

Application Note AN-NIR-125

采用近红外光谱技术测定橄榄油品质参数及掺假检测

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.

RESULTS

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.

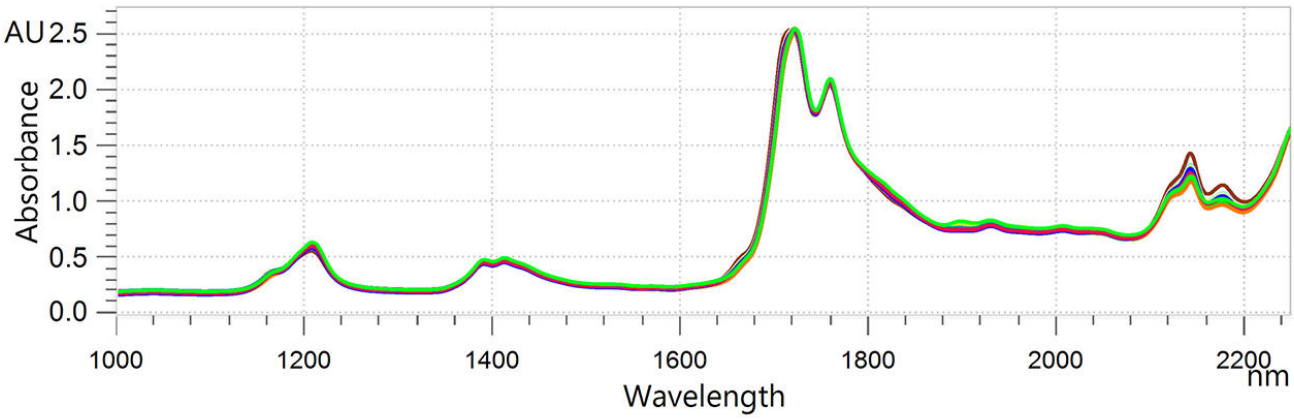


Figure 2. NIR spectra of olive oil samples analyzed on an OMNIS NIR Analyzer Liquid with 8 mm vials.

Result iodine value

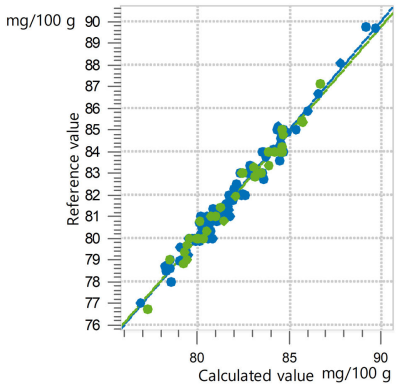


Figure 3. Correlation diagram and the respective FOMs for the prediction of iodine value in olive oil. Lab values were evaluated using GC.

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

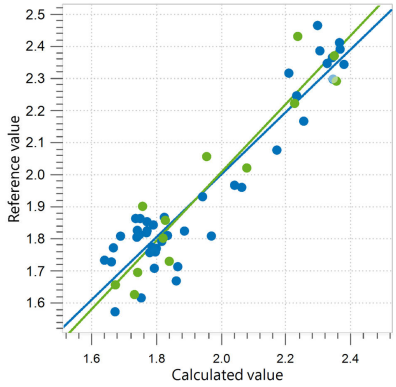


Figure 4. Correlation diagram and the respective FOMs for the prediction of K232 in olive oil. UV analysis was used to obtain the lab values.

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

Result C16:0 fatty acid content

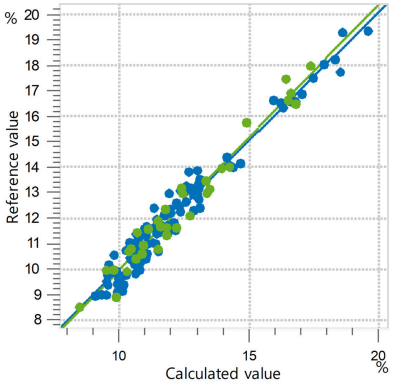


Figure 5. Correlation diagram and the respective FOMs for the prediction of C16:0 content in olive oil. Lab values were evaluated using GC.

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

Result C18:1 fatty acid content

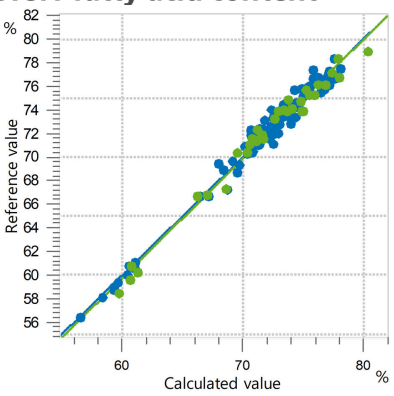


Figure 6. Correlation diagram and the respective FOMs for the prediction of C18:1 content in olive oil. Lab values were evaluated using GC.

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

Result C18:2 fatty acid content

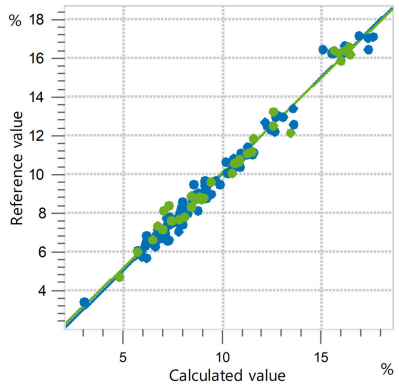


Figure 7. Correlation diagram and the respective FOMs for the prediction of C18:2 content in olive oil. Lab values were evaluated using GC.

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

Result induction time

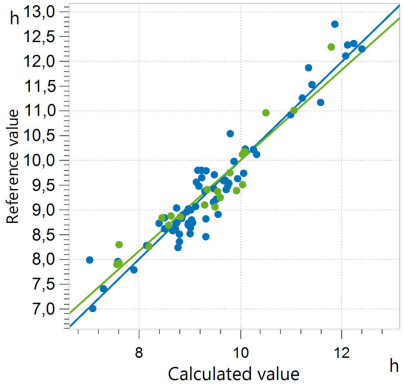


Figure 8. Correlation diagram and the respective FOMs for the prediction of olive oil induction time. Lab values were evaluated with a Rancimat.

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

用于液体样品的近红外光谱仪。

OMNIS NIR Analyzer 按照瑞士质量标准开发和制造,是用于整个生产链中常规分析的近红外光谱解决方案。最新技术的应用以及与现代 OMNIS Software 的集成,体现在这款 NIR 光谱仪的速度、操作性和灵活应用上。

OMNIS NIR Analyzer Liquid 的优势概览:

- 液体样品测量时间小于 10 秒
- 样品温度调节范围为 25–80 °C
- 自动检测样品容器的放入和移除
- 可轻松集成至自动系统,或与其它分析技术(滴定)联用。
- 支持多种不同路径长度的样品容器



OMNIS NIR8 mm

适合 8 mm 一次性小管的 OMNIS NIR Analyzer 的小管支架 (6.7402.240)。



8 mm 100

100 个玻璃(硼硅)一次性样品瓶,具有 8 mm 的光路长度,适用于分析透射中的液体。一次性样品瓶交付时带有所属的螺旋塞(件数 = 100)。

兼容:

- 支架 OMNIS NIR,小管,8 mm (6.07401.070)
- DS2500 支架适用于 8 mm 一次性样品瓶 (6.7492.020)

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