Application Note AN-NIR-098

Quality Control of PVC granulate

Determine molecular weight easily within seconds

PVC (polyvinyl chloride) has unique properties when compared to other olefin-derived plastics that only contain carbon and hydrogen atoms in their chemical structure. Some PVC features include increased chemical and mechanical stability as well as fire retardant properties. The molecular weight of the polymer has a significant influence on these properties. Molecular weight is defined here as the average weight of the molecules that make up a polymer, and this value gives an indication of the length of the polymer chains. To monitor the PVC quality, it is important to measure the molecular weight during the production process. The standard method to determine PVC molecular weight is by size exclusion chromatography (SEC). This analytical method is time-intensive and requires trained personnel to perform.

Determining the molecular weight of PVC is easier with near-infrared spectroscopy (NIRS). NIRS providesresultsin just a few seconds and can quickly indicate when adjustments to the production process are necessary.

EXPERIMENTAL EQUIPMENT

33 PVC samples with varying molecular weights from 113000–192000 g/mol were measured on the DS2500 Solid Analyzer. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

Figure 1. DS2500 Solid Analyzer

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
NIRS DS2500 Solid Analyzer	2.922.0010
Vision Air 2.0 Complete	6.6072.208
NIRS large sample cup	6.7402.050

RESULT

All 33 measured Vis-NIR spectra (Figure 2) were used to create a prediction model for quantification of molecular weight. The quality of the prediction model was evaluated using the cross-validation algorithm which displays a very high correlation between Vis-NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision of a NIRS prediction during routine analysis (Table 2).

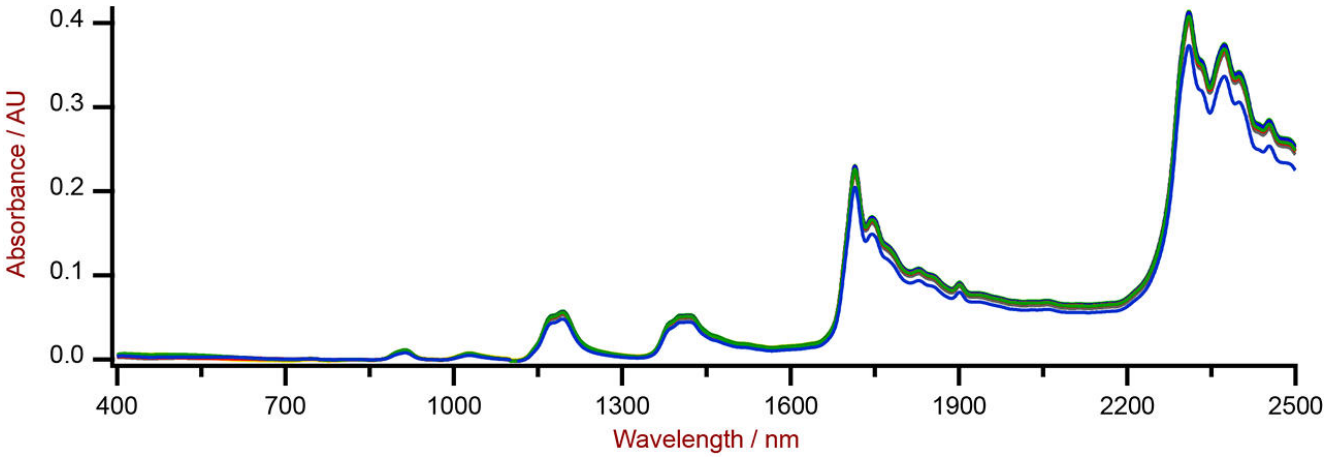

Figure 2. Vis-NIR spectra of PVC granulate samples with different molecular weights measured on a DS2500 Solid Analyzer.

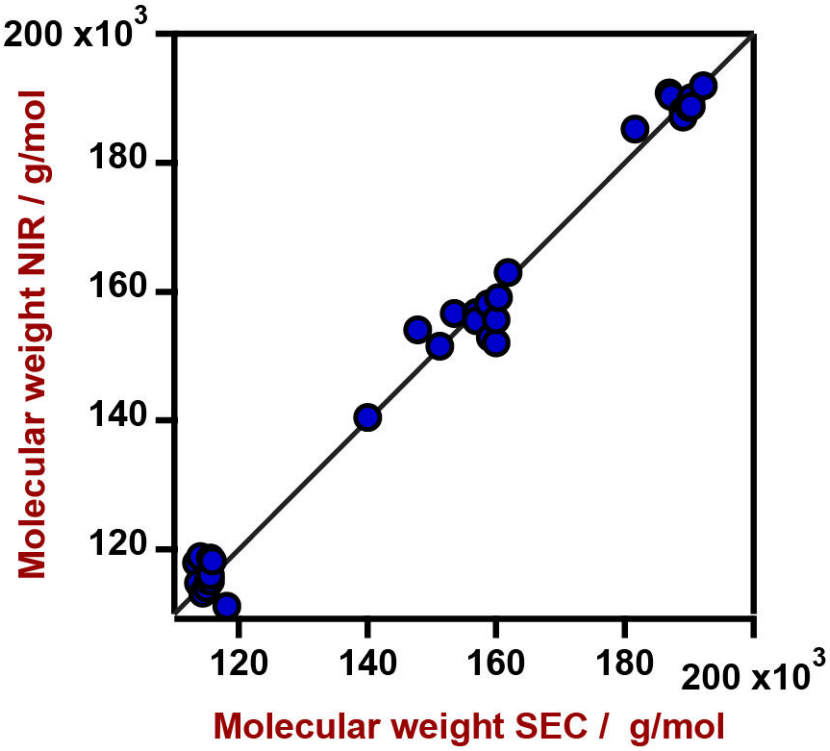

Figure 3. Correlation diagram for the prediction of PVC molecular weight using a DS2500 Solid Analyzer.

Table 2. Figures of merit for the prediction of the molecular weight of PVC granulate samples using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.988
Standard error of calibration	3640 g/mol
Standard error of cross-validation	5375 g/mol

CONCLUSION

This application note demonstrates the feasibility to determine the molecular weight in PVC granulate samples with NIR spectroscopy. The PVC molecular weight was successfully determined with NIRS only differing from the reference data with an average of 1.5%. Vis-NIR spectroscopy enables a fast determination with no sample preparation and has proven itself as a suitable method to measure PVC molecular weight.

CONTACT

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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.