Speed vs. accuracy in moisture sorption analysis: The hydration of L-lysine HCl

– Application Note 17-01

Introduction

Measurement of sorption kinetics by gravimetric moisture sorption is well known for not being a fast analysis method.

Whereas residual moisture content determination by Karl Fischer titration or water-activity measurements are completed within a relatively short period of time, water vapor sorption may take several days depending on the sorption kinetics of the sample material.

Providing an optimized surface to volume ratio by preparing the sample as a thin layer in a larger dish may at least save some valuable measuring time by avoiding bulk effects. The sorption kinetics of the material itself, however, cannot be changed.

Scope

L-lysine monohydrochloride (L-lysine HCl) was used as model system to demonstrate that speeding up a sorption analysis might lead to risky misinterpretation of results.

Hydration was measured in the Moisture Sorption Analyzer SPSx-1µ.

The measurement time and equilibrium conditions were varied to demonstrate their effects on moisture sorption and analysis results.

Speed up moisture sorption analysis?

With well-known samples, the cumulated measurement time for a test series can become very long. With a limited time-frame to finish a test series, one might be tempted to shorten the total required measurement time.

This can be achieved by:

1. Reducing the number of humidity steps per measurement
2. Weakening of the condition for detection of equilibrium (change of mass over time)
3. Limiting the maximum time per humidity step.

Such procedure may work for already well-known sample material. As sorption analysis is typically used in R&D with material of more or less unknown sorption properties, one might miss important information during a shortened measurement and thus rendering the whole test series to be useless.

1. Measurement under time pressure

Fig. 1 shows the sorption isotherm of anhydrous L-lysine HCl. Measurement parameters were chosen in order to finish the analysis within 24 hours.

Above 60 % RH, the results obtained provide insufficient information about the equilibrium state of the sample. No clear statements can be made about what happens between 60 % and 90 % RH in the sorption cycle.

One might conclude from Fig. 1 that there is no stable form of the dihydrate on the sorption branch of the isotherm.

- Too few humidity steps result in a bad resolution of the isotherm
- The maximum time per humidity step was set too short.
- Not sufficient time to reach equilibrium
- Too few humidity steps

2. Time pressure relieved measurement

For the sorption isotherm in Fig. 2, the time per moisture step was extended and a higher number of moisture steps was set. This allowed the samples to reach equilibrium, and a higher resolution of the sorption isotherm was achieved.

This clearly showed that the anhydrous form is stable up to 65 % RH. The dihydrate is stable in a relative humidity range of 70-85 %. Above 90 % RH the deliquescence point is exceeded, and the L-lysine starts to liquefy.

- Sufficient time to reach equilibrium
- Higher resolution

Conclusion

With a larger number of humidity steps and sufficient time for the sample to reach equilibrium at each step, high resolution sorption data is generated.

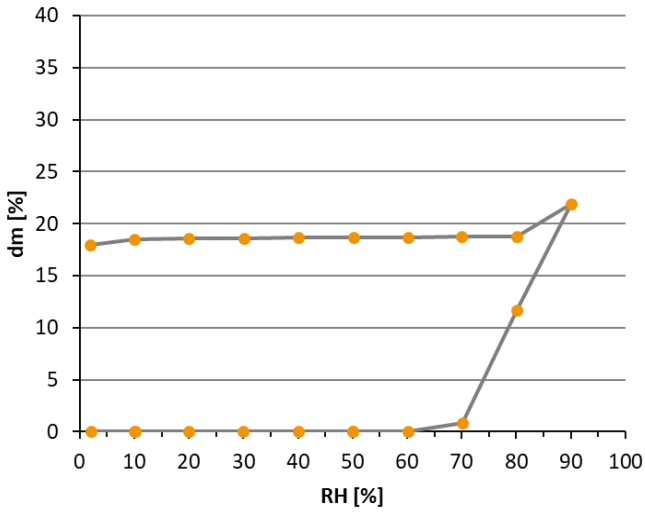

Fig. 1: Sorption isotherm of anhydrous L-lysine HCl from 0 % to 90 % RH at 25 °C. RH steps of 10 %, equ. condition 0.02 % mass change in 25 min, max. 2 h per step

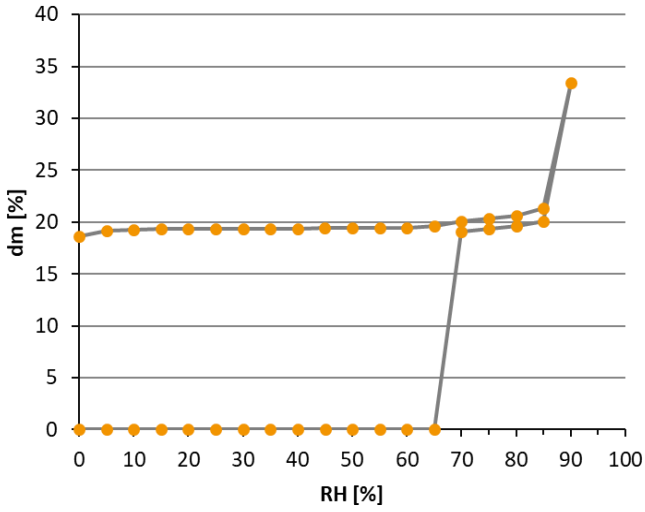

Fig. 2: Sorption isotherm of anhydrous L-lysine HCl from 0 % to 90 % RH at 25 °C. RH steps of 5 %, equ. condition 0.01 % mass change in 25 min, max. 20 h per step

"Time pressure relieved measurements generate valuable, high-resolution data, avoiding misinterpretation of sorption data"