1. Introduction
Thermal analysis is often used in the research and testing of pharmaceuticals. This brief presents an example of TG/DTA and DSC measurements of caffeine and anhydrous caffeine.

2. Measurements
2-1 Water of crystallization
Figure 1 shows the TG and DTG curves for caffeine and anhydrous caffeine. Samples weighing 10mg were measured under a nitrogen flow of 100ml/min at a heating rate of 10°C/min. The results show that the water of crystallization of caffeine desorbed and evaporated at approximately 80°C and was about 5% of the total weight. Furthermore, the anhydrous caffeine did not show a weight decrease until about 200°C, indicating that it does not contain any water.

Figure 1  TG and DTG curves for Caffeine and Anhydrous Caffeine

-------- Caffeine
----- Anhydrous caffeine
Figure 2 and Figure 3 show the DSC results for caffeine and anhydrous caffeine when measured in an open container and a sealed container, respectively. The sample weights were approximately 3mg and the heating rate was 10°C/min. In the open container results in Figure 2, there are peaks at 78°C and 86°C corresponding to the desorption and evaporation of the water of crystallization for the caffeine. The results in Figure 3 show an endothermic peak at 78°C, even though the container was sealed to control the evaporation of water. These results indicate that the 78°C peak for caffeine in Figure 2 is due to the desorption of water of crystallization and the 86°C peaks are due to the evaporation of the water.

2-2 Melting

In Figure 2, caffeine and anhydrous caffeine show an endothermic peak at 236°C due to melting. At the same time, the TG curve in Figure 1 shows a decrease in weight around the melting point. These results indicate that water continues to rapidly evaporate as the caffeine melts. The anhydrous caffeine showed a melting peak at 236°C for both the open and sealed conditions but the caffeine melting peak in the sealed condition was smaller and occurred at a lower temperature than the open condition. Likely, the water of crystallization that desorbed from the caffeine remained in the sealed container and this affected the crystal structure of the caffeine.