



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.

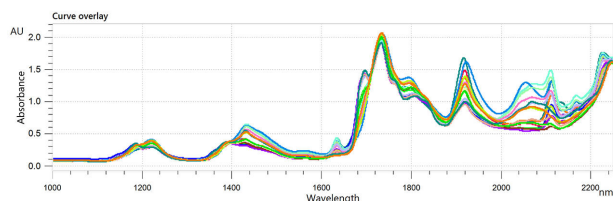


Figure 2. Stacked collection of NIR spectra from polyol samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT HYDROXYL NUMBER IN POLYOLS

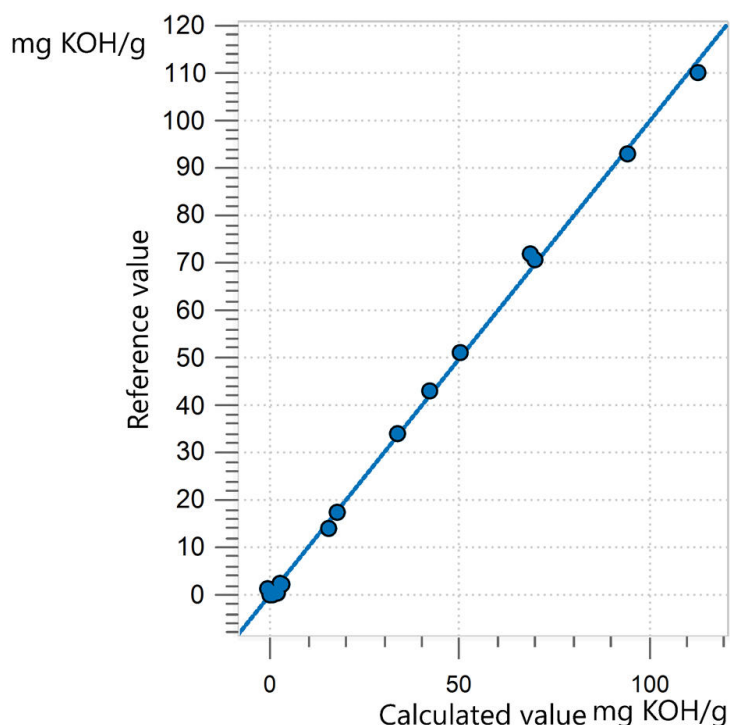


Figure 3. Correlation diagram and the respective figures of merit for the prediction of the hydroxyl number in polyols using an OMNIS NIR Analyzer Liquid. The hydroxyl number lab values were evaluated using titration.

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

RESULT ACID NUMBER IN POLYOLS

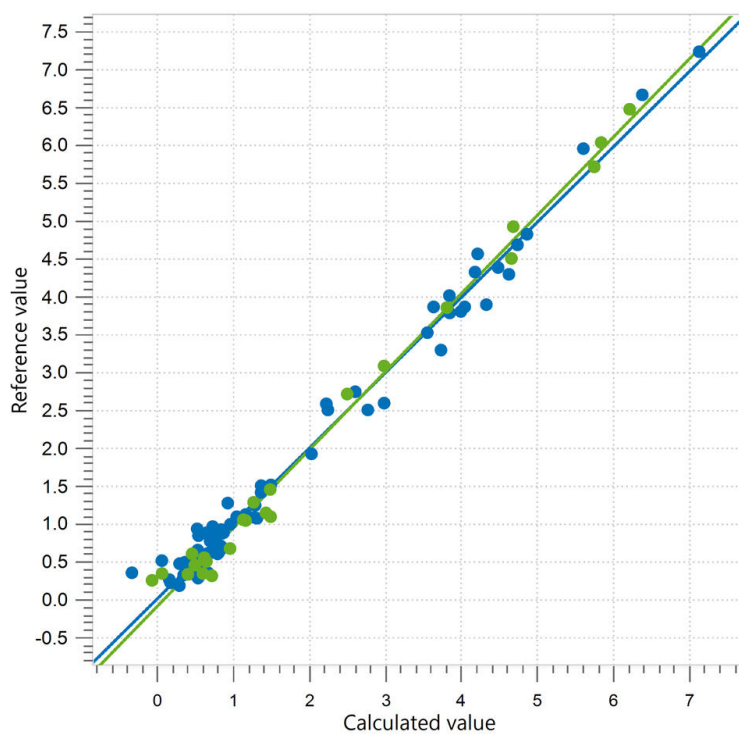


Figure 4. Correlation diagram and the respective figures of merit for the prediction of polyol acid number using an OMNIS NIR Analyzer Liquid. Titration was used to determine the laboratory acid number values.

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

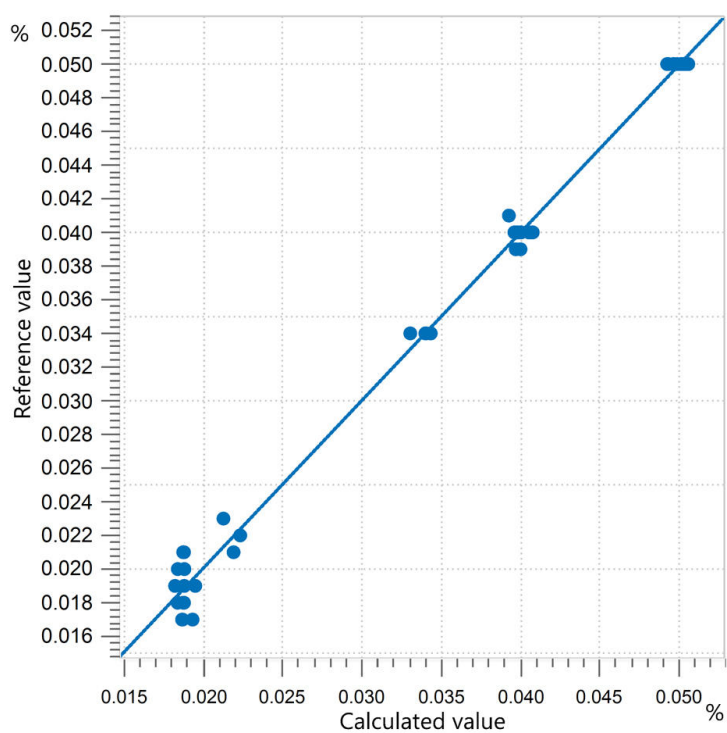


Figure 5. Correlation diagram and the respective figures of merit for the prediction of polyol water content using an OMNIS NIR Analyzer Liquid. Karl Fischer titration was used to determine the laboratory values for moisture content in polyols.

R ²	SEC (ppm)	SECV (ppm)
0.994	9	10

RESULT VISCOSITY IN POLYOLS

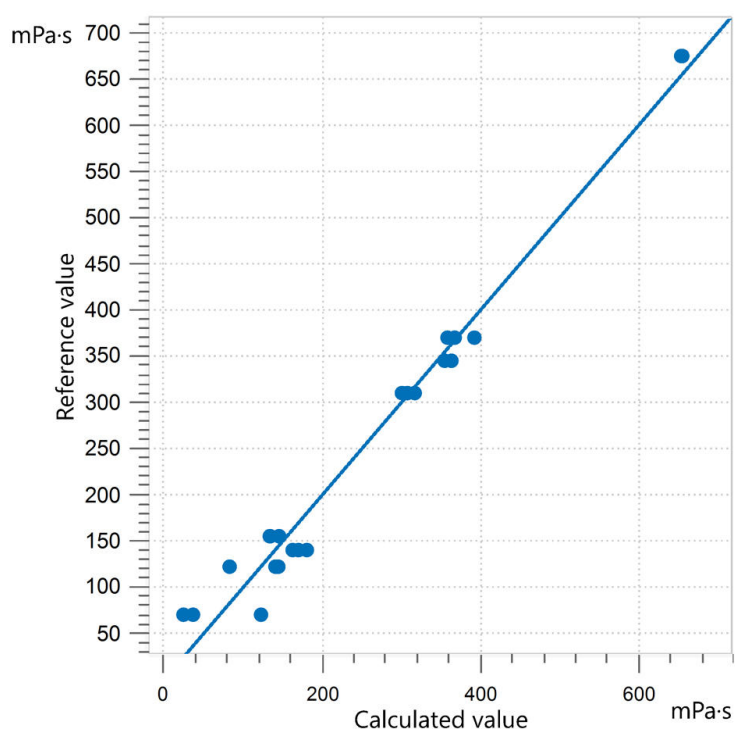


Figure 6. Correlation diagram and the respective figures of merit for the prediction of viscosity in polyols using an OMNIS NIR Analyzer Liquid. The viscosity lab values were evaluated with a viscometer.

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).

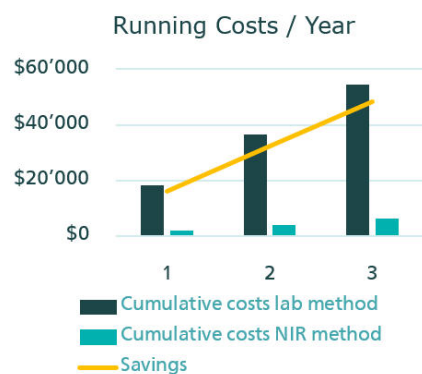


Figure 7. Comparison of running costs for the determination of the hydroxyl number with a standardized method (titration) and NIR spectroscopy.

	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

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CONFIGURATION



OMNIS NIR Analyzer Liquid

适合液体样品的近红外光谱仪。

OMNIS NIR Analyzer 是一种按照瑞士质量标准开发和生产的近红外光谱 (NIRS) 解决方案,用于整个生产链的常规分析。使用新技术和嵌入先进 OMNIS Software 反应在该 NIR 光谱仪的速度、可操作性和灵活使用上。

OMNIS NIR Analyzer Liquid 的优点概览:

- 可在 10 秒以内测量液体样品
- 对 25° C – 80° C 的样品进行温度控制
- 自动识别样品容器的插入和取出
- 方便地嵌入自动系统,或者与其它分析技术(滴定)关联
- 支持大量不同路径长度的样品容器



OMNIS NIR8 mm

适合 8 mm 一次性小管的 OMNIS NIR Analyzer 的小管支架 (6.7402.240)。



8 mm 100

100 个玻璃(硼硅)一次性样品瓶,具有 8 mm 的光路长度,适用于分析透射中的液体。一次性样品瓶交付时带有所属的螺旋塞(件数 = 100)。

兼容:

- 支架 OMNIS NIR,小管,8 mm (6.07401.070)
- DS2500 支架适用于 8 mm 一次性样品瓶 (6.7492.020)

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- 须通过万通许可授权平台进行激活。

Quant Development

用于在独立 OMNIS Software 安装套件中编写和编辑量化模型的软件许可证。