

Application Note AN-NIR-112

Intrinsic viscosity (IV) determination of recycled PET by NIR spectroscopy

Simple, chemical-free analysis with results in seconds

Determination of the intrinsic viscosity of recycled polyethylene terephthalate (rPET) is a time-consuming and challenging process. This is especially true if the sample is highly crystalline and needs to be dried before the analysis with the glass capillary as mentioned in the ASTM norm (ASTM D4603). The drying process often takes several hours until a constant weight is reached, and high crystallinity hinders solubility.

Near-infrared (NIR) spectroscopy is able to determine the intrinsic viscosity of rPET in less than one minute without any sample preparation. This Application Note demonstrates that the Metrohm DS2500 Solid Analyzer operating in the visible and near-infrared spectral region (Vis-NIR) offers users an easier way to perform this analysis without the use of toxic chemicals.

EXPERIMENTAL EQUIPMENT

48 different recycled PET samples with varying IV were measured on the Metrohm DS2500 Solid Analyzer, as well as with a glass capillary viscometer. All measurements on the DS2500 Solid Analyzer were performed in rotation to average the subsample spectra. This setup with the large sample cup reduces the influence of the particle size distribution of the polymer pellets (Figure 1). Data acquisition and prediction model development was performed with the software package Vision Air Complete.



Figure 1. Metrohm DS2500 Solid Analyzer with the DS2500 large sample cup for measuring the intrinsic viscosity of recycled polyethylene terephthalate (rPET).

Table 1. Hardware and software equipment overview.

Equipment	Article number
DS2500 Solid Analyzer	2.922.0010
DS2500 large sample cup	6.7402.050
Vision Air 2.0 Complete	6.6072.208

RESULT

All 48 measured Vis-NIR spectra (Figure 2) were used to create a prediction model for quantification of intrinsic viscosity. The quality of the prediction model was evaluated using correlation diagrams which display a high correlation (R^2 = 0.9061) between the Vis-NIR prediction and the reference viscosity values. The respective figures of merit (FOM) display the expected precision and confirm the feasibility during routine analysis (Figure 3).

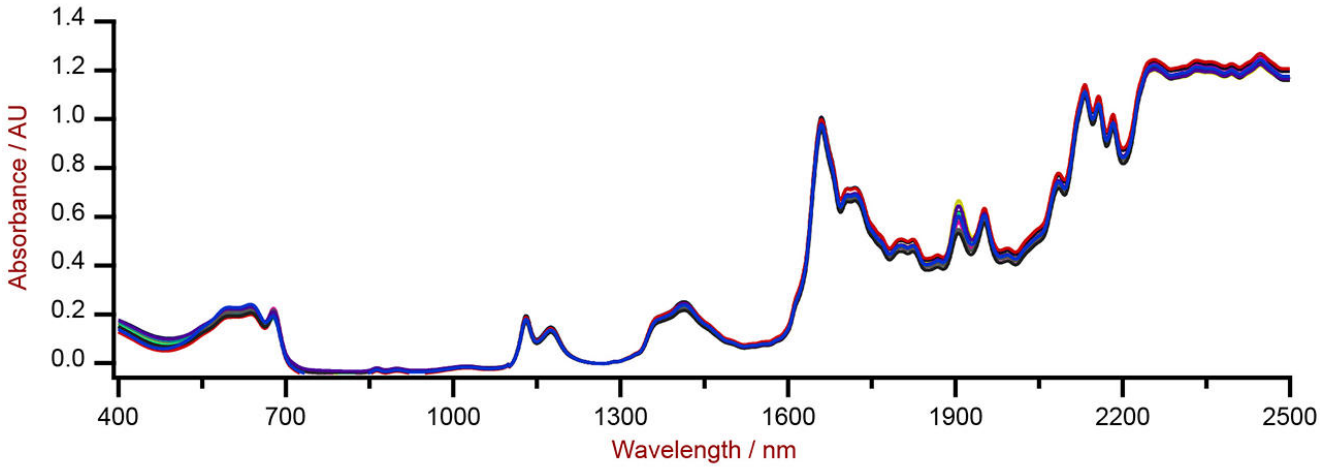


Figure 2. Selection of Vis-NIR spectra of rPET samples analyzed on a DS2500 Solid Analyzer with the large sample cup.

RESULT INTRINSIC VISCOSITY

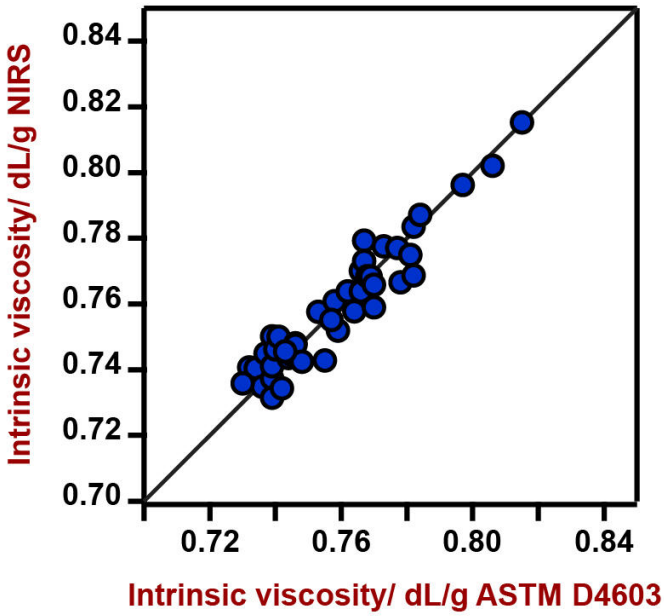


Figure 3. Correlation diagram and the respective figures of merit for the prediction of intrinsic viscosity in rPET using a DS2500 Solid Analyzer. The lab value was evaluated according to ASTM D4603.

Figures of Merit	Value
R^2	0.9061
Standard Error of Calibration	0.0068 dL/g
Standard Error of Cross-Validation	0.0084 dL/g

CONCLUSION

This Application Note demonstrates the feasibility of the Metrohm DS2500 Solid Analyzer for the determination of intrinsic viscosity in rPET. Vis-NIR spectroscopy enables fast determination (Table 2) without any sample preparation. In addition, the use of toxic chemicals is obsolete which increases lab safety and avoids chemical waste. Next to intrinsic viscosity, additional parameters like diethylene glycol or isophthalic acid can be determined in rPET with Vis-NIR spectroscopy.

Table 2. Time to result overview for the parameter intrinsic viscosity (IV) in recycled polyethylene terephthalate (rPET).

Parameter	Method	Time to result
Intrinsic viscosity	Viscometer (ASTM D4603)	2–3 h drying, dissolving, and measuring

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CONFIGURATION



DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.

The 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 DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.

The DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The 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 9 samples.



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.