

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 NIRS 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
Disposible 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.

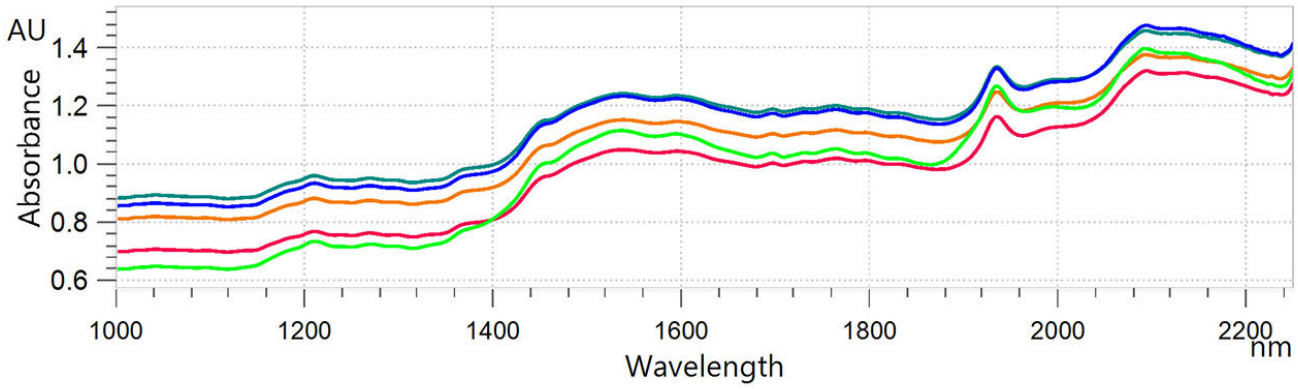


Figure 2. Overlaid NIR spectra of several lactose samples analyzed on an OMNIS NIR Analyzer Solid.

RESULT WATER CONTENT IN LACTOSE

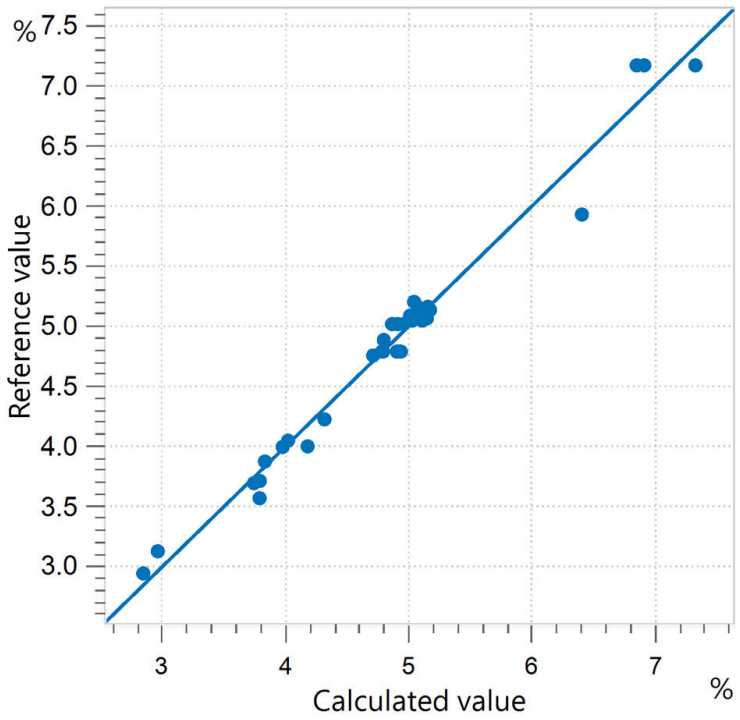


Figure 3. Correlation diagram and the respective figures of merit for the prediction of water in lactose using an OMNIS NIR Analyzer Solid. The reference water content was determined using a Karl Fischer (KF) oven method.

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
2. Lactose Monohydrate. DOI:10.31003/USPNF_M44190_04_01

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CONFIGURATION



OMNIS NIR Analyzer Solid

Near-infrared spectrometer for 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 Solid:

- Measurements of solids and viscous samples in less than 10 seconds
- Automated multi-position measurements for reproducible results, even with nonhomogeneous samples
- Simple integration in an automation system or link with additional analysis technologies (titration)
- Supports numerous sample vessels



Disposable vials, 19 mm, reflection

225 lockable disposable glass vials with a diameter of 19 mm for analyses of solids in reflection. Suitable for NIRS RapidContent (Solid) Analyzers of the XDS, DS2500 and OMNIS product families.



Flexible holder OMNIS NIR

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

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.