Application Note AN-NIR-082

Quality Control of Polypropylene

Non-destructive determination of melt flow rate without rheological tests

As a general purpose resin, polypropylene (PP) is widely used in industries such as electronic manufacturing and construction, and is used in packaging materials due to its insulating and processing properties. PP resins must be melted first in order to be formed into the intended shape, and therefore flow properties are important characteristics which affect the production process. One parameter that describes the flow characteristics is the melt flow rate (MFR). This is a measure of the mass of material that extrudes from the die over a given period of time (ASTM D1238). The standard procedure requires a significant amount of work with packing the sample, preheating, and cleaning. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows the analysis of MFR in **less than a minute**.

EXPERIMENTAL EQUIPMENT

PP pellets were measured with a DS2500 Solid Analyzer in reflection mode over the full wavelength range (400–2500 nm). To minimize particle size effects, a rotating DS2500 Large Sample Cup was employed. This accessory enables an automated measurement at different sample locations for a reproducible spectrum acquisition. As displayed in Figure 1, samples were measured without any sample preparation. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

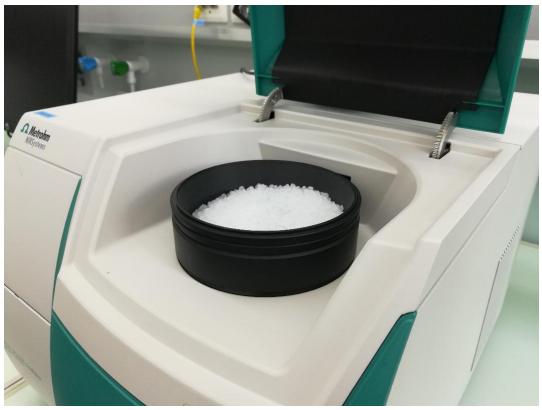

Figure 1. DS2500 Solid Analyzer with PP pellets filled in the rotating DS2500 Large Sample Cup.

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

RESULT

The obtained Vis-NIR spectra (Figure 2) were used to create prediction models for quantification of the density content. 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.

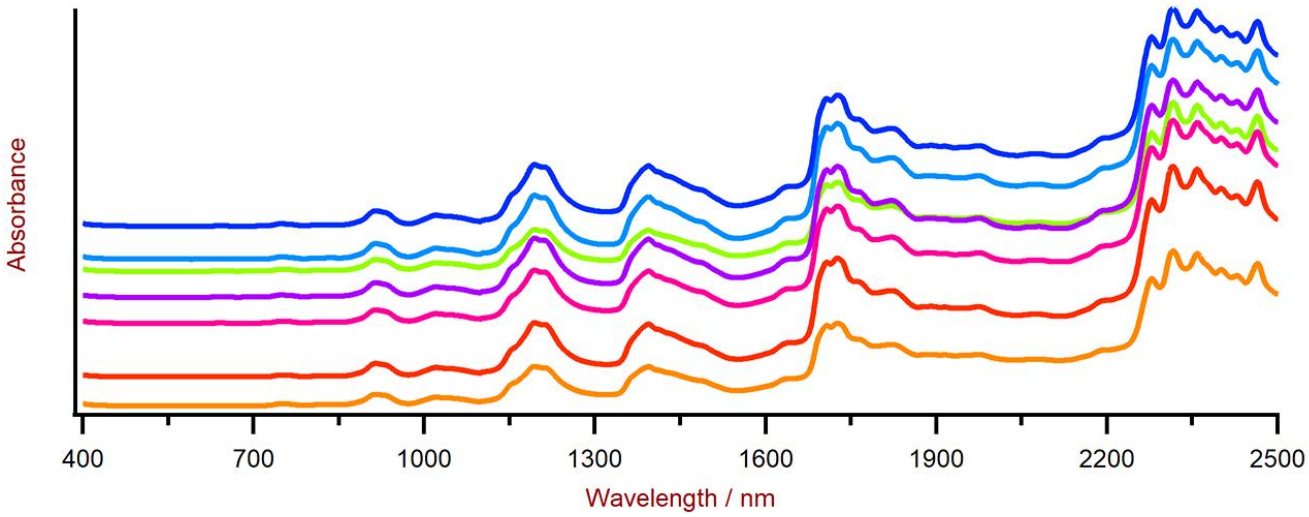

Figure 2. Display of a selection of PP Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Large Sample Cup. An offset has been applied to the spectra to make them easier to view.

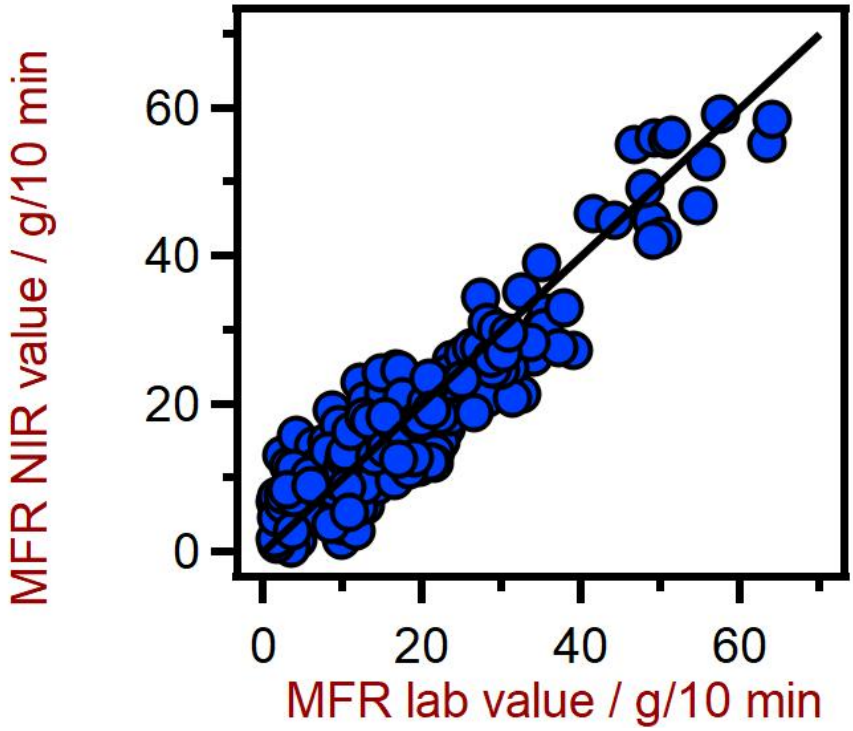

Figure 3. Correlation diagram for the prediction of the MFR using a DS2500 Solid Analyzer. The lab values were obtained using a melt flow indexer.

Table 2. Figures of merit for the prediction of the melt flow rate (MFR) of polypropylene samples using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.865
Standard error of calibration	4.99 g / 10 min
Standard error of cross-validation	7.00 g / 10 min

CONCLUSION

This application note demonstrates the feasibility of NIR spectroscopy for the analysis of MFR in polypropylene samples. In comparison to the standard method (ASTM D1238) (**Table 3**), the **reduction of analysis time and workload** is a major advantage of NIR spectroscopy.

Table 3. Time to result overview for the melt flow rate determination with the standard ASTM D1238 method.

Parameter	Method	Time to result and workflow
Melt flow rate	Extrusion ASTM D1238	~20 minutes; packing material, preheating, measuring, cleaning

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DS2500 Solid Analyzer

固耐用的近外光,用于生境和室中的量。

DS2500 分析是的活解决方案,用于整个生程中的固体、乳膏和液体行常分析。其固耐用的使 DS2500 Analyzer 分析不受灰、湿度、振和温度波的影,因此非常用于在劣的生境中使用。

DS2500 涵盖了从 400 到 2500 nm 的整个光范,并能在不到一分内提供准和可再的果。DS2500 Analyzer 足制行的要求,并由于操作便而能助用完成其日常工作任。

由于与完美匹配,附件可以承受任何具有挑性的品型,例如:粒料之的粗粒固体或乳膏之的半固体品,可得最佳果。量固体的候,使用 MultiSample Cup 可以提高生率,可以自批量量最多 9 个品。

Vision Air 2.0 Complete

Vision Air – 通用的光分析件。

Vision Air Complete 是用于管范境的先易用的件解决方案。

Vision Air 点一:

- 独特的件用和配的用界面保了直的操作方式
- 操作程的建与方式
- SQL 数据,可安全且地管理数据

Vision Air Complete (66072208) 版本包含所有用于可近外光分析量保程的用:

- 器和数据管理用
- 方法用
- 常分析用

其它 Vision Air Complete 解决方案:

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)

DS2500

用于在不同品位置使用 NIRS DS2500 Analyzer 采集粉末和粒反射光的大号品容器。