

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.

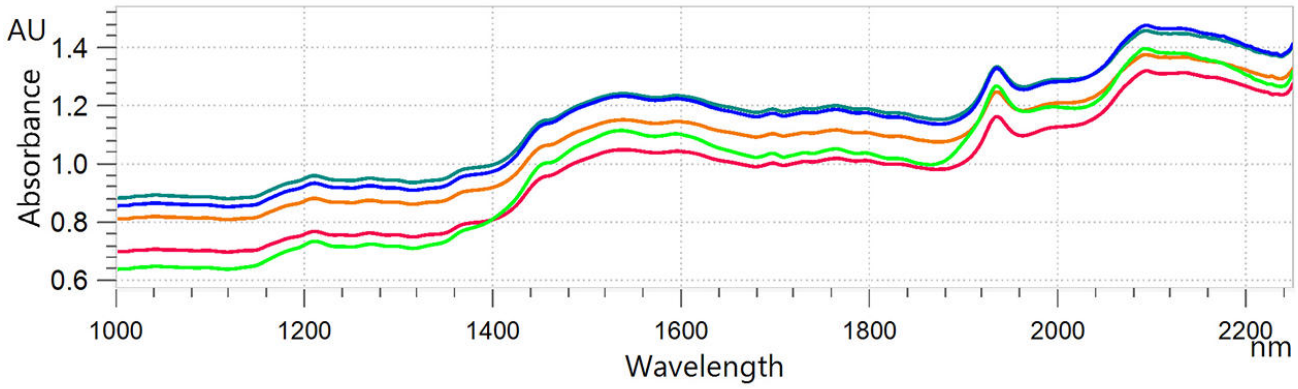


Figure 2. Overlaid NIR spectra of several lactose samples analyzed on an OMNIS NIR Analyzer Solid.

RESULT WATER CONTENT IN LACTOSE

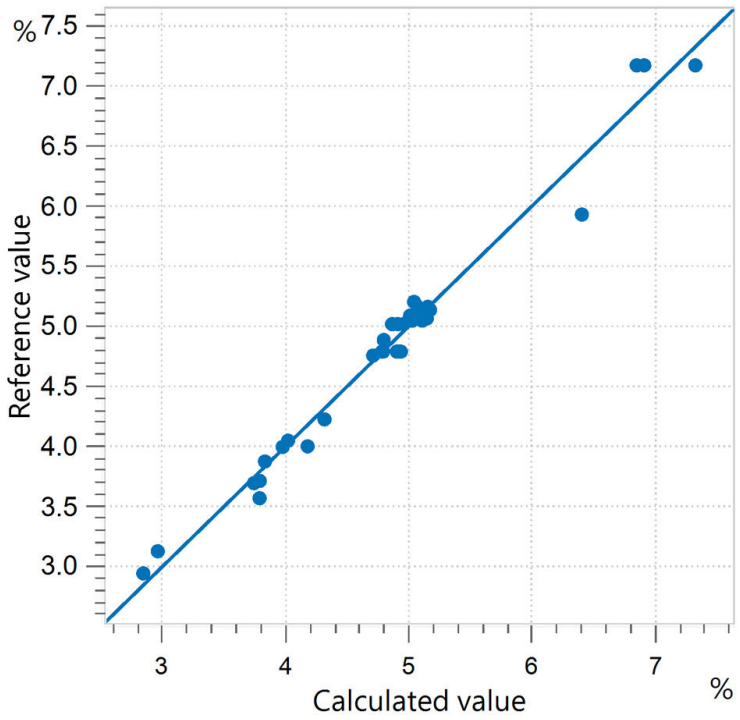


Figure 3. Correlation diagram and the respective figures of merit for the prediction of water in lactose using an OMNIS NIR Analyzer Solid. The reference water content was determined using a Karl Fischer (KF) oven method.

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

CONTACT

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CONFIGURATION



OMNIS NIR Analyzer Solid

Spectromètre proche infrarouge pour échantillons solides et visqueux.

L'OMNIS NIR Analyzer est la solution de spectroscopie proche infrarouge (NIRS) développée et produite selon les normes de qualité suisses pour les analyses de routine tout au long de la chaine de fabrication. L'application des technologies les plus récentes et l'intégration dans le logiciel OMNIS moderne se reflètent dans la vitesse, la facilité du maniement et la flexibilité d'utilisation de ce spectromètre NIR.

Apercu des avantages de l'OMNIS NIR Analyzer Solid :

- Mesures d'échantillons solides et visqueux en moins de 10 secondes
- Mesures multi-positions automatisées pour des résultats reproductibles, même avec des échantillons non homogènes
- Intégration simple dans un système d'automatisation ou liaison avec d'autres technologies d'analyse (titrage)
- Prise en charge de nombreux récipients d'échantillon



Flacons à usage unique, 19 mm, réflexion

225 flacons en verre à usage unique, refermables, d'un diamètre de 19 mm pour des analyses de matières solides par réflexion. Convient à l'appareil d'analyse de matières solides NIR des familles de produits XDS, DS2500 et OMNIS.



Support flexible OMNIS NIR

Support flexible avec un diamètre variable jusqu'à 30 mm pour l'examen des échantillons par réflexion dans des flacons.

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

Licence OMNIS autonome

Elle permet l'exploitation autonome du logiciel OMNIS sur un ordinateur WindowsTM.

Caractéristiques :

- La licence comprend déjà une licence pour appareils OMNIS.
- Elle doit être activée via le portail d'octroi de licences Metrohm.

OMNIS
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Licence logicielle Quant Development

Licence logicielle pour la création et l'édition de modèles de quantification dans une installation du logiciel OMNIS Stand-Alone.