Application Note AN-NIR-125

Determination of olive oil quality parameters and adulteration with NIR spectroscopy

Near-infrared spectroscopy reduces costs and chemical waste

Olive oil quality depends on many factors, such as time spent processing olives after harvest, the production process itself, and olive variety. Due to its high price, virgin olive oil in particular is one of the most vulnerable vegetable oils for food fraud. Many parameters are used to determine the oil quality including the iodine value, free fatty acids (FFA), refractive index, fatty acid composition, and aging indicators such as peroxide value (PV), K232, and induction time. Traditional analysis techniques for olive oil testing like titration or gas chromatography (GC) often require hazardous solvents which can pose health risks and increase laboratory costs. In contrast to these standard methods, the analysis with near-infrared spectroscopy (NIRS) helps to increase productivity and reduce costs, providing quick results for olive oil quality control

EXPERIMENTAL EQUIPMENT

A selection of olive oils with varying quality (137 samples) were measured on the OMNIS NIR Analyzer Liquid (Figure 1) in transmission mode (1000–2250 nm) using 8 mm disposable vials. The vial temperature was set and monitored at 40 °C with the built-in vial sensor to ensure consistent measurement performance. The OMNIS software was used for all data acquisition and prediction model development.

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

The obtained NIR spectra (Figure 2) were used to create a prediction model for quantification of all parameters: iodine value, FFA, refractive index, K232, PV, induction time, 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 (Figures 3–8) which display a high correlation between the NIR prediction and the standard reference methods for all parameters. Of the 137 samples measured, 25% were selected as validation set and 75% as calibration set. The respective figures of merit (FOM), shown for the following figures and in Table 2, display the expected precision and confirm the feasibility during routine analysis.

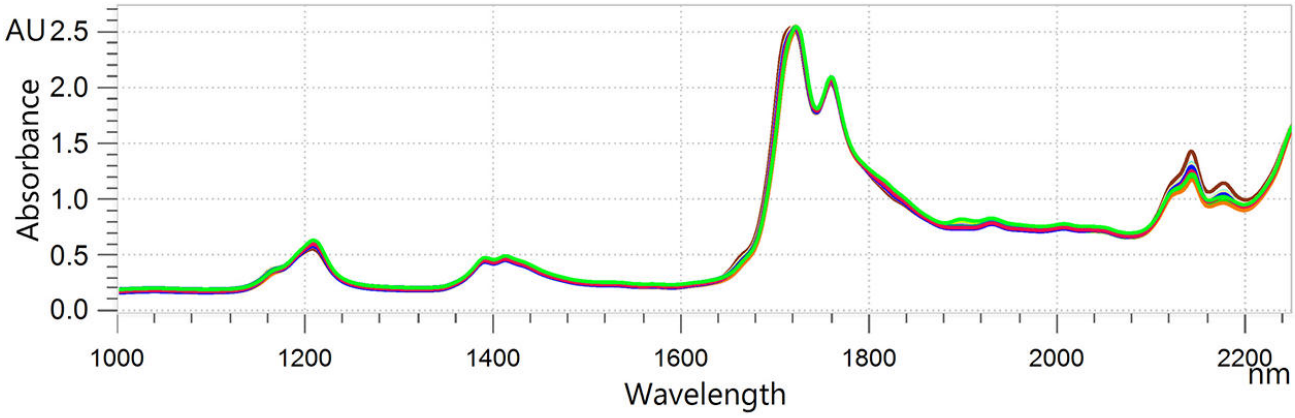

Figure 2. NIR spectra of olive oil samples analyzed on an OMNIS NIR Analyzer Liquid with 8 mm vials.

RESULTS

Result iodine value

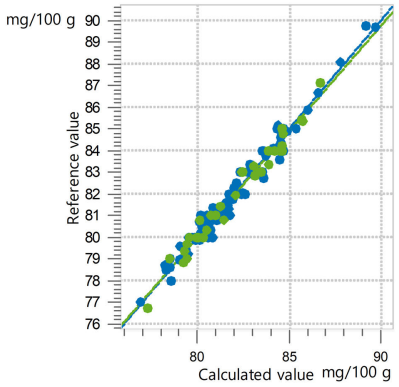

Figure 3. Correlation diagram and the respective FOMs for the prediction of iodine value in olive oil. Lab values were evaluated using GC.

Parameter	SEC (mg/100 g)	SECV (mg/100 g)	SEP (mg/100 g)	R^2CV
IV	0.38	0.40	0.38	0.974

Result K232

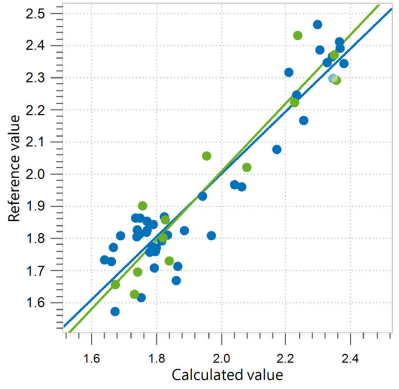

Figure 4. Correlation diagram and the respective FOMs for the prediction of K232 in olive oil. UV analysis was used to obtain the lab values.

Parameter	SEC	SECV	SEP	R^2CV
K232	0.067	0.086	0.090	0.864

Result C16:0 fatty acid content

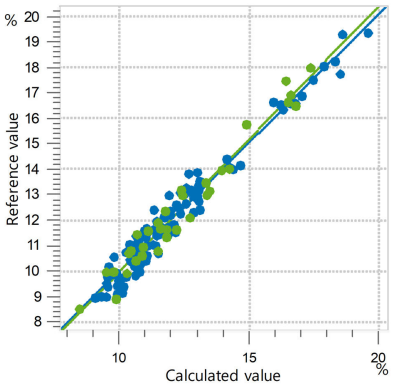

Figure 5. Correlation diagram and the respective FOMs for the prediction of C16:0 content in olive oil. Lab values were evaluated using GC.

Parameter	SEC (%)	SECV (%)	SEP (%)	R^2CV
C16:0	0.32	0.38	0.48	0.962

Result C18:1 fatty acid content

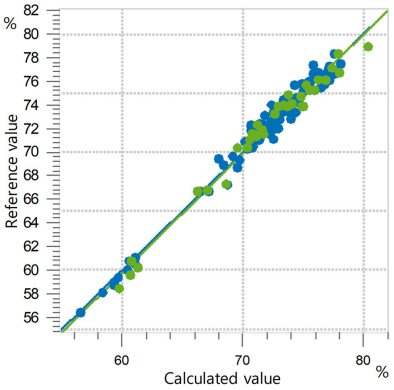

Figure 6. Correlation diagram and the respective FOMs for the prediction of C18:1 content in olive oil. Lab values were evaluated using GC.

Parameter	SEC (%)	SECV (%)	SEP (%)	R^2CV
C18:1	0.63	0.69	0.75	0.980

Result C18:2 fatty acid content

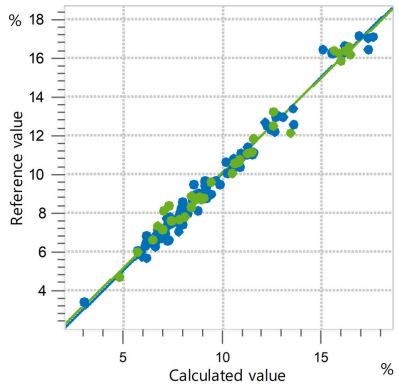

Figure 7. Correlation diagram and the respective FOMs for the prediction of C18:2 content in olive oil. Lab values were evaluated using GC.

Parameter	SEC (%)	SECV (%)	SEP (%)	R^2CV
C18:2	0.32	0.38	0.43	0.985

Result induction time

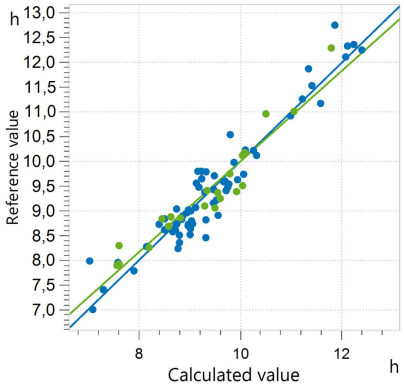

Figure 8. Correlation diagram and the respective FOMs for the prediction of olive oil induction time. Lab values were evaluated with a Rancimat.

Parameter	SEC (h)	SECV (h)	SEP (h)	R^2CV
Induction time	0.30	0.35	0.34	0.908

Table 2. Figures of merit for the parameters of stearic acid, α-linolenic acid, FFA, peroxide value, and refractive index in various olive oils.

Parameter	SEC	SECV	SEP	R2CV
Stearic acid (C18:0)	0.12%	0.22%	0.22%	0.778
α-linolenic acid (C18:3)	0.05%	0.05%	0.05%	0.633
FFA	0.03%	0.04%	0.04%	0.746
Peroxide value	0.72 meq/kg	0.83 meq/kg	1.01 meq/kg	0.719
Refractive index	0.00011	0.00012	0.00012	0.998

CONCLUSION

This Application Note displays the positive attributes of olive oil analysis with near-infrared spectroscopy. Compared to time-consuming conventional analytical methods, measurements performed with NIRS do not need any sample preparation. This ultimately leads to a workload reduction (Table 3) and reduced costs. Aside from the parameters shown in this Application Note, additional olive oil quality parameters like sterol content or moisture content can also be determined with NIRS.

Table 3. Time to result overview for the measurement of iodine value, FFA content, refractive index, K232, induction time, and fatty acid composition in olive oils by standard analytical methods.

Parameter	Method	Time to result
Iodine value	Gas chromatography	~30 minutes per sample
FFA content, Peroxide value	Titration	~15 minutes per sample
Refractive index	Refractometer	~5 minutes per sample
K232	UV absorption	~5 minutes per sample
Fatty acid composition	Gas chromatography	~30 minutes per sample
Induction time	Rancimat	~1–15 hours per sample

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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)

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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.
- Not transferable to another computer.

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Software license Quant Development

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