



Application Note AN-NIR-097

TBN determination in lubricants

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**).

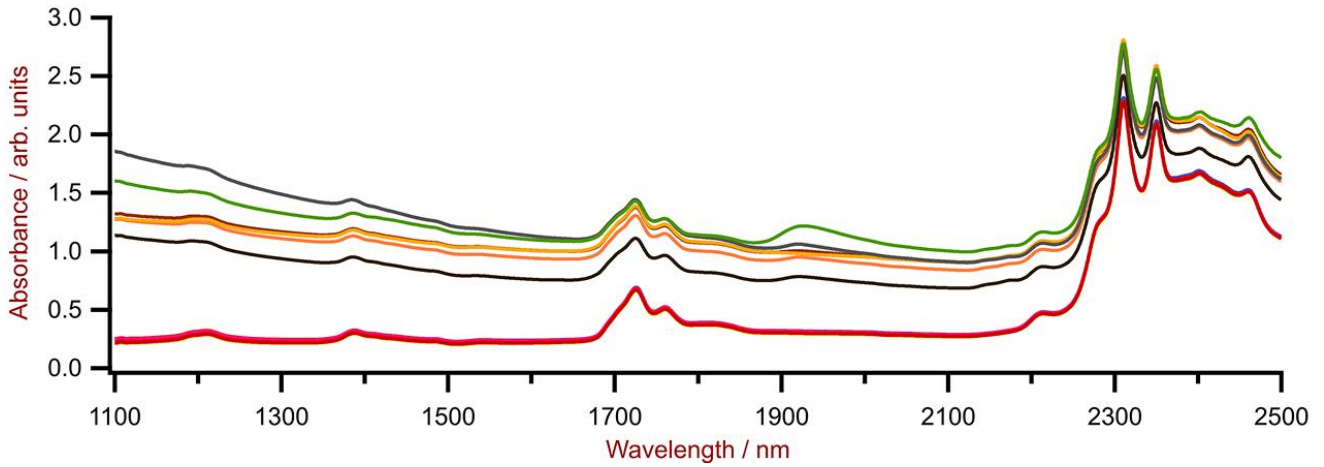


Figure 2. Selection of Vis-NIR spectra of marine cylinder lubricants and engine lubricants obtained using a DS2500 Liquid Analyzer with a 2.5 mm flow cell.

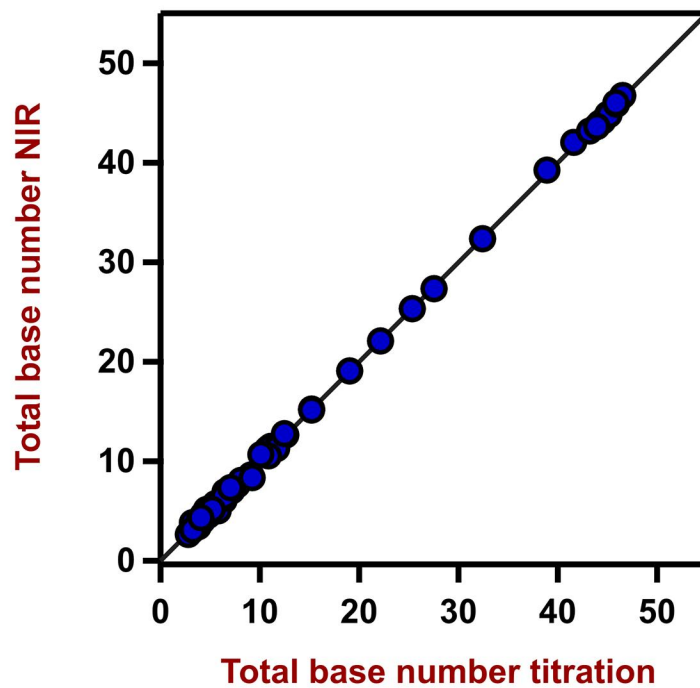


Figure 3. Correlation diagram for the prediction of TBN in lubricants using a DS2500 Liquid Analyzer. The lab values were determined using titration.

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 cylinder and 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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