

升温速率对美托拉脞分解的影响

Influences of Heating Rate on Decomposition, Metolazone

试样 美托拉脞

Sample Metolazone

应用 活性成分(利尿剂)

Application Active ingredient (diuretic)

条件 测试仪器: DSC

Conditions Measuring cell: DSC

坩埚: 40 μ l铝坩埚, 盖钻直径1mm的孔。

Pan: Al 40 μ l, with pierced lid with a hole of 1 mm diameter.

试样制备: 原样品

Sample preparation: As received

测试: 以2和5K/min由20 $^{\circ}$ C升温至200 $^{\circ}$ C。两次测试均进行空白修正。

Measurement: Heating from 20 $^{\circ}$ C to 200 $^{\circ}$ C at 2 und 5 K/min. Both measurements are blank curve corrected.

气氛: 氮气, 50 ml/min

Atmosphere: Nitrogen, 50 ml/min

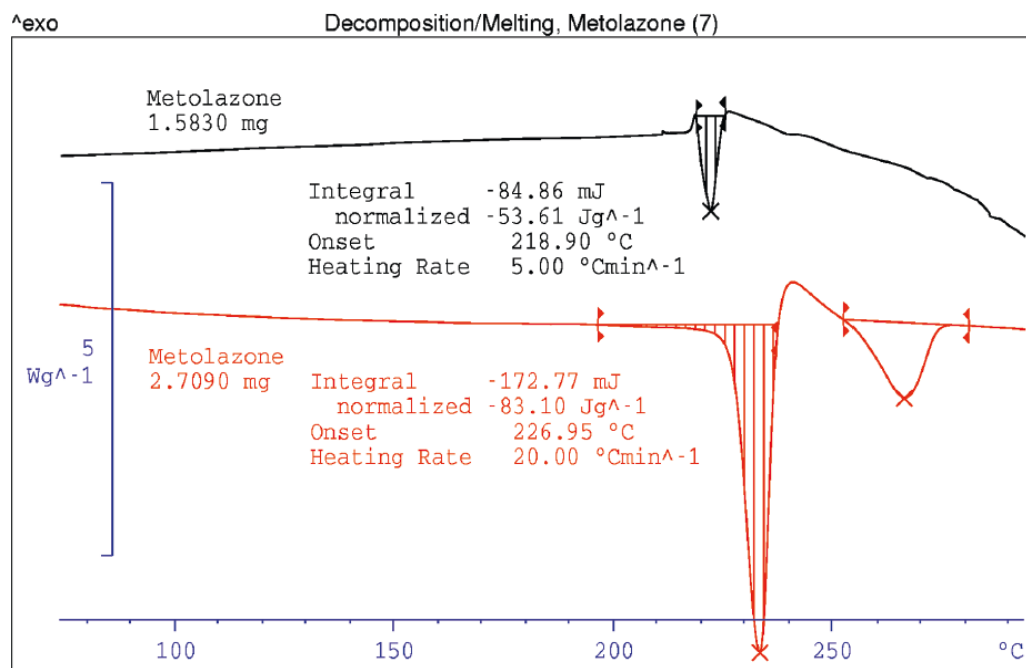


图 3.6 以两个不同升温速率测量的 α -美托拉脞 DSC 曲线

Figure 3.6 DSC curves of α -Metolazone measured at two different heating rates

解释

Interpretation

图 3.6 为以两个不同升温速率测量的 α -美托拉脞的 DSC 曲线的比较。以 20K/min，可观察到 α 晶型在约 227°C 熔融，随后是 γ 晶型的重结晶，在约 255°C 该晶型熔化，且伴有分解。

对于升温速率 5K/min，观察到一个小得多的吸热效应，熔点降低了大约 7°C。在 220°C，密封坩埚开裂(表现为基线偏移)。

The DSC curves of α -Metolazone measured at two different heating rates are compared in Fig. 3.6. At 20 K/min the melting of the α modification at approx. 227 °C followed by recrystallization in the γ modification can be observed. This melts with accompanying decomposition at approx. 255 °C.

With a heating rate 5 K/min, a much smaller endothermic effect and a depression of the melting point by approx. 7 °C is observed. At 220 °C the hermetically sealed pan bursts (shown as a displacement of the base line).

计算 Evaluation

仅计算吸热峰。选择“左水平”或“右水平”基线进行峰积分。

Only the endothermic peaks are evaluated. To integrate the peaks the baseline types 'horizontal left' or 'horizontal right' are selected.

升温速率	起始点 1	ΔH	起始点 2	ΔH
Heating rate	Onset 1	J/g	Onset 2	J/g
K/min	$^{\circ}\text{C}$		$^{\circ}\text{C}$	
5	218.9	53.2	-	-
20	227.0	83.1	255.1	31.0

结论 Conclusion

DSC 对在熔点处分解的物质只能有限表征。这时，用较快的升温速率常常可获得再现性较好的表征，因为分解反应移至较高温度。相同的方法也可用于 TGA，以得到更多的信息，尤其是当生成挥发性分解产物时。

Substances which decompose at the melting point can only characterized to a limited extent by DSC. In such cases, a more reproducible characterization is often possible by using faster heating rates, since the decomposition reaction is moved to higher temperatures. TGA can also be used in the same way to gain additional information, especially when volatile decomposition products are formed.