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?