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DSC Measurements of Mixed Water/Ethanol Solutions

1. Introduction

Since applications for estimating the maturity of whiskey were reported[1, 2], further research has been done regarding the DSC measurement of water and ethanol solutions[3].

The method involves lowering the temperature of the water and ethanol solution to -100°C or lower, heating it a constant rate and observing the melting peaks. According to these reports, in addition to ethanol (around -62°C) and water (around -40°C) melting peaks in the DSC curve during heating, there is a water/ ethanol compound endothermic peak between -60 and -50°C. The size of this peak is dependant on the maturity of the solution.

In this brief, DSC and an automatic gas cooling unit are used to measure a water and ethanol solution. The cooling unit injects vaporized gas of liquefied nitrogen into the furnace area. Due to its configuration, measurements during the raising and lowering of the temperature are performed automatically and the temperature is controlled accurately. Accordingly, this system is extremely effective when the measurement temperature is raised and lowered in the minus range, as in this measurement example.

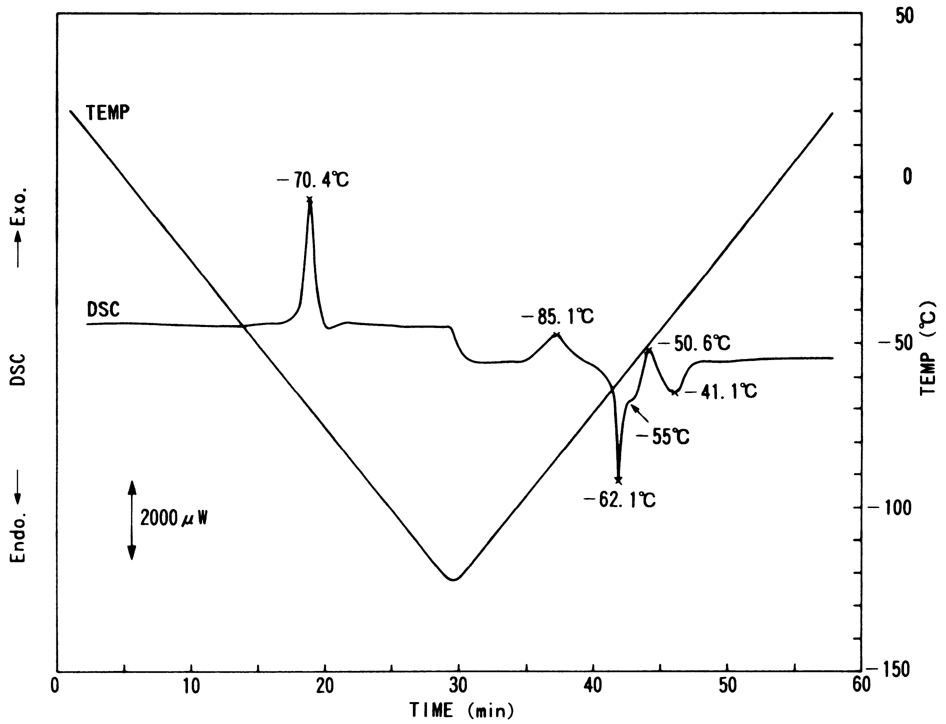

Figure 1 DSC Curve for water / ethanol solution during cooling and heating

2. Measurements

The measurement sample was a water and ethanol solution with 60% ethanol.

An automatic gas cooling unit was connected to the DSC220 High-sensitive Differential Scanning Calorimeter for the measurements.

Samples weighing 5mg and were measured in a simple hermetic aluminum container.

3. Results

Figure 1 shows the measurement results when the temperature of water and ethanol solution was cooled and heated. This is the DSC curve when the sample was cooled from room temperature to -130°C and then heated back to room temperature at a rate of 5°C/min. In the heating process, the ethanol melting peak appeared around -62°C and the water melting peak appeared around -41°C. Furthermore, the melting of the water and ethanol compound was observed as a shoulder peak at around -55°C. The exothermic peaks at -85°C and -50°C are considered to be the recrystallization peaks of the ethanol and water, respectively.

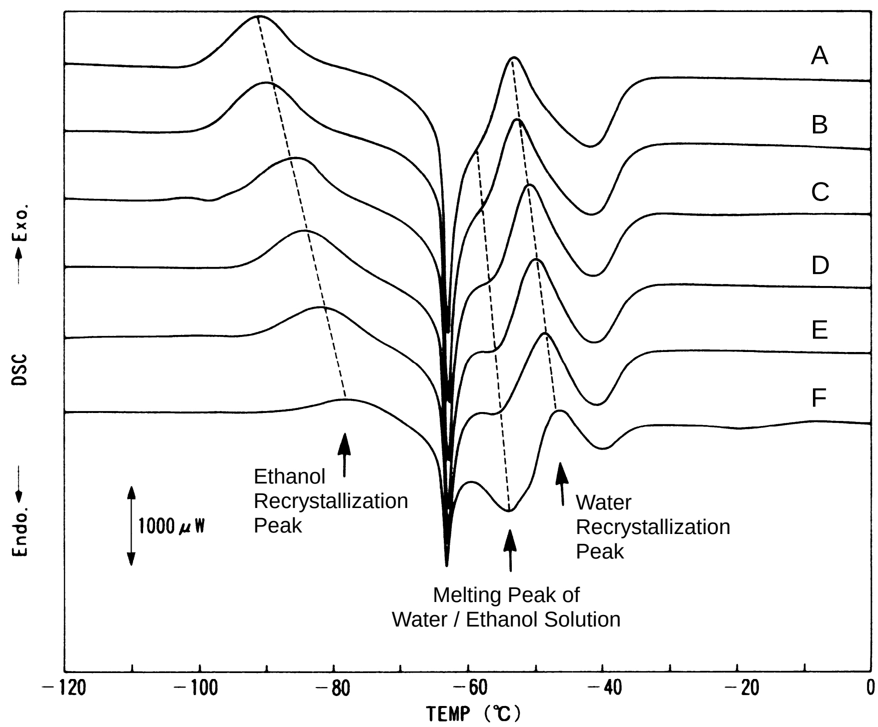

Figure 1 Cooling Rate Dependence in DSC Measurements of Water / Ethanol Solutions
Cooling rates before measurement
A 20°C/min
B 10°C/min
C 5°C/min
D 2°C/min
E 1°C/min
F 0.5°C/min

Figure 2 shows the DSC curves for samples that were cooled from room temperature at different rates and then heated at 5 °C /min. The data shows that the peak shapes and temperatures at the areas indicated by the arrows differed depending on the cooling rate before measurement. Furthermore, the melting peak of the water and ethanol compound seen around -50 °C was grew as the cooling rate lowered.

This indicates that the cooling rate before measurement is an important factor in the DSC measurement of water and ethanol solutions. When multiple samples are compared, it is important that cooling is performed at a uniform rate.

References

1) K.Koga and H.Yoshizumi, Differential Scanning Calorimetry (DSC) Studies on the Structures of Water-Ethanol Mixtures and Aged, J.Food Sci., **42**, 1213 (1977)
2) K.Koga and H.Yoshizumi, Differential Scanning Calorimetry (DSC) Studies on the Freezing Processes of Water-Ethanol Mixtures and Distilled Spirits, J.Food Sci., **44**, 1386 (1979)
3) R.Akaboshi and K.Okuma, Nippon Nogeikagaku Kaishi , **59**, 1 (1985)