



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

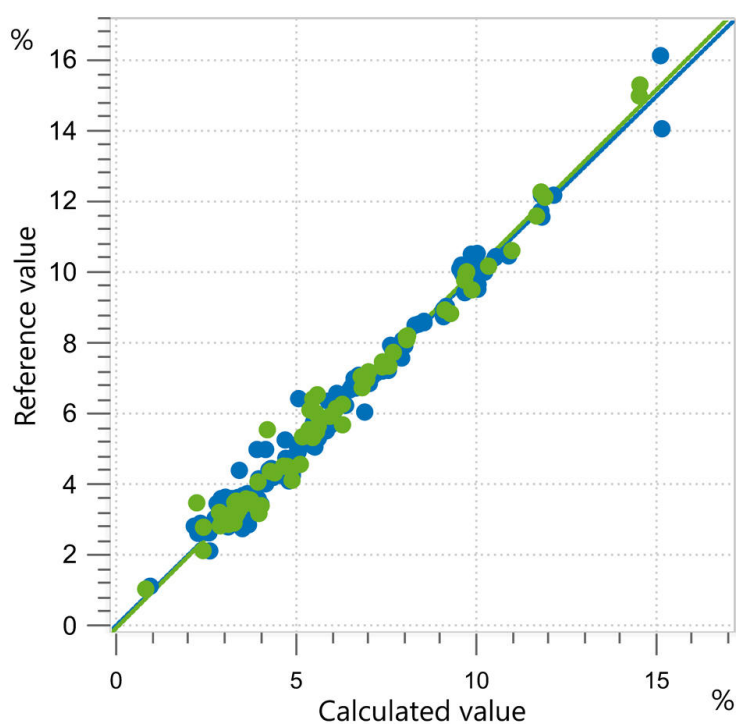


Figure 3. Correlation diagram and the respective figures of merit for the prediction of isocyanate content in polyurethane raw materials using an OMNIS NIR Liquid Analyzer. HPLC was used to evaluate isocyanate group content (% NCO) lab values.

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 适合液体样品的近红外光谱仪。

OMNIS NIR Analyzer 是一种按照瑞士质量标准开发和生产的近红外光谱 (NIRS) 解决方案,用于整个生产链的常规分析。使用新技术和嵌入先进 OMNIS Software 反应在该 NIR 光谱仪的速度、可操作性和灵活使用上。

OMNIS NIR Analyzer Liquid 的优点概览:

- 可在 10 秒以内测量液体样品
- 对 25° C – 80° C 的样品进行温度控制
- 自动识别样品容器的插入和取出
- 方便地嵌入自动系统,或者与其它分析技术(滴定)关联
- 支持大量不同路径长度的样品容器



OMNIS NIR8 mm

适合 8 mm 一次性小管的 OMNIS NIR Analyzer 的小管支架 (6.7402.240)。



8 mm 100

100 个玻璃(硼硅)一次性样品瓶,具有 8 mm 的光路长度,适用于分析透射中的液体。一次性样品瓶交付时带有所属的螺旋塞(件数 = 100)。

兼容:

- 支架 OMNIS NIR,小管,8 mm (6.07401.070)
- DS2500 支架适用于 8 mm 一次性样品瓶 (6.7492.020)

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