

Experimental Procedure:

1. Place a small sample of the organic compound on a piece of filter paper, a glass plate, a microscope slide, or in a clean small vial.
2. Gently place the open end of the capillary tube into the solid sample
3. When a small amount of solid is in the tube, invert the tube and gently tap the sample down toward the sealed end. Make sure the solid is firmly packed in the closed end of the tube. The tube should only have 1 to 2 mm of material in it.
4. Repeat steps 2 and 3 to fill a second capillary tube.
5. Secure the capillary tube, sample end down, with a rubber band to the side of a thermometer. Be sure to secure the tube near the bottom of the thermometer so that the closed end of the capillary tube is even with tip of the thermometer bulb. See Figure 6.
6. Fill a 250-mL beaker about two-thirds full with distilled water and place on a stirring hot plate. Add a stir bar and begin to heat the water bath. If using the immersion, place the heater in the water, plug in the heater, and begin to heat the sample. Use the magnetic stirrer or a stirring rod to stir the water bath. Do not use the thermometer as a stirring rod. The rate of heating should not exceed 10 °C per minute. Use an oil bath if the solid has a melting point over 90 °C.
7. As the water begins to heat, place the thermometer and the tube in the water bath so that the thermometer bulb is about an inch below the surface of the liquid. Hold the thermometer in place using a thermometer clamp. The open end of the capillary should not be near the surface of water bath.
8. When the first sign of melting occurs, write down the temperature. Keep watching the solid until the last crystal melts. Write this temperature down. These two temperatures are the melting point range of the sample.
9. Remove the thermometer and sample from the bath. Turn off the hot plate. Use insulated gloves or beaker tongs to carefully remove the water bath from the hot plate. If using an immersion heater, unplug the heater.
10. Discard about one-fourth of the water from the beaker. Add cool water to replace the discarded hot water. If using an oil bath, do not discard any oil, just add a little more cool oil and mix thoroughly.
11. Discard the first capillary tube and attach the second sample to the thermometer. Never reuse a sample; always discard the sample after it has melted.
12. Repeat steps 6 through 10. Use a slower heating rate (1-2 °C per minute) to achieve a more precise melting point.

Questions:

1. Why should a melting point determination sample never be reused?
2. Why is it important to use only small amounts of a solid in a melting point determination?
3. Does atmospheric pressure affect the melting point?
4. From the listed organic compounds, identify your unknown.