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

RESULT

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.

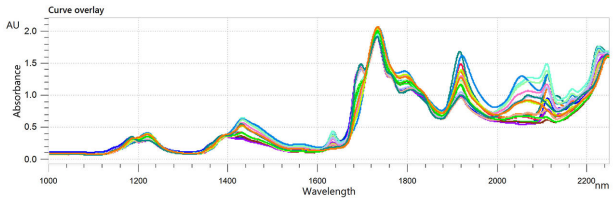

Figure 2. Stacked collection of NIR spectra from polyol samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT HYDROXYL NUMBER IN POLYOLS

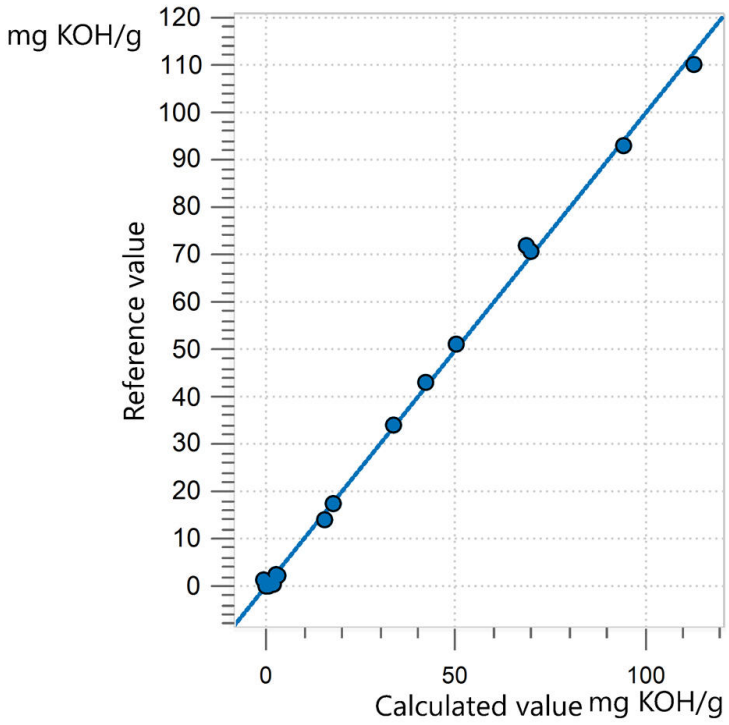

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the hydroxyl number in polyols using an OMNIS NIR Analyzer Liquid. The hydroxyl number lab values were evaluated using titration.

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

RESULT ACID NUMBER IN POLYOLS

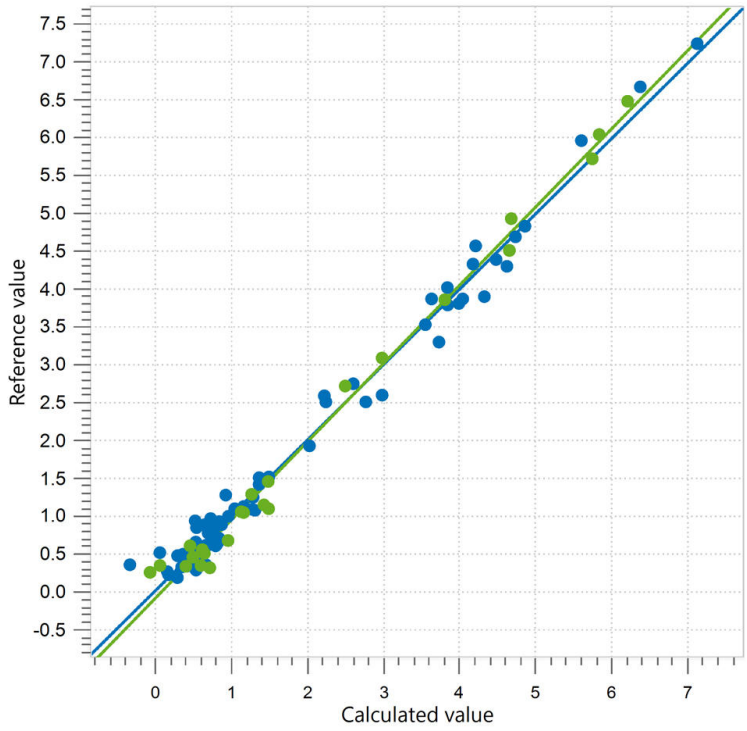

Figure 4. Correlation diagram and the respective figures of merit for the prediction of polyol acid number using an OMNIS NIR Analyzer Liquid. Titration was used to determine the laboratory acid number values.

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

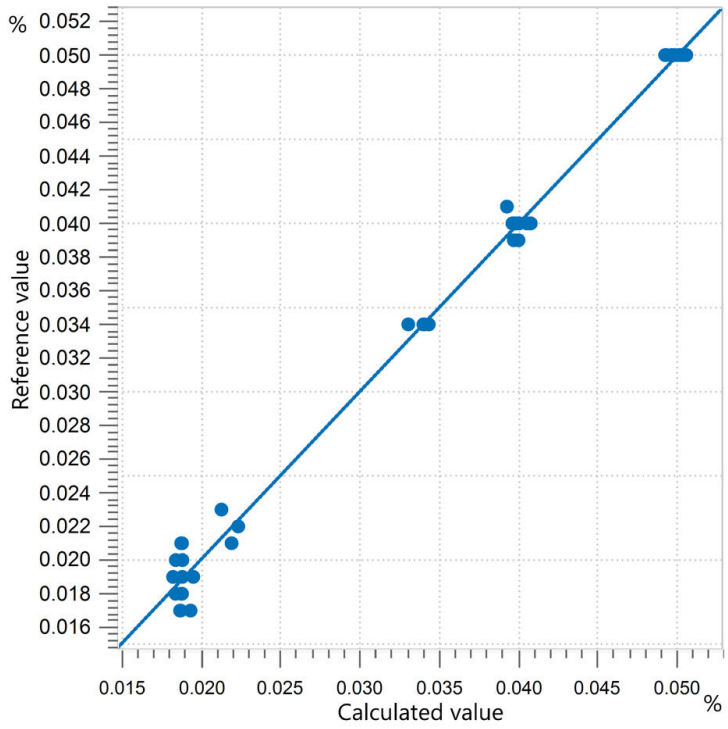

Figure 5. Correlation diagram and the respective figures of merit for the prediction of polyol water content using an OMNIS NIR Analyzer Liquid. Karl Fischer titration was used to determine the laboratory values for moisture content in polyols.

R^2	SEC (ppm)	SECV (ppm)
0.994	9	10

RESULT VISCOSITY IN POLYOLS

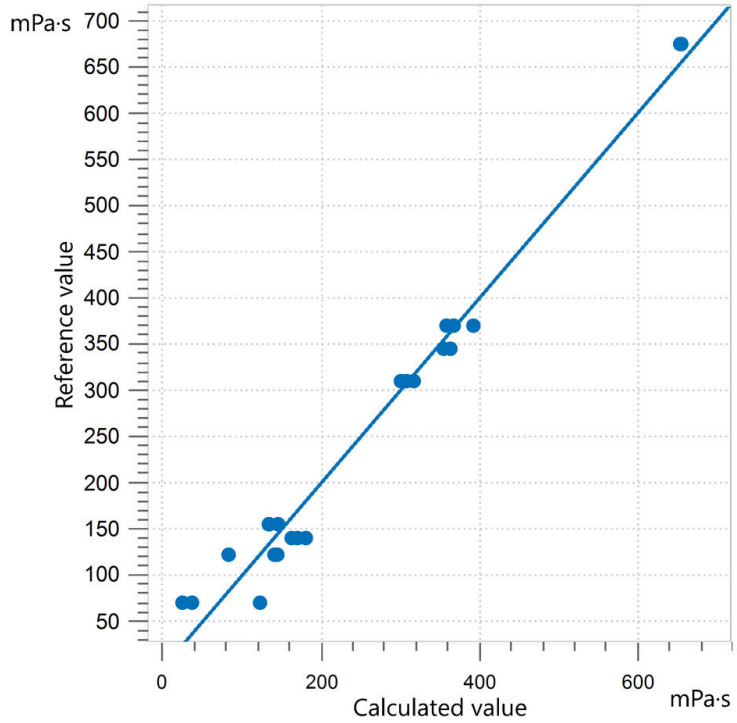

Figure 6. Correlation diagram and the respective figures of merit for the prediction of viscosity in polyols using an OMNIS NIR Analyzer Liquid. The viscosity lab values were evaluated with a viscometer.

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).

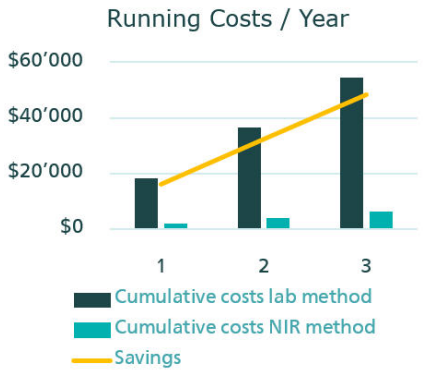

Figure 7. Comparison of running costs for the determination of the hydroxyl number with a standardized method (titration) and NIR spectroscopy.

	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

CONTACT

Metrohm Hispania
Calle Aguacate 15
28044 Madrid
mh@metrohm.es

CONFIGURATION

OMNIS NIR Analyzer Liquid

Espectrómetro del infrarrojo cercano para muestras líquidas.

El OMNIS NIR Analyzer es la solución de espectroscopía del infrarrojo cercano (NIRS) desarrollada y producida conforme a los estándares de calidad suizos para los análisis de rutina a lo largo de toda la cadena de producción. El empleo de las tecnologías más avanzadas y la integración en el moderno OMNIS Software se reflejan en la velocidad, la manejabilidad y el uso flexible de estos espectrómetros NIR.

Información general sobre las ventajas del OMNIS NIR Analyzer Liquid:

- Medidas de muestras líquidas en menos de 10 segundos
- Control de temperatura de la muestra: 25 °C...80 °C
- Detección automática de la colocación y la retirada del recipiente de muestras
- Fácil integración en un sistema de automatización o vinculación con otras tecnologías de análisis (titulación)
- Compatible con numerosos recipientes de muestras con distintas longitudes de recorrido

Soporte OMNIS NIR, vial, 8 mm

Soporte de viales para el OMNIS NIR Analyzer para viales desechables de 8 mm (6.7402.240).

Vial desechable, 8 mm, para transmisión, 100 unidades

100 viales desechables de vidrio (borosilicato) con una longitud de recorrido óptico de 8 mm para el análisis de líquidos en transmisión. Los viales desechables se suministran con los correspondientes tapones de cierre (100 unidades).

Compatibles con:

- Soporte OMNIS NIR para vial de 8 mm (6.07401.070)
- Soporte DS2500 para viales desechables de 8 mm (6.7492.020)

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Licencia "Stand-Alone" de OMNIS

Habilita el modo "Stand-Alone" del software OMNIS en un ordenador con WindowsTM.

Características:

- Se incluye una licencia de los aparatos OMNIS.
- Debe activarse en el portal de licencias de Metrohm.

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Licencia de software de Quant Development

Licencia de software para la creación y edición de modelos de cuantificación en una instalación de OMNIS Software "Stand-Alone".