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).

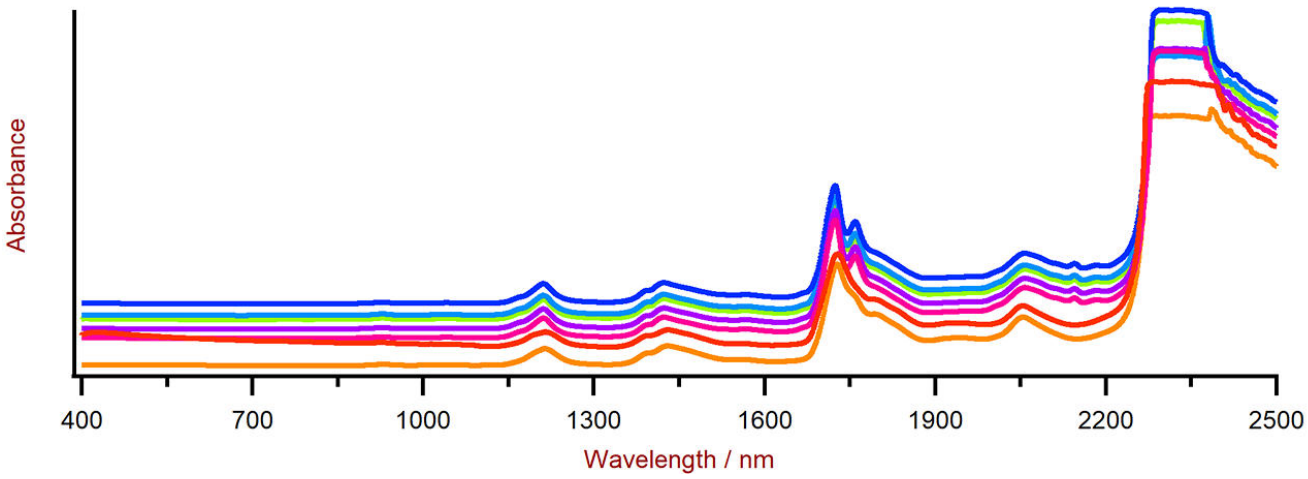

Figure 2. Selection of polyol Vis-NIR spectra obtained using an XDS RapidLiquid Analyzer and 4 mm disposable vials. For display reasons a spectra offset was applied.

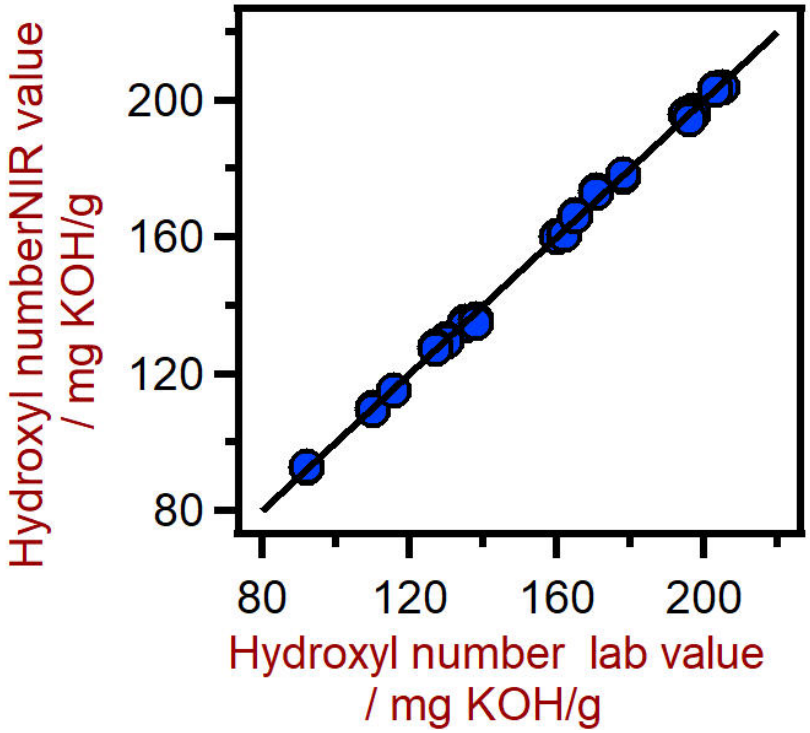

Figure 3. Correlation diagram for the prediction of the hydroxyl number in polyols using a XDS RapidLiquid Analyzer. The Hydroxyl Number lab value was evaluated using titration.

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

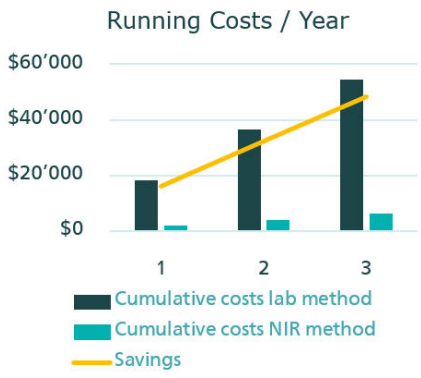

Figure 4. Comparison of the cumulative costs over three years for the determination of the hydroxyl number with titration and NIR spectroscopy.

CONTACT

Metrohm France
13, avenue du Québec - CS 90038
91978 VILLEBON COURTABOEUF CEDEX
info@metrohm.fr

NIRS XDS RapidLiquid Analyzer
Des analyses rapides et précises de liquides et suspensions de toutes sortes.

L'analyseur NIRS XDS RapidLiquid Analyzer permet des analyses rapides et précises de formules et substances liquides. Des résultats de mesure précis obtenus par simple pression d'une touche font du NIRS XDS RapidLiquid Analyzer une solution aussi fiable que simple pour le contrôle qualité en laboratoire et en production. Les échantillons sont présentés dans des cuvettes en quartz réutilisables ou des flacons en verre à usage unique ; une chambre à échantillons tempérée assure la reproductibilité des conditions d'analyse et, par conséquent, l'exactitude des résultats de mesure.

Vision Air 2.0 Complete

Vision Air - logiciel universel de spectroscopie.

Vision Air Complete est une solution logicielle moderne et simple d'utilisation pour une application dans un environnement réglementé.

Apercu des avantages de Vision Air :

- Des applications logicielles individuelles avec interface utilisateur adaptée sont le garant d'un maniement intuitif et simple
- Établissement et suivi simples des procédures de travail
- Base de données SQL pour une gestion sure et simple des données

La version Vision Air Complete (66072208) comprend toutes les applications d'assurance qualité par spectroscopie Vis-NIR :

- Application de gestion des instruments et des données
- Application de développement de méthodes
- Application d'analyse de routine

Autres solutions Vision Air Complete :

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)