Ministry of higher education and scientific research
AL-Mustaqbal University college
Department of medical physics

Organic chemistry(practical)

Lecture 3

Melting Point Determination

By
MSc. Elham Faisal

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.

✓ **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 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.

Melting Point Determination:-

- 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 149.5-150C or 189-190 C; impure samples of the same compounds melt at lower temperatures and over a wider range, for example 145-148C or 186-189C. .
- 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 2oC/min) until you obtain two consistent values.

- The general method is to the 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**

unlike boiling point, the melting point is relatively insensitive to pressure and no pressure correction needs to be made

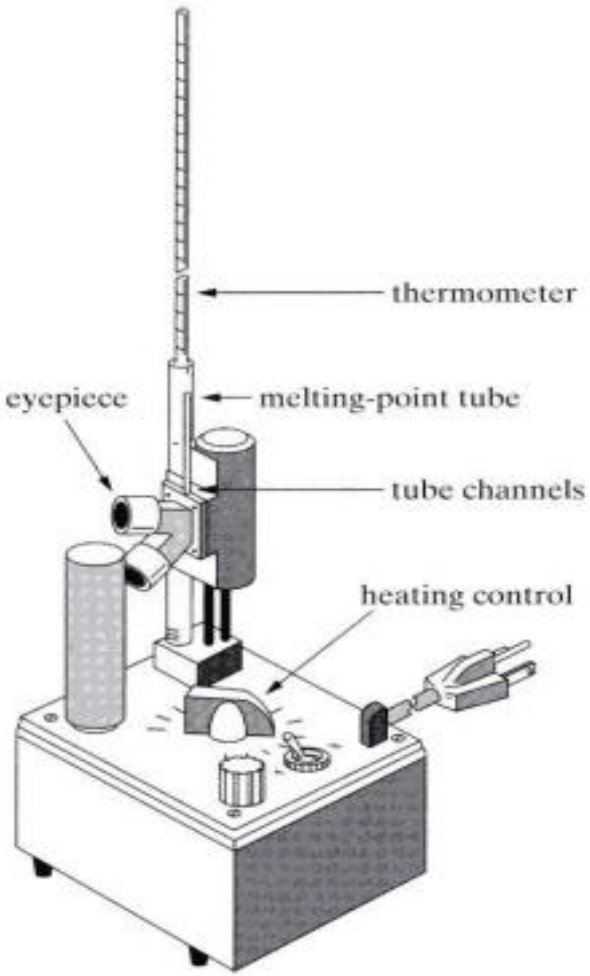
Mel-Temp Melting Point Apparatus

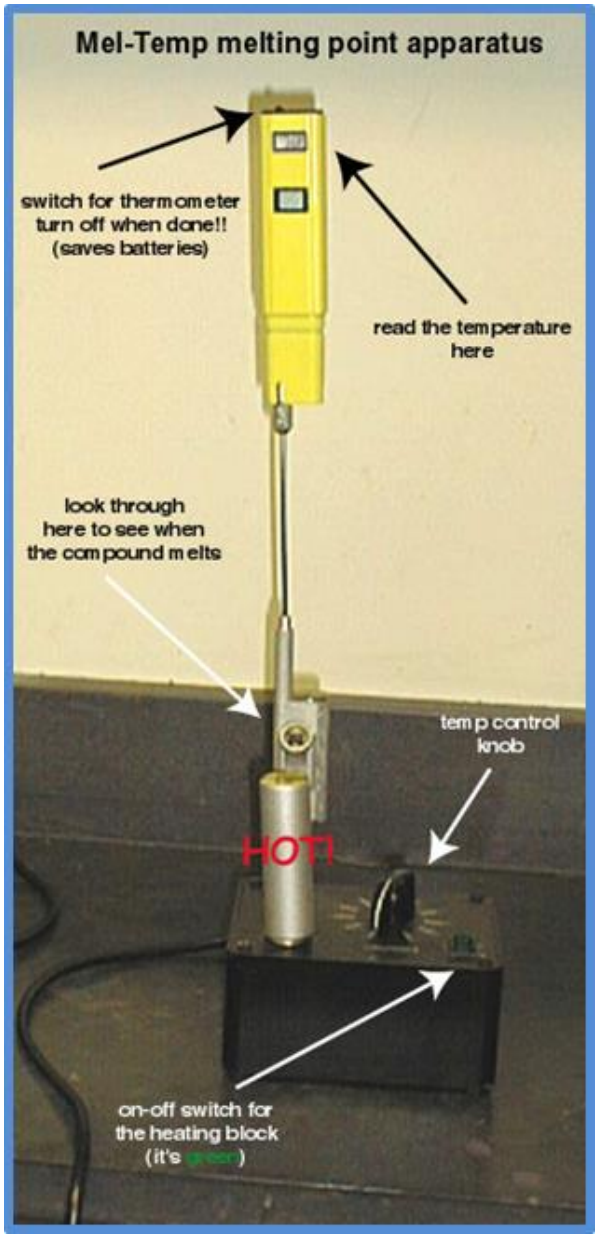
The diagram shows the apparatus with the heat shield removed to reveal the inside structure. The apparatus uses capillary packed samples .

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.

The sample can be observed through the lens on the front of the apparatus, the eye should be about 15cm from the lens.





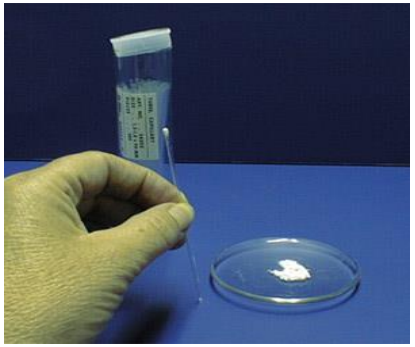
A Mel-Temp apparatus equipped with a digital thermometer

Technique for Taking a Melting Point

❖Thin-walled capillary melting point tubes are used to hold melting point samples.

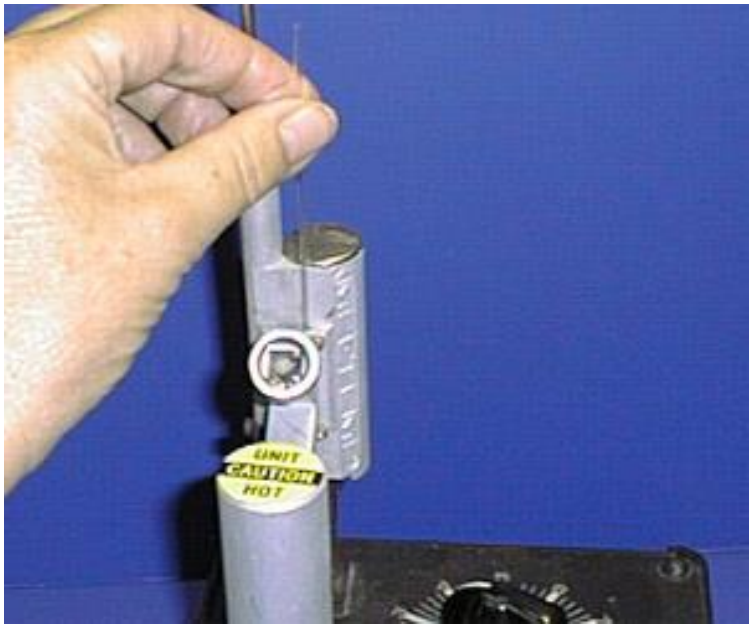
❖ This tube needs to be sealed at one end (sealed tubes).

1. Pack the capillary tube by pressing the open end gently into a sample of the compound to be analyzed. Crystals will stick in the open end of the tube.
2. The solid should fill the tube to a depth of 2-3 mm. Tap the bottom of the capillary on a hard surface so that the crystals pack down into the bottom of the tube.



3. Drop the capillary tube down a length of glass tubing to pack the crystals into the bottom of the tube.

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



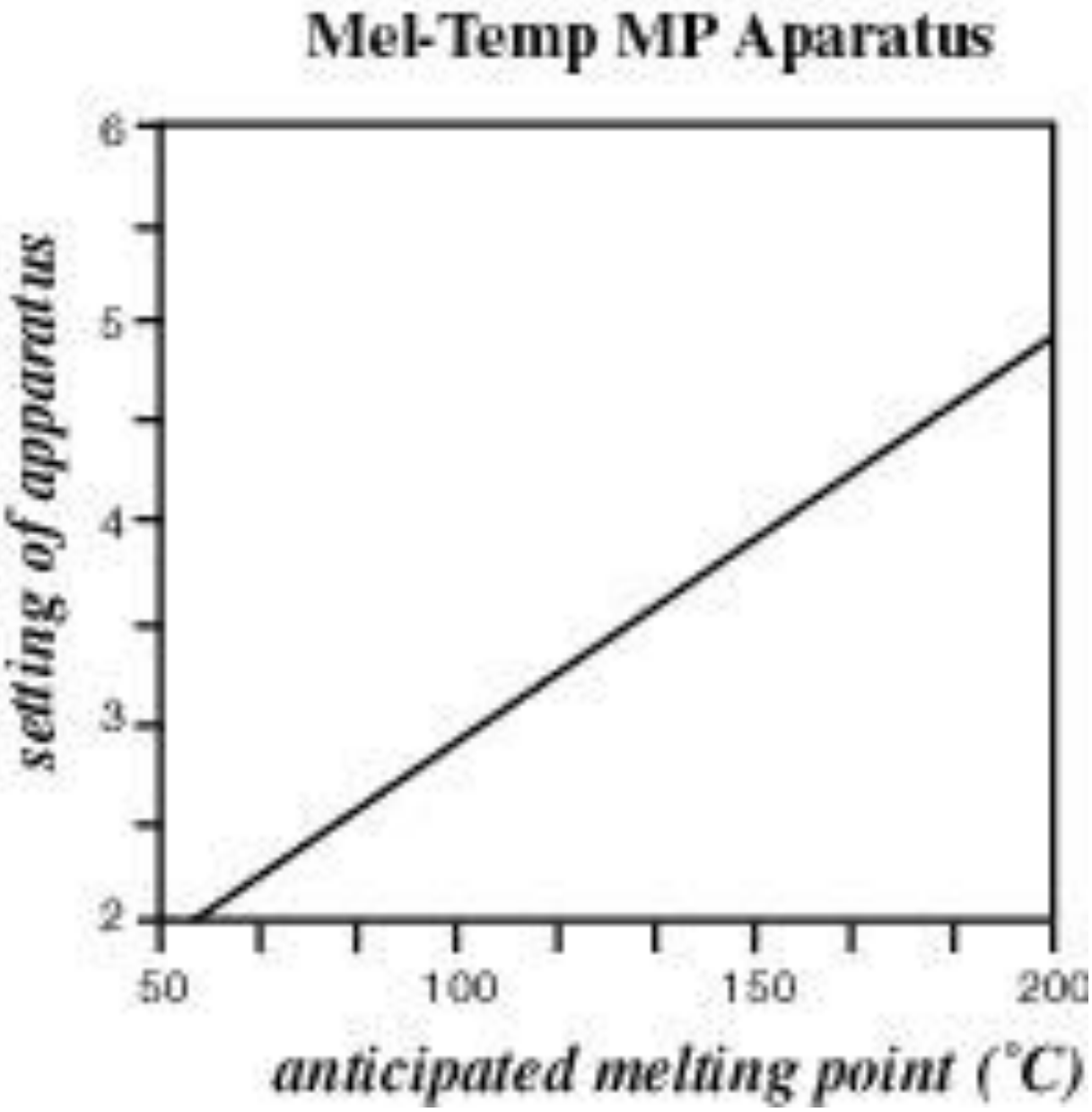
4. When the crystals are packed into the bottom of the tube, place the tube into the slot behind the eyepiece on the Mel-Temp.

   Make sure the unit is plugged in and set to zero, then turn it on.

5. To determine an appropriate heating rate. turn off both the unit and the thermometer and place the used melting point tube in the used melting point capillary tube receptacle.

- The rate of temperature increase in the vicinity of the melting point must be small, about 2 degrees C per min. This insures that the temperature of the hot plate, thermometer, and sample will be in thermal equilibrium. Increase the temperature rapidly at first and then slowly as the melting point is approached in the following manner:

I. Set the power level to 5.
II. When the temperature is about 15 degrees below the anticipated melting point, change the setting to that indicated on the graph below.
III. Observe the crystals with your eye about 6" from the lens to prevent accidentally touching the hot apparatus.
IV. Record the temperatures at which melting begins and at which the last crystal disappears.
V. If you do not know the melting point of a compound, first take a crude melting point by heating rapidly.
VI. Then cool the plate to 20° below the crude melting point, and proceed to take a more careful melting point on a second sample of the compound.


Mel-Temp MP Aparatus
setting of apparatus
6
5
4
3
2
50
100
150
200
anticipated melting point (°C)

I. That a slow heating rate at the melting point is needed in order to get an accurate measurement.

II. 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).

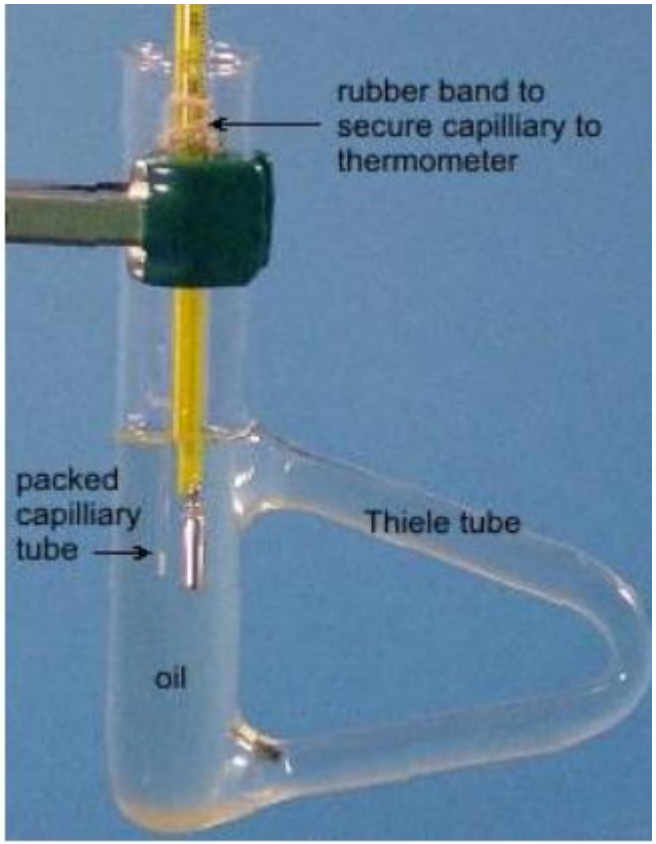
III. Once the sample has melted, turn the power to OFF and remove the capillary and dispose of it in the special container for this purpose.

IV. 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.

Thiele tube method

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.

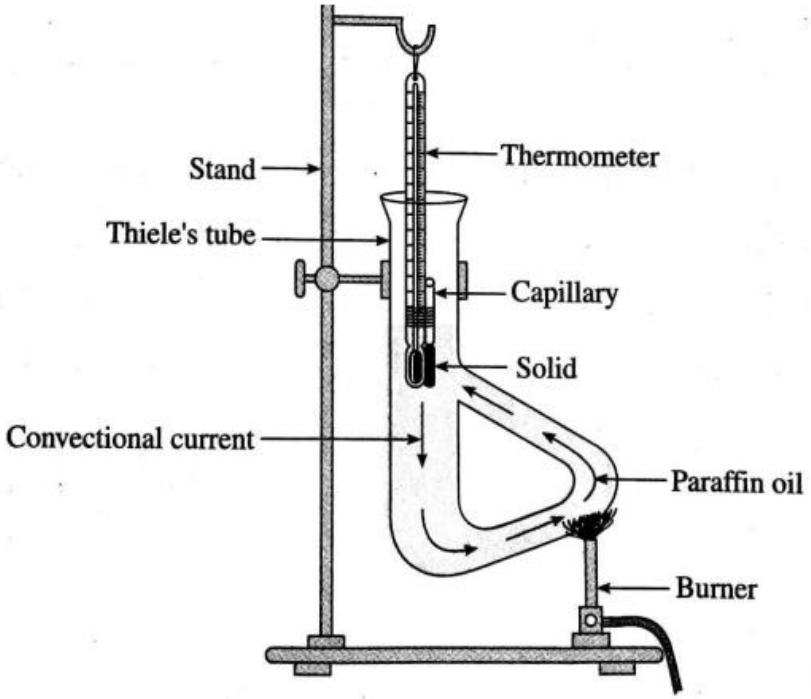


The Thiele tube is usually heated using a **microburner** with a small flame but a Bunsen burner can also be used. When heating, the rate of temperature increase should be carefully controlled. Usually one holds the burner by its base and, using a small, gentle flame, moves the burner slowly back and 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 1-2C 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.

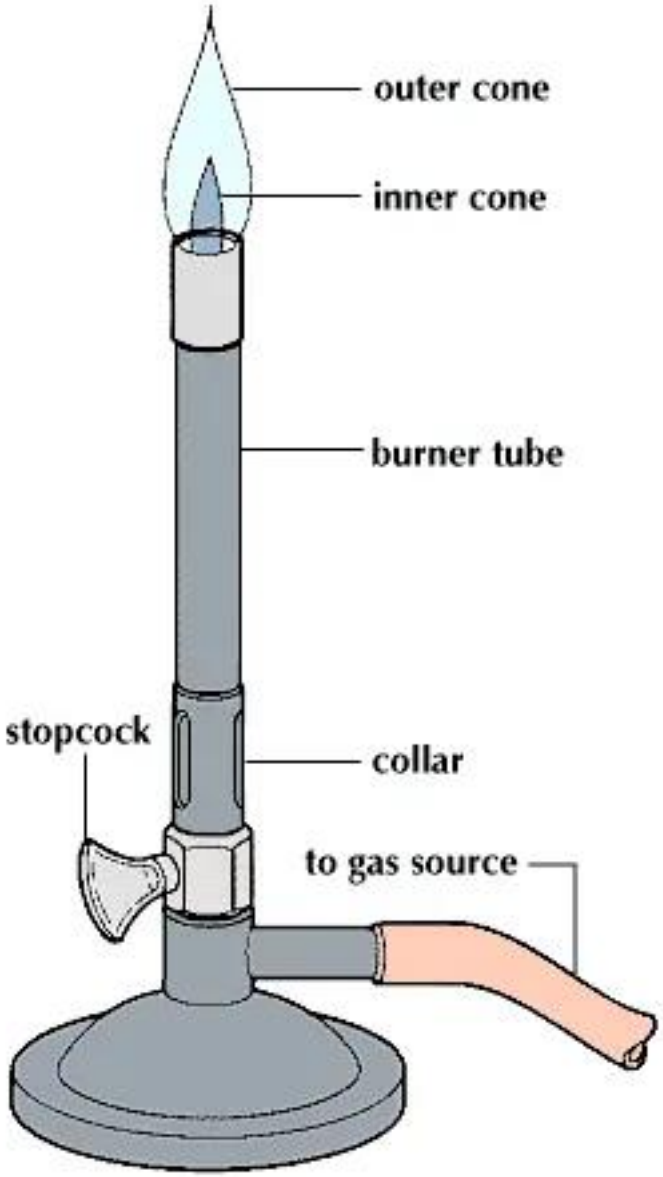
How do you use the Thiele melting point tube?

1. Fill a small tube about half-full with sample and insert a capillary tube, closed end up. Attach the tube to a thermometer with a small rubber band.
2. Insert the sample into a Thiele tube, so that the sample is near the middle of the oil.
3. Heat the arm of the Thiele tube with a burner, gently and continuously.



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.



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