



Application Note AN-NIR-076

Quality control of polyvinyl alcohol

Improved polymer quality control with NIR spectroscopy

Polyvinyl alcohol (PVA) is used in a variety of medical products (e.g. eye drops) due to its low toxicity, low protein adhesion, and film forming properties. PVA is a linear polymer, which forms copolymers of vinyl acetate and vinyl alcohol. The degree of alcoholysis is the percentage of hydroxyl functional groups compared to the total functional groups accessible in the molecule. It is an important index for the water solubility, viscosity, and adhesion of the product. Conventional alcoholysis determination requires each

sample to be weighed, dissolved, heated, cooled, and titrated. This procedure can take up to six hours per sample. Compared to the primary method, analysis with near-infrared spectroscopy (NIRS) only takes one minute. The following application note describes the determination of the degree of alcoholysis by NIRS. Aside from alcoholysis, additional quantification methods for sodium acetate and volatiles can be established.

EXPERIMENTAL CONDITIONS

54 spectra from 18 different sample batches were collected using a Metrohm DS2500 Solid Analyzer in combination with the Vision Air Complete spectroscopy software. To overcome sample inhomogeneity, the measurement was performed with a large sample cup in rotation. The reference values were obtained by titration. Outlier detection was performed on pre-processed spectra (2nd derivative) using a maximum distance in wavelength space algorithm. The NIRS prediction model was created with the settings described in the following table, and validated using cross validation.



Figure 1. The DS2500 Solid Analyzer was used to collect the spectra of PVA polymer.

Pre-processing	Algorithm	Validation type
2 nd derivative	PLS	Cross-validation

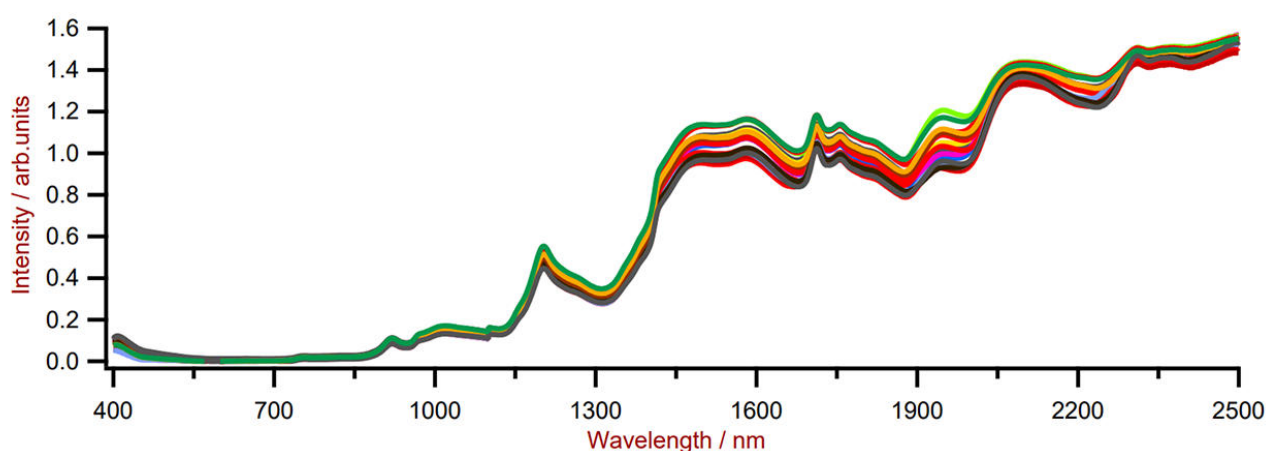


Figure 2. Selection of polymer spectra with varying degrees of alcoholysis.

RESULT & CONCLUSION

The obtained correlation graph displays a high correlation ($R^2 = 0.98$) between alcoholysis degree predicted by NIRS and the primary lab method. A

close ratio between SEC and SECV (< 10%), proves that the method is valid.

# Factors	R^2	SEC	SECV
3	0.98	0.24%	0.25%

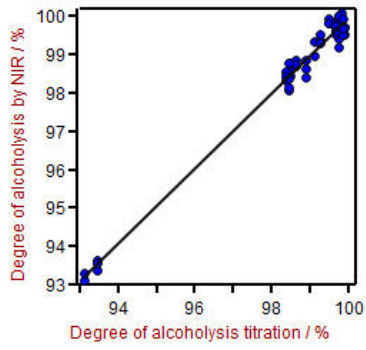


Figure 3. Correlation graph for alcoholysis degree predicted by NIRS vs. lab method.

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CONFIGURATION



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.