DSC Laboratory Experiment – Determining the Thermal Properties of Organic Hydrocarbons

Purpose

The purpose of this laboratory is to introduce the primary concepts of organic materials. This lab will include:

1. An introduction to the techniques involved in using a Differential Scanning Calorimeter (DSC)
2. How to determine melting point (T_m) and the heat of fusion (ΔH_{fusion}) of various hydrocarbons (Octadecane, Dodecane, Cyclohexane)
3. Calculation of the entropy of fusion (ΔS_{fusion}) of each compound
4. Establish the relationship between the hydrocarbon's molecular weight and the ΔS_{fusion}
5. Comparison of experimental results to NIST standards

Materials

Chemicals of 99% purity are to be obtained from Sigma Aldrich.

Procedure

A Q250 DSC is utilized under a nitrogenous flow at 20 cm^3/m to prevent oxidation of the samples. In order to obtain sufficient results, 5 to 15 mg of each sample is weighed on an analytical balance. The samples are weighed at room temperature and placed in hermetic DSC pans. The pans are then crimped to seal the liquid. The samples are ran under a heat/cool/heat cycle at 20°C/min three times (-20°C to 60°C) to erase the sample's thermal history. After the completion of the last thermal cycle the samples are then subjected to a temperature ramp (-20°C to 60°C) at which point the data is collected.

Analysis

The melting point of each sample is determined by finding the onset of the heating curve (Figure 1). The heat of fusion is calculated by the software by measuring the area under the curve.

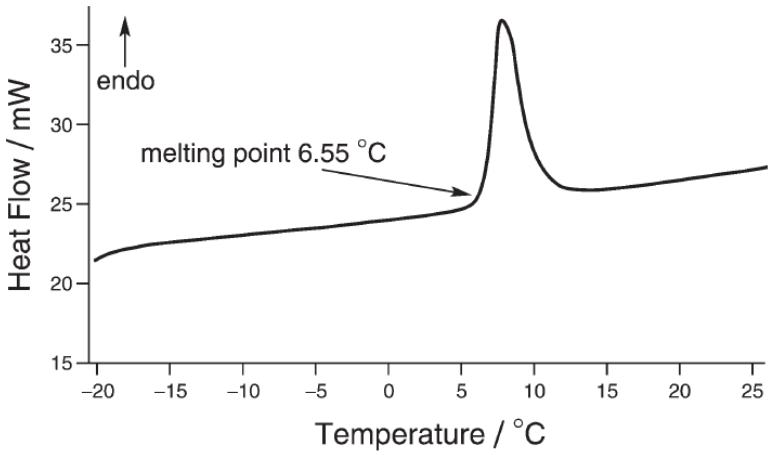

Figure 1. An example of a DSC thermogram of pure cyclohexane. (Ronald D'Amelia, 2007)

The entropy of fusion is calculated by utilizing the following equation:

$$\Delta S_{fusion} = \frac{\Delta H_{fusion}}{T_m}$$

The experimentally obtained results are compared to the NIST data (table 1) for each sample. From this comparison, the procedural efficacy can be determined for each student/group by calculating the error.

Sample	**T_m (K)**	**ΔH_{fusion} (kJ/mol)**	**ΔS_{fusion} (J/mol K)**
Cyclohexane	279.6	2.68	9.57
Dodecane	263.5	36.8	161.5
Octadecane	301.0	60.8	203.6
Table 1. The accepted value are from the NIST database.			

Homework

1. Determine the relationship between the entropy of fusion and the molecular weight of a hydrocarbon.
2. From the relationship established in question 1, predict the ΔS_{fusion} and ΔH_{fusion} for Octane (C_8H_{18}) and compare it to NIST data.

References

Ronald D'Amelia, T. F. (2007). Introduction of Differential Scanning Calorimetry in a General Chemistry Laboratory Course: Determination of Thermal Properties of Organic Hydrocarbons. *Journal of Chemical Education*, 453-455.