

How to Determine the Boiling Point

Sample method (when 0.5–3 g of compound are available).

When relatively large amounts of liquid are available, boiling points can be determined using a distillation method or by simply heating the liquid in a test tube in which a thermometer is inserted so that it does not touch the walls of the test tube. When the liquid is boiling freely, then that is the boiling temperature.

Only the lower portion of the test tube must be heated. A sand bath may also be used, if preferred. Do not use a direct flame on the test tube.

Figure 1 is self-explanatory. A cork with a small slit cut in it may be used instead of the thermometer adapter and syringe needle shown here. Be sure there is a way for vapor to vent.

After the setup is complete, drop a fresh boiling chip into the test tube. Then add the liquid to be tested. Insert the thermometer carefully so as not to break it. Be sure the thermometer is not touching the glass test tube—and that there is enough liquid to cover the thermometer bulb a short way up the stem.

The boiling temperature is the steady temperature observed (for about one minute), when refluxing is occurring over the lower part of the thermometer, yet not all the way up to the top of the test tube.

This procedure is not designed to provide accurately calibrated absolute values for boiling points, but the values will be quite close and the method will do an excellent job of providing relative values for solutions/mixtures of varying concentrations, or solutions vs. pure substances.

Micro-method. When only small amounts of liquid (a few drops) are available, or it desired to keep amounts of chemicals used and waste disposal to a minimum, the following method may be used.

Two glass tubes are needed for this setup (Fig. 2). One tube is a normal melting point capillary, the second is a 3–5 mm internal diameter glass tube that is sealed at one end, and which is a little shorter than the melting-point capillary. Place the melting-point capillary in a moderately hot, but small, burner flame. Warm it while rotating the tube

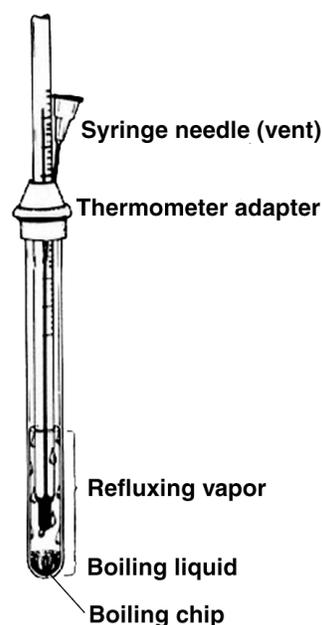


Fig. 1

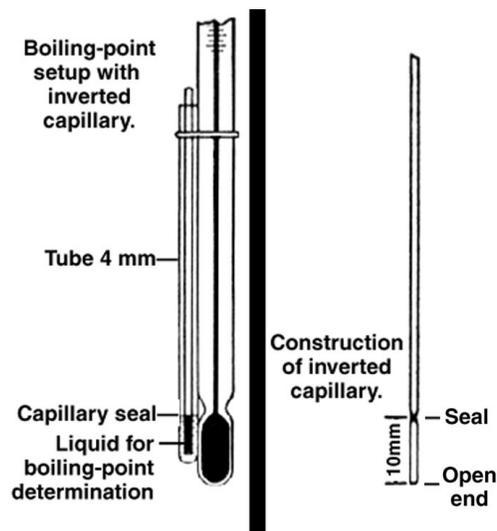


Fig. 2

about 1 cm from the open end (so that it seals at that point). Place the liquid to be measured in the larger tube and the small *boiling detector* in the tube. Attach these to a thermometer, using one or more elastic bands made by slicing a ring from the end of a piece of latex rubber tubing.

Place the thermometer in a small heating bath heated by a low flame (or a hot plate). Depending on the temperature range needed, the bath can contain water (0–95°C), ethylene glycol or antifreeze (up to 198°C), or mineral oil (up to 230°C). Stir the bath well to keep it at a uniform temperature. Observe the liquid and thermometer with the aid of a hand lens. Bubbles will be created as air is expelled from the small capillary. When bubbling increases greatly, the liquid is boiling. Stop heating and allow the bath to cool 1–3°. The bubbles should cease. Record the temperature at which the bubbles cease. That should just be the boiling point. You can repeat this heating-cooling/bubbling-no bubbling cycling several times, and narrow down the mean temperature, which will be the appropriate boiling point. The quality of measurements probably depends on the time available and your patience! The slower the rate of heating and cooling, the more precise may be the boiling temperature.