



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.

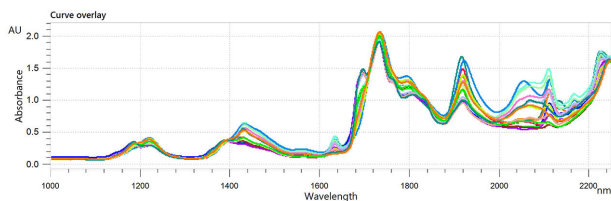


Figure 2. Stacked collection of NIR spectra from polyol samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT HYDROXYL NUMBER IN POLYOLS

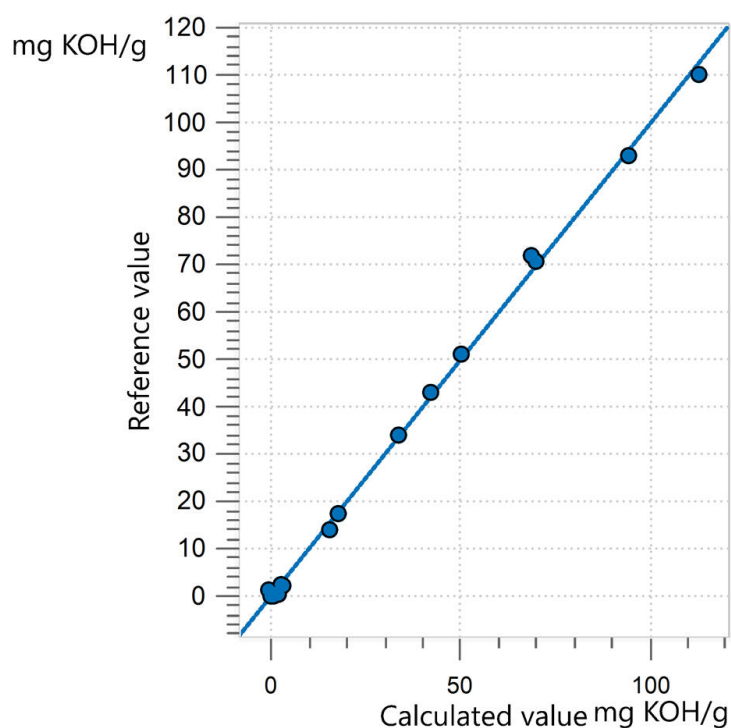


Figure 3. Correlation diagram and the respective figures of merit for the prediction of the hydroxyl number in polyols using an OMNIS NIR Analyzer Liquid. The hydroxyl number lab values were evaluated using titration.

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

RESULT ACID NUMBER IN POLYOLS

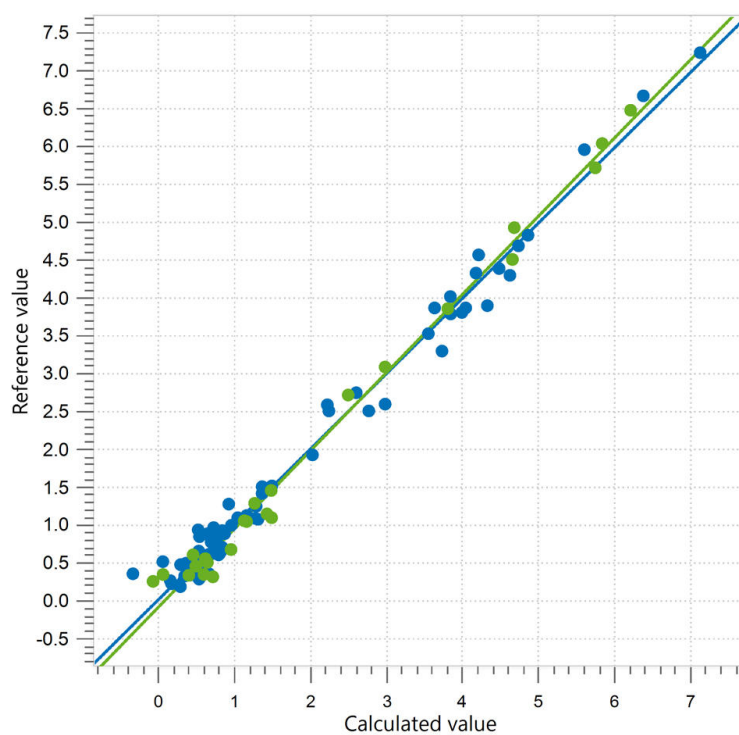


Figure 4. Correlation diagram and the respective figures of merit for the prediction of polyol acid number using an OMNIS NIR Analyzer Liquid. Titration was used to determine the laboratory acid number values.

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

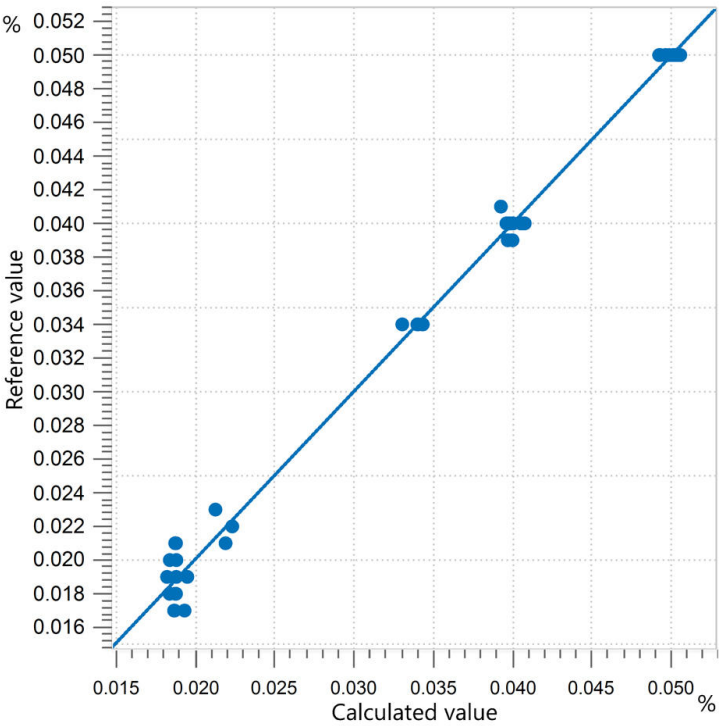


Figure 5. Correlation diagram and the respective figures of merit for the prediction of polyol water content using an OMNIS NIR Analyzer Liquid. Karl Fischer titration was used to determine the laboratory values for moisture content in polyols.

R ²	SEC (ppm)	SECV (ppm)
0.994	9	10

RESULT VISCOSITY IN POLYOLS

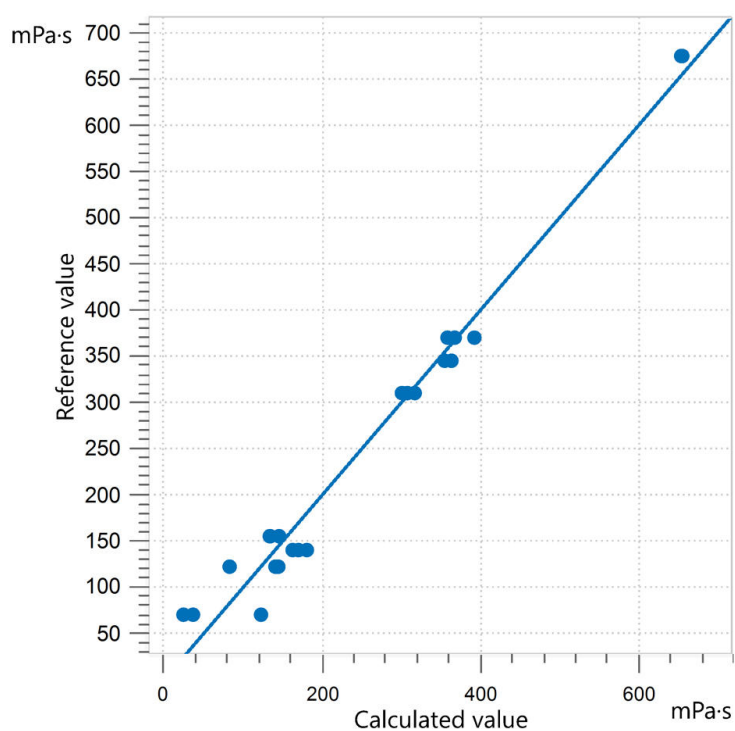


Figure 6. Correlation diagram and the respective figures of merit for the prediction of viscosity in polyols using an OMNIS NIR Analyzer Liquid. The viscosity lab values were evaluated with a viscometer.

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).

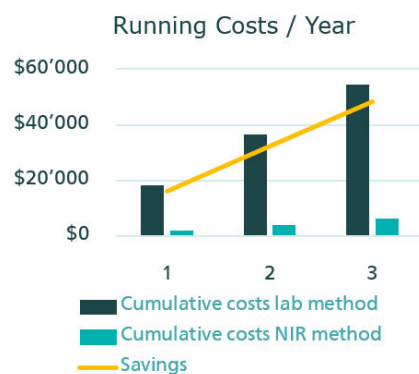


Figure 7. Comparison of running costs for the determination of the hydroxyl number with a standardized method (titration) and NIR spectroscopy.

	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 France
13, avenue du Québec - CS
90038
91978 VILLEBON
COURTABOEUF CEDEX

info@metrohm.fr

CONFIGURATION



OMNIS NIR Analyzer Liquid

Spectromètre proche infrarouge pour échantillons liquides.

L'OMNIS NIR Analyzer est la solution de spectroscopie proche infrarouge (NIRS) développée et produite selon les normes de qualité suisses pour les analyses de routine tout au long de la chaîne de fabrication. L'utilisation des technologies les plus récentes et l'intégration dans le logiciel OMNIS moderne se reflètent dans la vitesse, la facilité d'utilisation et la flexibilité d'utilisation de ces spectromètres NIR.

Vue d'ensemble des avantages de l'OMNIS NIR Analyzer Liquid :

- Mesures d'échantillons liquides en moins de 10 secondes
- Contrôle de la température sur l'échantillon de 25 °C à 80 °C
- Détection automatique de l'insertion et du retrait d'échantillons du récipient d'échantillon
- Intégration simple dans un système d'automatisation ou liaison avec d'autres technologies d'analyse (titrage)
- Prise en charge de nombreux récipients d'échantillon de différentes longueurs de chemin

Support OMNIS NIR, flacon, 8 mm

Support de flacon pour l'OMNIS NIR Analyzer pour flacons à usage unique de 8 mm (6.7402.240).





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Flacon à usage unique, 8 mm, transmission, Qté 100
100 flacons en verre (borosilicate) à usage unique avec une longueur de chemin de 8 mm pour des analyses de liquides lors d'une transmission. Les flacons à usage unique sont fournis avec les bouchons de fermeture correspondants (quantité = 100).

Compatible avec :

- Support OMNIS NIR, flacon, 8 mm (6.07401.070)
- Support DS2500 pour flacons à usage unique 8 mm (6.7492.020)

Licence OMNIS autonome

Elle permet l'exploitation autonome du logiciel OMNIS sur un ordinateur Windows™.

Caractéristiques :

- La licence comprend déjà une licence pour appareils OMNIS.
- Elle doit être activée via le portail d'octroi de licences Metrohm.

Licence logicielle Quant Development

Licence logicielle pour la création et l'édition de modèles de quantification dans une installation du logiciel OMNIS Stand-Alone.