



Application Note AN-NIR-077

Moisture analysis in caprolactam

Moisture control without chemical waste

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.

EXPERIMENTAL CONDITIONS

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 (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.

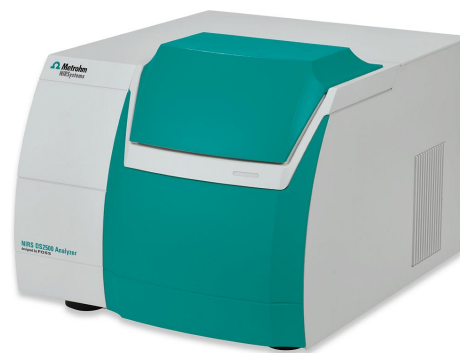


Figure 1. The DS2500 Solid Analyzer was used to collect spectra of caprolactam samples.

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

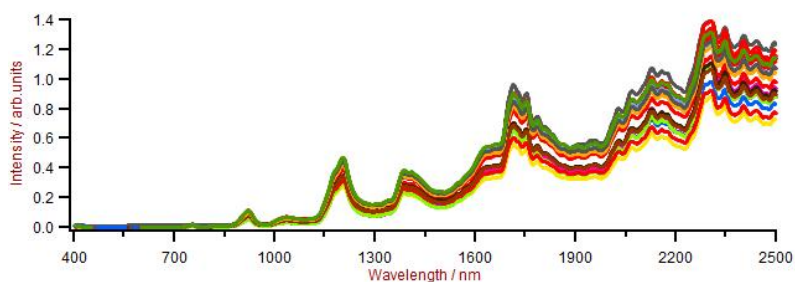


Figure 2. Caprolactam samples with varying water content.

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 |

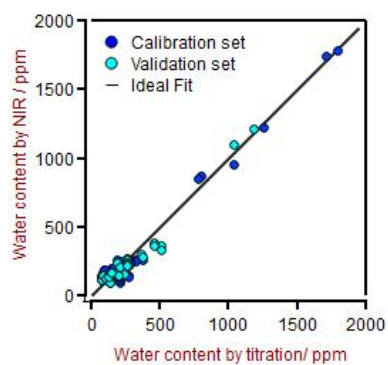


Figure 3. Correlation graph for moisture predicted by NIRS vs titration.

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