



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.

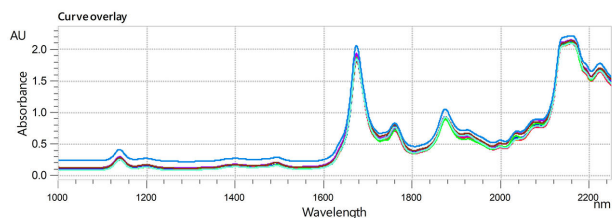


Figure 2. Stacked collection of NIR spectra from isocyanate samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT NCO CONTENT IN ISOCYANATES

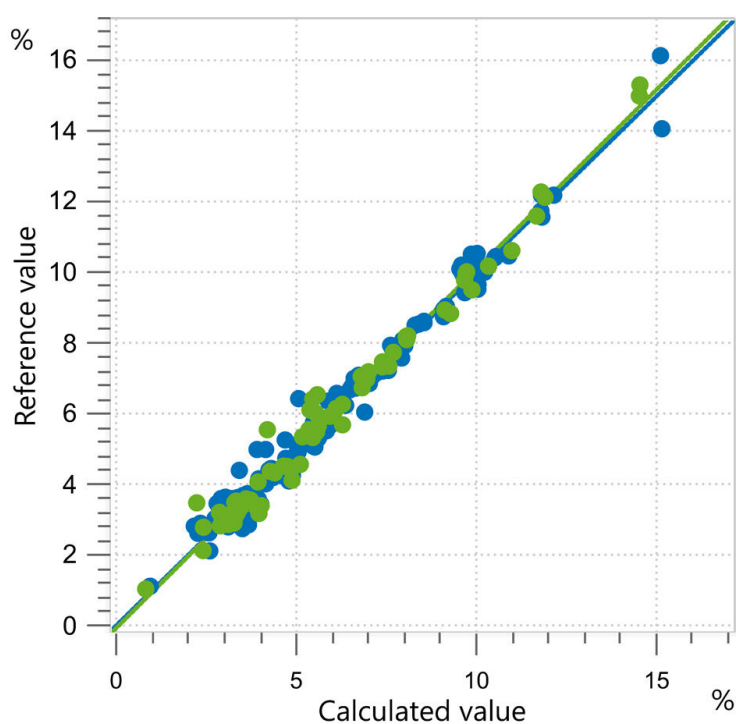


Figure 3. Correlation diagram and the respective figures of merit for the prediction of isocyanate content in polyurethane raw materials using an OMNIS NIR Liquid Analyzer. HPLC was used to evaluate isocyanate group content (% NCO) lab values.

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)

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CONFIGURATION



OMNIS NIR Analyzer Liquid

Spectromètre proche infrarouge pour échantillons liquides.

L'OMNIS NIR Analyzer est la solution de spectroscopie proche infrarouge (NIRS) développée et produite selon les normes de qualité suisses pour les analyses de routine tout au long de la chaîne de fabrication. L'utilisation des technologies les plus récentes et l'intégration dans le logiciel OMNIS moderne se reflètent dans la vitesse, la facilité d'utilisation et la flexibilité d'utilisation de ces spectromètres NIR.

Vue d'ensemble des avantages de l'OMNIS NIR Analyzer Liquid :

- Mesures d'échantillons liquides en moins de 10 secondes
- Contrôle de la température sur l'échantillon de 25 °C à 80 °C
- Détection automatique de l'insertion et du retrait d'échantillons du récipient d'échantillon
- Intégration simple dans un système d'automatisation ou liaison avec d'autres technologies d'analyse (titrage)
- Prise en charge de nombreux récipients d'échantillon de différentes longueurs de chemin

Support OMNIS NIR, flacon, 8 mm

Support de flacon pour l'OMNIS NIR Analyzer pour flacons à usage unique de 8 mm (6.7402.240).





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Flacon à usage unique, 8 mm, transmission, Qté 100
100 flacons en verre (borosilicate) à usage unique avec une longueur de chemin de 8 mm pour des analyses de liquides lors d'une transmission. Les flacons à usage unique sont fournis avec les bouchons de fermeture correspondants (quantité = 100).

Compatible avec :

- Support OMNIS NIR, flacon, 8 mm (6.07401.070)
- Support DS2500 pour flacons à usage unique 8 mm (6.7492.020)

Licence OMNIS autonome

Elle permet l'exploitation autonome du logiciel OMNIS sur un ordinateur Windows™.

Caractéristiques :

- La licence comprend déjà une licence pour appareils OMNIS.
- Elle doit être activée via le portail d'octroi de licences Metrohm.

Licence logicielle Quant Development

Licence logicielle pour la création et l'édition de modèles de quantification dans une installation du logiciel OMNIS Stand-Alone.