



Application Note AN-NIR-041

润滑油质量控制

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.

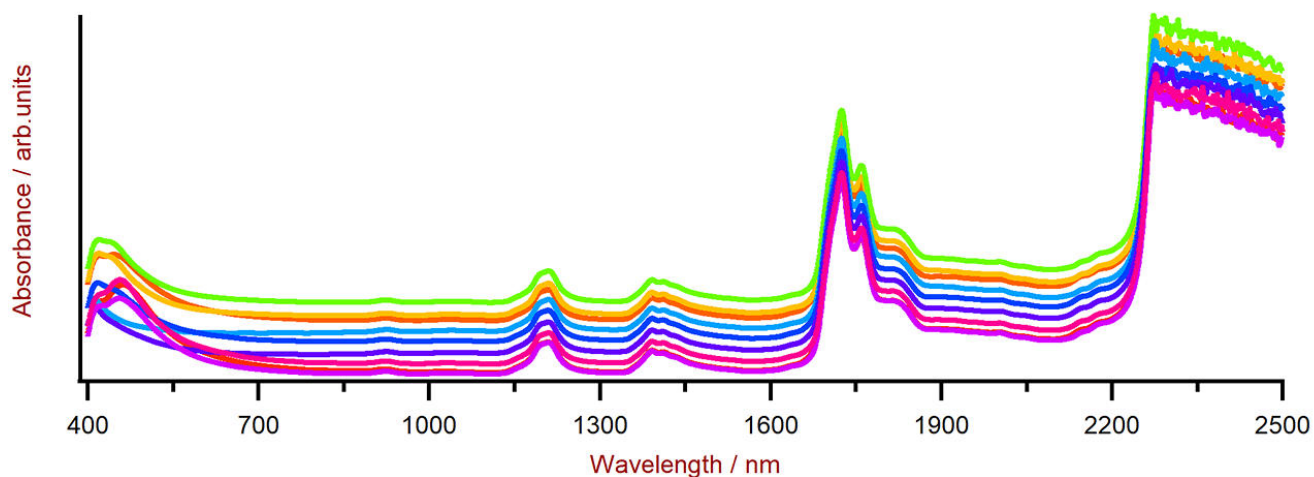


Figure 2. Selection of lubricant oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.

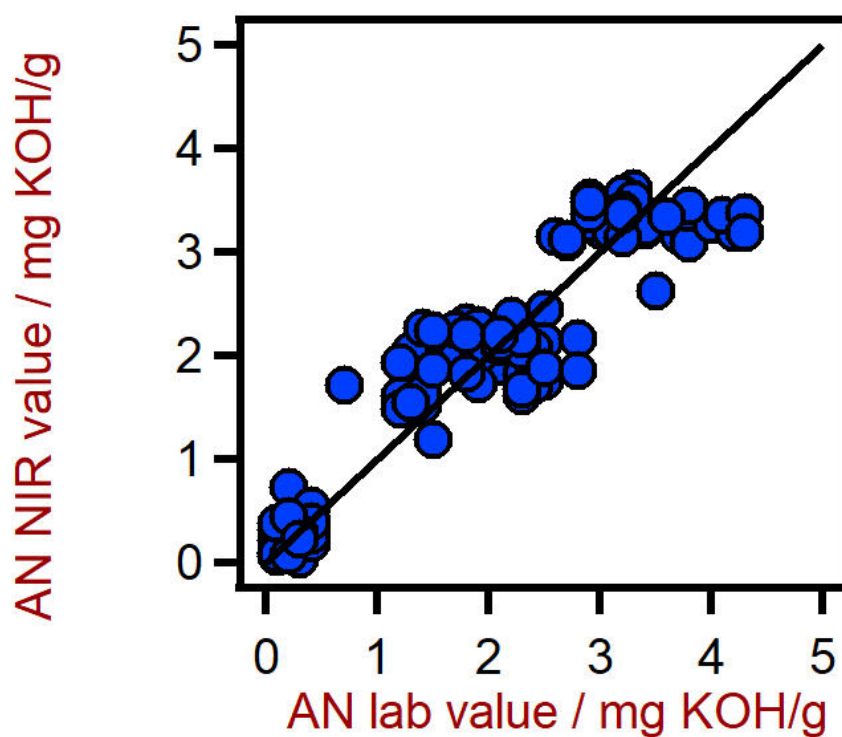


Figure 3. Correlation diagram for the prediction of the acid number (AN) in lubricants using a XDS RapidLiquid Analyzer. The AN lab value was evaluated using titration.

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

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

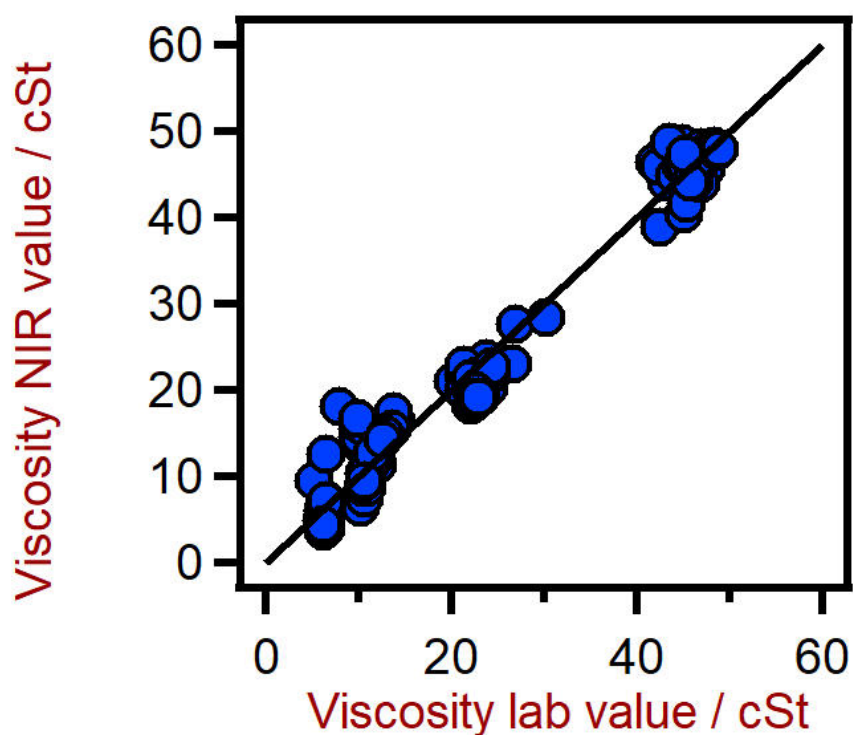


Figure 4. Correlation diagram for the prediction of the viscosity of lubricants using a XDS RapidLiquid Analyzer. The viscosity lab value was evaluated using viscometry.

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

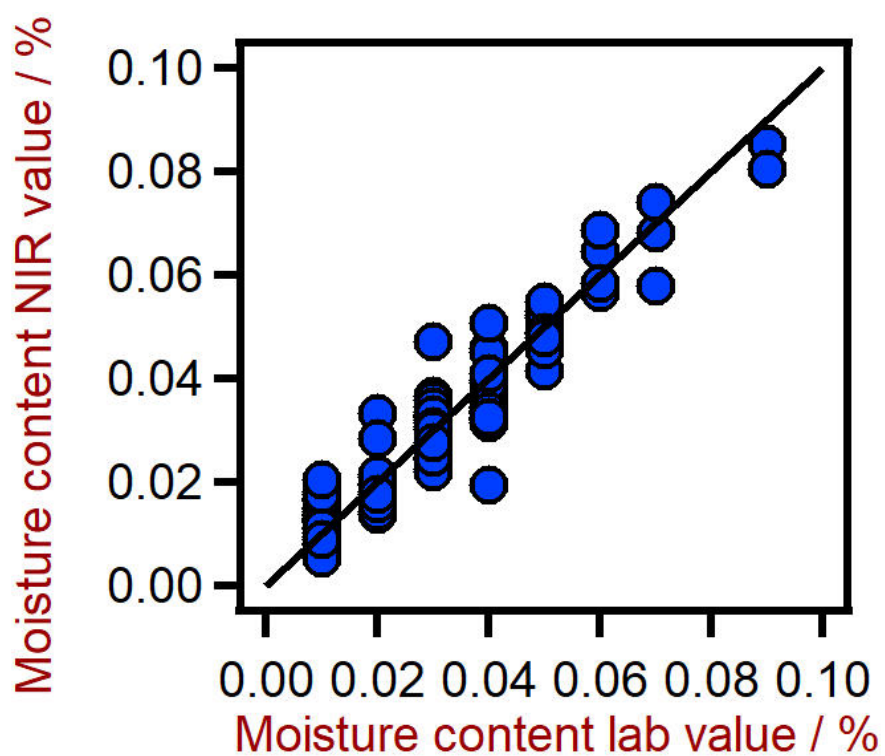


Figure 5. Correlation diagram for the prediction of the moisture content in lubricants using a XDS RapidLiquid Analyzer. The moisture content lab value was evaluated using Karl Fischer (KF) titration.

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%

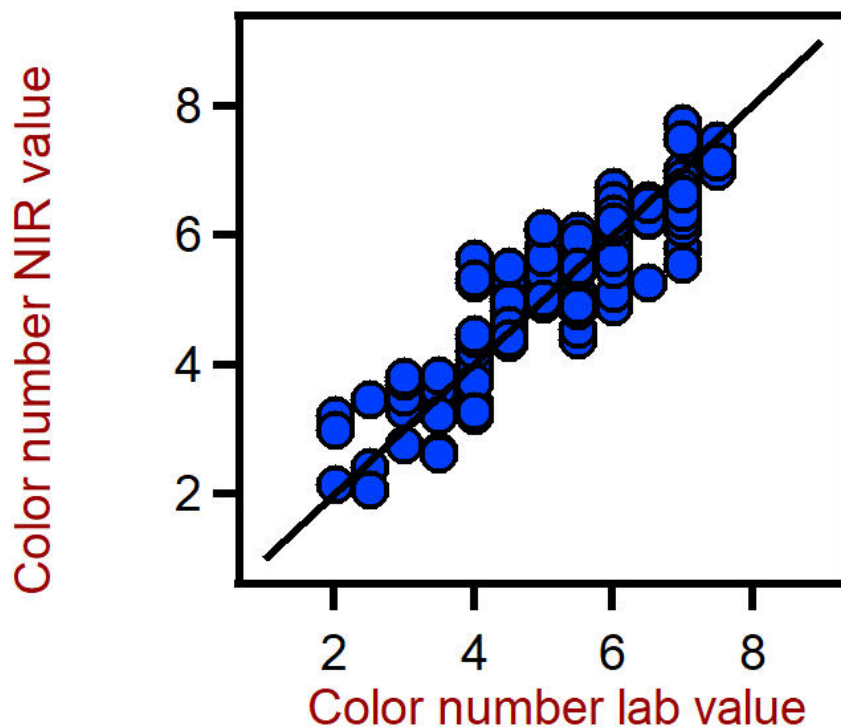


Figure 6. Correlation diagram for the prediction of the color number in lubricants using a XDS RapidLiquid Analyzer. The hydroxyl number lab value was evaluated using photometry.

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 快速精确地分析各种液体和悬浮液.

NIRS XDS RapidLiquid Analyzer 分析仪可快速精确地分析液体制剂和物质。按下按钮即可得到精确的测量结果,NIRS XDS RapidLiquid Analyzer 是适用于实验室和工艺过程中质量控制的可靠且方便的解决方案。样品将被预置在可重复使用的石英比色皿或一次性玻璃瓶中;可控制温度的样品室确保可重现的分析条件,由此得到精确的测量结果。



Vision Air 2.0 Complete

Vision Air – 通用的光谱分析软件。

Vision Air Complete 是用于监管规范环境的先进易用的软件解决方案。

Vision Air 优点一览:

- 便捷的软件应用和经过适配的用户界面保证了直观简单的操作方式
- 操作过程的创建与维护方式简单
- SQL 数据库,可安全且简单地管理数据

Vision Air Complete (66072208) 版本包含所有用于可见近红外光谱分析质量保证过程的应用:

- 仪器和数据管理应用
- 方法开发应用
- 常规分析应用

其它 Vision Air Complete 解决方案:

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