Microcrystalline Cellulose as a Humidity Reference for Gravimetric DVS Instruments

– Application Note 18-03

Introduction

Frequent validation of the humidity sensor calibration of gravimetric DVS instruments is highly recommended. With the proUmid MCC reference, a certified factory standard for precise and reliable validation results was made available.

The reference material was certified in a certification study including a comprehensive Europe-wide round robin test with 10 participating laboratories [1].

With a multi-sample DVS instrument, the periodically required humidity validation can be conveniently accomplished in-line with regularly scheduled measurements, using a free sample dish. Running time-consuming separate validation tests is not required.

In-line validation measurement

Requirements for in-line humidity validation, parallel to a standard routine measurement:

- free sample dish on the sample carousel,
- constant 25 °C measurement temperature,
- sorption and desorption cycle, ideally over an extended RH range, e.g. from 0 % RH to 90 % RH,
- addition of an extra 3 % RH step to the sorption cycle.

During sample preparation, 100 mg of the MCC reference are distributed evenly on the bottom of the sample pan designated for the humidity validation.

The other sample and measurement settings are prepared and started according to the applying standard operation procedure (SOP).

Validation procedure

The MCC sorption data is evaluated separately from the other samples. For each humidity step x, the change of mass in percent, dm_x, based on the equilibrium net weight of the additional 3 % RH step, $\mathrm{m}_{3\%\,\mathrm{RH}}$, is determined as

$$\mathrm{dm}_x = \frac{\mathrm{m}_x - \mathrm{m}_{3\%\,\mathrm{RH}}}{\mathrm{m}_{3\%\,\mathrm{RH}}} \times 100 \qquad [1]$$

with m_x being the measured equilibrium net weight of the corresponding humidity step x.

The dm_x values of all humidity steps used for validation are compared with the values given in the MCC certificate (Fig. 1). If the allowed deviation is exceeded, re-calibration of the humidity sensor is recommended.

sorption/ desorption	relative humidity in %	change of mass* dm in %	uncertainty** dm in %	allowed deviation dm in %
sorption	10	0.890	0.051	0.4
	20	1.828	0.075	0.4
	30	2.637	0.066	0.4
	40	3.467	0.076	0.4
	50	4.410	0.074	0.4
	60	5.475	0.085	0.6
	70	6.780	0.105	0.6
	80	8.507	0.129	0.7
	90	11.459	0.305	1.0
desorption	80	9.782	0.127	0.7
	70	8.093	0.132	0.6
	60	6.732	0.109	0.6
	50	5.560	0.095	0.4
	40	4.516	0.072	0.4
	30	3.550	0.070	0.4
	20	2.548	0.062	0.4
	10	1.372	0.071	0.4

* unweighted mean value of the means of accepted sets of data for certification.
** half-width of the 99.5% confidence interval of the mean.

Fig. 1: proUmid MCC certificate for batch no. 5611262924.

In-line validation example

A SPS11-10µ instrument was validated in-line during a regular measurement with several food ingredients. 100 mg of MCC reference material were placed in a free dish on the sample carousel and an extra 3 % RH step was added to the sorption cycle (Tab. 1). Sorption kinetics data of the measurement are shown in Fig. 2.

Table 1: Humidity settings at constant 25 °C

cycle	RH steps [%]
sorption	0 – **3** - 10 - 20 - 30 - 35 - 40 - 50 - 60 - 65 - 70 - 80 - 85 - 90
desorption	85 - 80 - 70 - 65 - 60 - 50 - 40 - 35 - 30 - 20 - 10 - 0

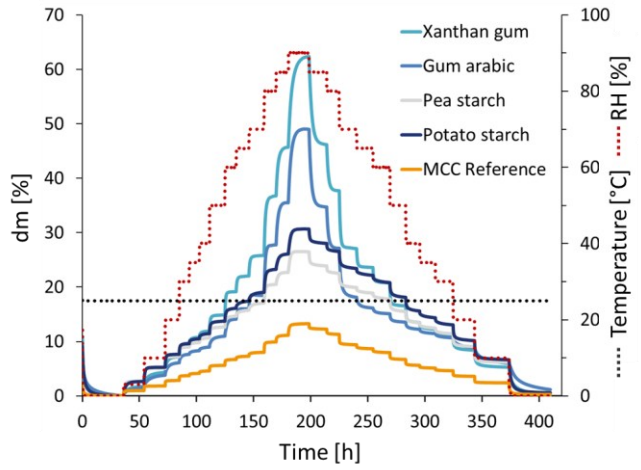

Fig. 2: Sorption kinetics data of a food ingredients measurement with in-line humidity validation using an extra MCC reference sample.

Evaluation and results

The change in mass of the MCC reference sample for all RH steps required for validation (Tab. 2), based on the equilibrium net weight recorded at the end of the additional 3 % RH step, was calculated according to equation (1) and compared with the values from the certificate. All results for the MCC were found to be within the limit for the maximum allowed deviation.

Table 2: Validation of the humidity calibration

RH [%]	dm [%] certificate	dm_x [%]	deviation	max. allowed	result
10	0.995	0.842	0.153	0.4	☑
20	1.910	1.849	0.061	0.4	☑
30	2.716	2.716	0.000	0.4	☑
40	3.564	3.586	0.022	0.4	☑
50	4.515	4.602	0.087	0.4	☑
60	5.610	5.665	0.055	0.6	☑
70	6.934	6.964	0.030	0.6	☑
80	8.721	8.847	0.126	0.7	☑
90	11.623	12.184	0.561	1.0	☑
80	9.929	10.232	0.303	0.7	☑
70	8.197	8.343	0.146	0.6	☑
60	6.813	6.923	0.110	0.6	☑
50	5.644	5.677	0.033	0.4	☑
40	4.592	4.596	0.004	0.4	☑
30	3.610	3.614	0.004	0.4	☑
20	2.645	2.598	0.047	0.4	☑
10	1.434	1.417	0.017	0.4	☑

Summary

With this dual-purpose measurement, not only the calibration of the instrument, but also the generated measurement data of the food ingredient samples itself were successfully validated.

The in-line validation saves valuable measurement time, especially when doing long measurements as in this example, with many RH steps, slow sorption kinetics and a total measurement time of 420 hours.

Practical note: With a single or a few values slightly exceeding the allowed deviation, it is required to check whether the humidity calibration is still acceptable or if the measurement needs to be rejected and repeated after re-calibration.
For very sensitive applications, e.g. in pharma, user defined maximum allowed deviation smaller than on the certificate values can be specified.

References

[1] proUmid Application Note 18-02, Certification of Microcrystalline Cellulose as a humidity reference for gravimetric DVS instruments.