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.

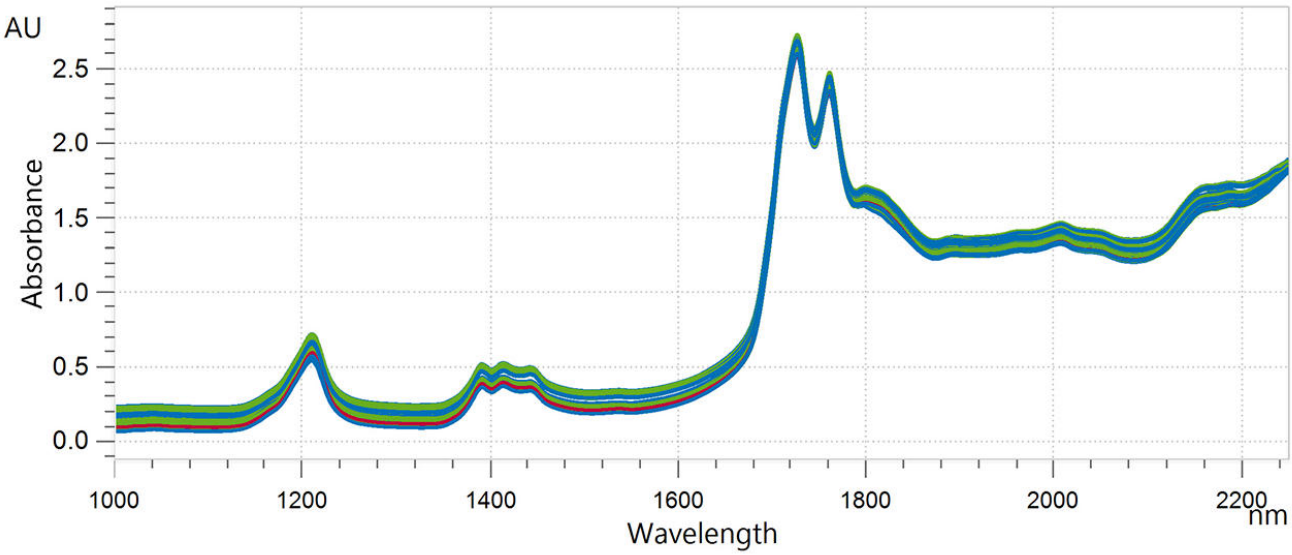

Figure 2. NIR spectra of the stearate cleavage samples collected by transmission mode in an 8 mm vial at 75 °C. These spectra were collected with the OMNIS NIR Analyzer in the liquid sample configuration.

RESULT ACID NUMBER IN PRE-ENTRY SAMPLES

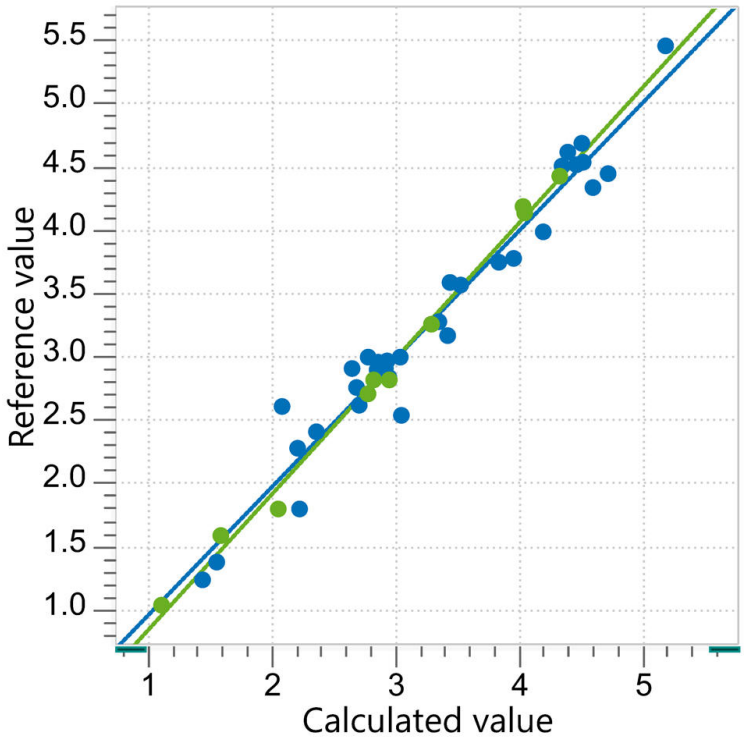

Figure 3. Correlation diagram and the respective FOMs for the prediction of acid number in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

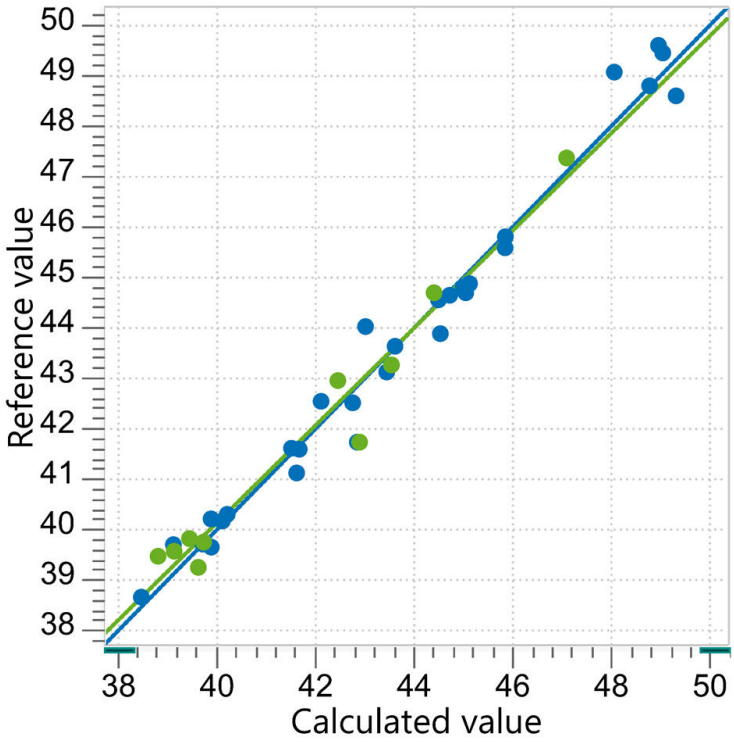

Figure 4. Correlation diagram and the respective FOMs for the prediction of iodine value in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

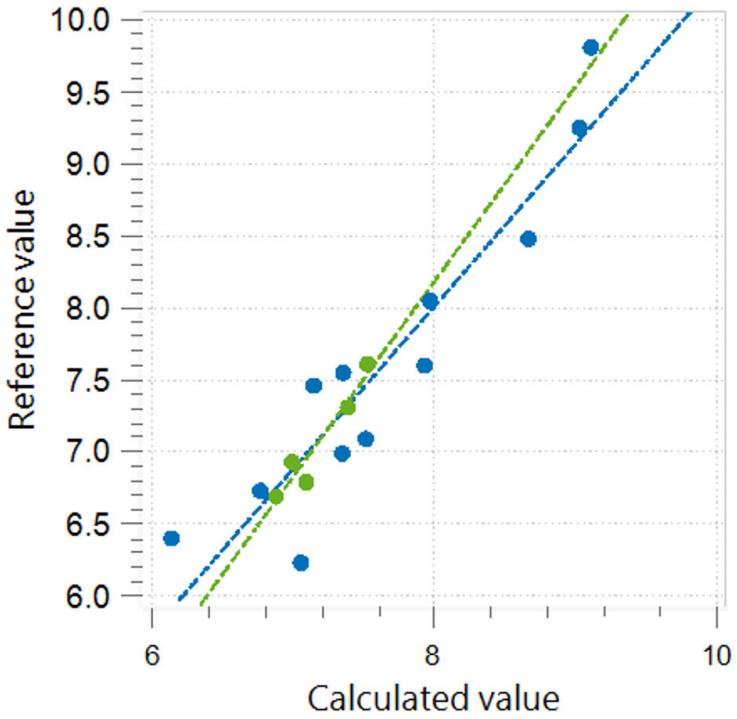

Figure 5. Correlation diagram and the respective FOMs for the prediction of acid number in the stearate hydrogenation process with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

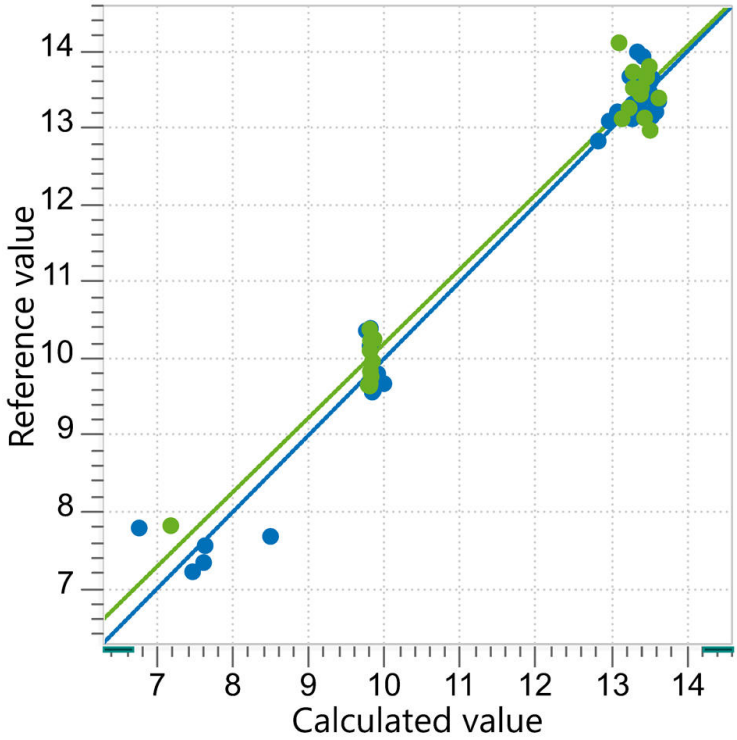

Figure 6. Correlation diagram and the respective FOMs for the prediction of ash content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT MOISTURE IN STEARATES

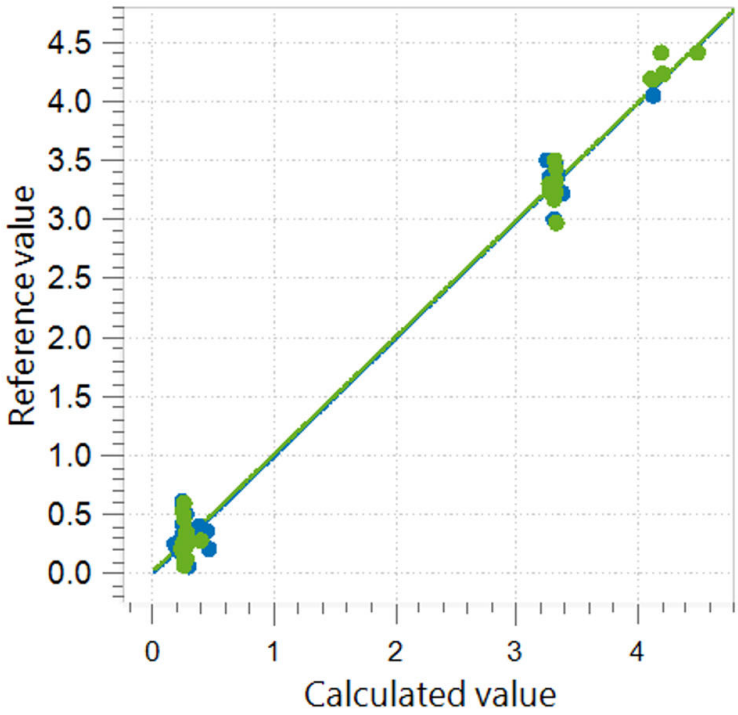

Figure 7. Correlation diagram and the respective FOMs for the prediction of moisture content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT MELTING POINT IN STEARATES

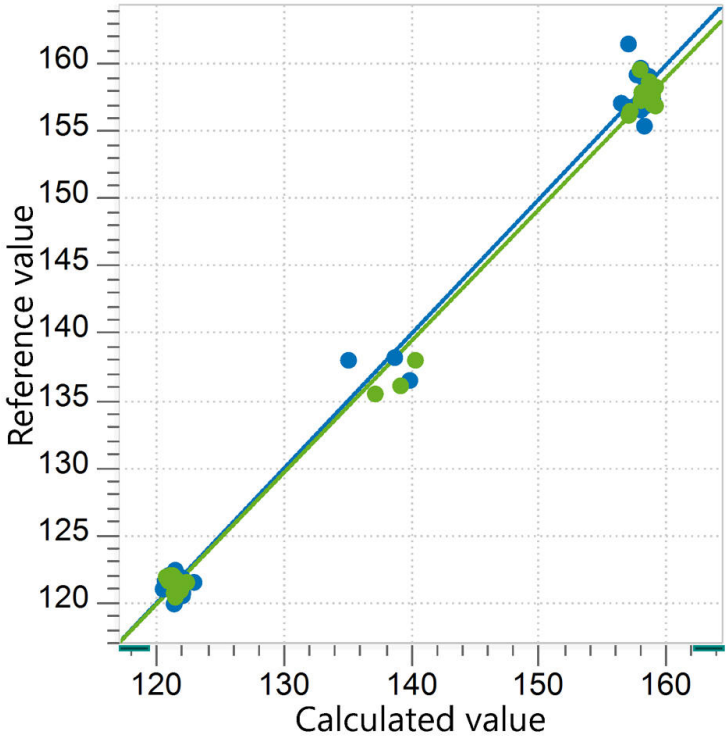

Figure 8. Correlation diagram and the respective FOMs for the prediction of the melting point in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT GRANULOMETRY IN STEARATES

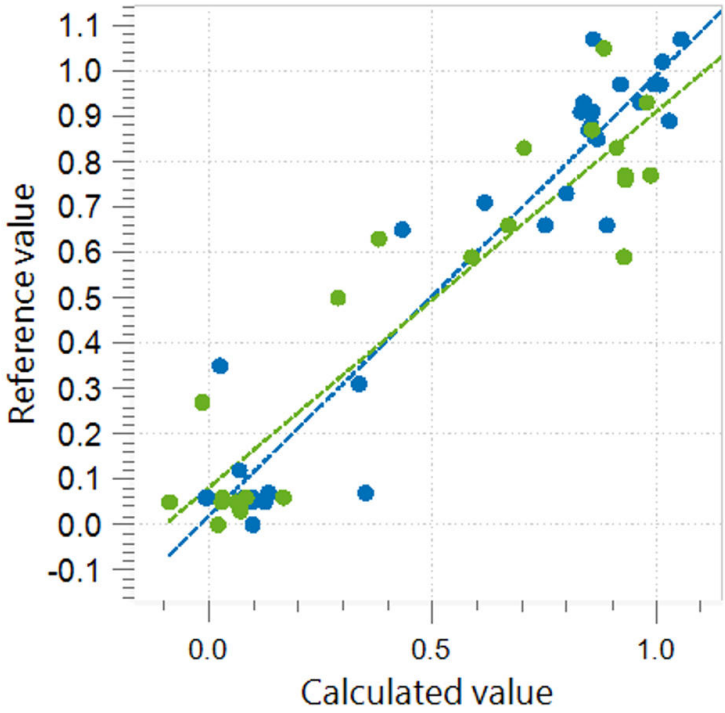

Figure 9. Correlation diagram and the respective FOMs for the prediction of the granulometry in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

Near-infrared spectrometer for liquid, solid, and viscous 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/Solid:

- Measurements of fluid, solid, and viscous samples in less than 10 seconds
- Fast sequential measurement of liquid and solid samples without conversion of the system
- Simple integration in an automation system or link with additional analysis technologies (titration)
- Supports numerous sample vessels

Highlights of measurements of liquids:

- Temperature control on the sample from 25–80 °C
- Automatic detection of the sample

Highlights of measurements of solids:

- Automated multi-position measurements for reproducible results, even with nonhomogeneous samples

Disposable vials, 28 mm, reflection

216 lockable disposable glass vials with a diameter of 28 mm for analyses of solids in reflection. Suitable for the following analyzers:

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

Flexible holder OMNIS NIR

Flexible holder with a variable diameter of up to 30 mm for the examination of samples in vials in reflection.

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)

Holder OMNIS NIR, vial, 8 mm

Vial Holder for the OMNIS NIR Analyzer for 8 mm disposable vials (6.7402.240).

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.

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Software license Quant Development

Software license for the creation and editing of quantification models in a stand-alone OMNIS Software installation.