

- **MELTING POINT**

The physical properties of a compound, such as melting point and boiling point can provide useful information which can help in the identification of a sample or to establish its purity. These pages describe two common methods for determining melting point using **i** a Meltemp apparatus and **ii** a Thiele tube set up.

The temperature at which a solid melts and becomes a liquid is the melting point. Since this requires that the intermolecular forces that hold the solid together have to be overcome, the temperature at which melting occurs will depend on the structure of the molecule involved - an example of the relationship between structure and properties. Hence, different compounds tend to have different melting points.

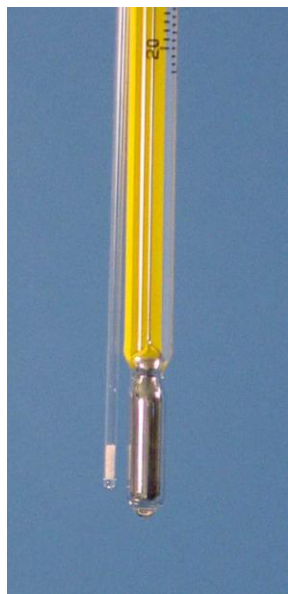
A pure, nonionic, crystalline organic compound usually has a sharp and characteristic melting point (usually 0.5-1.0°C range). A mixture of very small amounts of miscible impurities will produce a depression of the melting point and an increase in the melting point range. Consequently, the melting point of a compound is a criterion for purity as well as for identification.

The melting point of an organic solid can be determined by introducing a tiny amount into a small capillary tube, attaching this to the stem of a thermometer centred in a heating bath, heating the bath slowly, and observing the temperatures at which melting begins and is complete. Pure samples usually have sharp melting points, for example 149.5-150°C or 189-190°C; impure samples of the same compounds melt at lower temperatures and over a wider range, for example 145-148°C or 186-189°C. .

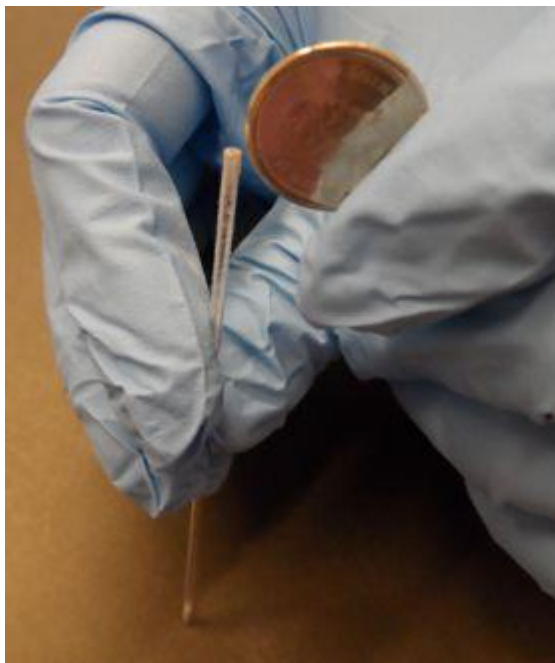
It is standard practice (in order to make the most effective use of time) to carry out a rapid melting point determination initially (by heating rapidly) to establish an *approximate* melting point and then carry out at least two further careful determinations (by heating more gently, *i.e.* temperature changing only about 2°C/min) until you obtain two consistent values.

The general method is to heat the sample indirectly by placing the prepared sample (either packed in a glass capillary or on a glass cover slip) in or on a heated medium, these days this is most commonly a heated metal block such as a Mel-Temp apparatus. There are other designs such as the Fisher-Johns apparatus. A more basic, but just as effective method is the Thiele tube method where the capillary is immersed in a heated oil bath. Note that the Thiele tube system is also used for [boiling point determination](#).

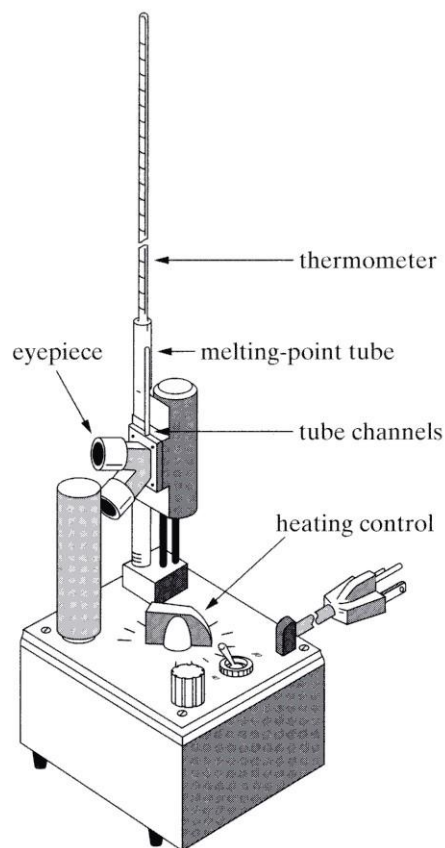
**IMPORTANT** : Note that *unlike* boiling point, the melting point is relatively insensitive to pressure and no pressure correction needs to be made.

**Packing a capillary for melting point determination**

Thin-walled capillary melting point tubes are used to hold melting point samples. This tube needs to be sealed at one end (pre-sealed tubes should be available in the laboratory, or, an open capillary can be sealed by inserting the tip into a Bunsen flame near the base of the flame and turning the tube in your fingers.... ask your TA to show you how to do this). To pack the tube, the open end is pressed gently into a small amount of the sample of the crystalline material on a watch glass or weighing paper. To transfer the crystals from the open end to the bottom of the tube, tap the bottom gently on the bench top or scratch the top edge of the tube on a small file or a coin with a milled edge (the [video on melting points](#) illustrates how to do this). A densely packed column of crystals about 3 mm high in the tube is all that is required. A packed capillary attached to a normal mercury thermometer is shown to the left.



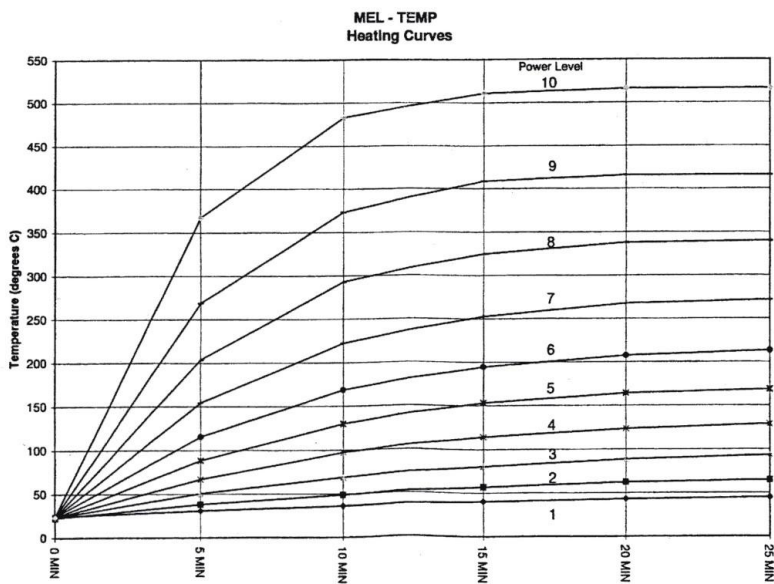
Using a coin with a milled edge to transfer the solid to the bottom of the capillary tube

**Mel-Temp Apparatus**

A Mel-Temp apparatus equipped with a digital thermometer is shown above. Note that we have switched to using a simple digital thermometer inserted into a conductive metal sleeve in place of a conventional mercury or alcohol thermometer. The diagram on the right shows the apparatus with the heat shield removed to reveal the inside structure. The apparatus uses capillary packed samples (see above). Insert the thermometer into the thermometer well and the capillary into the channels located on the front of the thermometer tube (there are slots for three capillaries). Turn the green LED power switch on (the "1" position) and turn the black heating control knob in order to set the power level to obtain the desired heating rate (see graph below). The sample can be observed through the lens on the front of the apparatus, the eye should be about 15cm from the lens.

Remember that a slow heating rate at the melting point is needed in order to get an accurate measurement. Record the temperature on the thermometer when the sample starts to melt and record the temperature again when all of the sample has melted (this gives you the melting point range). Once the sample has melted, turn the power to OFF and remove the capillary and dispose of it in the special container for this purpose. The block will require a little time to cool - the cooling process can be facilitated by connecting a hose to the in-house compressed air and blowing the air across the block.

Here is a link to a video showing [how to carry out a melting point determination using a MelTemp apparatus](#).



Graph of Mel-Temp heating power level profiles

**Thiele tube method** (included for completeness) (quick link to videos : [version 1](#), [version 2](#))

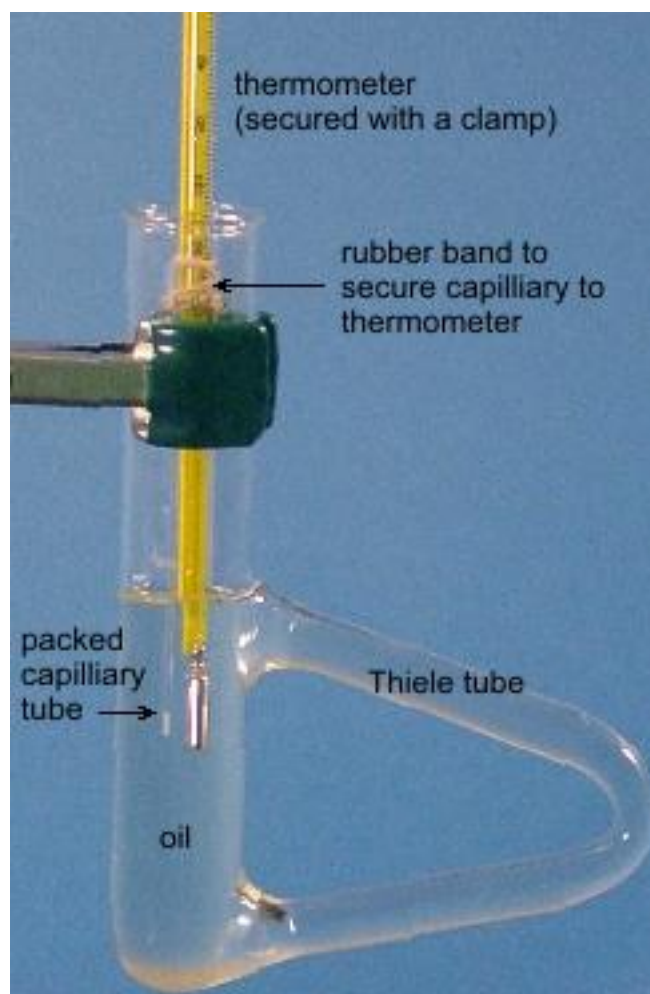


**Before using a Bunsen burner make sure all flammable materials (e.g. solvents) are removed from the area around the Bunsen burner. This means not only your workspace but also the students near to you.**

**When using the Bunsen burner, make sure that you adjust it to a small flame.**

If you are unsure of how to set up and use a Bunsen burner, see the "[heat sources](#)" techniques document and the video on the [Bunsen burner](#).

The Thiele tube is a glass tube designed to contain heating oil and a thermometer to which a capillary tube containing the sample is attached. The shape of the Thiele tube allows for formation of convection currents in the oil when it is heated. These currents maintain a fairly uniform temperature distribution throughout the oil in the tube. The side arm of the tube is designed to generate these convection currents and thus transfer the heat from the flame evenly and rapidly throughout the heating oil. The sample, packed in a capillary tube is attached to the thermometer, and held by means of a rubber band or a small slice of rubber tubing. It is important that this rubber band be above the level of the oil (allowing for expansion of the oil on heating). Otherwise, the oil softens the rubber and allows the capillary tubing to fall into the oil.



The Thiele tube is usually heated using a microburner with a small flame but a Bunsen burner can also be used. When heating, the rate of temperature increase should be carefully controlled. Usually one holds the burner by its base and, using a small, gentle flame, moves the burner slowly back and forth along the bottom of the side arm of the Thiele tube. If the heating rate is too fast, the burner is removed for a few seconds before resuming the heating process. The rate of heating should be slow near the

melting point (about 1-2°C per minute) to ensure that the rate of temperature increase is not faster than the ability of the heat to be transferred to the sample being observed. At the melting point it is necessary that the thermometer bulb and the sample in the capillary tube be at thermal equilibrium.

Here are two very similar video showing how the use the Thiele tube method for measuring melting point., [version 1](#), [version 2](#) (newer).