



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.

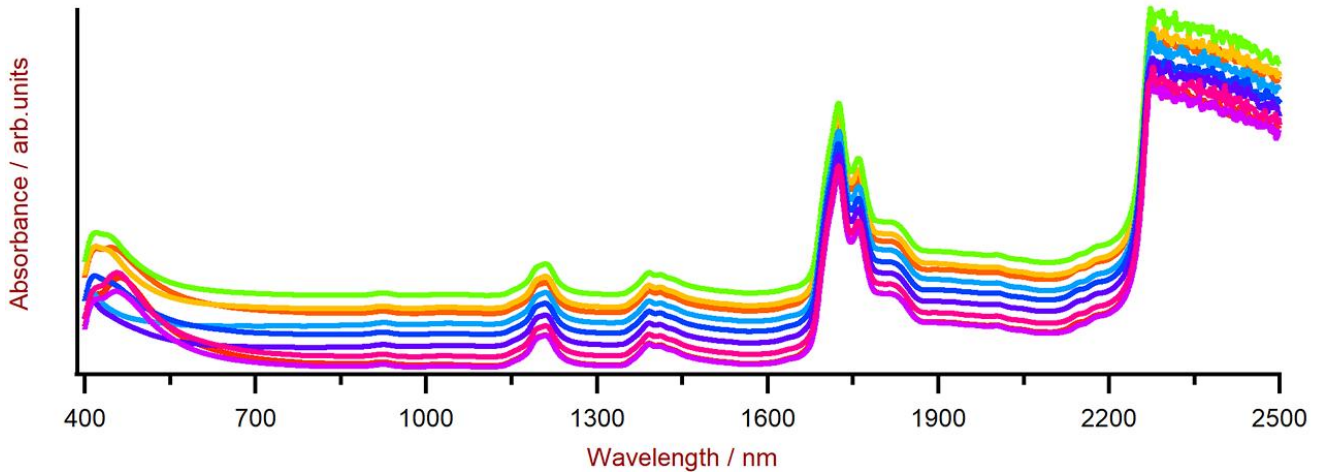


Figure 2. Selection of lubricant oil Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 8 mm disposable vials. For display reasons a spectra offset was applied.

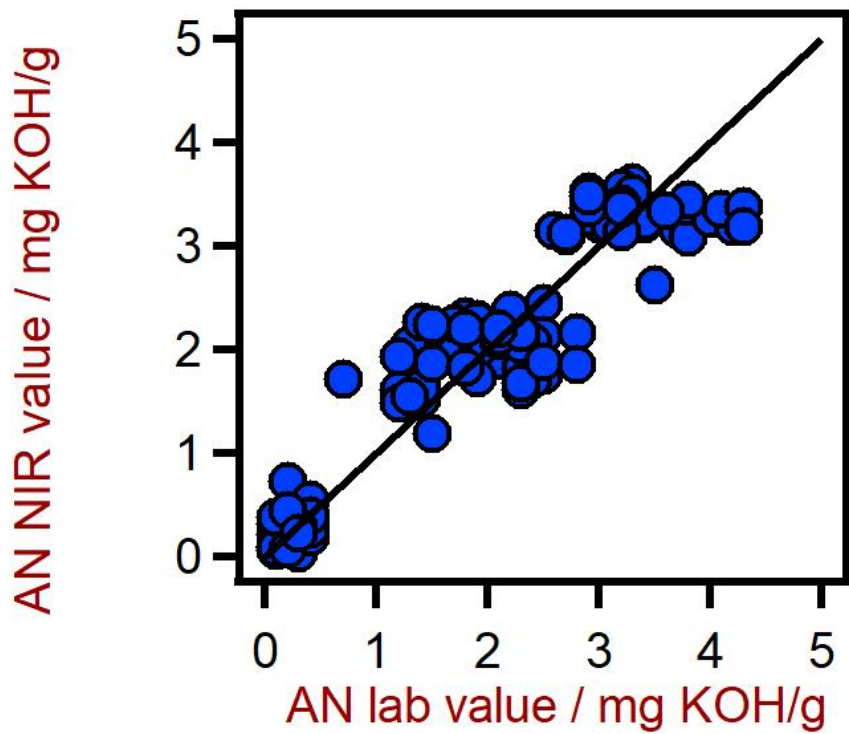


Figure 3. Correlation diagram for the prediction of the acid number (AN) in lubricants using a XDS RapidLiquid Analyzer. The AN lab value was evaluated using titration.

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

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

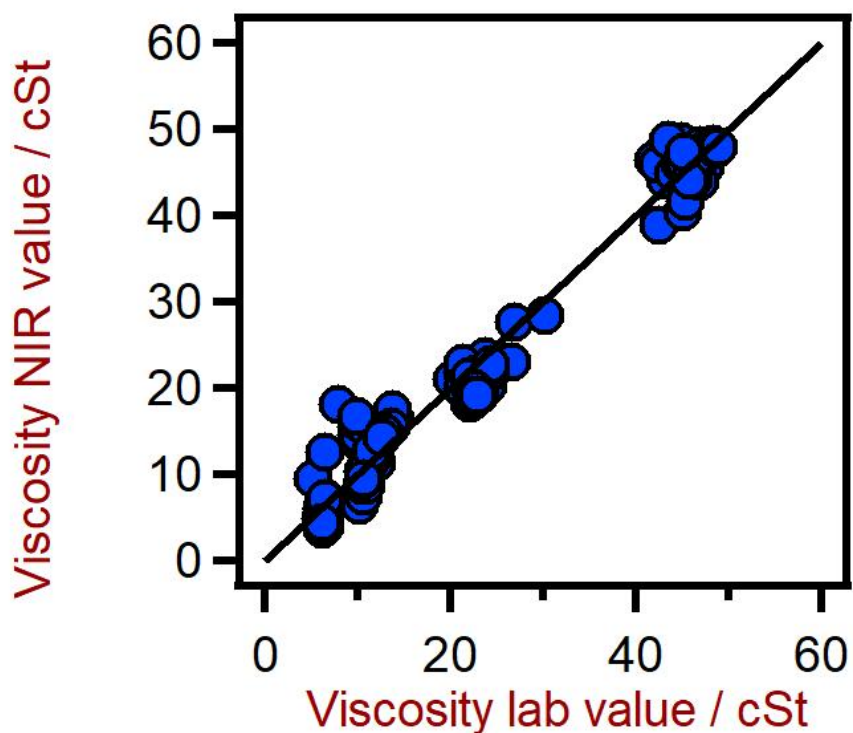


Figure 4. Correlation diagram for the prediction of the viscosity of lubricants using a XDS RapidLiquid Analyzer. The viscosity lab value was evaluated using viscometry.

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

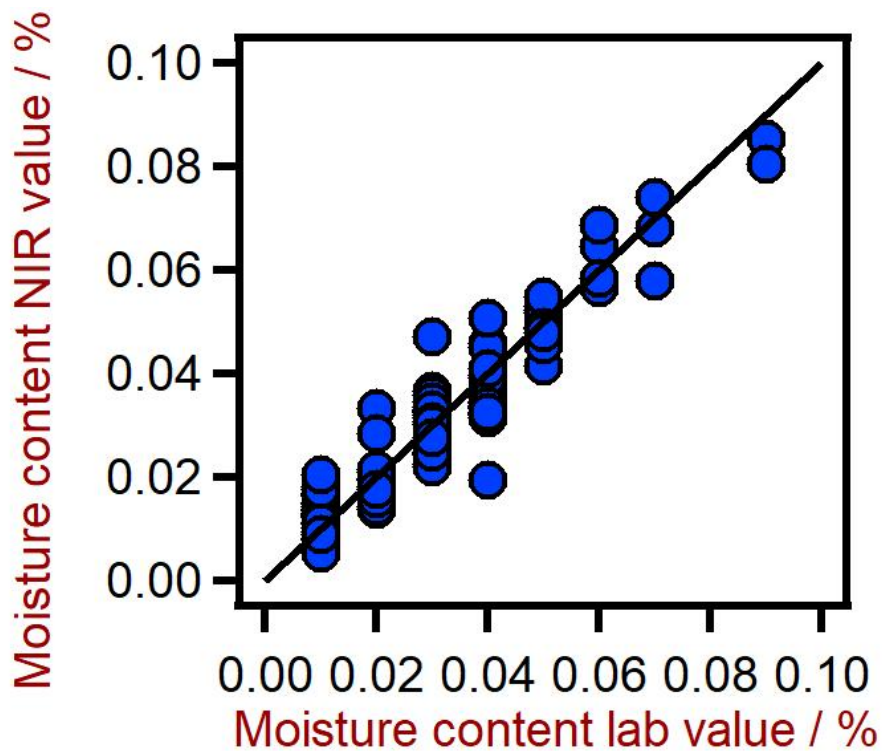


Figure 5. Correlation diagram for the prediction of the moisture content in lubricants using a XDS RapidLiquid Analyzer. The moisture content lab value was evaluated using Karl Fischer (KF) titration.

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%

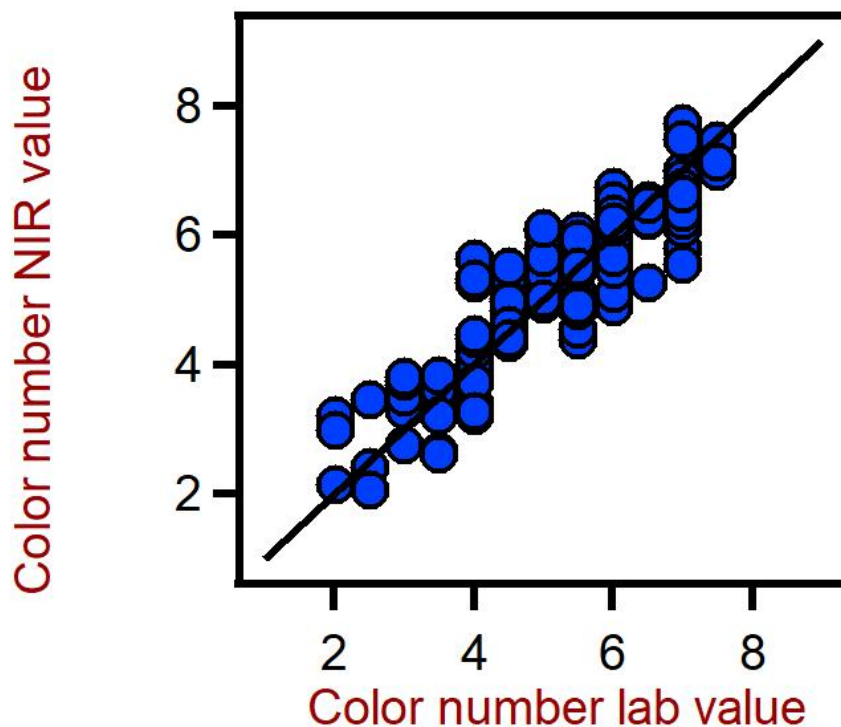


Figure 6. Correlation diagram for the prediction of the color number in lubricants using a XDS RapidLiquid Analyzer. The hydroxyl number lab value was evaluated using photometry.

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

CONTACT

メトロームジャパン株式会社
143-0006 東京都大田区平
和島6-1-1
null 東京流通センター アネ
ックス9階

metrohm.jp@metrohm.jp



NIRS XDS RapidLiquid Analyzer

あらゆる種類の液体および懸濁液の迅速で正確な分析。

NIRS XDS RapidLiquid Analyzerは、液体製剤および物質の迅速で正確な分析を可能にします。ボタンを押すだけで正確な測定結果が得られるため、NIRS XDS RapidLiquid Analyzerはラボおよびプロセスにおける品質管理のための信頼性が高くシンプルなソリューションです。サンプルは、再使用可能な石英製キュベットまたは使い捨てガラス製ハイアルに置かれます。温度調整されたサンプルコンパートメントは、再現性のある分析条件、およびそれによる正確な測定結果を保証します。



Vision Air 2.0 Complete

Vision Air - 汎用性に優れた分光法ソフトウェア。

Vision Air Complete は、規制環境下での使用のための、操作の容易な最新のソフトウェアソリューションです。

Vision Air の利点の概要:

- 調整済みのユーザーインターフェースを伴う個別のソフトウェアアプリケーションにより、直観的かつ容易な操作が保証されます。
- 作業手順の容易な作成およびメンテナンス
- 安全かつ容易なデータ管理のための SQL データベース

バージョン Vision Air Complete (66072208) には、可視近赤外分光法を用いた品質管理のための全てのアプリケーションが含まれています:

- 装置管理およびデータ管理のためのアプリケーション
- メソッド開発のためのアプリケーション
- ルーチン分析のためのアプリケーション

その他の Vision Air Complete ソリューション:

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