Quality Control of Lubricants

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**.

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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 path length 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.

Figure 1. XDS RapidLiquid Analyzer and lubricant samples.

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

RESULTS

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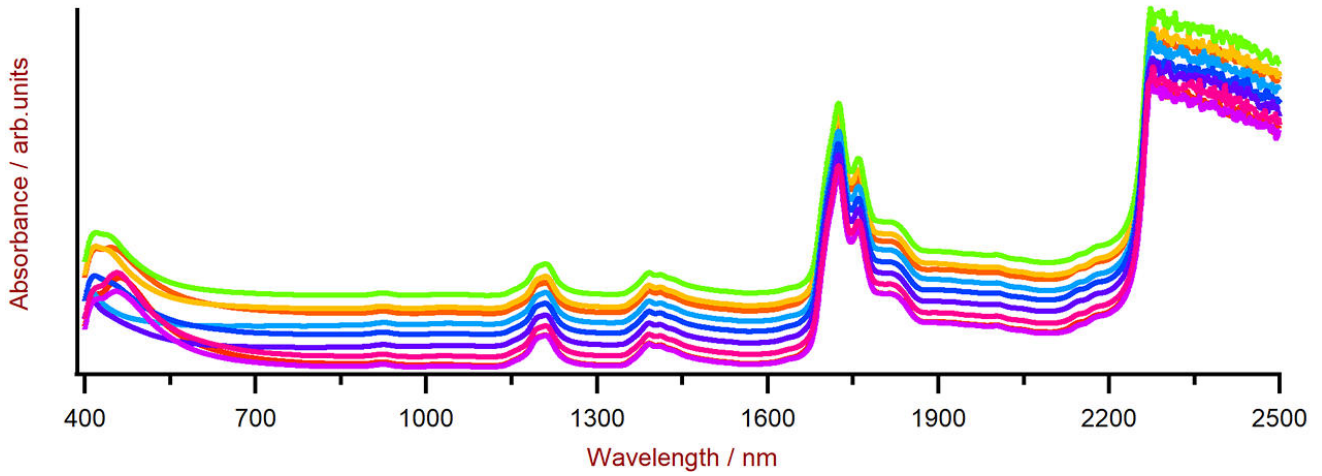
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Figure 2. Selection of lubricant oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.
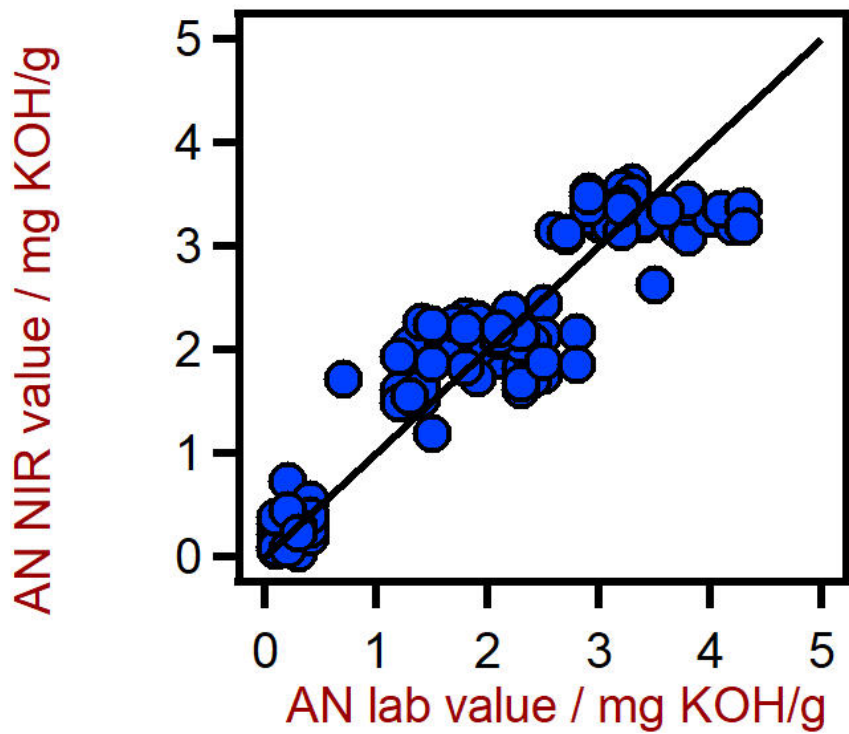

Figure 3. Correlation diagram for the prediction of the acid number (AN) in lubricants using a XDS RapidLiquid Analyzer. The AN lab value was evaluated using titration.

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Table 2. Figures of merit for the prediction of the acid number in lubricants using a XDS RapidLiquid Analyzer.

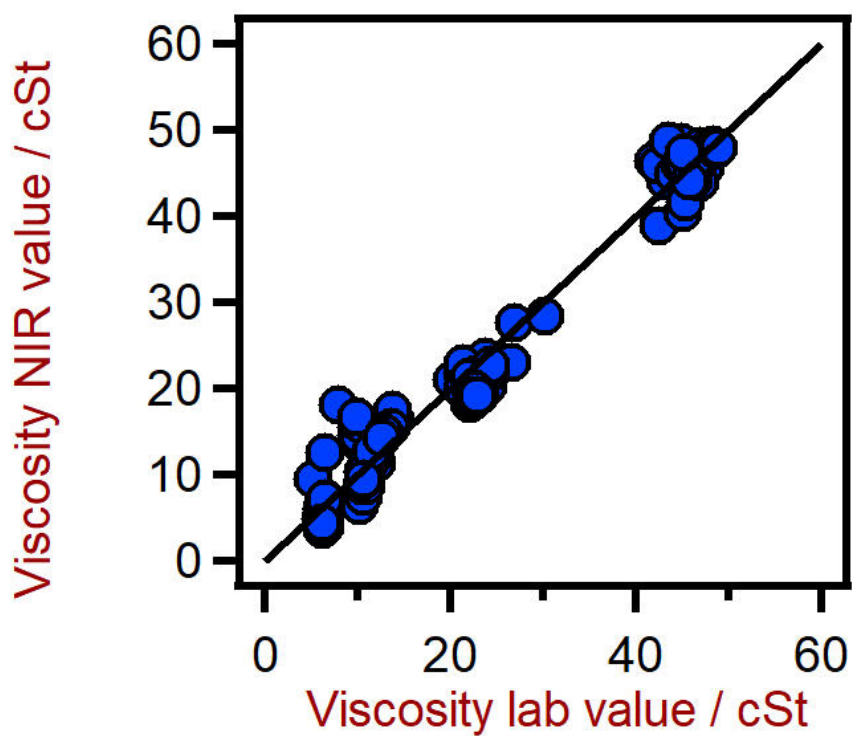| Figures of merit	Value
R_2	0.898
Standard error of calibration	0.422 mg KOH/g
Standard error of cross-validation	0.439 mg KOH/g

Figure 4. Correlation diagram for the prediction of the viscosity of lubricants using a XDS RapidLiquid Analyzer. The viscosity lab value was evaluated using viscometry.

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Table 3. Figures of merit for the prediction of the viscosity of lubricants using a XDS RapidLiquid Analyzer.

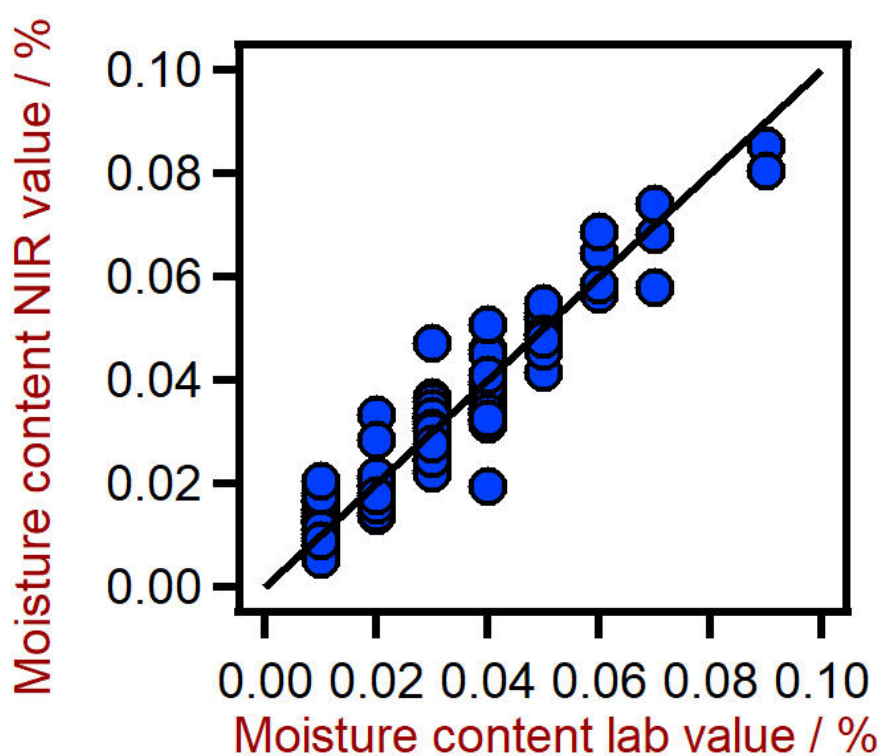| Figures of merit	Value
R_2	0.987
Standard error of calibration	1.77 cSt
Standard error of cross-validation	1.84 cSt

Figure 5. Correlation diagram 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.

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Table 4. Figures of merit for the prediction of the moisture content in lubricants using a XDS RapidLiquid Analyzer.

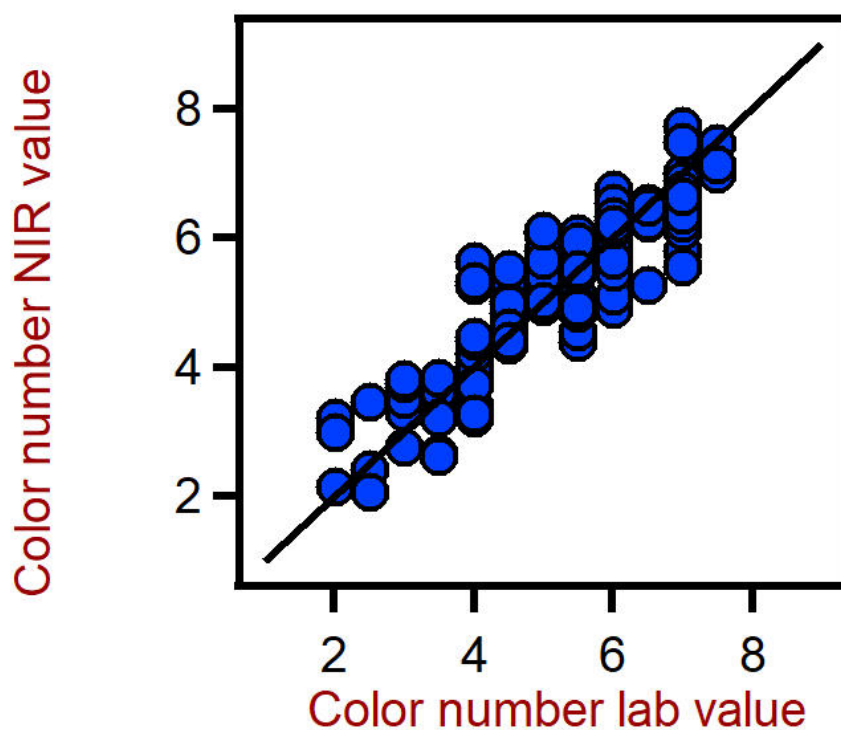| Figures of merit	Value
R_2	0.907
Standard error of calibration	0.0059%
Standard error of cross-validation	0.0062%

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

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Table 5. Figures of merit for the prediction of the color number in lubricants using a XDS RapidLiquid Analyzer.

Figures of merit	Value
R_2	0.700
Standard error of calibration	0.841
Standard error of cross-validation	0.916

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 6), 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 6. 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

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NIRS XDS RapidLiquid Analyzer
Rapid, precise analyses of liquids and suspensions of all types.

The NIRS XDS RapidLiquid Analyzer enables rapid, precise analyses of liquid formulations and substances. Precise measurement results at the push of a button make the NIRS XDS RapidLiquid Analyzer an equally reliable and simple solution for quality monitoring in laboratories and processes. The samples are transferred to quartz cuvettes designed for multiple use or disposable glass vials; a tempered sample compartment ensures reproducible analysis conditions and thus accurate measurement results.

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)

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