



Application Note AN-NIR-068

Quality Control of Isocyanates

Chemical-free and fast determination of isocyanate content with NIRS

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.



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

Table 1. Hardware and software equipment overview.

Equipment	Metrohm number
XDS RapidLiquid Analyzer	2.921.1410
Disposable Vials, 8 mm diameter, transmission	6.7402.000
Vision Air 2.0 Complete	6.6072.208

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.

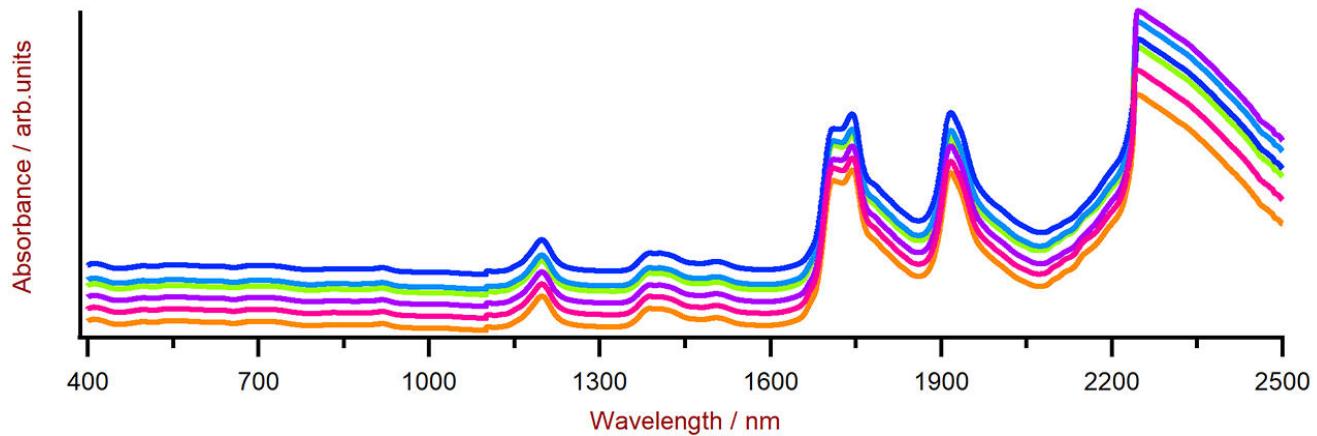


Figure 2. Selection of Isocyanate Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm Disposable Vials. For display reasons a spectra offset was applied.

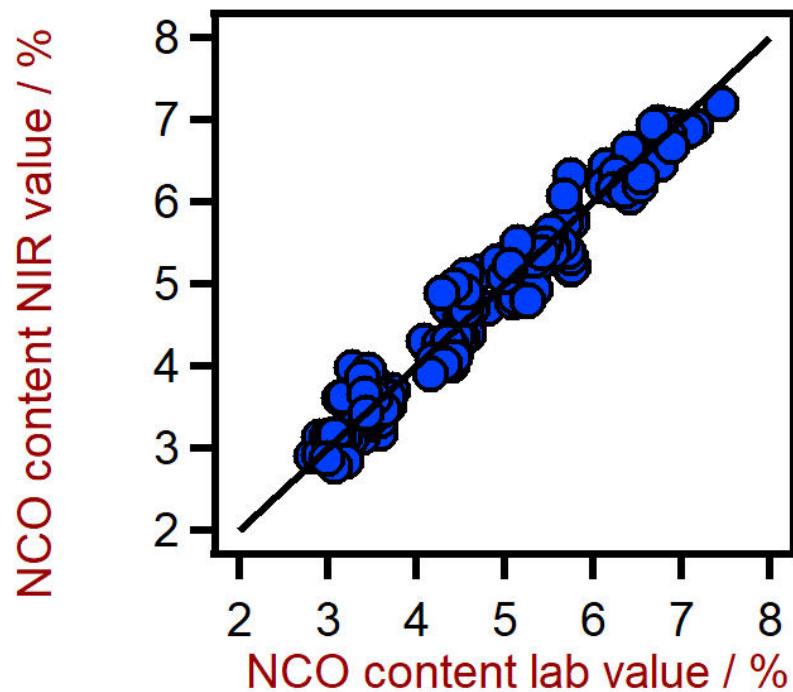


Figure 3. Correlation diagram for the prediction of the isocyanate (NCO) content using a XDS RapidLiquid Analyzer. The isocyanate content lab value was evaluated using titration.

Table 2. Figures of merit for the prediction of the isocyanate (NCO) content using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.968
Standard error of calibration	0.24
Standard error of cross-validation	0.26

CONCLUSION

This study demonstrates the feasibility of NIR spectroscopy for the analysis of isocyanates. In comparison to wet chemical methods (Table 3), 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 3. Time to result overview for the different parameters

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

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EXPERIMENTAL EQUIPMENT



NIRS XDS RapidLiquid Analyzer

Rapid, precise analyses of liquids and suspensions of all types.

The NIRS XDS RapidLiquid Analyzer enables rapid, precise analyses of liquid formulations and substances. Precise measurement results at the push of a button make the NIRS XDS RapidLiquid Analyzer an equally reliable and simple solution for quality monitoring in laboratories and processes. The samples are transferred to quartz cuvettes designed for multiple use or disposable glass vials; a tempered sample compartment ensures reproducible analysis conditions and thus accurate measurement results.



Vision Air 2.0 Complete

Vision Air - Universal spectroscopy software.

Vision Air Complete is a modern and simple-to-operate software solution for use in a regulated environment.

Overview of the advantages of Vision Air:

- Individual software applications with adapted user interfaces ensure intuitive and simple operation
- Simple creation and maintenance of operating procedures
- SQL database for secure and simple data management

The Vision Air Complete version (66072208) includes all applications for quality assurance using Vis-NIR spectroscopy:

- Application for instrument and data management
- Application for method development
- Application for routine analysis

Additional Vision Air Complete solutions:

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)