

Polymorphism in Dotriacontane

1. Introduction

It is known that past heat treatments (thermal history) of samples produce crystal polymorphism in long-chain hydrocarbon compounds. Organic compounds exhibiting crystal polymorphism frequently appear in pharmaceuticals and are an important factor in determining the effectiveness of pharmaceuticals.

In this brief, dotriacontane samples ($\text{CH}_3(\text{CH}_2)_{30}\text{CH}_3$) with various thermal histories are measured to provide an example of DSC applications for crystal polymorphism.

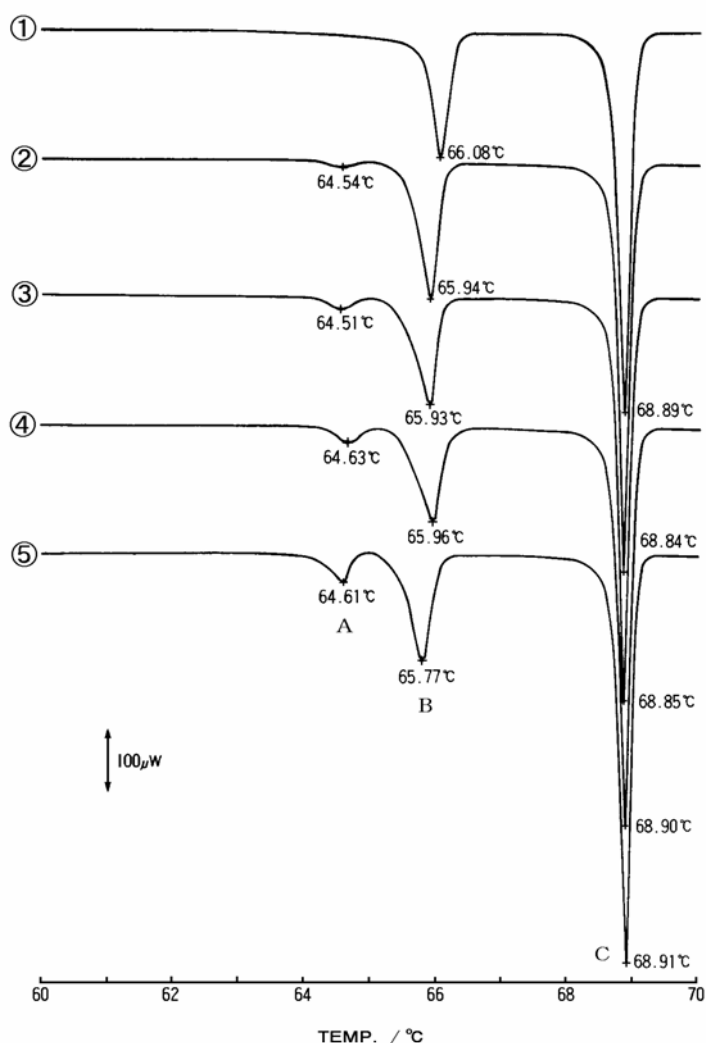


Figure 1 DSC Curves of dotriacontane

- ① 1st run
- ② Cooling rate : 0.05°C/min
- ③ Cooling rate : 0.5°C/min
- ④ Cooling rate : 1°C/min
- ⑤ Cooling rate : 10°C/min

2. Measurements

The samples were commercially-available dotriacontane.

For the measurements, a DSC200 High-sensitive Differential Scanning Calorimeter was connected to a SSC5000 Disk Station.

The sample weight was approximately 0.1mg. The samples were heated between 60°C and 70°C at a heating rate of 0.5°C/min.

3. Results

Figure 1 shows the DSC results for dotriacontane. Number ① is the result for the sample as purchased. Numbers ② to ⑤ are the results for the treated samples. Samples were heated until melting was completed and then cooled at various rates (0.05, 0.5, 1 and 10°C/min for numbers ② to ⑤, respectively). There are 2 peaks for number ① but 3 peaks for numbers ② to ⑤. The peak temperatures ascended from A to B to C. Figure 2 shows the dependence of heat of transition on cooling rate for each peak.

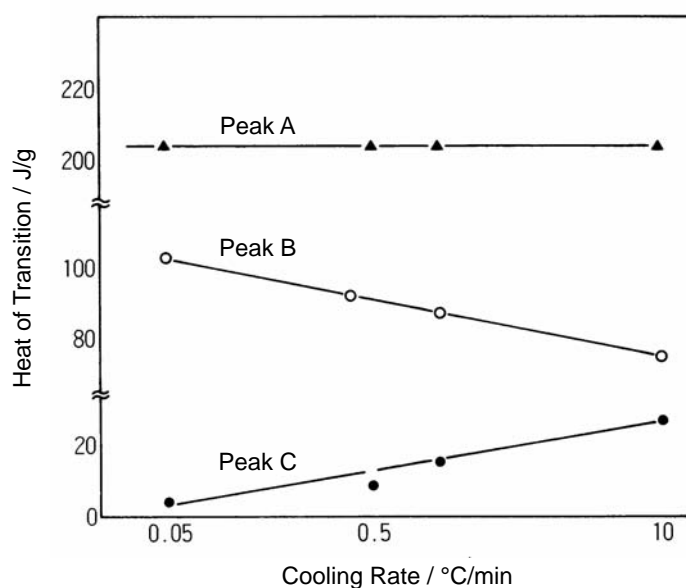


Figure 2 Dependence of Heat of Transition on Cooling Rate in Dotriacontane

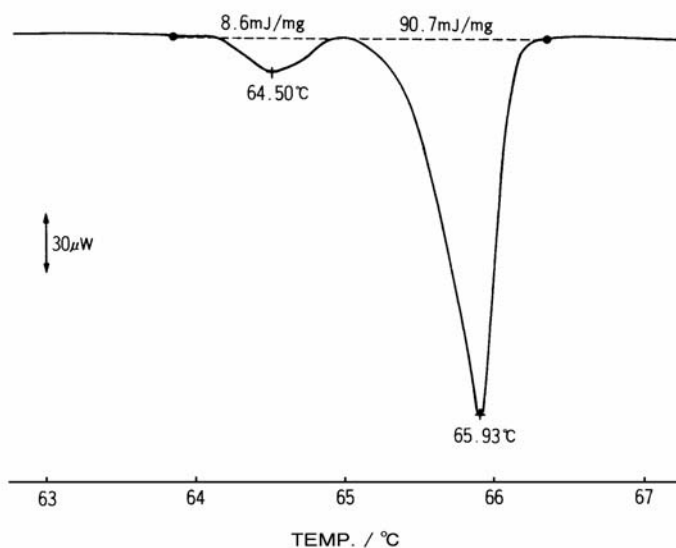


Figure 3 DSC result for dotriacontane with a cooling rate of 0.5°C/min

This data shows that the faster the cooling rate, the greater the increase of metastable crystals, shown by transition A, and the greater the decrease of crystals, shown by transition B.

Figure 3 shows an enlarged version of peaks A and B for the 0.5°C/min cooling condition. This data shows that the DSC200 accurately measures peaks at sample weights as low as 0.1mg.

4. Summary

In this brief, the effects of thermal history on crystal polymorphism in dotriacontane were investigated as an example of crystal polymorphs measurement using DSC. DSC can accurately investigate the relationship of crystal polymorphism and thermal history because it can accurately identify minute changes caused by different crystal polymorphs, as well as measure heat.