

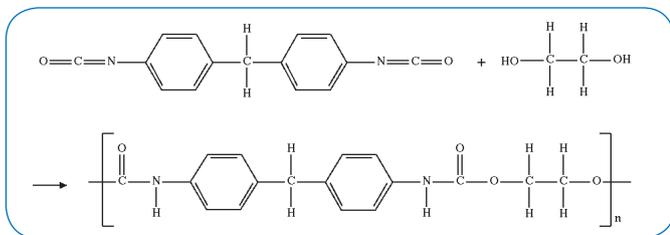


## Application Note AN N521

# On-line Monitoring of NCO Content during Polyurethane Production

Polyurethanes are extremely popular due to their diversity in usage. No other plastic group opens up such a wide range of applications. Depending on the starting material used, it is possible to obtain linear or crosslinked polyurethanes which are used in various applications in foams, elastomers, lacquers, insulation, adhesives, fibers, etc.

Polyurethanes are formed by polyaddition of bi- or higher-functional alkanols and isocyanates with a catalyst and various additives in a reactor.



The first step is to produce a precursor to the final product by reaction of a polyol with an excess of an isocyanate, resulting in an isocyanate-terminated pre-polymer.

Here, the ratio of the reactive groups of the isocyanate and the polyol need to be closely measured during this process to ensure that the resulting product has an excess of reactive isocyanate left over for post-processing in order to complete the reaction.

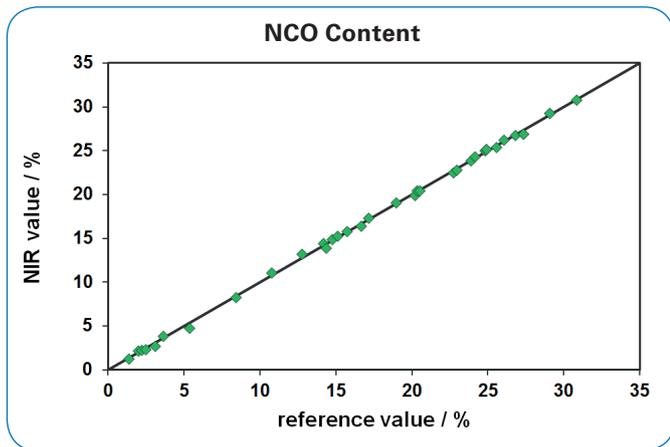
These reactive isocyanate groups that remain are measured as “%NCO”. This value is important for the next processing step to calculate the correct amount of curing agent needed to complete the reaction and produce high quality polyurethane materials.

However, isocyanates are potentially hazardous materials and require extremely careful handling, making the sampling difficult for analysis. An in-line approach would minimize the OSH risk involved in pulling samples. Moreover, a 24/7 control of the process leads to a better process understanding and reduces the risk of potential rework or disposal costs.

### FT-NIR Measurement

Since the pre-polymer is typically a transparent liquid, it can be easily analyzed using a fiber optic transmission probe. Such probes are available for temperatures up to 300°C or higher as well as in different lengths and equipped with different kind of flanges. With the MATRIX-F process spectrometer up to six measuring points can be monitored with only one spectrometer optimizing the return on investment of such a project.

Process communication protocols like Profibus DP, Modbus, OPC DA or 4-20mA allow the transfer of all necessary information to the DCS for optimized process control.



Cross validation results of a PLS based model for the online prediction of NCO content during Polyurethane reaction



The MATRIX-F FT-NIR spectrometer is designed to easily fit into 19" racks and enclosures. The spectrometer for this experiment was housed in a temperature controlled enclosure 100 m from the sample point along with a touch-screen computer. The results are continuously transmitted to the Process Control system via a 4-20mA interface and the reaction is controlled by a closed loop based on the NIR results.

## Quantitative Analysis

Near-infrared spectra result from combination and overtone bands of C-H, N-H, and O-H vibrations. Since the reaction mixtures contain organic components containing these bonds, they are ideal for near-infrared analysis.

The OPUS/QUANT quantitative analysis software package uses partial least squares (PLS) to develop quantitative models. Typically the development of a model requires measuring samples that contain a range of concentrations of the components of interest. In this example, NIR spectra of the reaction mixture were collected continuously in a pilot plant and correlated via their time stamp to samples pulled from the reactor and analyzed off-line.

## About Bruker Optics

Bruker Optics offers a wide variety of instrumentation to meet your specific needs:

- The TANGO offers an efficient and cost-effective analysis for material identification and quantification of constituents in a wide variety of application fields.
- The MPA II (Multi Purpose Analyzer) is ideal as a laboratory or at-line system for quality control. It offers a complete solution for all analysis sampling needs, including liquids, and powders.
- The MATRIX-F is a rugged FT-NIR spectrometer ideally suited to on-line/in-line process monitoring of liquids and solids based on fibre optic probe technologies.

**FT-NIR Spectrometers:** Bruker Optics offers various FT-NIR spectrometer models for lab, at-line and on-line applications:

**TANGO**

FT-NIR analyzer for routine use in the lab.

**MPA II**

Multi Purpose Analyzer for maximum flexibility.

**MATRIX-I**

At-line analysis with optional NEMA4/IP66 protection.

**MATRIX-F**

Process monitoring with probes and measurement heads.

### ● Bruker Optik GmbH

Ettlingen · Deutschland  
 Phone +49 (7243) 504-2000  
 Fax +49 (7243) 504-2050  
 info.bopt.de@bruker.com

[www.bruker.com/optics](http://www.bruker.com/optics)

### Bruker Optics Inc.

Billerica, MA · USA  
 Phone +1 (978) 439-9899  
 Fax +1 (978) 663-9177  
 info.bopt.us@bruker.com

### Bruker Shanghai Ltd.

Shanghai · China  
 Phone +86 21 51720-890  
 Fax +86 21 51720-899  
 info.bopt.cn@bruker.com