

Application Note AN-NIR-150

采用近红外光谱技术监测硬脂酸及其盐类的生产过程

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.

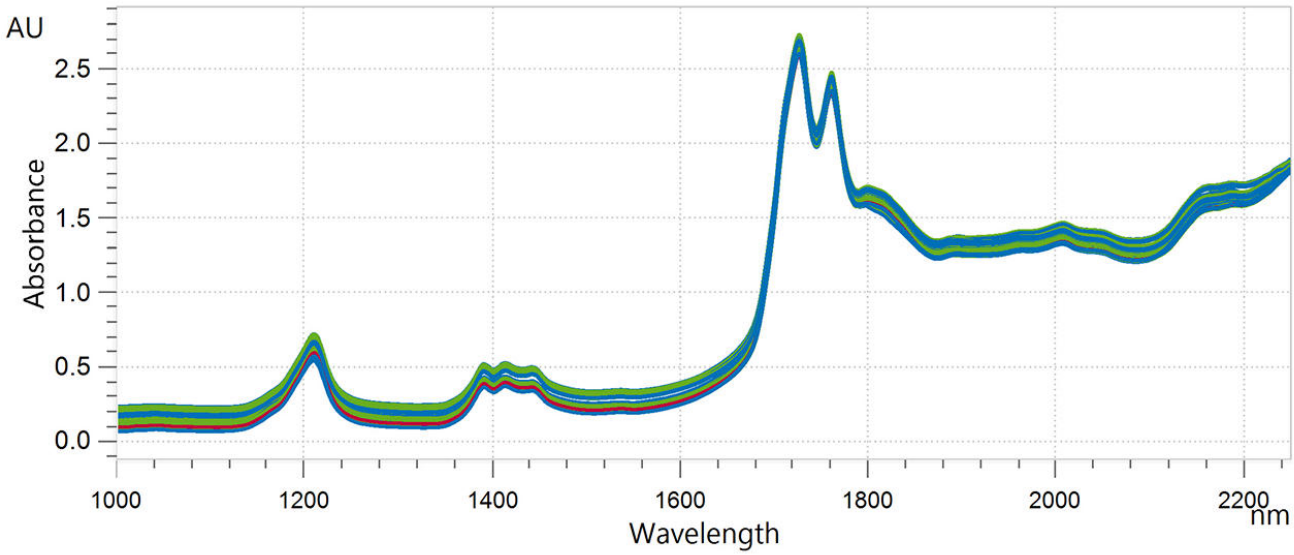


Figure 2. NIR spectra of the stearate cleavage samples collected by transmission mode in an 8 mm vial at 75 ° C. These spectra were collected with the OMNIS NIR Analyzer in the liquid sample configuration.

RESULT ACID NUMBER IN PRE-ENTRY SAMPLES

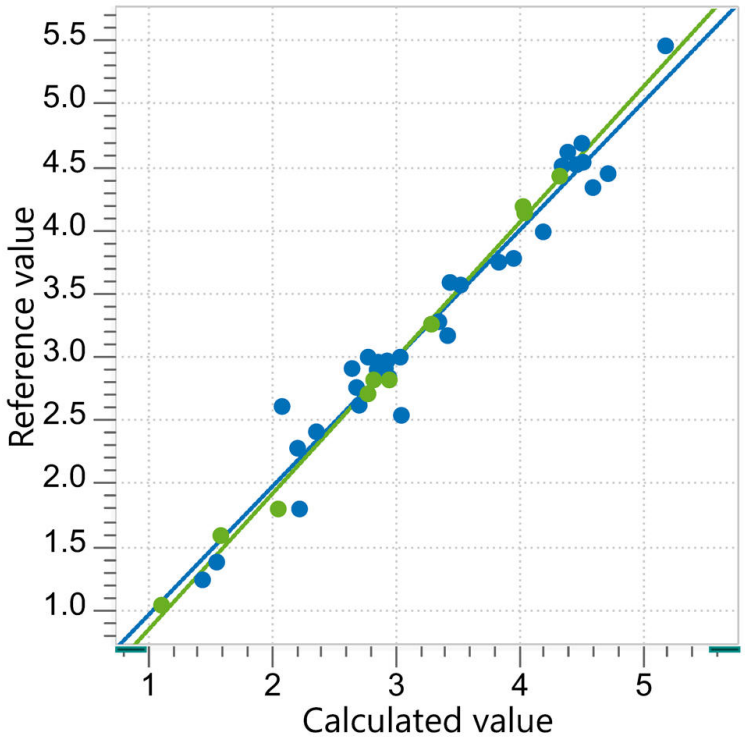


Figure 3. Correlation diagram and the respective FOMs for the prediction of acid number in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

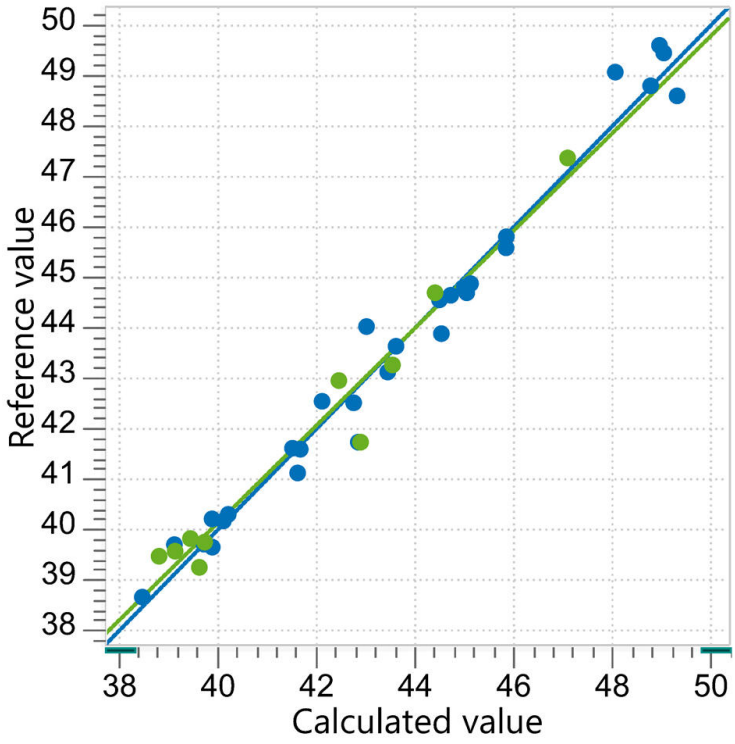


Figure 4. Correlation diagram and the respective FOMs for the prediction of iodine value in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

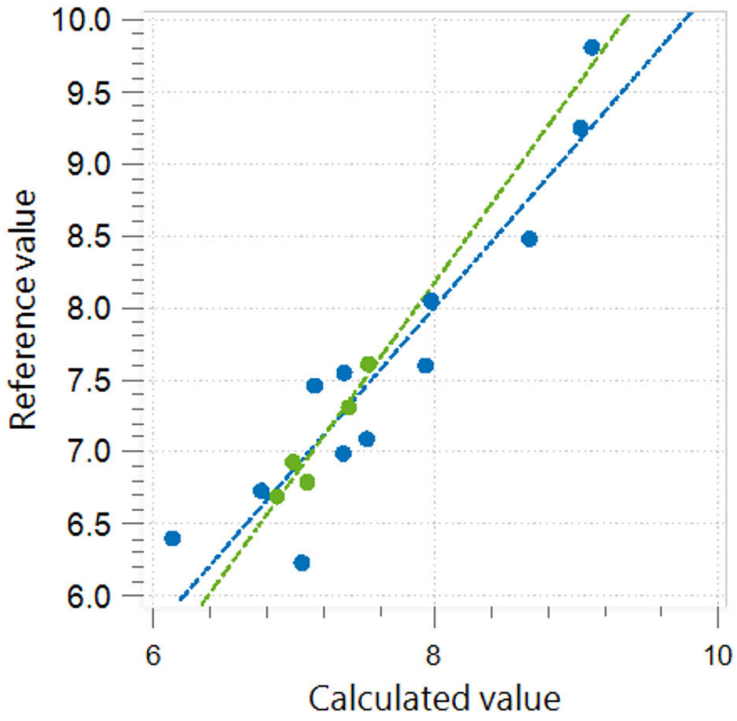


Figure 5. Correlation diagram and the respective FOMs for the prediction of acid number in the stearate hydrogenation process with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

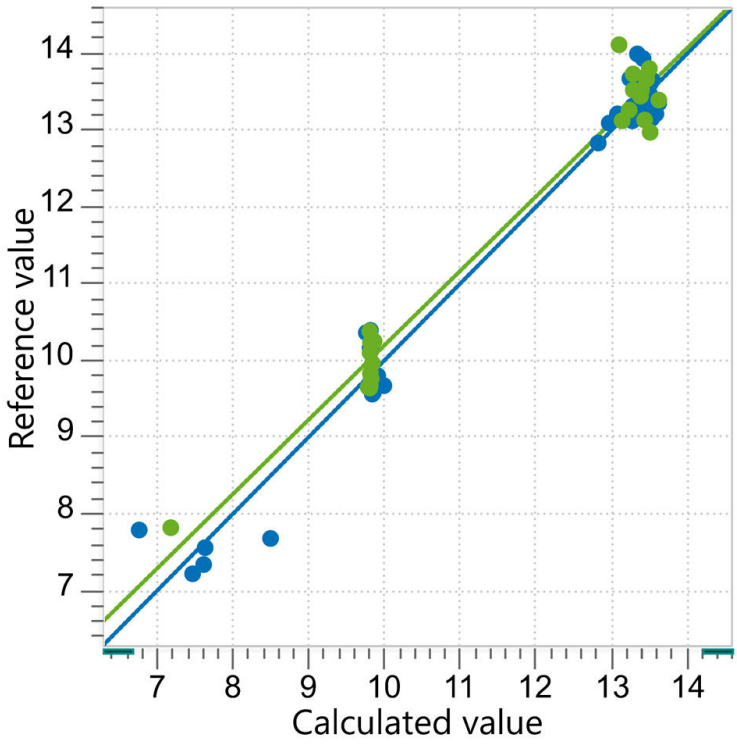


Figure 6. Correlation diagram and the respective FOMs for the prediction of ash content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT MOISTURE IN STEARATES

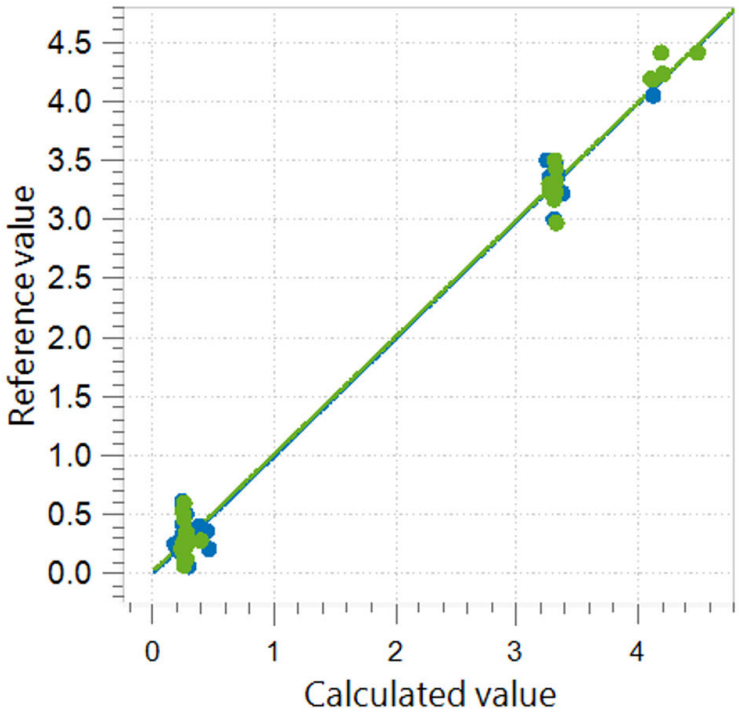


Figure 7. Correlation diagram and the respective FOMs for the prediction of moisture content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT MELTING POINT IN STEARATES

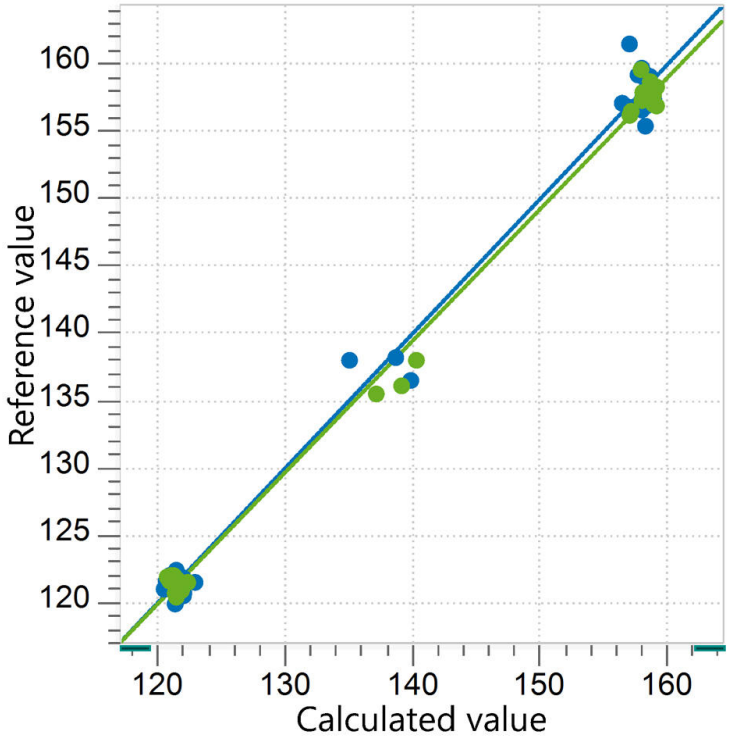


Figure 8. Correlation diagram and the respective FOMs for the prediction of the melting point in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT GRANULOMETRY IN STEARATES

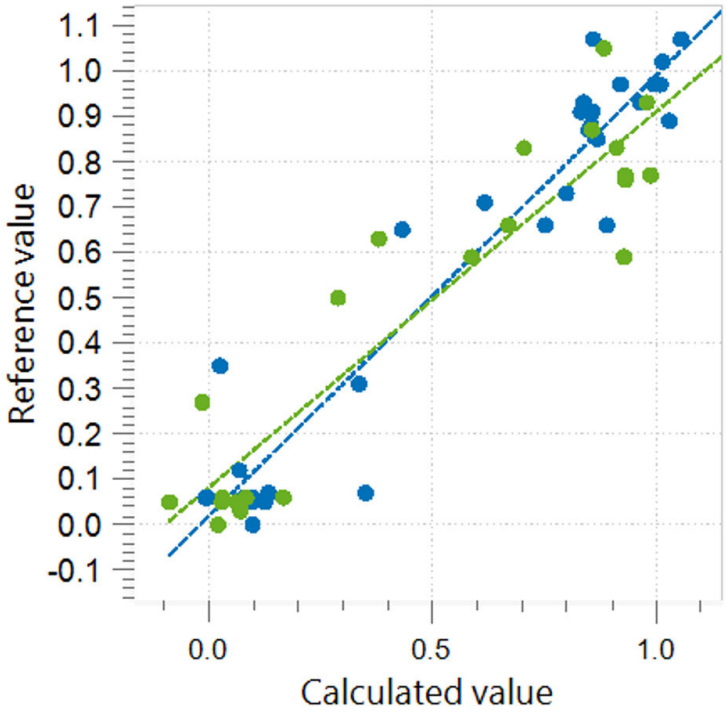


Figure 9. Correlation diagram and the respective FOMs for the prediction of the granulometry in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

Metrohm Taiwan Ltd.
開封街二段 46 號 5 樓
108 台北市
info@metrohm.com.tw

CONFIGURATION



OMNIS NIR Analyzer Liquid/Solid

用于液体、固体和粘稠样品的近红外光谱仪。

OMNIS NIR Analyzer 按照瑞士质量标准开发和制造,是用于整个生产链中常规分析的近红外光谱(NIRS)解决方案。最新技术的应用以及与现代 OMNIS Software 的集成,体现在这款 NIR 光谱仪的速度、操作性和灵活应用上。

OMNIS NIR Analyzer Liquid/Solid 的优势概览:

- 液体、固体和粘稠样品测量时间小于 10 秒
- 无需系统转换即可快速连续测量液体和固体样品
- 可轻松集成至自动系统,或与其它分析技术(滴定)联用。
- 支持多种样品容器

液体测量的亮点:

- 样品温度调节范围为 25–80 °C
- 样品的自动检测

固体测量的亮点:

- 自动多位置测量,确保即使在非均质样品中也能获得可重现的结果



28 mm

216 个可封闭的玻璃一次性样品瓶,直径为 28 mm,适用于分析反射中的固体。适用于以下分析仪:

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



OMNIS NIR

可变直径高达 30 mm 的活动支架,用于通过反射检查样品。



8 mm 100

100 个玻璃(硼硅)一次性样品瓶,具有 8 mm 的光路长度,适用于分析透射中的液体。一次性样品瓶交付时带有所属的螺旋塞(件数 = 100)。

兼容:

- 支架 OMNIS NIR,小管,8 mm (**6.07401.070**)
- DS2500 支架适用于 8 mm 一次性样品瓶 (**6.7492.020**)



OMNIS NIR8 mm

适合 8 mm 一次性小管的 OMNIS NIR Analyzer 的小管支架 (6.7402.240)。

OMNIS

A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS

允许单机版 OMNIS 软件在一台 WindowsTM 计算机上运行。

特性:

- 该许可证已含有一份 OMNIS 设备许可证。
- 须通过万通许可授权平台进行激活。

OMNIS

A WHOLE NEW LEVEL OF PERFORMANCE

Quant Development

用于在独立 OMNIS Software 安装套件中编写和编辑量化模型的软件许可证。