Quality Control of Lubricants

Unassisted, rapid determination of the Acid Number by automated NIR spectroscopy according to ASTM E1655

Summary

Acid Number (AN) analysis of lubricants (ASTM D664) can be a lengthy and costly process due to usage of large amounts of chemicals and required cleaning steps of the analytical equipment between each measurement.

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

Experimental Equipment

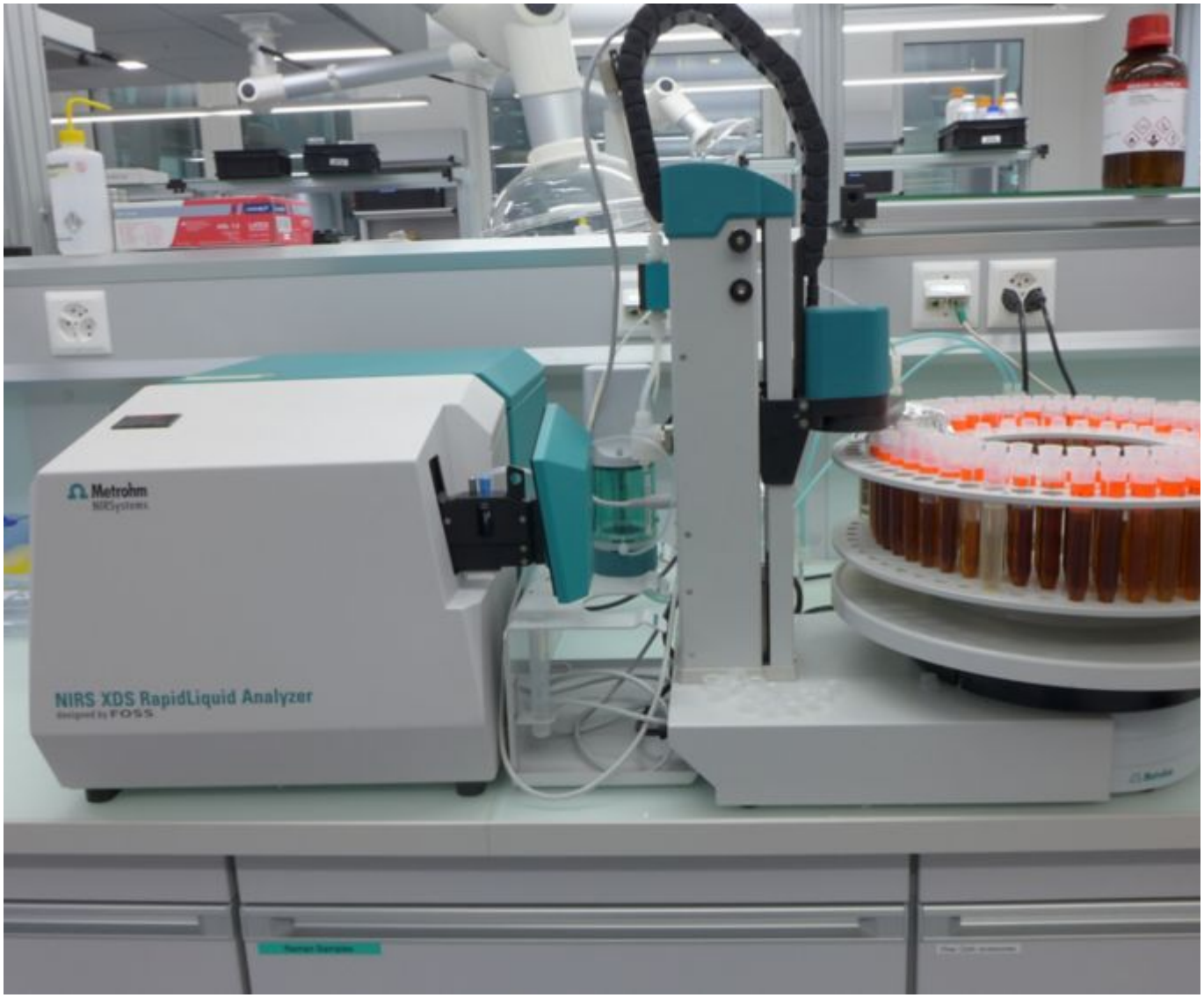

Figure 1. XDS RapidLiquid Analyzer with 5.0 mm flow cell and the 815 Sample Processor.

Lubricant samples were measured in transmission mode over the full wavelength range (400 nm to 2500 nm) using a XDS RapidLiquid Analyzer in combination with an 815 Robotic USB Sample Processor, which can carry a total of 141 samples. Reproducible spectrum acquisition was achieved using the built-in temperature control (at 30 °C) of the XDS RapidLiquid Analyzer. The Metrohm software packages ***tiamo*** and Vision Air Complete were 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
815 Robotic USB Sample Processor XL (Sample Rack 141 x 11 ml)	2.815.0010
800 Dosino	2.800.0020
5.0 mm flow cell	Hellma
Vision Air complete	6.6072.208
tiamo	6.6056.301

2.921.1410 - 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.

2.815.0010 - 815 Robotic USB Sample Processor XL (1T/1P)

Robotic USB Sample Processor XL with one workstation and one built-in membrane pump for the automatic processing of routine samples in series with large quantities and for complex sample preparation or parallel runs. In addition to the built-in pump, an additional one (membrane or peristaltic) and up to three dosing devices for Liquid Handling tasks can be connected.Because of the multitude of application variants, rack, stirrer, titration head, robotic arm, Swing Head and sample vessels must be tailored to the application and ordered separately.The control is "stand alone" using Touch Control. The following software products can be selected for the PC control: tiamo™ titration software, MagIC Net chromatography software, viva voltammetry software, or OMNIS.

2.800.0020 - 800 Dosino

Drive with write/read hardware for intelligent dosing units. With permanently attached cable (length 0.65 m).

6.6072.208 - 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);

6.6056.301 - tiamo™ 3.0 light USB: 1 license

tiamo™ 3.0 light computer program for controlling a titration system.Up to two Metrohm instruments (Titrino, Titrando, etc.) can be connected; balances and other generic (i.e. non-Metrohm) instruments can be included without limitation Graphical method editor with numerous templates Layout manager for individual monitor interface Professional database with reevaluation High-performance report generator Data export as PDF file, CSV, SLK No parallel titration No data export in XML format for LIMS Dialog languages: German, English, French, Italian, Spanish, Czech, Portuguese, Polish, Russian, Slovakian, Japanese, Chinese, Traditional Chinese

Result

The obtained Vis-NIR spectra (**Figure 2**) were used to create prediction models for quantification of the Acid Number in lubricants. The quality of the prediction models was evaluated using correlation diagrams, which display the relationship between Vis-NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

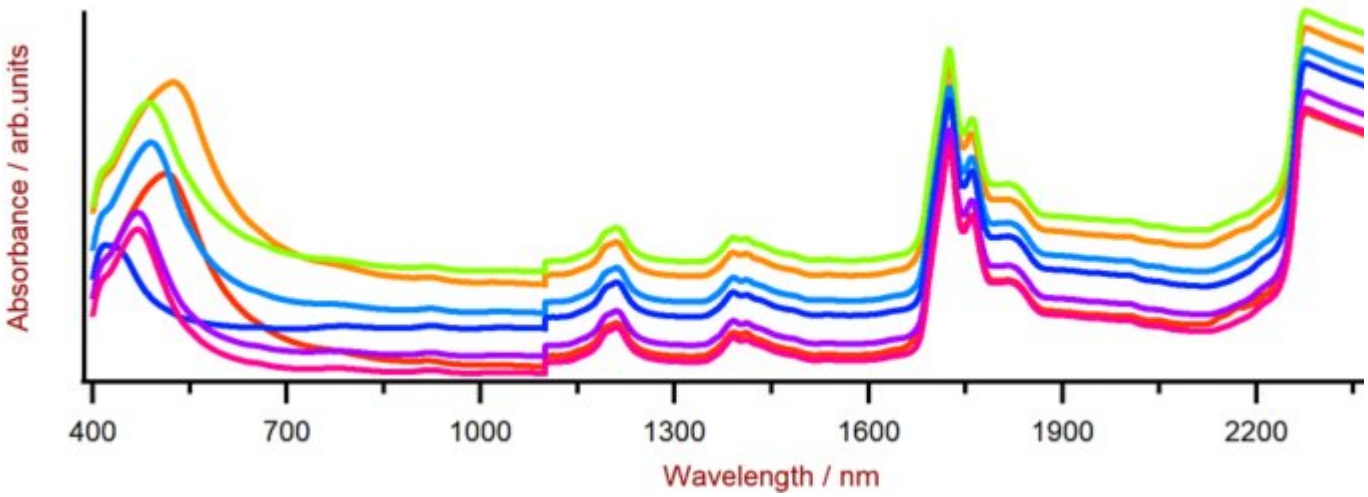

Figure 2. Selection of lubricant Vis-NIR spectra obtained using a XDS RapidLiquid Analyzer and 5.0 mm flow cell. For display reasons a spectra offset was applied.

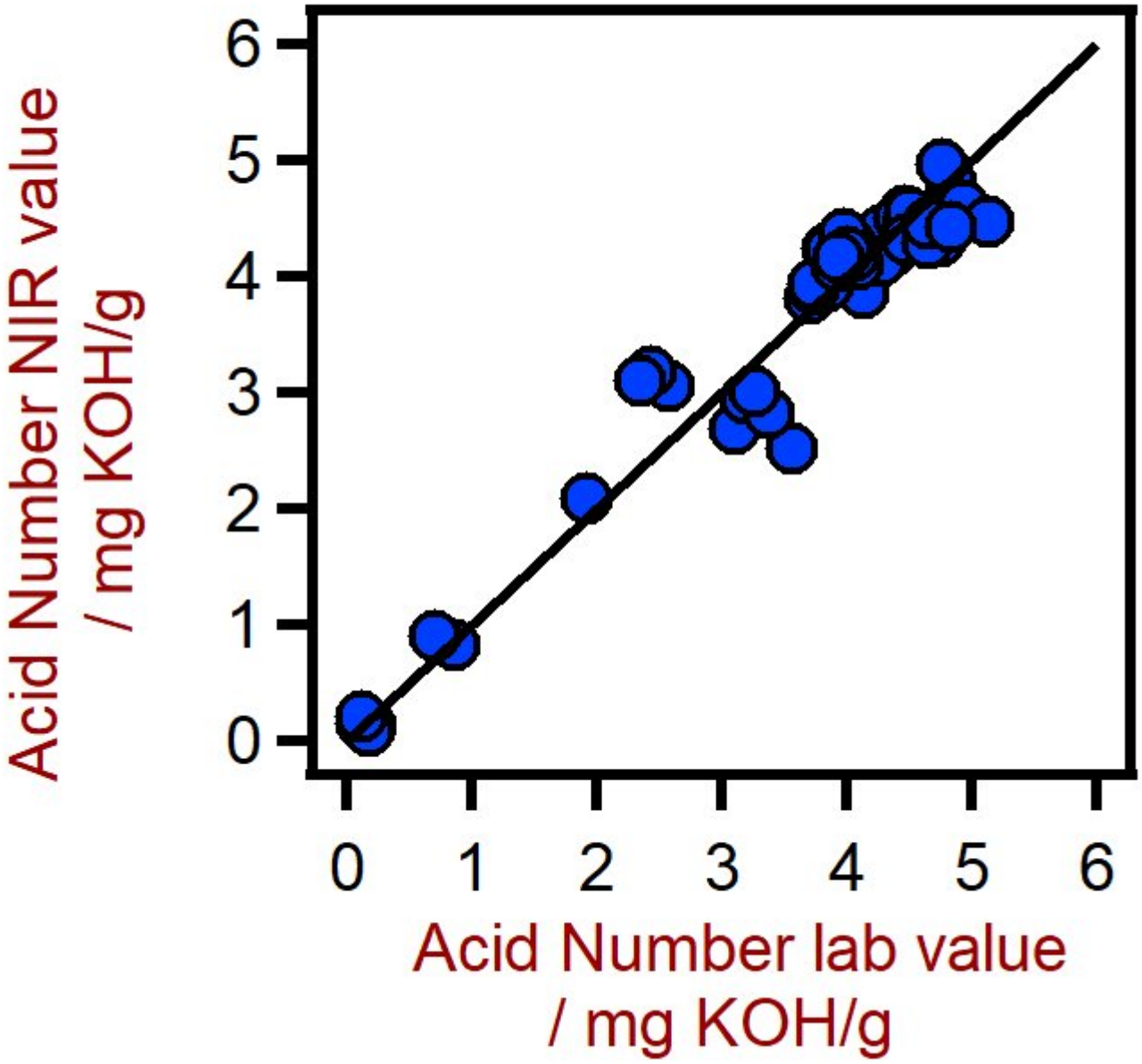

Figure 3. Correlation diagram for the prediction of the Acid Number in lubricants using a XDS RapidLiquid Analyzer. The Acid Number content lab value was evaluated using titration.

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	0.950
Standard error of calibration	0.344 mg KOH/g
Standard error of cross-validation	0.395 mg KOH/g

Conclusion

This study demonstrates the feasibility of NIR spectroscopy for the analysis of the Acid Number in lubricants. In comparison to wet chemical methods **running costs are significantly reduced** when using NIR spectroscopy (**Table 3** and **Figure 4**).

Table 3. Comparison of running costs for the determination of the acid number with titration (ASTM D664) and NIR spectroscopy.

	Lab method	NIR method
Number of analyses (per day)	10	10
Cost of operator (per hour)	$25	$25
Costs of consumables and chemicals OH number	$10	$1.50
Time spent per analysis	10 min	4 min
Total running costs (per year)	**$31,875**	**$7,125**

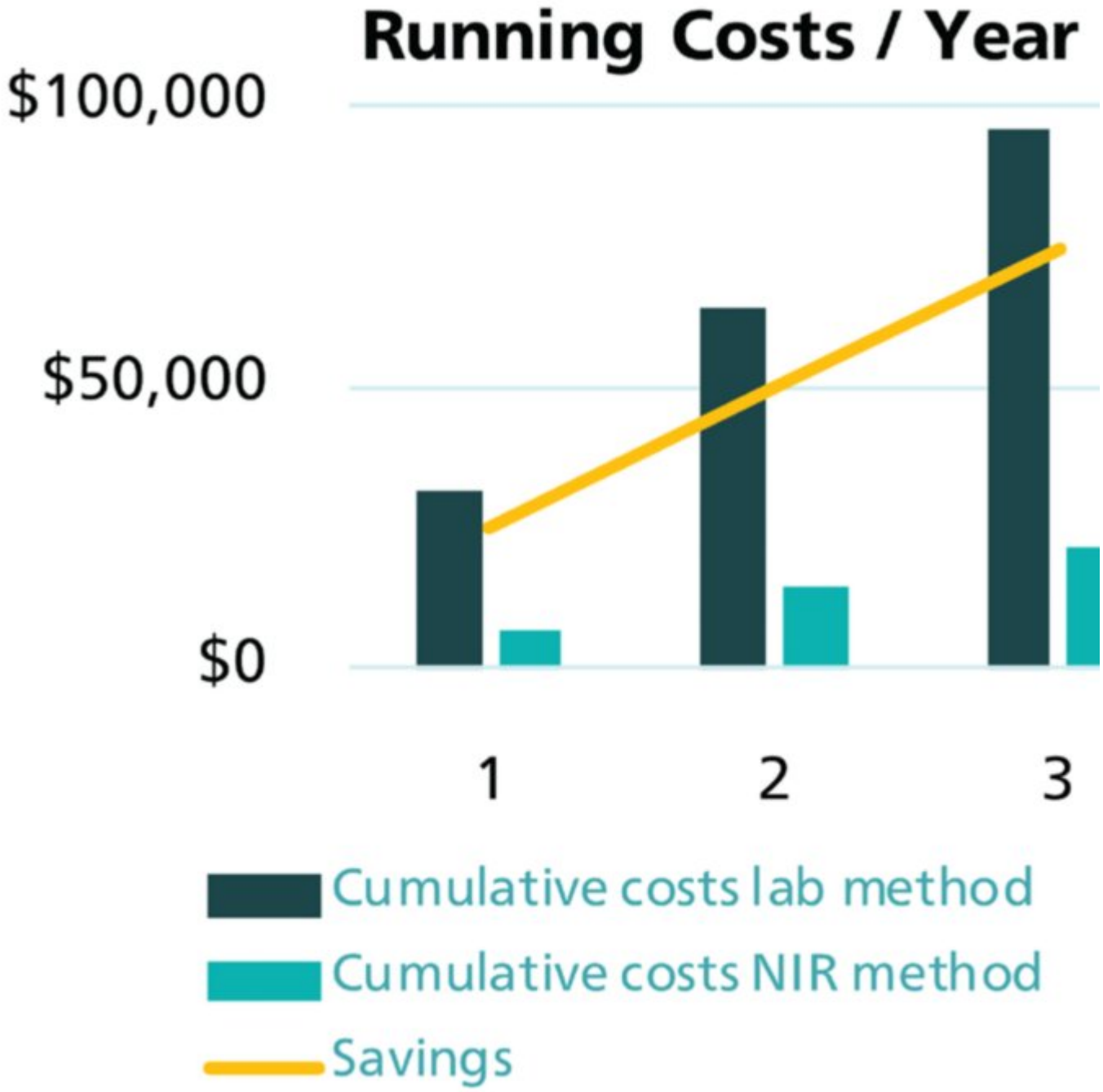

Figure 4. Comparison of the cumulative costs over three years for the determination of the acid number with titration and NIR spectroscopy.

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