

Application Note AN-NIR-150

Monitoring the production of stearic acid and its salts with NIR spectroscopy

Acid number, iodine value, granulometry, moisture, and more

Stearic acid and its derivatives (e.g., magnesium, calcium, and zinc stearates) are widely used in the lubricants, pharmaceutical, cosmetics, plastics, and food industries. Producing these materials involves several steps: clarification, hydrogenation, cleavage, and distillation, followed by converting stearic acid into its respective stearates. This Application Note demonstrates how the OMNIS NIR Analyzer can be used to monitor the stearic acid iodine value, acid value, moisture, ash content, melting point, and granulometry during the production process and in the final product. Near-infrared (NIR) spectroscopy is a rapid, nondestructive analytical technique that is fast becoming a preferred method over other slower, more costly alternatives.

EXPERIMENTAL EQUIPMENT

Quality control during stearic acid production is essential to guarantee consistency and compliance with industry standards. Each stage of the process, from initial raw material preparation to the final conversion into stearates, requires analytical checks. The samples used in this study include:

Pre-entry (raw material):
Acid number/iodine value – 44/40 samples
Hydrogenation:
Acid number – 17 samples
Cleavage:
Acid number – 99 samples
Mg, Ca, and Zn stearates:
Ash, granulometry, melting point, and moisture – 69 samples

During stearic acid production, samples occur in two forms depending on the process stage. Early steps involve liquid samples (a viscous mixture of saturated fatty acids and/or animal fat). After purification, the product solidifies into flakes, powder, or pellets, representing the final stearic acids and their salts. Since quality control is required at different stages of production, both liquid and solid sample types can be measured with the OMNIS NIR Analyzer Liquid/Solid (Figure 1). This allows an easy transition from monitoring intermediate products to performing quality control of final stearic acids.



Figure 1. OMNIS NIR Analyzer Liquid/Solid.

The OMNIS Software (Table 1) was used for all data acquisition, and prediction model development was done with the help of the OMD (OMNIS Model Developer).

Liquid samples (pre-entry, hydrogenation, cleavage):
- Each sample was measured in transmission mode using an 8 mm disposable vial.
- Spectra were collected at 75 °C using the vessel temperature control mode.

Solid samples (Mg, Ca, and Zn stearates):
- Each sample was measured by reflectance mode in a 28 mm disposable vial.

Table 1. Hardware and software equipment overview.

Equipment	Article Number
OMNIS NIR Analyzer Liquid/Solid	2.1072.0010
Disposable vials, 28 mm, reflection	6.7402.140
Flexible holder OMNIS NIR	6.07402.300
Disposable vial, 8 mm, transmission	6.7402.240
Holder OMNIS NIR, vial, 8 mm	6.07401.070
OMNIS Stand-Alone license	6.06003.010
Software license Quant Development	6.06008.002

RESULT

NIR spectroscopy was successfully applied to monitor both process samples and final stearate products. Figure 2 shows NIR spectra collected from cleavage samples, illustrating the spectral information used for model development. Quantitative models were developed for stearic acid's acid value, iodine value, moisture, ash content, melting point, and granulometry. The reference values used were obtained using the routine quality control methods employed during stearic acid and stearate production. Figures 3–9 present the correlation plots and corresponding figures of merit (FOMs) for each parameter. The data points along the regression line indicate good correlation between the NIR model and the reference method. The FOMs provide additional information about model performance, including calibration accuracy and robustness.

Overall, the developed models demonstrated good correlation with the laboratory reference values, indicating that the selected NIR spectral range contains sufficient information for reliable quality control throughout the production process.

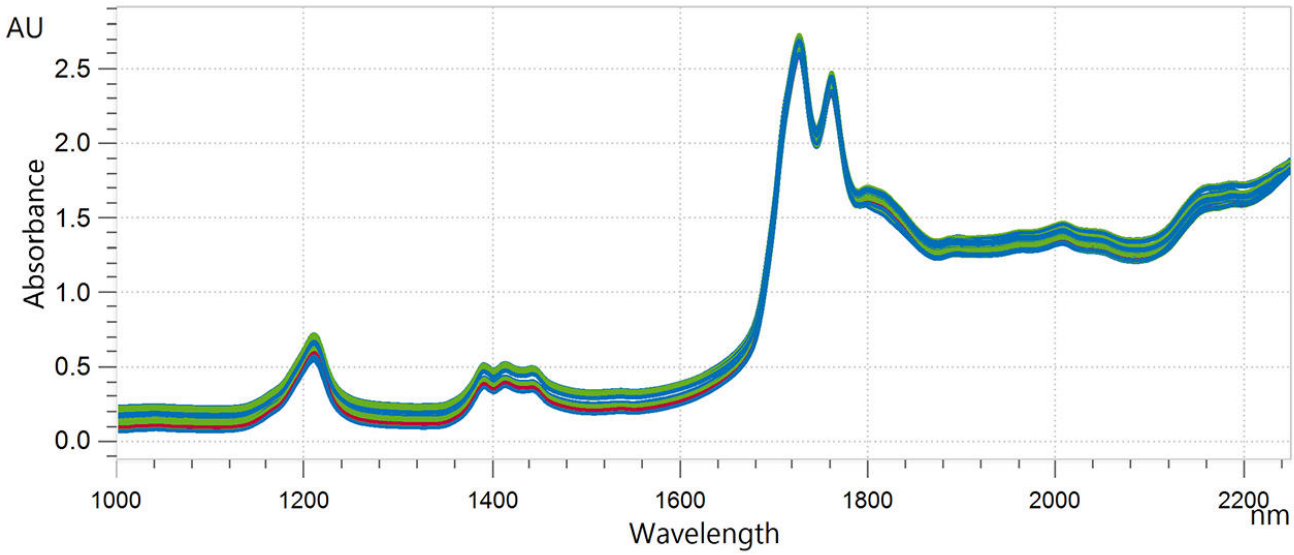


Figure 2. NIR spectra of the stearate cleavage samples collected by transmission mode in an 8 mm vial at 75 °C. These spectra were collected with the OMNIS NIR Analyzer in the liquid sample configuration.

RESULT ACID NUMBER IN PRE-ENTRY SAMPLES

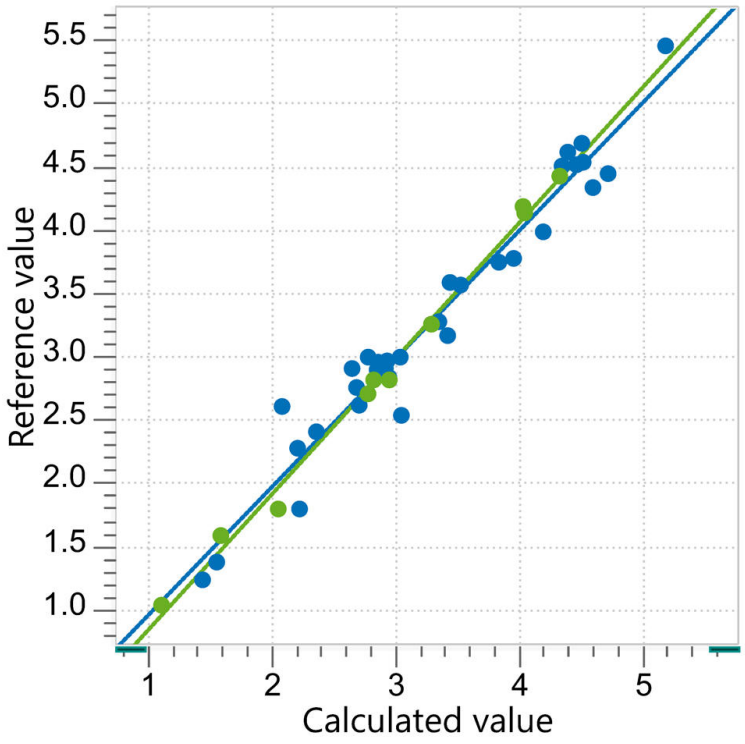


Figure 3. Correlation diagram and the respective FOMs for the prediction of acid number in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP

0.994	0.16 mg KOH/g	0.21 mg KOH/g	0.12 mg KOH/g

RESULT IODINE VALUE IN PRE-ENTRY SAMPLES

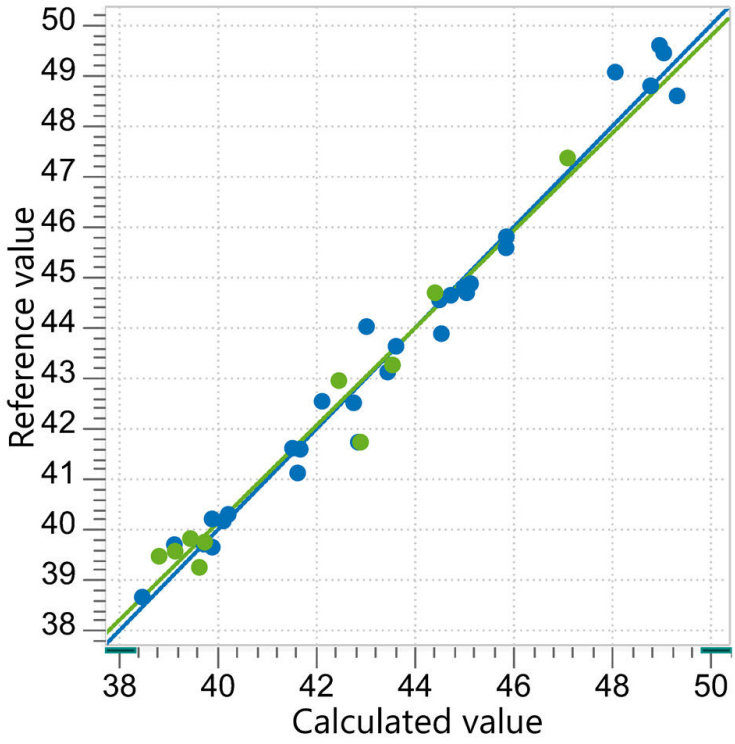


Figure 4. Correlation diagram and the respective FOMs for the prediction of iodine value in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP
0.962	0.40 g I_2/100 g	0.46 g I_2/100 g	0.52 g I_2/100 g

RESULT ACID NUMBER IN THE HYDROGENATION PROCESS

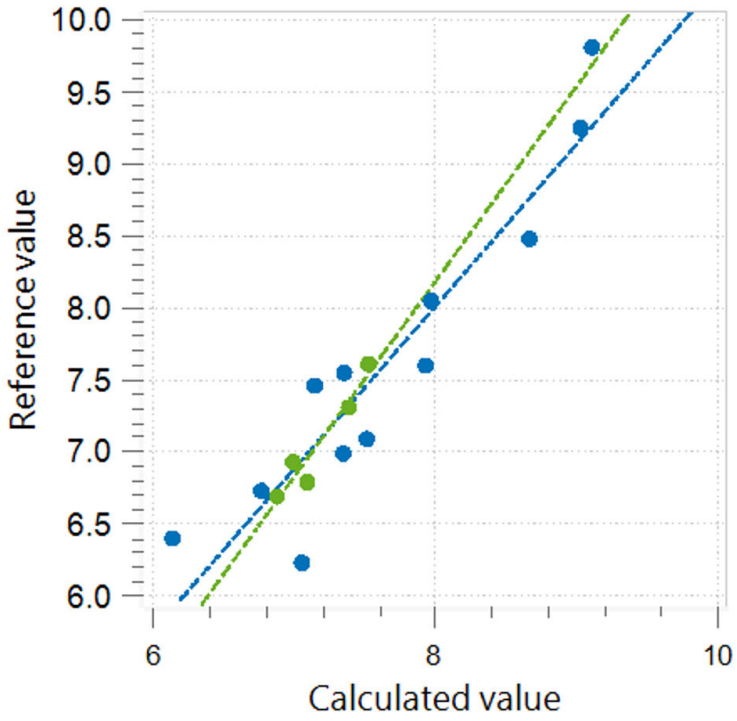


Figure 5. Correlation diagram and the respective FOMs for the prediction of acid number in the stearate hydrogenation process with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP
0.921	0.11 mg KOH/g	0.40 mg KOH/g	0.17 mg KOH/g

RESULT ASH IN STEARATES

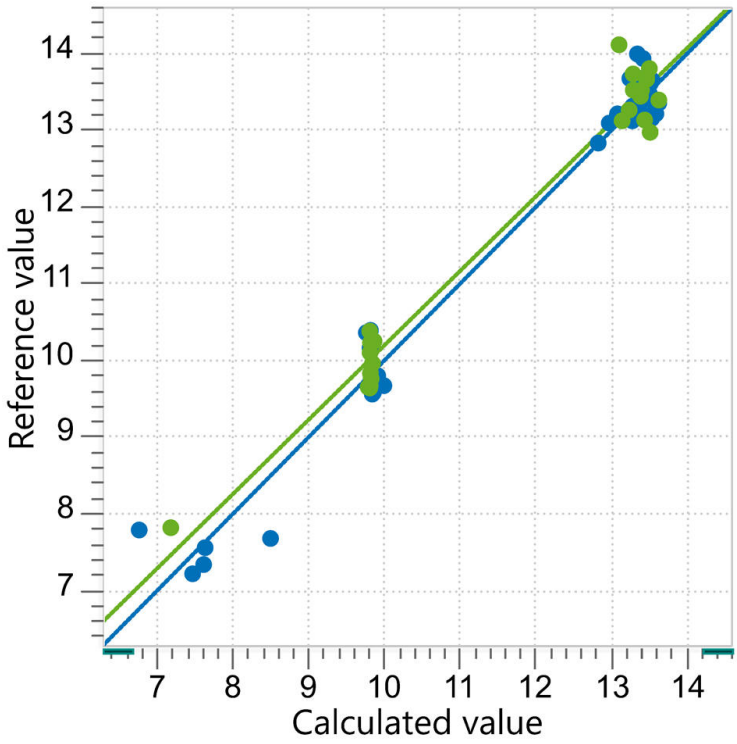


Figure 6. Correlation diagram and the respective FOMs for the prediction of ash content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP
0.971	0.29%	0.33%	0.36%

RESULT MOISTURE IN STEARATES

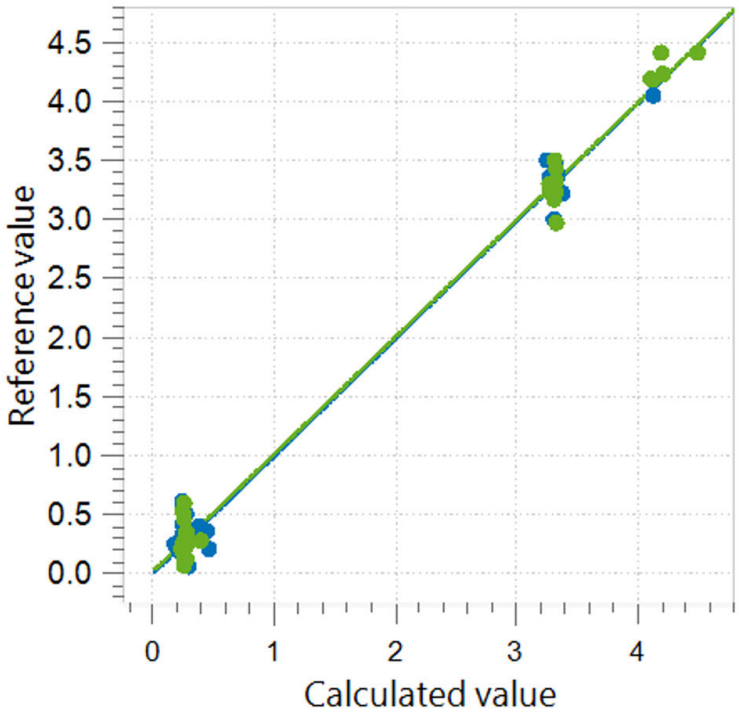


Figure 7. Correlation diagram and the respective FOMs for the prediction of moisture content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP
0.992	0.14%	0.14%	0.15%

RESULT MELTING POINT IN STEARATES

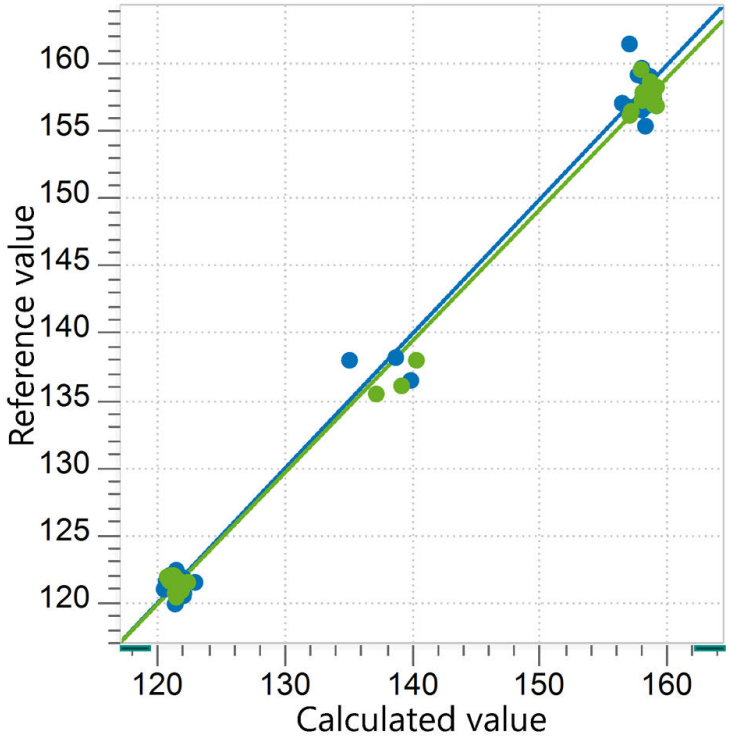


Figure 8. Correlation diagram and the respective FOMs for the prediction of the melting point in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP
0.996	1.05 °C	1.40 °C	1.25 °C

RESULT GRANULOMETRY IN STEARATES

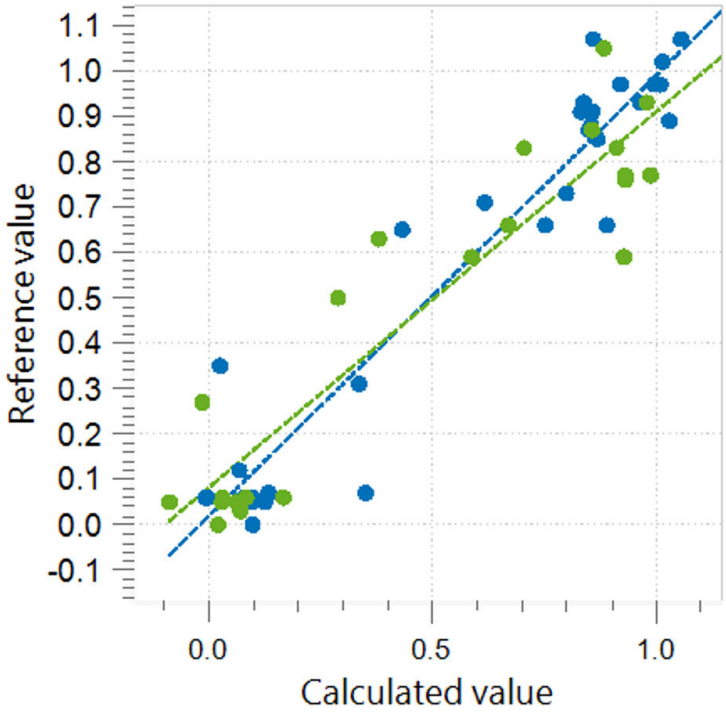


Figure 9. Correlation diagram and the respective FOMs for the prediction of the granulometry in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

R^2	SEC	SECV	SEP
0.862	0.09 mm	0.12 mm	0.15 mm

CONCLUSION

The results obtained in this study demonstrate the strong applicability of NIR spectroscopy for quantitative analysis of both process samples and final products across multiple parameters during the production of stearic acid and its salts.

The OMNIS NIR Analyzer offers significant operational advantages by combining liquid and solid measurement modules in a single instrument, making sample analysis simple and efficient. The liquid module's ability to heat samples up to 80 °C with precise temperature control ensures complete melting of samples with higher melting points, which improves measurement accuracy and reproducibility.

The final Mg, Ca, and Zn stearates are widely used in pharmaceuticals (as tablet lubricants and excipients), cosmetics (in creams, lotions, and soaps), food (as emulsifiers and coating agents), lubricants (as thickeners and stabilizers), and other sectors such as plastics and rubber (as stabilizers and release agents). This versatility shows the importance of robust analytical solutions like NIRS for quality control across diverse industries.

CONTACT

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CONFIGURATION



OMNIS NIR Analyzer Liquid/Solid

Spectromètre proche infrarouge pour échantillons liquides, solides et visqueux.

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'application des technologies les plus récentes et l'intégration dans le logiciel OMNIS moderne se reflètent dans la vitesse, le maniement et la flexibilité d'utilisation de ce spectromètre NIR.

Apercu des avantages de l'OMNIS NIR Analyzer Liquid/Solid :

- Mesures d'échantillons liquides, solides et visqueux en moins de 10 secondes
- Mesure séquentielle rapide d'échantillons liquides et solides sans modification du système
- Intégration simple dans un système d'automatisation ou liaison avec d'autres technologies d'analyse (titrage)
- Prise en charge de nombreux récipients d'échantillon

Points forts des mesures de liquides :

- Régulation de température sur l'échantillon de 25 à 80 °C
- Détection automatique de l'échantillon

Points forts des mesures de matières solides :

- Mesures multi-positions automatisées pour des résultats reproductibles, même avec des échantillons non homogènes



Flacons à usage unique, 28 mm, réflexion

216 flacons en verre à usage unique, pouvant être refermés, d'un diamètre de 28 mm pour des analyses de matières solides par réflexion. Convient aux appareils d'analyse suivants :

- NIRS DS2500 Analyzer
- NIRS XDS RapidContent (Solid) Analyzer
- NIRS XDS MultiVial Analyzer
- NIRS XDS Masterlab Analyzer



Support flexible OMNIS NIR

Support flexible avec un diamètre variable jusqu'à 30 mm pour l'examen des échantillons par réflexion dans des flacons.



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**)



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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Licence OMNIS autonome

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

Caractéristiques :

- La licence comprend déjà une licence pour appareils OMNIS.
- Elle doit être activée via le portail d'octroi de licences Metrohm.

OMNIS
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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.