Application Note AN-NIR-064

Quality control of Ammonium Nitrate

Rapid and non-destructive moisture determination

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.

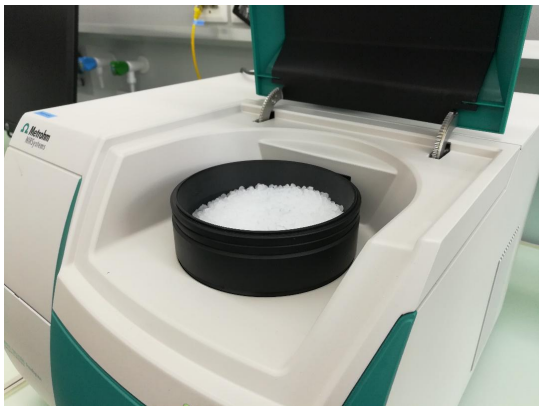

Figure 1. Samples filled in the rotating DS2500 Large Sample Cup.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Solid 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.

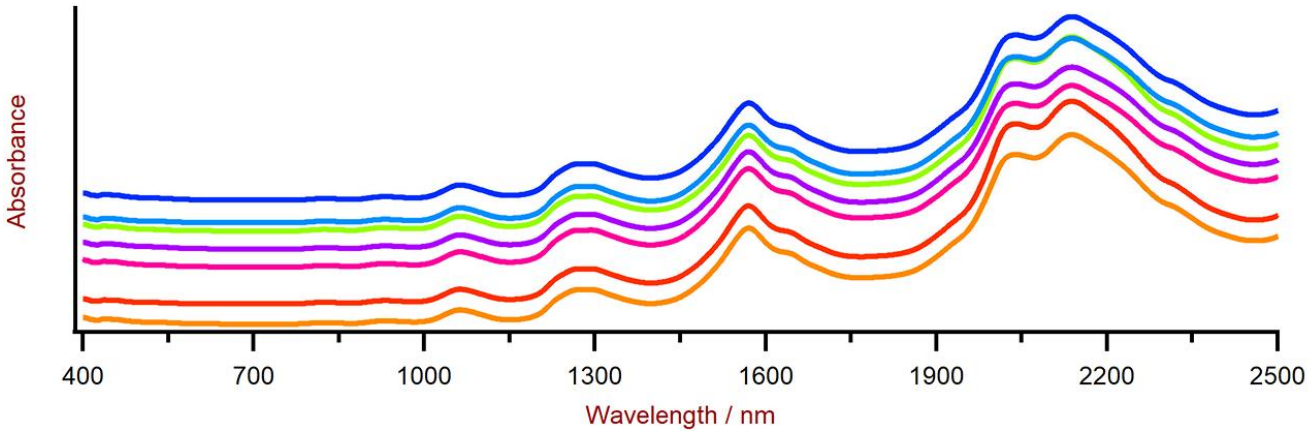

Figure 2. A selection of ammonium nitrate Vis-NIR spectra obtained using a DS2500 Analyzer and a rotating sample cup. For display reasons a spectra offset was applied.

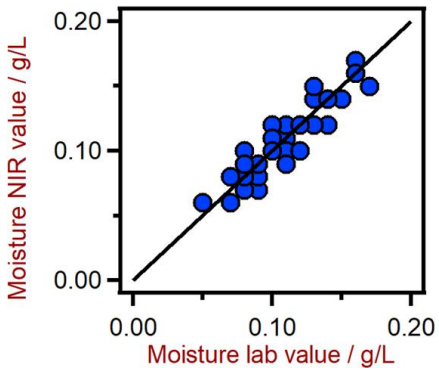

Figure 3. Correlation diagram for the prediction of moisture content using a DS2500 Analyzer. The reference lab values were evaluated with volumetric Karl Fischer titration.

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 mg/L
Standard error of cross-validation	0.012 mg/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

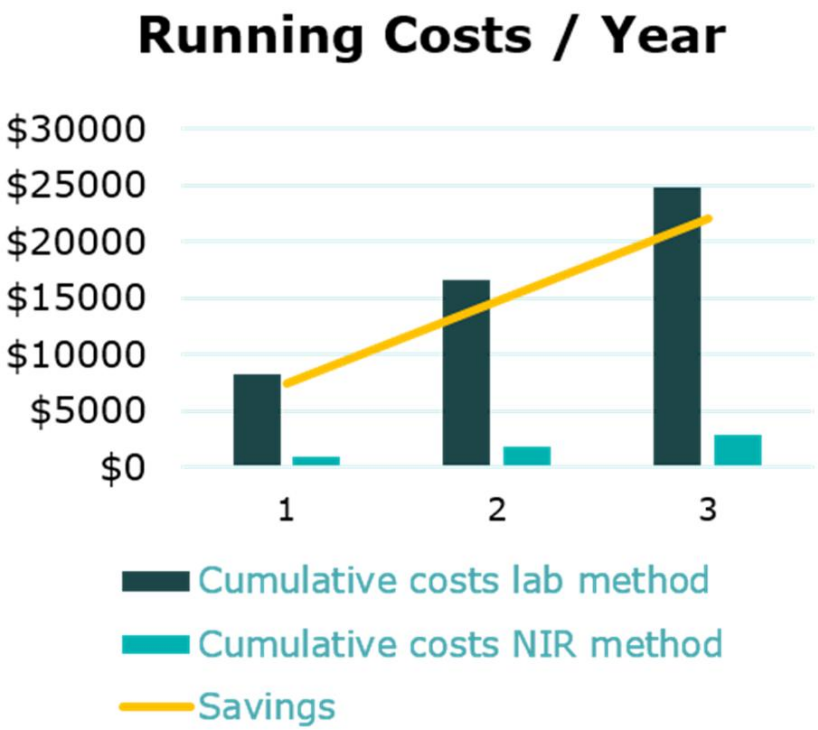

Figure 4. Comparison of the cumulative costs other three years for the determination of water content with volumetric titration and NIR spectroscopy.

CONTACT

Metrohm Brasil
Rua Minerva, 161
05007-030 São Paulo
metrohm@metrohm.com.br