

Application Note AN-NIR-097

Total base number in lubricants with near-infrared spectroscopy

Quality control of total base number without toxic chemicals

Alkaline additives in engine lubricants are used to prevent the build-up of acids and as a result, they inhibit corrosion. The total base number (TBN) indicates the amount of basic additives present in samples and thus can be used as a measure for the degradation of the lubricant. Depending on the application, the TBN value varies from 7 mg KOH/g in lubricants for combustion engines up to 80 mg KOH/g for marine grade lubricants.

The standard test method for TBN in lubricants is potentiometric titration according to ASTM D2896. This method requires the use of toxic reagents (e.g., tetraethylammonium bromide) and the cleaning procedure is labor-intensive. In contrast to the primary method, near-infrared spectroscopy (NIRS) is a fast analytical technique which does not produce any chemical waste and completes the TBN analysis in less than one minute.

EXPERIMENTAL EQUIPMENT

23 marine cylinder lubricants and 37 engine lubricants were analyzed on a Metrohm DS2500 Liquid Analyzer equipped with 2.5 mm flow cell. All measurements were performed in transmission mode from 400 nm to 2500 nm. In this feasibility study, a flow cell was used to automate the sample handling and measurement. Data acquisition and prediction model development was performed with the software package Vision Air complete.



Figure 1. DS2500 Liquid Analyzer.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Liquid Analyzer	2.929.0010
DS2500 Holder for flow cells	6.7493.000
Vision Air 2.0 Complete	6.6072.208

RESULTS

The obtained Vis-NIR spectra (Figure 2) were used to create a prediction model for the TBN determination. To verify the quality of the prediction model, correlation diagrams were created which display the correlation 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 3).

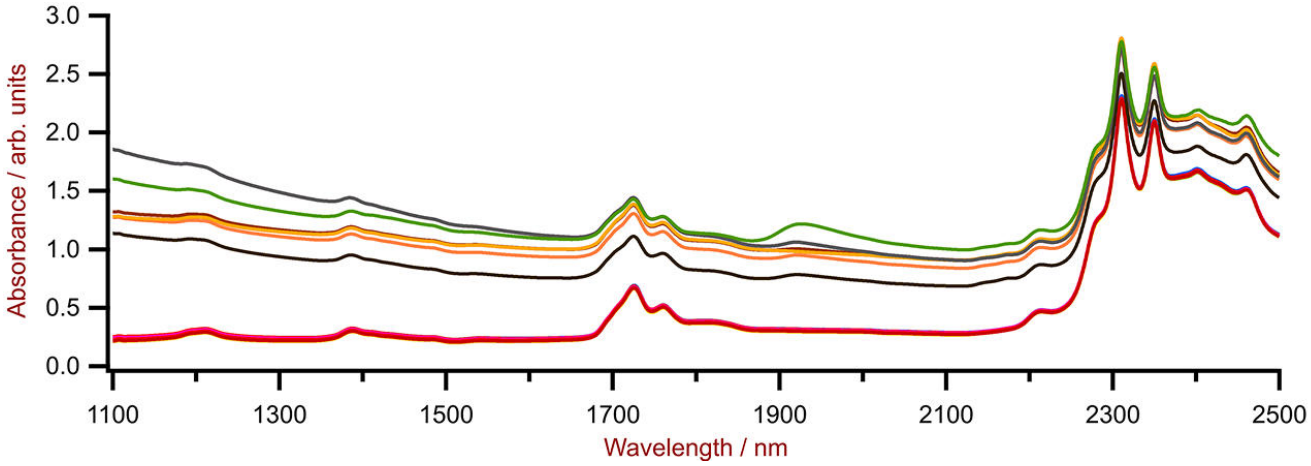

Figure 2. Selection of Vis-NIR spectra of marine cylinder lubricants and engine lubricants obtained using a DS2500 Liquid Analyzer with a 2.5 mm flow cell.

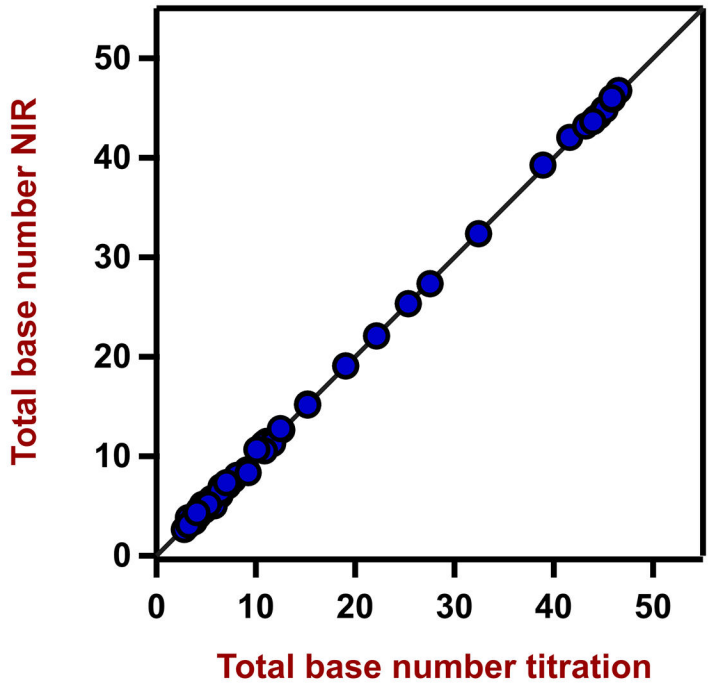

Figure 3. Correlation diagram for the prediction of TBN in lubricants using a DS2500 Liquid Analyzer. The lab values were determined using titration.

Table 2. Figures of merit for the prediction of TBN in lubricants using a DS2500 Liquid Analyzer.

Figures of merit	Value
R^2	0.998
Standard error of calibration	1.1
Standard error of cross-validation	1.2

CONCLUSION

This application note shows the feasibility of NIR spectroscopy for the analysis of total base number in marine cylinderand engine lubricants. In comparison to the wet chemical method (Table 3), no sample preparation or chemicals are needed with NIR spectroscopy.

Table 3. Time to result with conventional titration method ASTM D2896

Parameter	Method	Time to result
Total base number	Titration	5–10 minutes

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DS2500 Liquid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.

The DS2500 Liquid Analyzer is the tried and tested, flexible solution for routine analysis of liquids along the entire production chain. Its robust design makes the DS2500 Liquid Analyzer resistant to dust, moisture and vibrations, which means that it is eminently suited for use in harsh production environments.

The DS2500 Liquid Analyzer covers the full spectral range from 400 to 2500 nm, heats samples up to 80°C and is compatible with various disposable vials and quartz cuvettes. The DS2500 Liquid Analyzer is thus adaptable to your individual sample requirements and helps you obtain accurate and reproducible results in less than one minute. The integrated sample holder detection and the self-explanatory Vision Air Software also ensure simple and safe operation by the user.

In the case of larger-sized sample quantities, productivity can be considerably increased by using a flow-through cell in combination with a Metrohm sample robot.



DS2500 Holder Flow Cell

Intelligent holder for 12.5 mm flow cells



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)