



Application Note AN-NIR-122

Quantification of water in lactose with the OMNIS NIRS Analyzer

Fast, non-destructive determination of water with NIRS

Lactose is an important pharmaceutical product. Approximately 60% to 70% of pharmaceutical dosage forms contain lactose [1], and it is one of the largest pharmaceutical excipients by volume. Lactose can be used as a bulk filler for pharmaceutical tablets, as a binder to provide more strength to a dosage form, and it can also be added to facilitate the flow of a formulation during the production process. Moisture in lactose is undesirable at high levels as it causes

the material to become sticky and bind to itself, forming hard clusters that may be difficult to break apart. USP specifies the water content range in lactose monohydrate from 4.5 % to 5.5 % [2].

The analysis of water in lactose is usually done with time-consuming methods. Near-infrared spectroscopy (NIRS) is a faster alternative. This Application Note shows the determination of water content in lactose with NIRS.

EXPERIMENTAL EQUIPMENT

In this study, samples of lactose with varying water content were analyzed to create a NIRs prediction model for quantification. Lactose monohydrate samples either spiked with water or dried in an oven were measured on an OMNIS NIR Analyzer (Figure 1) in reflection mode (1000–2250 nm) in 19 mm vials using a flexible holder. Single measurement was selected as the measuring mode. Data acquisition and prediction model development were performed with OMNIS software.



Figure 1. The OMNIS NIR Analyzer Solid from Metrohm.

Table 1. Hardware and software equipment overview.

Equipment	Article number
OMNIS NIR Analyzer Solid	2.1071.0010
Disposable vials, 19 mm, reflection	6.7402.120
Flexible holder OMNIS NIR	6.07402.300
OMNIS Stand-Alone license	6.06003.010
Quant Development software license	6.06008.002

RESULT

The measured NIR spectra (Figure 2) were used to create a quantification prediction model for the percentage of water in lactose. The quality of the prediction model was evaluated using the correlation diagram which displays a very high correlation between the NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision and

confirm the feasibility during routine analysis (Figure 3).

The water content of Hydranal Water Standard KF Oven, lactose monohydrate, (water content 5.10 ± 0.04 %) was predicted using the mentioned prediction model. The result is shown in the Table 2.

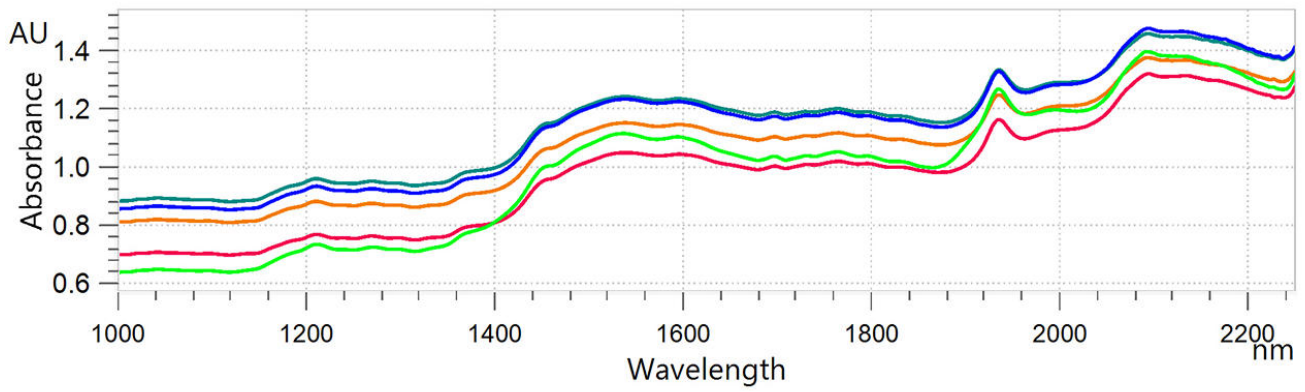


Figure 2. Overlaid NIR spectra of several lactose samples analyzed on an OMNIS NIR Analyzer Solid.

RESULT WATER CONTENT IN LACTOSE

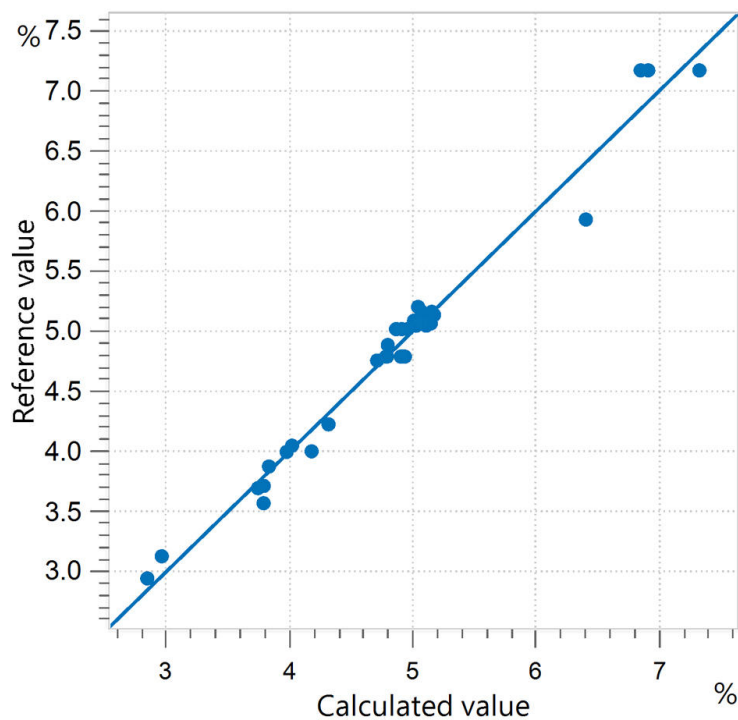


Figure 3. Correlation diagram and the respective figures of merit for the prediction of water in lactose using an OMNIS NIR Analyzer Solid. The reference water content was determined using a Karl Fischer (KF) oven method.

R^2	SEC (%)	SECV (%)
0.977	0.12	0.14

Table 2. Mean predicted water content for Hydranal Water Standard KF Oven, Lactose monohydrate, as determined with an OMNIS NIR Analyzer Solid (n = 3).

Hydranal Water Standard	
Water content (%)	5.1380
SD (rel) in %	0.029

CONCLUSION

This Application Note demonstrates the feasibility to determine water content in lactose quickly and easily. NIR spectroscopy offers users a fast, cost-effective, and highly accurate

alternative to other standard testing methods. Additionally, NIRS analysis is non-destructive, completely reagent-free, and gives results in only a few seconds.

REFERENCES

1. Hebbink, G. A.; Dickhoff, B. H. J. Chapter 5 - Application of Lactose in the Pharmaceutical Industry. In *Lactose*; Paques, M., Lindner, C., Eds.; Academic Press, 2019; pp 175–229.
[DOI:10.1016/B978-0-12-811720-0.00005-2](https://doi.org/10.1016/B978-0-12-811720-0.00005-2)
2. Lactose Monohydrate.
[DOI:10.31003/USPNF_M44190_04_01](https://doi.org/10.31003/USPNF_M44190_04_01)

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CONFIGURATION



OMNIS NIR Analyzer Solid

固体および粘性のサンプルのための近赤外スペクトロメーター。

OMNIS NIR Analyzer は、スイスの品質基準に従って開発・製造された、生産チェーン全体に沿ったルーチン分析のための近赤外分光法 (NIRS) ソリューションです。最新技術の適用と最新の OMNIS Software への統合は、この NIR スペクトロメーターの速度、ユーザビリティ、柔軟な使用に反映されています。

OMNIS NIR Analyzer Solid の利点の概要:

- 10秒未満で固体サンプルと粘性サンプルを測定
- 不均質なサンプルでも再現性のある結果を得るための自動マルチホジション測定
- オートメーションシステムへの統合、またはその他の分析技術 (滴定) との連結が容易
- 多数のサンプル容器に対応



19 mm

反射された固形物を分析するための直径 19mm の密封可能なガラス製使い捨てハイアル 225 個。製品ファミリー XDS、DS2500、OMNIS の NIR 固形物分析に適しています。

OMNIS NIR

反射中のハイアルのサンプルを調べるためのフレキシブルホルター、30 mm まで直径を調節できます。



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Windows™コンピュータ上のOMNISソフトウェアをスタント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テハ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。
- 他のコンピュータに移行することはできません。

Quant Development

スタントアロン型 OMNIS Software のインストールにおける定量化モデルの作成と編集のためのソフトウェアライセンス。