坩埚对一水葡萄糖失水的影响

Influence of the Pan on Dehydration, Glucose Monohydrate

试样 α-D-一水葡萄糖

Sample α-D-Glucose monohydrate

应用 非活性成分，药片和胶囊的填料

Application Inactive ingredient, filler for tablets and capsules

条件 测试仪器：DSC和TGA

Conditions Measuring cell: DSC and TGA

DSC坩埚：40μl铝坩埚，密封或盖钻孔。

Pan DSC: Al 40μl, hermetically sealed or with pierced lid.

TGA坩埚：100μl铝坩埚，盖钻孔。

Pan TGA: Al 100μl, with pierced lid.

试样制备：原样品

Sample preparation: As received

DSC 测试：以20K/min由30°C升温至250°C。

DSC Measurement: Heating from 30°C to 250°C at 20 K/min.

TGA 测试：以20K/min由30°C升温至300°C。

TGA Measurement: Heating from 30°C to 300°C at 20 K/min.

气氛：氮气，DSC：50 ml/min，TGA：80 ml/min

Atmosphere: Nitrogen, DSC: 50 ml/min，TGA: 80 ml/min

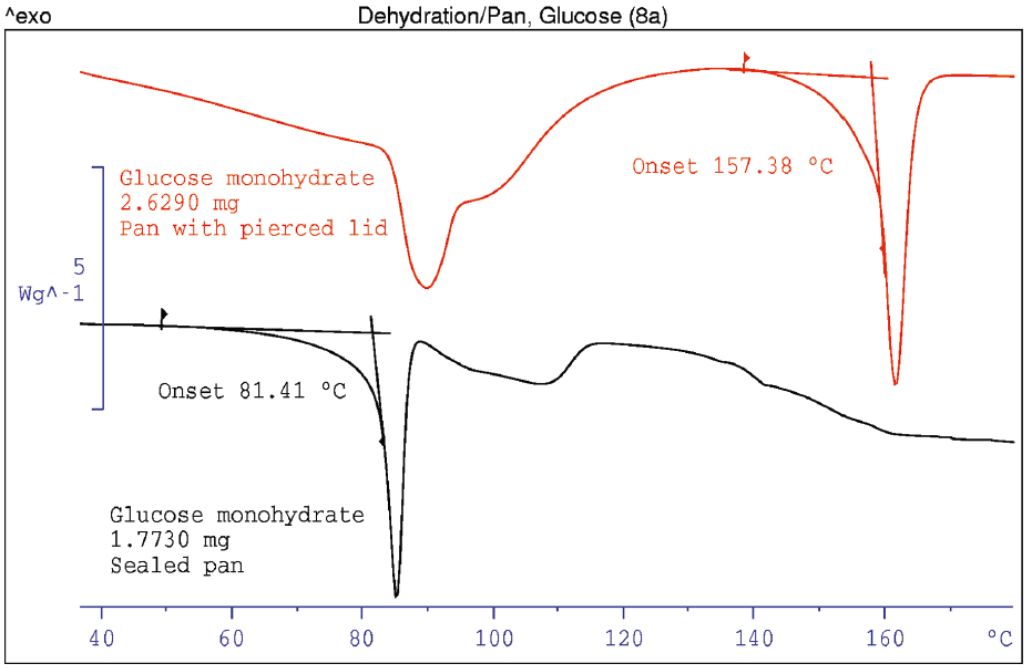

图 3.7　α-D-一水葡萄糖在钻孔盖坩埚(上)和密封坩埚(下)中测试得到的 DSC 曲线

Figure 3.7 DSC curves of α- D-Glucose monohydrate measured at pan with pierced lid (above) and sealed pan (below)

解释

Interpretation

图 3.7 所示α-D-一水葡萄糖两条 DSC 曲线的比较，表明了试样在密封坩埚或在盖钻孔的坩埚中测试时产生的变化。如果用盖钻孔的坩埚，结晶水可以逃逸，这从测试开始时 DSC 曲线的移动和宽泛的蒸发峰可明显观察到。同时，出现向β-D-无水葡萄糖的转变，它的熔点约为 158°C。200°C 以上，葡萄糖开始熔化变焦。

A comparison of the two DSC curves of α-D-Glucose monohydrate in Fig. 3.7 shows the changes that arise when the sample is measured in a sealed pan or in a pan with a pierced lid. In a hermetically sealed pan the sharp melting peak of the monohydrate can be observed. If a pan with a pierced lid is used, the water of crystallization can escape. This is noticeable as a shift of the DSC curve at the beginning of the measurement and as a broad evaporation peak. At the same time, a transition to β-D-Glucose anhydrate occurs, the melting point of which is at about 158 °C. Above 200 °C the glucose starts to caramelize.

DSC 计算

Evaluation DSC

测试条件 Measuring conditions	起始点 Onset °C	效应 Effect

密封坩埚 Sealed pan	81.4	熔融
盖钻孔的坩埚 Pan with pierced lid	157.4	熔融

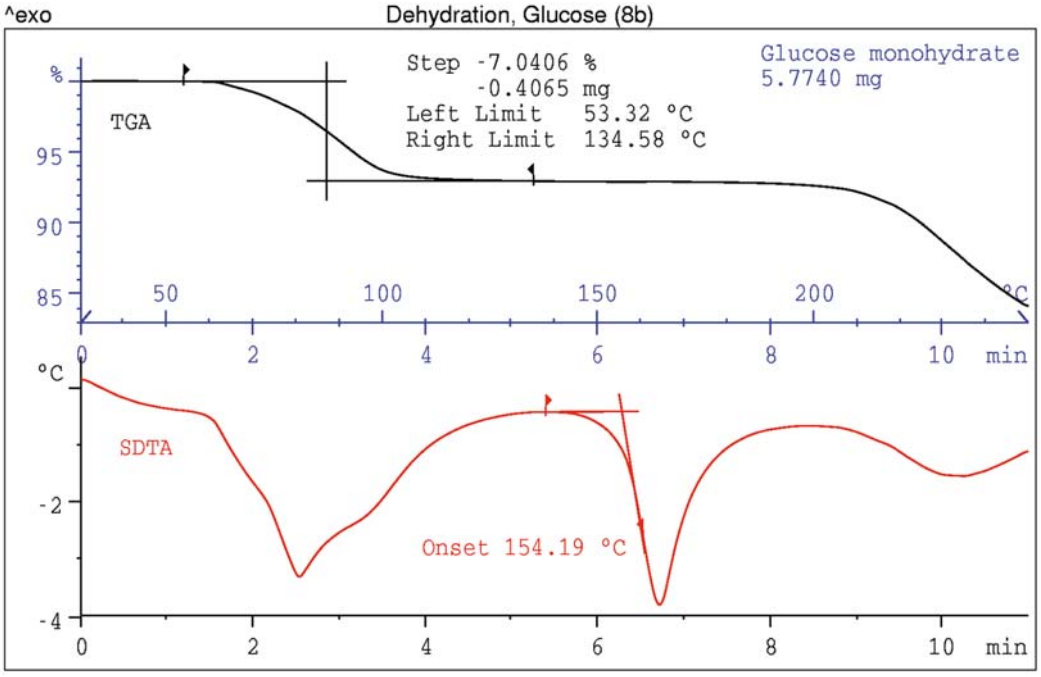

图 3.8　α-D-一水葡萄糖在钻孔盖坩埚)中测量的 TGA 和 SDTA 曲线

Figure 3.8 TGA and SDTA curves of α-D-Glucose monohydrate measured at a pan with pierced lid

解释

Interpretation

图 3.8 为α-D-一水葡萄糖的 TGA 和 SDTA 曲线。在盖钻孔的坩埚中的热重测试，与由 DSC 曲线得到的结果的解释一致，尤其是由结晶水蒸发产生的失重以及随后的β-D-无水葡萄糖的熔融。53°C 至 134°C 间 7%的失重台阶比化学计量值略低，不过，可解释为样品贮存期间结晶水的损失。

Figure 3.8 shows the TGA and SDTA cures of α-D-Glucose monohydrate measured at a pan with pierced lid.

Thermogravimetric measurements using a pan with a pierced lid confirm the interpretation of the results obtained from the DSC curves, in particular the weight loss caused by the evaporation of the water of crystallization as well as the melting of the β-D-Glucose anhydrate afterwards. The weight loss step of 7% between 53 °C and 134 °C is somewhat less than that expected stoichiometrically. It can be explained however by a loss of water of crystallization during storage of the sample.

TGA 计算
Evaluation TGA

	温度 Temperature °C	效应 Effect
TGA 台阶 TGA step	53~134	7%失重(结晶水) 7.0% weight loss (water of crystallization)
SDTA 起始点 SDTA onset	59	吸热峰 endothermic peaks
SDTA 起始点 SDTA onset	154.2	熔融峰 melting peak

结论
Conclusion

含结晶水的物质和它的无水形式通常具有不同的熔点(假多晶型)。如果含结晶水的形式不发生分解，则可在密封坩埚中测定其熔点。在敞口坩埚中，结晶水可逃逸，因而测得的是无水形式的熔点。始终应该通过测量失重来确认含结晶水形式的存在。
A substance that contains water of crystallization and its anhydrous form normally have different melting points (pseudopolymorphism). The melting point of the form containing the water of crystallization can be determined in a hermetically sealed pan, provided that no decomposition occurs. In an open pan the water of crystallization can escape so that the melting point of the anhydrous form is measured. The presence of a form with water of crystallization should always be confirmed by measuring the weight loss.