How to Determine the Melting or Freezing Point

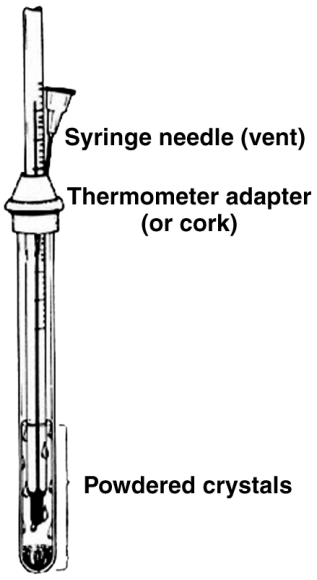


Fig. 1

These methods are not designed to provide accurately calibrated absolute values for melting or freezing points, but 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.

Sample method (when 0.5–3 g of compound are available). When relatively large amounts of compound are available, melting points can be determined using a distillation method or by simply heating the compound in a test tube in which a thermometer is inserted so that the thermometer does not touch the walls of the test tube. The heating/cooling bath set-up is essentially similar to that used in the micro method. The beaker may need to be a little larger so the bath fluid is sure to cover the lower portion of the test tube used. A cork with a slit cut in it may be used instead of the "thermometer adapter" and syringe. Insert the thermometer carefully so as not to break it. Make sure that the thermometer is resting among the crystals/solid material that is to be melted. Only the lower portion of the test tube must be heated. Do not use a direct flame on the test tube. If you are starting with solid materials, powder them as finely as possible.

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. When the compound begins to soften, that is the melting temperature.

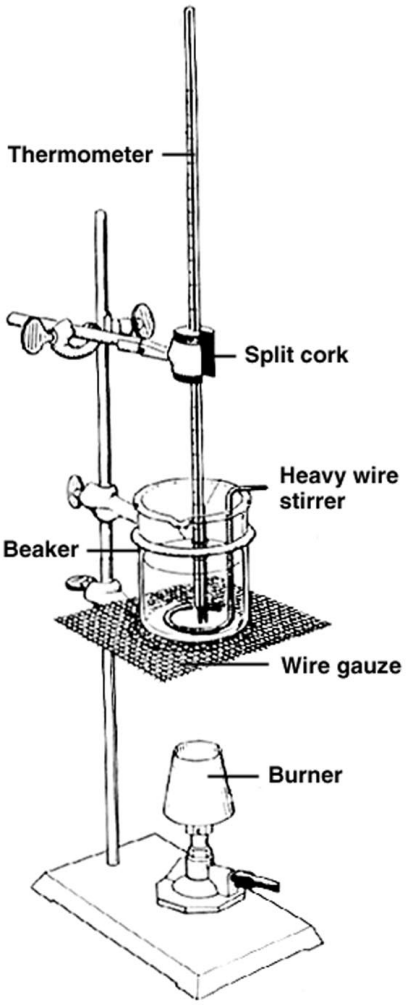
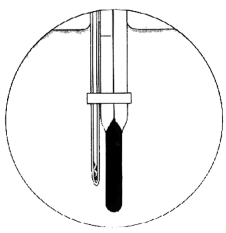


Fig. 2

Micro-method for determining the melting point. Take a clean glass microscope slide or object with similar hard, clean surface. Place a few crystals of the substance or mixture of



substances to be measured. Crush them carefully with the end of a small hard spatula or stirring rod. *Be careful not to spill the crystals, especially on your hands, as some may be toxic.* Use the end of the spatula to persuade some of the powder you now have into the end of the melting point capillary tube (Fig. 2 inset). Tap the tube gently on the bench to get powder to fall to the closed end. Repeat until you have about 5 mm of crystals in the bottom of the tube. If you do not have enough for this, the measurement can be done on less. Use one or more elastic bands made by slicing a ring from the end of a piece of latex rubber tubing to fasten the tube to a thermometer. 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 crystals and thermometer with the aid of a hand lens. Record the temperature at which the crystals appear to start to soften, and then at which the last crystal seems to have melted. Immediately stop heating. The quality of your measurements probably depends on the time available and your patience! The slower the rate of heating, the more precise may be the melting temperature.

Method for determining the freezing point. Start with a liquid and use a micropipette or syringe to place solution in the capillary tube, (or warm it in your hands). Invert in a drop of the solution, and then cool with ice. Turn it back upright, and tap to get air bubbles to go to the surface. In this way, you should be able to get some solution down to the closed end of the tube. Proceed as above, but using an ice, or ice-salt, or other bath depending upon the expected freezing point (Fig. 3).

If you are measuring the freezing point of a liquid or a solution, then it is necessary only to add enough liquid to cover the thermometer bulb, and a short way up the stem.

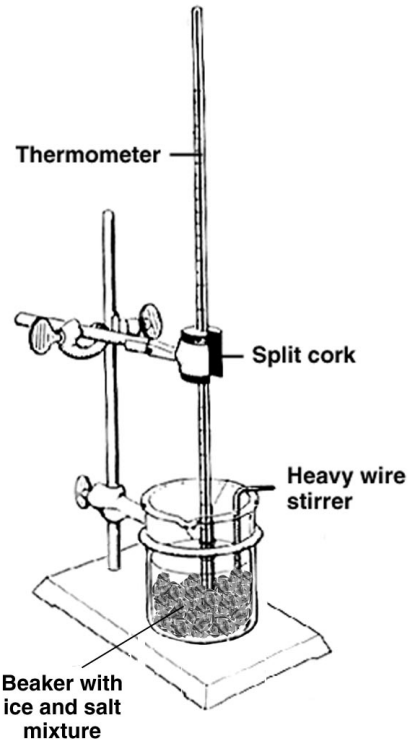


Fig. 3