

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.

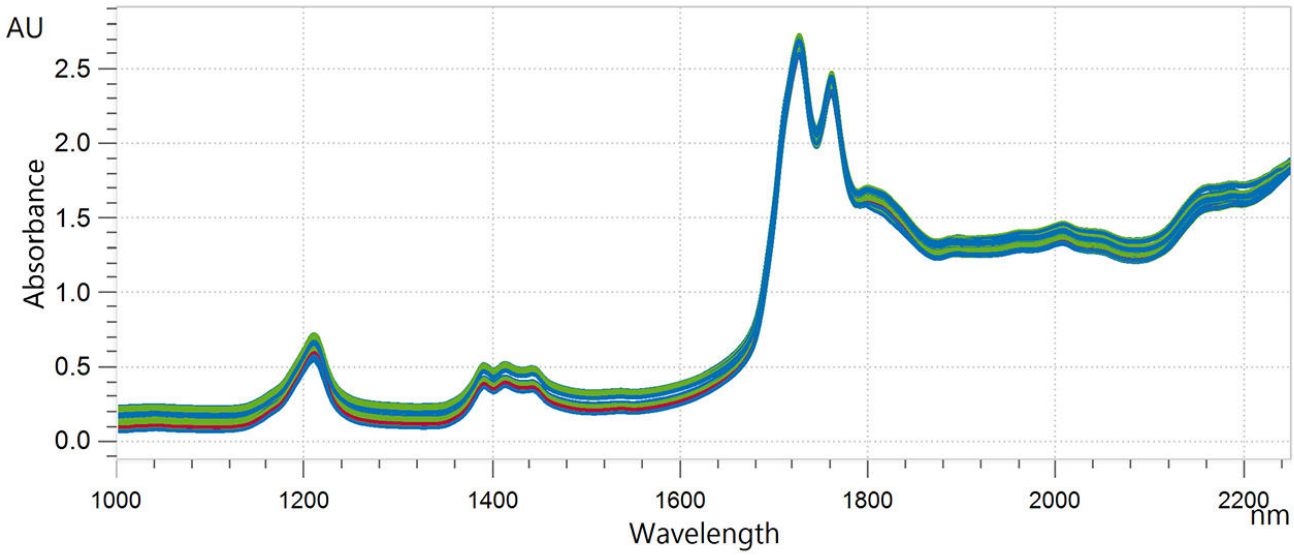


Figure 2. NIR spectra of the stearate cleavage samples collected by transmission mode in an 8 mm vial at 75 ° C. These spectra were collected with the OMNIS NIR Analyzer in the liquid sample configuration.

RESULT ACID NUMBER IN PRE-ENTRY SAMPLES

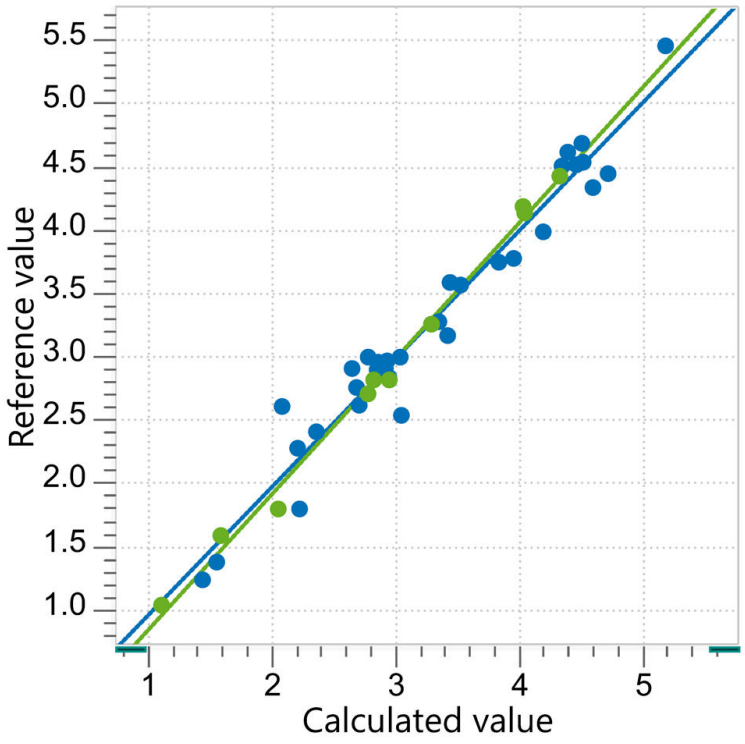


Figure 3. Correlation diagram and the respective FOMs for the prediction of acid number in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

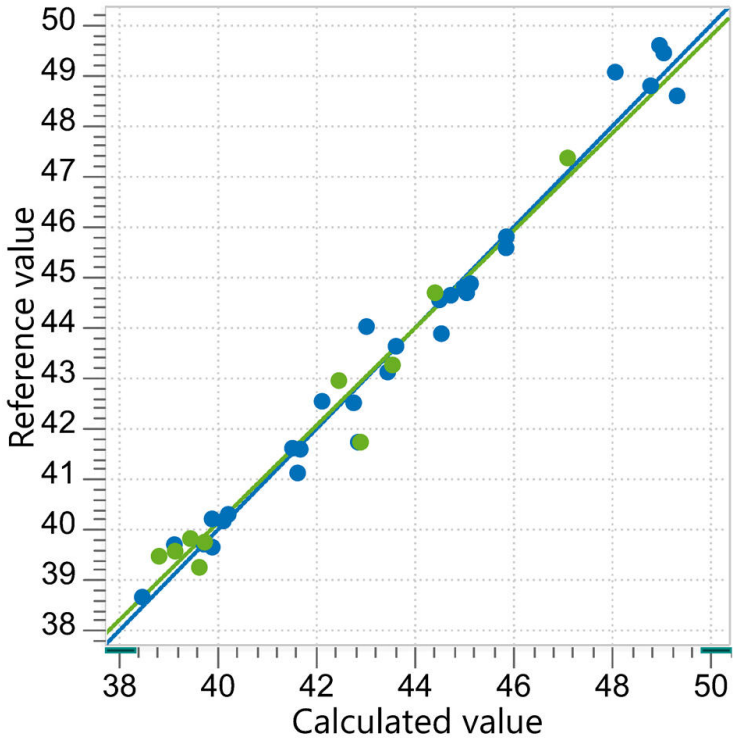


Figure 4. Correlation diagram and the respective FOMs for the prediction of iodine value in stearate pre-entry samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

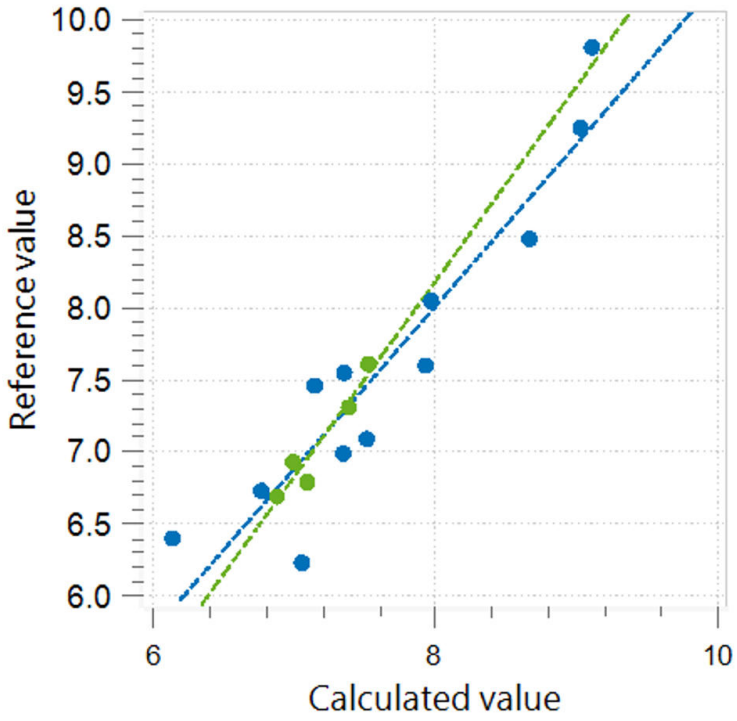


Figure 5. Correlation diagram and the respective FOMs for the prediction of acid number in the stearate hydrogenation process with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

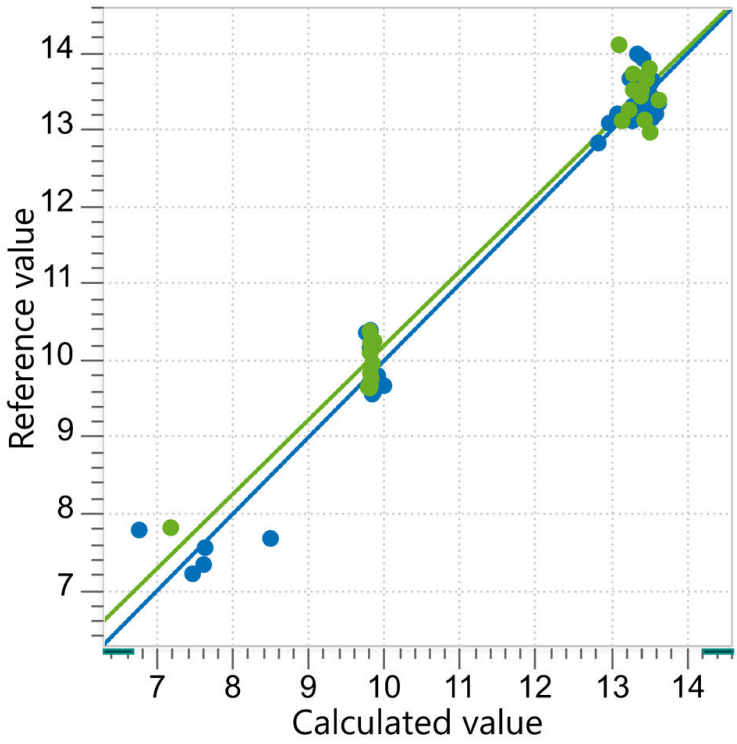


Figure 6. Correlation diagram and the respective FOMs for the prediction of ash content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT MOISTURE IN STEARATES

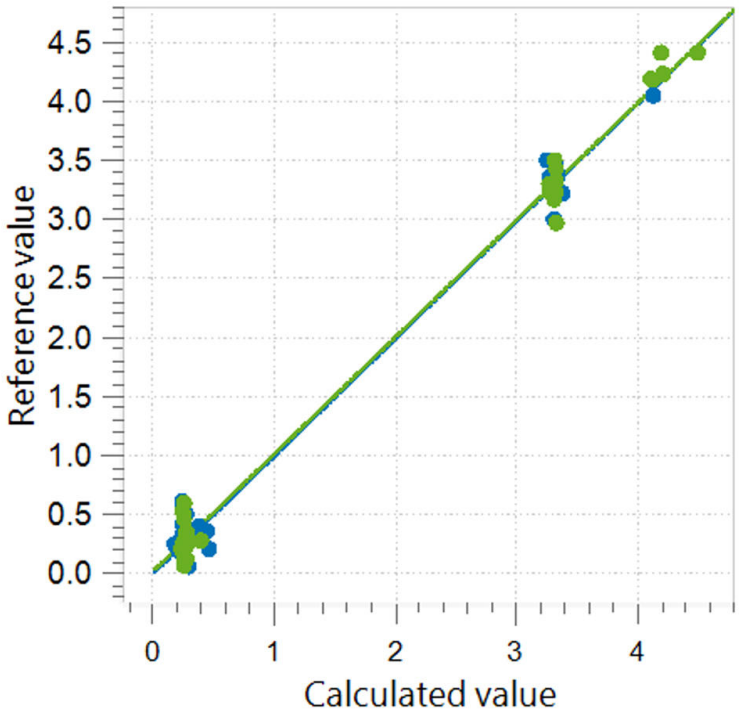


Figure 7. Correlation diagram and the respective FOMs for the prediction of moisture content in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT MELTING POINT IN STEARATES

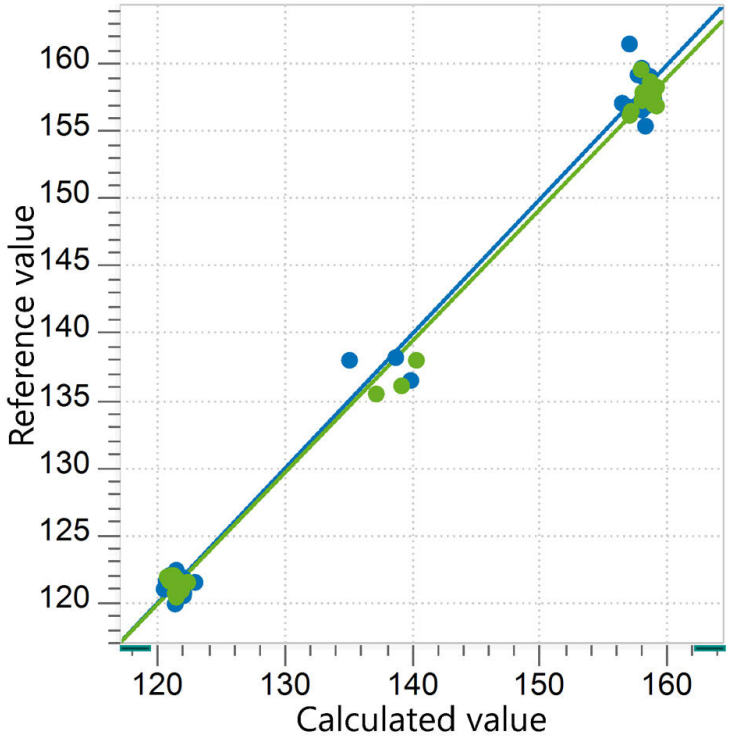


Figure 8. Correlation diagram and the respective FOMs for the prediction of the melting point in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

RESULT GRANULOMETRY IN STEARATES

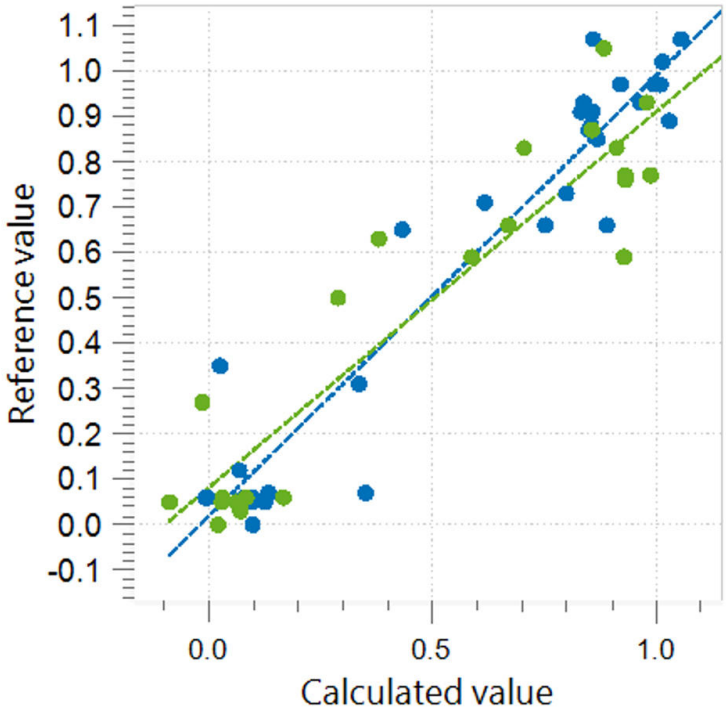


Figure 9. Correlation diagram and the respective FOMs for the prediction of the granulometry in Mg, Ca, and Zn stearate samples with the OMNIS NIR Analyzer Liquid/Solid. Blue dots represent the calibration dataset, and green dots represent the validation dataset.

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

メトロームジャパン株式会社
143-0006 東京都大田区平和島6-1-1
null 東京流通センター アネックス9階
metrohm.jp@metrohm.jp

CONFIGURATION



OMNIS NIR Analyzer Liquid/Solid

液体、固形物、および粘性のあるサンプル用の近赤外分光計。

スイスの品質規格に基づいて開発および製造されたOMNIS NIR Analyzerは、生産工程全体にわたる日常的な分析に対応する近赤外分光法(NIRS)ソリューションです。最新技術のアプリケーションと、最新のOMNIS Softwareとの統合により、このNIR分光計は高速性、操作性、そして柔軟な活用が可能となっています。

OMNIS NIR Analyzer Liquid/Solidの利点の概要:

- 液体、固形物、粘性サンプルの測定を10秒未満で実施
- システムの切り替えなしで、液体および固形物サンプルを高速かつ連続的に測定
- オートメーションシステムへの容易な統合、または追加の分析技術(滴定)との連携
- 多数のサンプル容器に対応

液体測定のハイライト:

- 25~80 °Cの範囲でのサンプルの温度調節
- サンプルの自動検出

固形物の液体測定のハイライト:

- 不均一なサンプルであっても再現性のある結果が得られる、自動多点測定



28 mm

反射された固形物を分析するための直径 28mm の密封可能なガラス製使い捨てバイアル 216 個。以下のアナライザーに適しています:

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



OMNIS NIR

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



8 mm100

透過した液体を分析するための光学的距離 8 mm のガラス製 (ホウケイ酸塩) 使い捨てバイアル 100 個。使い捨てバイアルには閉じ蓋 (個数 = 100) が付属されています。

次の製品と互換性があります:

- ホルダー OMNIS NIR、バイアル、8 mm (6.07401.070)
- 使い捨てバイアル 8 mm 用の DS2500 ホルダー (6.7492.020)



OMNIS NIR8 mm

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

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS

WindowsTMコンピューター上のOMNISソフトウェアをスタンドアローン操作することが可能になります。

特徴:

- ライセンスには、既に1つのOMNISデバイスライセンスが含まれています。
- メトローム・ライセンシングポータルにて、アクティブ化する必要があります。

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Quant Development

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