Lab 1 – Melting Point Determination

Introduction:
Melting Points are done routinely in chemical laboratories. The melting point of a sample provides an indication of the purity of the sample. A pure sample has a sharp melting point range: usually between 0.5 to 1.0ºC. Impurities will generally cause the sample to melt over a range of several degrees and at a significantly lower temperature than that of the pure compound. These concepts have important applications with regard to determining the identity of a compound. Even though several compounds may have the same melting point, accurately determining the melting point of a pure compound will eliminate the possibility of it being many others.

Procedure:
There are various methods used to determine melting points. One of the most common methods employs a Mel-temp apparatus. As many as three different sample tubes can be inserted into the apparatus at the same time. Voltage settings can be chosen by using the time vs temperature chart provided.

1. Choose any two (2) of the eight (8) known compounds that <u>have melting points more than twenty (20) degrees apart.</u> Determine and record the melting point range for each compound. Your Experimental values should agree with the literature values of the known compounds tested.

2. Choose two (2) of the eight (8) known compounds that <u>have melting points less than six (6) degrees apart.</u> Intimately mix approximately equal amounts of the two known compounds on a clean and dry watch glass before putting the mixture into the capillary tube. Place 1-2mm of the mixture into the capillary tube by pressing the open end into the mixture a few times and then dropping the capillary tube, closed end down, through about 15 inches of glass tubing. The capillary tube should bounce a few times until the sample gets packed into the closed end of the tube. Determine and record the melting point range for the mixture. Your experimental value should be significantly below the literature values of the two known compounds and the mixture should be observed to melt over a wide temperature range. The mixing of two samples and observing the melting point of them combined is known as the "Mixed Melting Point Technique". This technique will be used to confirm the identity of an unknown.

3. Determine and record the melting point range for the mixture. Your experimental value should be significantly below the literature values of the two known compounds themselves and the mixture should be observed to melt over a wide temperature range.

4. Obtain an Unknown, record the Unknown Number, then determine and record its melting point range.

5. Choose the two known compounds that melt less than 6ºC above or below the melting point of your unknown. Do mixed melting points for mixtures of your

unknown compound mixed with each of these known compounds. To prepare a sample for a mixed melting point, intimately mix approximately equal amounts of the unknown compound with the known compound on a clean and dry watch glass before packing the mixture into a capillary tube.

6. Since your Unknown is one of the 8 known compounds, you should be able to identify the Unknown on the basis of the data you have recorded.

    Note: When determining the identity of an unknown, it is not sufficient to just compare melting points. Many compounds may have the same melting point. Confirmation must be made by the mixed melting point technique. For example, one of the two known compounds when mixed with the unknown should have a lower melting point than its literature value. The other, since it is being mixed with itself should melt at the literature melting point.

The data sheet outline below is provided as a guide to the student. The student should keep data in a bound notebook and a full laboratory report must be submitted to the instructor within one week of the completion of the lab work.

Data:

In the table below record the name and the literature melting points of all of the known compounds available. List the known compounds in order of increasing melting points.

Name of Known   Lit. M.P.   Name of Known   Lit. M.P.

_____   _____

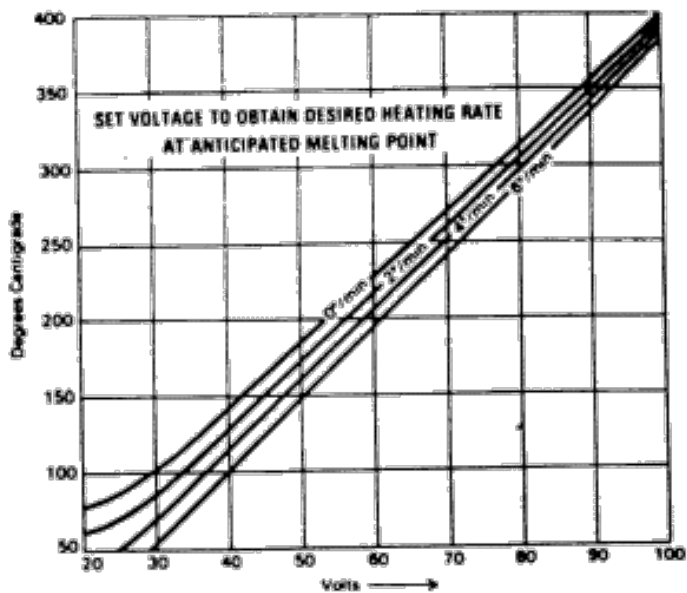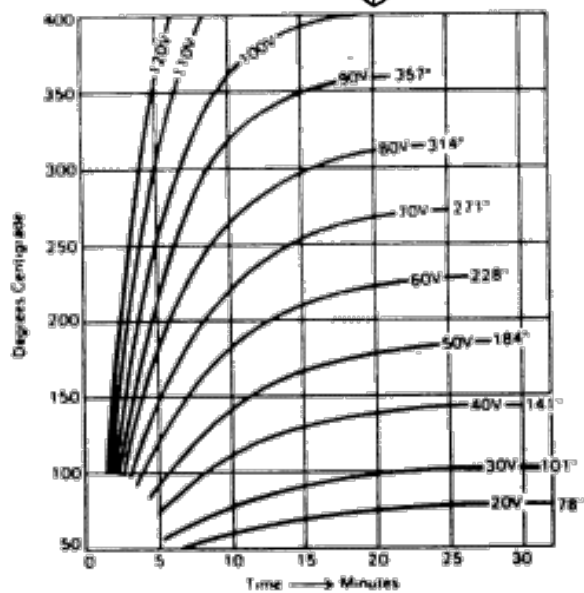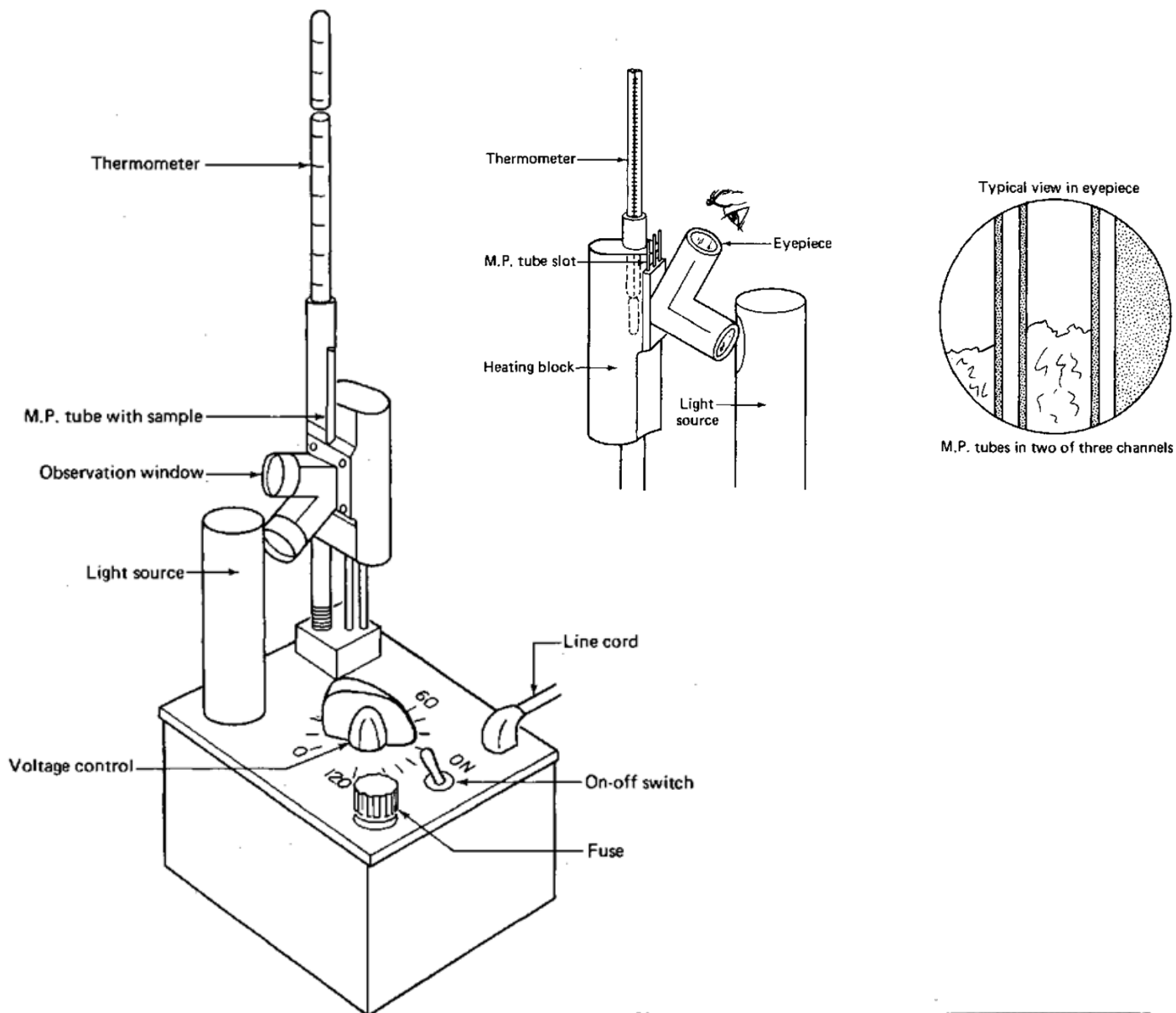_____   _____

_____   _____

_____   _____

Experimental Data:
Enter the name, the literature melting range, and your observed melting point range for the two known compounds you chose.

I.	Known Compounds	Lit. M.P.	Obs. M.P.	Deviation (Lit.-Obs.)
	_____	_____ºC	_____ºC	_____ºC
	_____	_____ºC	_____ºC	_____ºC

II.   Determine a mixed melting point for two known compounds.

Compound A: _____   _____ºC (Lit. M.P.)

Compound B: _____   _____ºC (Lit. M.P.)

Mixture melting range: _____º C

III.   Unknown #_____   Observed M.P._____ºC

IV.   Mixed Melting Points – Unknowns mixed with possible known compounds.
        Select possible known compounds from the table.

Unknown + _____   _____ºC (Obs. M.P.)
                    (Possible known)

Unknown + _____   _____ºC (Obs. M.P.)
                    (Possible known)

Conclusions:
Include the identity of your unknown and your reasons for this choice. Be sure to explain
how the mixed melting point technique confirmed your conclusion.

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Thermometer

M.P. tube with sample

Observation window

Light source

Voltage control

Line cord

On-off switch

Fuse

Thermometer

M.P. tube slot

Heating block

Eyepiece

Light source

Typical view in eyepiece

M.P. tubes in two of three channels

120V
110V
100V

90V — 357°

80V — 314°

70V — 271°

60V — 228°

50V — 184°

40V — 141°

30V — 101°

20V — 78°

Degrees Centigrade

Time ——> Minutes

SET VOLTAGE TO OBTAIN DESIRED HEATING RATE
AT ANTICIPATED MELTING POINT

Degrees Centigrade

Volts ——>

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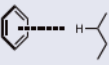
Safety Precautions
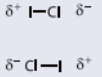
1. Never insert a room temperature thermometer into a hot Mel-Temp. It may shatter.
2. Do not leave a Mel-Temp on if you are not actually using it, or leave it running unattended.
3. Do not heat the Mel-Temp beyond the upper temperature of the thermometer.
4. Do not touch the heating block – it gets very HOT!

General Information

1. Use no more than 2 mm of sample in the capillary tube. A larger sample may lead to non- uniform heating, and poor results.
2. It is virtually impossible to determine precisely a melting *point* (i. e. 123 ºC). Rather, chemists actually determine a melting *range* (i. e. 122 – 124 ºC). The first temperature in the melting range is recorded when the first signs of liquid droplets appear in the sample, and the second temperature is recorded when the entire sample becomes liquid.

3. Sometimes, the following may be observed:
   a. The sample will "settle" in the melting point tube
   b. Solvent will condense on the cooler portions of the tube

   It is important to note that these phenomena are **not** melting.

Intermolecular forces explained

Interaction	Image	Strength / kJ M⁻¹	Comments
Ion – ion		100–350	Comparable to a covalent bond in strength. However, non-directional and non-selective.
Ion – dipole		50–200	Strong attraction between a molecule with a permanent dipole and an ion.
Dipole – dipole		5–50	Electrostatic interaction between two molecules with permanent dipoles.
Hydrogen bonds		4–120	Arise between an atom bearing a lone pair of electrons (here, O) and a hydrogen atom bonded to an electronegative atom. Useful because of their high degree of directionality. Several H-bonds positioned correctly can add up to give very strong attractive forces.
π–π stacking		0–50	Arise from sharing of electron density between two aromatic (benzene-like) units. The effect is most powerful when there is a significant difference between the partial charges of the interacting units.
CH–π		4–12	Arise from dispersion forces. Controversial forces which are not easy to differentiate from the hydrophobic effect.
Van der Waals		< 5	Arise from random fluctuations in electron density, leading to brief moments of electromagnetic attraction between molecules. Extremely weak and non-directional. Not generally useful to supramolecular designers.

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Using the Mel-Temp Melting Point Apparatus

I. Establishing an **Approximate** Melting Point

The Mel-Temp apparatus is the most efficient way to quickly establish an approximate melting point for substances whose melting point is unknown. An approximate melting point can be taken using a rate of temperature rise of about 7 – 10 ºC/min.

1. Make sure a thermometer is inserted into the Mel-Temp. If not, place the bulb end of a thermometer (with an upper temperature of at least 250 ºC) in the opening.

2. Turn on the apparatus using the on/off switch 3. Insert melting point capillary tube into the holder 4. Turn the voltage control knob to achieve a rate of temperature rise of about 7 – 10 ºC/min.

4. Observe the approximate melting range of the sample by looking through the observation window. If the rate of temperature rise slows to less than 5 ºC/min, turn the voltage control knob to a higher value to increase the rate.

5. When finished, turn the voltage control knob to "0" or turn the unit off.

II. Establishing a **Precise** Melting Point
Once an approximate melting point has been established (or if the melting point of the substance is known from the literature) the Mel-Temp apparatus can be used to find the precise melting range for the substance. To establish a precise melting point, a rate of temperature rise of 1 – 2 ºC/min must be used.

1. Make sure a thermometer is inserted into the Mel-Temp. If not, place the bulb end of a thermometer (with an upper temperature of at least 250 ºC) in the opening.

2. Turn on the apparatus using the on/off switch

3. Turn the voltage control knob so as to establish a starting temperature about 5 ºC below the approximate melting point found from part I (or about 5 ºC below the literature value for the substance)

4. Insert melting point capillary tube into the holder.

5. Adjust the voltage control knob so as to establish the rate of temperature rise to be 1 – 2 ºC/min

6. Observe the precise melting range of the sample by looking through the observation window. If the rate of temperature rise slows to less than 0.3 ºC/min, turn the voltage control knob to a higher value to increase the rate.

7. When finished, turn the voltage control knob to "0" or turn the unit off.