 1 digital error collection line for user defined alarms

- Automatically created result files with time stamp for recording measuring results, error messages and (optional) NIR spectra
- Real time trend chart for simultaneous monitoring of measuring results
- Interfaces for data exchange with PCS: RS232 interface/RK512 protocol; MPI interface; NIRLinePLC interface: ASCII protocol (RS232).
- Automatic check instrument during routine operation and a comprehensive service diagnostic software tool guarantee reliable operation and easy maintenance of the entire NIR on-line system.
- High stability and robustness because of using a programmable logic controller (PLC): active control of the hardware components; long-lasting PLC components; state-of-the-art in process control engineering

Accessory components:

The accessory components consist of process probes for integration into pipes and reactors, optical multiplexers for analysing up to 16 measuring points and high quality, OH-low silica light fibres for bridging distances of up to 100 m.

Application

In a distillation plant for separation of benzene and its homologues, three process streams (xylene, toluene, C9-C10 aromatics) should be monitored by a quantitative analysis with a maximum of seven components per process stream.

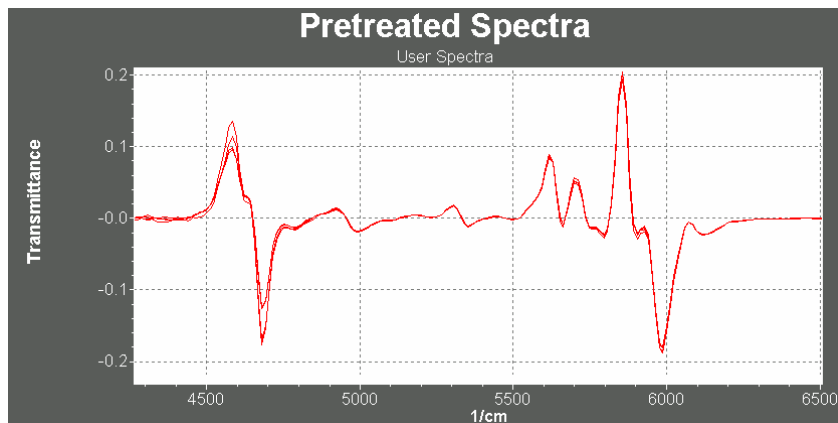


Figure 2. NIR spectra (first derivative) for the xylene process stream.

Set-up

Installed equipment: probe for each process stream integrated in a bypass; ex-proved, optical multiplexer; light fibres for bridging distances of up to 45 m. The predicted data values are visualised in the process control room.

Calibration

To involve environmental influences such as temperature (winter/summer) and changing composition of the incoming product streams the calibration models were updated along a year. The reference values for the calibration samples were determined by gas chromatography.

As an example for the multicomponent analysis - NIR calibration data for the separation of *o*-, *m*- and *p*-xylene in the xylene process stream are listed in the table below. The calibration models were validated by using a test set of samples, which were not included in the calibration set.

Table 1. Calibration data for *ortho*-, *meta*-, *para*-xylene in the xylene process stream (testseries)

Product parameter	Number of calibration/validation samples	Calibration range in %	Regression coefficient for calibration / validation set	Standard deviation for calibration / validation set in %
<i>o</i> -xylene	157 / 65	6.0-19	0.99 / 0.99	0.18 / 0.18
<i>m</i> -xylene	174 / 70	39-49	0.99 / 0.99	0.16 / 0.17
<i>p</i> -xylene	158 / 63	10-23	0.99 / 0.99	0.10 / 0.12

Routine operation

The process streams are monitored every two hours; the analysing time is below two minutes. Handling and maintenance during routine operation is quite easy: performance of a check instrument test in irregular intervals (> 14 days); reference measurements within intervals of 4–12 weeks; lamp change as sole maintenance work.

Summary

The robustness, easy-to-use and reliability of the introduced NIR on-line instrumentation, predestinate it as a process analyser for the chemical industry.

Since no time consuming lab analysis is anymore necessary the user benefits from fast analysing times and low operating costs.

There are additional economical advantages because complex multi-component analysis can easily be performed by evaluation of just a single NIR spectrum. Moreover—information about physical parameters, for example, density, particle size, distillation points can also be obtained from NIR spectra.