



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

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**).

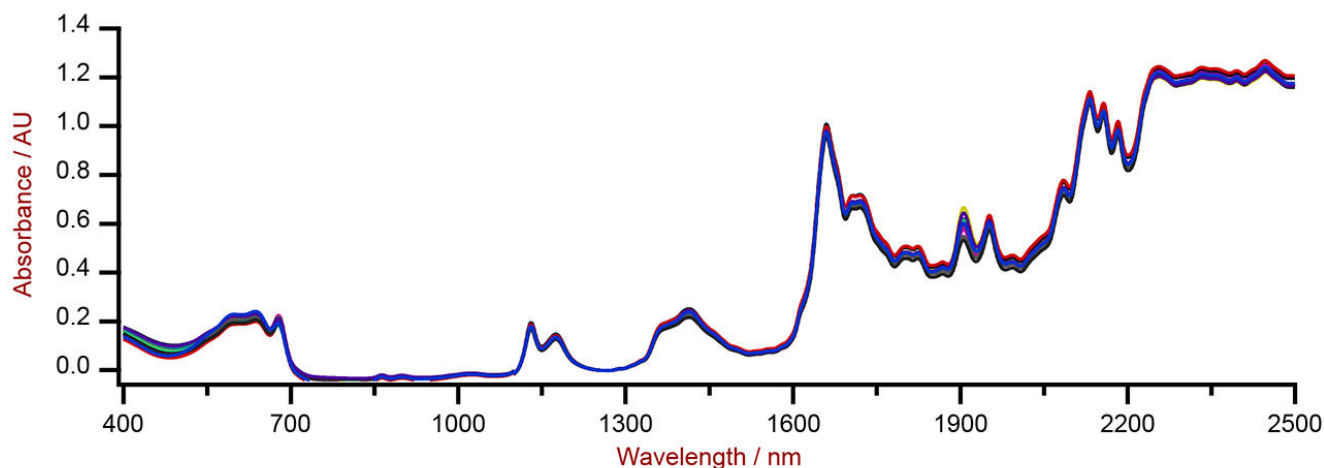


Figure 2. Selection of Vis-NIR spectra of rPET samples analyzed on a DS2500 Solid Analyzer with the large sample cup.

RESULT INTRINSIC VISCOSITY

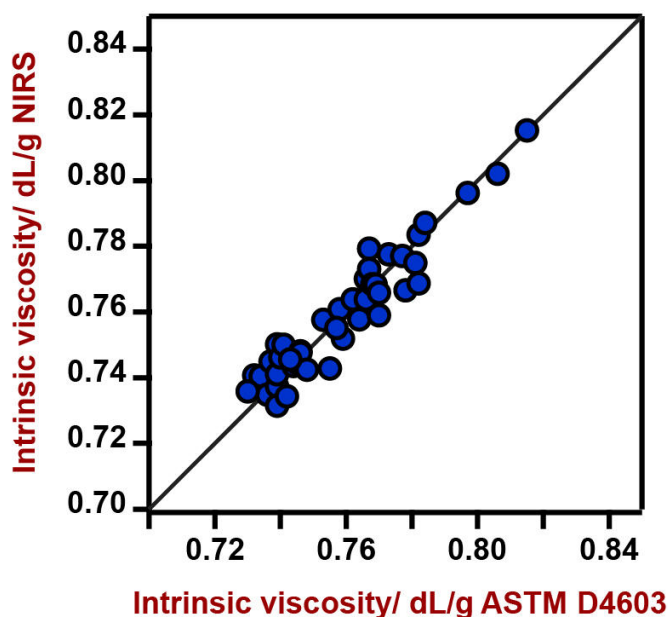


Figure 3. Correlation diagram and the respective figures of merit for the prediction of intrinsic viscosity in rPET using a DS2500 Solid Analyzer. The lab value was evaluated according to ASTM D4603.

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

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

ラボおよび生産環境における品質管理用の堅牢な近赤外分光法。

DS2500 Analyzerは、生産チェーン全体に沿った固形物、クリーム、およびオプションとしての液体のルーチン分析に実績のあるフレキシブルなソリューションです。頑丈な仕様により、DS2500 Analyzerは粉塵、湿気、振動や温度変動に強い為、過酷な生産環境での使用に理想的です。

DS2500は400 ~ 2500 nmのスペクトル範囲全体をカバーし、1分以内に正確で再現性の高い結果を提供します。DS2500 Analyzerは製薬業界の要件を満たしており、簡単な操作により日常的な作業においてユーザーをサポートします。

装置に完全に適応した付属品により、顆粒のような粒の荒い固形物、またはクリームのような半固形液体サンプルなどのあらゆる困難なタイプのサンプルにおいても、最良の結果を得ることができます。固形物の測定においては、9つまでのサンプルのシリーズの自動測定を可能にするMultiSample Cupを使用することで、生産性を高めることができます。



DS2500

NIRS DS2500 Analyzerを用いた、様々なサンプル位置における反射中の粉末および顆粒のスペクトル記録のための、大きなサンプル容器です。