Quality Control of Diesel Exhaust Fluid

Fast determination of urea content with high accuracy

Summary

The quality control of diesel exhaust fluids (DEF) is key to ensure the optimal catalytic performance and prevent damage to the exhaust system in diesel vehicles. The standard method to determine urea content is measuring the refractive index (ISO 22241-2:2019). The issue is that although this method is fast, it is not as accurate as other methods (e.g., HPLC). This application note demonstrates that the DS2500 Liquid Analyzer provides a **fast solution with high accuracy** for the determination of urea in DEF. With **no sample preparation or chemicals needed**, visible near infrared (Vis-NIR) spectroscopy allows for the analysis of diesel exhaust fluids in **less than a minute**.



Experimental equipment



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

Aqueous urea samples with different urea content from 0.5% to 40% (v/v) were measured in transmission mode with a DS2500 Liquid Analyzer over the full wavelength range (400–2500 nm). Reproducible spectrum acquisition was achieved using the built-in temperature control at 40 °C. For convenience, disposable vials with a path length of 2 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
Disposable vials, 2 mm diameter, transmission	6.7492.000
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.



6.7492.000 - DS2500 Holder 2 mm vials

Intelligent holder for disposable glass vials with 2 mm diameter



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);



Results

All 16 measured Vis-NIR spectra (**Figure 2**) were used to create a prediction model for quantification of the urea content. The quality of the prediction models was evaluated using correlation diagrams, which display a very high 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.

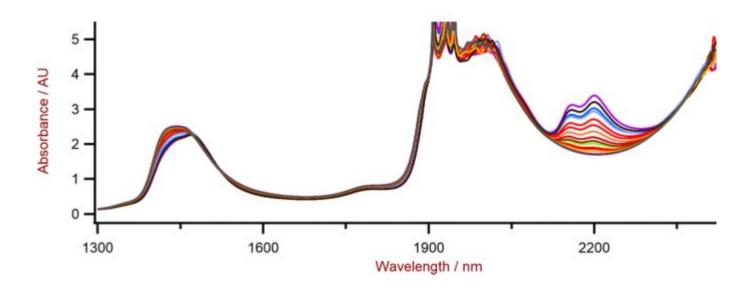


Figure 2. Vis-NIR spectra of diesel exhaust fluids with varying urea content measured on a DS2500 Liquid Analyzer.

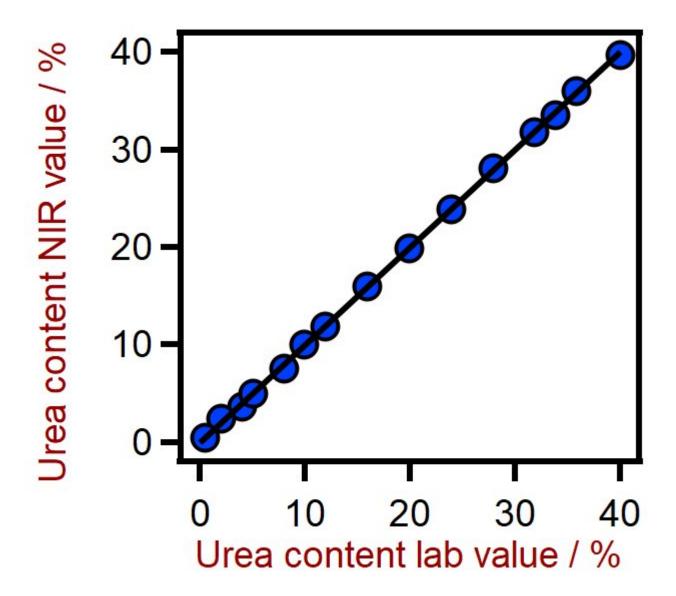


Figure 3. Correlation diagram for the prediction of urea content in diesel exhaust fluids using a DS2500 Liquid Analyzer.

Table 2. Figures of merit for the prediction of urea content in diesel exhaust fluids using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.999
Standard error of calibration	0.23%
Standard error of cross-validation	0.25%

CONCLUSION

This application note demonstrates the feasibility of the DS2500 Liquid Analyzer for the determination of urea content in diesel exhaust fluids. Vis-NIR spectroscopy enables a fast determination with high accuracy, and therefore represents a suitable alternative to the standard method. Additionally, it should be pointed out, that for further parameters such as density, similar NIR methods can be developed.

Table 3. Time to result for the urea content determination in aqueous urea solutions using HPLC

Parameter	Method	Time to result and workflow
Urea content	HPLC	5 min (preparation) + 10 min (HPLC)
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