



NIR APPLICATION NOTE NIR-068

Quality Control of Isocyanates

Chemical-free and fast determination of isocyanate content

Determination of isocyanates (ASTM D7252) is a challenging procedure due to the reactivity of these organic species with atmospheric moisture, as well as their toxicity. Furthermore, HPLC analysis typically used for this kind of analysis involves sample preparation steps and chemicals, with each measurement taking up to 20 minutes to complete.

This application note demonstrates that the XDS RapidLiquid Analyzer operating in the visible and near infrared spectral region (Vis-NIR) provides a **chemical-free and fast solution** for determination of isocyanate content. With **no sample preparation needed**, Vis-NIR spectroscopy allows the analysis of isocyanates in **less than a minute**.

EXPERIMENTAL EQUIPMENT

Isocyanate samples were measured with a XDS RapidLiquid Analyzer in transmission mode over the full wavelength range (400–2500 nm). Reproducible spectrum acquisition was achieved using the built-in temperature control (at 30°C) of the XDS RapidLiquid Analyzer. For convenience, disposable vials with a path length of 8 mm were used, which made cleaning of the sample vessels unnecessary. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

Table 1. Hardware and software equipment overview.

Equipment	Metrohm number
XDS RapidLiquid Analyzer	2.921.1410
Disposable Vials, 8 mm diameter, transmission	6.7402.010
Vision Air 2.0 Complete	6.6072.208



Figure 1. XDS RapidLiquid Analyzer and an isocyanate sample present in the 8 mm Disposable Vial.

RESULT

The obtained Vis-NIR spectra (**Figure 2**) were used to create prediction models for quantification of the isocyanate content. The quality of the prediction models was evaluated using correlation diagrams, which display the relationship between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

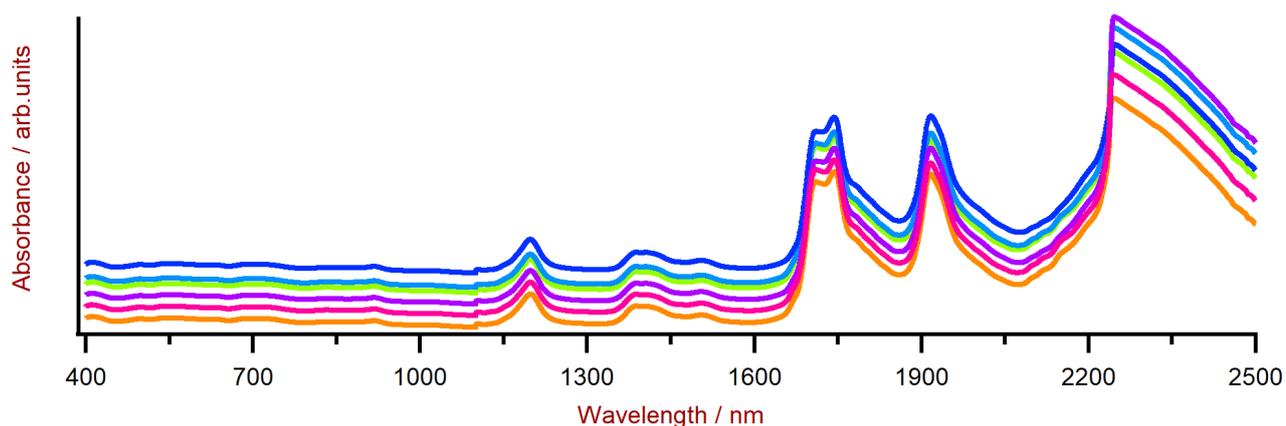
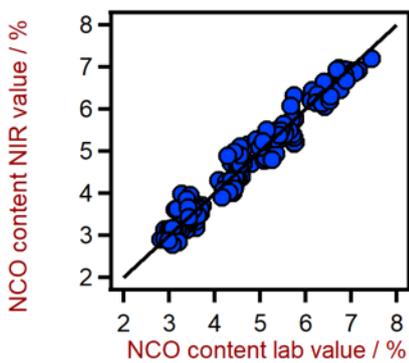


Figure 2. Selection of Isocyanate Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm Disposable Vials. For display reasons a spectra offset was applied.

RESULT ISOCYANATE CONTENT



Figures of Merit	Value
R ²	0.968
Standard Error of Calibration	0.24
Standard Error of Cross-Validation	0.26

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the isocyanate (NCO) content using a XDS RapidLiquid Analyzer. The isocyanate content lab value was evaluated using titration.

CONCLUSION

This study demonstrates the feasibility of NIR spectroscopy for the analysis of isocyanates. In comparison to wet chemical methods (Table 2), the

time to result is a major advantage of NIR spectroscopy, since the isocyanate content is determined **with no sample preparation needed in less than a minute.**

Table 2. Time to result overview for the different parameters

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

Analytes: Isocyanate content
Matrix: Isocyanate
Industry: Chemical industry
Standards: ASTM E1655