TA NO.27 MAR.1986   Purity Determination of Phenacetin

1. Introduction

Measuring the melting point of a material, identifying that material and qualitatively checking the purity has been performed for a long time. Recently, the DSC method has been used to determine the purity of high purity materials using small samples of 1 ~ 3 mg in a short period of time.

This application brief shows the results of Purity Determination of a Standard Reference Material for DSC (No.1514 Phenacetin) through NBS.

2. Purity Determination Calculation Method

Based on the system of impurities melting to liquids and not melting to solids (solids solution are not produced), think of the leading van`t Hoff equation (1) using the Raoult law.

$$T_S = T_0 - \frac{RT_0X}{\Delta Hf} \; \frac{1}{F} \qquad (1)$$

T_S : Observed sample temperature
T_0 : Fusion temperature of pure main components
R : Gas constant
X : Molar fraction of impurities
Hf : Fusion heat of main components
F : Fusion fraction for temperature T_S

In equation (1), T_S is measured as the F function. If T_S is plotted for 1/F, X is obtained from that slope. In other words, Purity $P = 100 \times (1-X)$. However, the following corrections are necessary to calculate purity from DSC data.

1) Heat Resistance

It is necessary to correct the slowness caused by heat resistance of the DSC calculation system. To correct, measure the fusion curved lines of high purity metal samples beforehand and use the slope of that data to calculate temperature.

2) Area Correction

When calculating fusion fraction F, the peak area calculated from DSC data is under evaluated because the melting start is not clear. Because of this, the plot of Ts and 1/F is plotted as a curved line, not a straight line. In order to correct this effect, the trial and error method is used and area b is added to the excess. F is then calculated and the 1/F and Ts plot becomes a straight line. (The % of correction is for the complete area of correction area b.)

3. Results

Figure 1 shows the DSC data of the different purity of Phenacetin. From the figure, we learn that as the purity lessens, the peak broadens and the melting start temperature and the peak top temperature also decrease.

Figure 2 shows the purity calculation results from data processing using a sample with a purity of 99.3%. Table 1 shows the results from the purity calculations for the standard samples.
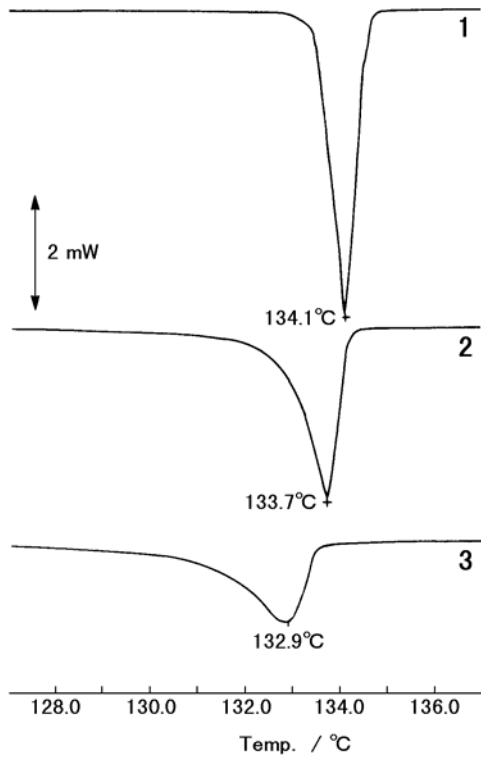
Figure 1   DSC Curve of Phenacetin
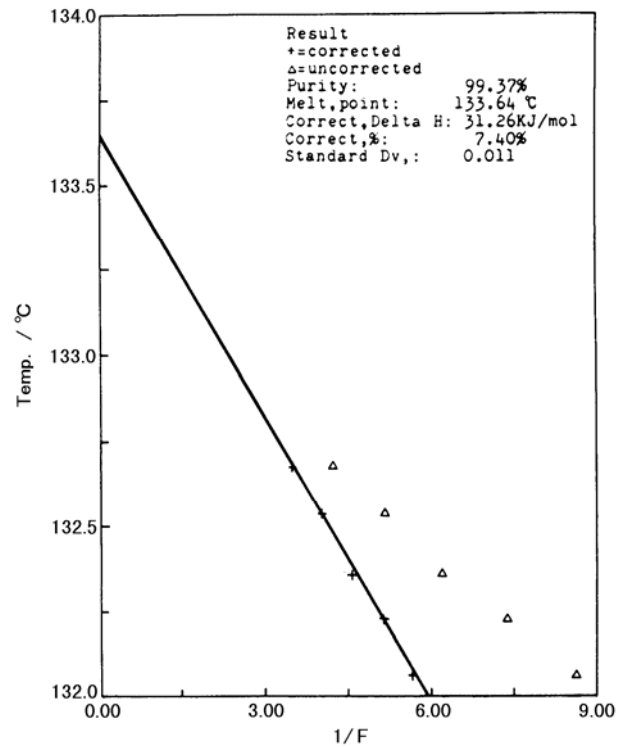Purity   1 : 100%
2 : 99.3%
3 : 98%

Figure 2   Purity Calculation Result

Table 1   Purity Calculation Result of Phenacetin

Purity	Guarantee Range (M%) of Impurity Concentration	Calculation Result	
		X (n=5)	Σ_{n-1}/n
100%	0.00	99.94%	4.5×10^{-5}
99.3%	0.67 ± 0.07	99.37%	4.5×10^{-5}
98%	1.91 ± 0.12	98.05%	4.9×10^{-3}