



NIR APPLICATION NOTE NIR-041

# Multiparameter Quality Control of Lubricants with NIRS

Measure Acid Number, viscosity, water content, and color number

For lubricant analysis, determination of the Acid Number (ASTM D664), viscosity (ASTM D445), moisture content (ASTM D6304), and color number (ASTM D1500) require the use of multiple analytical technologies and, in part, large volumes of chemicals. Time to result can therefore be quite a long and costly process.

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

## EXPERIMENTAL EQUIPMENT

Lubricant samples were measured with a XDS RapidLiquid Analyzer in transmission mode over the full wavelength range (400–2500 nm) (Figure 1). Reproducible spectrum acquisition was achieved using the instrument's built-in temperature control (at 40 °C). For convenience, disposable vials with a pathlength of 8 mm were used, which made cleaning of the sample vessels unnecessary. The Metrohm software package Vision Air Complete was used for all data acquisition and prediction model development.

**Table 1.** Hardware and software equipment overview

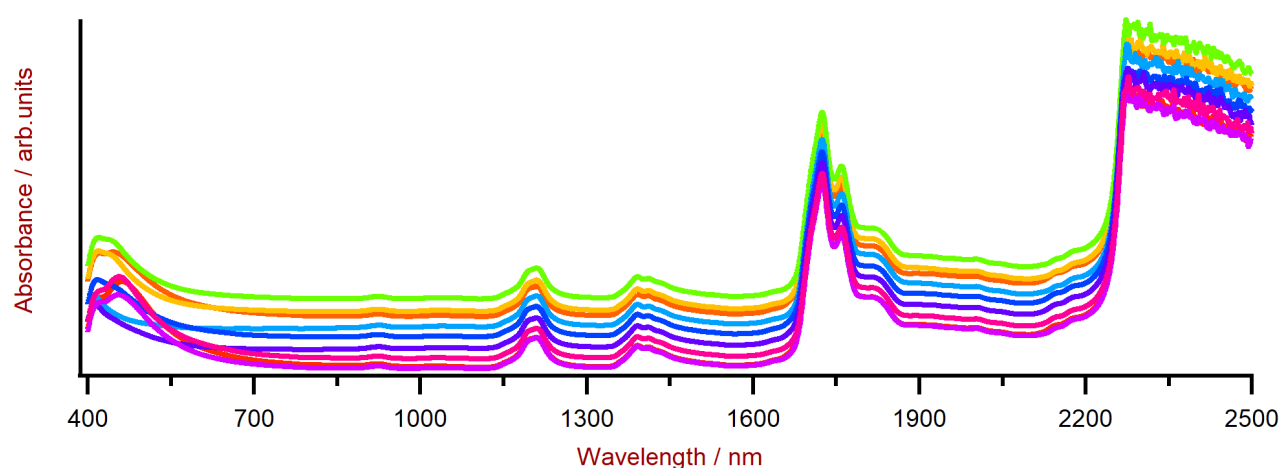
Equipment	Metrohm number
XDS RapidLiquid Analyzer	2.921.1410
Disposable vials, 8 mm diameter, transmission	6.7402.000
Vision Air Complete	6.6072.208



**Figure 1.** XDS RapidLiquid Analyzer and lubricant samples.

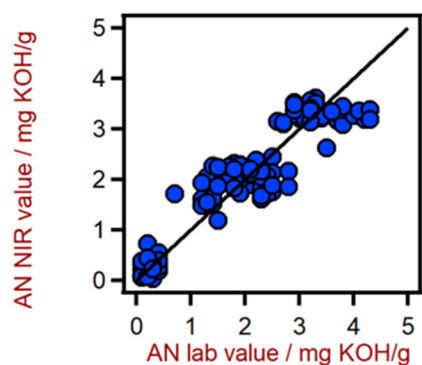
## RESULT

The obtained Vis-NIR spectra (Figure 2) were used to create prediction models for quantification of the Acid Number, viscosity, moisture content, and color number in lubricants. Correlation diagrams, which display the relationship between Vis-NIR prediction and primary method values, are used to determine the quality of the prediction models. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.



**Figure 2.** Selection of lubricant oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.

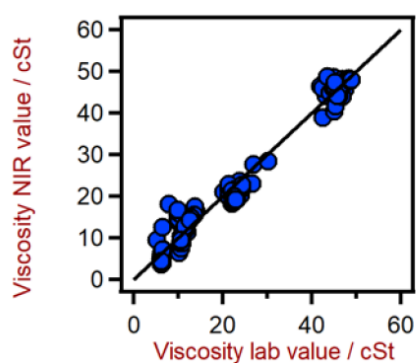
## RESULT ACID NUMBER



Figures of Merit	Value
$R^2$	0.898
Standard Error of Calibration	0.422 mg KOH/g
Standard Error of Cross-Validation	0.439 mg KOH/g

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

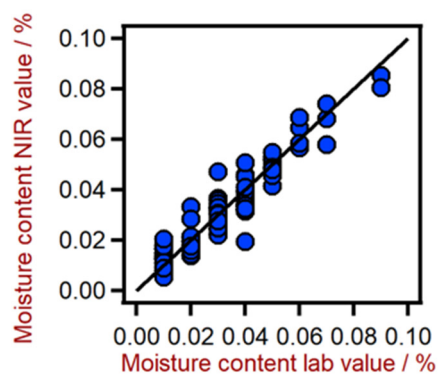
## RESULT VISCOSITY



Figures of Merit	Value
$R^2$	0.700
Standard Error of Calibration	0.841 cSt
Standard Error of Cross-Validation	0.916 cSt

**Figure 4.** Correlation diagram and the respective figures of merit for the prediction of the viscosity in lubricants using a XDS RapidLiquid Analyzer. The viscosity lab value was evaluated using viscometry.

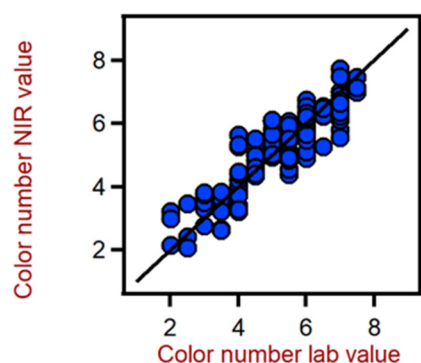
## RESULT MOISTURE CONTENT



Figures of Merit	Value
$R^2$	0.907
Standard Error of Calibration	0.0059 %
Standard Error of Cross-Validation	0.0062 %

**Figure 5.** Correlation diagram and the respective figures of merit for the prediction of the moisture content in lubricants using a XDS RapidLiquid Analyzer. The moisture content lab value was evaluated using Karl Fischer (KF) titration.

## RESULT COLOR NUMBER



Figures of Merit	Value
$R^2$	0.700
Standard Error of Calibration	0.841
Standard Error of Cross-Validation	0.916

**Figure 6.** Correlation diagram and the respective figures of merit for the prediction of the color number in lubricants using a XDS RapidLiquid Analyzer. The lab value was evaluated using photometry.

## CONCLUSION

The following application note demonstrates the feasibility of NIR spectroscopy for the analysis of key quality parameters in lubricants. In comparison to

wet chemical methods (**Table 2**), the time to result is a major advantage of NIR spectroscopy, since all parameters are determined **in a single measurement in less than a minute**.

**Table 2.** Time to result overview for the different quality control parameters.

Parameter	Method	Time to result
Acid Number	Titration	~5 min
Viscosity	Viscometry	~4 min
Moisture content	KF Titration	~5 min
Color number	UV-Vis Photometer	~1 min

Analytes:	Acid number; Physical parameters – viscosity, density, etc.; Moisture/water
Matrix:	Lubricants
Industry:	Petrochemical
Standards:	ASTM E1655