



Application Note AN-NIR-112

近红外光谱法测定回收PET的特性粘度(IV)

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**).

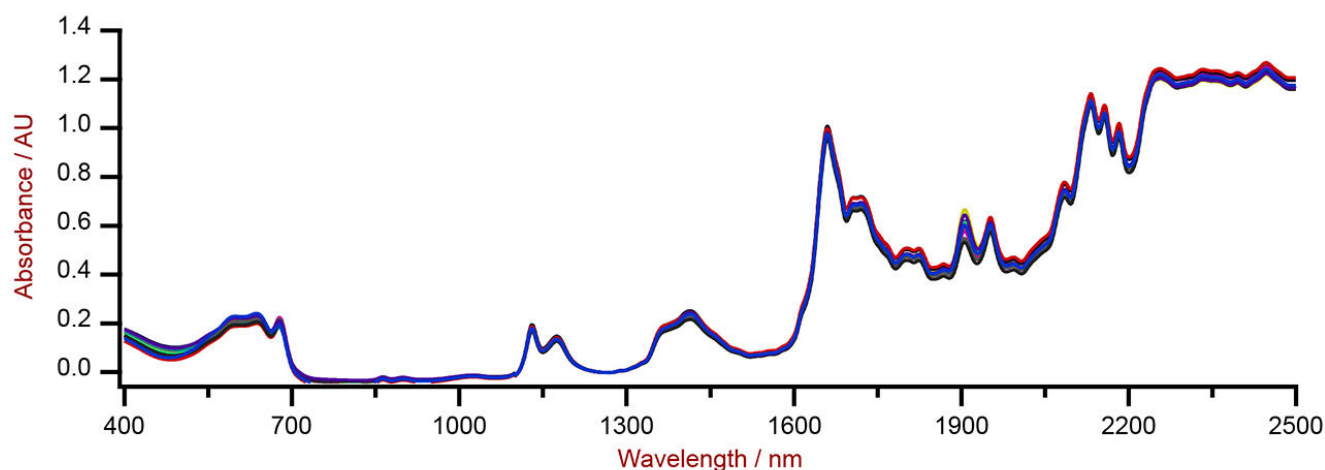


Figure 2. Selection of Vis-NIR spectra of rPET samples analyzed on a DS2500 Solid Analyzer with the large sample cup.

RESULT INTRINSIC VISCOSITY

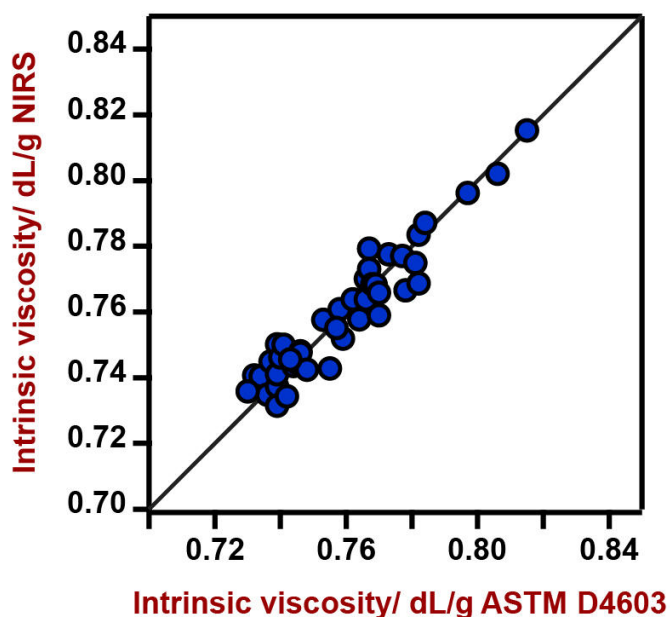


Figure 3. Correlation diagram and the respective figures of merit for the prediction of intrinsic viscosity in rPET using a DS2500 Solid Analyzer. The lab value was evaluated according to ASTM D4603.

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

坚固结实的近红外光谱仪,用于生产环境和试验室中的质量检查。

DS2500 分析仪是经过验证的灵活解决方案,用于对整个生产链过程中的固体、乳膏和液体进行常规分析。其坚固结实的设计使 DS2500 Analyzer 分析仪不受灰尘、湿度、振动和温度波动的影响,因此非常适用于在恶劣的生产环境中使用。

DS2500 涵盖了从 400 到 2500 nm 的整个光谱范围,并能在不到一分钟时间内提供准确和可再现的结果。

DS2500 Analyzer 满足制药行业的要求,并由于操作简便而能帮助用户完成其日常工作任务。

由于与设备匹配,附件可以承受任何具有挑战性的样品类型,例如:粒料之类的粗颗粒固体或乳膏之类的半固体样品,可获得结果。测量固体的时候,使用 MultiSample Cup 可以提高生产率,可以自动批量测量多 9 个样品。



DS2500

用于在不同样品位置处使用 NIRS DS2500 Analyzer
采集粉末和颗粒反射光谱的大号样品容器。