

Quality Control of Polyamides

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

Summary

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

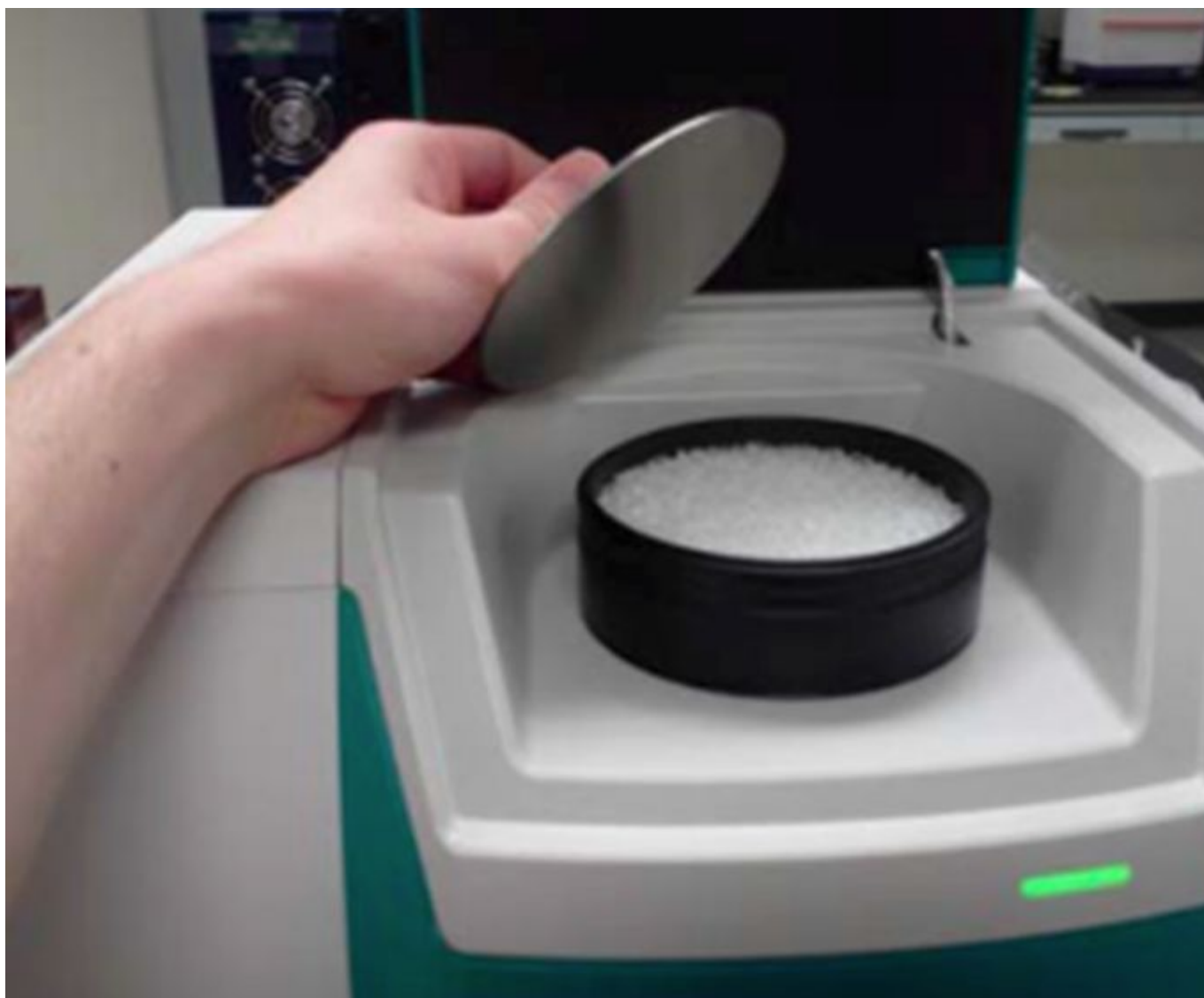


Figure 1. DS2500 Solid Analyzer and polyamide pellets present in the rotating DS2500 Large Sample Cup.

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.

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



2.922.0010 - DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments. The NIRS DS2500 Analyzer is the tried and tested, flexible solution for routine analysis of solids, creams, and optionally also liquids along the entire production chain. Its robust design makes the NIRS DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments. The NIRS DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The NIRS DS2500 Analyzer meets the demands of the pharmaceutical industry and supports users in their day-to-day routine tasks thanks to its simple operation. Thanks to accessories tailored perfectly to the instrument, optimum results are achieved with every sample type, no matter how challenging it is, e.g. coarse-grained solids such as granulates or semi-solid samples such as creams. The MultiSample Cup can help improve productivity when measuring solids, as it enables automated measurements of series containing up to nine samples.



6.7402.050 - DS2500 large sample cup

Large sample cup for the spectral recording of powders and granulates in reflection at various sample positions using the NIRS DS2500 Analyzer.



6.6072.208 - 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);

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.

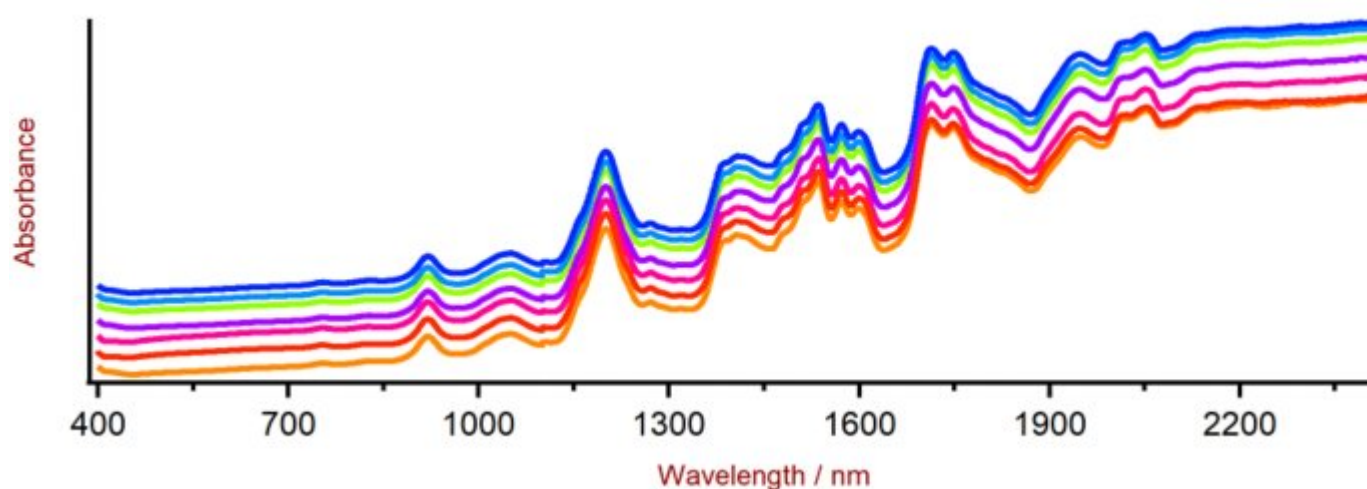


Figure 2. Selection of polyamide Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Large Sample Cup. For display reasons spectra are shown with an offset applied.

Result relative viscosity

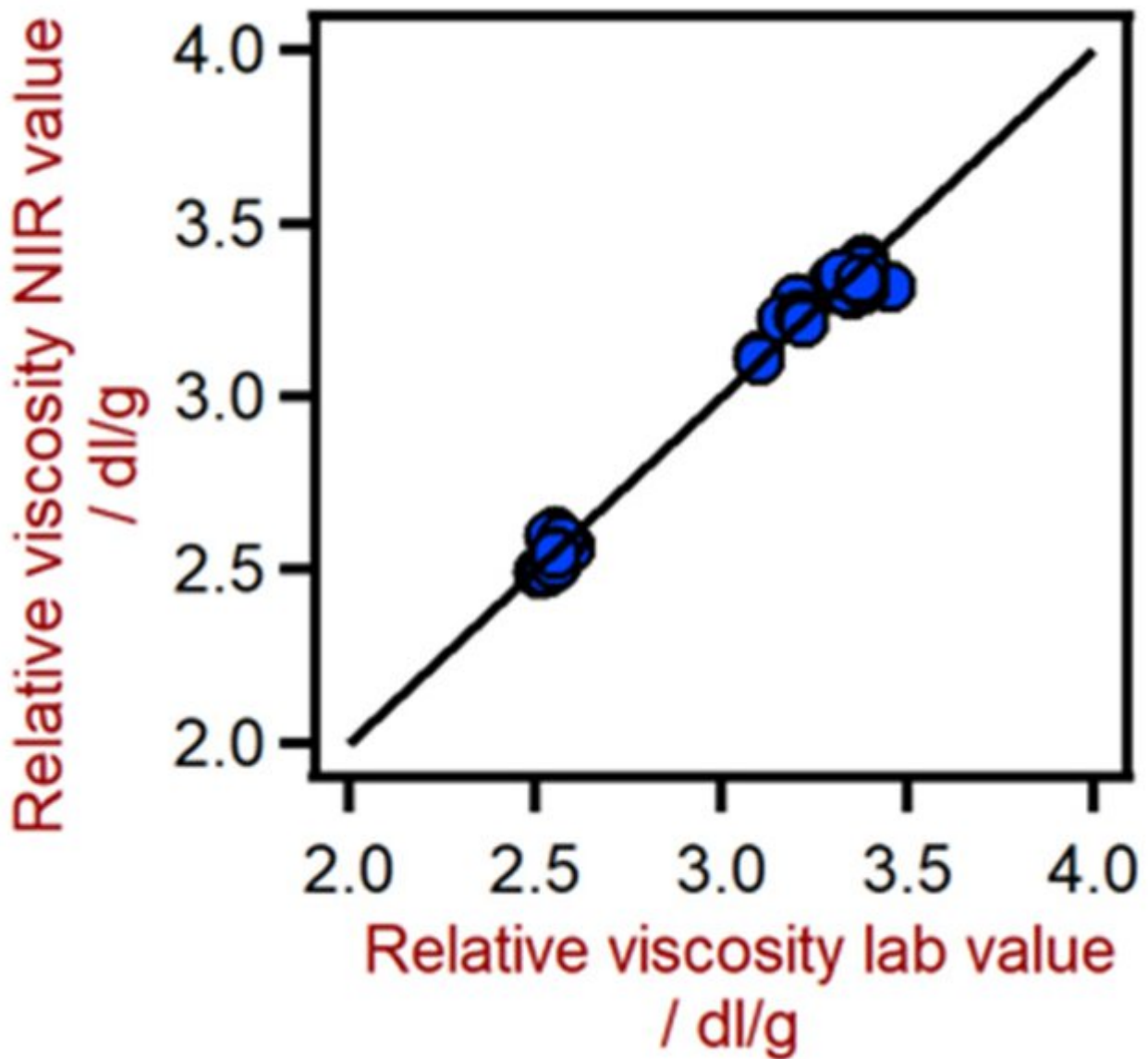


Figure 3. Correlation diagram for the prediction of the relative viscosity of polyamides using a DS2500 Solid Analyzer. The relative viscosity lab value was evaluated using viscometry.

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

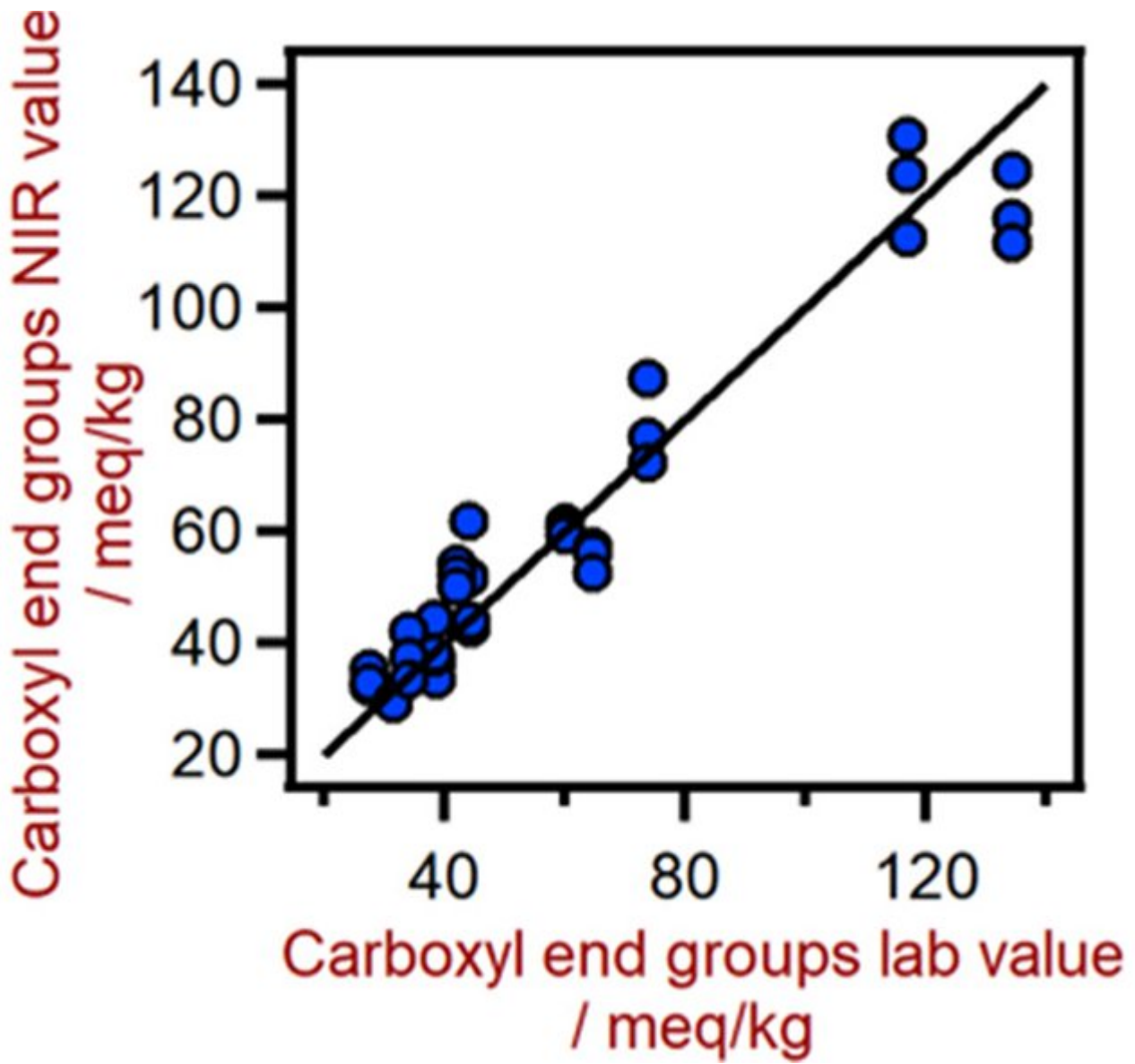


Figure 4. Correlation diagram for the prediction of carboxylic end group content in polyamides using a DS2500 Solid Analyzer. The carboxyl end group lab value was evaluated using titration.

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

Result amine end group content

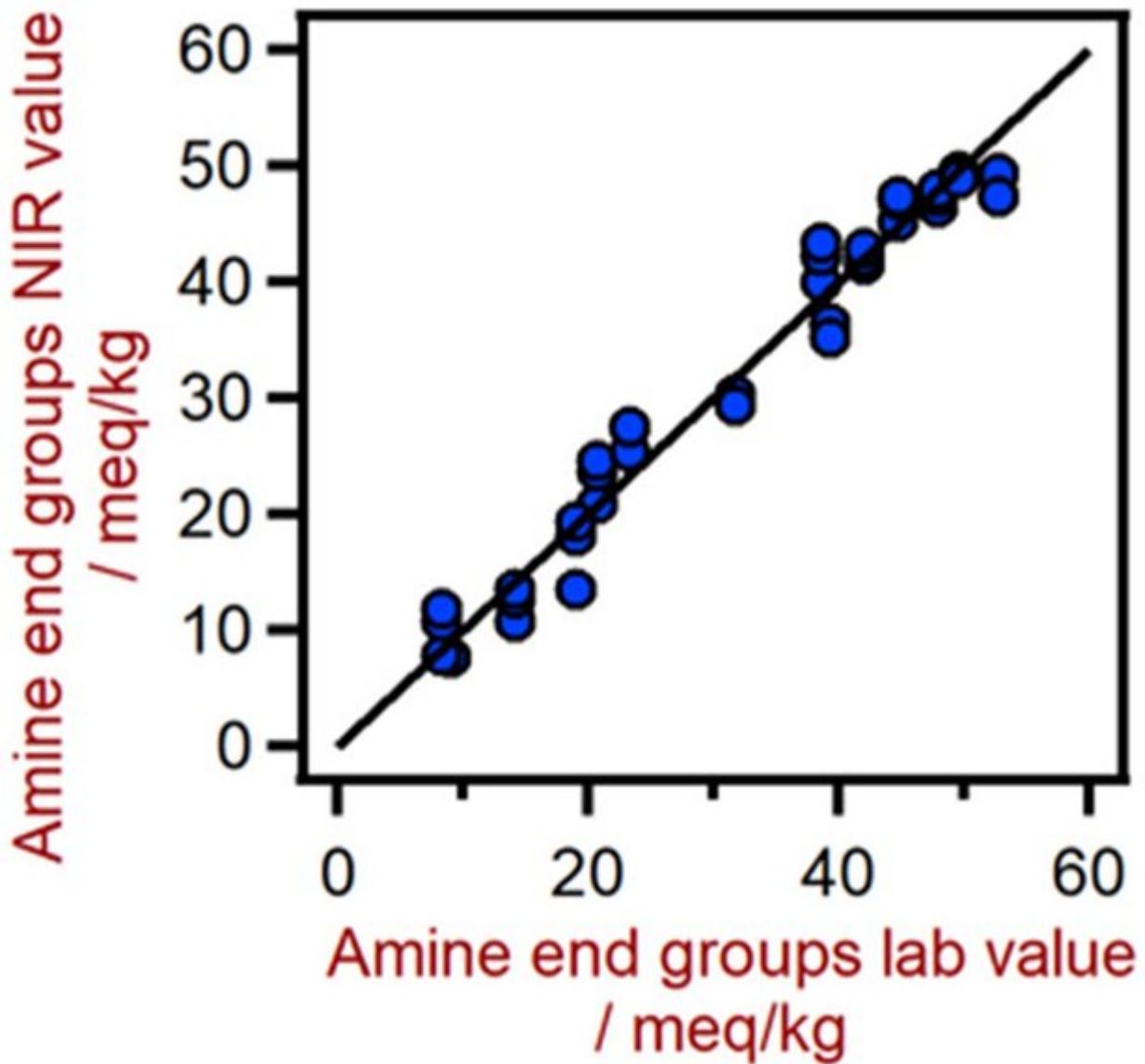


Figure 5. Correlation diagram for the prediction of amine end group content in polyamides using a DS2500 Solid Analyzer. The amine end group lab value was evaluated using titration.

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

Result moisture content

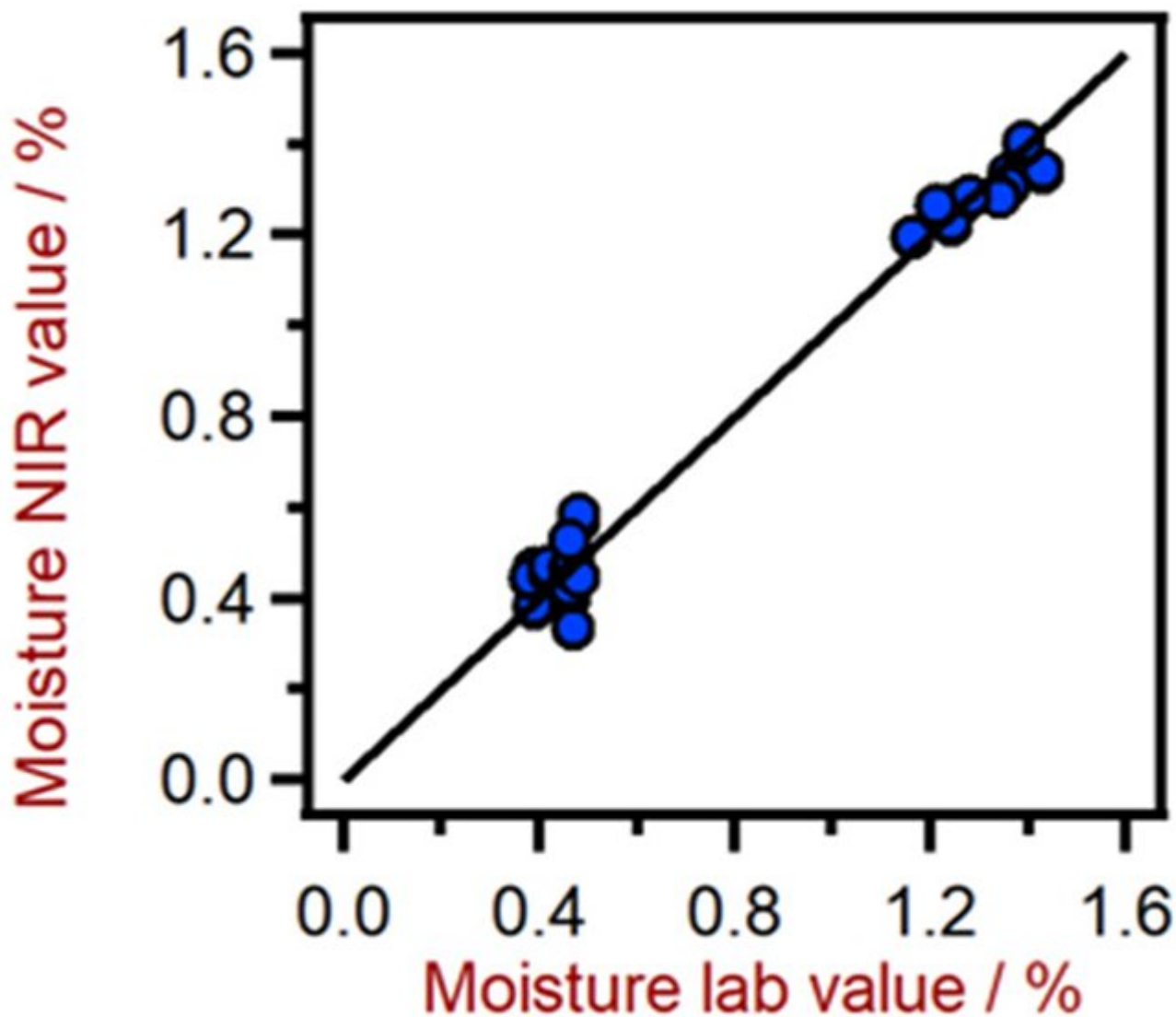


Figure 6. Correlation diagram for the prediction of moisture content in polyamides using a DS2500 Solid Analyzer.

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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