



Application Note AN-NIR-111

Iodine value, FFA, refractive index, and fatty acid composition in edible oils

Multiparameter analysis within one minute using NIRS

Edible oils are considered essential nutrients for our daily diet. Various parameters are used to determine the oil quality including the iodine value, free fatty acids (FFA), refractive index, and fatty acid composition. The fatty acid profile provides a detailed view regarding the concentration of different fatty acids present in the oil. The content of essential linoleic acid (C18:2) and alpha-linolenic acid (C18:3) is especially interesting for producers.

Traditional analysis techniques like titration or gas chromatography often require hazardous solvents which can pose health risks and increase laboratory costs. In contrast to these standard methods, all edible oil quality parameters can be analyzed with the Metrohm NIRS DS2500 Liquid Analyzer. Analysis via near-infrared spectroscopy (NIRS) helps to increase productivity and reduce costs, providing quick results without any sample preparation.

EXPERIMENTAL EQUIPMENT

In total, 397 different samples from several types of edible oils (including sunflower-, rapeseed-, sesame-, and soybean oil) were measured on the Metrohm NIRS DS2500 Liquid Analyzer (Figure 1). All measurements were performed in transmission mode using 8 mm disposable vials. The temperature control of the analyzer was set to 40 °C to ensure consistent

measurement performance. Iodine values were calculated based on fatty acid composition data obtained from gas chromatography analysis. Spectral data acquisition and prediction model development were performed with the Metrohm software package Vision Air Complete.

Equipment	Article number
DS2500 Liquid Analyzer	2.929.0010
DS2500 Holder 8 mm vials	6.7492.020
Vision Air 2.0 Complete	6.6072.208



Figure 1. Metrohm NIRS DS2500 Liquid Analyzer used for the measurement of several quality control parameters in edible oils.

RESULT

The measured Vis-NIR spectra (Figure 2) were used to create a prediction model for quantification of all eight parameters: iodine value, FFA, refractive index, palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2), and alpha-linolenic acid (C18:3). The quality of the prediction models was

evaluated using correlation diagrams which display a high correlation ($R^2 > 0.94$) between the Vis-NIR prediction and the standard reference methods for all parameters. The respective figures of merit (FOM) display the expected precision and confirm the feasibility during routine analysis (Figures 3–10).

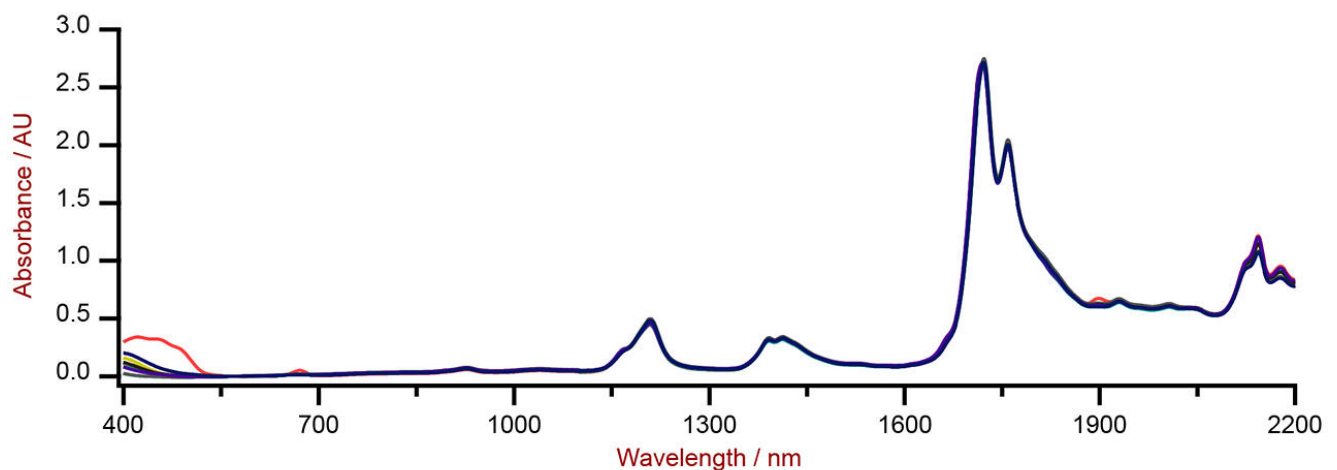


Figure 2. Selection of Vis-NIR spectra of edible oil samples analyzed on a Metrohm NIRS DS2500 Liquid Analyzer with 8 mm disposable vials.

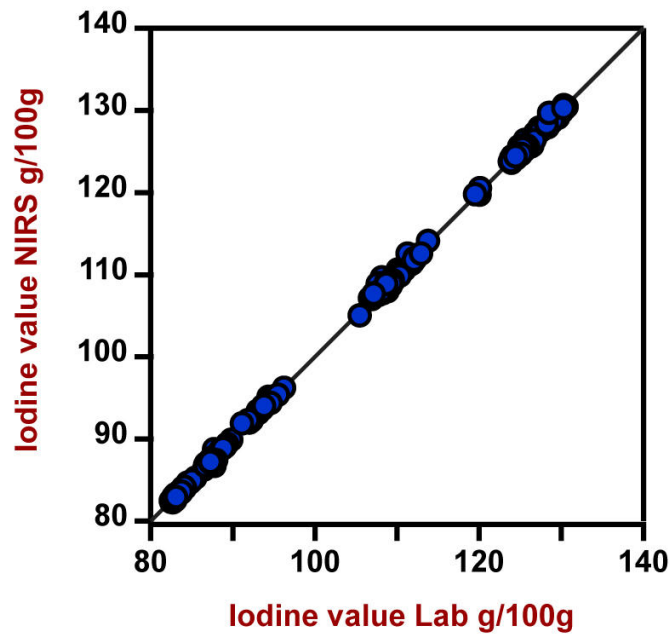


Figure 3. Correlation diagram and the respective figures of merit for the prediction of iodine value in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using gas chromatography.

Figures of Merit	Value
R ²	0.999
Standard Error of Calibration	0.29 g/100 g
Standard Error of Cross-Validation	0.30 g/100 g

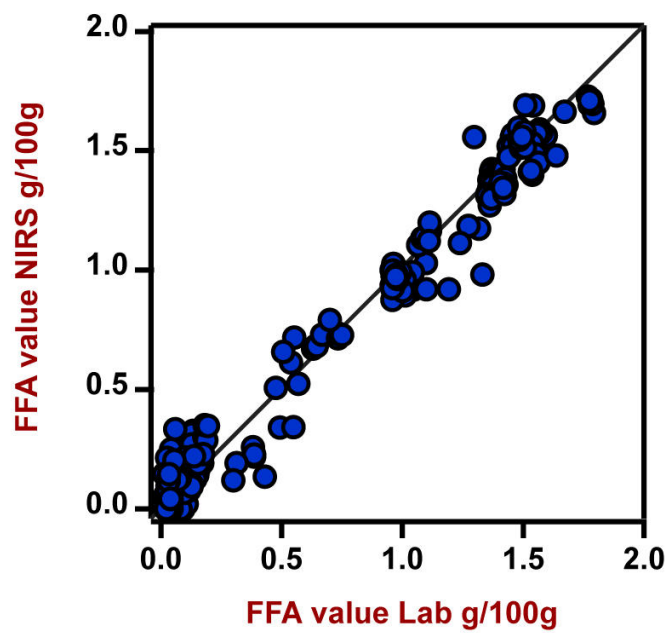


Figure 4. Correlation diagram and the respective figures of merit for the prediction of FFA value in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using a titration method.

Figures of Merit	Value
R ²	0.983
Standard Error of Calibration	0.08 g/100 g
Standard Error of Cross-Validation	0.08 g/100 g

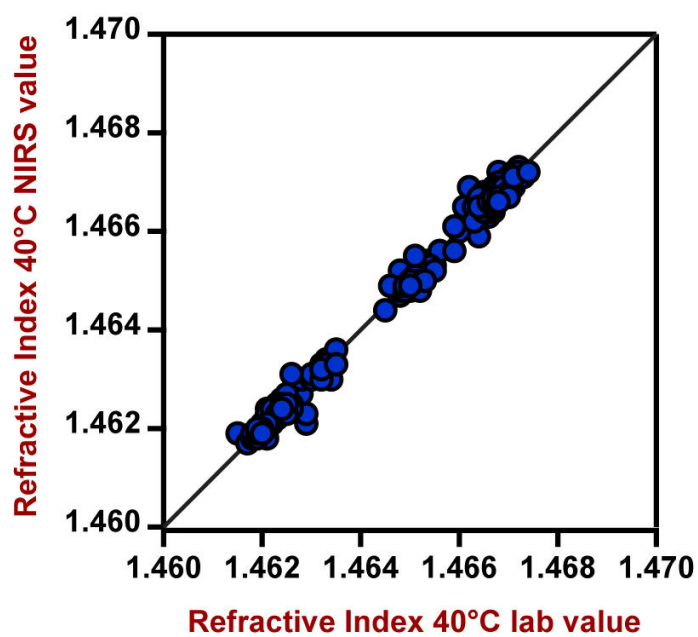


Figure 5. Correlation diagram and the respective figures of merit for the prediction of refractive index in edible oils using a DS2500 Liquid Analyzer. The lab value was evaluated using a refractometer.

Figures of Merit	Value
R2	0.996
Standard Error of Calibration	0.0001
Standard Error of Cross-Validation	0.0001

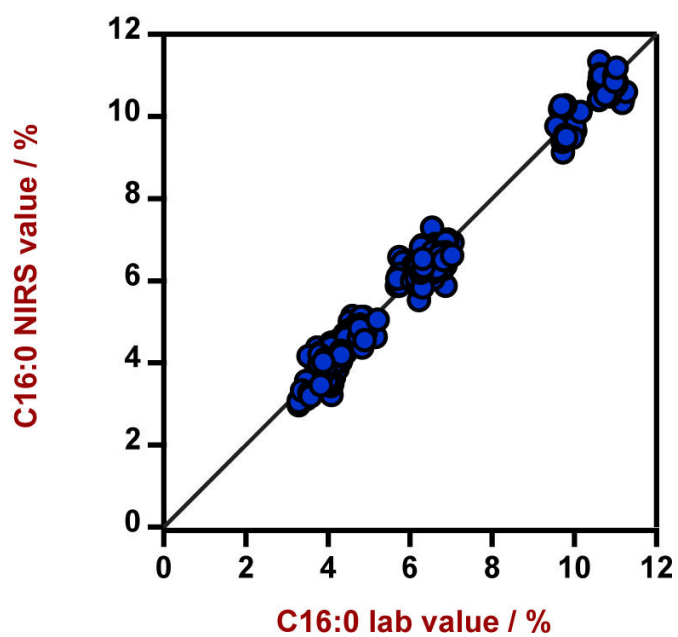


Figure 6. Correlation diagram and the respective figures of merit for the prediction of relative C16:0 fatty acid (palmitic acid) content in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using gas chromatography.

Figures of Merit	Value
R ²	0.980
Standard Error of Calibration	0.29%
Standard Error of Cross-Validation	0.30%

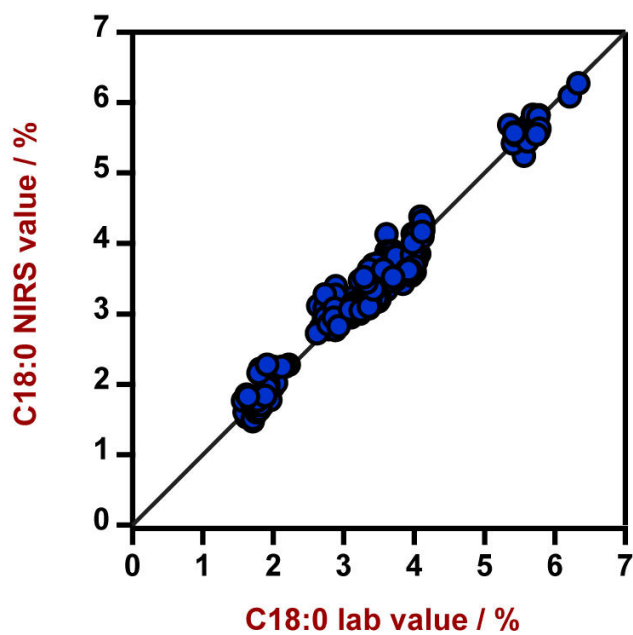


Figure 7. Correlation diagram and the respective figures of merit for the prediction of relative C18:0 fatty acid (stearic acid) content in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using gas chromatography.

Figures of Merit	Value
R2	0.967
Standard Error of Calibration	0.18%
Standard Error of Cross-Validation	0.18%

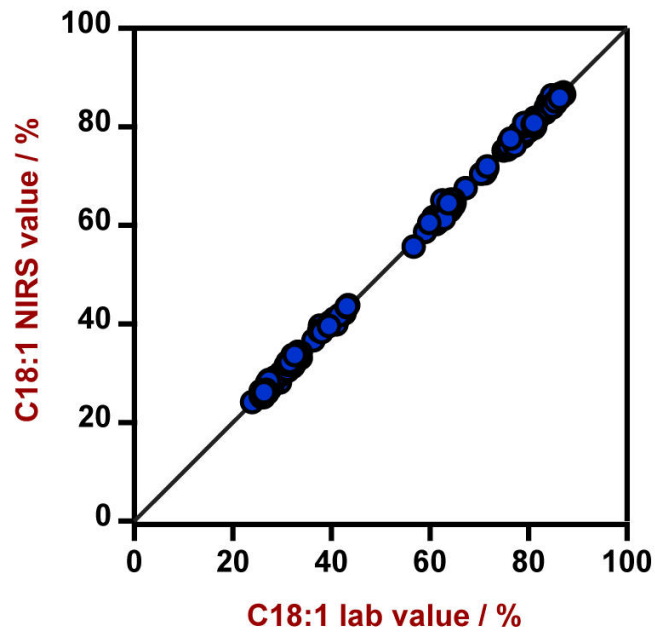


Figure 8. Correlation diagram and the respective figures of merit for the prediction of relative C18:1 fatty acid (oleic acid) content in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using gas chromatography.

Figures of Merit	Value
R ²	0.999
Standard Error of Calibration	0.57%
Standard Error of Cross-Validation	0.62%

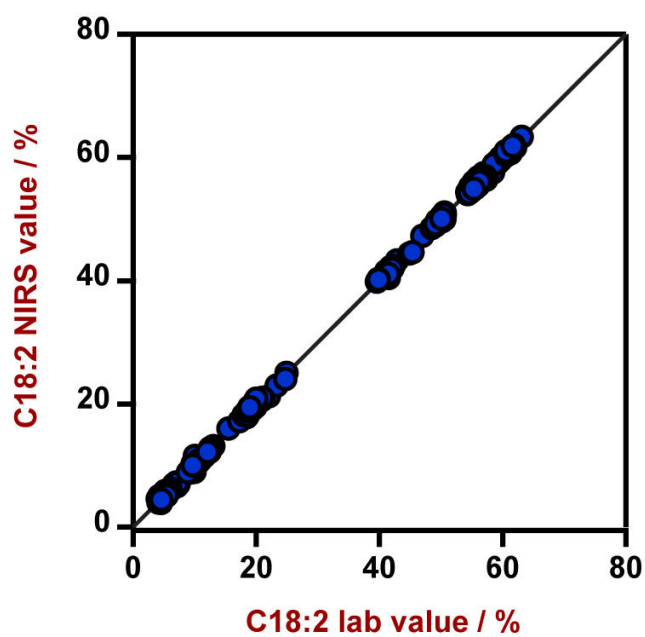


Figure 9. Correlation diagram and the respective figures of merit for the prediction of relative C18:2 fatty acid (linoleic acid) content in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using gas chromatography.

Figures of Merit	Value
R ²	0.999
Standard Error of Calibration	0.33%
Standard Error of Cross-Validation	0.37%

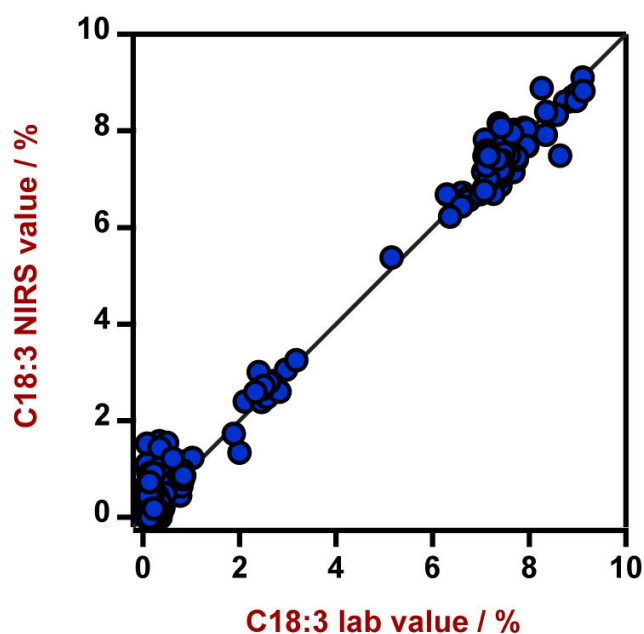


Figure 10. Correlation diagram and the respective figures of merit for the prediction of relative C18:3 fatty acid (alpha-linolenic acid) content in edible oils using a DS2500 Liquid Analyzer. The lab value was measured using gas chromatography.

Figures of Merit	Value
R2	0.990
Standard Error of Calibration	0.32%
Standard Error of Cross-Validation	0.34%

CONCLUSION

This Application Note displays the benefit of using the Metrohm NIRS DS2500 Liquid Analyzer for routine analysis of QC parameters in the laboratories of edible oil manufacturers. Compared to other conventional

methods, measurements performed with Vis-NIR spectroscopy do not need any sample preparation. This ultimately leads to a reduction of workload (Table 2) and related costs.

Table 2. Time to result overview for the measurement of iodine value, FFA content, refractive index, and fatty acid composition in edible oils by standard analytical methods.

Parameter	Method	Time to result
Iodine value	Gas Chromatography	30 minutes per sample
FFA content	Titration	10 minutes per sample
Refractive index	Refractometer	2 minutes per sample
Fatty acid composition	Gas Chromatography	30 minutes per sample

CONTACT

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CONFIGURATION



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.