Application Note AN-NIR-111

Iodine value, FFA, refractive index, and fatty acid composition with NIRS

Multiparameter analysis of edible oils within a few seconds

Edible oils are essential for our diet. Various parameters are used to assess oil quality including the determination of iodine value, free fatty acids (FFA), refractive index, and fatty acid composition. Fatty acid composition analysis 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) are especially interesting for edible oil producers.

Traditional analysis techniques like titration or gas chromatography can be time consuming and often require the use of hazardous solvents which can pose health risks and increase analysis costs. In contrast to these standard methods, each of the mentioned edible oil quality parameters can be analyzed simultaneously, without sample preparation, and in a few seconds via near-infrared spectroscopy (NIRS) with the OMNIS NIRS Analyzer.

EXPERIMENTAL EQUIPMENT

More than 1000 samples of several types of edible oils (including sunflower-, rapeseed-, sesame-, and soybean oil) were measured on the OMNIS NIR Analyzer Liquid in transmission mode (1000–2250 nm) using 8 mm disposable vials. The temperature control of the NIRS analyzer was set to 40 °C to ensure consistent measurement performance. The OMNIS software was used for all data acquisition and prediction model development.

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

Table 1. Hardware and software equipment overview.

Equipment	Article number
OMNIS NIR Analyzer Liquid	2.1070.0010
Holder OMNIS NIR, vial, 8 mm	6.07401.070
Disposable vial, 8 mm, transmission	6.7402.240
OMNIS Stand-Alone license	6.06003.010
Quant Development software license	6.06008.002

The obtained NIR spectra (Figure 2) were used to create prediction models to quantify all eight oil quality parameters: iodine value (IV), 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 NIR prediction and the standard reference methods for all parameters. Out of the total, 25% of the samples were selected as the validation set and the other 75% as a calibration set. The respective figures of merit (FOM) display the expected precision and confirm the feasibility during routine analysis (Figures 3–10).

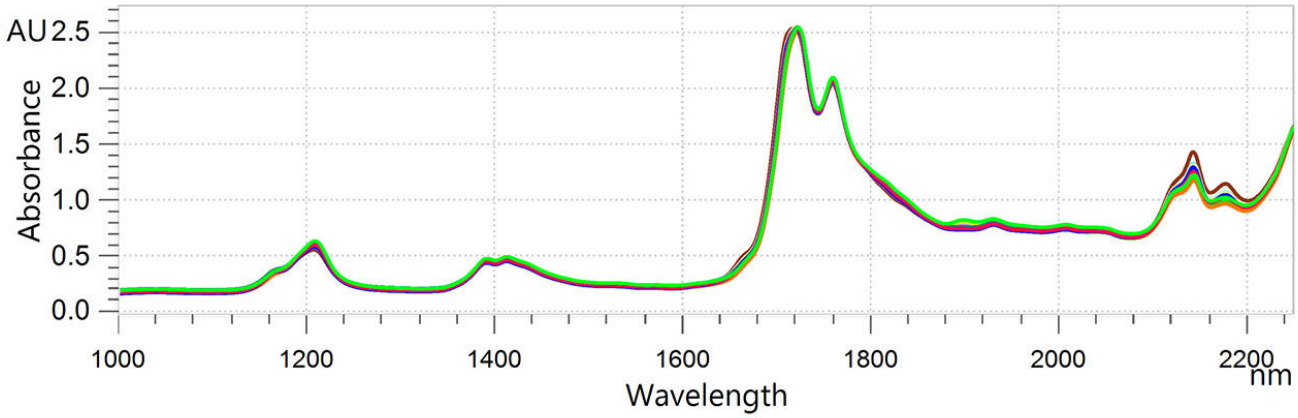

Figure 2. Overlaid NIR spectra of edible oil samples which were analyzed on an OMNIS NIR Analyzer Liquid at 40 °C with 8 mm vials.

RESULT IODINE VALUE

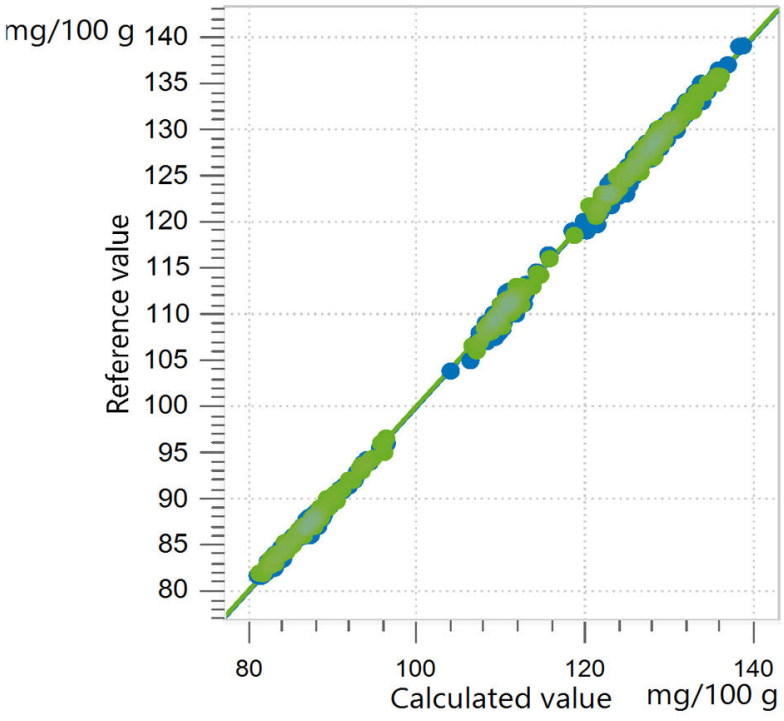

Figure 3. Correlation diagram and the respective figures of merit for the prediction of iodine value (also known as iodine number) in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

Parameter	SEC (mg/100g)	SECV (mg/100g)	SEP (mg/100g)	R2CV
IV	0.47	0.48	0.50	0.999

RESULT FREE FATTY ACIDS

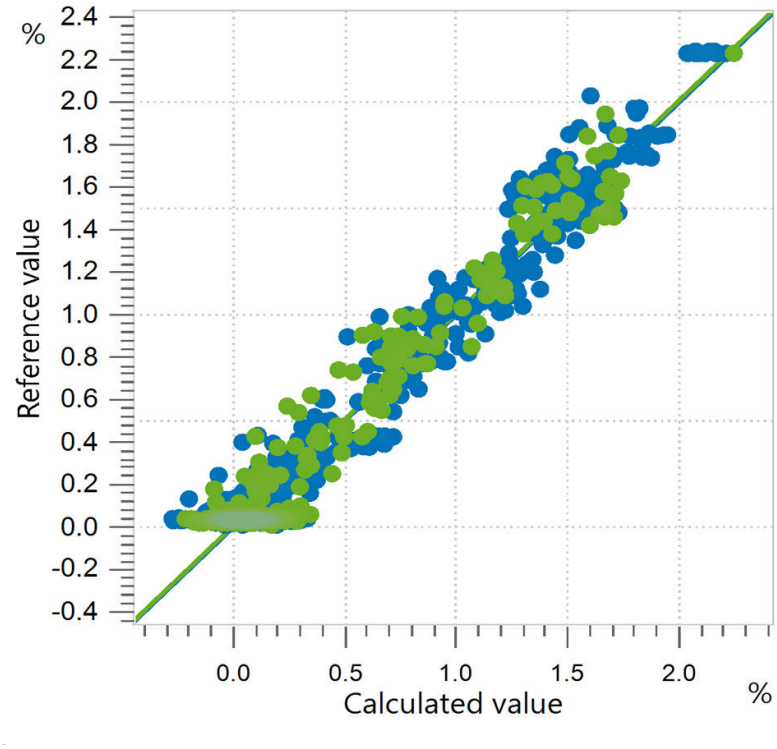

Figure 4. Correlation diagram and the respective figures of merit for the prediction of FFA in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using a titration method.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
FFA	0.12	0.12	0.13	0.946

RESULT REFRACTIVE INDEX

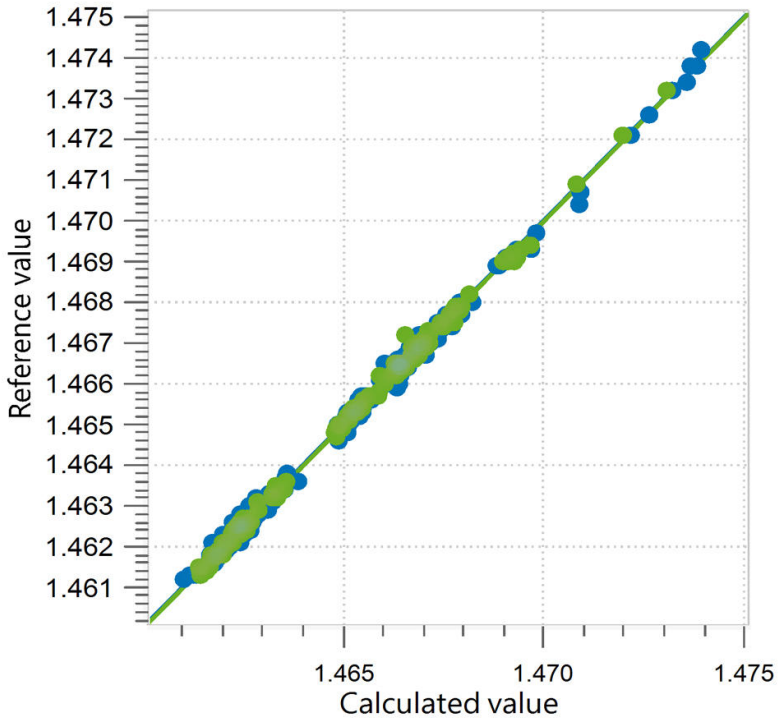

Figure 5. Correlation diagram and the respective figures of merit for the prediction of refractive index (RI) in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using a refractometer.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
RI	0.00011	0.00012	0.00012	0.998

RESULT C16:0 FATTY ACID CONTENT

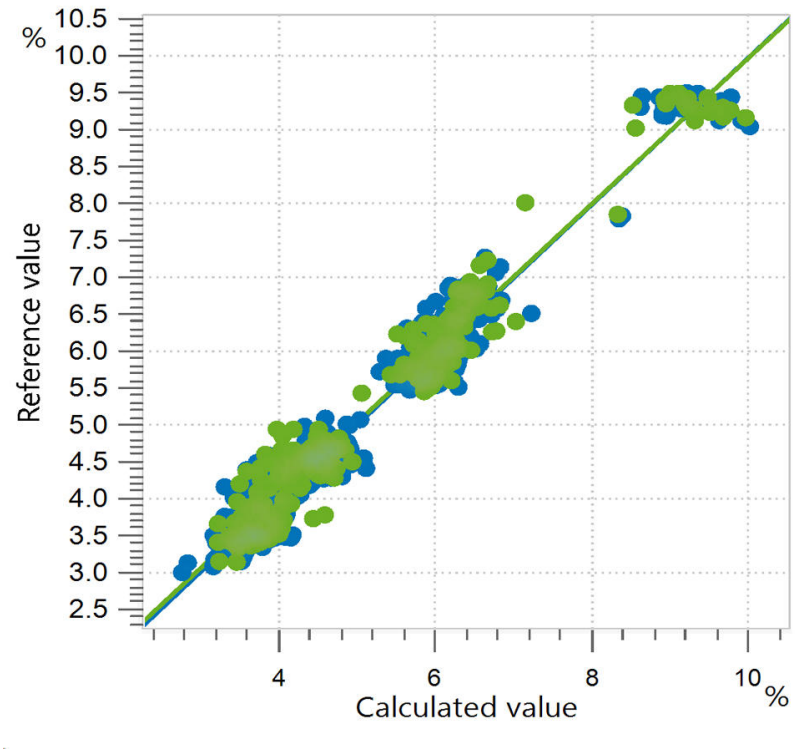

Figure 6. Correlation diagram and the respective figures of merit for the prediction of relative C16:0 fatty acid (palmitic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
C16:0	0.26	0.27	0.31	0.958

RESULT C18:0 FATTY ACID CONTENT

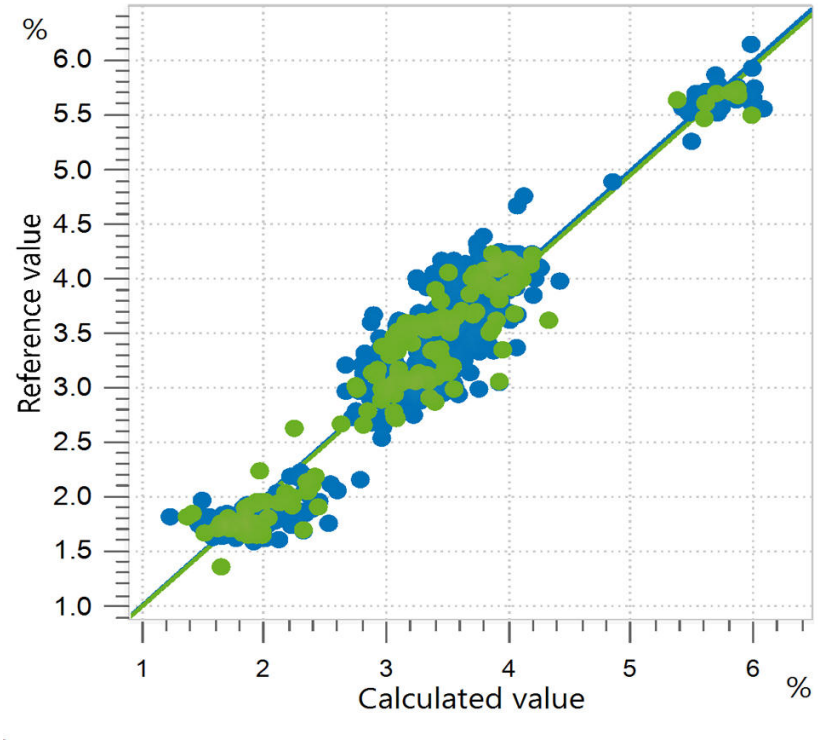

Figure 7. Correlation diagram and the respective figures of merit for the prediction of C18:0 fatty acid (stearic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
C18:0	0.26	0.27	0.25	0.936

RESULT C18:1 FATTY ACID CONTENT

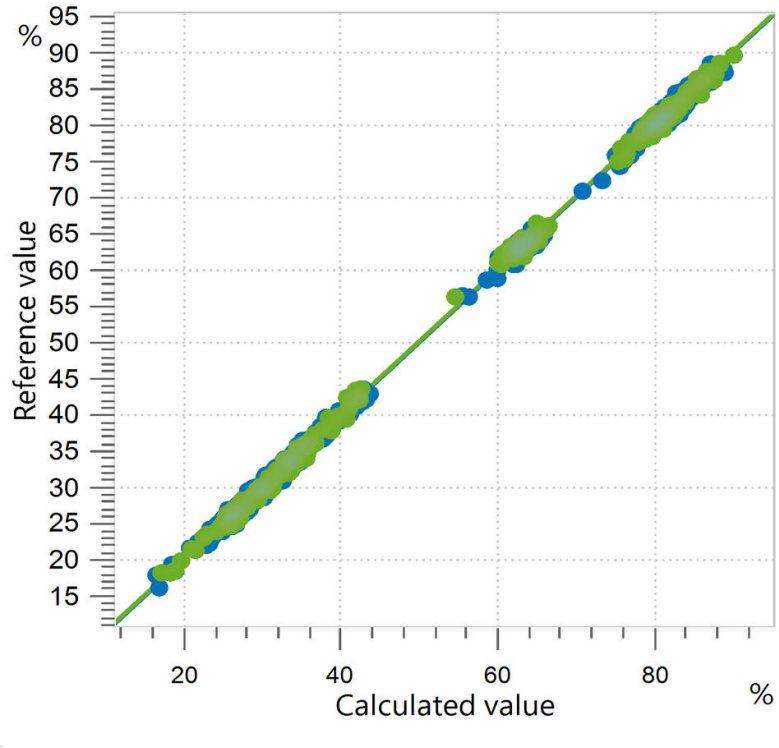

Figure 8. Correlation diagram and the respective figures of merit for the prediction of relative C18:1 fatty acid (oleic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
C18:1	0.64	0.67	0.71	0.999

RESULT C18:2 FATTY ACID CONTENT

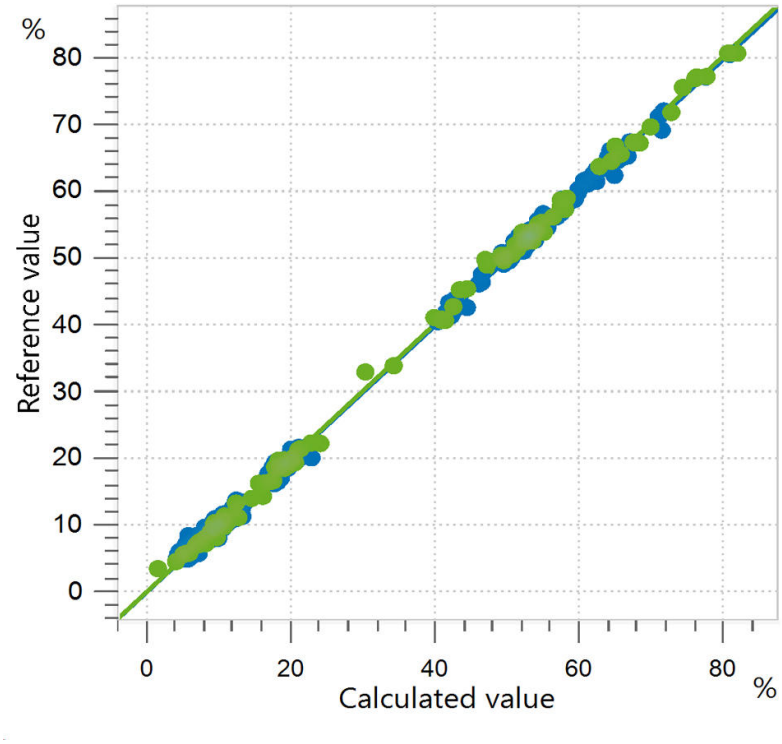

Figure 9. Correlation diagram and the respective figures of merit for the prediction of relative C18:2 fatty acid (linoleic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
C18:2	0.63	0.77	0.84	0.999

RESULT C18:3 FATTY ACID CONTENT

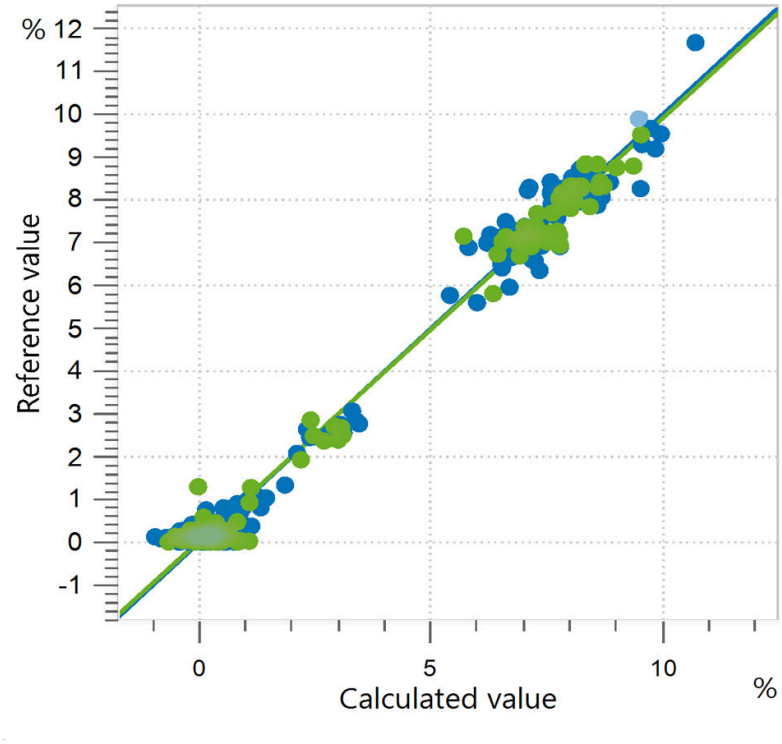

Figure 10. Correlation diagram and the respective figures of merit for the prediction of relative C18:3 fatty acid (alpha-linolenic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

Parameter	SEC (%)	SECV (%)	SEP (%)	R2CV
C18:3	0.32	0.36	0.36	0.989

CONCLUSION

This Application Note displays the benefits of using the OMNIS NIR Analyzer Liquid for routine analysis of several QC parameters in the laboratories of edible oil manufacturers. All quality parameters can be measured simultaneously in only a few seconds. Compared to other conventional methods, measurements performed with NIR spectroscopy do not need any sample preparation or solvents. This ultimately leads to a reduction in workload (Table 2) and related costs, as well as keeping lab personnel safer.

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 sample preparation (Methyl esterification + sample preparation) + 20 GC
FFA content	Titration	10 minutes per sample
Refractive index	Refractometer	2 minutes per sample
Fatty acid composition	Gas chromatography	30 minutes per sample

Internal reference: AW NIR CH-0074-042023

CONTACT

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CONFIGURATION

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)

OMNIS
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.

OMNIS
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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.