



Application Note AN-NIR-082

聚丙烯的质量控制

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

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。



Vision Air 2.0 Complete

Vision Air – 通用的光谱分析软件。

Vision Air Complete 是用于监管规范环境的先进易用的软件解决方案。

Vision Air 优点一览:

- 便捷的软件应用和经过适配的用户界面保证了直观简单的操作方式
- 操作过程的创建与维护方式简单
- SQL 数据库,可安全且简单地管理数据

Vision Air Complete (66072208) 版本包含所有用于可见近红外光谱分析质量保证过程的应用:

- 仪器和数据管理应用
- 方法开发应用
- 常规分析应用

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。