

1. Prepare a tiny sealed capillary (one that will fit inside of a standard melting point capillary) by heating the middle of a Pasteur pipet in a small blue flame. Hold the pipet by the ends and when the glass starts to sag in the flame, remove it from the flame and pull the two ends until about 5 inches of a capillary of the desired diameter is made. Allow the pipet to cool; then, break off approximately 2 inches of the fine capillary and seal both ends. Break this 2-inch capillary in half for use.

2. Use the very fine tip of the Pasteur pipet that you just melted to introduce a small amount (about  $\frac{1}{2}$ " ) of the liquid sample into a melting-point capillary. Add one of the (now 1-inch) capillaries to the melting-point capillary, open end DOWN (see Fig. 5.12). Be sure there are no

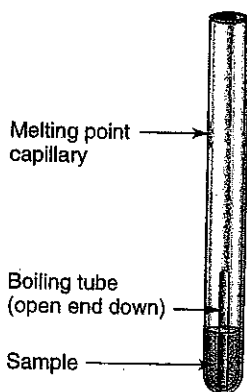


Figure 5.12 Capillary boiling point setup.

air bubbles in the melting point capillary. If air bubbles are present, tap the melting point capillary with a pencil or with your finger until they disappear.

3. Place the melting point capillary in a standard melting point apparatus and heat it gently. Bubbles (mostly expanded air) will start to come out of the bottom of the tiny capillary. Occasionally observe the temperature on thermometer. When a *stream* of bubbles (mostly vapors from the boiling liquid) start to come from the bottom (open end) of the tiny capillary, turn off the heater. The temperature of the sample tube will start to cool. When the boiling point (condensation point) is reached, the vapor will suddenly condense and a vacuum will be formed. This causes the liquid to be suddenly sucked up into the tiny capillary. Read the temperature at which this occurs. This is the "BOILING POINT."

*Note: Before you try an unknown liquid, try two or three knowns such as water, isopropyl alcohol, or hexane. You should not be satisfied with more than 2 degrees error.*