



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.

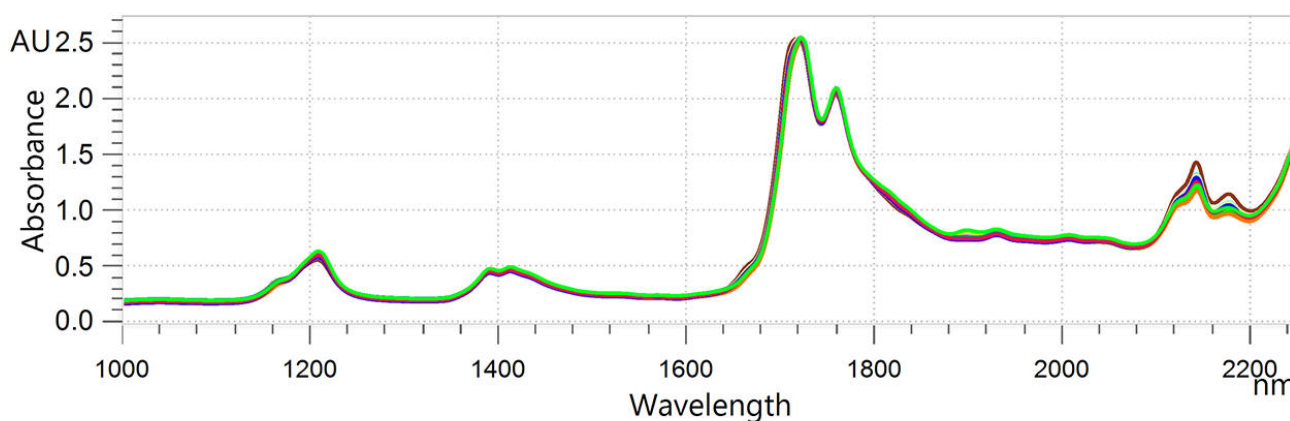


**Figure 1.** The OMNIS NIR Analyzer and a sample filled in a disposable vial.

## RESULTS

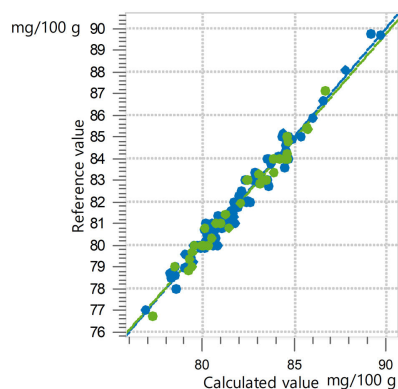
The obtained NIR spectra (Figure 2) were used to create a prediction model for quantification of all parameters: iodine value, FFA, refractive index, K232, PV, induction time, palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2), and alpha-linolenic acid (C18:3). The 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 1, 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.

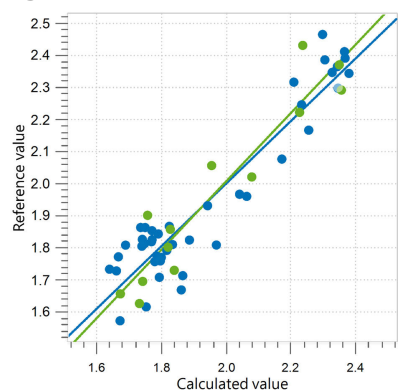
### Result iodine value



**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 <sup>2</sup> CV |
|-----------|----------------|-----------------|----------------|-------------------|
| IV        | 0.38           | 0.40            | 0.38           | 0.974             |

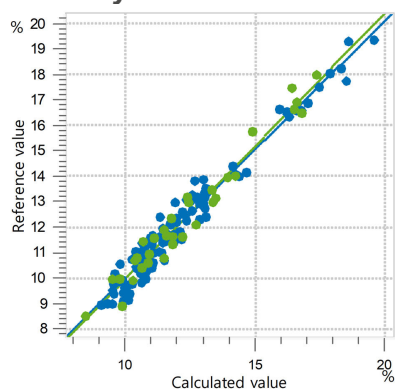
### Result K232



**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 <sup>2</sup> CV |
|-----------|-------|-------|-------|-------------------|
| K232      | 0.067 | 0.086 | 0.090 | 0.864             |

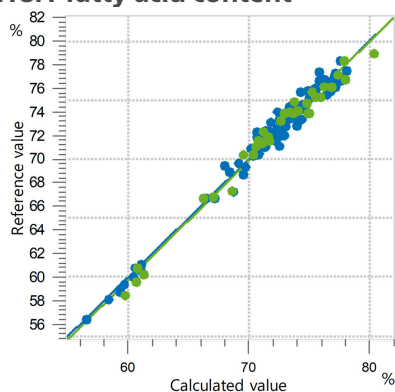
### Result C16:0 fatty acid content



**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 <sup>2</sup> CV |
|-----------|---------|----------|---------|-------------------|
| C16:0     | 0.32    | 0.38     | 0.48    | 0.962             |

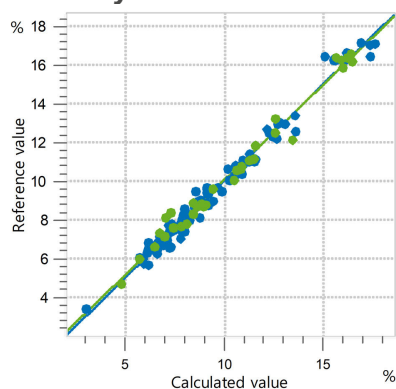
### Result C18:1 fatty acid content



**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 <sup>2</sup> CV |
|-----------|---------|----------|---------|-------------------|
| C18:1     | 0.63    | 0.69     | 0.75    | 0.980             |

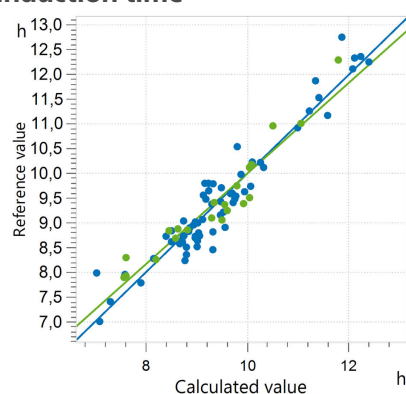
## Result C18:2 fatty acid content



**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 <sup>2</sup> CV |
|-----------|---------|----------|---------|-------------------|
| 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 <sup>2</sup> CV |
|----------------|---------|----------|---------|-------------------|
| Induction time | 0.30    | 0.35     | 0.34    | 0.908             |

**Table 1.** Figures of merit for the parameters of stearic acid,  $\alpha$ -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 |
| $\alpha$ -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 2**) 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 2.** 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 |

## CONTACT

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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).





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#### 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)

#### OMNIS Stand-Alone license

Enables stand-alone operation of the OMNIS software on a Windows<sup>TM</sup> computer.

Features:

- The license already includes one OMNIS instrument license.
- Must be activated via the Metrohm licensing portal.

#### Software license Quant Development

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