Application Note AN-NIR-115

Multiparameter quality control of palm oil with NIR spectroscopy

Obtain fast and reliable results without using any chemicals

Palm oil is currently the most widely produced and consumed vegetable oil globally and is used as a raw material in many industries. Crude palm oil (CPO) is used in various edible products, including cooking oil, margarine, and processed foods. CPO can be refined to remove impurities and improve color, flavor, and odor. Several steps are involved (e.g., degumming, neutralization, bleaching, deodorization) to produce refined bleached deodorized palm oil (RBDPO). RBDPO is used for frying purposes and is also found in foods like instant noodles and ice cream.

If the palm oil composition is found to be out of specification during production, the process stops, and the oil is recycled. Determination of key quality parameters like iodine value and the fatty acid profile of palm oil can be determined easily in just a few seconds without sample preparation using near-infrared spectroscopy (NIRS).

EXPERIMENTAL EQUIPMENT

In this application, 20 samples of crude palm oil (CPO) and 30 samples of refined, bleached, deodorized palm oil (RBDPO) were kept in a water bath at 60 °C for at least 30 minutes to liquify them. All samples were measured with an OMNIS NIR Analyzer Liquid in transmission mode at 60 °C using 8 mm disposable vials. Data acquisition and prediction model development were performed with OMNIS software.

Gas chromatography (GC) was used as a reference method after the methyl esterification of the fatty acids. The concentration of the fatty acids was derived from the corresponding peak area. Determination of iodine value in palm oil was possible by a calculation from the combined concentrations of oleic acid (18:1) and linoleic acid (18:2) according to the obtained fatty acid composition of palm oil.

Another set of CPO samples (681) was used to determine the water content. These samples were treated in the same manner as described above and measured using NIR spectroscopy. Karl Fischer (KF) titration was used as a standard method in this case.

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

Table 1. Hardware and software equipment overview.

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

RESULT

The measured NIR spectra of CPO (Figure 2) and of RBDPO (Figure 3) were used to create prediction models for the quantification of iodine value (IV), linoleic acid (18:2), oleic acid (18:1), and palmitic acid (16:0) for the two different palm oils. A quantification model was created for water content present in palm oil using another set of CPO samples.

The quality of the prediction models was evaluated using correlation diagrams (Figures 4–12) which display a high correlation between the NIR predictions and the results given by primary methods (i.e., GC and KF titration). The respective figures of merit (FOM) display the expected precision and confirm the feasibility during routine analysis.

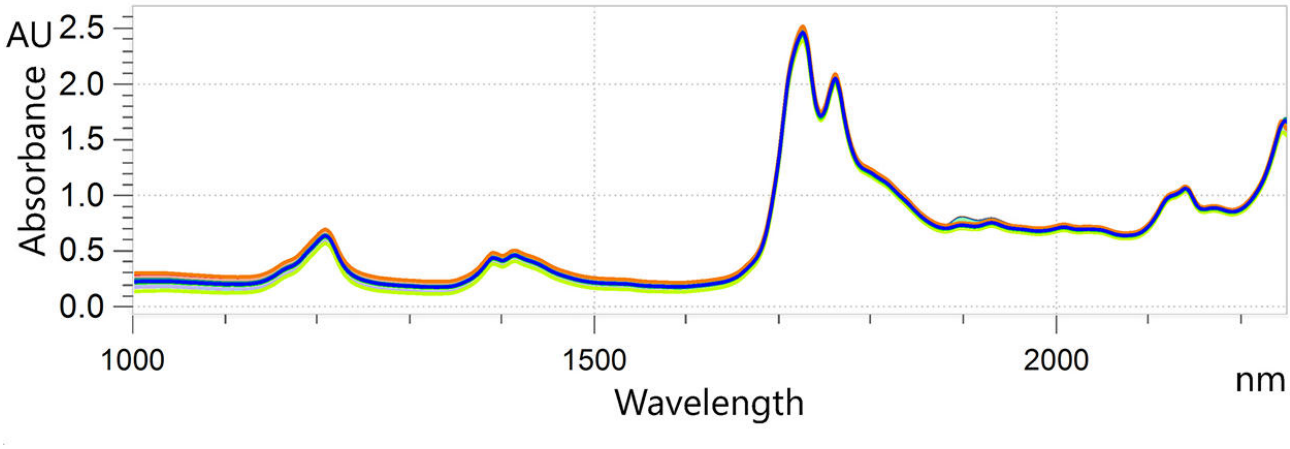

Figure 2. Overlay of NIR spectra from CPO samples analyzed on an OMNIS NIR Analyzer Liquid at 60 °C.

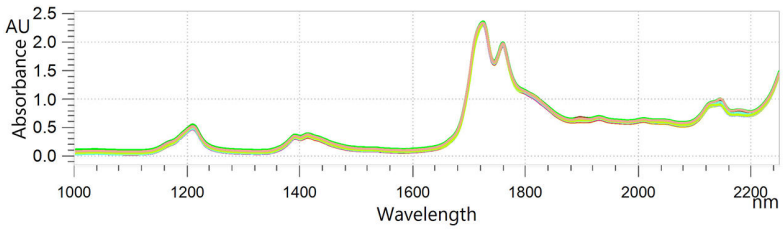

Figure 3. Overlaid NIR spectra of RBDPO samples analyzed on an OMNIS NIR Analyzer Liquid at 60 °C.

RESULT IV IN RBDPO

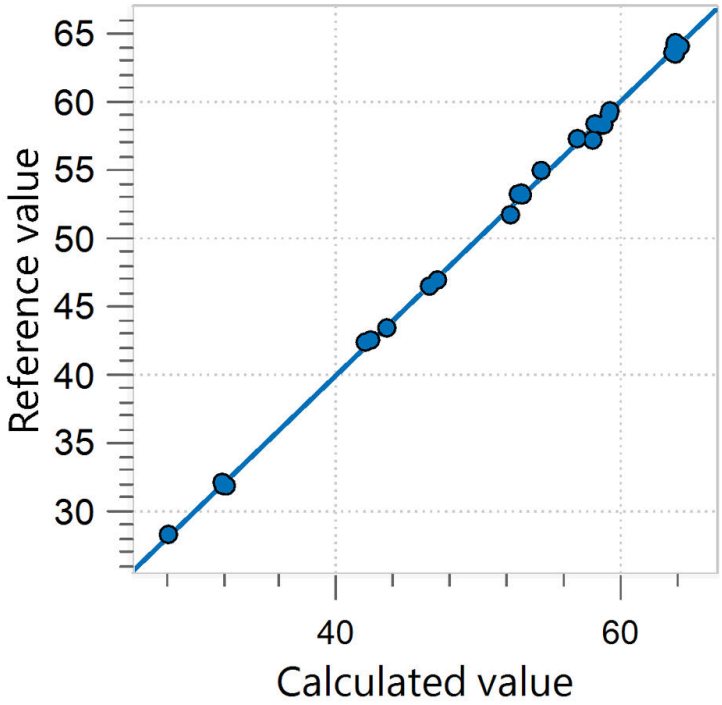

Figure 4. Correlation diagram and the respective figures of merit for the prediction of iodine value in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC(mg/100g)	SECV(mg/100g)	R2CV
IV	0.31	0.34	0.999

RESULT PALMITIC ACID (16:0) IN RBDPO

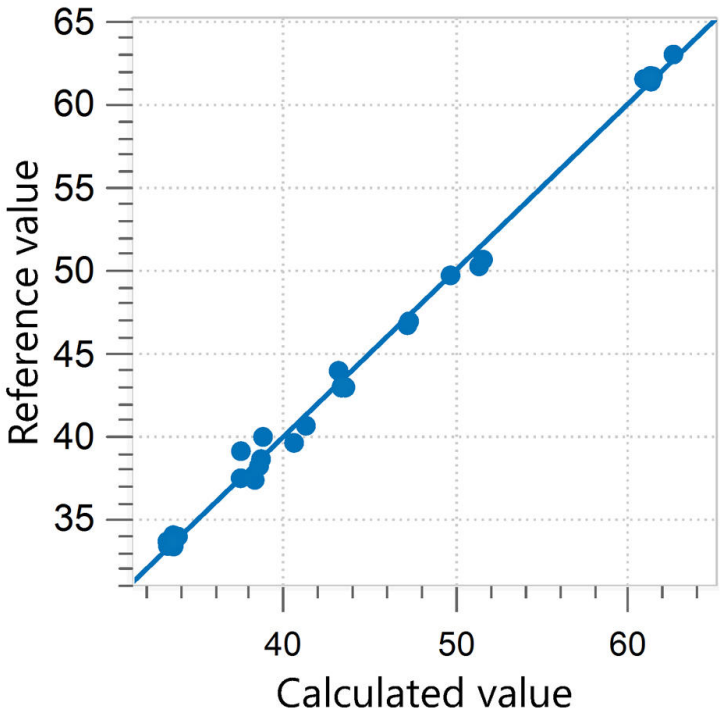

Figure 5. Correlation diagram and the respective figures of merit for the prediction of palmitic acid (16:0) in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC (%)	SECV (%)	R2CV
16:0	0.53	0.63	0.996

RESULT OLEIC ACID (18:1) IN RBDPO

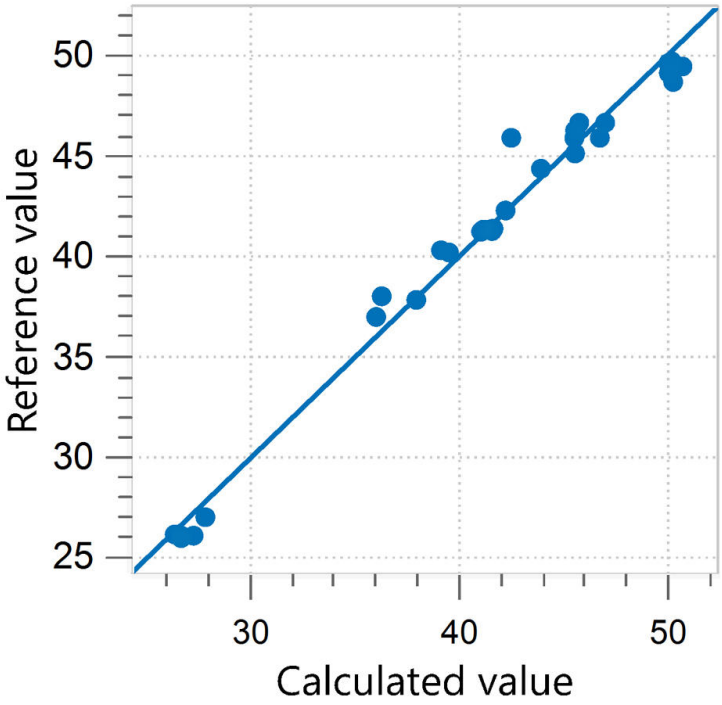

Figure 6. Correlation diagram and the respective figures of merit for the prediction of oleic acid (18:1) in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC (%)	SECV (%)	R2CV
18:1	0.75	0.98	0.985

RESULT LINOLEIC ACID (18:2) IN RBDPO

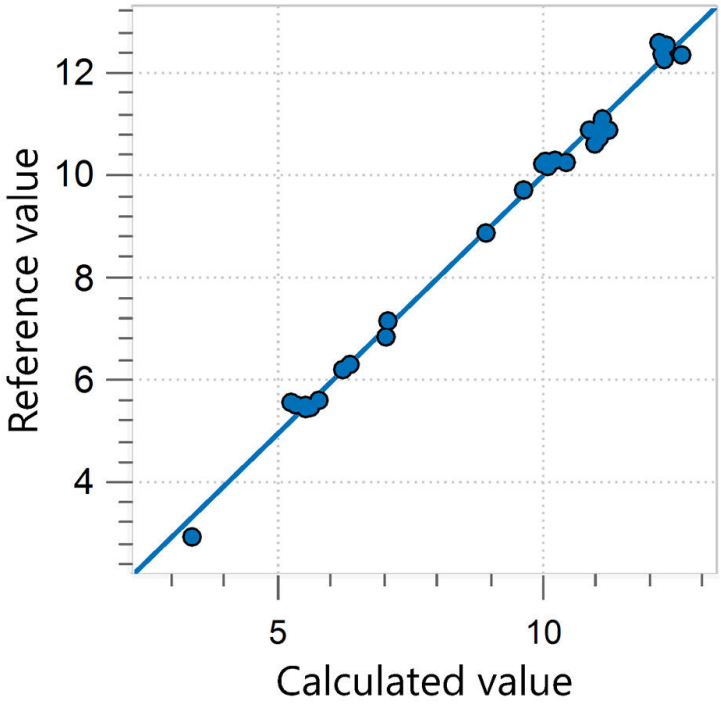

Figure 7. Correlation diagram and the respective figures of merit for the prediction of linoleic acid (18:2) in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC (%)	SECV (%)	R2CV
18:2	0.14	0.21	0.994

RESULT IV IN CPO

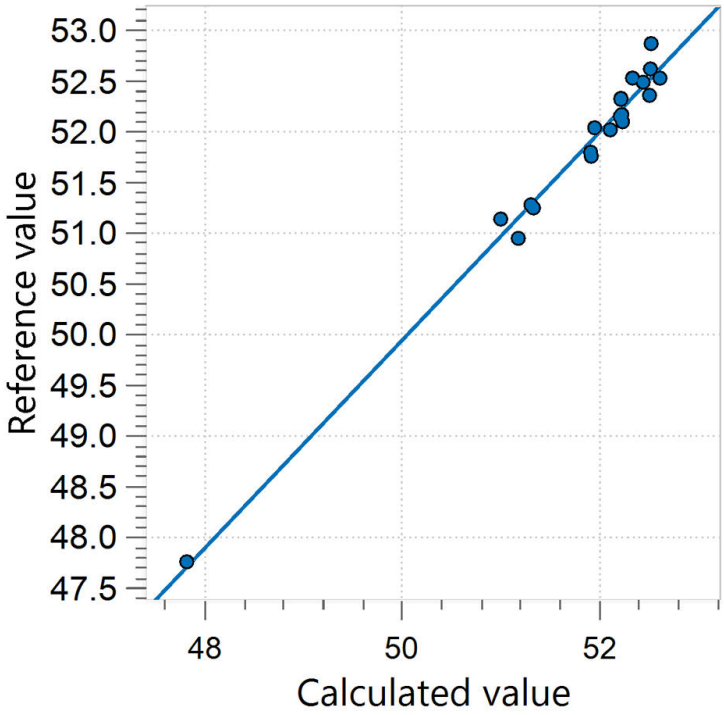

Figure 8. Correlation diagram and the respective figures of merit for the prediction of iodine value in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC(mg/100g)	SECV(mg/100g)	R2CV
IV	0.11	0.14	0.984

RESULT PALMITIC ACID (16:0) IN CPO

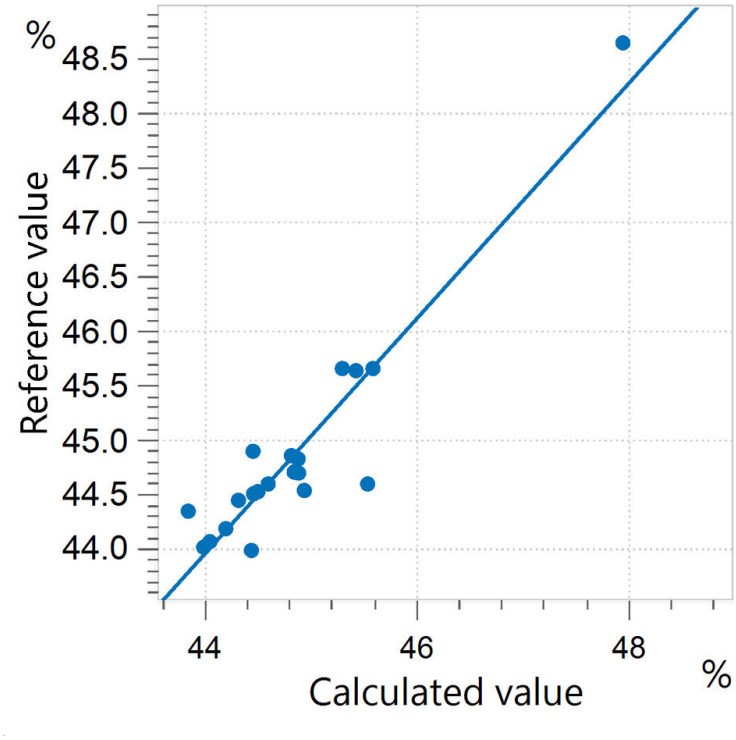

Figure 9. Correlation diagram and the respective figures of merit for the prediction of palmitic acid (16:0) in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC(%)	SECV(%)	R2CV
16:0	0.11	0.14	0.984

RESULT OLEIC ACID (18:1) IN CPO

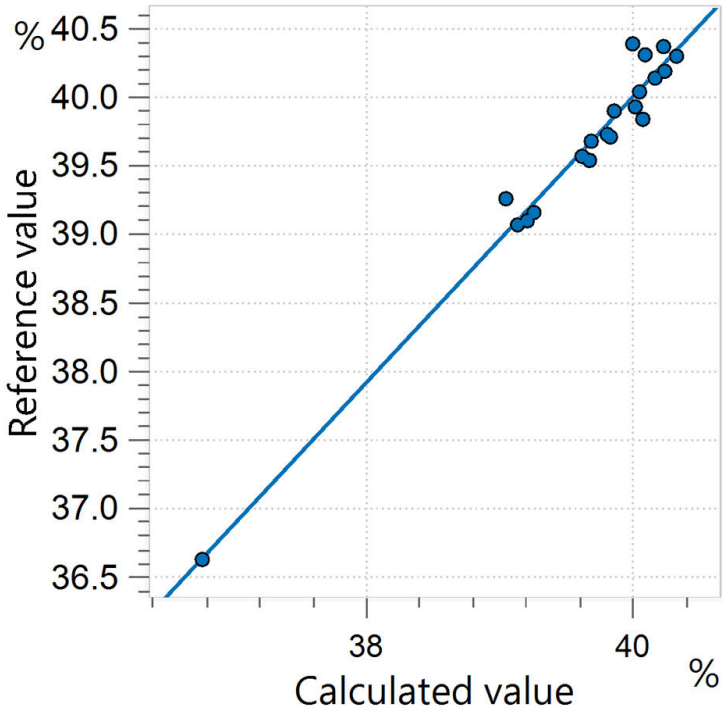

Figure 10. Correlation diagram and the respective figures of merit for the prediction of oleic acid (18:1) in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC(%)	SECV(%)	R2CV
18:1	0.12	0.14	0.969

RESULT LINOLEIC ACID (18:2) IN CPO

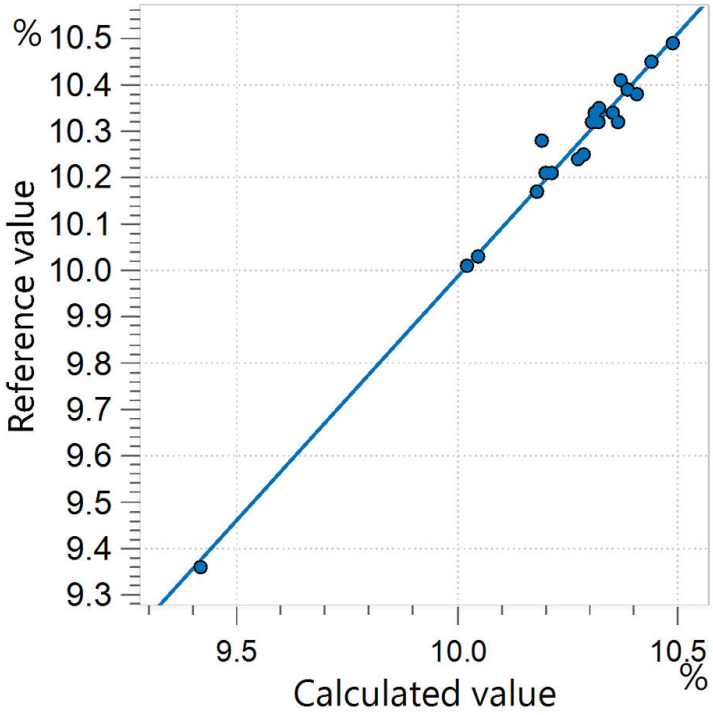

Figure 11. Correlation diagram and the respective figures of merit for the prediction of linoleic acid (18:2) in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

Parameter	SEC(%)	SECV(%)	R2CV
18:2	0.02	0.03	0.984

RESULT WATER CONTENT IN CPO

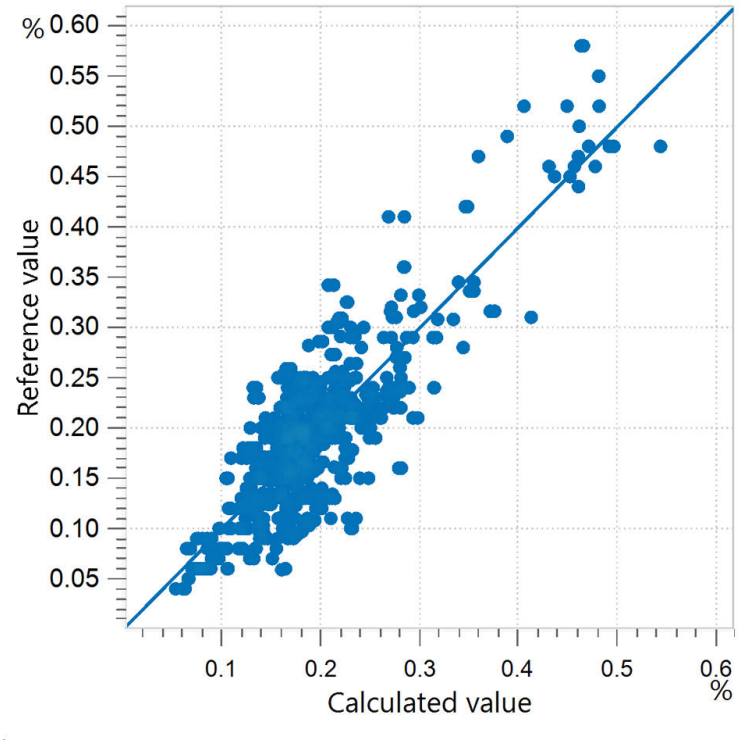

Figure 12. Correlation diagram and the respective figures of merit for the prediction of water content in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using KF titration.

Parameter	SEC(%)	SECV(%)	R2CV
Water	0.044	0.044	0.714

CONCLUSION

This Application Note displays the benefit of using the OMNIS NIR Analyzer Liquid for routine analysis in laboratories that measure various palm oil quality parameters. Compared to other conventional methods like Karl Fischer titration and GC, determinations with NIR spectroscopy do not require any sample preparation or chemical reagents. This ultimately leads to a reduction in workload (Table 2) and costs.

Table 2. Time to result overview for the parameters of iodine value, fatty acid composition, and water content in palm oil by standard methods.

Parameter	Method	Time to result
Iodine value, Fatty acid composition	Gas chromatography	30 min sample preparation (Methyl esterification + sample preparation) + 20 min GC
Water content	KF titration	10 min per sample

Internal reference: AW NIR CH-0066-042023

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.