



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.

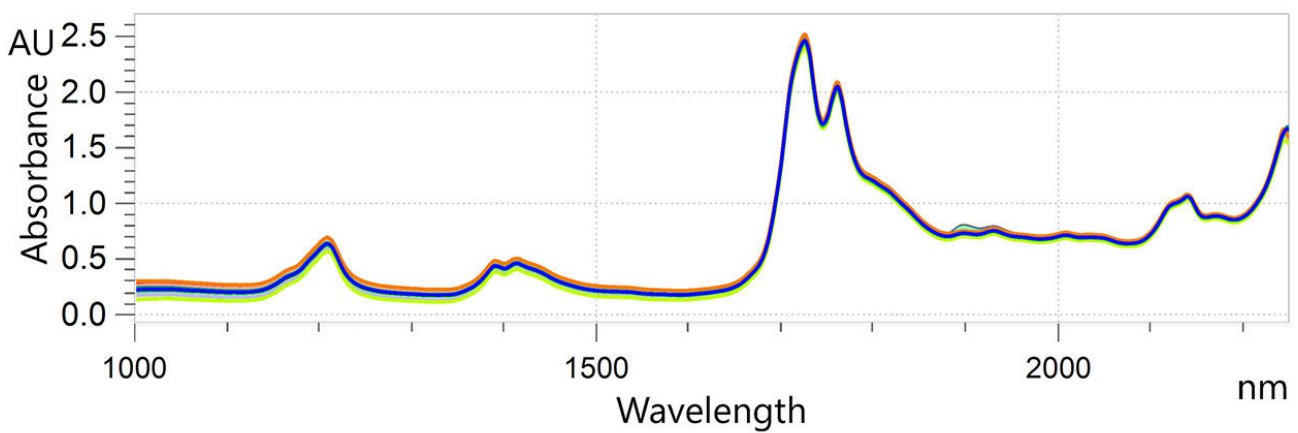


Figure 2. Overlay of NIR spectra from CPO samples analyzed on an OMNIS NIR Analyzer Liquid at 60 °C.

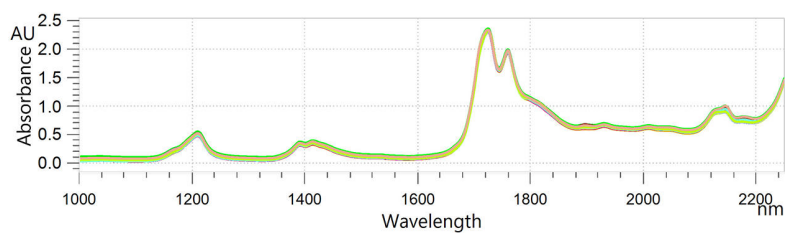


Figure 3. Overlaid NIR spectra of RBDPO samples analyzed on an OMNIS NIR Analyzer Liquid at 60 °C.

RESULT IV IN RBDPO

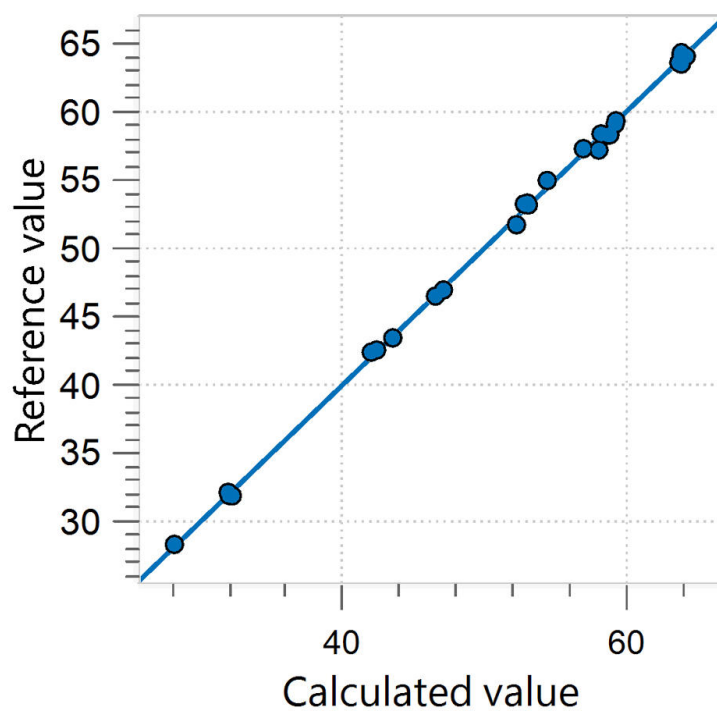


Figure 4. Correlation diagram and the respective figures of merit for the prediction of iodine value in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

RESULT PALMITIC ACID (16:0) IN RBDPO

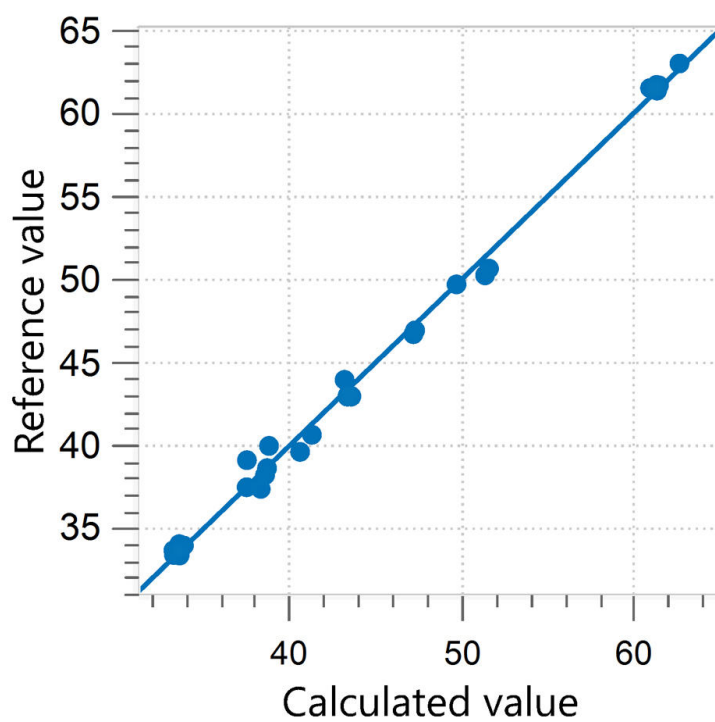


Figure 5. Correlation diagram and the respective figures of merit for the prediction of palmitic acid (16:0) in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

RESULT OLEIC ACID (18:1) IN RBDPO

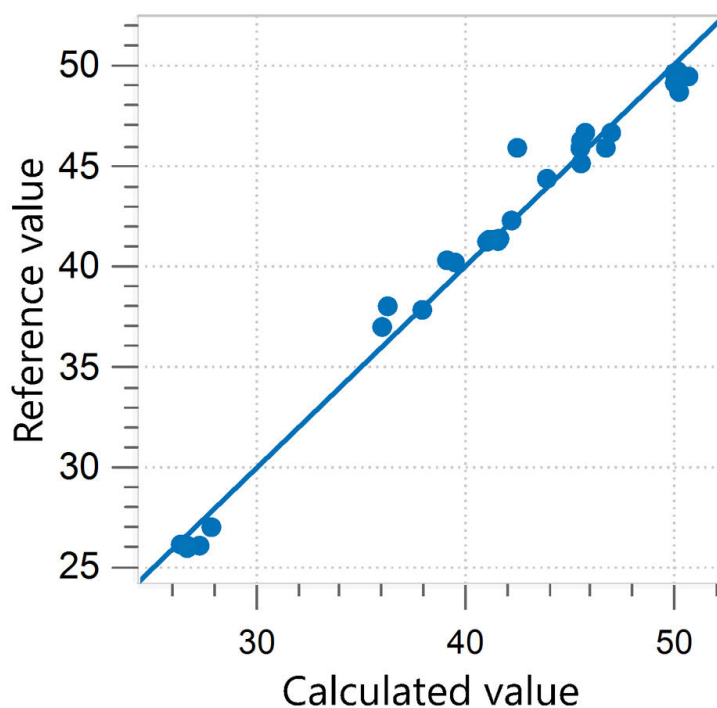


Figure 6. Correlation diagram and the respective figures of merit for the prediction of oleic acid (18:1) in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

RESULT LINOLEIC ACID (18:2) IN RBDPO

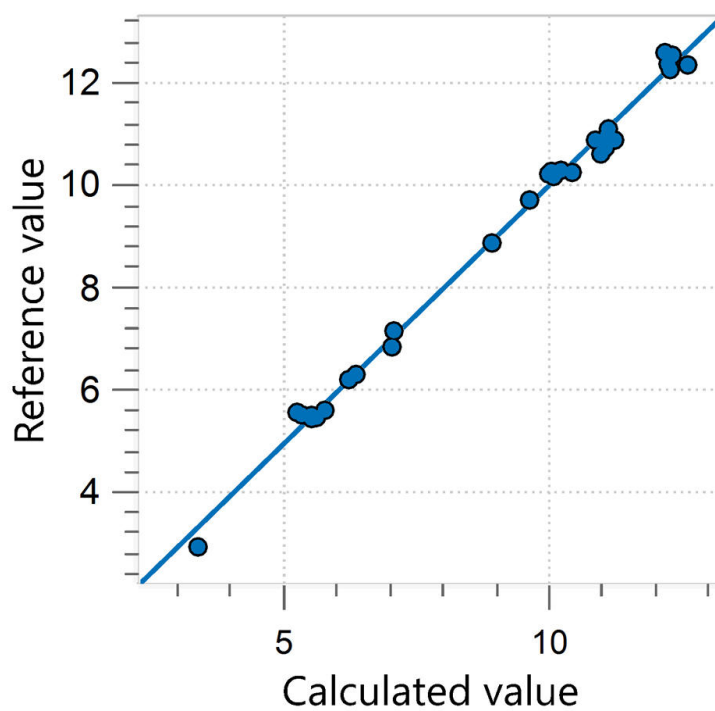


Figure 7. Correlation diagram and the respective figures of merit for the prediction of linoleic acid (18:2) in RBDPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

RESULT IV IN CPO

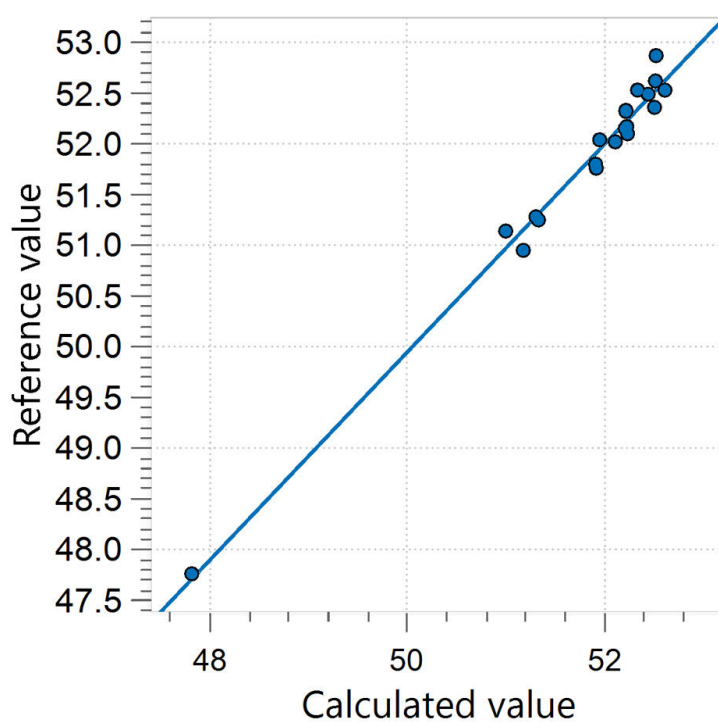


Figure 8. Correlation diagram and the respective figures of merit for the prediction of iodine value in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

RESULT PALMITIC ACID (16:0) IN CPO

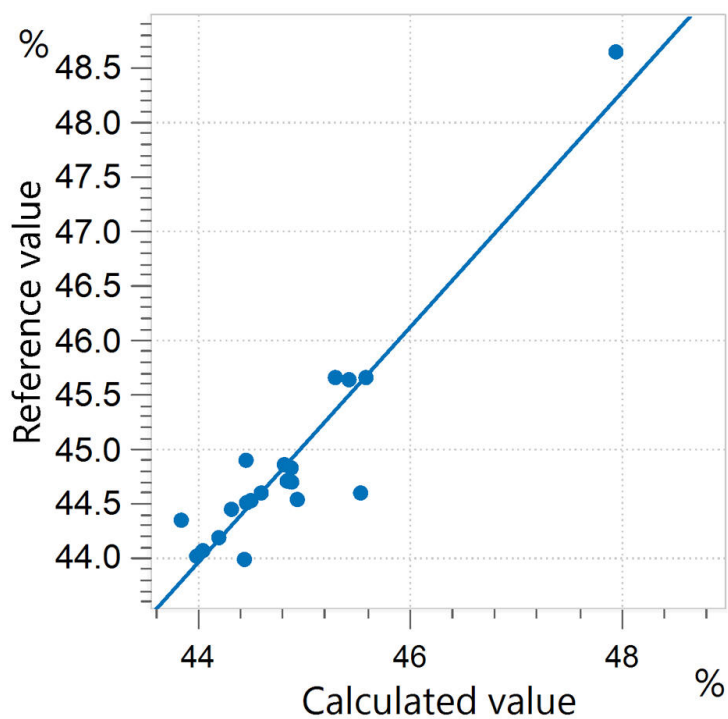


Figure 9. Correlation diagram and the respective figures of merit for the prediction of palmitic acid (16:0) in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

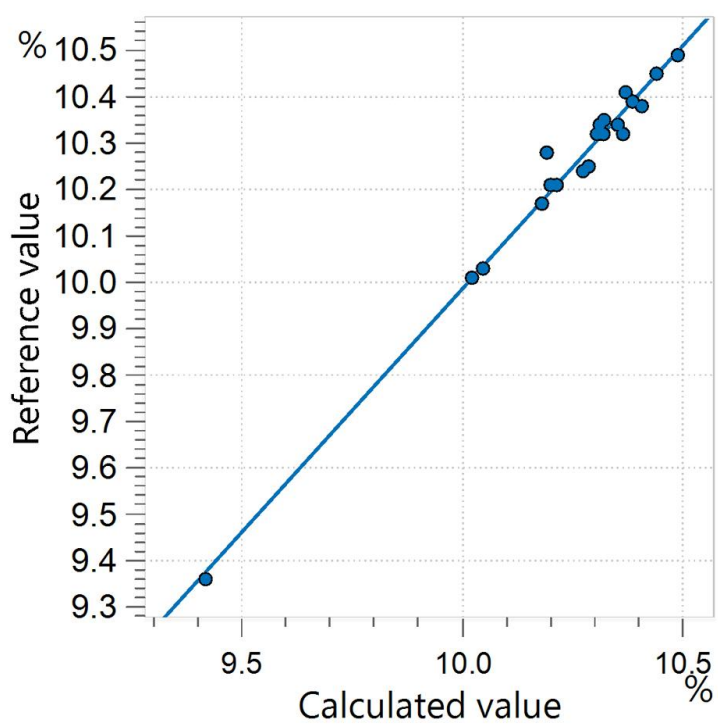


Figure 11. Correlation diagram and the respective figures of merit for the prediction of linoleic acid (18:2) in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using GC.

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

RESULT WATER CONTENT IN CPO

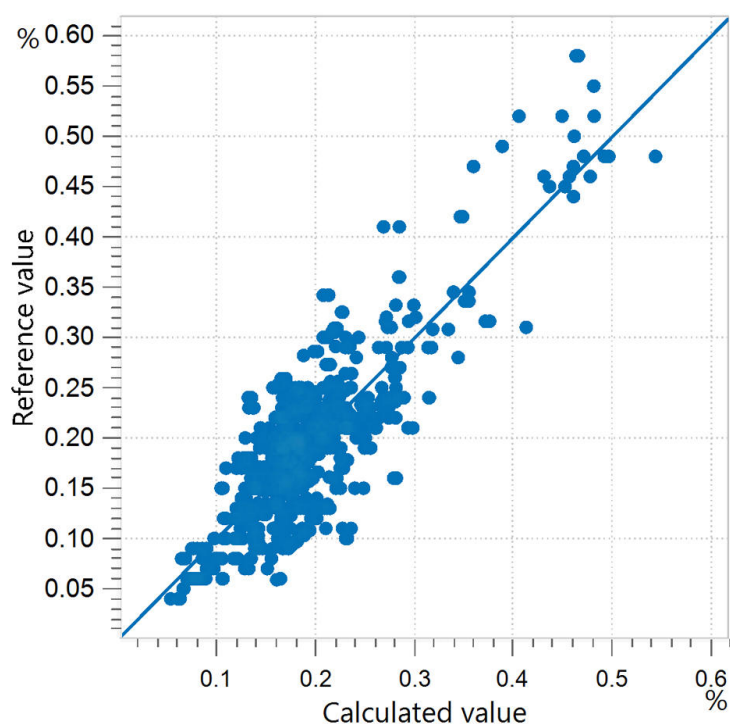


Figure 12. Correlation diagram and the respective figures of merit for the prediction of water content in CPO using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using KF titration.

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

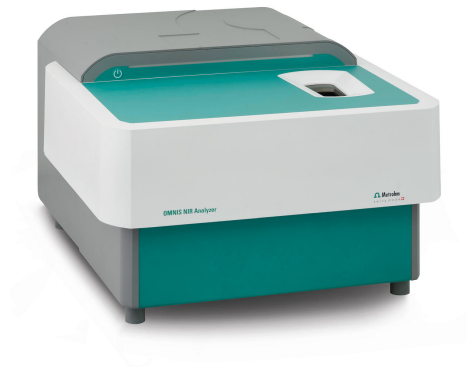
Internal reference: AW NIR CH-0066-042023

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CONFIGURATION



OMNIS NIR Analyzer Liquid

Spektromètre proche infrarouge pour échantillons liquides.

L'OMNIS NIR Analyzer est la solution de spectroscopie proche infrarouge (NIRS) développée et produite selon les normes de qualité suisses pour les analyses de routine tout au long de la chaîne de fabrication. L'utilisation des technologies les plus récentes et l'intégration dans le logiciel OMNIS moderne se reflètent dans la vitesse, la facilité d'utilisation et la flexibilité d'utilisation de ces spectromètres NIR.

Vue d'ensemble des avantages de l'OMNIS NIR Analyzer Liquid :

- Mesures d'échantillons liquides en moins de 10 secondes
- Contrôle de la température sur l'échantillon de 25 °C à 80 °C
- Détection automatique de l'insertion et du retrait d'échantillons du récipient d'échantillon
- Intégration simple dans un système d'automatisation ou liaison avec d'autres technologies d'analyse (titrage)
- Prise en charge de nombreux récipients d'échantillon de différentes longueurs de chemin

Support OMNIS NIR, flacon, 8 mm

Support de flacon pour l'OMNIS NIR Analyzer pour flacons à usage unique de 8 mm (6.7402.240).





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A WHOLE NEW LEVEL OF PERFORMANCE

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A WHOLE NEW LEVEL OF PERFORMANCE

Flacon à usage unique, 8 mm, transmission, Qté 100
100 flacons en verre (borosilicate) à usage unique avec une longueur de chemin de 8 mm pour des analyses de liquides lors d'une transmission. Les flacons à usage unique sont fournis avec les bouchons de fermeture correspondants (quantité = 100).

Compatible avec :

- Support OMNIS NIR, flacon, 8 mm (6.07401.070)
- Support DS2500 pour flacons à usage unique 8 mm (6.7492.020)

Licence OMNIS autonome

Elle permet l'exploitation autonome du logiciel OMNIS sur un ordinateur Windows™.

Caractéristiques :

- La licence comprend déjà une licence pour appareils OMNIS.
- Elle doit être activée via le portail d'octroi de licences Metrohm.
- Elle ne peut pas être transférée sur un autre ordinateur.

Licence logicielle Quant Development

Licence logicielle pour la création et l'édition de modèles de quantification dans une installation du logiciel OMNIS Stand-Alone.