

Melting Point

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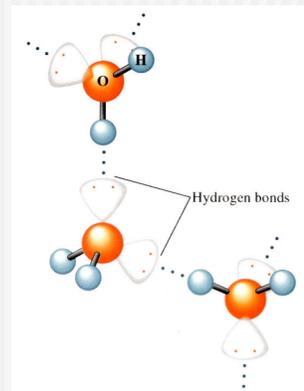
Phase Changes

- | | |
|------------------|----------------|
| ➤ Solid → Liquid | ➤ Melting |
| ➤ Liquid → Solid | ➤ Freezing |
| ➤ Liquid → Gas | ➤ Vaporization |
| ➤ Gas → Liquid | ➤ Condensation |
| ➤ Solid → Gas | ➤ Sublimation |
| ➤ Gas → Solid | ➤ Deposition |

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Intermolecular Forces

- Many properties of liquids, solids & gases can be explained on basis of weak attractive forces between molecules: Hydrogen bonds & Dipole-Dipole forces.
- H bonds: attractive force between positive H on N, O, or F & a N, O, F on another molecule. Example: Water



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Intermolecular Forces van der Waals forces

- Dipole-Dipole Interactions: Weak attractive forces between opposite charged dipoles. Occurs with polar molecules such as H-Cl
- Induced Dipole Interactions: Weaker attractive forces (London Forces) between temporarily induced dipoles. Occurs with non-polar molecules such as Br₂
- Strengths:
 - Covalent Bond ~ 400 kJ/mol
 - H Bond ~ 20 - 60 kJ/mol
 - van der Waals < 10 kJ/mol

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Attractive Forces & Solids

- Solids can be:
 - 1) Amorphous with no ordered structure (glass)
 - 2) Crystalline with a well defined 3D structure
- Types of Solids:
 - Molecular – molecules held together by intermolecular forces (Ice).
 - Metallic – metal atoms in sea of electrons (Fe).
 - Ionic – cations & anions held together by opposite charge attractions (NaCl).
 - Covalent – Solids held together by large networks of covalent bonds (graphite).

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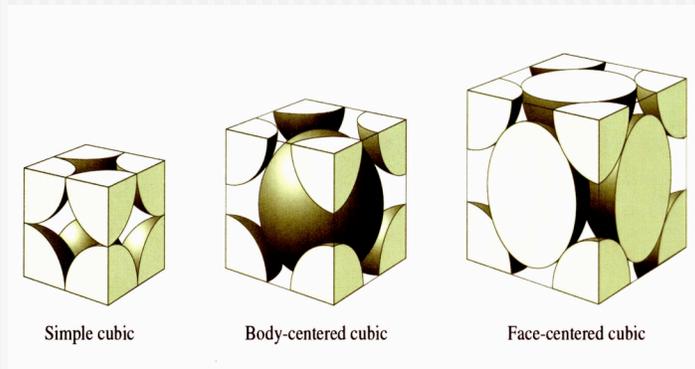
Attractive Forces & Solids

- Crystalline solids have ordered, repetitive 3d structure that we call the crystal lattice.
- Smallest boxlike unit that makes up the crystal lattice is called the unit cell.
- There are several unit cell shapes: Cubic, Hexagonal, etc.
- Three types of Cubic Unit Cells are:
 - ❖ **Simple Cubic Unit Cell**
 - ❖ **Body-Centered Cubic Unit Cell**
 - ❖ **Face-Centered Cubic Unit Cell**
- Exact crystal structure can be determined by X-Ray Crystallography.

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Attractive Forces & Solids

Cubic Unit Cells



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Melting Point

- **Temperature at which a transition occurs between solid and liquid phases**
- **Temperature at which an equilibrium exists between the well-ordered crystalline state and the more random liquid state.**

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Melting Point Range

- **The first point (lower temperature) is the temperature at which the first drop of liquid forms amongst the crystals.**
- **The second point (higher temperature) is the temperature at which the entire mass of solid turns to a clear liquid.**

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Application of Melting Point

- **Identify Compounds**
- **Establish Purity of Compounds**

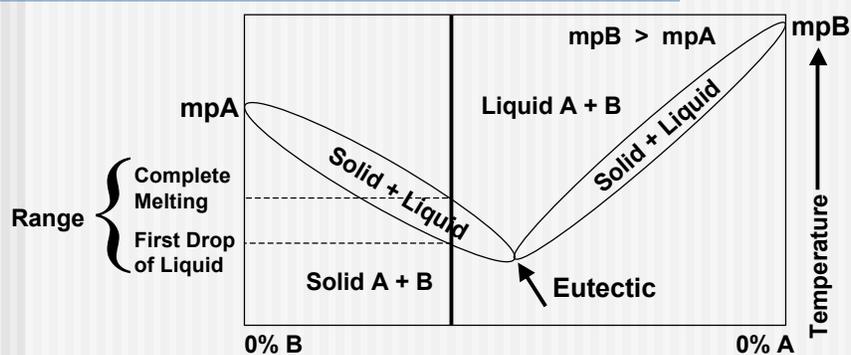
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Melting Point

- **Melting point indicates purity in two ways**
 - ❖ The purer the compound, the higher the melting point
 - ❖ The purer the compound, the narrower the melting point range
- **Melting point of A decreases as impurity B is added**
- **Eutectic point is the solubility limit of B in A; thus, it is the lowest melting point of an A/B mixture**
(Note: sharp melting point – no range at eutectic point)

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Phase Diagram of Mixture



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Sample Preparation Procedure

- **Crush sample**
- **Loading the capillary tube**
- **Tap open end of tube into sample (1-2 mm of sample)**
- **Drop tube (closed end down) down a length of glass tubing letting it bounce on table – sample is transferred to closed end of capillary tube. Repeat, if necessary.**

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Obtain the “Melting Point Range”

- **Place capillary tube with sample at the bottom of the tube in a Mel-Temp apparatus.**
- **Adjust temperature knob until temperature rises about (2-3°C per minute)**
- **Determine rough melting point.**
- **Allow capillary tube to cool until liquid solidifies.**
- **Reset temperature knob for a slower rate of temperature increase.**
- **Allow temperature to rise to 10°C below “rough MP”**
- **Reset temperature knob so that temperature rises no more than 0.5°C/Min.**

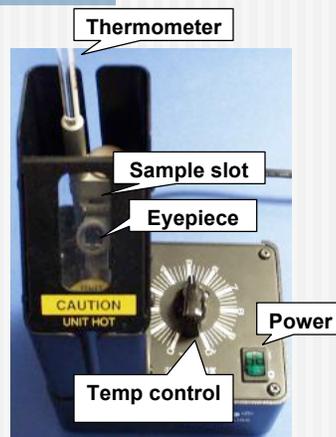
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Experimental Procedure

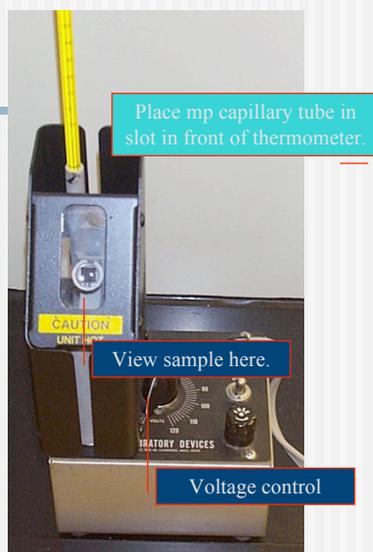
Turn the Mel-Temp power on and adjust the temp control to the desired rate of heating.

In order to obtain an accurate mp, it is necessary to heat **SLOWLY**, at a rate of 2-3°C/min.

Heating too fast may lead to inaccurate results because of insufficient time for heat transfer.



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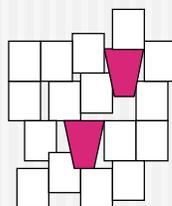
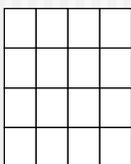


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Theory of Melting Point

> Impurities lower melting point:

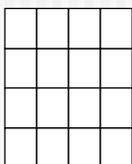
- ❖ takes less energy to disrupt crystal lattice when impurities are present
- ❖ melting point will be lower
- ❖ melting point range will be broader



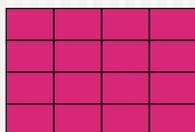
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Theory of Melting Point

> Mixed melting point - used to determine identity of compound:



Urea
MP 120-121°C



Cinnamic acid
MP 120-121°C

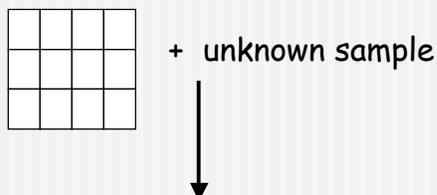
Have unknown compound
that melts at 120-121.
What is the unknown?

How could you tell?

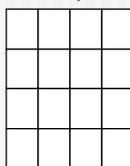
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Mixed Melting Point

- **Mix unknown compound with a little urea and measure melting point**



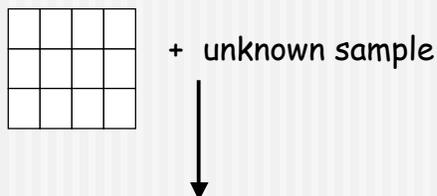
If melting point is still 120-121°C, unknown compound was urea.



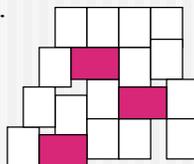
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Mixed Melting Point

- **Mix unknown compound with a little urea and measure melting point**



If melting point is lower and broader, i.e. 110-116°C, unknown compound was NOT urea.



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