



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.

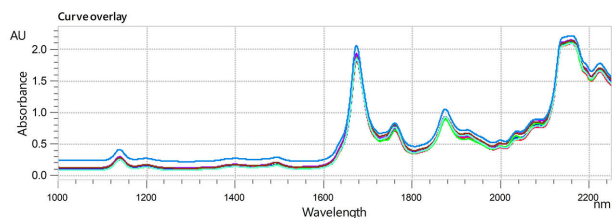


Figure 2. Stacked collection of NIR spectra from isocyanate samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT NCO CONTENT IN ISOCYANATES

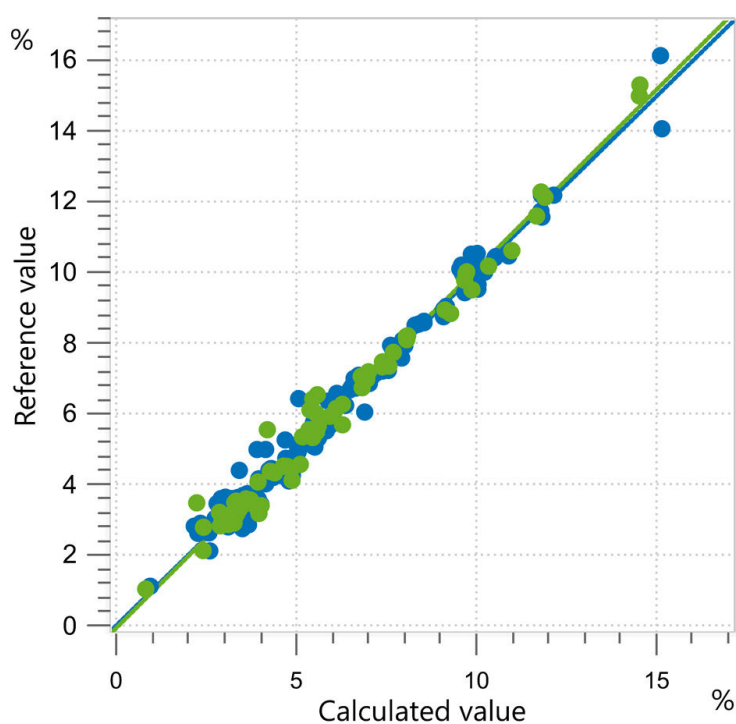


Figure 3. Correlation diagram and the respective figures of merit for the prediction of isocyanate content in polyurethane raw materials using an OMNIS NIR Liquid Analyzer. HPLC was used to evaluate isocyanate group content (% NCO) lab values.

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

CONCLUSION

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

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CONFIGURATION



OMNIS NIR Analyzer Liquid

Spettrometro nel vicino infrarosso per campioni liquidi.

OMNIS NIR Analyzer è la soluzione per spettroscopia nel vicino infrarosso (NIRS) sviluppata e prodotta secondo gli standard di qualità svizzeri utilizzabile per l'analisi di routine lungo l'intera catena di produzione. L'uso delle più recenti tecnologie e l'integrazione nel moderno OMNIS Software si riflettono nella velocità, operatività e flessibilità d'uso di questo spettrometro NIR.

Panoramica dei vantaggi di OMNIS NIR Analyzer Liquid:

- Misurazioni di campioni liquidi in meno di 10 secondi
- Controllo della temperatura sul campione da 25 °C a 80 °C
- Rilevamento automatico dell'inserimento e del prelievo del contenitore portacampione
- Facile integrazione in un sistema di automazione o collegamento con altre tecnologie di analisi (titolazione)
- Supporto di numerosi contenitori portacampione con diverse lunghezze del cammino



Supporto OMNIS NIR, vial, 8 mm

Supporto per vial per OMNIS NIR Analyzer per vial monouso da 8 mm (6.7402.240).



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Vial monouso, 8 mm, trasmissione, num. 100

100 vial monouso in vetro (borosilicato) con lunghezza del cammino ottico di 8 mm per l'analisi di liquidi in trasmissione. I vial monouso sono forniti con i relativi tappi di chiusura (numero di pezzi = 100).

Compatibile con:

- Supporto OMNIS NIR, vial, 8 mm (6.07401.070)
- DS2500 holder per vial usa e getta da 8 mm (6.7492.020)

Licenza OMNIS Stand-Alone

Consente l'utilizzo stand-alone del software OMNIS su un computer Windows™.

Caratteristiche:

- la licenza contiene già una licenza per strumenti OMNIS.
- Deve essere attivata tramite il portale licenze Metrohm.

Licenza software Quant Development

Licenza software per la creazione e l'elaborazione di modelli di quantificazione in un'installazione Stand-Alone di OMNIS Software.