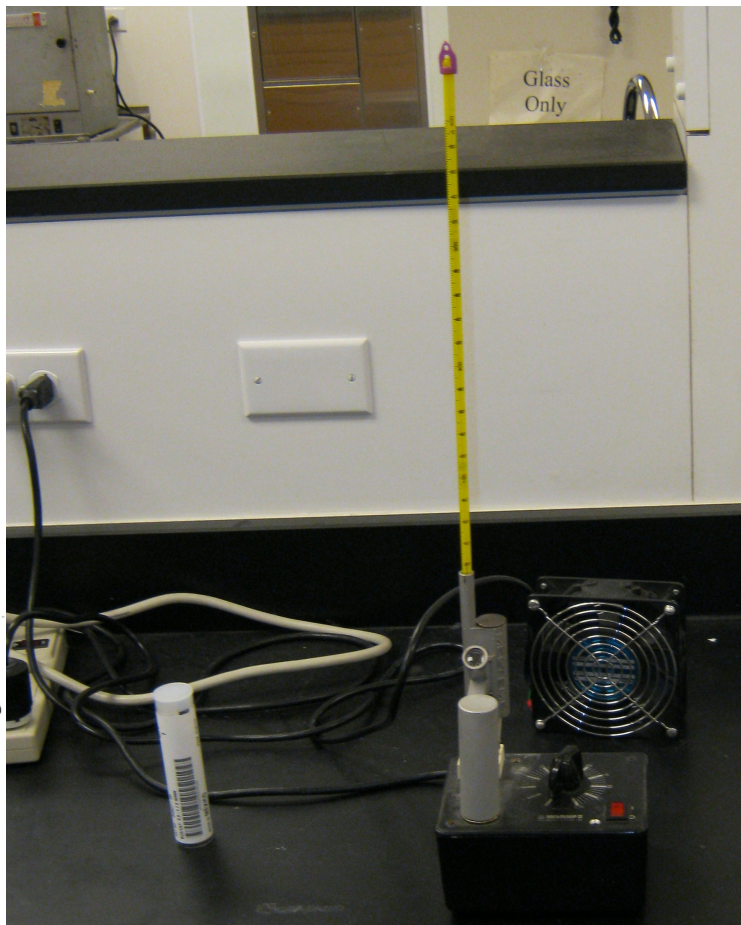
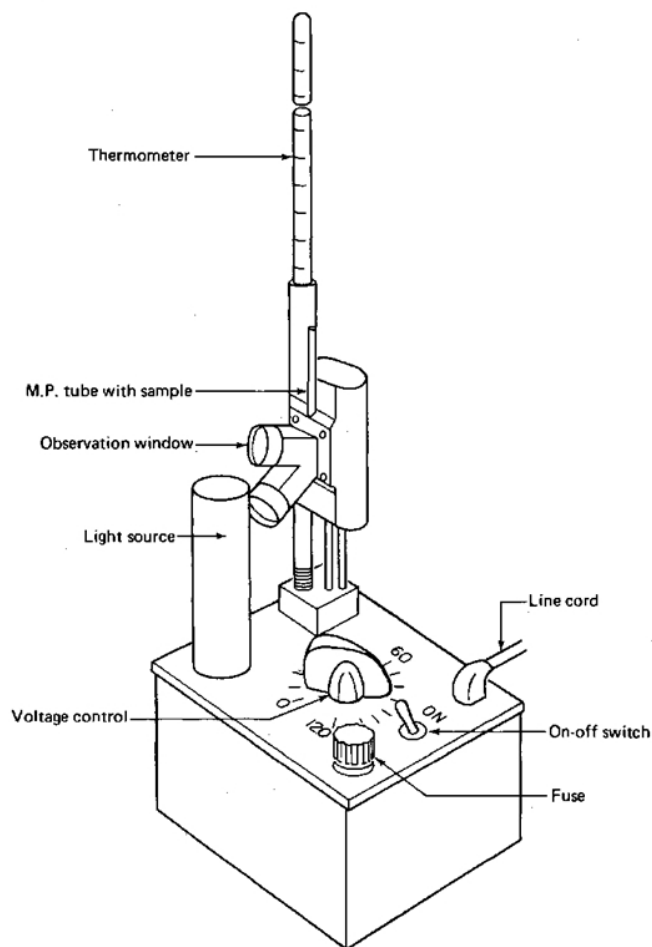


## MELTING POINT DETERMINATION

For 1<sup>st</sup> semester organic lab (CHEM 30121), a single melting point determination is generally sufficient. In the 2<sup>nd</sup> semester organic lab (CHEM 30143), you might need to do two measurements. The first one should be done with a fast ramp rate (fast rate of heating) to get a rough number. You then might need to do a second measurement with a slower ramp rate about 10-20 °C below what you measured the first time. You CANNOT use the same capillary again. Some compounds decompose and besides cooling the capillary will not give nice crystals.

### ANALOG INSTRUMENT (Mel-Temp II)



This instrument is very easy to use. In our case, the fuse is not visible, unlike the above scheme on the left. The rate of heating (ramp rate) is controlled with the knob ("voltage control"). For high melting solids ( $> 200\text{ }^{\circ}\text{C}$ ), this should be set to perhaps 5-6. Three capillaries can be handled at the same time. Once the compound has been placed in the capillary (this is done by tapping the open end of the melting capillary into the solid until there is 1-2 mm of solid in it), the tube needs to be packed. This is done by dropping the capillary open end at the top (where the solid is) down a narrow glass tube 2-3 times depending on the length of the glass tube. The solid should fall to the bottom of the capillary. The very long glass tubes can be placed vertically against the floor, the shorter ones can be placed vertically against the bench.

Place the capillary into one of the three slots. Turn the apparatus on (red or green switch), turn the knob to the desired value (ramp rate).

The solid will start "sweating" (ie the crystals on the outer side of the capillary will look "wet" when the sample is starting to melt or very close to it). Then there will be a clearly identified point where both liquid and solid are present, and finally it will be all liquid (the end point of your melting point/range).

Once your measurement is complete (all capillaries in the instrument have melted), turn off the green power switch, turn the knob down to zero. Then turn on the power strip nearby so that the fan helps cool the melting point apparatus. Remove the capillaries and throw them away in the reddish plastic box (“sharps disposal”).

The next person needs to switch off the fan (switch off the power strip) and then start the procedure from the top.

### DIGITAL INSTRUMENT (SRS DigiMelt)

We also have a digital melting point apparatus. It can also run 3 samples at once.

For the sample prep, the only difference is that when you have 1-2 mm of sample in the capillary you must wipe the capillary clean on the outside with a piece of paper (Kim Wipe or paper towel). Then the capillary should be placed in one of the three holes in the bottom right hand corner (#5 on the photo below) and then press “Tube Tapper”. This will pack the sample.

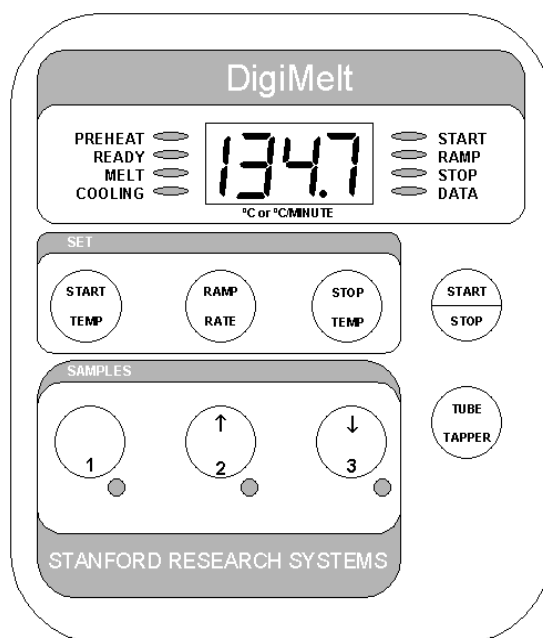
Place the capillary in one of the three holes above the viewing window (#2 on the photo below).

Instructions follow:

## Instrument Overview



# DigiMelt Quick Start Guide



*Schematic of DigiMelt Keypad*

- 1) Push **Start Temp** and use the **↑/2** and **↓/3** buttons to set the starting temperature (generally 20 degrees below the expected melting point).
- 2) Push **Ramp Rate** and use the **↑/2** and **↓/3** buttons to set the ramp rate (2 deg/min is suggested).
- 3) Push **Stop Temp** and use the **↑/2** and **↓/3** buttons to set the stop temperature (at least 5 degrees above the expected melting point).
- 4) Push **Stop Temp** again to return to the current temperature display.
- 5) Load capillaries with sample. Insert capillaries into the chassis holes near the **Tube Tapper** button. Press the **Tube Tapper** button to pack your samples.
- 6) Push **Start/Stop** to preheat the block to the starting temperature. The **Preheat** LED will light.
- 7) When the **Ready** LED becomes lit, the oven is holding at the start temperature. Insert your samples into the DigiMelt oven.
- 8) Push **Start/Stop** to begin ramping the temperature at the ramp rate. The **Melt** LED will light.
- 9) Observe your samples during the ramp.
- 10) Push the **1**, **↑/2** and **↓/3** buttons to record data (up to 4 temperatures per sample) during the melt. (To end the experiment before the stop temperature is reached, push the **Start / Stop** button.)
- 11) When the **Cooling** LED is lit, the experiment is over. If you recorded data, the **Data** LED is also lit.
- 12) To read back the data, push the **1**, **↑/2** and **↓/3** buttons (make sure the **Cooling** LED is lit).

A short version of these instructions is printed on the side of the instrument that faces the user.

The Start Temp should be significantly below (20-30 °C) what you are expecting, and if you do not know, it should be the lowest (perhaps room temperature to 35 °C). If you know what to expect, use a ramp rate of 10 °C/min and then decrease this to 2- or 5 °C/min when you are 10-20 °C below what you are expecting.

See the “ANALOG INSTRUMENT” section above about what a melting sample will look like.

**IF YOU ARE USING THE DIGITAL INSTRUMENT, THE TEMPERATURE SHOULD BE ROUNDED TO THE NEAREST INTEGER, even though the display gives one decimal place.**

#### **NOTE**

Analog or digital instruments seem equally good. However, the digital apparatus is limited to a maximum temperature of 250 °C, which should still work for 90% of the cases. Personally - call me old-fashioned - I still like the analog instruments best: I feel it is easier to turn a single knob clockwise or counterclockwise than pressing three buttons. But then again, I do not own an iPhone!

In your case, you should not have any particular preference. The students who used the digital instrument seemed to like it. Given the chance, use either one based on availability so you are getting experience on both instruments this semester.

If you take the second semester laboratory, you will need to measure a lot of melting points and in this case you will often not know the melting point. Again, this means you will need to run two measurements on different capillaries, the first time with a fast ramp rate and the second with a slow ramp rate starting below (~ 10-20 °C) what you measured the first time.