Change in Structure of a Pharmaceutical Material by Vapor Sorption Analysis

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ABSTRACT

Vapor sorption (VS) can be used to distinguish structural changes in powdered pharmaceutical materials. The moisture sorption isotherm plot can be used to calculate the stoichiometry of this change in structure. Results of an anhydrous to monohydrate formation are discussed.

INTRODUCTION

Vapor Sorption (VS) instruments are designed to measure gravimetrically the effect of moisture on materials. The tendency of a material to absorb water when being exposed to a humid environment can provide important information related to the structure of that material. This information is very useful when evaluating materials, such as pharmaceuticals for active ingredient screening and characterization.

EXPERIMENTAL & RESULTS

The TA Instruments Q5000 SA was used to aid in the characterization of a pharmaceutical powder material. The typical sample mass used for VS experiments is from 1 to 10 mg, since this reduces experimental equilibration time. The temperature of the Q5000 SA was set to 25 °C and the relative humidity (RH) method was programmed as shown in Figure 1. Once the sample (5.5 mg) was loaded, the humidity was held at 50 % for up to 6 hrs (data not shown). Then the sample was exposed to humidity that was increased and decreased in 5 % increments between 50 % and 75 %RH. The sample mass increased approximately 15.5 % upon the first stepped increase in humidity to 75 %RH (Figure 1). When the humidity was decreased back to 50 %RH, the sample mass decreased only 10 % and did not return to the original weight. Another sequence of increased humidity shows the material gaining back this 10 % weight loss.

Figure 2 shows the moisture isothermal plot of the data presented in Figure 1. From the plot it is evident that the sample gained little mass on the first humidity increase (blue curve) until the humidity surpassed 65 %RH. First suspicions would indicate the original material is in a crystalline state. However, after desorption (red curve) the material retains 5 % mass due to water, which is a good indication that the material formed a hydrate. From the molecular weight of the material, the stoichiometry of the hydrate can then be calculated. Here the material is assumed to form a monohydrate,

further proof for which is given by the second adsorption (green curve). The sample mass profile increased in the same manner as the desorption profile, which is a good indication that the original material has indeed changed form.

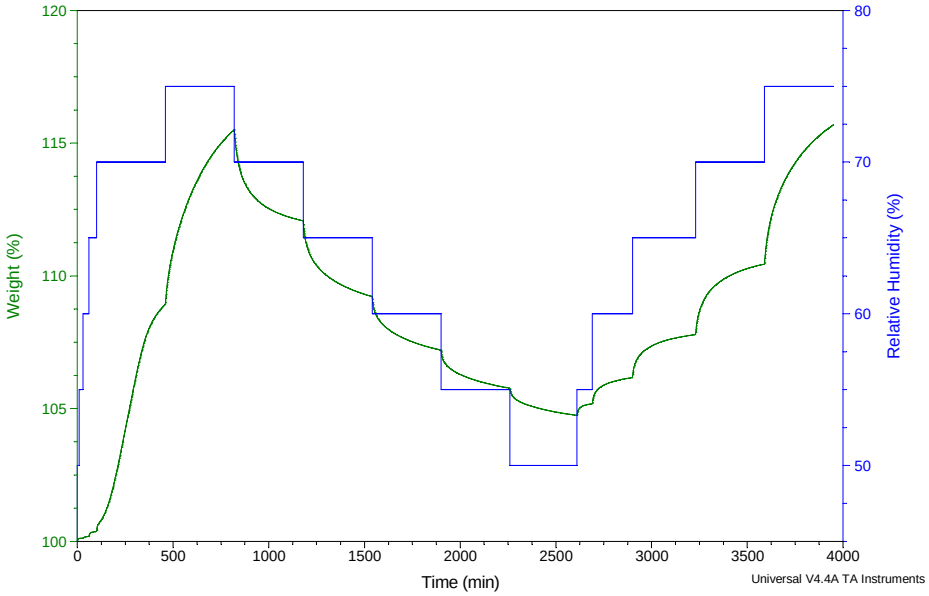

Figure 1: VS plot of API

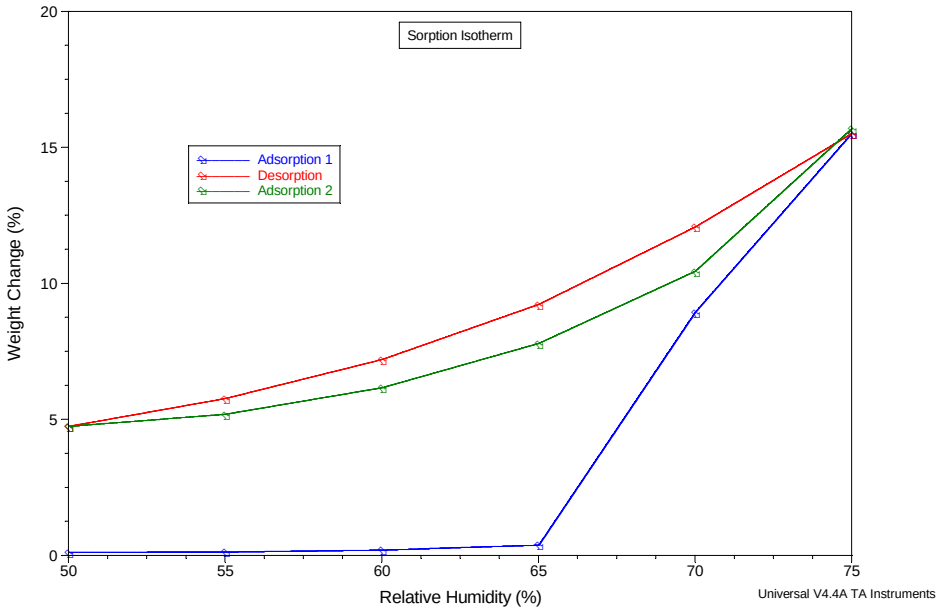

Figure 2: Moisture Sorption Isotherm plot of API

CONCLUSIONS

Vapor sorption experiments using the Q5000 SA represent an important analytical technique for the characterization of pharmaceutical powders. Calculations from the moisture sorption isotherm plot can be used to deduce structural information.

REFERENCE

1. Moisture Sorption Analysis of Pharmaceuticals, Technical Application Note 329 (TA 329)

KEY WORDS

Q5000 SA, VS, Vapor sorption, Moisture sorption isotherm, Adsorption, Desorption, Humidity, Relative humidity, Pharmaceuticals, API, Active ingredients

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