Application Note AN-NIR-064

Quality control of ammonium nitrate

Rapid and non-destructive moisture determination with NIRS

Specialty chemicals have to fulfill multiple quality requirements. One of these quality parameters, which can be found in almost all certificates of analysis and specifications, is the moisture content. The standard method for the determination of moisture content is Karl Fischer titration.

This method requires reproducible sample preparation, chemicals, and waste disposal. Alternatively, near-infrared spectroscopy (NIRS) can be used for the determination of moisture content. With this technique, samples can be analyzed without any preparation and without using any chemicals.

EXPERIMENTAL EQUIPMENT

Ammonium nitrate samples were measured with a DS2500 Solid Analyzer in reflection mode over the full wavelength range (400–2500 nm). To minimize particle size effects, a rotating DS2500 large sample cup was employed. This accessory enables an automated measurement at different sample locations for a reproducible spectrum acquisition. As displayed in Figure 1, samples were measured without any preparation steps. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

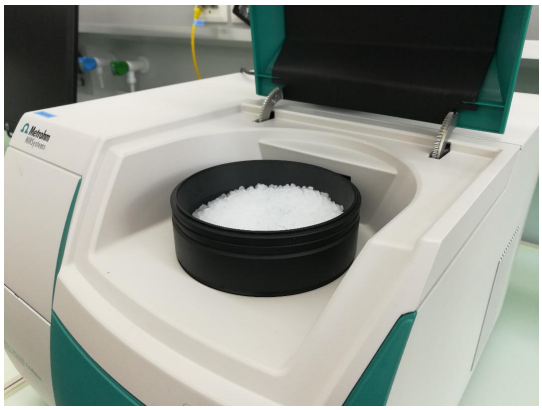

Figure 1. Samples filled in the rotating DS2500 large sample cup.

Table 1. Hardware and software equipment overview.

Equipment	Article number
DS2500 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 a prediction model for the moisture value determination. To verify the quality of the prediction model, correlation diagrams were created which display the correlation between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

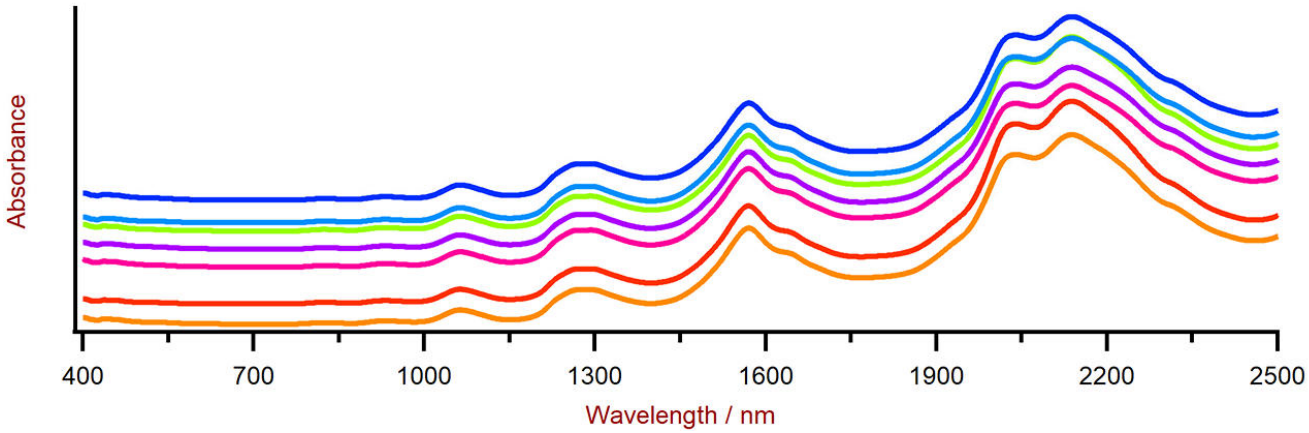

Figure 2. A selection of ammonium nitrate Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating sample cup. For display reasons a spectra offset was applied.

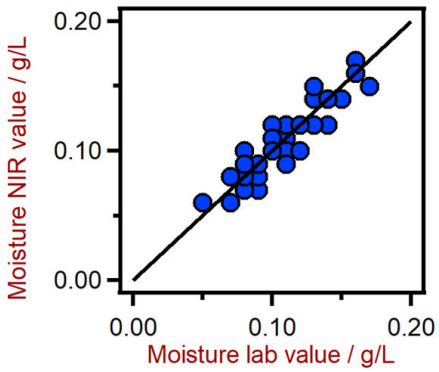

Figure 3. Correlation diagram for the prediction of moisture content using a DS2500 Analyzer. The reference lab values were evaluated with coulometric Karl Fischer titration.

Table 2. Figures of merit for the prediction of the moisture content using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.840
Standard error of calibration	0.011 g/L
Standard error of cross-validation	0.012 g/L

CONCLUSION

This Application Note demonstrates the feasibility of NIR spectroscopy for the analysis of low moisture content in specialty chemicals, specifically ammonium nitrate. In comparison to the standard titration method, the **reduction of analysis time and chemicals** is a major advantage of NIR spectroscopy.

Table 3. Comparison of running costs for the determination of the moisture content with titration and NIR spectroscopy.

	Lab method	NIR method
Number of analyses (per day)	10	10
Cost of operator (per hour)	$25	$25
Costs of chemicals for water determination	$2	$0
Time spent per analysis	5 min	1 min
Total running costs (per year)	**$8,288**	**$938**

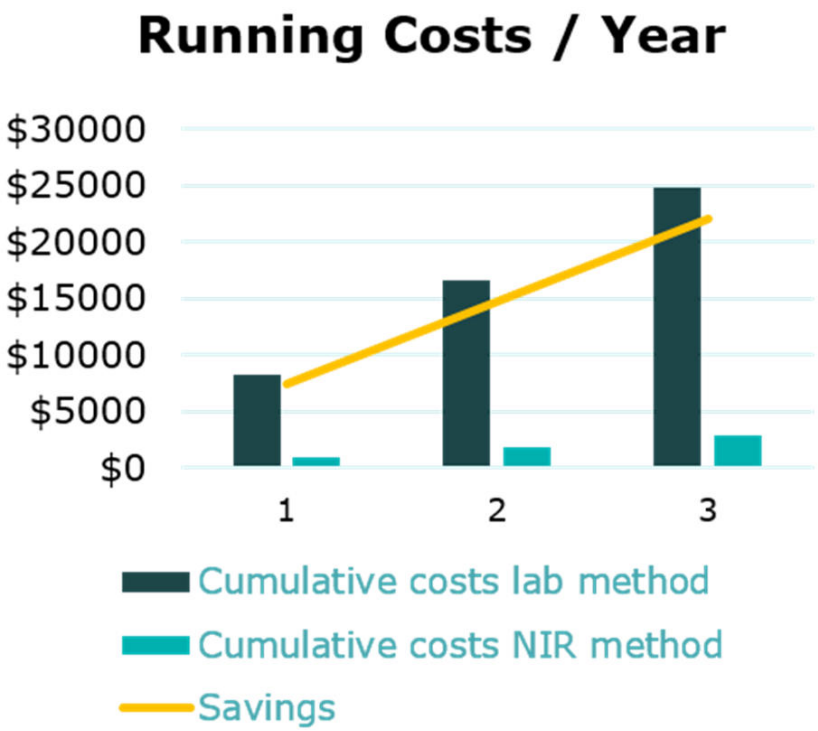

Figure 4. Comparison of the cumulative costs over three years for the determination of water content with coulometric Karl Fischer titration and NIR spectroscopy.

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.

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.

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)