



Moisture Sorption of EC Standard Reference Material RM 302 on a DVS Instrument

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The Dynamic Vapour Sorption (DVS) provides potential benefit over the COST 90 procedure. This application note compares DVS Results to COST 90 procedure.

Introduction

Microcrystalline cellulose (RM 302) is a standard EC reference material for validation of the measurement of water sorption isotherms of food materials following the COST 90 procedure [1]. This procedure involves the periodic weighing of samples stored over saturated salt solutions until equilibrium is established (nominally after 7 days). The moisture content RM 302 at ten specified relative humidities (from Stokes and Robinson [2] has been certified in a study between ten independent labs within the EC [1]. The DVS instrument provides many potential benefits over the COST 90 procedure for isotherm measurement including much faster equilibration times, independently validated humidity generation, and fully automated weighing of samples, thus eliminating some sources of error in the measurement. This application note therefore presents data from two independent laboratories using DVS instruments to measure water sorption isotherms on RM 302 for comparison with the COST 90 procedure.

Method

Samples of microcrystalline cellulose RM 302 supplied by the Laboratory of the Government Chemist, UK, in sealed sachets were stored at 4°C until ready for use. Sample sizes between 10-15 mg were used to give the optimum balance between sensitivity, equilibration time and homogeneity. Ten humidity steps were chosen to correspond to the saturated salt solutions used in the EC study, and equilibration times of between 2 - 6 hours for each step were chosen. All samples were analysed at 25°C, with 20-second data collection intervals and a total flow rate of 200 sccm. The total acquisition time for a complete sorption isotherm including drying curve was 33.5 hours.

Results

Figure 1 shows typical kinetic data for a sorption isotherm on RM 302 using the method outlined above and indicates that equilibrium is established quite rapidly for all the sorption steps.



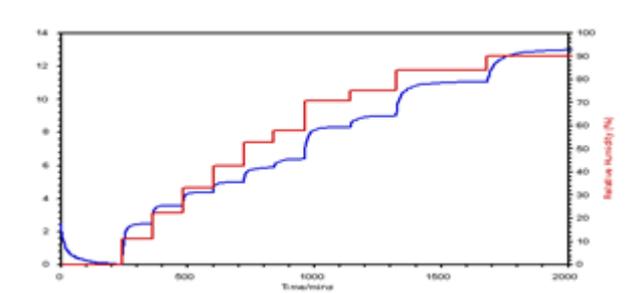


Figure 1. Kinetic data for moisture sorption of RM 302 on a DVS instrument.

Table 1 is a summary of the certified equilibrium moisture content of RM 302 as measured by the COST 90 procedure and using a DVS gravimetric vapour sorption instrument. The DVS data shown is the mean of 4 data sets, 3 from one instrument in the same laboratory and one from a different instrument in another laboratory. The standard deviation of the four data sets is also shown to give some indication of the repeatability of the DVS measurement.

Table 1. Moisture sorption isotherm data for RM 302.

Relative Humidity (%)	Mean % H2O Content COST 90	Mean % H2O Content DVS	Standard Deviation DVS
11.05	2.13 ± 0.11	2.45	0.026
22.45	3.24 ± 0.12	3.59	0.041
33.00	4.15 ± 0.09	4.40	0.029
42.76	5.16 ± 0.09	5.02	0.027
52.86	5.97 ± 0.14	5.89	0.026
57.70	6.48 ± 0.15	6.43	0.029
70.83	8.25 ± 0.17	8.29	0.060
75.28	8.90 ± 0.24	8.84	0.081
84.26	11.00 ± 0.33	10.98	0.071
90.19	13.27 ± 0.43	12.82	0.160

Conclusion

The DVS data agrees well with the COST 90 data for humidities above 33%, where all the data points fall within the 95% confidence limits of the EC study. For humidities of 33% and lower, the DVS data is consistently above this confidence limit, indicating that there may be systematic differences between the two procedures, since the standard deviation of the DVS data set is very small over this range.

In conjunction with saturated salt solutions (App. Note 01), this data provides a rapid means of validating humidity generation in the DVS instrument.

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References

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