



Application Note AN-NIR-041

# Quality Control of Lubricants

Fast and chemical-free determination of the acid number, viscosity, moisture content, and color number of lubricants with NIRS

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 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.

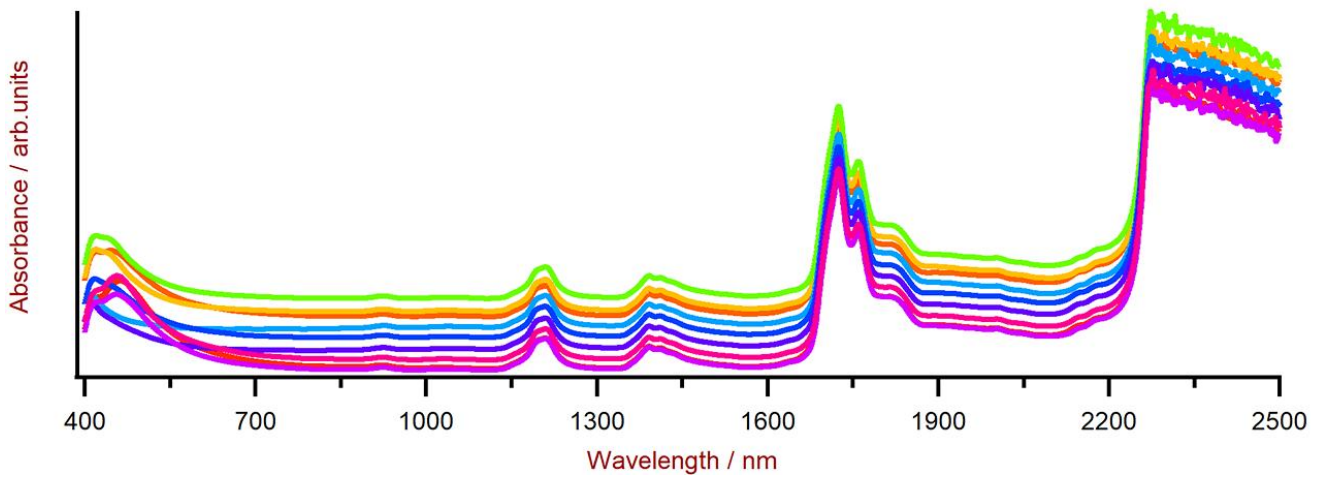
Table 1. Hardware and software equipment overview

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

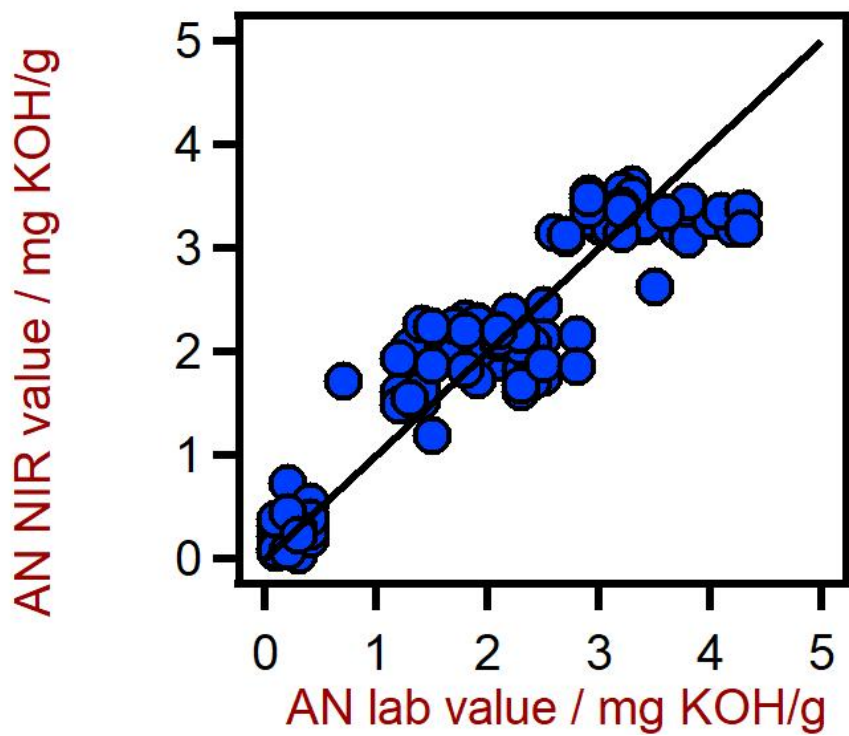
## RESULTS

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.



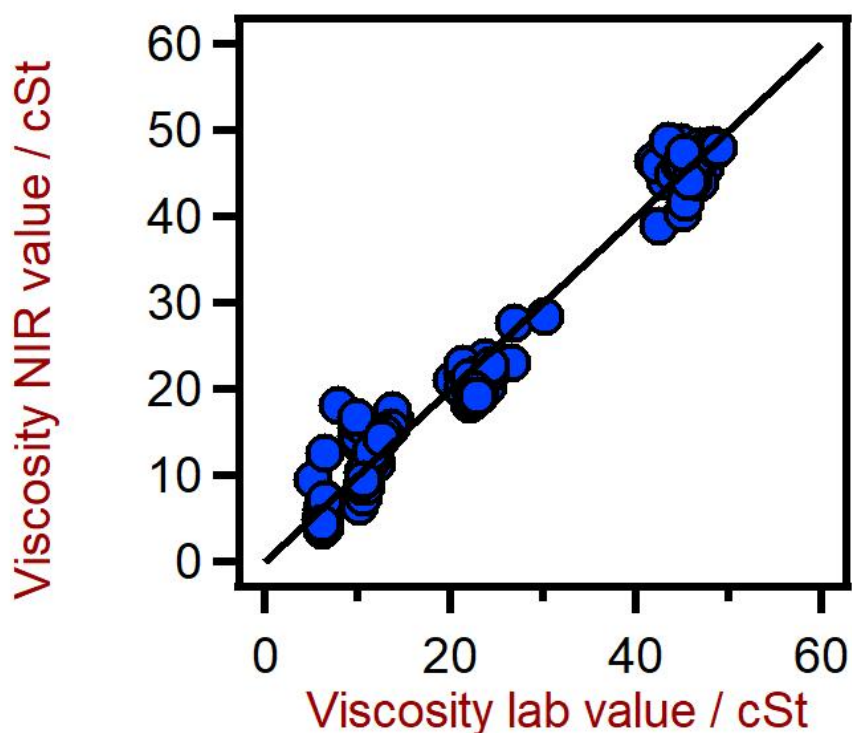
**Figure 2.** Selection of lubricant oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.



**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.

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

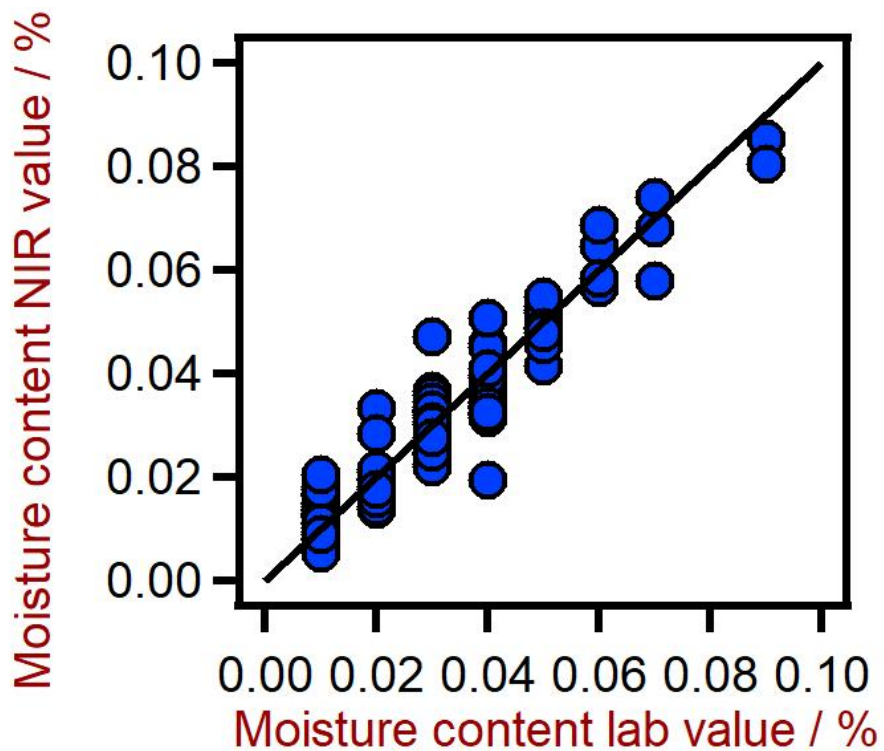
Figures of merit	Value
R <sup>2</sup>	0.898
Standard error of calibration	0.422 mg KOH/g
Standard error of cross-validation	0.439 mg KOH/g



**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.

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

Figures of merit	Value
R <sup>2</sup>	0.987
Standard error of calibration	1.77 cSt
Standard error of cross-validation	1.84 cSt

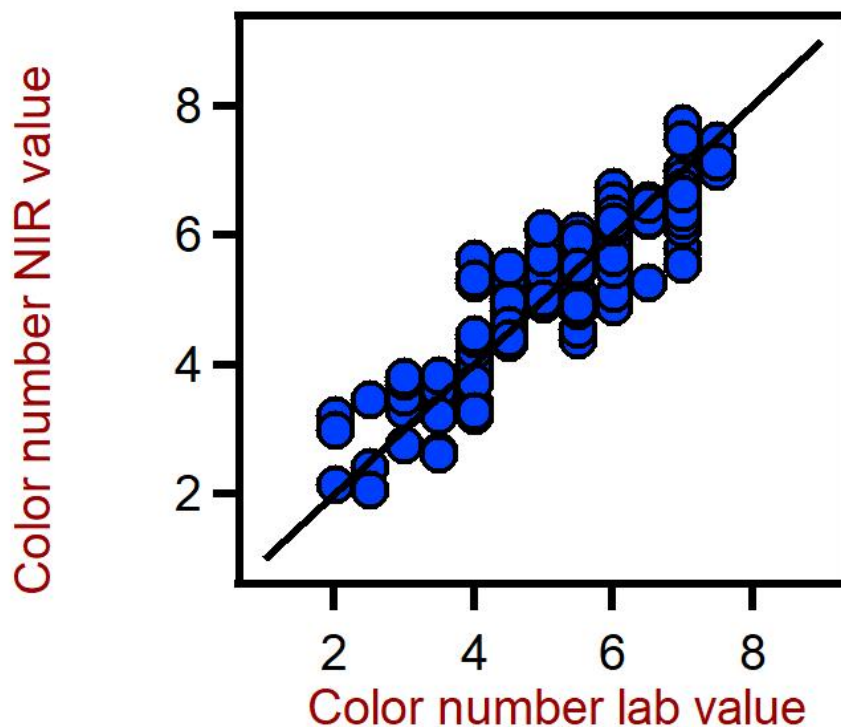


**Figure 5.** Correlation diagram for the prediction of the moisture content in lubricants using a XDS RapidLiquid Analyzer. The moisture content lab value was evaluated using Karl Fischer (KF) titration.

**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.

**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

Des analyses rapides et précises de liquides et suspensions de toutes sortes.

L'analyseur NIRS XDS RapidLiquid Analyzer permet des analyses rapides et précises de formules et substances liquides. Des résultats de mesure précis obtenus par simple pression d'une touche font du NIRS XDS RapidLiquid Analyzer une solution aussi fiable que simple pour le contrôle qualité en laboratoire et en production. Les échantillons sont présentés dans des cuvettes en quartz réutilisables ou des flacons en verre à usage unique ; une chambre à échantillons tempérée assure la reproductibilité des conditions d'analyse et, par conséquent, l'exactitude des résultats de mesure.



## Vision Air 2.0 Complete

### Vision Air - logiciel universel de spectroscopie.

Vision Air Complete est une solution logicielle moderne et simple d'utilisation pour une application dans un environnement réglementé.

Aperçu des avantages de Vision Air :

- Des applications logicielles individuelles avec interface utilisateur adaptée sont le garant d'un maniement intuitif et simple
- Établissement et suivi simples des procédures de travail
- Base de données SQL pour une gestion sûre et simple des données

La version Vision Air Complete (66072208) comprend toutes les applications d'assurance qualité par spectroscopie Vis-NIR :

- Application de gestion des instruments et des données
- Application de développement de méthodes
- Application d'analyse de routine

Autres solutions Vision Air Complete :

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)