

Application Note AN-NIR-096

Water in diesel with NIRS

Moisture in diesel fuel within seconds using NIR spectroscopy

Fuels can incorporate traces of water during the production process, in transport, and while in storage. Excessive water in fuels leads to several problems. For example, elevated water content in diesel fuel promotes biological growth in storage fuel tanks, which could lead to metal corrosion and formation of sludge and biofilms. This in turn clogs filters in fuel systems and results in damage to diesel engines.

The standard specification for diesel fuel quality includes multiple parameters, but dissolved water is the biggest risk factor. According to the European Committee for Standardization, the maximum acceptable amount of water in diesel fuel for commercialization is 200 mg/L (ppm) (EN 590). Usually, this is determined by Karl Fischer (KF) titration, yet this method requires chemicals and takes about five minutes to perform. This Application Note describes how near-infrared spectroscopy (NIRS) is a faster and more cost-efficient alternative to KF titration for the prediction of water content in diesel fuel.

EXPERIMENTAL EQUIPMENT

Samples of diesel with varying water content (from 66 to 362 mg/L) were measured with an OMNIS NIR Analyzer Liquid in transmission mode (1000–2250 nm). Reproducible spectrum acquisition was achieved using the built-in temperature control. For convenience, disposable vials with a pathlength of 8 mm were used, which made cleaning of the sample vessels unnecessary. The OMNIS software was used for all data acquisition and prediction model development.



Figure 1. OMNIS NIR Analyzer and a sample filled in a disposable vial.

Table 1. Hardware and software equipment overview.

Equipment	Article number
OMNIS NIR Analyzer Liquid	2.1070.0010
Holder OMNIS NIR, vial, 8 mm	6.07401.070
Disposable vial, 8 mm, transmission	6.7402.240
OMNIS Stand-Alone license	6.06003.010
Quant Development software license	6.06008.002

RESULT

The NIR spectra (Figure 2), along with the corresponding reference values, were used to create a prediction model for quantification of the moisture content in diesel samples. The quality of the prediction model was evaluated using the correlation diagram shown in Figure 3 which displays a very high correlation between the NIR prediction and the measured values of water content using KF titration (reference values). The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

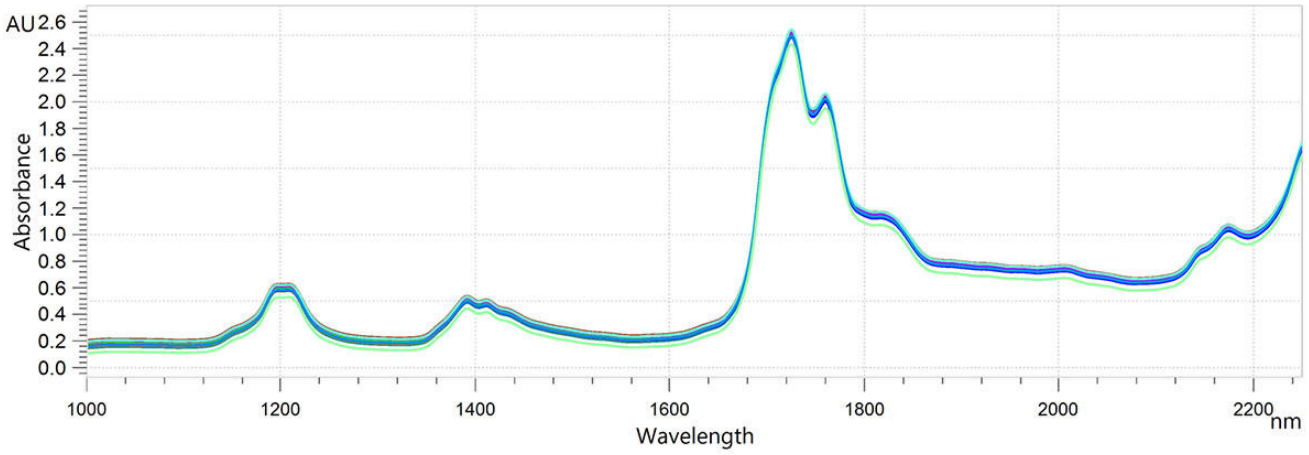

Figure 2. Stacked collection of NIR spectra from diesel samples analyzed with the OMNIS NIR Analyzer Liquid.

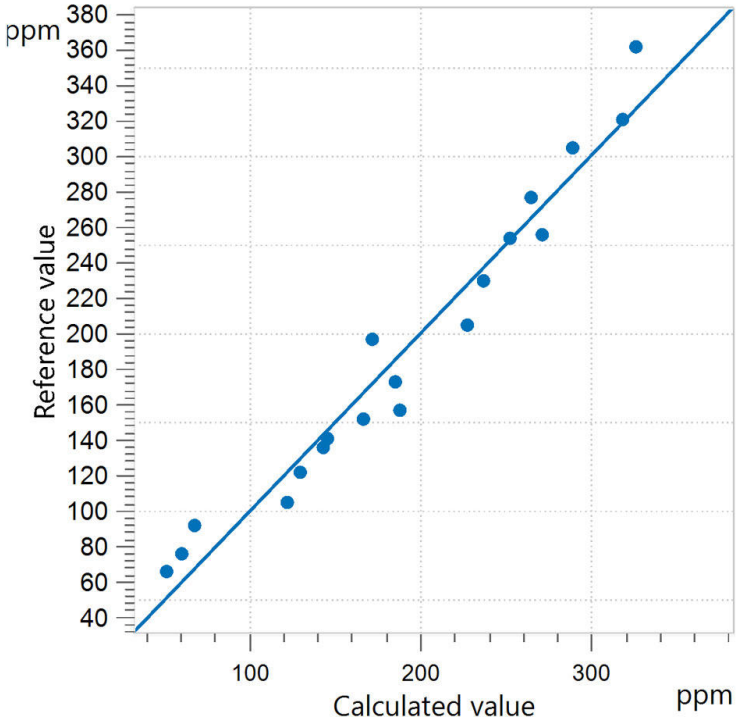

Figure 3. Correlation diagram and the respective figures of merit for the prediction of water content in diesel using an OMNIS NIR Analyzer Liquid. The lab values were evaluated using KF titration.

R^2	SEC (mg/L)	SECV (mg/L)
0.957	12	17

CONCLUSION

This Application Note demonstrates the feasibility to determine a key parameter for the quality control of diesel fuel – water content – with NIR spectroscopy. The main advantages of NIR spectroscopy over wet chemical methods like KF titration are that running costs are significantly lower and time-to-result is significantly reduced to a few seconds. Additionally, no chemicals are required, and the technique is non-destructive to samples.

Table 2. Time to result overview for KF titration.

Parameter	Method	Time to result
Water	Karl Fischer titration	5 minutes

Internal reference: AW NIR CH-0064-112021

CONTACT

Metrohm SA
1 Bridle Close
2052 Woodmead
sales@metrohm.co.za

CONFIGURATION



OMNIS NIR Analyzer Liquid

Near-infrared spectrometer for liquid samples.

Developed and produced in accordance with Swiss quality standards, the OMNIS NIR Analyzer is the near-infrared spectroscopy (NIRS) solution for routine analysis along the entire production chain. Its application of the latest technologies and its integration in the modern OMNIS Software are reflected in its speed, operability, and flexible utilization of this NIR spectrometer.

Overview of the advantages of the OMNIS NIR Analyzer Liquid:

- Measurements of liquid samples in less than 10 seconds
- Temperature control on the sample from 25–80 °C
- Automatic detection of the insertion and removal of the sample vessel
- Simple integration in an automation system or link with additional analysis technologies (titration)
- Supports numerous sample vessels with different path lengths



Holder OMNIS NIR, vial, 8 mm

Vial Holder for the OMNIS NIR Analyzer for 8 mm disposable vials (6.7402.240).



Disposable vial, 8 mm, transmission, qty. 100

100 disposable glass vials (borosilicate) with an optical path length of 8 mm for analyses of liquids in transmission. The disposable vials are supplied with the associated stoppers (number of pieces = 100).

Compatible with:

- Holder OMNIS NIR, vial, 8 mm (6.07401.070)
- DS2500 holder for 8 mm disposable vials (6.7492.020)

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS Stand-Alone license

Enables stand-alone operation of the OMNIS software on a WindowsTM computer.

Features:

- The license already includes one OMNIS instrument license.
- Must be activated via the Metrohm licensing portal.

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Software license Quant Development

Software license for the creation and editing of quantification models in a stand-alone OMNIS Software installation.