



Application Note AN-NIR-035

Quality control of polyols with NIRS

Determination of hydroxyl number according to ASTM D6342

Polyols are a broad class of organic compounds characterized by the presence of two or more hydroxyl groups. Synthetic polyols with varying chemical and physical properties are often used as raw materials in the production of polyurethanes. Quality control of these raw materials is critical prior to polymerization to avoid creating out-of-specification products. Several quality parameters can be determined in polyols like hydroxyl value, acid value (or acid number), moisture content, and viscosity. Titration is usually preferred for the analysis (ASTM D4274 for hydroxyl number, and ASTM D4662

and ASTM D7253 for acid number). This time-consuming method requires reproducible sample preparation, additional toxic and corrosive chemicals, and waste disposal, resulting in elevated running costs. Near-infrared spectroscopy (NIRS) offers a safer, faster alternative for multiparameter analysis of polyols without any sample preparation or chemical reagents. NIRS is recognized by regulatory bodies as a tool for quality control and can be used for the determination of hydroxyl values of polyols (ASTM D6342).

EXPERIMENTAL EQUIPMENT

Samples of polyols were measured in an OMNIS NIR Analyzer Liquid at 35 °C in transmission mode (1000–2250 nm). Disposable vials with a pathlength of 8 mm were used for convenience. All data acquisition and prediction model development was performed with OMNIS Software.



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

The NIR spectra (**Figure 2**), along with the corresponding reference values, were used to create prediction models for quantification of viscosity, hydroxyl number, acid value, and moisture in polyol samples.

The quality of the prediction models was evaluated

using correlation diagrams (**Figures 3–6**). These diagrams display very high correlations between the NIR predictions and the measured values of different parameters using wet chemical methods. The respective figures of merit (FOM) display the expected precision during routine analysis.

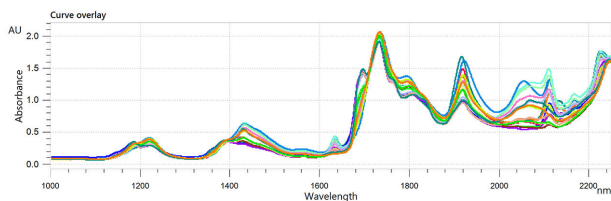


Figure 2. Stacked collection of NIR spectra from polyol samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT HYDROXYL NUMBER IN POLYOLS

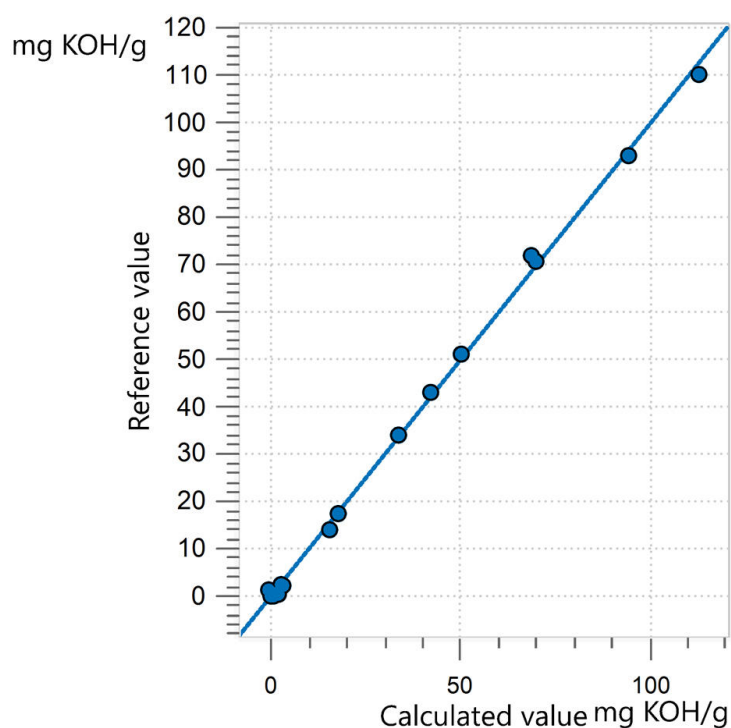


Figure 3. Correlation diagram and the respective figures of merit for the prediction of the hydroxyl number in polyols using an OMNIS NIR Analyzer Liquid. The hydroxyl number lab values were evaluated using titration.

R^2	SEC (mg KOH/g)	SECV (mg KOH/g)
0.998	1.17	1.42

RESULT ACID NUMBER IN POLYOLS

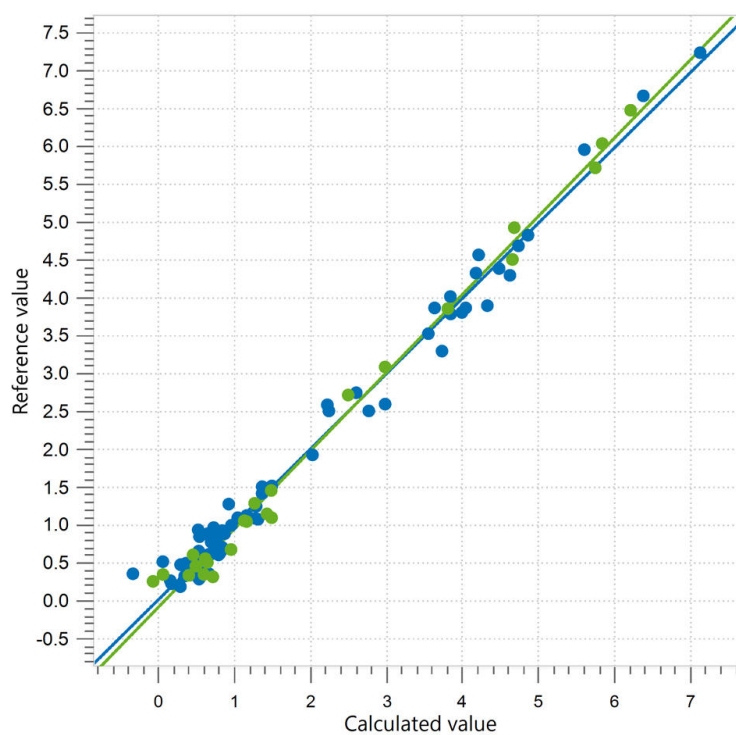


Figure 4. Correlation diagram and the respective figures of merit for the prediction of polyol acid number using an OMNIS NIR Analyzer Liquid. Titration was used to determine the laboratory acid number values.

R^2	SEC (mg KOH/g)	SECV (mg KOH/g)	SEP (mg KOH/g)
0.990	0.13	0.20	0.21

RESULT MOISTURE CONTENT IN POLYOLS

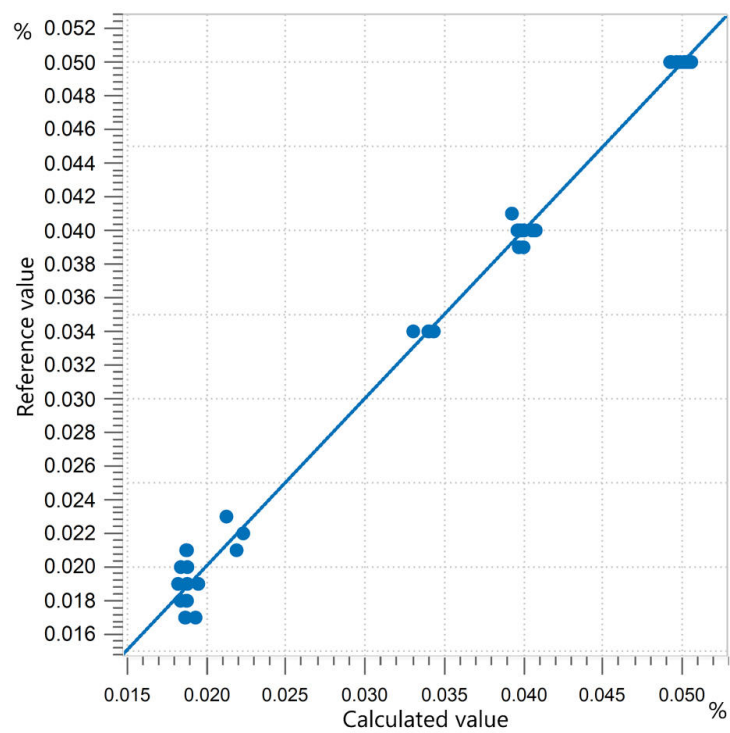


Figure 5. Correlation diagram and the respective figures of merit for the prediction of polyol water content using an OMNIS NIR Analyzer Liquid. Karl Fischer titration was used to determine the laboratory values for moisture content in polyols.

R ²	SEC (ppm)	SECV (ppm)
0.994	9	10

RESULT VISCOSITY IN POLYOLS

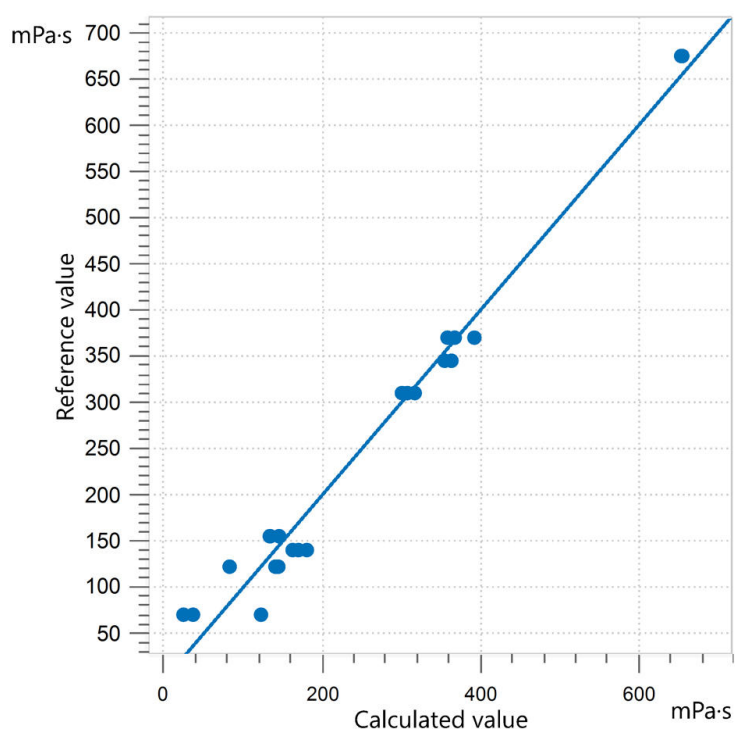


Figure 6. Correlation diagram and the respective figures of merit for the prediction of viscosity in polyols using an OMNIS NIR Analyzer Liquid. The viscosity lab values were evaluated with a viscometer.

R ²	SEC (mPa·s)	SECV (mPa·s)
0.978	20.25	25.59

CONCLUSION

This Application Note shows the feasibility of using NIR spectroscopy for the analysis of hydroxyl number, moisture, acid number, and viscosity in polyols.

Compared to wet chemical methods, running costs are significantly lower when using NIR spectroscopy (Figure 7).

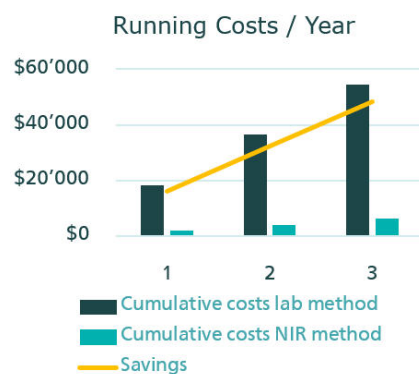


Figure 7. Comparison of running costs for the determination of the hydroxyl number with a standardized method (titration) and NIR spectroscopy.

	Lab method	NIR method
Number of analyses (per day)	10	10
Cost of operator (per hour)	\$25	\$25
Costs of consumables and chemicals OH-number	\$6	\$1
Time spent per analysis	5 min	1 min
Total running costs (per year)	\$18,188	\$2,063

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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



Holder OMNIS NIR, vial, 8 mm

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



Disposable vial, 8 mm, transmission, qty. 100

100 disposable glass vials (borosilicate) with an optical path length of 8 mm for analyses of liquids in transmission. The disposable vials are supplied with the associated stoppers (number of pieces = 100).

Compatible with:

- Holder OMNIS NIR, vial, 8 mm (6.07401.070)
- DS2500 holder for 8 mm disposable vials (6.7492.020)

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OMNIS Stand-Alone license

Enables stand-alone operation of the OMNIS software on a WindowsTM computer.

Features:

- The license already includes one OMNIS instrument license.
- Must be activated via the Metrohm licensing portal.

Software license Quant Development

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