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

RESULT

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).

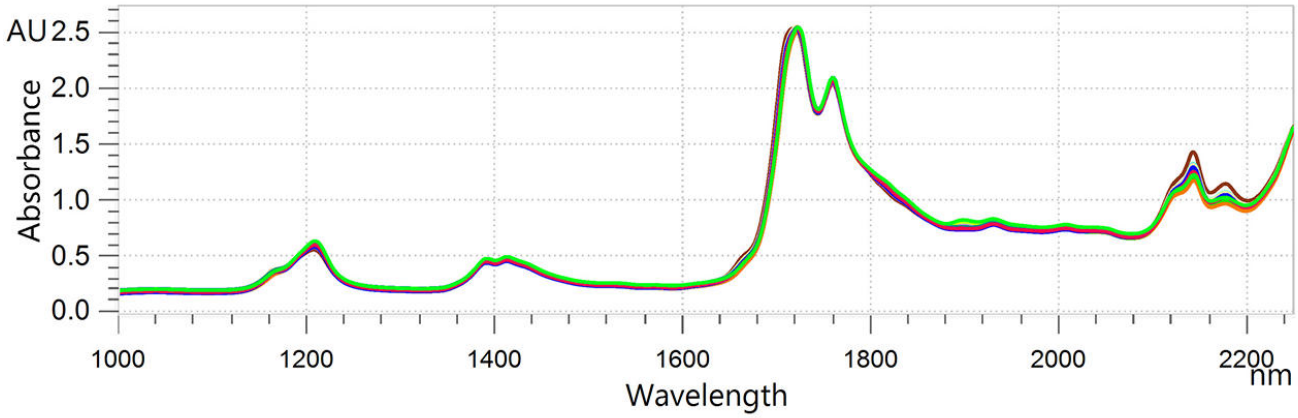

Figure 2. Overlaid NIR spectra of edible oil samples which were analyzed on an OMNIS NIR Analyzer Liquid at 40 °C with 8 mm vials.

RESULT IODINE VALUE

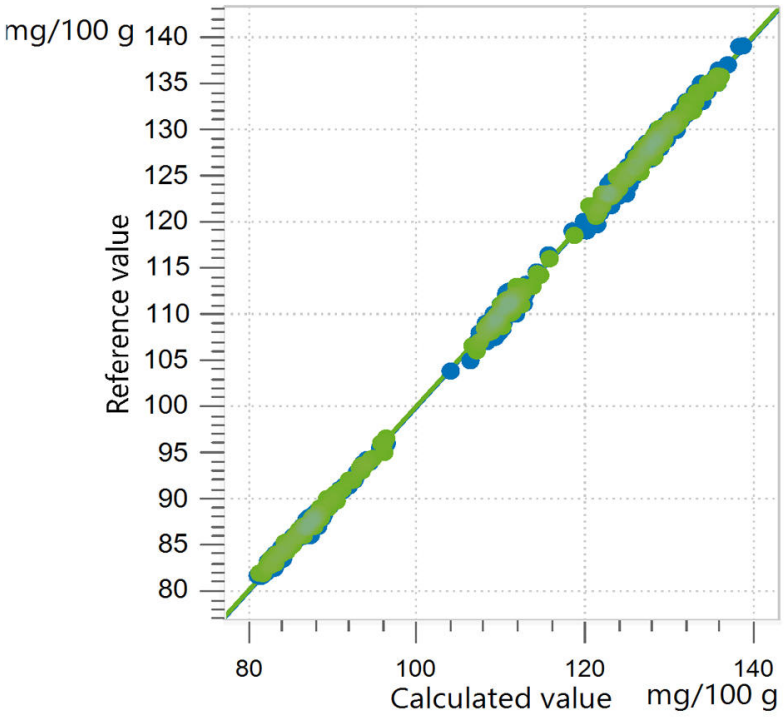

Figure 3. Correlation diagram and the respective figures of merit for the prediction of iodine value (also known as iodine number) in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

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

RESULT FREE FATTY ACIDS

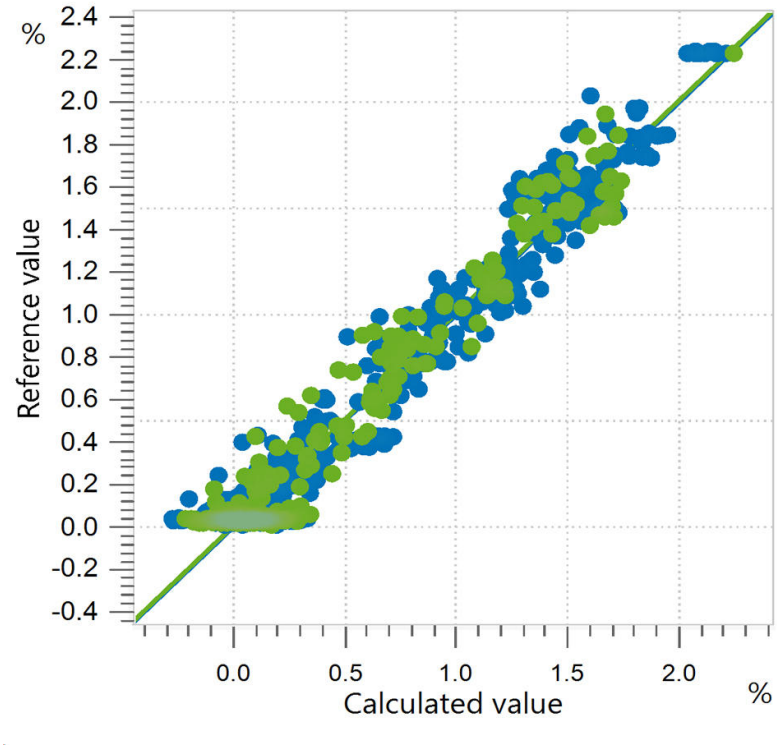

Figure 4. Correlation diagram and the respective figures of merit for the prediction of FFA in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using a titration method.

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

RESULT REFRACTIVE INDEX

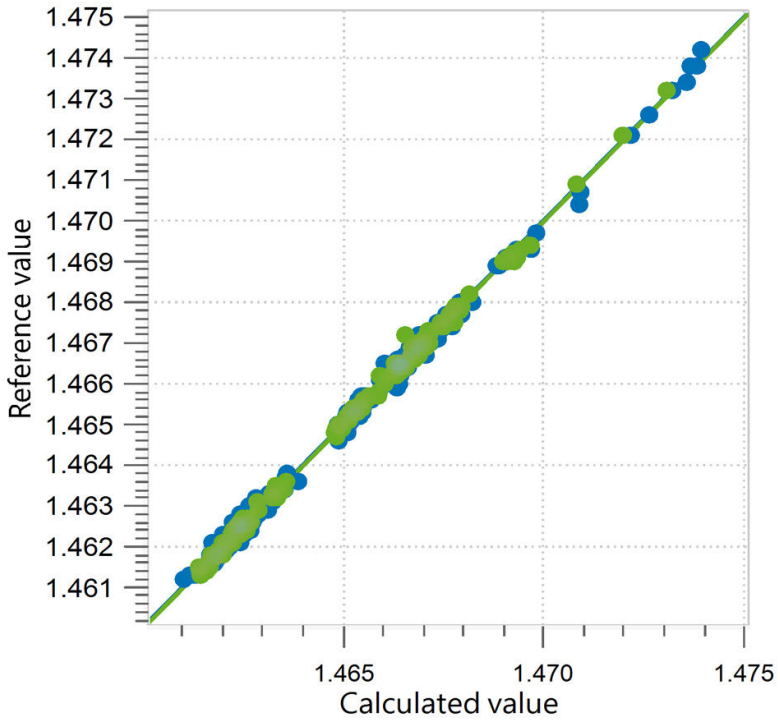

Figure 5. Correlation diagram and the respective figures of merit for the prediction of refractive index (RI) in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using a refractometer.

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

RESULT C16:0 FATTY ACID CONTENT

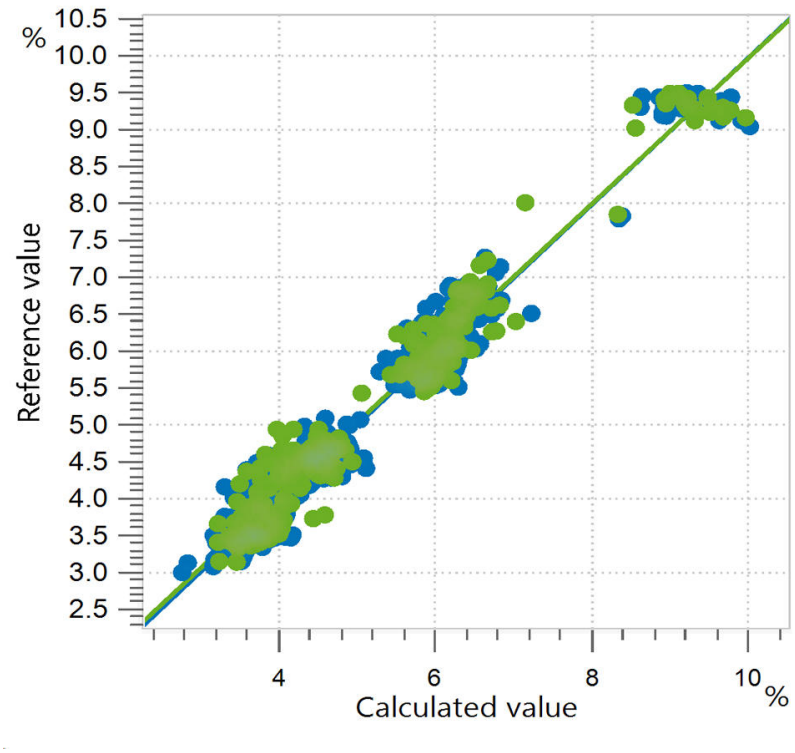

Figure 6. Correlation diagram and the respective figures of merit for the prediction of relative C16:0 fatty acid (palmitic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

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

RESULT C18:0 FATTY ACID CONTENT

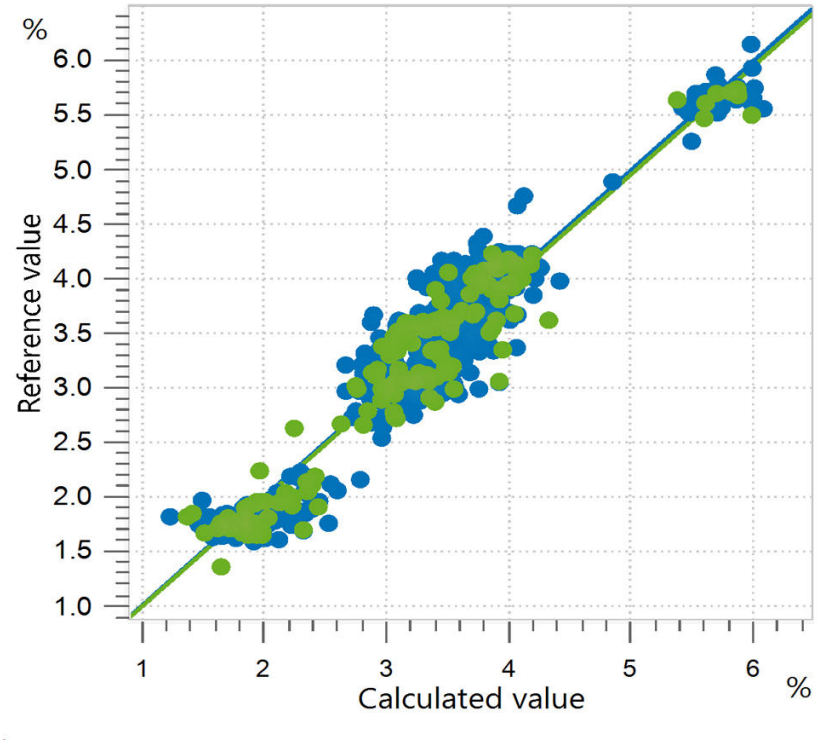

Figure 7. Correlation diagram and the respective figures of merit for the prediction of C18:0 fatty acid (stearic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

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

RESULT C18:1 FATTY ACID CONTENT

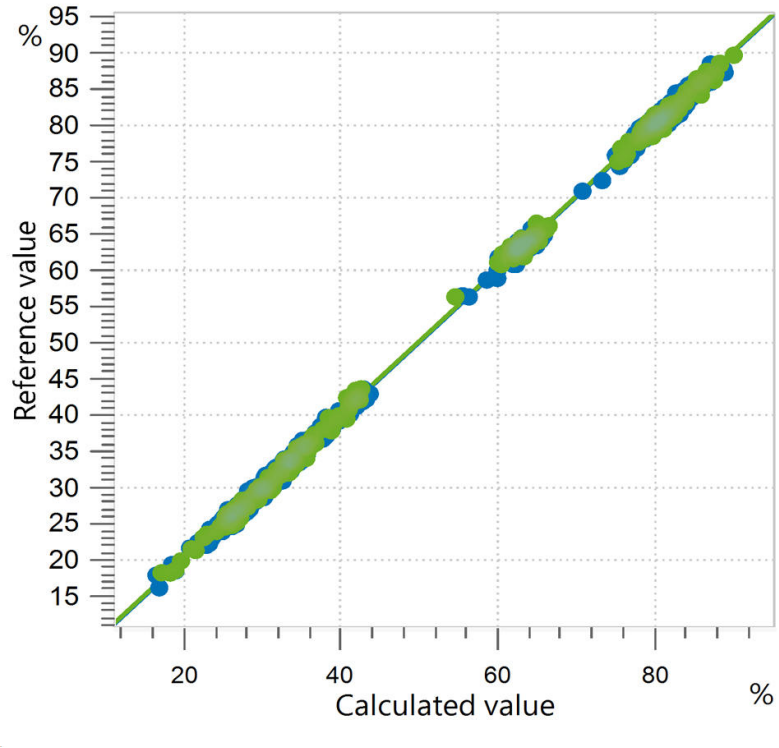

Figure 8. Correlation diagram and the respective figures of merit for the prediction of relative C18:1 fatty acid (oleic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

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

RESULT C18:2 FATTY ACID CONTENT

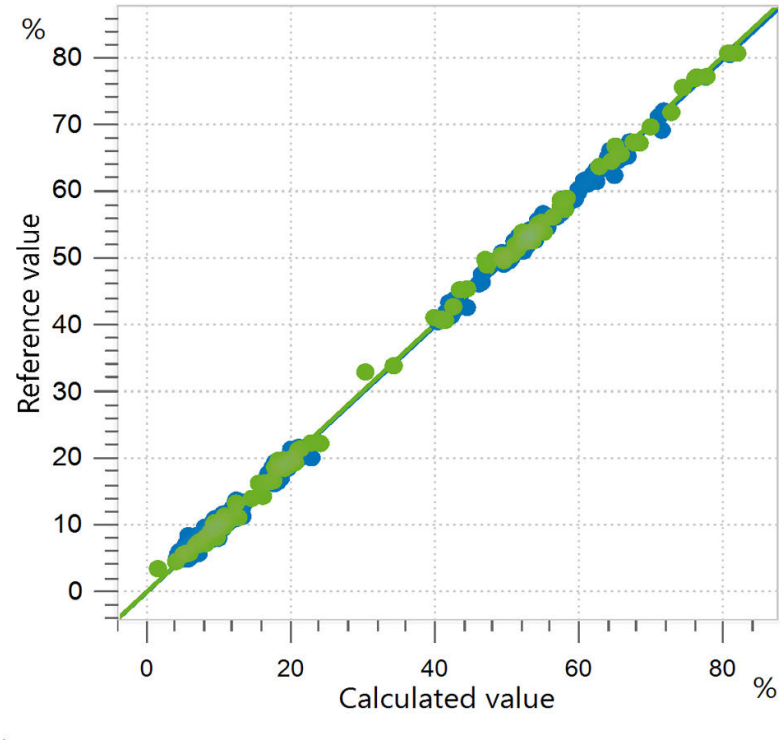

Figure 9. Correlation diagram and the respective figures of merit for the prediction of relative C18:2 fatty acid (linoleic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

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

RESULT C18:3 FATTY ACID CONTENT

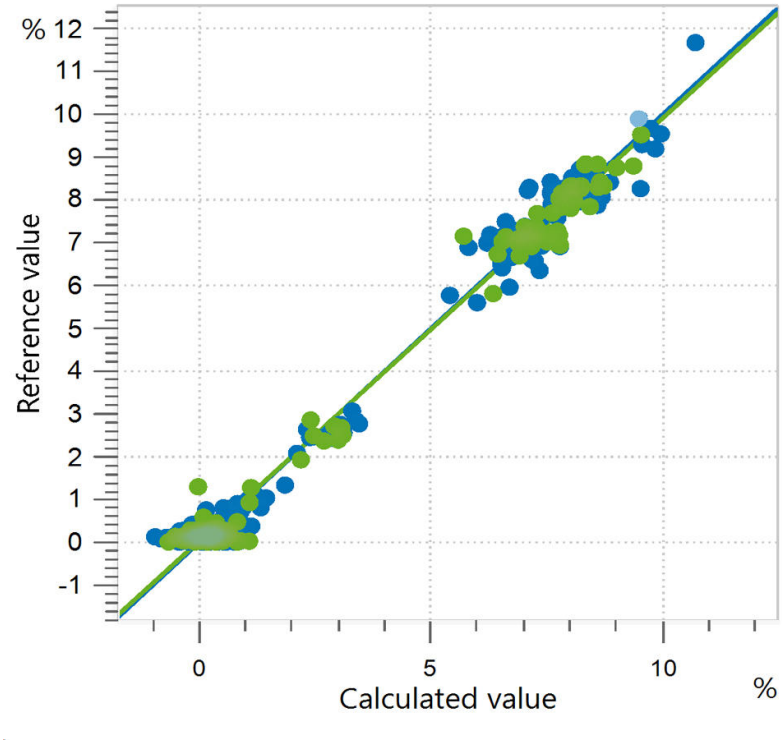

Figure 10. Correlation diagram and the respective figures of merit for the prediction of relative C18:3 fatty acid (alpha-linolenic acid) content in edible oils using an OMNIS NIR Analyzer Liquid. The reference values were evaluated using gas chromatography.

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

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CONFIGURATION

OMNIS NIR Analyzer Liquid

Spectromètre 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 chaine 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
- Controle 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).

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)

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Licence OMNIS autonome

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Licence logicielle Quant Development

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