Application Note AN-NIR-060

Quality Control of Polyamides

Determination of viscosity, functional groups, and moisture within one minute using NIR Spectroscopy

Functional group and viscosity analysis (ASTM D789) of polyamides can be a lengthy and challenging process due to the sample's limited solubility.
This application note demonstrates that the DS2500 Solid Analyzer operating in the visible and near-infrared spectral region (Vis-NIR) provides a cost-efficient and fast solution for a simultaneous determination of the relative viscosity as well as the amine, carboxylic, and moisture content in polyamides. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows for the analysis of polyamides in **less than a minute**.

EXPERIMENTAL EQUIPMENT

Polyamide pellets were measured with a DS2500 Solid Analyzer in reflection mode over the full wavelength range (400–2500 nm). A rotating DS2500 Large Sample Cup was employed to overcome the distribution of the particle sizes and chemical components. This allowed for an automated measurement at different sample locations for a reproducible spectrum acquisition. As displayed in Figure 1, samples were measured without any preparation. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

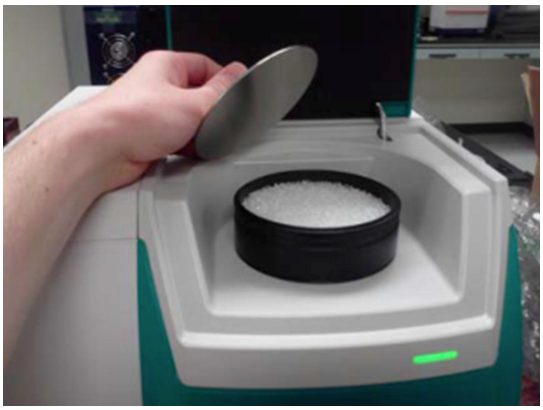

Figure 1. DS2500 Solid Analyzer and polyamide pellets present in the rotating DS2500 Large Sample Cup.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Solid Analyzer	2.922.0010
DS2500 Large Sample Cup	6.7402.050
Vision Air 2.0 Complete	6.6072.208

RESULTS

The obtained Vis-NIR spectra (Figure 2) were used to create prediction models for quantification of the relative viscosity and amine end group, carboxylic end group, and moisture content in polyamides. The quality of the prediction models was evaluated using correlation diagrams, which display the relationship between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

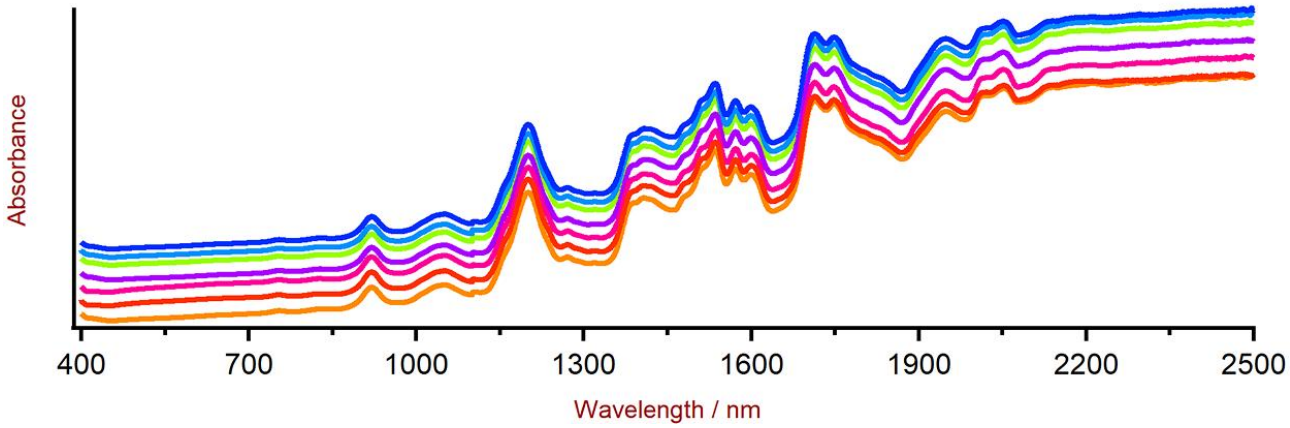

Figure 2. Selection of polyamide Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Large Sample Cup. For display reasons spectra are shown with an offset applied.

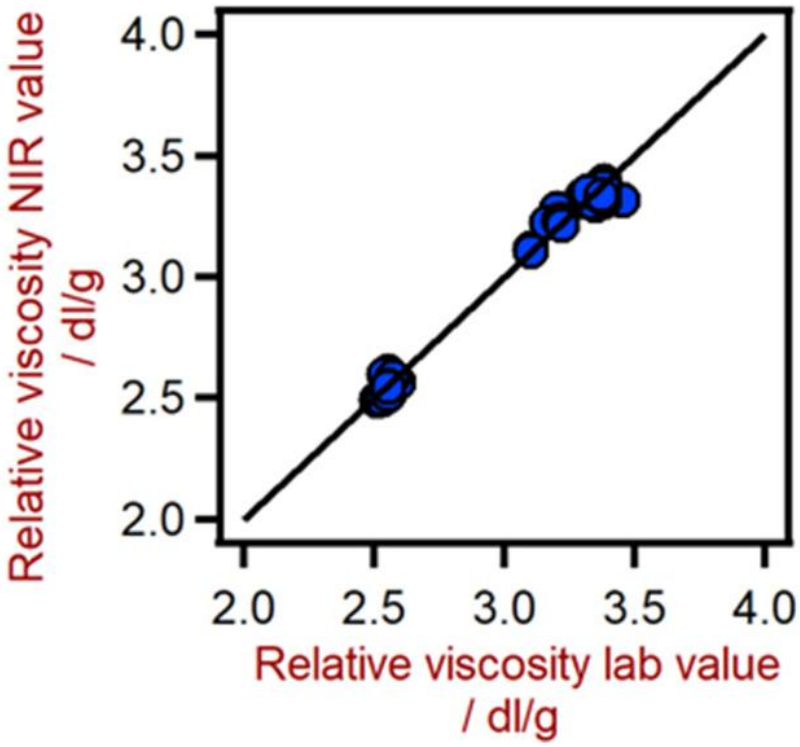

Figure 3. Correlation diagram for the prediction of the relative viscosity of polyamides using a DS2500 Solid Analyzer. The relative viscosity lab value was evaluated using viscometry.

Table 2. Figures of merit for the prediction of the relative viscosity of polyamides using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.986
Standard error of calibration	0.046 dl/g
Standard error of cross-validation	0.055 dl/g

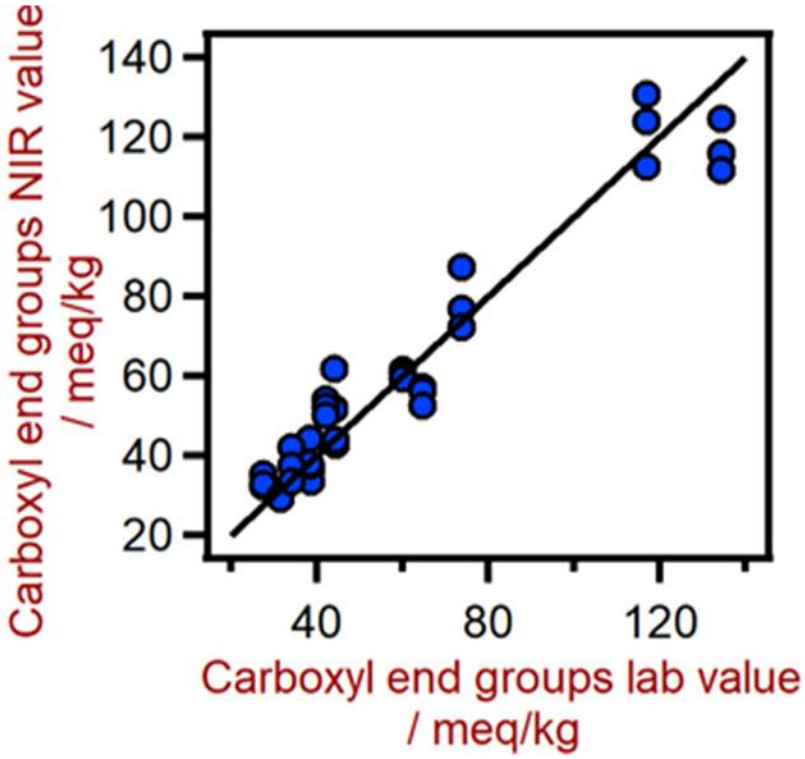

Figure 4. Correlation diagram for the prediction of carboxylic end group content in polyamides using a DS2500 Solid Analyzer. The carboxyl end group lab value was evaluated using titration.

Table 3. Figures of merit for the prediction of the carboxylic end group content in polyamides using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.972
Standard error of calibration	6.1 meq/kg
Standard error of cross-validation	11.1 meq/kg

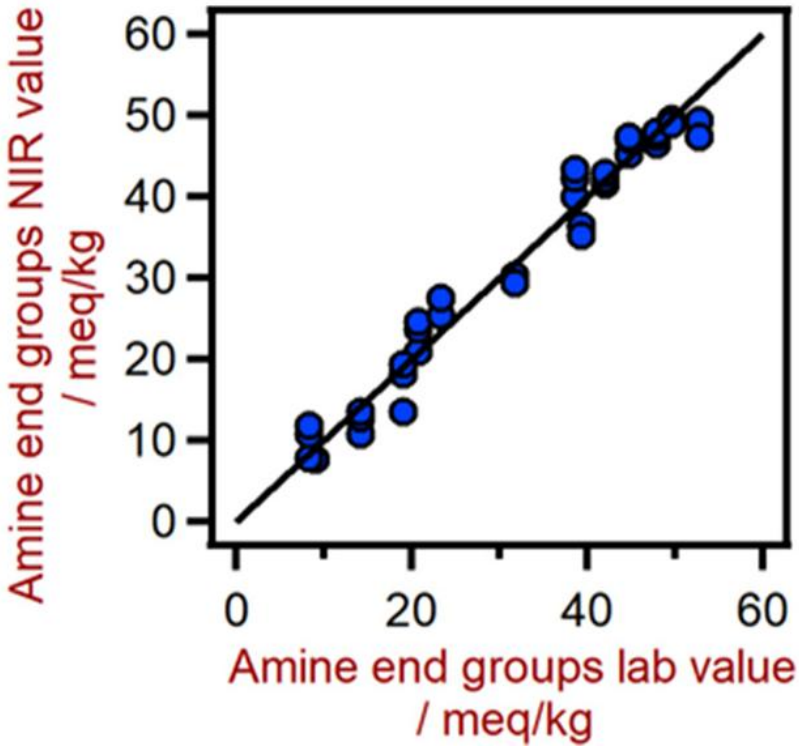

Figure 5. Correlation diagram for the prediction of amine end group content in polyamides using a DS2500 Solid Analyzer. The amine end group lab value was evaluated using titration.

Table 4. Figures of merit for the prediction of the amine end group content in polyamides using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.981
Standard error of calibration	2.5 meq/kg
Standard error of cross-validation	4.1 meq/kg

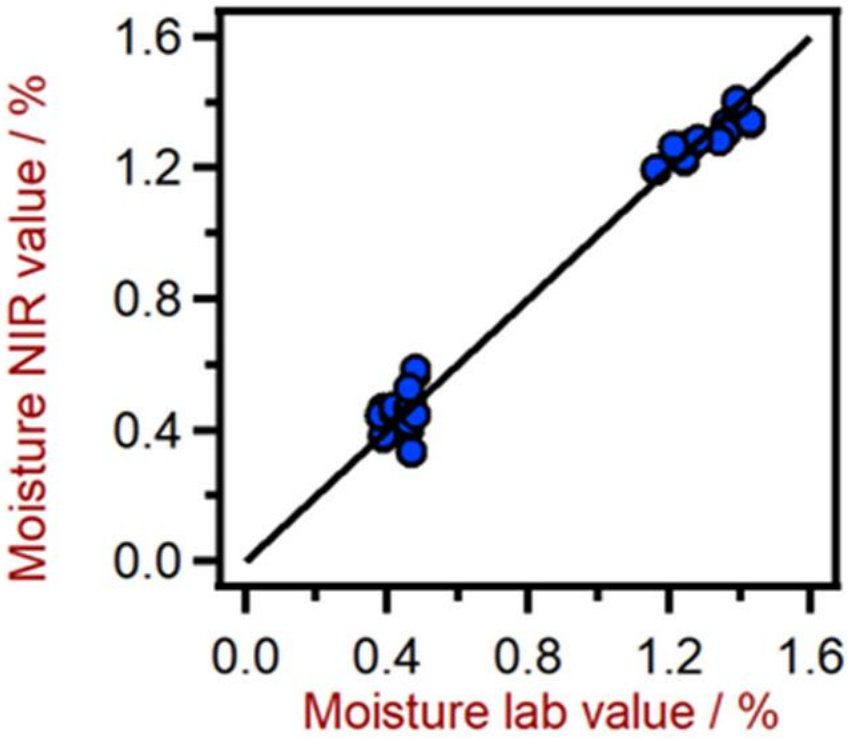

Figure 6. Correlation diagram for the prediction of moisture content in polyamides using a DS2500 Solid Analyzer.

Table 5. Figures of merit for the prediction of the moisture content in polyamides using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.991
Standard error of calibration	0.041%
Standard error of cross-validation	0.067%

CONCLUSION

This study demonstrates the feasibility of NIR spectroscopy for the analysis of some key quality parameters of polyamides. In comparison to wet chemical methods (Table 6), the time to result is a major advantage of NIR spectroscopy, since all parameters are determined in a single measurement in less than a minute.

Table 6. Time to result overview for the different parameters.

Parameter	Procedure	Time to result
Relative viscosity	Viscometry	90 min (preparation) + 1 min (viscometry)
Carboxyl end group	Titration	90 min (preparation) + 20 min (titration)
Amine end group	Titration	90 min (preparation) + 20 min (titration)
Moisture	KF Titration	25 min (preparation) + 5 min (Karl Fischer titration)

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