



## Humidity Calibration of Dynamic Vapor Sorption (DVS) Instruments

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### ABSTRACT

Methods for verification or calibration of humidity generators are described in ASTM International standard E 2551 (1). This ASTM standard describes a gravimetric measurement of deliquescent salts to verify the humidity control at a given temperature over the relative humidity range of 5 to 98 %RH (2). Humidity control for the Q5000SA Dynamic Vapor Sorption (DVS) instrument is exemplified using sodium bromide salt at 25 °C.

### INTRODUCTION

Adsorption of water, which is everywhere, significantly affects the properties of many materials. Dynamic Vapor Sorption (DVS) instruments are designed to measure a material's propensity to absorb water when exposed to a humid environment. DVS instruments consist of a microbalance to measure sample weight, temperature control and humidity generation systems, plus a way to measure the actual humidity. Figure 1 shows a schematic of the Q5000 SA DVS.

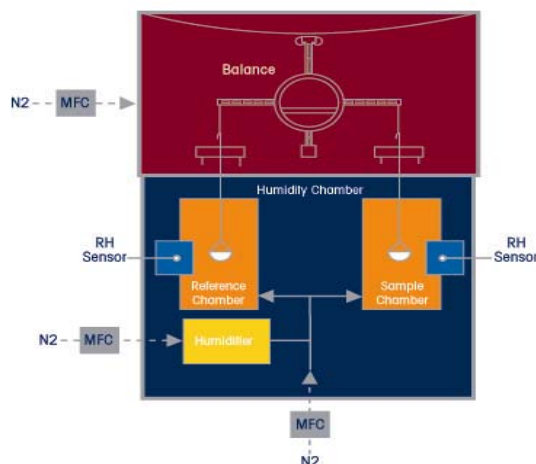


Figure 1 – Schematic Diagram of the Q5000SA Dynamic Sorption Instrument

Most commercial instruments use relative humidity sensors to measure and control the atmosphere to which the sample is exposed. TA Instruments Q5000 SA takes another approach, which avoids the use of relative humidity sensors for control due to their unreliability and short lifespan. The Q5000 SA utilizes mass flow controllers to control the humidity, by mixing a saturated gas stream with a dry gas to attain the required humidity.



Deliquescent salts are used to verify the humidity of the environment. Deliquescent materials absorb very little water as the humidity is raised until the percent relative humidity (%RH) reaches a critical level or deliquescence point. At a given temperature and that specific %RH, the material suddenly starts absorbing any available moisture. Table 1 shows some common deliquescent salts and their deliquescence points (2).

## EXPERIMENTAL and RESULTS

There are three experimental methods described in ASTM E 2551 that may be used for determining the deliquescence point:

### *Method A - Step Humidity Up*

- Begin at least 3-7 %RH below the expected deliquescence point and increase the relative humidity in small incremental steps.
- The deliquescence point is defined as the percent relative humidity (%RH) where the weight curve first deviates from baseline.

### *Method B - Step Humidity Down*

- Begin at least 3-7 %RH above expected deliquescence point and decrease the relative humidity in small decremental steps.
- The deliquescence point is defined as the percent relative humidity at the maximum in the weight curve.

### *Method C - Ramp Humidity Down*

- Begin at least 3-7 %RH above the expected deliquescence point and linearly decrease the relative humidity.
- The deliquescence point is defined as the percent relative humidity where the derivative of mass change with respect to the relative humidity ( $dm/dRH$ ) is zero.

Salt	Theoretical (% RH)	Experimental (Method C) (% RH)
Lithium Chloride	11.30	10.96
Magnesium Chloride	32.80	32.46
Magnesium Nitrate	52.90	52.70
Sodium Bromide	57.60	57.77
Sodium Chloride	75.30	75.50
Potassium Chloride	84.20	84.49
Potassium Nitrate	93.60	92.85

Table 1- Q5000SA Humidity Calibration Verification

Method C utilizes the ability to ramp the relative humidity to reduce the length of the experiment. Only a total of 125 min is required for this method including 60 min of equilibration



time at the beginning of the experiment. Experimental results were collected at 25 °C using Method C with a humidity ramp rate of 0.2 %RH/min. A sample mass of 1-5 mg is recommended for analysis. Figure 2 shows an example of this test for sodium bromide, while Table 1 shows additional results for various salts.

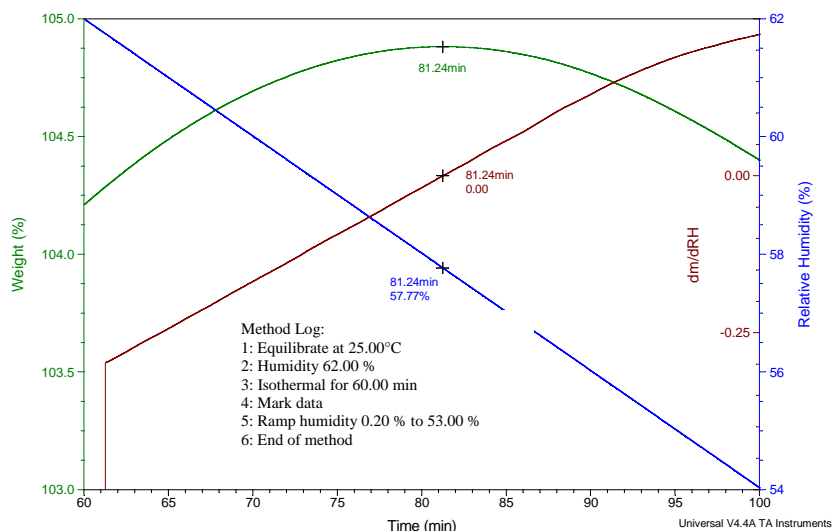


Figure 2 – Effect of Specimen Mass and Rate of Mass Change on Relative Humidity

The Q5000 SA software has a built-in humidity verification feature and deliquescence method that allows the user to choose from six different salts at six different temperatures (Figure 3). Humidity verification by using Method C is performed as described. Once the salt and temperature are selected, the humidity verification is initiated manually or automatically and unattended using the scheduling feature of the software.

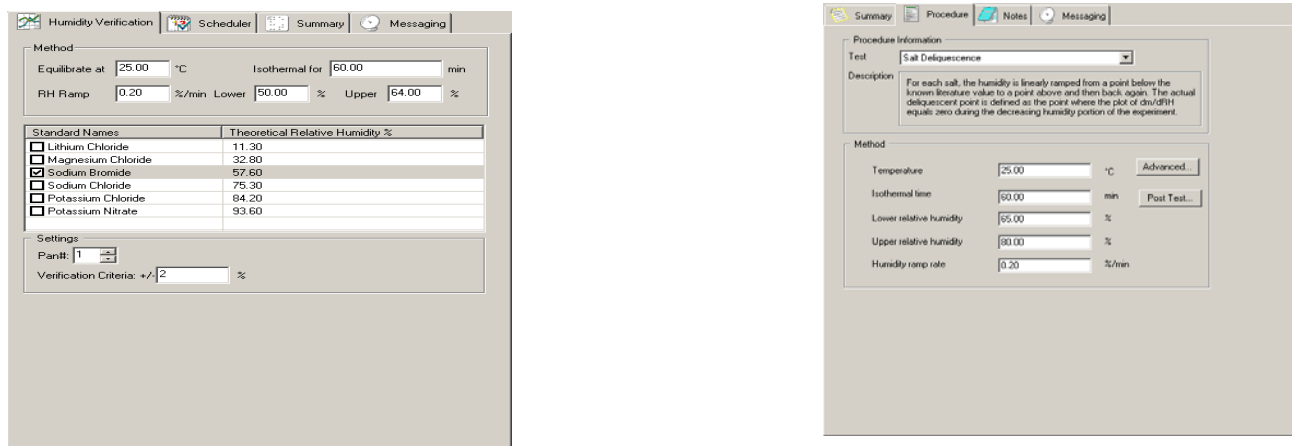


Figure 3 – Instrument Control Screens for Humidity Calibration



## CONCLUSION

The use of digital mass flow controllers of the Q5000SA design provide excellent control of the humidity without the errors induced by standard humidity sensors and their short life spans. This performance can be easily and automatically verified on a routine basis with the Q5000SA software.

## REFERENCES

1. E2551, “Standard Test Method for Humidity Calibration (or Conformation) of Humidity Generators for Use with Thermogravimetric Analyzers.” American Society for Testing and Materials International, West Conshohocken, PA.
2. L. Greenspan, “Humidity Fixed Points of Binary Saturated Aqueous Solutions”, *Journal of Research of the National Bureau of Standards - A. Physics and Chemistry*, **1977**, 81A (1), pp. 89-96.

## KEYWORDS

Calibration, Moisture, Water, Deliquescence

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