B-XRD1021 - Phase changes of pharmaceuticals as a function of temperature and humidity

Introduction

The crystal systems of pharmaceuticals and foods may change due to factors such as temperature and humidity. The climate of Japan in particular exhibits extreme changes in temperature and humidity, with hot and humid summers, and dry, low-temperature winters, and these are poor conditions as an environment for synthesizing pharmaceuticals or storing foods. Therefore, there is a need to conduct measurement beforehand under various atmospheric conditions, and determine what sort of changes these materials undergo in the actual environment. Thus we evaluated thermal changes and changes in the crystal structure of pharmaceuticals by simultaneously measuring X-ray diffraction (XRD) and differential scanning calorimetry (DSC) while varying humidity.

Measurements and results

It is possible to simultaneously determine thermal changes and changes in crystal structure by simultaneously measuring XRD and DSC. Therefore, we measured carbamazepine—a substance known as a therapeutic drug for epilepsy. Carbamazepine exhibits an endothermic reaction immediately before the melting point, and is known to transition to a high-temperature phase. Figure 1 shows the result of heating in a high-humidity atmosphere (water vapor partial pressure 8.6 kPa). It was found that, at around 70°C, there is a change from the low-temperature phase to low-temperature phase 1, then, via a small endothermic reaction at around 145°C, to low-temperature phase 2, and finally to the high-temperature phase immediately before the melting point. By measuring while varying the atmosphere in this way, it was found that a new crystal phase appears[1].
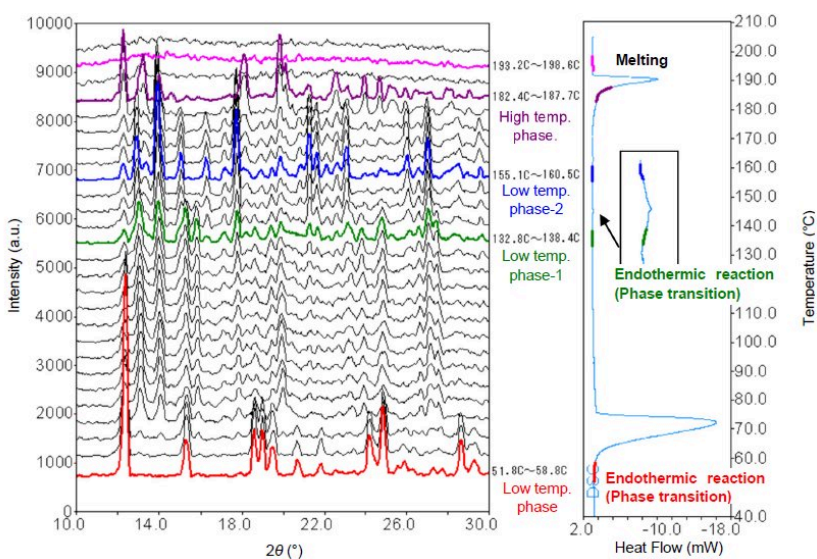
Figure 1: Results of simultaneous XRD-DSC measurement of carbamazepine (in a high-humidity atmosphere)

Reference:

(1) Jun Han, Geoff G.Z. Zhang, Devalina Law, Weili Wang, Michelle A. Long: The 31st NATAS meeting, Sep. 22-24, 2003.

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