Melting Point Lab (V-A)

Background Reading

Beran J. Lab Manual for Principles of General Chemistry (10th Ed).
Experiment 14 (involves freezing/melting point depression).
Ebbing D., and Gammon S. General Chemistry (11th Ed).
Section 12.6 (freezing/melting point depression).
Zanger M., and McKee J.R. Small Scale Syntheses. pp 15-17 and 57-8.
Zubrick, J.W. The Organic Chem Lab Survival Manual.
Chapter 12 (Sample Preparation, MP Hints, and Mel-Temp).

Key Words

melting point (MP), melting range, FP/MP depression (Review notes on pages 6-7), mixed MP test, decomposition point

Experimental Data

- No reaction section or yield information is needed.
- Discuss the melting process and how purity of samples affects the melting range in the Principles section.
- Create an original labeled diagram of the instrument in the Apparatus section.
- Describe how the melting process appears to you in the Observations section.
- Make a table of experimental and reference melting point ranges for each assigned substance and mixture in the Data section.
- Compare the experimental ranges with reference values in the Conclusion.

Procedure

1. Obtain 100-200 mg of acetanilide (melting range: 114 - 116 ^{o}C). If it is not already a fine powder, then place inside of a folded piece of filter paper and grind with a clean spatula.

2. Press the open end of a capillary tube into the powder to obtain a small aliquot.

3. Hold the long glass tube vertically with the bottom end resting on the lab counter. Drop the capillary upright into the long tube several times to pack the sample into the bottom of the capillary. Ideally, the capillary should contain 2 to 3 mm of sample packed into the bottom.

4. Turn on melting point apparatus. Insert capillary tube with sample into an empty slot in the capillary guide. Observe the sample's initial appearance through the magnifying lens.

5. Enter the set point temperature as 5 ^{o}C below the compound's MP, if known. Enter the ramp rate as 1.0 to 2.0 ^{o}C per minute. The first trial for a substance may be performed with a more rapid ramp rate to determine an approximate melting range. If so, the subsequent trials would need a lower ramp rate to improve accuracy, and then those melting ranges may occur at lower temperatures than for the first trial.

   Caution – The apparatus is now hot, avoid touching the capillary or the heated block.

6. To determine MP range, record temperature when first drop of clear liquid is observed, then record temperature when no solid remains. Prior to initially melting, the sample may appear to deform (shrink, soften, or sag). Watch the sample vigilantly to avoid missing the beginning or end of the MP range. Also, decomposition may occur instead of melting, and cause a color change or gas evolution. Observe sample's appearance during entire melting process. Press the stop button or turn off unit once the melting process is complete.

7. Allow unit to cool to at least 10 ^{o}C below MP before inserting next sample. Perform at least two trials with consistent values. If sample MP is > 2 ^{o}C below tabulated value, the sample may either be contaminated (causing MP depression) or have a different identity. If MP is above tabulated range, the sample may have been heated too quickly.

8. Repeat the procedure for urea (132 − 134 ^{o}C), benzoic acid (122 − 123 ^{o}C), 2-naphthol (121 − 123 ^{o}C), and the mixture of 2-naphthol (121 − 123 ^{o}C) and benzoic acid. The melting range for the mixture will be well below that of either pure compound. Enter an initial set point of 80 ^{o}C for the mixture.

Waste Disposal

Place all used chemicals in the appropriately-labeled waste container(s).
Place all used capillary tubes in the broken glass container.
Never reuse a sample in a capillary tube, as that sample
may have become degraded, decomposed, or contaminated.

Post-Lab Q's

1. Describe how to determine not only if a sample is a suspected chemical substance, but also if it is relatively free of impurities. Refer to key words.

2. Describe the potential consequence of heating a sample too rapidly. What happens to the melting range?

3. Describe the potential consequence of reusing a previously melted sample.

4. What visual observations can be expected if the sample is decomposing, rather than melting? What is decomposition? Refer to lab procedure.

5. What is the potential consequence of using a sample that is not finely powdered? What happens to the melting range?