Application Note AN-NIR-035

Quality Control of Polyols

Chemical-free determination of Hydroxyl Number according to ASTM D6342-12

Toxic and corrosive chemicals such as p-toluenesulfonyl isocyanate (TSI) and tetrabutylammonium hydroxide are used for the Hydroxyl Number analysis of polyols by titration according to ASTM D4274-16.

This application note demonstrates how the XDS RapidLiquid Analyzer operating in the visible and near-infrared spectral region (Vis-NIR) provides a cost-efficient and fast solution for the determination of the hydroxyl (OH) number of polyols without such toxic materials. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows for the analysis of polyols in **less than a minute**.

EXPERIMENTAL EQUIPMENT

Polyol samples were measured with the 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 4 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 a polyol sample present in a 4 mm disposable vial.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
XDS RapidLiquid Analyzer	2.921.1410
Disposable vials, 4 mm diameter, transmission	6.7402.010
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 hydroxyl number in polyol samples. The quality of the prediction models was evaluated using correlation diagrams, which display the relationship between the Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis (Figure 3).

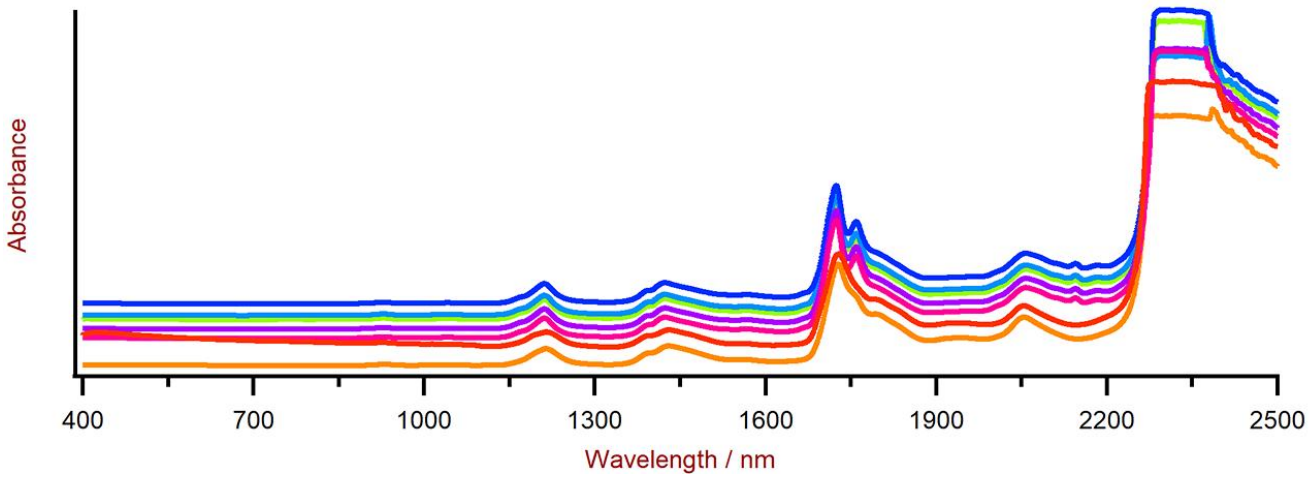

Figure 2. Selection of polyol Vis-NIR spectra obtained using an XDS RapidLiquid Analyzer and 4 mm disposable vials. For display reasons a spectra offset was applied.

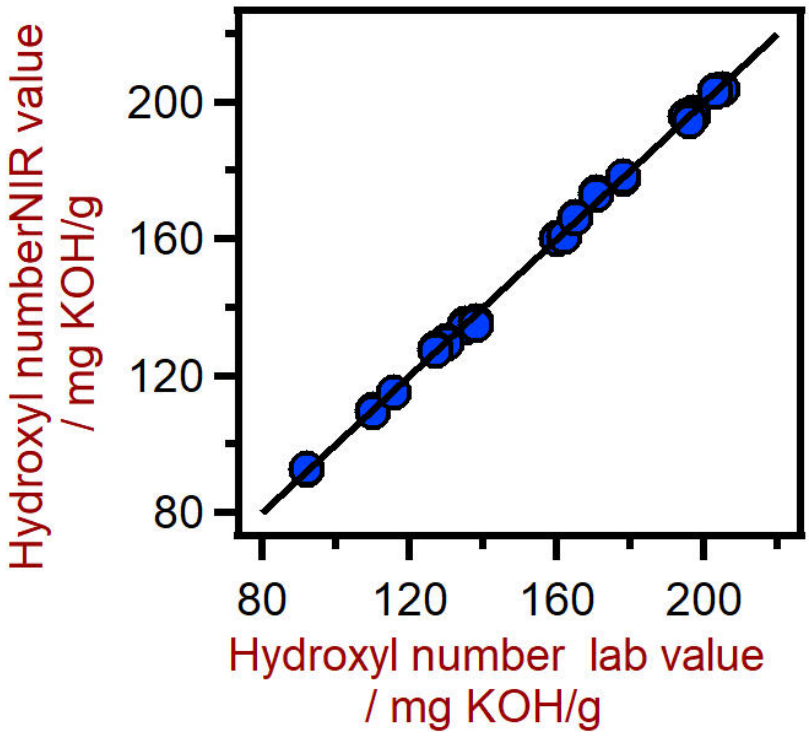

Figure 3. Correlation diagram for the prediction of the hydroxyl number in polyols using a XDS RapidLiquid Analyzer. The Hydroxyl Number lab value was evaluated using titration.

Table 2. Figures of merit for the prediction of the hydroxyl number in polyols using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R^2	0.998
Standard error of calibration	1.28 mg KOH/g
Standard error of cross-validation	1.42 mg KOH/g

CONCLUSION

This application note demonstrates the feasibility of NIR spectroscopy for the analysis of the Hydroxyl Number in polyols according to ASTM D6342-12. In comparison to wet chemical methods, running costs are significantly lower when using NIR spectroscopy (Table 3 and Figure 4). Additionally, there is no need to use dangerous chemicals for the analysis as with ASTM D4274-16.

Table 3. Comparison of running costs for the determination of the hydroxyl number with 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

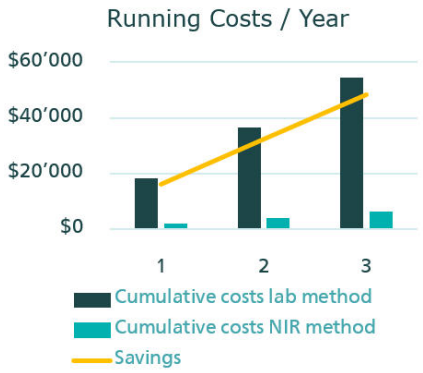

Figure 4. Comparison of the cumulative costs over three years for the determination of the hydroxyl number with titration and NIR spectroscopy.

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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)