

Quality Control of PET

Determination of diethylene glycol, isophthalic acid, intrinsic viscosity, and acid number within one minute with NIRS

Summary

Determination of the diethylene glycol content, isophthalic acid content, intrinsic viscosity (ASTM D4603), and the acid number (AN) of polyethylene terephthalate (PET) is a lengthy and challenging process due to the sample's limited solubility and the need to use different analytical methods.

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 diethylene glycol content, isophthalic acid content, intrinsic viscosity, and the acid number in PET. Vis-NIR spectroscopy allows for the analysis of PET in **less than one minute without sample preparation or using any chemical reagents**.

Experimental Equipment

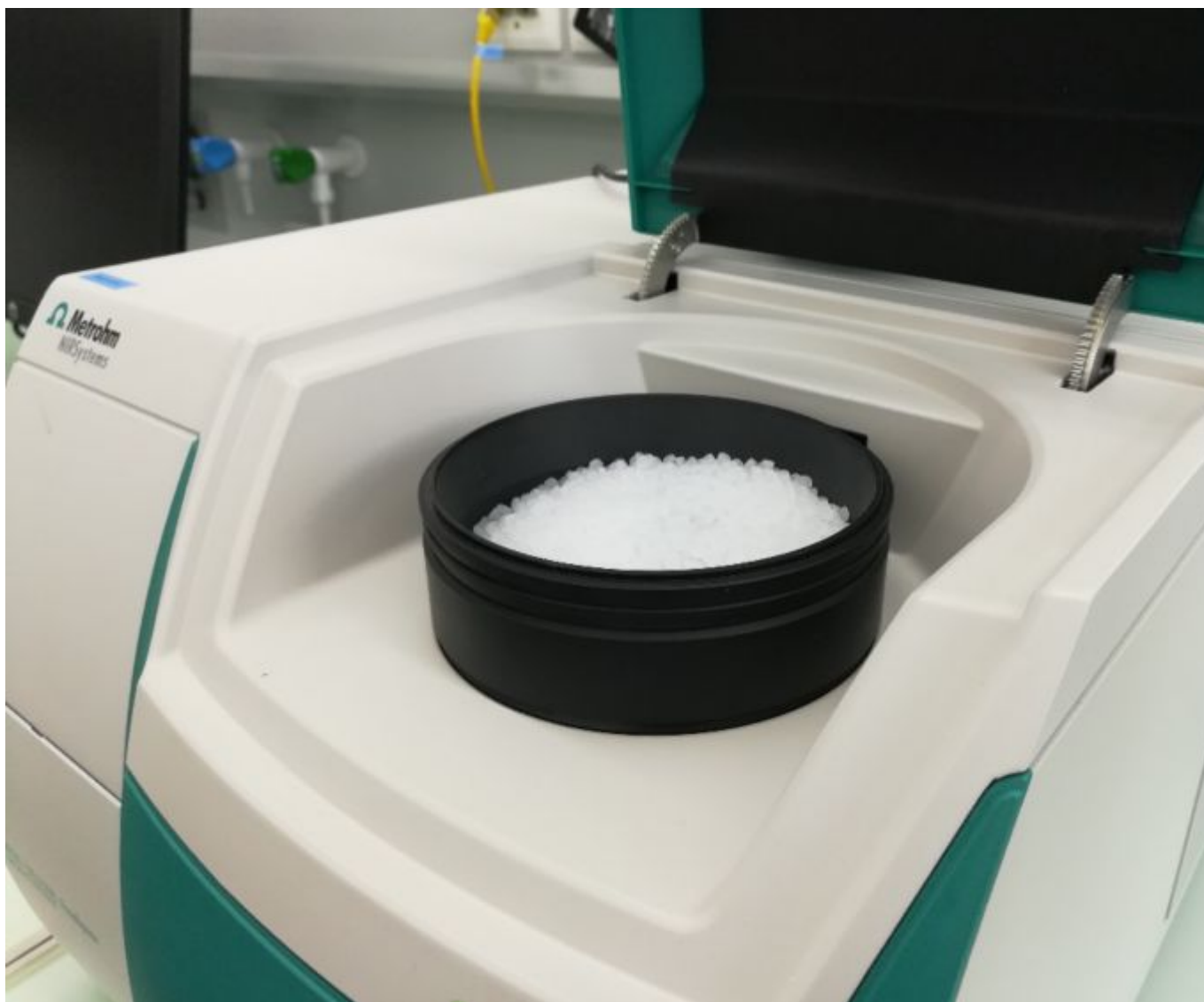


Figure 1. DS2500 Solid Analyzer with PET pellets present in the rotating DS2500 Large Sample Cup.

PET 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 varied particle sizes and chemical components. This allowed automated measurements at different sample locations for a reproducible spectrum acquisition. As displayed in **Figure 1**, samples were measured without any preparation step. 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 diethylene glycol, isophthalic acid, intrinsic viscosity, and acid number. The quality of the prediction models was evaluated using correlation diagrams, which display the correlation between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

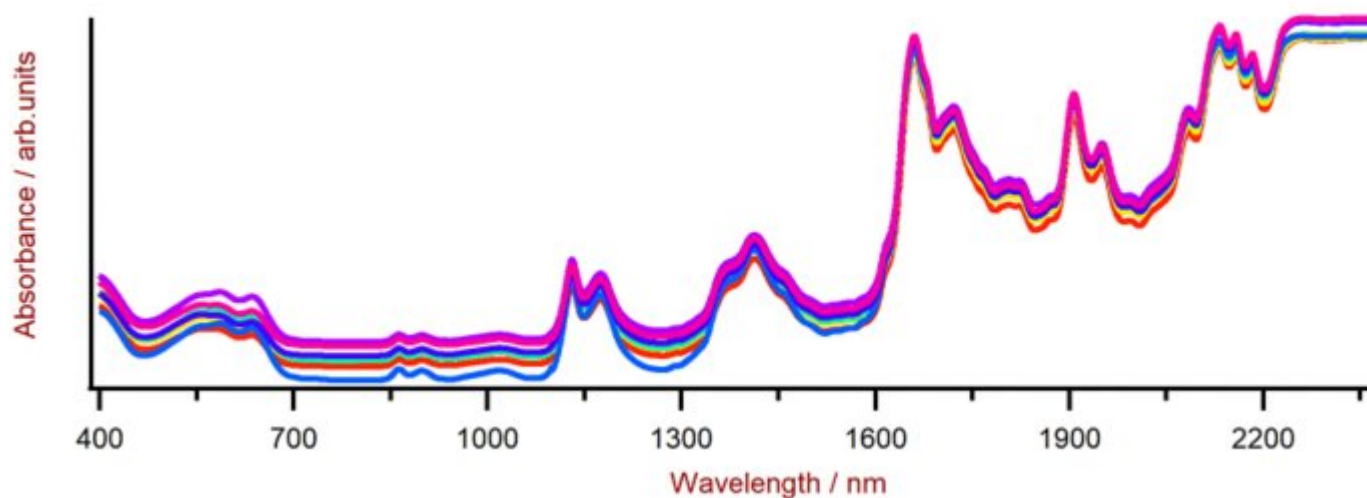


Figure 2. Selection of PET Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Large Sample Cup. For display reasons a spectra offset was applied.

Result diethylene glycol content

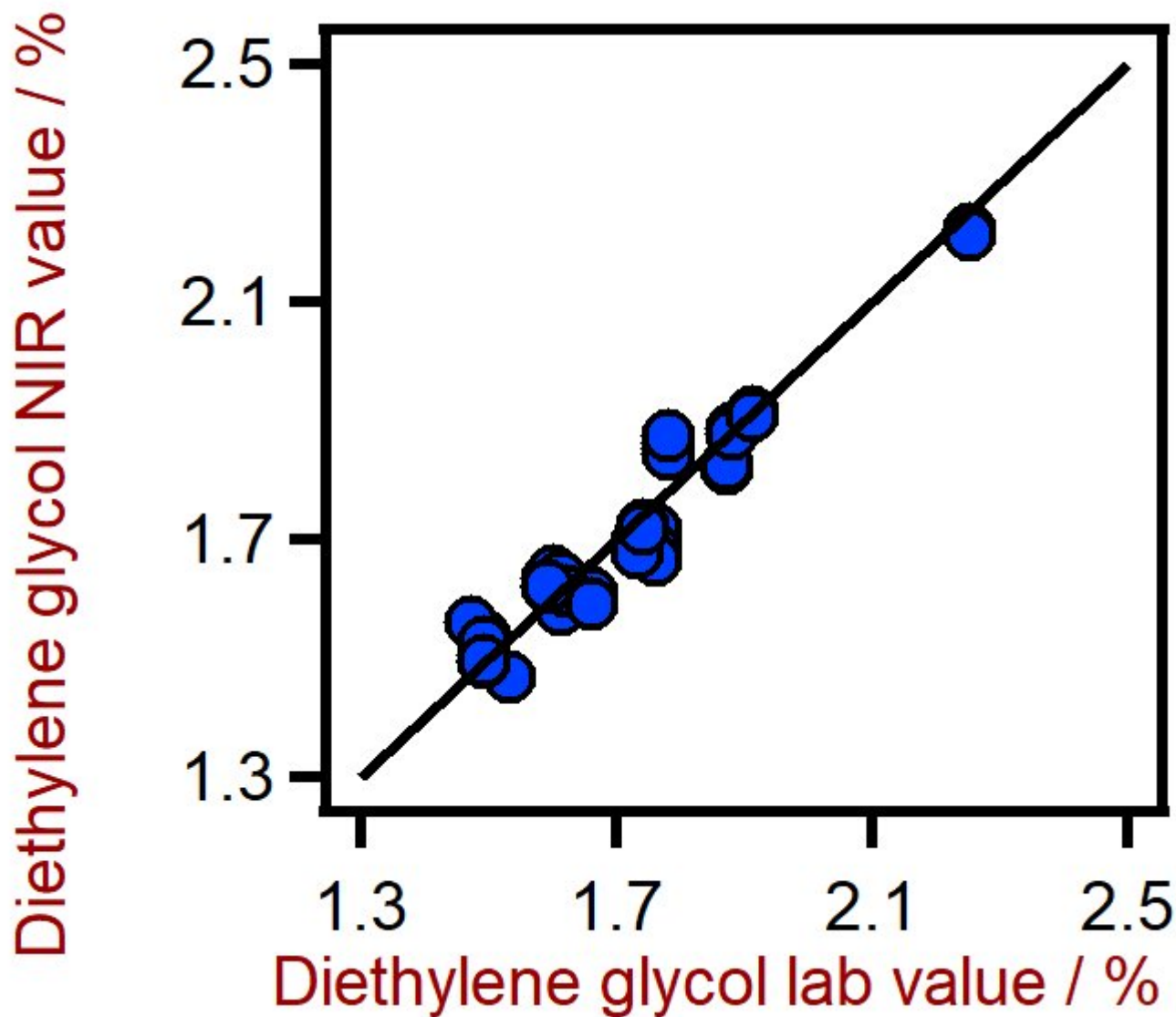


Figure 3. Correlation diagram for the prediction of the diethylene glycol content in PET using a DS2500 Solid Analyzer. The diethylene glycol lab value was evaluated using HPLC-MS.

Table 2. Figures of merit for the prediction of the diethylene glycol content in PET using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.931
Standard error of calibration	0.052%
Standard error of cross-validation	0.066%

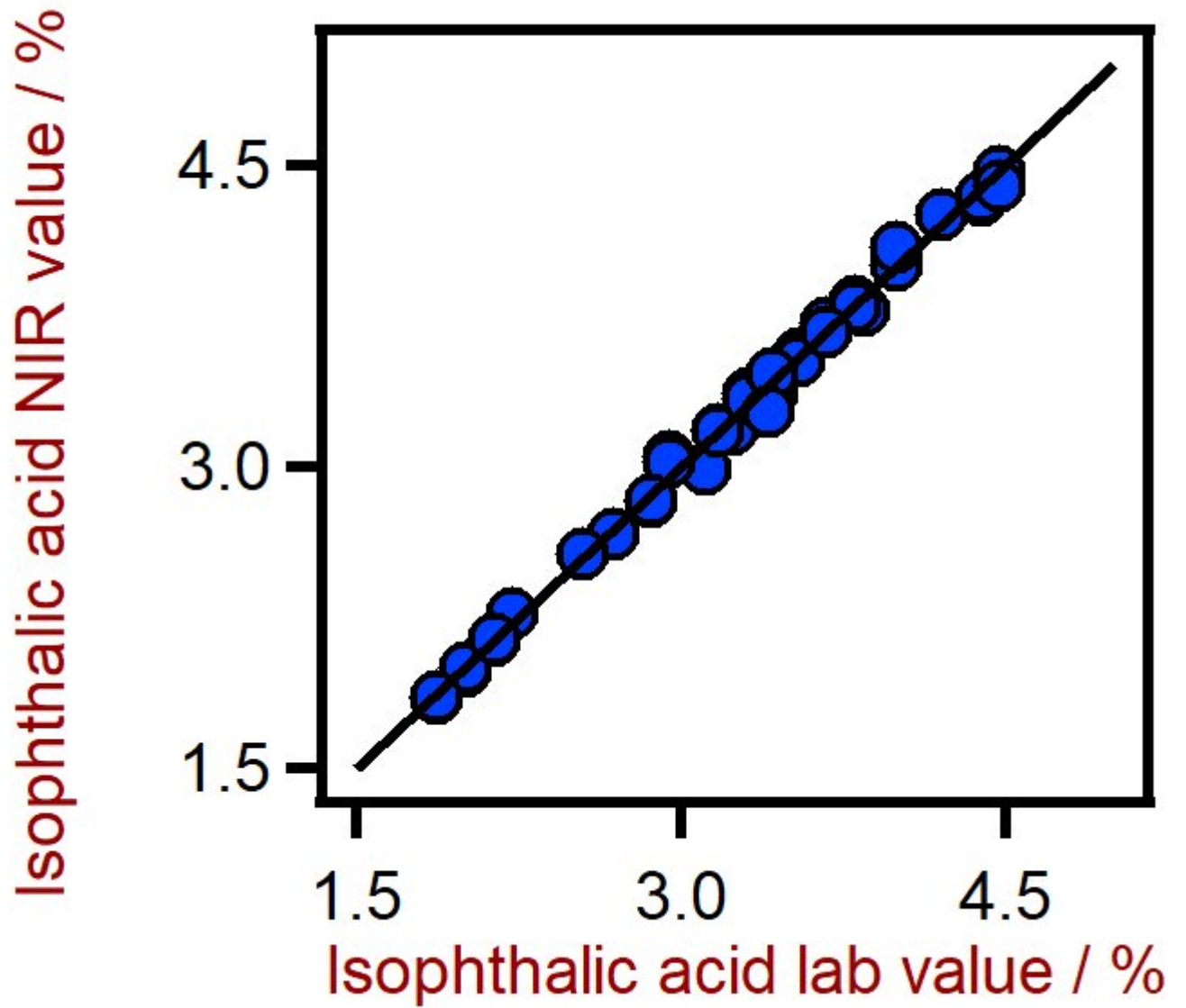


Figure 4. Correlation diagram for the prediction of the isophthalic acid content in PET using a DS2500 Solid Analyzer. The isophthalic acid lab value was evaluated using HPLC.

Table 3. Figures of merit for the prediction of the isophthalic acid content in PET using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.995
Standard error of calibration	0.059%
Standard error of cross-validation	0.085%

Result intrinsic viscosity

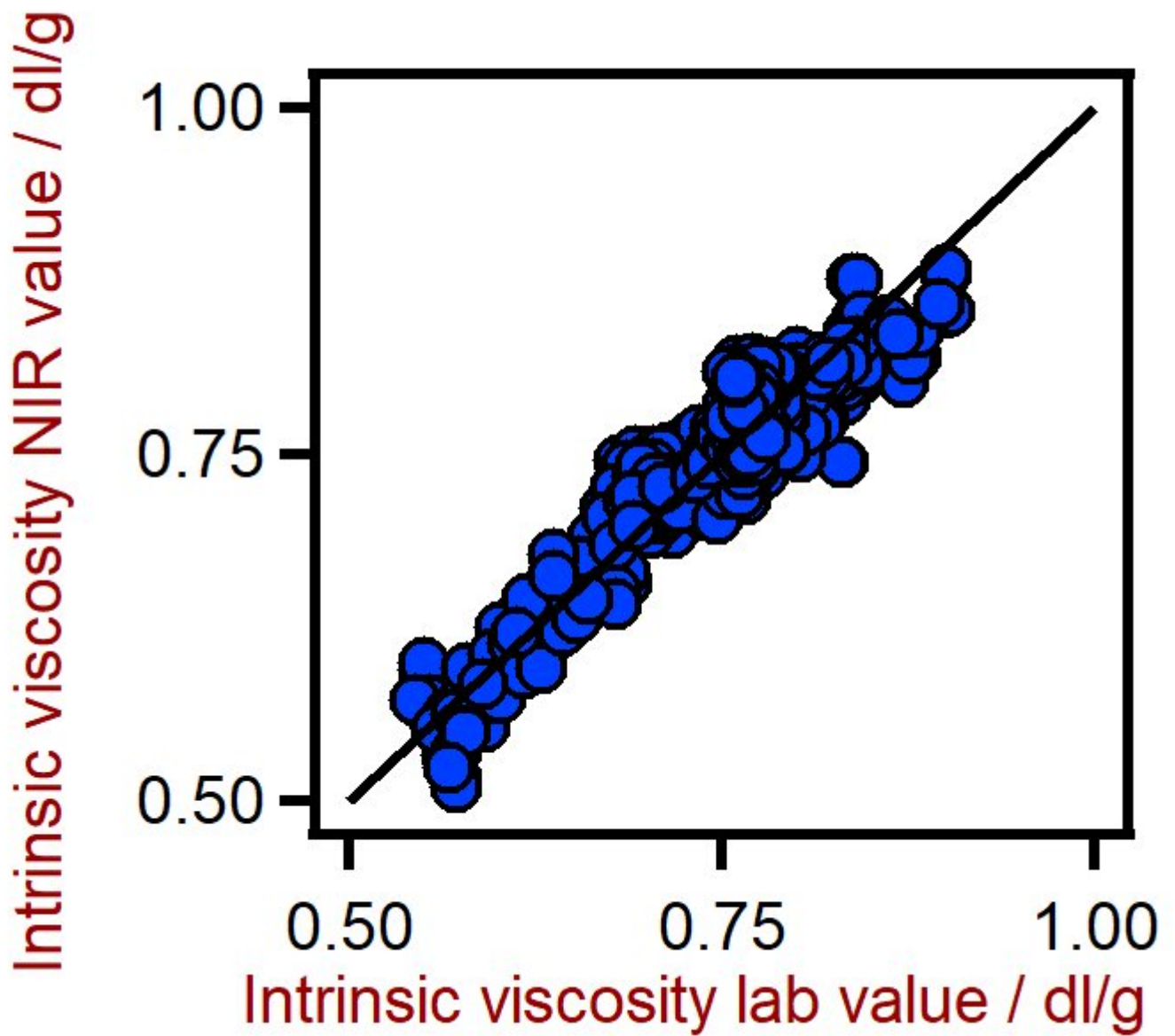


Figure 5. Correlation diagram for the prediction of the intrinsic viscosity of PET using a DS2500 Solid Analyzer. The intrinsic viscosity lab value was evaluated using viscometry.

Table 4. Figures of merit for the prediction of the intrinsic viscosity of PET using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.873
Standard error of calibration	0.0236
Standard error of cross-validation	0.0238

Result acid value

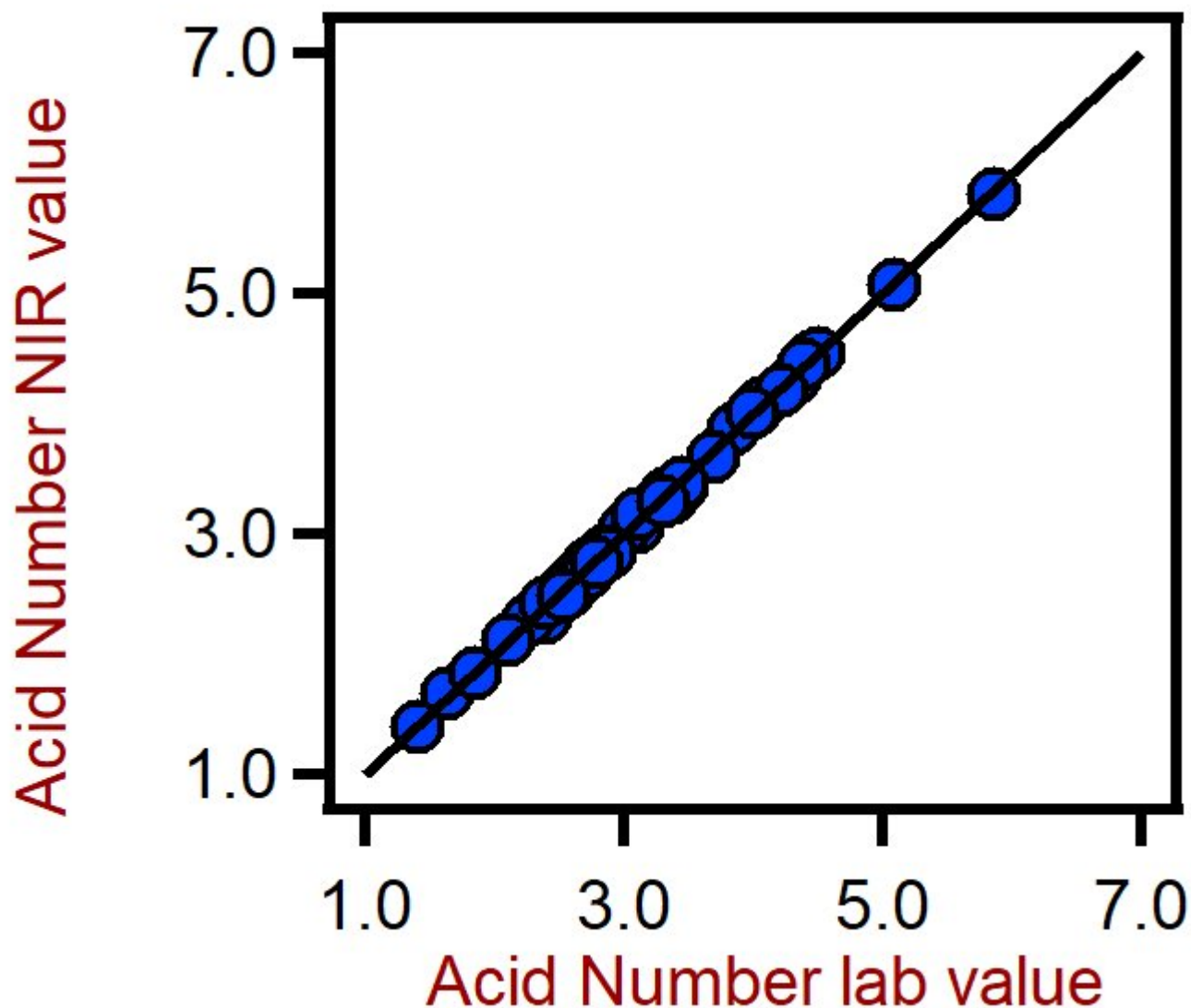


Figure 6. Correlation diagram for the prediction of the acid number in PET using a DS2500 Solid Analyzer. The Acid Number lab value was evaluated using titration.

Table 5. Figures of merit for the prediction of the acid number in PET using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.991
Standard error of calibration	0.093

Conclusion

This study demonstrates the feasibility of NIR spectroscopy for the analysis of key quality parameters of PET. 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 under a minute**.

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

Parameter	Method	Time to result
Diethylene glycol	Extraction + analysis HPLC-MS	45 min (preparation) + 40 min (HPLC)
Isophthalic acid	Dissolve + HPLC	45 min (preparation) + 40 min (HPLC)
Intrinsic viscosity	Dissolve + viscometry	90 min (preparation) + 1 min (viscometry)
Acid Number	Dissolve + titration	90 min (preparation) + 10 min (titration)

Metrohm AG

*Ionenstrasse
9100 Herisau*

<mailto:info@metrohm.com>