Melting Point[1]

Make sure to read the melting point chapter in the lab text (chapter 3).

Several devices are available for measuring melting points. Our lab is equipped with a computer-controlled digital instrument. There is a handout posted on the course website with instructions on how to operate it. Your TA will also demonstrate how to use it.

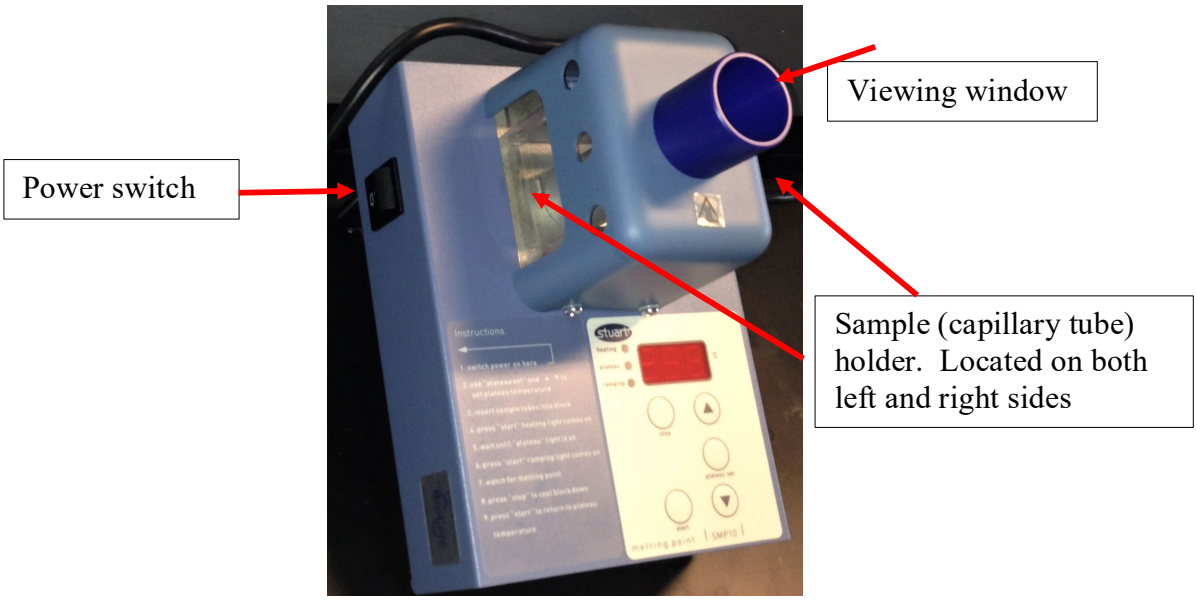

Figure 1. The digital melting point apparatus

To summarize chapter 3 of the lab text, melting points can provide information about the identity and the purity of a solid sample.

Prelab: You may either print out your prelab and bring it with you to lab, or bring your computer. Your TA will grade it on the spot before you begin the experiment. For the in lab observations, you may use scratch paper and record later in your ELN, or bring your computer and record directly in your ELN.

Postlab Report: Make sure to use the non-formal postlab report template on the course website!

Procedure[2]

The purpose of this experiment is to learn to determine melting points (MPs) accurately. This is an important technique that will be used in many of the experiments in the organic lab. Always record a MP range: the temperature at which the first drop of liquid appears, to the temperature at which all sample has melted. Be sure to distinguish between melting and movement of the solid due to expansion. The MP range begins when the first tiny drop of liquid is observed. Remember

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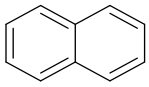
that an impure sample melts over a wide range and at a temperature lower than that of the pure material. For the purposes of this course, a compound with a MP range of 2 °C or less will be considered to be sufficiently pure.

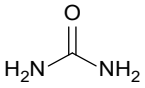
Filling the capillary tube: Adding sample is accomplished by pushing the open end of the capillary down into the powdered sample, then tapping the sample down into the closed end of the capillary tube by dropping the tube, closed end first, into a 2' length of glass tubing so the sample bounces and allows the solid to pack into the closed end. This can also be done by tapping the closed end gently on the desktop but care must be taken to not break the fragile tube. The height of sample in the capillary should be about 2-3 mm (thickness of two 25 cent coins). Too much sample will result in poor results.

Using the Mel-Temp device: See document posted on course website. **When finished using the apparatus, always turn off the power switch.**

(1.) Knowns: Determine the MPs of two known compounds. You will be using naphthalene and urea. Note that the MPs of samples used in the lab may differ slightly from those given in a handbook. Therefore, use the MP values given below. Samples will be dispensed in labeled dishes on the side bench. Do not move the dishes. Fill the capillaries right there. Determine the MPs of these known compounds in order of increasing MP. This will obviate the need to allow the apparatus to cool between determinations. The values that you find should agree well with those listed and the range should be narrow. If you find a wide range (> about 2 °C) or a value different from that expected (within ± 2 °C - remember that the thermometers used here are good only to about ± 2 °C), do a second determination on a fresh sample. It is important to use a small sample and to raise the temperature very slowly (about 1°C/minute) when you are near the MP of the sample. If a MP must be redetermined always use a fresh sample; once a sample has melted, you must assume that it may have decomposed, and therefore become contaminated with impurities, causing the MP to become depressed and the range widened. Never dispose of used capillaries or any glass in the regular trash. The person who empties the trash could be injured by broken glass. Place the used capillaries in the dishes provided. ALWAYS turn the device and digital thermometer off when finished.



Naphthalene
MP 79 - 80 °C



Urea
MP 132 - 134 °C

(2.) Determine the MP of one unknown compound. Your TA will assign your unknown to you. To save time, when you determine the MP of an unknown sample, first find an approximate MP by setting the temperature to a certain value to see if it melts. If it does not, again set the temperature at 10 degrees higher and see if it melts. Keep repeating until you get an approximate MP, and then redo it using a fresh sample by setting the temperature to about 10 degrees less the observed approximate MP. Using the MP, identify your compound using the table of possible compounds at end of this document.

BEFORE LEAVING THE LAB: Turn the power switch off. Clean up your work area and ask your TA for their signature.

WASTE DISPOSAL: Place used MP capillary tubes in the evaporating dishes on the side benches. Never place any glass into the trash. The custodian could become injured with broken glass.

SAFETY: Keep all lab chemicals off of your skin.

Postlab Questions

1.) A solid sample has a MP of 133 - 137°C. What can one conclude about the sample?

2.) For question 1, if the sample is one of four possible compounds the melting points of which are 133°, 135°, 137°, and 139°C, which is it most likely to be? Why?

3.) Two test tubes contain compounds having the same MP. Using MPs, how could you determine whether the two test tubes contain the same or different compounds?

4.) In a recrystallization (a technique that you will encounter later in the semester), a solid is dissolved in a solvent and later the solvent is removed. If a MP of the sample is taken while the sample is still moist with solvent, what effect would that have on the MP of the sample?

5.) What two pieces of information can a MP determination provide?

Possible Unknown Compounds

Compound	Melting Point (°C)
4-Methylphenol	35
Benzophenone	48-50
Maleic anhydride	54-56
4-Bromophenol	64-66
4-Aminobenzaldehyde	71
Naphthalene	79-80
3,4-Diaminotoluene	89-90
Acenaphthene	94-96
Isobutyranilide	106-107
Acetanilide	113-115
Benzoic acid	122-123
Urea	132-134
d,l-Glyceraldehyde	145
Adipic acid	152-154
Sulfanilamide	165-167
2-Aminophenol	174
4-Toluic acid	180-182
Succinic acid	187-189

The melting points listed here vary slightly from those found in reference texts. These are closer to what you will observe because our compounds are not ultra-pure as ultra-pure compounds are ultra-expensive!!