Moisture analysis in caprolactam

Moisture control without chemical waste

Summary

Caprolactam is an important polymer used for the production of Nylon 6, which is the base material for industrial fibers. Other application areas for caprolactam are in the resin or plastic /polymer industry. Due to its commercial significance, many different synthesis methods have been developed over the years. Around 90% of the global production is synthesized from cyclohexanone, which is first converted to its oxime and then treated with acid to create the final product. Caprolactam is hygroscopic and water soluble, therefore it is important to have a reliable analysis technique for water determination.

Analyzing the water content by conventional methods requires each sample to be weighed, dissolved, heated, and titrated. Compared to the primary method, near-infrared spectroscopy (NIRS) offers unique advantages: it generates reliable results within seconds, but it does not need any sample preparation nor does it create chemical waste.

Configuration

2.922.0010 - DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.The NIRS 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 NIRS DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.The NIRS DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The NIRS 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 nine samples.

6.6072.208 - 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);

6.7402.050 - 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.

Experimental Conditions

Figure 1. The DS2500 Solid Analyzer was used to collect spectra of caprolactam samples.

44 spectra of samples with different moisture content 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 KF-titration. Outlier detection was performed on pre-treated spectra (2^{nd} 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.

Pre-Processing	Algorithm	Validation Type
2nd derivative	PLS	Validation Set

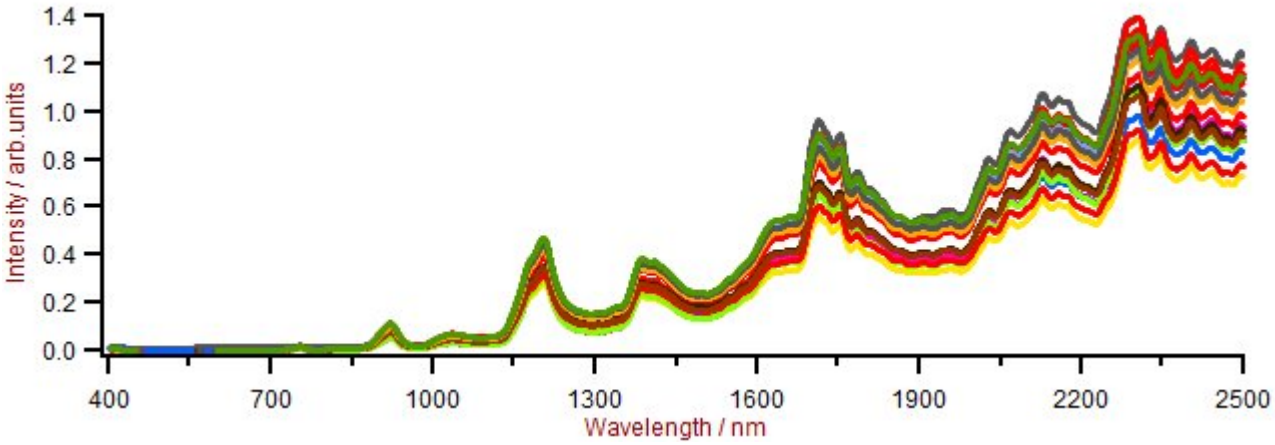

Figure 2. Caprolactam samples with varying water content.

Result & Conclusion

The obtained correlation graph displays a high correlation (R^2 = 0.98) between moisture predicted by NIRS and the titration method. SEC and SECV values are in the range of 60 mg/L, which proves that NIRS is a sensitive and suitable technique for moisture determination.

# Factors	R^2	SEC	SECV
2	0.98	53 mg/L	58 mg/L

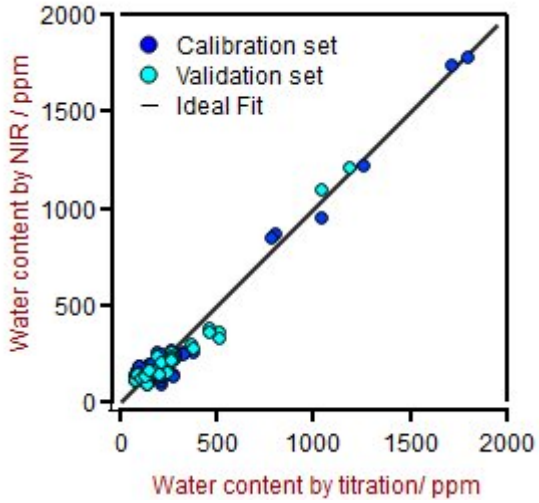

Figure 3. Correlation graph for moisture predicted by NIRS vs titration.

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