Melting Points of Compounds and Mixtures

Objectives

- Measure the melting point of a compound or mixture
- Determine the purity of products based on melting point

Background

Melting Points of Compounds and Mixtures

The definition of a melting point for a compound is the temperature at which both the solid and liquid phases both exist. In practice, a melting point measurement does not find a single temperature. Instead, a melting point range is reported from the temperature when liquid first appears to the temperature when the sample is completely liquid.

Measuring Melting Points

To measure melting points in the lab, most people use a melting point apparatus. There are both analog and digital versions of the apparatus, but they all follow the same general structure. The sample is loaded into a small capillary tube and placed in a chamber of the apparatus. You then start the heat and view the sample through the viewing window. The apparatus also includes a thermometer or temperature display to read the temperature when the first liquid appears in the sample and the temperature when the last visible solid melts into liquid.

There are different methods to load a sample into a capillary tube. However, the first step is always to tap a small amount of finely ground sample into the open end of the tube. Then, the sample needs to be moved to the bottom, closed end of the tube. One way to do so is to tap the closed end of the tube on a hard surface, such as dropping the capillary tube down a packing tube. Other methods are also possible.

Melting Points of Pure Compounds

Melting points for pure compounds are often easily found in literature and therefore can be used to help determine the identity of the compound. For example, if you know a couple possible options for the identity, you can compare a melting point measurement to the expected melting point to determine which compound is present.

The melting point measurement also gives an indication of how pure a sample is. Pure compounds normally have a very narrow melting point range of 1-2 ^{o}C. The range should also be very close to the expected melting point for the compound. Therefore, measuring the melting point can indicate the purity of a product from a synthesis.

Melting Points of Mixtures

Melting points of mixtures have different characteristics than either pure compound. Thus, using melting points to identify compounds in a mixture is challenging. One of the compounds needs to

be known so that the other can be determined. The melting point range of the mixture will normally have an upper limit that is close to the higher melting point of either compound.

In general, the melting point range of a mixture is broader than either of the two melting points separately as the different compounds interfere with the melting behavior. The melting points of mixtures are also generally lower than the melting point of either solid separately. Consider the plot of melting point of a two-component mixture in Figure 1. The melting point of the pure compounds are at each side of the plot and are higher than any other part of the line.

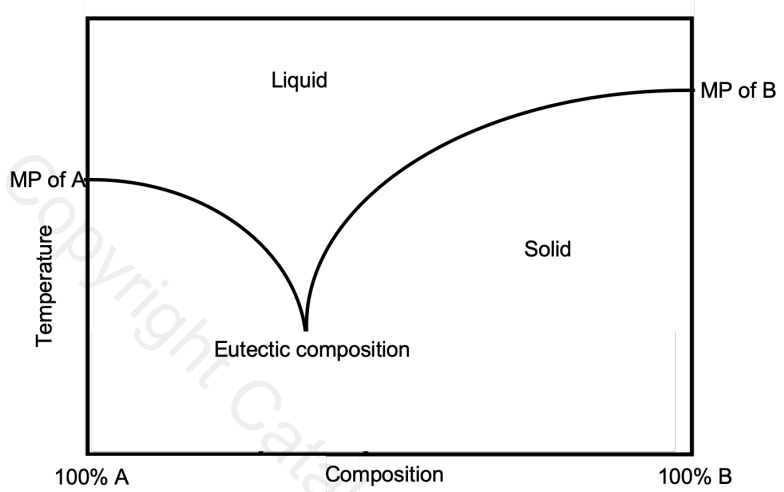

Figure 1 Plot of melting point behavior for a mixture of compounds A and B at different compositions

The graph of the melting point versus the percent composition of the mixture, like that in Figure 1, helps you determine the eutectic composition. The eutectic composition is the percent composition of a binary mixture with the lowest melting point. This is the lowest temperature at which the mixture will begin to melt. With this information, one can better determine the percent composition of a mixture when only knowing the melting point range.

Materials

- Benzoic acid
- Mandelic acid
- Mixtures of the two acids
- Melting point apparatus
- Capillary tubes

> ***Safety goggles are required!***
>
> *Benzoic acid and mandelic acid are both weak acids and irritants to the skin and eyes.*

Procedure

Melting Points of Compounds and Mixtures

In this procedure, you will determine the melting point of two pure compounds, benzoic acid and mandelic acid along with two mixtures of the two. Your TA will assign you two of the mixtures to study. Then, you will report your findings of the mixtures to the class so that everyone will be able to complete the graph.

1. Put a small amount of the sample, which could be benzoic acid, mandelic acid, or a mixture, on weigh paper (approximately 25.0 mg should be plenty).
2. Tap the open end of a capillary tube into the powder until about 2.0 mm of powder is in the end of the tube.
3. Take the packing tube (a long clear plastic tube) and place one end flat on the table. Then, hold the capillary tube over the open end of the packing tube with the closed end down and drop the capillary tube. Once the capillary tube stops bouncing, remove the capillary. Check if the solid has reached the end of the capillary tube. If not, repeat the drop. You want the powder as close to the end as possible, but it does not have to be touching the end.
4. Place the capillary tube in the melting point apparatus and set the temperature at 95ºC. Allow the capillary tube to come up to that temperature. You will always want the temperature set below the actual melting temperature, so that you can monitor the melting of the sample in the capillary tube.
5. After allowing the capillary tube to sit in the apparatus for about 5 minutes at 95 ºC, start ramping up the temperature. The rate of increase should be about 1-2 ºC per minute.
6. Watch the sample while the temperature rises. Record the temperature when the sample starts to melt and the temperature when the sample has completely melted.
7. Discard capillary tubes in the glass waste once you have finished determining the melting point.
8. Repeat the melting point sample preparation and measurement for each sample, recording the melting point range for each sample.
9. Write the mixture melting point range on the board to be shared with all student pairs.

10. Once you have completed taking melting points, dispose of weigh paper and all leftover powder in the trash.
11. Graph your results of the pure compound and mixture melting points. For each point, use the temperature in the middle of your melting point range. After plotting all the points, draw two best fit straight lines to connect at the eutectic composition.

Pre-lab Questions

Prepare for lab by understanding the answers to these questions. Refer to the Background or another resource, such as your textbook, if necessary.

1. Why would a sample that is not finely ground create difficulties in a melting point determination? Give two reasons.

2. What is the definition of the eutectic composition?

3. What is the structure of benzoic acid and what is its expected melting point?

4. What is the structure of mandelic acid and what is its expected melting point?

Lab 3: Melting Points of Compounds and Mixtures Report Sheet

Name _____ Section _____

Date _____ Instructor _____

Melting Points of Pure Compounds

Benzoic Acid

Temperature when melting starts _____

Temperature when melting is complete _____

Melting point temperature range _____

Mandelic Acid

Temperature when melting starts _____

Temperature when melting is complete _____

Melting point temperature range _____

Melting Points of Mixtures

80:20 Benzoic Acid: Mandelic Acid Mixture

Temperature when melting starts _____

Temperature when melting is complete _____

Melting point temperature range _____

60:40 Benzoic Acid: Mandelic Acid Mixture

Temperature when melting starts _____

Temperature when melting is complete _____

Melting point temperature range _____

40:60 Benzoic Acid: Mandelic Acid Mixture

Temperature when melting starts _____

Temperature when melting is complete _____

Melting point temperature range _____

20:80 Benzoic Acid: Mandelic Acid Mixture

Temperature when melting starts _____

Temperature when melting is complete _____

Melting point temperature range _____

Post-lab Questions

1. What is the melting point of benzoic acid that you determined?

2. What does the benzoic acid melting point tell you about the purity of the compound?

3. Based on the intersection of the lines from your graph, what was the eutectic temperature for the mixture?

4. Based on the intersection of the lines from your graph, what was the composition of the eutectic mixture?

5. Where should you dispose of capillary tubes when you have finished determining melting points?