Quality Control of Diesel

Moisture determination within one minute using NIRS

Summary

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

The standard specification for diesel fuel quality includes multiple parameters, but water contamination is the biggest risk factor. According to the European Committee for Standardization, the maximal acceptable amount of water in diesel 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.

Metrohm

Experimental equipment

Figure 1. DS2500 Liquid Analyzer and a sample filled in a disposable vial.

Samples of diesel with varying water contents (from 103 to 379 mg/L) were measured with a DS2500 Liquid Analyzer in transmission mode (400–2500 nm). Reproducible spectrum acquisition was achieved using the built-in temperature control at 40 °C. For convenience, disposable vials with a pathlength 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.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Liquid Analyzer	2.929.0010
DS2500 Holder 8 mm vials	6.7492.020
Disposable vials, 8 mm	6.7402.000
Vision Air 2.0 Complete	6.6072.208

2.929.0010 - 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.

6.7492.020 - DS2500 Holder 8 mm vials

Intelligent holder for disposable glass vials with 8 mm diameter

6.7402.000 - Disposable vials, 8 mm diameter, transmission

250 lockable disposable glass vials (borosilicate) with a diameter of 8 mm for analyses of liquid samples in transmission mode. Suitable for the following Analyzers:NIRS XDS RapidLiquid Analyzer NIRS XDS VialHeater + NIRS XDS Transmission OptiProbe Analyzer

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);

Result

The obtained Vis-NIR spectra (**Figure 2**) 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, which displays a very high correlation between the Vis-NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.
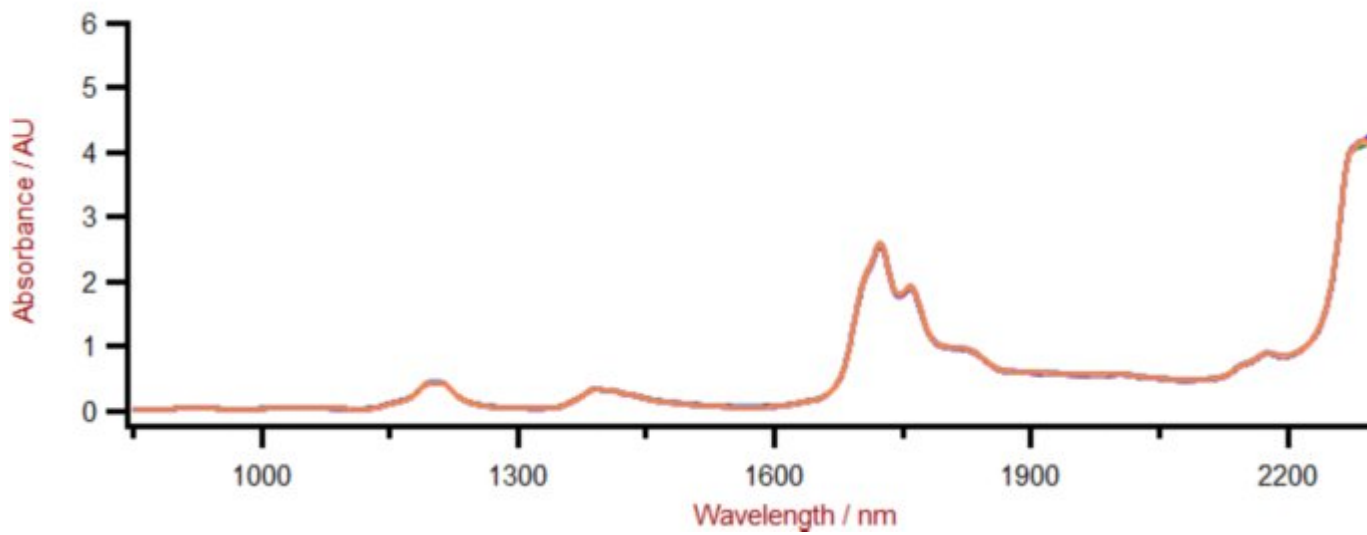
Figure 2. Vis-NIR spectra of diesel samples analyzed on a DS2500 Liquid Analyzer.
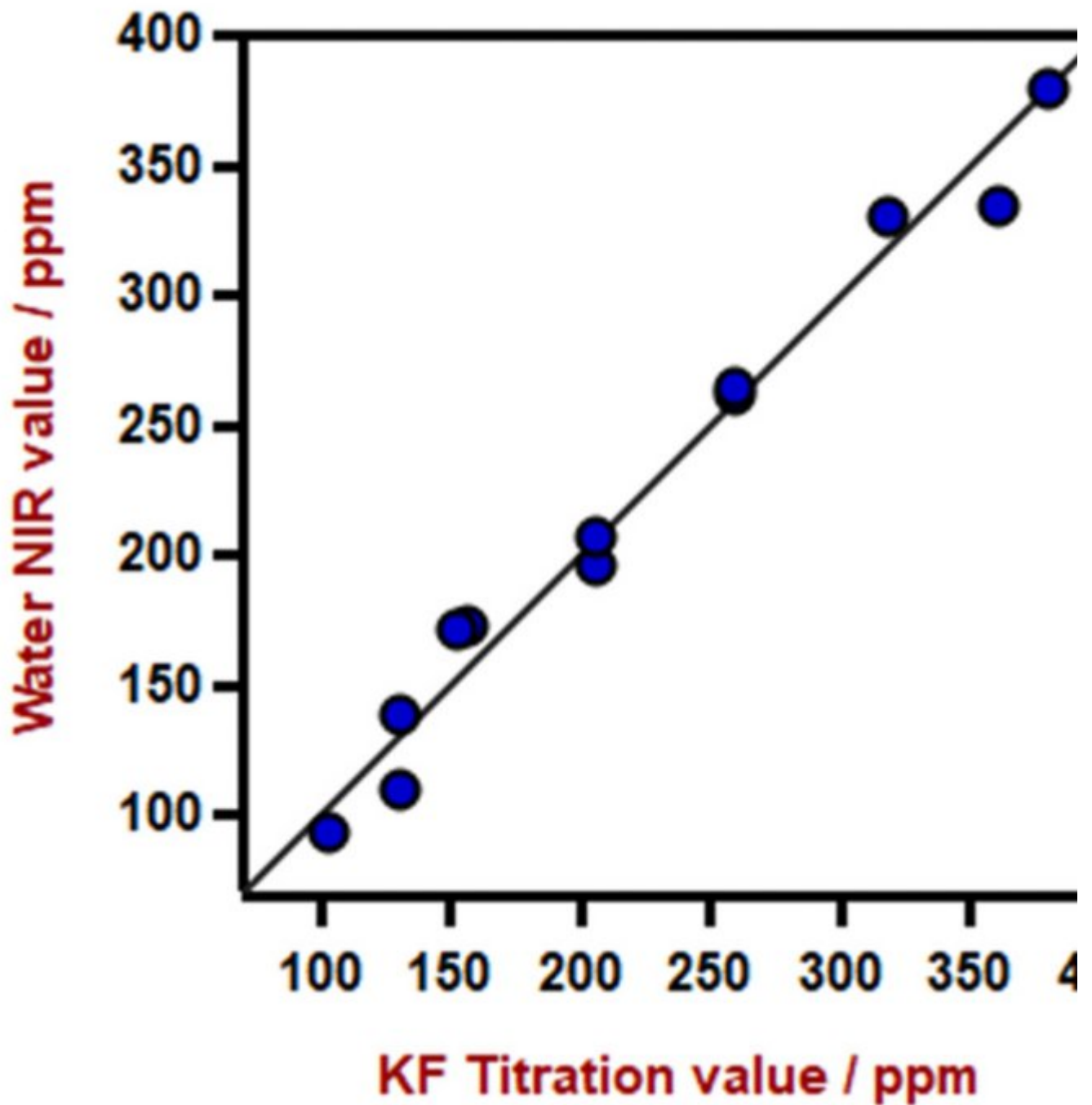
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Figure 3. Correlation diagram for the prediction of water content in diesel using a DS2500 Liquid Analyzer. The lab value was evaluated using KF titration.

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Table 2. Figures of merit for the prediction of water content in diesel using a DS2500 Liquid Analyzer.

Figures of merit	Value
R^2	0.9776
Standard error of calibration	16 ppm
Standard error of cross-validation	21 ppm

Conclusion

This application note demonstrates the feasibility to determine a key parameter of the quality control of diesel fuel (water content) with NIR spectroscopy. The main advantages of Vis-NIR spectroscopy over wet chemical methods are that running costs are significantly lower and time-to-result is significantly reduced. 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

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