NIR APPLICATION NOTE NIR-071

Quality Control of Lubricants

Unassisted, rapid determination of the Acid Number by automated NIR spectroscopy according to ASTM E1655

Acid Number (AN) analysis of lubricants (ASTM D664) can be a lengthy and costly process due to usage of large amounts of chemicals and required cleaning steps of the analytical equipment between each measurement.

This application note demonstrates that the XDS RapidLiquid Analyzer operating in the visible and near-infrared spectral region (Vis-NIR) provides a cost-efficient, fast alternative for the determination of the acid number of lubricants. With **no sample preparation or chemicals needed**, Vis-NIR spectroscopy allows for the analysis of AN in **less than a minute**.

EXPERIMENTAL EQUIPMENT

Lubricant samples were measured in transmission mode over the full wavelength range (400 nm to 2500 nm) using a XDS RapidLiquid Analyzer in combination with an 815 Robotic USB Sample Processor, which can carry a total of 141 samples. Reproducible spectrum acquisition was achieved using the built-in temperature control (at 30°C) of the XDS RapidLiquid Analyzer. The Metrohm software packages Tiamo and Vision Air Complete were used for all data acquisition and prediction model development.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
XDS RapidLiquid Analyzer	2.921.1410
815 Robotic USB Sample Processor XL (Sample Rack 141 x 11 ml)	2.815.0010
800 Dosino	2.800.0020
5.0 mm flow cell	Hellma
Vision Air complete	6.6072.208
Tiamo	6.6056.021

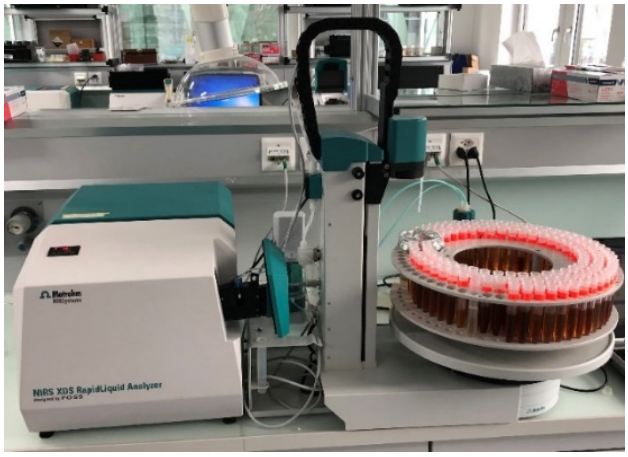


Figure 1. XDS RapidLiquid Analyzer with 5.0 mm flow cell and the 815 Sample Processor.

RESULT

The obtained Vis-NIR spectra (**Figure 2**) were used to create prediction models for quantification of the Acid Number in lubricants. The quality of the prediction models was evaluated using correlation diagrams, which display the relationship between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

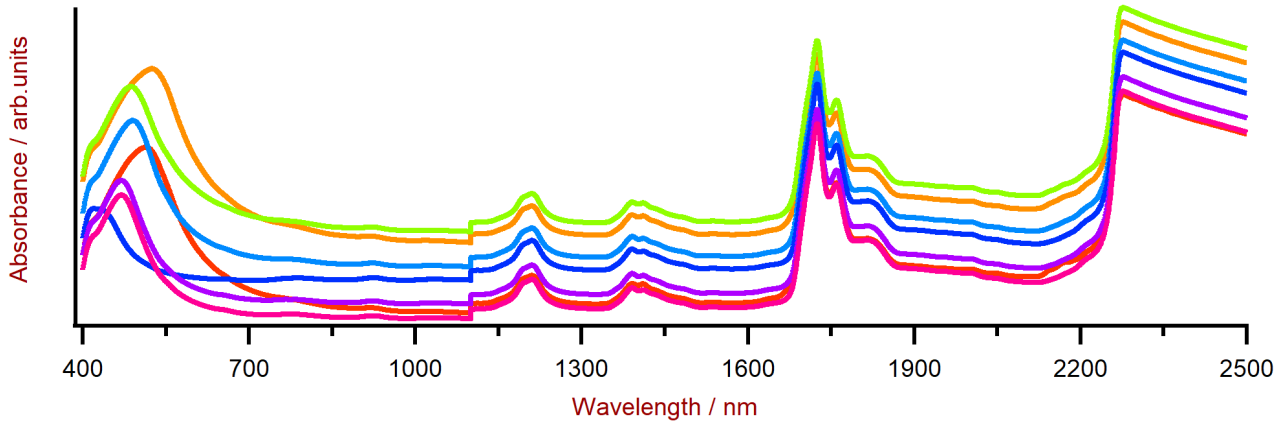
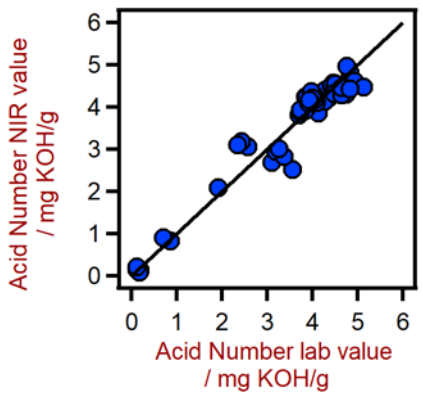


Figure 2. Selection of lubricant Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 5.0 mm flow cell. For display reasons a spectra offset was applied.

RESULT ACID NUMBER

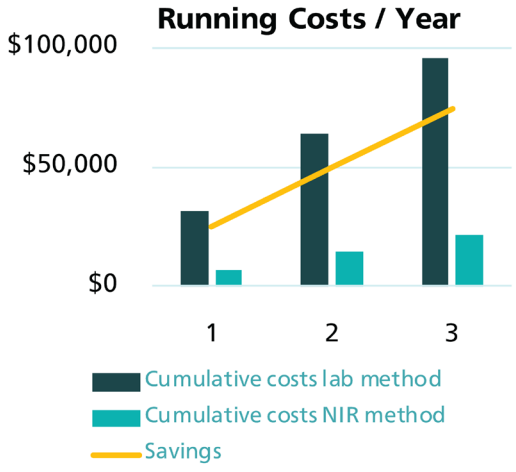


Figures of Merit	Value
R^2	0.950
Standard Error of Calibration	0.344 mg KOH/g
Standard Error of Cross-Validation	0.395 mg KOH/g

Figure 3. Correlation diagram and the respective figures of merit for the prediction of the Acid Number in lubricants using a XDS RapidLiquid Analyzer. The Acid Number content lab value was evaluated using titration.

CONCLUSION

This study demonstrates the feasibility of NIR spectroscopy for the analysis of the Acid Number in lubricants. In comparison to wet chemical methods **running costs are significantly reduced** when using NIR spectroscopy (**Figure 4**).



	Lab method	NIR method
Number of analysis (per day)	10	10
Cost of operator (per hour)	$25	$25
Costs of consumables and chemicals: Acid Number	$10	$1.50
Time spent per analysis	10 min	4 min
Total running costs / year	$31,875	$7,125

Figure 4. Comparison of running costs for the determination of the Acid Number with titration (ASTM D664) and NIR spectroscopy.

Analytes:	**Acid Number**
Matrix:	**Lubricants**
Industry:	**Petrochemical**
Standards:	**ASTM E1655**