MICRO BOILING POINT DETERMINATION

The boiling point, like the melting point, is used to characterize a liquid substance and is particularly useful for identifying organic liquids. Many organic liquids, however, are flammable and this determination must be done with care. This procedure provides a safe and simple method for determining the boiling point of a flammable, volatile liquid.

Safety Precautions:

The liquids used in this procedure are flammable. Although the danger of fire is greatly reduced by the use of small samples, it is **not eliminated. Keep all liquid samples away from open flames.** Avoid inhaling vapors from volatile liquids. Avoid skin contact with the volatile liquids.

Know the flash point of the oil used in this experiment. Keep the oil temperature at least 20°C below the flash point.

Materials Needed:

Test tube, 6 x 50 mm
Glass capillary tube, closed one end
Thiele tube with mineral oil (you can substitute a 150-mL beaker for the Thiele tube)
Dropper
Thermometer, 150°C or higher, if needed
Rubber tubing
Scissors
Triangular file
Test tube holder

Procedure for Determining Micro Boiling Point:

The set-up for this procedure is shown in Figure BP-1.

Obtain a 6 x 50 mm test tube. Holding it with a test tube holder, slowly heat the test tube from the bottom to the open top to remove any water condensed on the interior of the tube. Place the tube on a ceramic hot pad to cool before use.

Attach the 6 x 50 mm test tube, or a micro boiling point tube, to the thermometer using a rubber band made by cutting a piece of rubber tubing about 2 mm thick.. The bottom of the test tube should be close to the center of the thermometer bulb.

Obtain a glass capillary tube. Using the file, carefully cut a piece 3 to 4 cm long, measuring from the closed end of the tube. Place this tube, open end down, into the micro boiling point tube prepared above.

Obtain a sample of a volatile liquid (about 1 mL needed). Using a clean, dry dropper, add a few drops of the liquid to the micro boiling point tube attached to

the thermometer. The depth of the liquid in the tube should be about 1.5 to 2.0 cm high (this is a volume of about 0.2 mL).

Assemble the apparatus as shown in Figure BP-1. If the Thiele tube is not filled with mineral oil, add mineral oil to the tube. The oil level must be above the upper opening of the side arm. Position the thermometer so that the bulb is just below the upper opening of the side arm of the Thiele tube.

Using a Bunsen burner, gently heat the side arm of the Thiele tube to produce a temperature increase of between 5 to 10 degrees a minute. While the system is being heated, bubbles will come out of the open end of the capillary tube.

When a rapid and continuous stream of bubbles comes out of the small capillary tube and passes through the liquid, discontinue heating and allow the apparatus to cool.
The stream of bubbles will slow as the apparatus cools. When the bubbles stop coming out of the capillary tube and just before the liquid enters it, read and record the temperature on the thermometer. This temperature is the boiling point of the liquid.

If all the liquid evaporates, remove the burner, allow the apparatus to cool, and add additional liquid to the test tube. Repeat the heating process.

Clean-up and Disposal:

Do not discard the oil in the Thiele Tube. It can be reused many times.

The 6 x 50 mm test tube can be cleaned and dried in the burner flame (CAUTION: Flammable liquid residue may be present), and reused. Be sure to hold the test tube with a test tube holder.

Preparation of a micro boiling point tube:

If a 6 x 50 mm test tube is not available, a micro boiling point tube can be prepared from a piece of 6 mm glass tubing.

Cut a piece of 6-mm glass tubing about 6 cm long. (Tubing should be clean)

Using a test tube holder to hold the piece of glass tubing, heat one end in the Bunsen burner flame until it is sealed. Fire polish the other end of the tube.

Starting at the closed end of the glass tube, slowly heat the entire tube in the burner flame to evaporate any water condensed in the tube. Failure to do this will result in erroneous values of boiling points.

Allow the tube to cool on a ceramic hot pad.

Reference:

This procedure is a modification of one described in Shriner, R. L., Fusion, R. C. and Curtin, D. Y., **The Systematic Identification of Organic Compounds**, 5th Ed., John Wiley and Sons, Inc., New York, 1964, pp. 37-8.

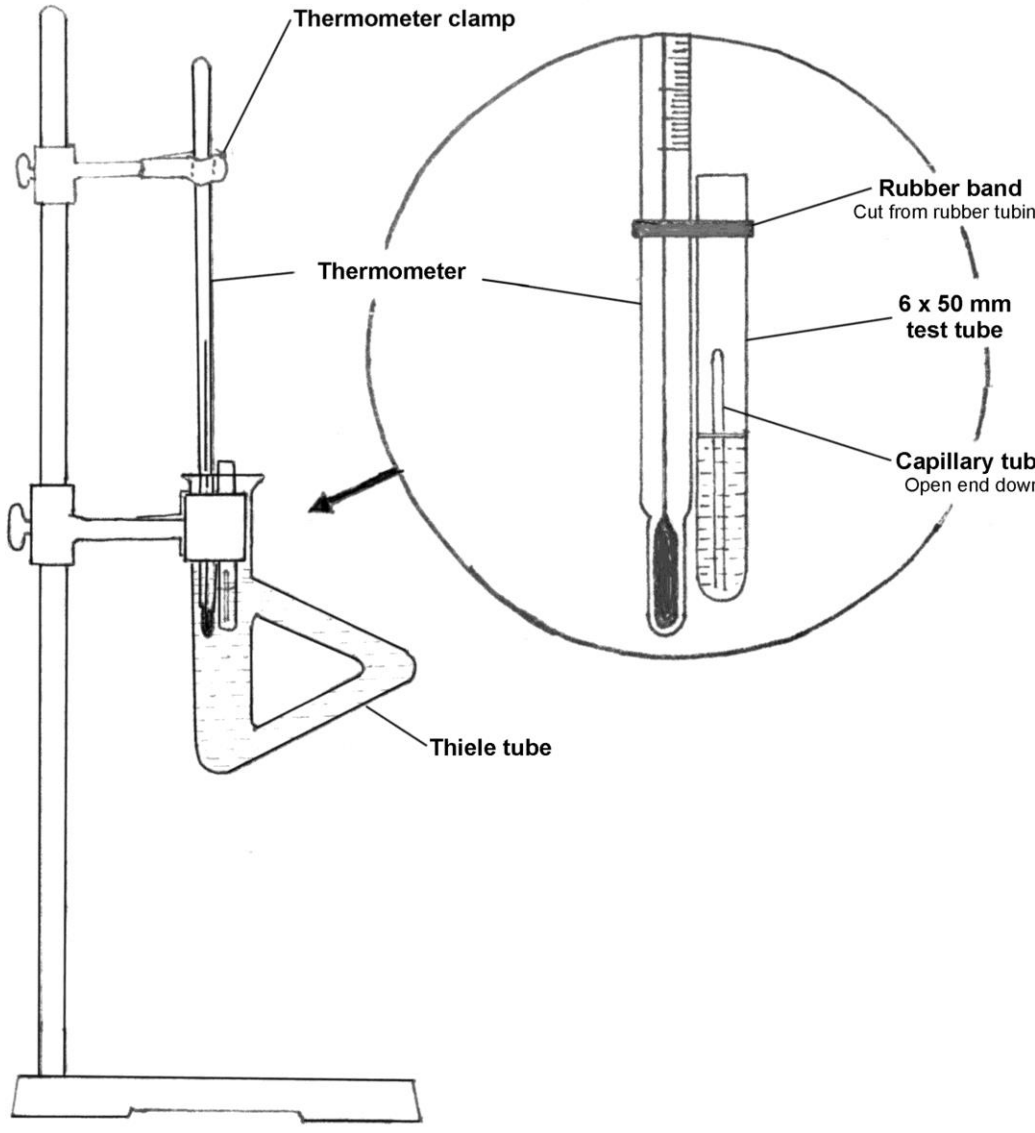

Figure BP-1. Micro boiling point apparatus