



Application Note AN-NIR-096

Moisture Determination in Diesel

Moisture determination within one minute using NIRS

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.

EXPERIMENTAL EQUIPMENT

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.



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

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

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.

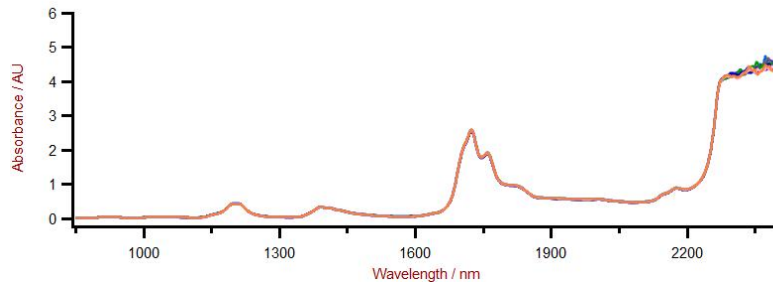


Figure 2. Vis-NIR spectra of diesel samples analyzed on a DS2500 Liquid Analyzer.

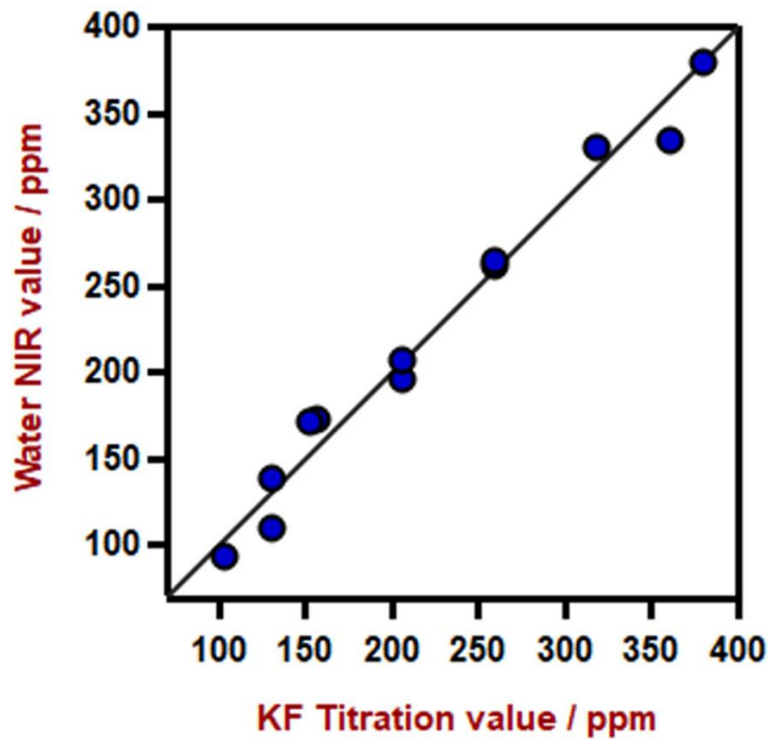


Figure 3. Correlation diagram for the prediction of water content in diesel using a DS2500 Liquid Analyzer. The lab value was evaluated using KF titration.

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

Internal reference: AW NIR CH-0064-112021

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DS2500 Liquid Analyzer

ラボおよび生産環境における品質管理用の堅牢な近赤外分光法。

NIRS DS2500 Liquid Analyzerは、生産チェーン全体に沿った液体のルーチン分析に実績のあるフレキシブルなソリューションです。頑丈な仕様により、NIRS DS2500 Liquid Analyzerは粉塵、湿気、および振動に強い為、過酷な生産環境での使用に理想的です。

DS2500 Liquid Analyzer は400~2500 nmのスペクトル範囲全体をカバーし、サンプルを80° Cまで加熱し、様々な使い捨てハイアルやクオーツキューベットとの互換性を有します。このようにして個々のサンプル要件に対応し得るDS2500 Liquid Analyzerは、1分未満で正確かつ再現性ある結果を得られるようサポートします。さらに一体型のサンプルホルダー検出、および説明不要のVision Airソフトウェアを用いることで、ユーザーが簡単かつ安全に操作できることが保証されます。

サンプル量が多い場合、Metrohmサンプルロケットと組み合わされたフローセルの導入によって、生産性を著しく向上させることができます。



8mmDS2500

直径8 mmのガラス製使い捨てハイアル用インテリジェントホルダー



Vision Air 2.0 Complete

Vision Air - 汎用性に優れた分光法ソフトウェア。

Vision Air Complete は、規制環境下での使用のための、操作の容易な最新のソフトウェアソリューションです。

Vision Air の利点の概要:

- 調整済みのユーザーインターフェースを伴う個別のソフトウェアアプリケーションにより、直観的かつ容易な操作が保証されます。
- 作業手順の容易な作成およびメンテナンス
- 安全かつ容易なデータ管理のための SQL データベース

バージョン Vision Air Complete (66072208) には、可視近赤外分光法を用いた品質管理のための全てのアプリケーションが含まれています:

- 装置管理およびデータ管理のためのアプリケーション
- メソッド開発のためのアプリケーション
- ルーチン分析のためのアプリケーション

その他の Vision Air Complete ソリューション:

- 66072207 (Vision Air Network Complete)
- 66072209 (Vision Air Pharma Complete)
- 66072210 (Vision Air Pharma Network Complete)