

Application Note AN-NIR-081

Quality Control of Polyethylene

Reliable determination of PE density within one minute using NIRS

SUMMARY

Determination of the density of polyethylene (PE) (ASTM D792) is normally a challenging procedure due to reproducibility difficulties. Since this parameter is most frequently used to determine the PE type, alternative methods which are sensitive to molecular structure such as FT-IR spectroscopy are also common. However, measurement via FT-IR can also be problematic when larger sample sizes must be analyzed due to sample inhomogeneity.

This application note demonstrates that the DS2500 Solid Analyzer operating in the visible and near-infrared spectral region (Vis-NIR) provides a **reliable and fast solution** for determination of the density of PE. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows the analysis of larger, inhomogeneous sample sizes of PE in **less than a minute**.

EXPERIMENTAL EQUIPMENT

PE pellets were measured in reflection mode over the full wavelength range (400–2500 nm) of the DS2500 Solid Analyzer. A rotating DS2500 Large Sample Cup was employed to overcome the distribution of varied particle sizes and chemical components. This allowed automated measurements at different sample locations for a reproducible spectrum acquisition. As displayed in **Figure 1**, samples were measured without any preparation. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.



Figure 1. DS2500 Solid Analyzer and PE pellets present 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 relationship between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

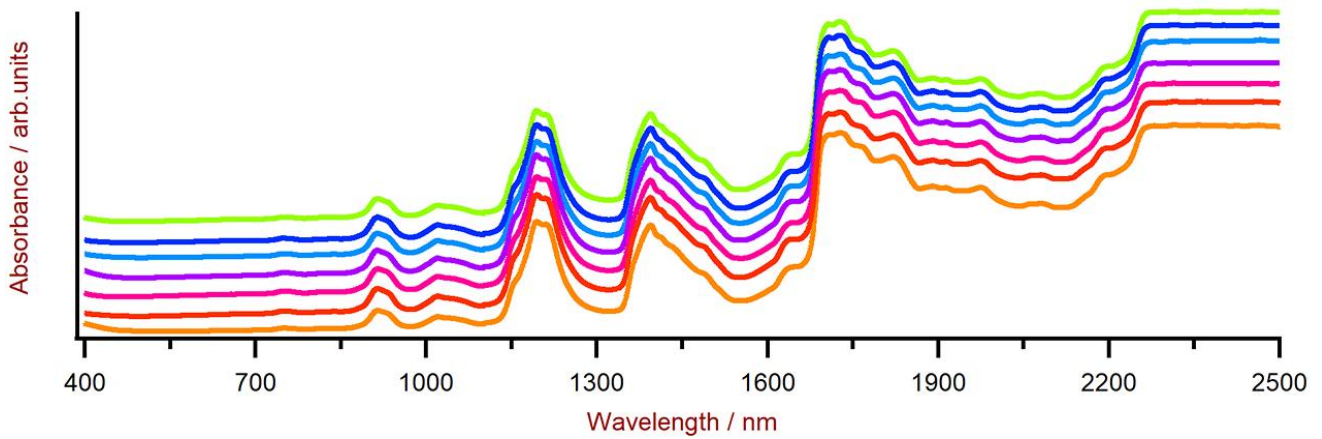


Figure 2. Selection of PE Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating DS2500 Large Sample Cup. For display reasons a spectra offset was applied.

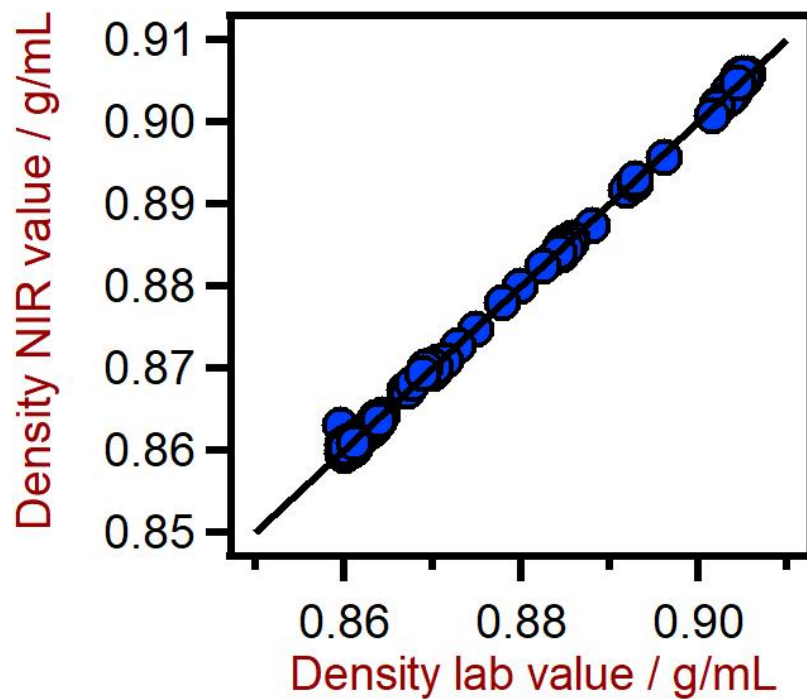


Figure 3. Correlation diagram for the prediction of the density of PE using a DS2500 Solid Analyzer. The density lab value was evaluated using densimetry.

Table 2. Figures of merit for the prediction of the density of PE using a DS2500 Solid Analyzer.

Figures of merit	Value
R ²	0.991
Standard error of calibration	0.0005 g/mL
Standard error of cross-validation	0.0005 g/mL

CONCLUSION

This application note demonstrates that the density of PE can be determined easily with NIR spectroscopy. Since **no sample preparation is needed**, samples are

analyzed as they are, which allows for simple operation leading to highly precise results (0.0005 g/mL, see **Table 2**).

CONTACT

Metrohm AG
Ionenstrasse
9100 Herisau

info@metrohm.com

EXPERIMENTAL EQUIPMENT



DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.

The DS2500 Analyzer is the tried and tested, flexible solution for routine analysis of solids, creams, and optionally also liquids along the entire production chain. Its robust design makes the DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.

The DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The DS2500 Analyzer meets the demands of the pharmaceutical industry and supports users in their day-to-day routine tasks thanks to its simple operation.

Thanks to accessories tailored perfectly to the instrument, optimum results are achieved with every sample type, no matter how challenging it is, e.g. coarse-grained solids such as granulates or semi-solid samples such as creams. The MultiSample Cup can help improve productivity when measuring solids, as it enables automated measurements of series containing up to 9 samples.



Vision Air 2.0 Complete

Vision Air - Universal spectroscopy software.

Vision Air Complete is a modern and simple-to-operate software solution for use in a regulated environment.

Overview of the advantages of Vision Air:

- Individual software applications with adapted user interfaces ensure intuitive and simple operation
- Simple creation and maintenance of operating procedures
- SQL database for secure and simple data management

The Vision Air Complete version (66072208) includes all applications for quality assurance using Vis-NIR spectroscopy:

- Application for instrument and data management
- Application for method development
- Application for routine analysis

Additional Vision Air Complete solutions:

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



DS2500 large sample cup

Large sample cup for the spectral recording of powders and granulates in reflection at various sample positions using the NIRS DS2500 Analyzer.