

Purity Determination by DSC

INTRODUCTION

The Van't Hoff method allows for the determination of the purity of substances based on a DSC curve. An example of this method is introduced in the present document, based on samples of Phenacetin with certified impurity levels and provided by NIST. Check the application note "ANS006 - Purity Determination by DSC – 1 (Theory)" for more details about the mathematics involved and the limitations of the method.

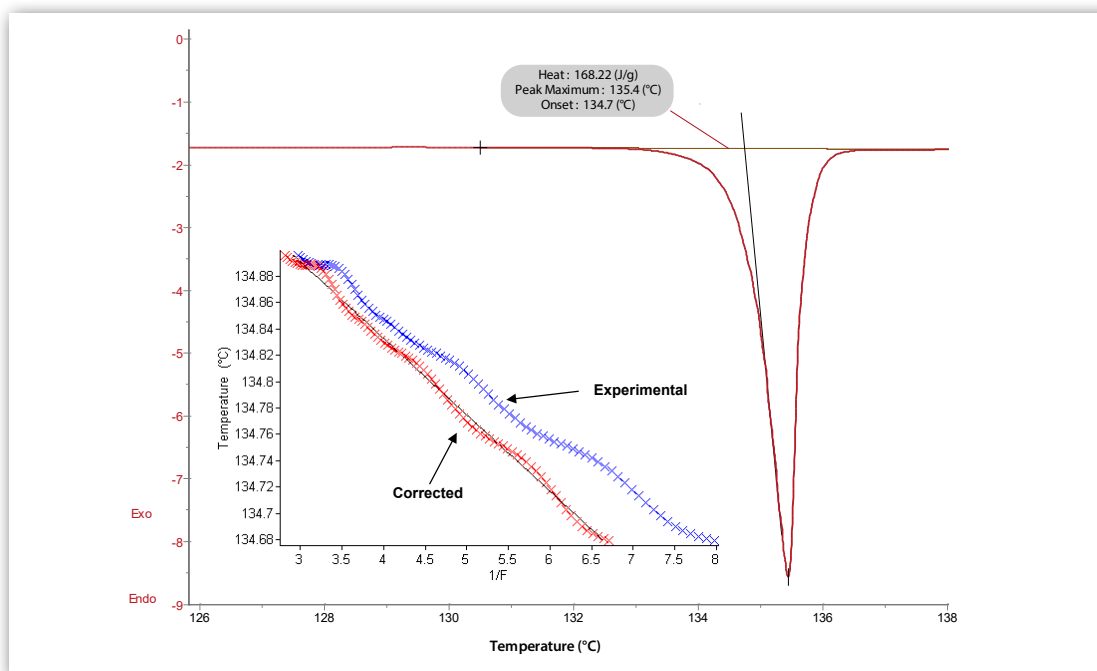


Figure 1 - Purity determination of 'pure' phenacetin by the Calisto software tool

EXPERIMENT

Sample:

Set of certified Phenacetin samples with different purity rates (pure, 99.3%, 98% and 95%)

Experimental conditions:

- Atmosphere: Nitrogen, atmospheric pressure
- Sample mass: About 2 mg in a 30µl aluminum crucible sealed with a lid.
- 110°C ➔ 150°C at 1°C/min

RESULTS AND CONCLUSION

The green curve on Figure 1 corresponds to the melting peak of the certified "pure" Phenacetin sample. The inset plot represents the experimental function $T_s = f(1/F)$ and the corrected function by the SONDACK method.

$T_o = 135.07^\circ\text{C}$

Correction $K = 9.31$ mJ or 2.78 %

Slope = -0.06°C

Molar purity = 99.87%

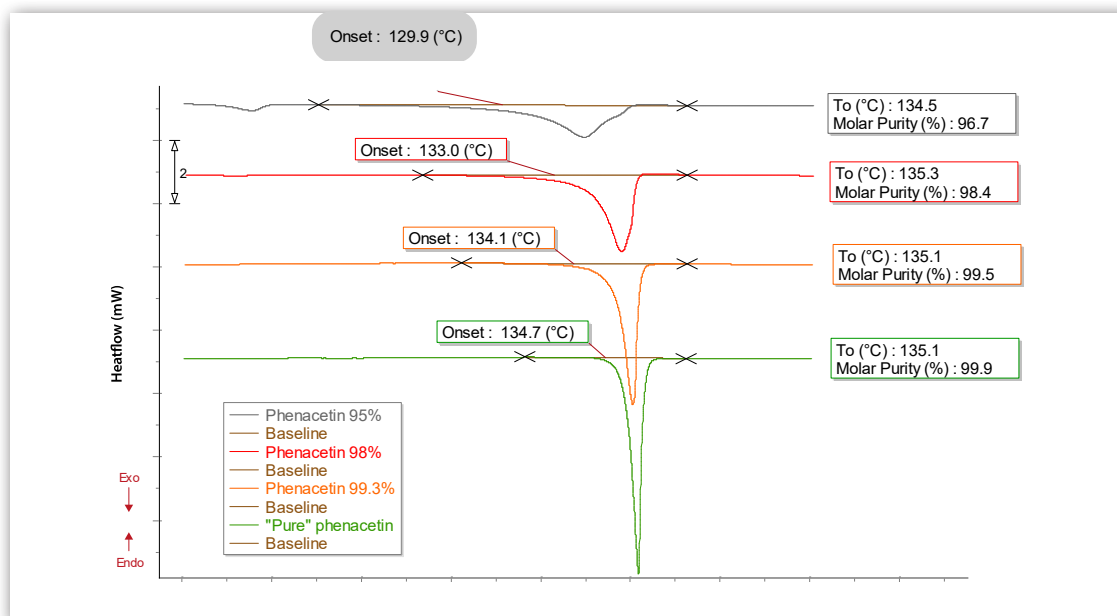


Figure 2 - Melting peaks superposition of phenacetin samples at different purity ratios

RESULTS AND CONCLUSION

Figure 2 corresponds to the melting peaks of all the certified Phenacetin samples.

The purity determination function of Calisto Data Processing software automatically applies the Van't Hoff relationship and the SONDAK correction method, leading to the evaluation of the molar impurity percentage of the tested Phenacetin samples. Check the application note "ANS006- Purity Determination by DSC – 1 (Theory)" for more details.

When the impurity rate increases, the melting peak is spread over a larger range of temperature.

On the thermogram corresponding to phenacetin with 5% impurity, the eutectic melting peak is observable prior to the melting of phenacetin. In this case, the purity measurement is much less precise.

This observation, and the comparison of the certified purity values vs. the values obtained by DSC, can confirm that this method is accurate for relatively pure compounds (less than 2% of impurity).

INSTRUMENT

SETLINE DSC / DSC+

-170 to 700°C



EASY TO USE WITH ROBUST SENSOR TECHNOLOGY
ensuring quality, consistent and reliable data

AVAILABLE WITH HIGH PRESSURE CRUCIBLES
up to 500 bar at 600°C

REASONABLY PRICED INSTRUMENT & SENSOR
for easy, cost effective replacement

LOWER COST OF OWNERSHIP
through simplified maintenance and a Replacement Parts Guarantee

TECHNICAL & APPLICATION SUPPORT
for fast expert help with any questions

CALISTO 2.0 EXCLUSIVE SOFTWARE
for intuitive and easy data handling