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.

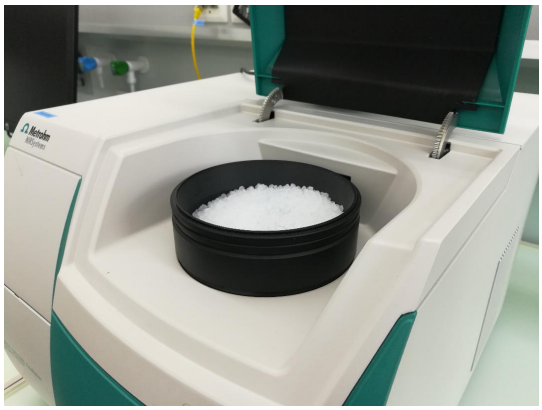

Figure 1. DS2500 Solid Analyzer with PP pellets filled in the rotating DS2500 Large Sample Cup.

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.

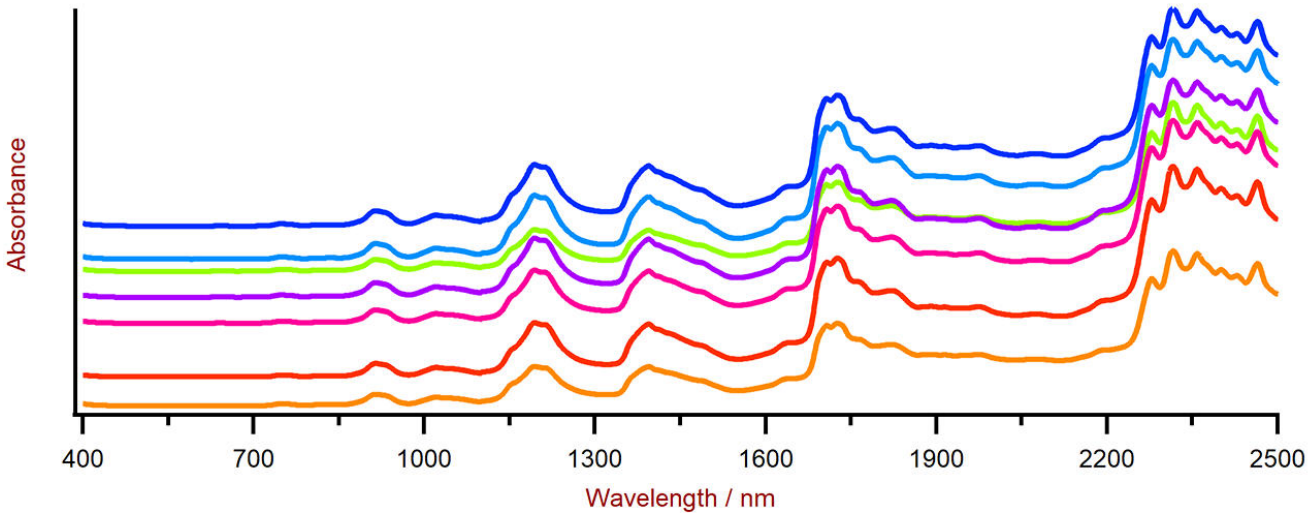

Figure 2. Display of a selection of PP Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Large Sample Cup. An offset has been applied to the spectra to make them easier to view.

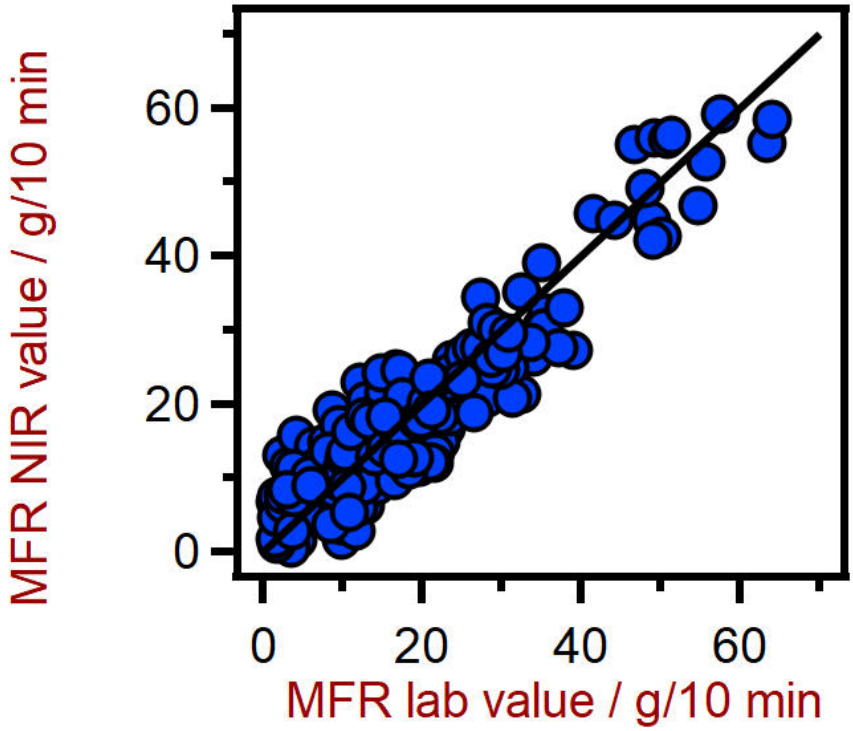

Figure 3. Correlation diagram for the prediction of the MFR using a DS2500 Solid Analyzer. The lab values were obtained using a melt flow indexer.

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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