



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

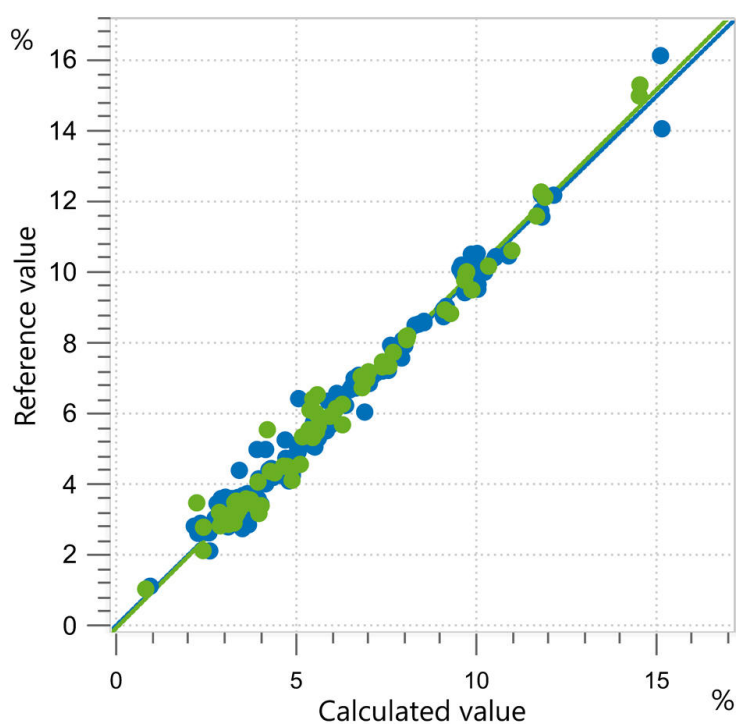


Figure 3. Correlation diagram and the respective figures of merit for the prediction of isocyanate content in polyurethane raw materials using an OMNIS NIR Liquid Analyzer. HPLC was used to evaluate isocyanate group content (% NCO) lab values.

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

メトロームジャパン株式会
社

143-0006 東京都大田区平
和島6-1-1

null 東京流通センター アネ
ックス9階

metrohm.jp@metrohm.jp

CONFIGURATION



OMNIS NIR Analyzer Liquid

Near-infrared spectrometer for liquid samples. Developed and produced in accordance with Swiss quality standards, the OMNIS NIR Analyzer is the near-infrared spectroscopy (NIRS) solution for routine analysis along the entire production chain. Its application of the latest technologies and its integration in the modern OMNIS Software are reflected in its speed, operability, and flexible utilization of this NIR spectrometer. Overview of the advantages of the OMNIS NIR Analyzer Liquid: Measurements of liquid samples in less than 10 seconds; Temperature control on the sample from 25–80 °C; Automatic detection of the insertion and removal of the sample vessel; Simple integration in an automation system or link with additional analysis technologies (titration); Supports numerous sample vessels with different path lengths;



OMNIS NIR8 mm

Vial Holder for the OMNIS NIR Analyzer for 8 mm disposable vials (6.7402.240).



8 mm100

透過した液体を分析するための光学的距離 8 mm のガラス製 (ホウケイ酸塩) 使い捨てバイアル 100 個。使い捨てバイアルには閉じ蓋 (個数 = 100) が付属されています。

次の製品と互換性があります:

- ホルダー OMNIS NIR、バイアル、8 mm (6.07401.070)
- 使い捨てバイアル 8 mm 用の DS2500 ホルダー (6.7492.020)

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WindowsTMコンピュータ上のOMNISソフトウェアをスタンドアローン操作することが可能になります。

特徴:

- ライセンスには、既に1つのOMNISデバイスライセンスが含まれています。
- メトローム・ライセンシングポータルにて、アクティブ化する必要があります。

Quant Development

Software license for the creation and editing of quantification models in a stand-alone OMNIS Software installation.