



Application Note AN-NIR-068

Isocyanates quality control with NIRS

Fast, chemical-free determination of isocyanate content for polyurethane production

Analyzing NCO content in isocyanates as raw materials for polyurethane (PU) is crucial to control properties of the final polymer. Wet analytical methods such as those used in ASTM D7252 are challenging due to the reactivity of these organic species with atmospheric moisture, as well as their toxicity. Furthermore, analysis of NCO content with HPLC (high-performance liquid chromatography)

involves many sample preparation steps and chemical reagents, with each measurement taking up to 20 minutes to complete.

This Application Note demonstrates that near-infrared spectroscopy (NIRS) provides a reagent-free and faster alternative to determine NCO content in isocyanates. The OMNIS NIR Analyzer Liquid analyzes isocyanates in a few seconds without any sample preparation.

EXPERIMENTAL EQUIPMENT

Liquid samples of isocyanates were measured with an OMNIS NIR Analyzer Liquid in transmission mode over a wavelength range of 1000–2250 nm. Reproducible spectrum acquisition was achieved using the built-in temperature control at 30 °C. For convenience, disposable vials with a path length of 8 mm were used. OMNIS Software was used for all data acquisition and prediction model development.



Figure 1. OMNIS NIR Analyzer and a sample filled in a disposable vial.

Table 1. Hardware and software equipment overview.

Equipment	Article number
OMNIS NIR Analyzer Liquid	2.1070.0010
Holder OMNIS NIR, vial, 8 mm	6.07401.070
Disposable vial, 8 mm, transmission	6.7402.240
OMNIS Stand-Alone	6.06003.010
Software license Quant Development	6.06008.002

RESULT

The NIR spectra (**Figure 2**), along with the corresponding reference values, were used to create a prediction model for isocyanate quantitative analysis. The quality of the prediction model was evaluated using the correlation diagram shown in **Figure 3**

which displays a very high correlation between the NIR prediction and the measured values using HPLC. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

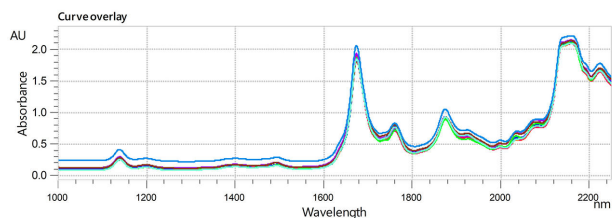


Figure 2. Stacked collection of NIR spectra from isocyanate samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT NCO CONTENT IN ISOCYANATES

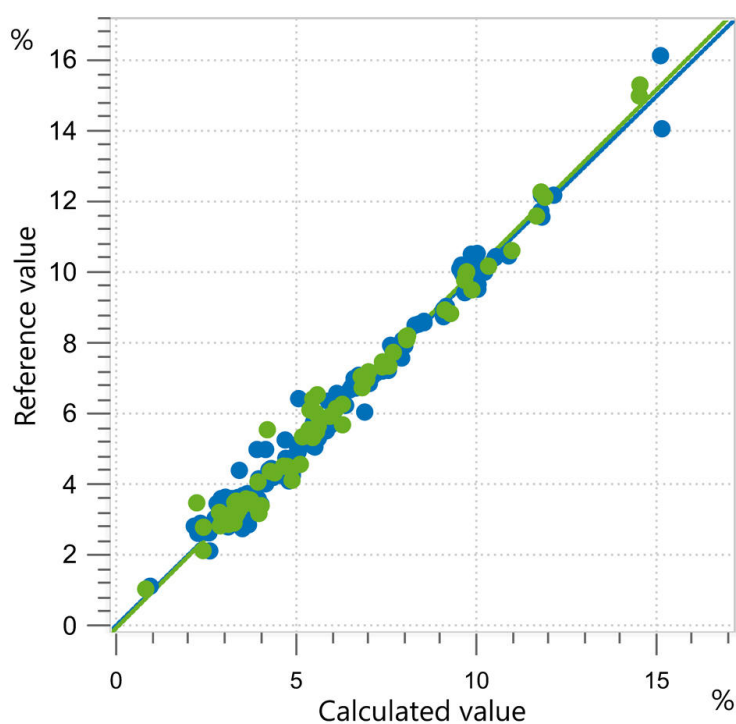


Figure 3. Correlation diagram and the respective figures of merit for the prediction of isocyanate content in polyurethane raw materials using an OMNIS NIR Liquid Analyzer. HPLC was used to evaluate isocyanate group content (% NCO) lab values.

R^2	SEC (%)	SECV (%)	SEP (%)
0.983	0.30	0.33	0.39

This Application Note shows the feasibility of using NIR spectroscopy for the quality control of isocyanates for PU production. NCO content can be determined in only a few seconds without any sample preparation. Compared to wet chemical methods (Table 2), the

time to result is a major advantage of NIR spectroscopy. Also, no chemicals are required, helping to better adhere to environmental, chemical, and health regulations.

Table 2. Time to result overview for the analysis of isocyanate content with HPLC.

Parameter	Method	Time to result
Isocyanate content	HPLC	20 min (preparation) + 20 min (HPLC)

CONTACT

Metrohm Deutschland
In den Birken 3
70794 Filderstadt

info@metrohm.de

CONFIGURATION



OMNIS NIR Analyzer Liquid

Nahinfrarot-Spektrometer für Flüssigproben.

Der OMNIS NIR Analyzer ist die nach Schweizer Qualitätsstandards entwickelte und produzierte Nahinfrarot-Spektroskopie (NIRS) Lösung für die Routineanalytik entlang der gesamten Produktionskette. Die Nutzung neuester Technologien und die Einbindung in die moderne OMNIS Software spiegeln sich in der Geschwindigkeit, der Bedienbarkeit und dem flexiblen Einsatz dieser NIR-Spektrometer wider.

Die Vorteile des OMNIS NIR Analyzer Liquid im Überblick:

- Messungen von Flüssigproben in weniger als 10 Sekunden
- Temperaturkontrolle an der Probe von 25°C – 80°C
- Automatische Erkennung des Einsetzen und der Entnahme des Probengefäßes
- Einfache Einbindung in ein Automationssystem oder Verknüpfung mit weiteren Analysetechnologien (Titration)
- Unterstützung zahlreicher Probengefäße mit unterschiedlicher Pfadlänge



Halter OMNIS NIR, Vial, 8 mm

Vialhalter für den OMNIS NIR Analyzer für 8 mm Einwegvials (6.7402.240).



OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Einwegvial, 8 mm, Transmission, Anz. 100

100 Einwegvials aus Glas (Borosilikat) mit einer optischen Pfadlänge von 8 mm für Analysen von Flüssigkeiten in Transmission. Die Einwegvials werden mit den zugehörigen Verschlussstopfen (Stückzahl = 100) geliefert.

Kompatibel mit:

- Halter OMNIS NIR, Vial, 8 mm (6.07401.070)
- DS2500 Halter für 8 mm Einwegvials (6.7492.020)

OMNIS Stand-Alone Lizenz

Ermöglicht den Stand-Alone Betrieb der OMNIS Software auf einem Windows™ Computer.

Merkmale:

- Die Lizenz enthält bereits eine OMNIS Geräte Lizenz.
- Muss über das Metrohm Lizenzierungsportal aktiviert werden.

Softwarelizenz Quant Development

Softwarelizenz für die Erstellung und Bearbeitung von Quantifizierungsmodellen in einer Stand-Alone OMNIS Software Installation.