PROBEWARE INVESTIGATION 10 • A

Determining Melting Points

The melting point of a compound is the temperature at which the solid and liquid phases are in equilibrium. This is a physical property that is often used to identify compounds, or to check the purity of a compound.

Question

How can the melting points of various organic compounds be found?

Prediction

Look up the chemical structures of the following compounds, and predict their relative melting points: vanillin, acetanilide, benzoic acid, 2-naphthol, urea, maleic acid, cholesterol, citric acid, salicylic acid.

Materials

computer system and interface
temperature probe
melting point capillary tubes (closed at one end)
filter papers
samples (1 g each) of vanillin, acetanilide, benzoic acid, 2-naphthol, urea, maleic acid, cholesterol, citric acid, and salicylic acid
clean cloth
Thiele tube filled with mineral oil (filled to a level no more than 2 cm above the upper inlet of the sidearm)
two-holed stopper to fit the top of the Thiele tube
3 cm (3 mm outside diameter) rubber tubing
scissors
Bunsen burner

Safety Precautions

- Wear safety goggles, gloves, and a lab coat while carrying out the experiment.
- Before starting, check that the room is well ventilated.
- Work with care, in order to avoid burns and/or broken glass.
- Dispose of all chemicals safely and appropriately, as directed by your teacher.

Procedure

1. Set up the computer system with the temperature probe set to record at a rate of once per second.

2. Display the sensor with a graph (temperature vs. time) and digits display.

3. Obtain a melting point capillary tube and a sample of the known compound.

4. To load the melting point capillary tube, place a small amount of the compound on a piece of clean filter paper. Push the open end of the melting point capillary tube into the middle of the pile of compound. Some solid should be trapped in the tube — use the smallest amount of material that can be seen.

5. Turn the melting point capillary tube over, closed end down. Use a clean cloth to remove any of the compound that may be sticking to the outside of the tube.

6. Keeping the melting point capillary tube vertical, drop it several times from a height of 2 cm perpendicularly onto a solid surface. The melting point capillary tube is "loaded" when the entire compound has reached the bottom of the melting point capillary tube.

7. Clamp the Thiele tube just under the mouth with the utility clamp that is attached midway to the support stand.

8. Fit the temperature probe through the two-holed stopper. When placed into the Thiele tube in step 11, the tip of the temperature probe will be immersed in the oil beside the upper inlet of the sidearm.

9. Using scissors, cut a 2 mm section of the rubber tubing. This will be used as a small rubber band.

10. Place the closed end of the loaded melting point capillary tube next to the end of the temperature probe. Place the rubber band around the temperature probe and the melting point capillary tube. The rubber band should be 1 cm from the top of the melting point capillary tube.

11. Keeping the temperature probe vertical, place the stopper apparatus into the Thiele tube.

12. Start the temperature sensor to monitor the temperature.

13. Heat the mineral oil with a moderate burner flame, directing the flame at the curved side of the Thiele tube.

14. Allow the temperature to rise rapidly to within 15–20°C below the expected melting point of the compound. (Your teacher will give you an approximate melting point for the compound.)

15. Adjust the flame size so that the temperature rises slowly, no more than 2–3°C per minute. Keep the temperature rising at this slow rate just before, during, and just after the period in which the compound melts.

16. Record the temperature at the first visible sign of liquid (the sample appears moist, or a tiny drop of liquid is observed). Next, record the temperature at which the sample is completely melted. The range formed by these two temperatures is the melting point of the compound.

17. After the sample has melted, lift the temperature probe and attached sample tube carefully (it may be hot) until they are just out of the oil. Wait for the temperature probe to cool to about room temperature before you remove it entirely from the tube.

18. Remove the melting point capillary tube and wipe off some of the oil from the temperature probe.

19. Reload a new melting point capillary tube (never re-melt melted samples), and repeat steps 3 to 18 for each of the other samples provided.

20. Stop recording the data.

21. Clean up and discard the materials as directed by your teacher. Do not pour anything down the drain.

Analyze and Conclude

1. Why does the sample in the melting point capillary tube have to be packed tightly?

2. Why should the filled portion of the capillary tube be placed immediately beside the temperature probe?

3. What were the melting temperatures of the compounds you analyzed?

Applications

4. List two ways in which the melting point of a solid organic compound could be useful to organic chemists.

5. What might be the effect of a small amount of impurity on the melting point of an organic compound?

6. The freezing point of a substance has the same numerical value as its melting point. Melting points are routinely measured, but freezing points are not. Why?

7. Why is the method outlined in this investigation not used for finding the melting points of inorganic compounds?

Teacher Information

Notes

- If necessary, use a spatula to grind the compound into a fine powder so that it can enter the melting point capillary tube.
- Remind students to pack the compound into the melting point capillary tubes. If left loose, the compound will heat unevenly. You may want to use a dropping tube to assist in packing the compound into the melting point capillary tubes.
- The hot oil in the Thiele tube is used to transfer heat evenly to your sample in the melting point capillary tube. Oil in the side arm is heated, and expands to become less dense. The hot oil goes up the sidearm, warming the sample and the temperature probe as it touches them. The oil cools and becomes denser, and falls to the bottom of the tube where it is heated again. This cycle goes on automatically as you do the melting point test in the Thiele tube. **Caution:** Do not allow water to get into the Thiele tube. If this happens, the water can boil and throw hot oil out.
- Take care that the rubber band securing the melting point capillary tube to the temperature probe remains above the mineral oil throughout the experiment.
- Never re-melt any sample. The sample may undergo chemical changes such as oxidation, rearrangement, and decomposition.
- The melting point is defined as the temperature range over which a small amount of solid in a thin-walled capillary tube first visibly softens (first drop of liquid) and then completely liquefies. Thus, the melting point is actually a melting range. Melting points recorded in the chemical journals are capillary melting points unless otherwise stated.

Answers to Analyze and Conclude Questions

1. Students may suggest various reasons, but they should be aware that packing the sample leads to a more accurate and reproducible result. The sample must be packed tightly so that the melting can be observed within the same area in which the temperature is being monitored. The temperature lower or higher in the tube may not be the same as the temperature being monitored.

2. The filled portion of the tube is placed beside the temperature probe to allow the melting temperature of the sample to be monitored with accuracy.

3. Answers will vary. Suggested results:

Compound	**Melting Point (°C)**
vanillin	77–82
acetanilide	113–114
benzoic acid	121–122
2-naphthol	121–122
urea	132–133
maleic acid	136–137
cholesterol	148–150
citric acid	150–153
salicylic acid	156–158

Answers to Applications Questions

4. Melting points are useful for the determination of purity. Impurities generally have two effects: they lower the melting point from what it would be for the pure compound; and they cause the melting point range to broaden. Melting points are also useful for identifying unknown compounds. The melting point of an unknown sample can be found by students, who can then compare their findings to a list of known melting points of different organic compounds.

5. Students may suggest various results, but they should be aware that the presence of an impurity will change the melting point. In fact, a small amount of an impurity will broaden the melting point temperature range so that it will be wider than 2°C.

6. Freezing points are rarely measured in practice because they are more difficult to determine. It is much easier to heat a compound gradually than to cool it gradually. Another reason why freezing points are difficult to obtain is that solidification may not occur at the correct temperature due to the phenomenon of supercooling.

7. This method will not work for most inorganic compounds because they have extremely high melting points.