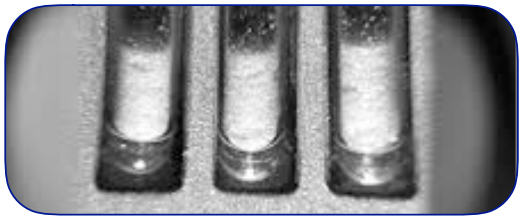
Ex10

Purity and Melting Point

- Melting Point
  - Characterization
  - Purity & Melting Point Depression
  - Observations
  - Techniques



- The Experiment
  - A - Powder
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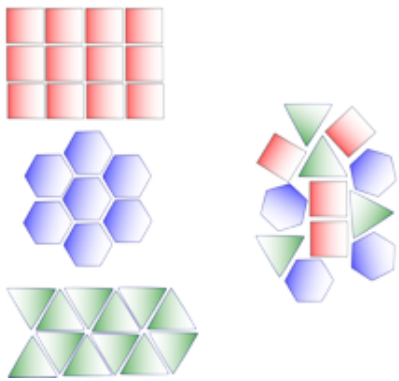
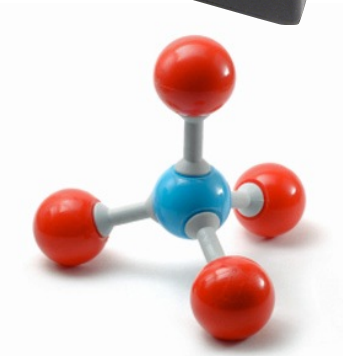
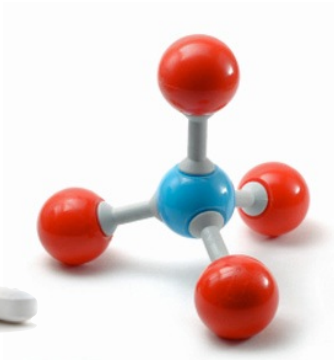
- For Next Week





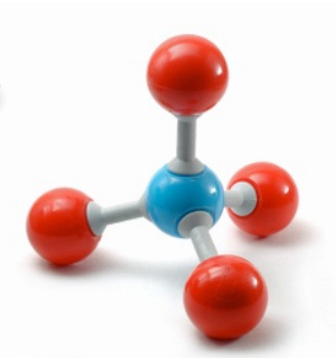
Purity and Melting Point

- Chemistry lets us accurately and effectively predict the properties of new substances.
- We design and build materials that have a wide range of applications in biology, medicine, nutrition, construction and other fields.
- But those processes rarely produce a pure material.
- Some of the greatest challenges chemists face is not producing the right material, but getting it sufficiently pure.
  - The construction of the new bay bridge in San Francisco was recently halted because metal pins used in it's construction were found to not have been made sufficiently pure.
  - The prescription drug thalidomide resulted in children being born with malformed limbs — not because of the drug itself, but because chemists failed to sufficiently purify it.



Purity and Melting Point

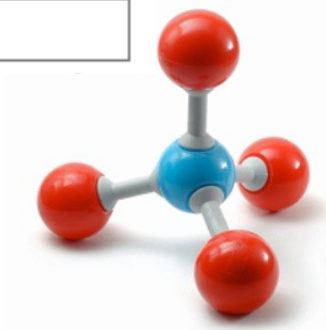
- One of the oldest and still most reliable means of characterizing a substance is it's melting point.
  - It's usually the first property we measure of any new substance we discover or create.
  - It often defines a substance we hope to reproduce or discover elsewhere.



Purity and Melting Point

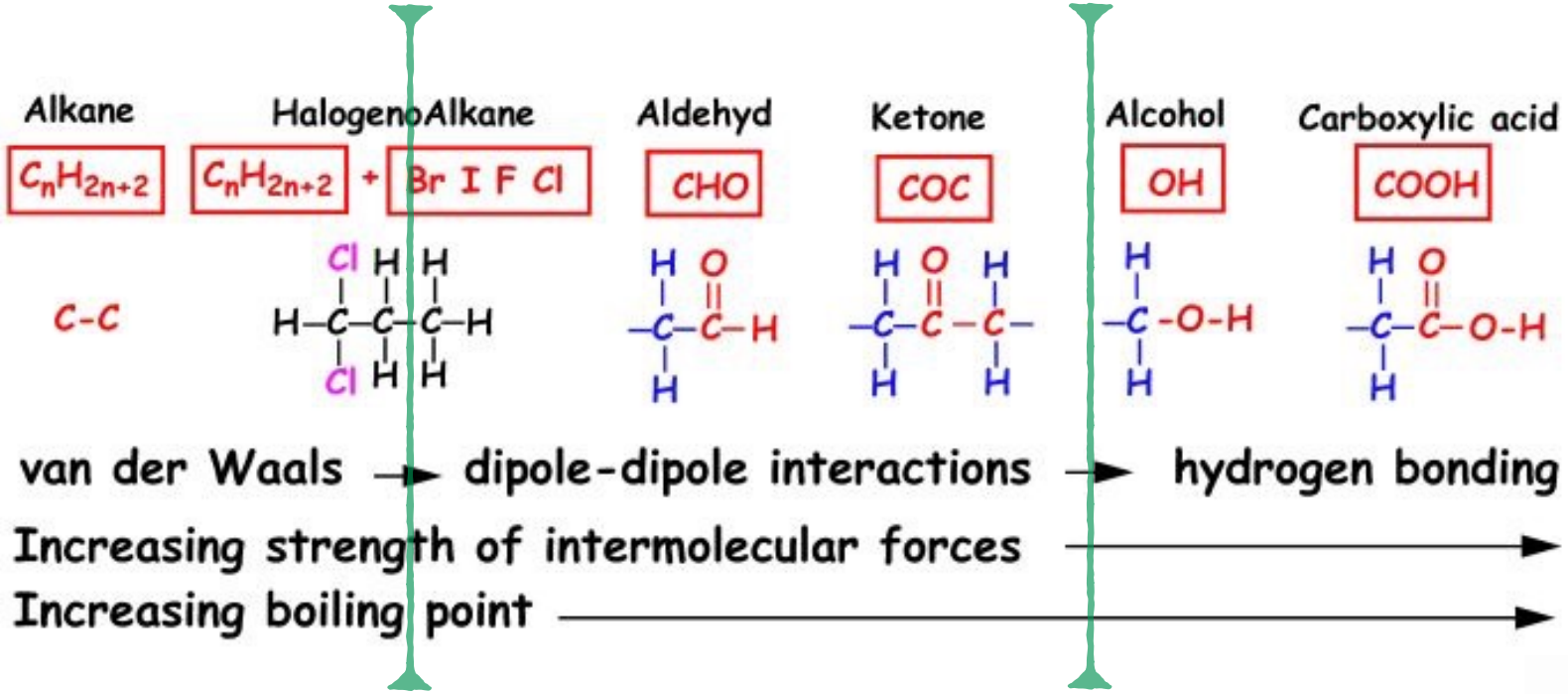
- Most of the 50 million unique substances the human race knows about are uniform white solids when pure.
- But even very similar compounds can have very different melting and boiling points.

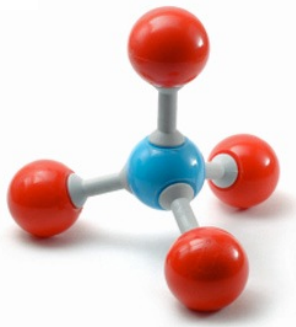
	Melting Point (°C)	Boiling Point (°C)
$LiCl$	610	1382
$BeCl_2$	405	488
CCl_4	-23	77
NCl_3	-40	71
OCl_2	-20	4
FCl	-154	-101
$NaCl$	808	1465
$MgCl_2$	714	1418
$SiCl_4$	-68	57
PCl_3	-91	74
SCl_2	-122	59
Cl_2	-102	-35
KCl	772	1407
$CaCl_2$	772	>1600



Purity and Melting Point

- For organic compounds it can reveal subtle differences in structure.
- The same trends we observe in boiling point apply to melting points.

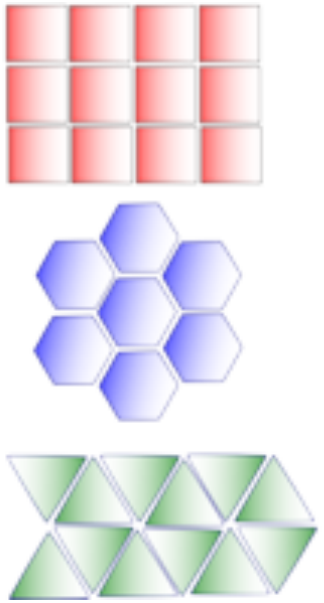


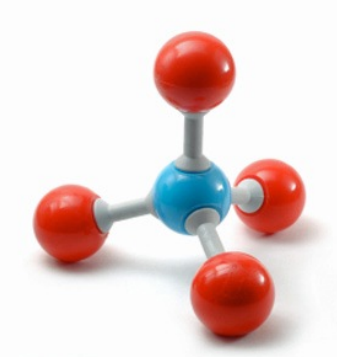


Purity and Melting Point

- Because each molecule has a unique shape, mixtures of substances always have a lower melting point that either substance when pure.
- This is why we salt frozen roads, to make the ice on them melt at lower temperatures.

- Melting point depression is therefore a measure of how pure a substance is. Even very small impurities can substantially reduce a melting point.





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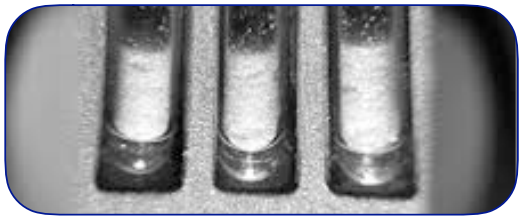

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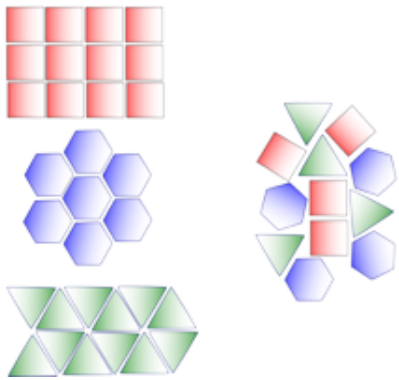

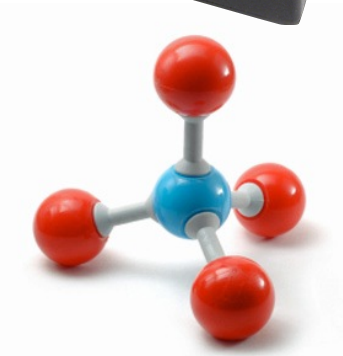

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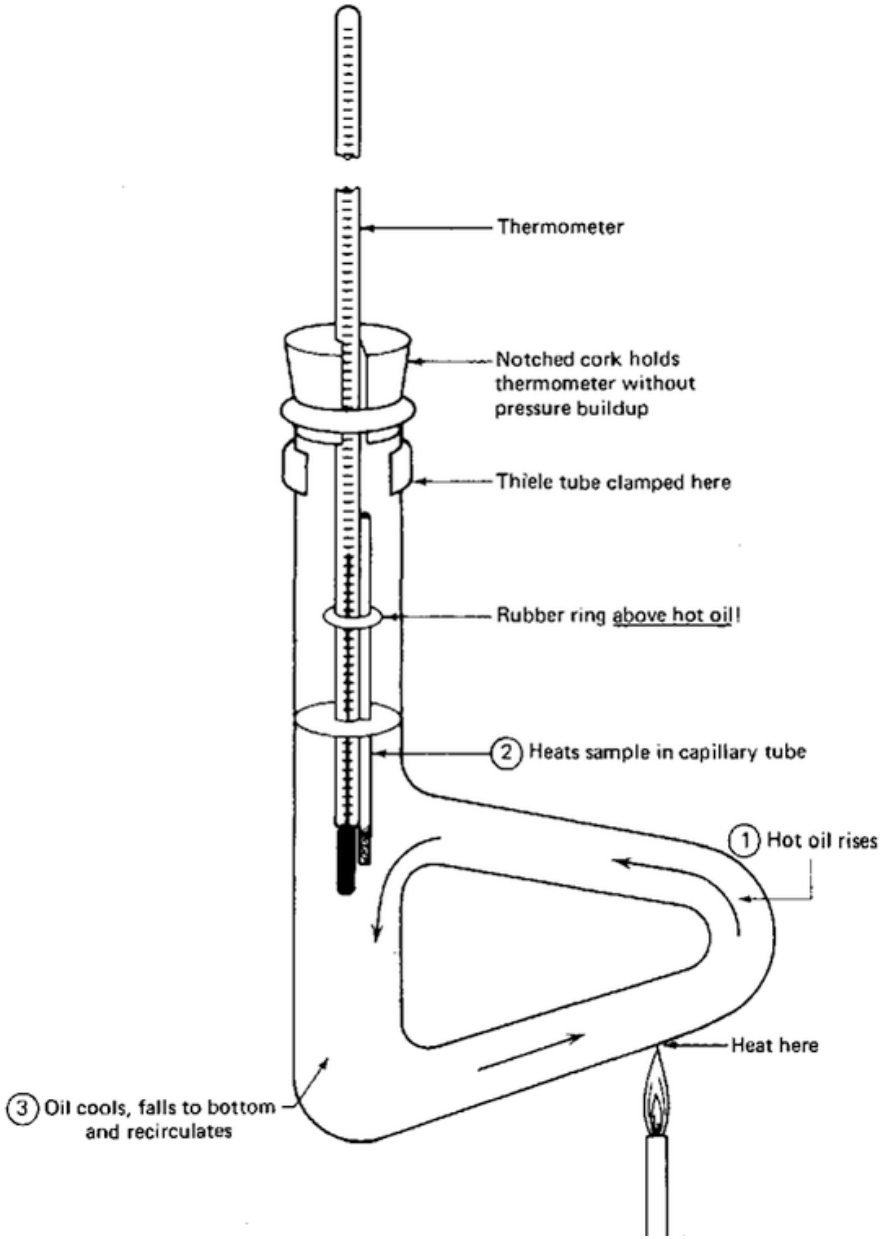
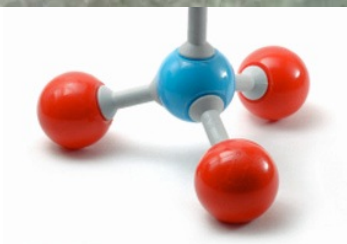


- Melting point determination involves slowly heating a substance in a thin capillary and observing it's behavior.
- Melting points are pressure dependent and pressure will change as you heat a sample.

Thiele Tube



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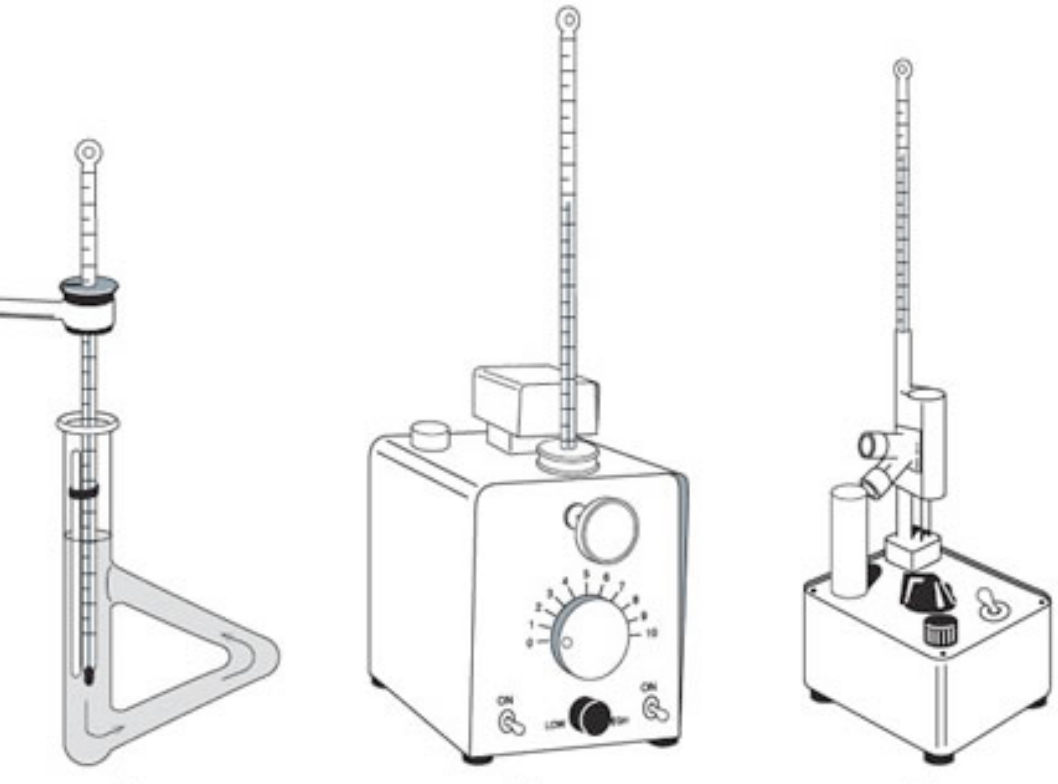

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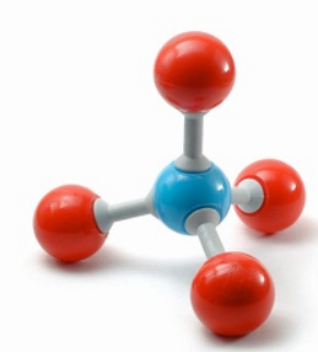

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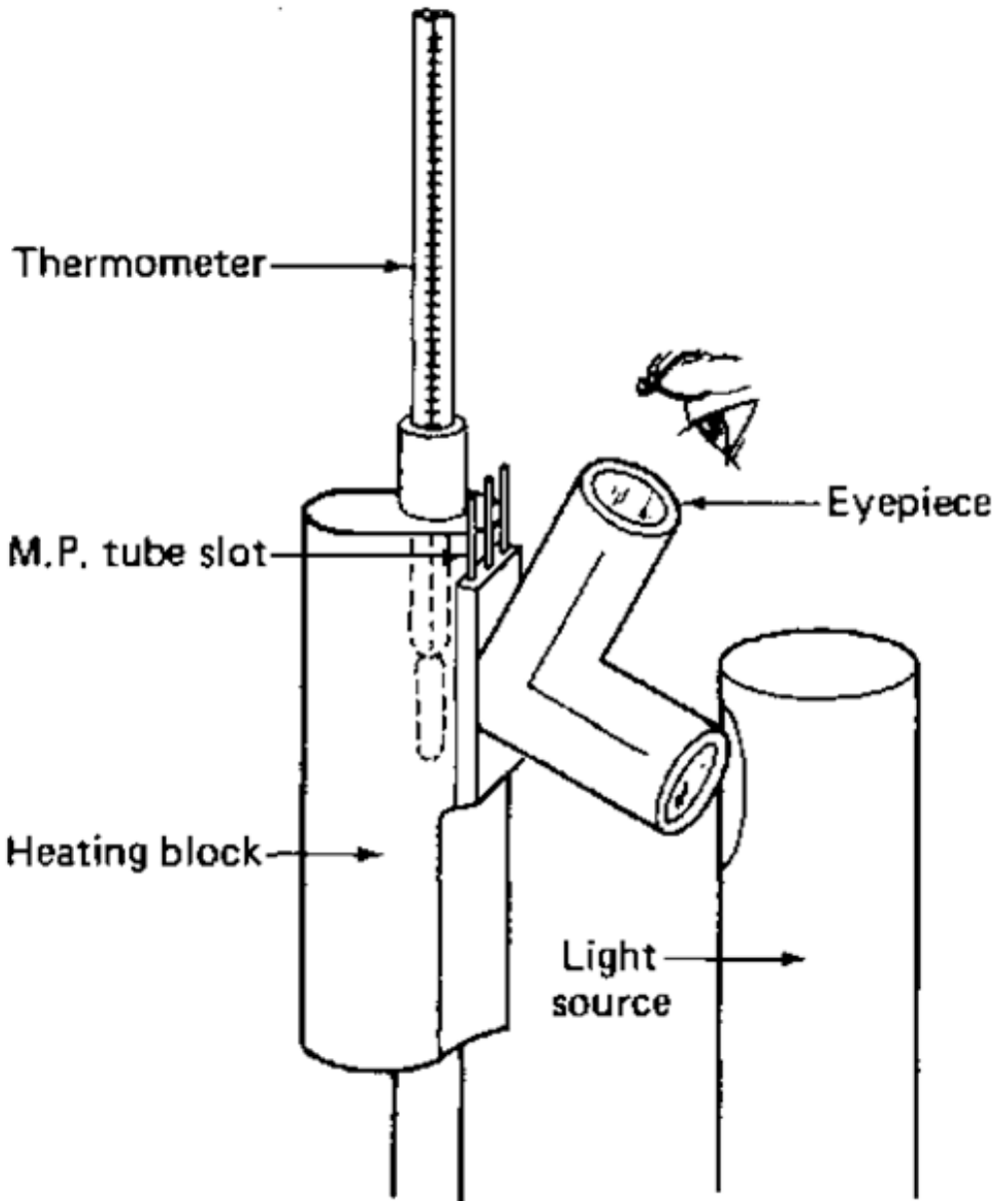

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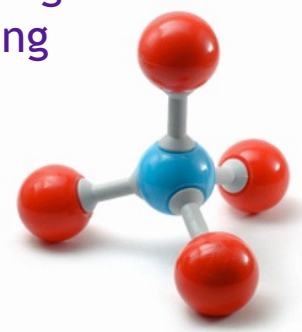
- We've improved on the process by adding magnifying glasses and light sources, but capturing a melting point is otherwise the same process we've used for thousands of years.



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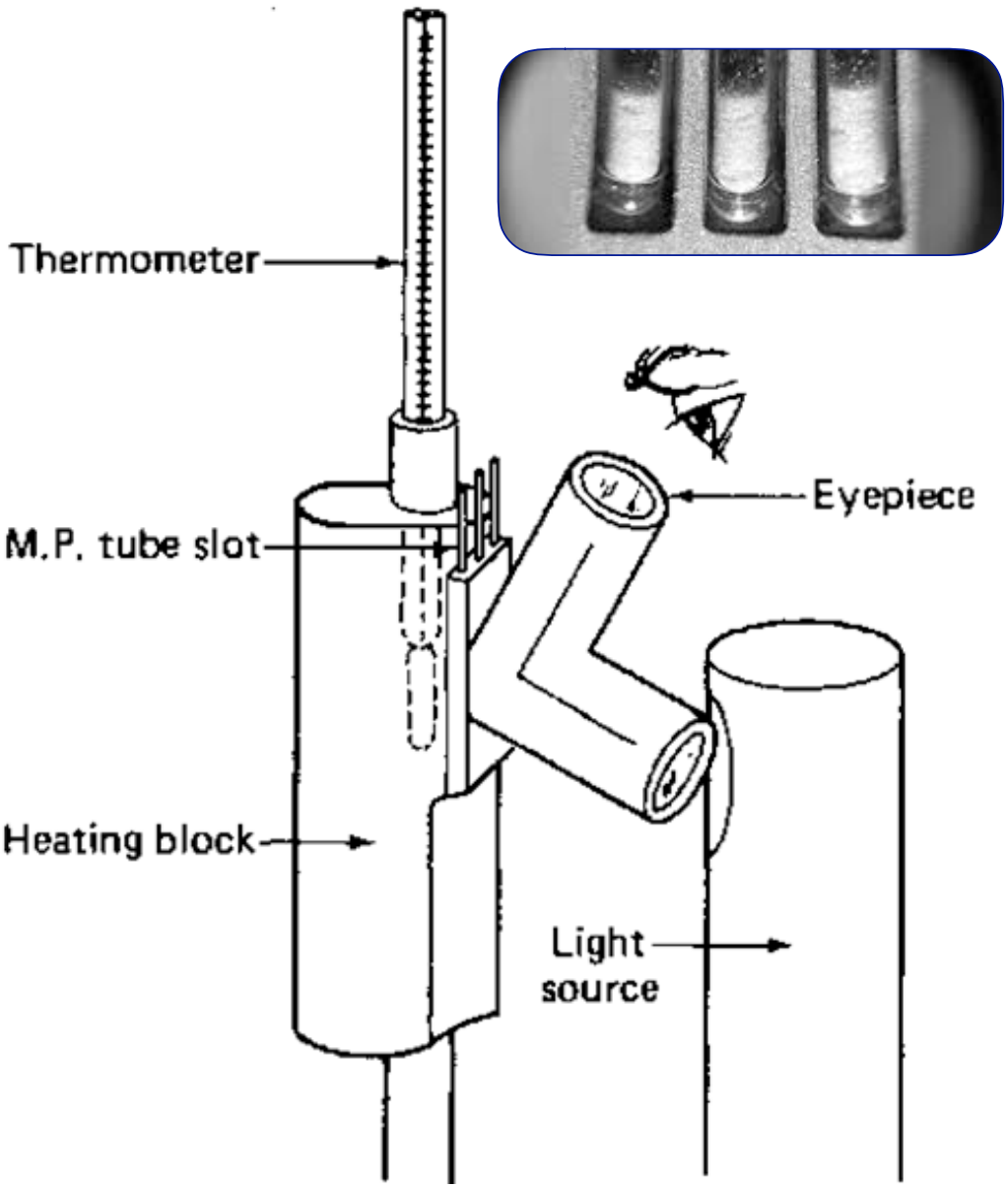

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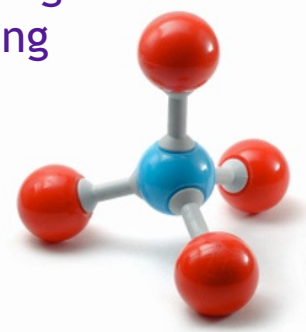
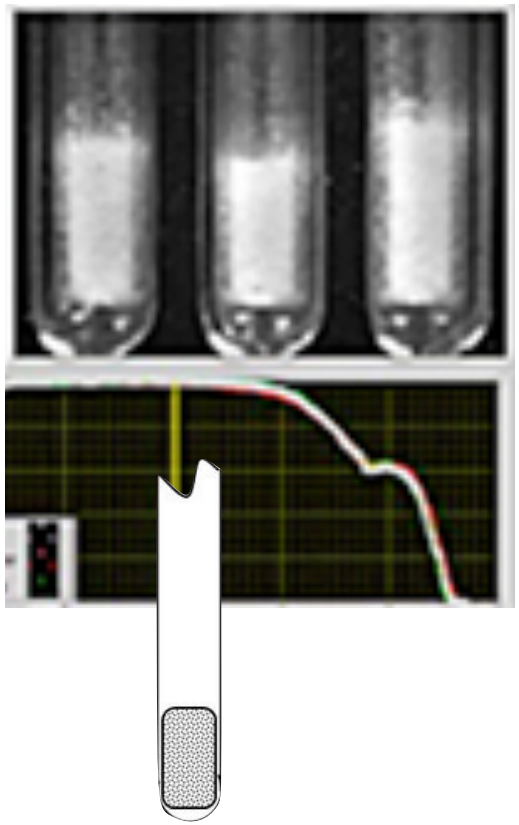
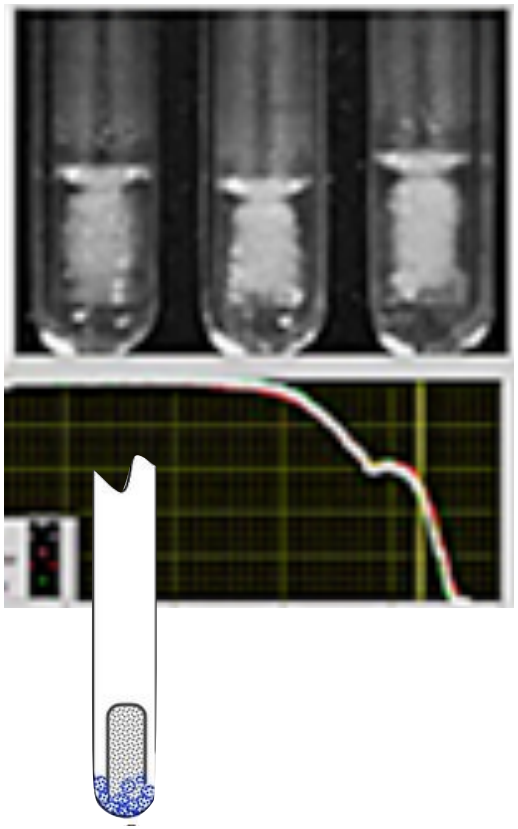
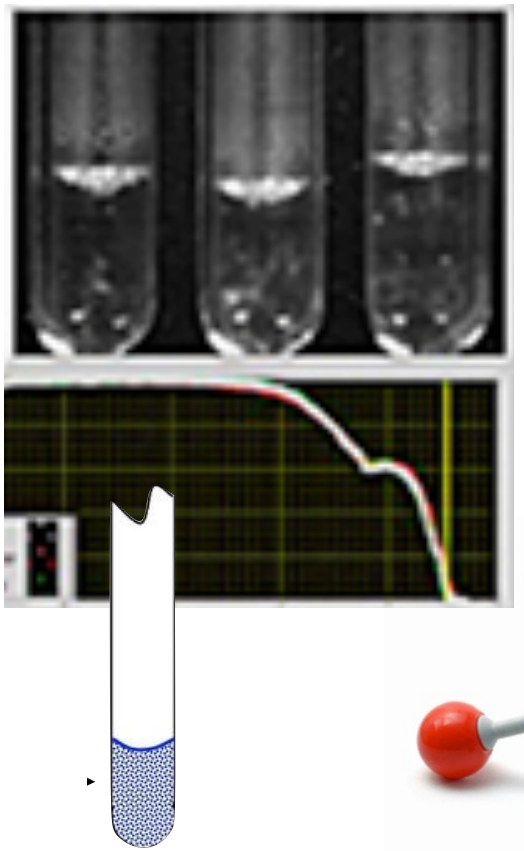


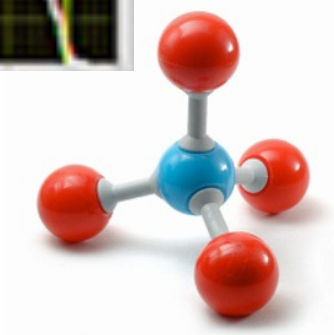
Purity and Melting Point

- Record a Range for your melting point.
- Each range will have a
  - start temperature, where you first see the material melting.
  - end temperature, where the last solid becomes liquid.



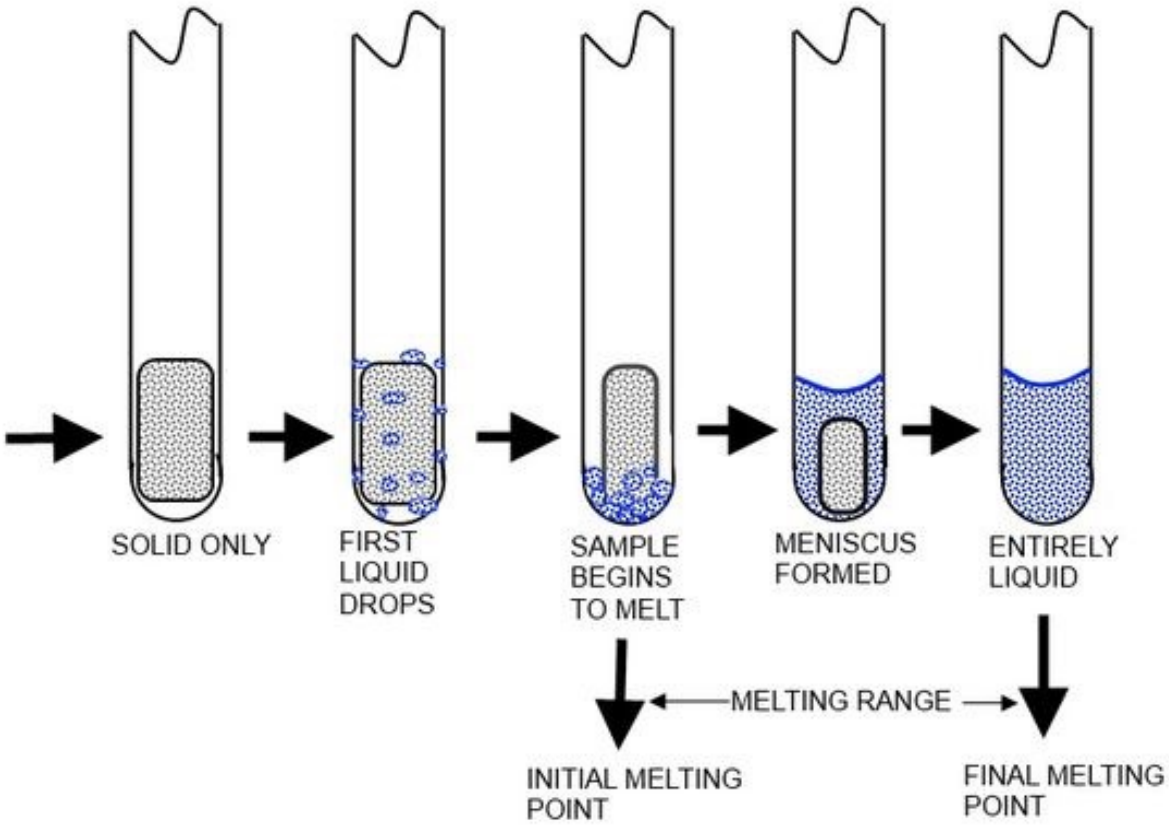


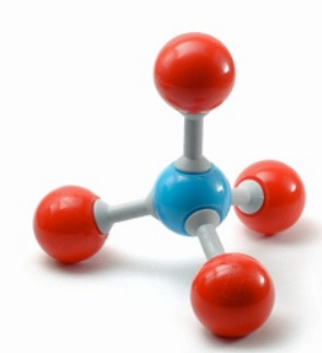


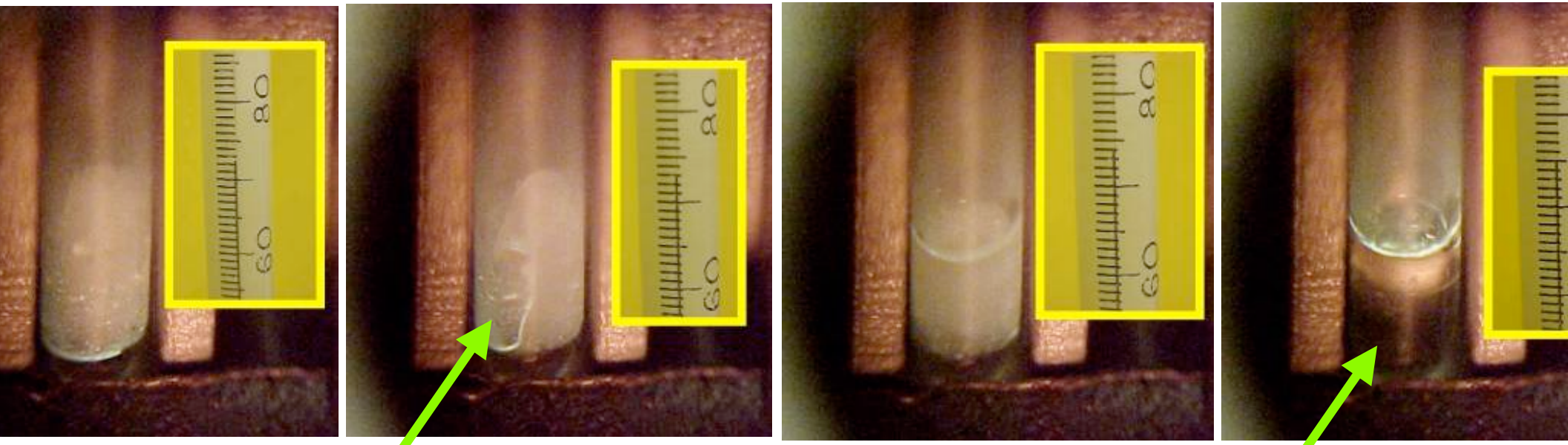


Purity and Melting Point

- Record a Range for your melting point.
- Each range will have a
  - start temperature, where you first see the material melting.
  - end temperature, where the last solid becomes liquid.
    - the start temperature is not when you first see crystals shaking or sweating, but when liquid starts to pool at the bottom of the tube



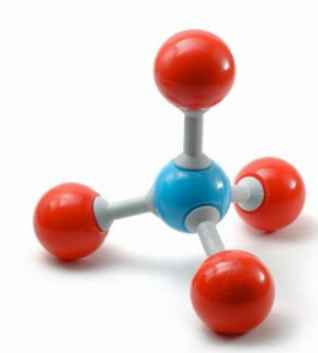




72˚ liquid observed

75˚ last solid is now liquid

Record the melting point as 72-75˚C



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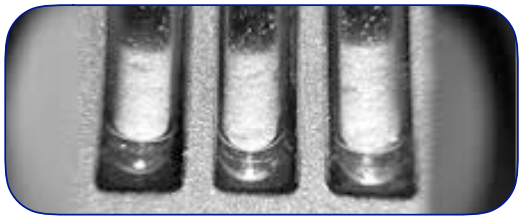

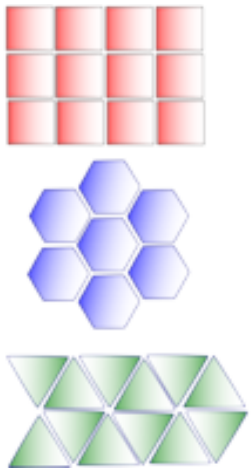

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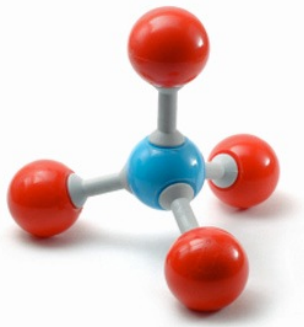
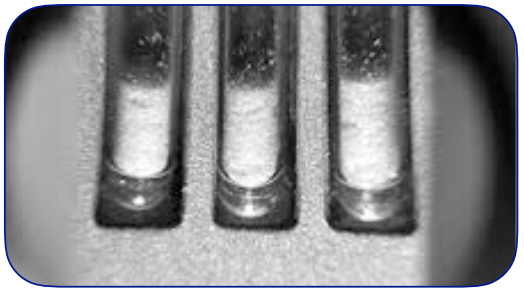
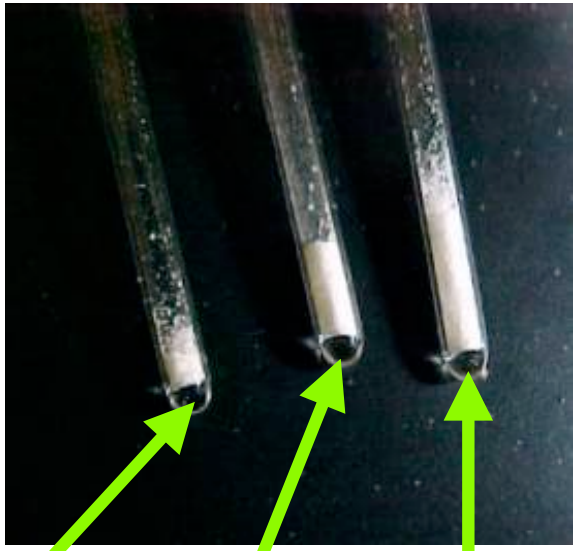
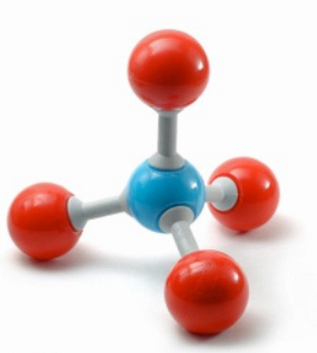


Purity and Melting Point

- Load about 1 cm of sample, tightly packed into a capillary.
- To load the sample, spread out some of your solid on a watch glass and stab at it with the open end of your capillary.
- The knock the closed end against a table to cause it to settle into the other end.

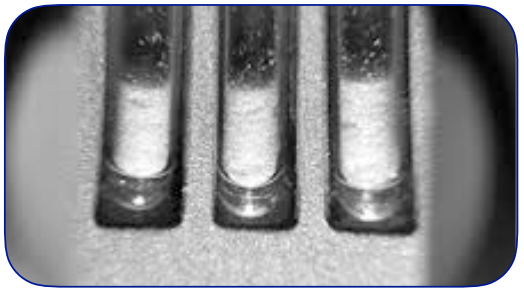




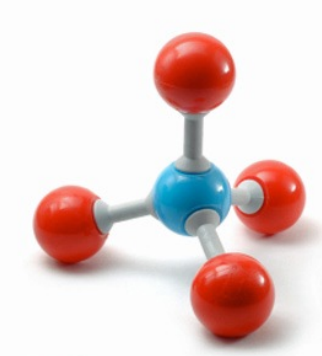


Purity and Melting Point

- Use a shaker on your DigiMelt or drop the tube through a buret against a table to pack it well.
- If there substance isn't firmly packed into the close end of the tube you will get a very large range and inconsistent results.

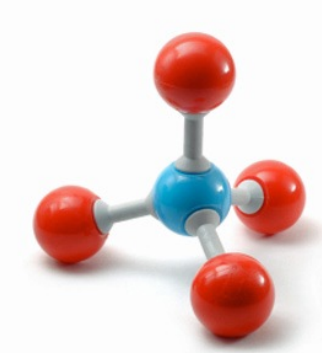


shaker



Purity and Melting Point

- Start your observations well below your expected melting point.
- Set your end rate well above your final temperature so the device doesn't start slowing down before you reach your desired temperature.
- Using a MeltTemp device use a:
  - Start Temp of 20-30 degrees below your expected melting point.
    - More for samples you know are less pure.
  - Use a ramp rate of 5 degrees for most observations.
  - End Temp of 20 degrees above your expected melting point.



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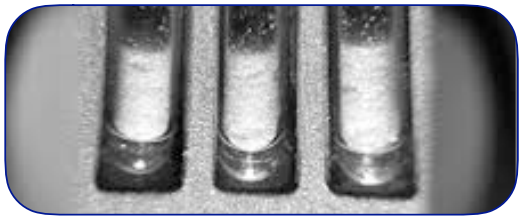

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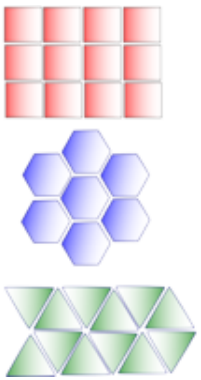
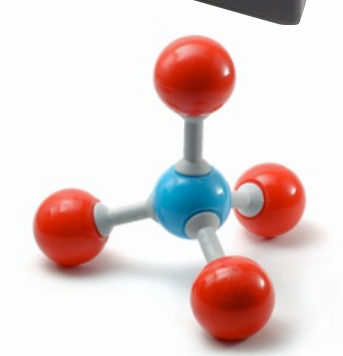
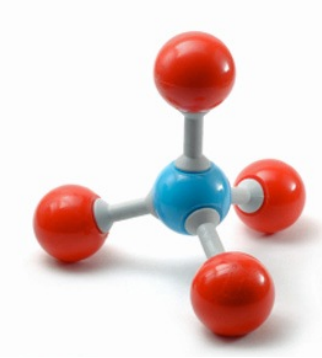




Purity and Melting Point

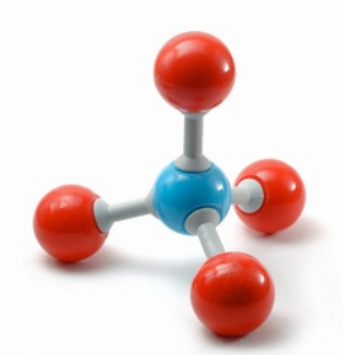
- OBJECTIVE: To process a sample of over the counter “pure” medicine and comment on it’s relative purity after each of the two separation techniques you apply.

- GOAL: To practice your extraction, crystallization and chromatography separation techniques and refine your skill at identifying melting points.



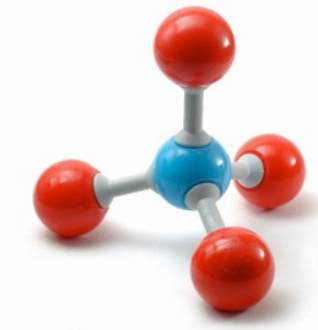
Purity and Melting Point

- Prepare Powder
  - Using pestle crush the tablet(s)
  - Separate a small portion for taking an initial melting point.
    - Call this sample A.



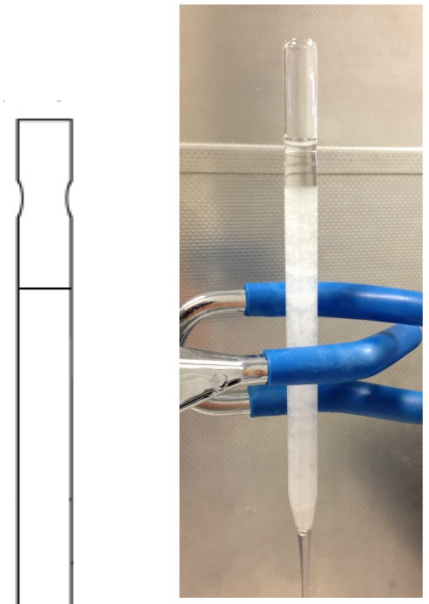
Purity and Melting Point

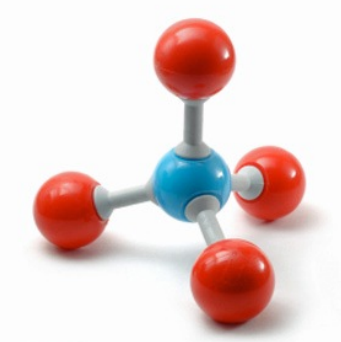
- Extraction
  - Calibrate and mark a pipette for a volume of 2 mL
  - Add the remainder of your powder to a 3 mL conical vial
  - Using the calibrated pipette
    - Add 2 mL of methanol to the vial
  - Cap and shake vigorously
  - Let settle (~ 5min)
  - With a second pipette transfer liquid to a capped centrifuge tube
  - Repeat extraction with a second 2 mL of methanol (reused your first calibrated pipette)
  - Centrifuge the extracted methanol for 3-5 minutes.



Purity and Melting Point

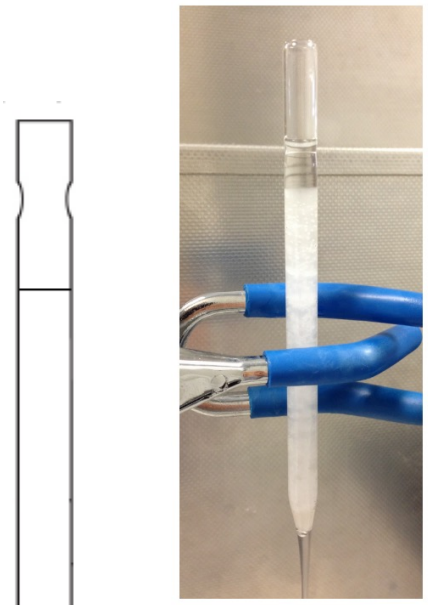
- Extraction
  - With a pipette carefully decant the methanol into a clean test tube.
  - Put about 20% of this solution into an Erlenmeyer flask and concentrate it down to a white power using a 50˚C hot water bath.
    - Label the resulting white powder **sample B**.
- Take the remaining 80% of your extract on to the next step.

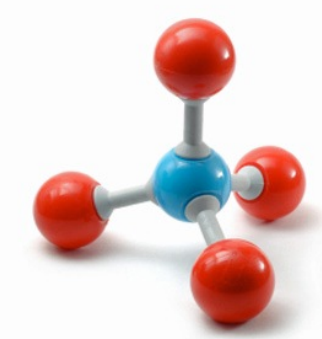




Purity and Melting Point

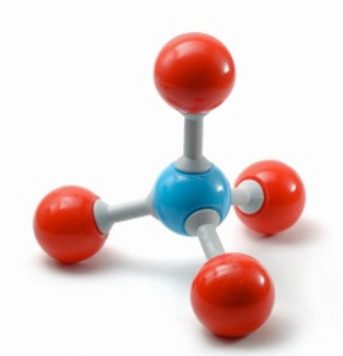
- Column Separation
  - Pack end of pipette with Cotton
  - Ad 0.50 g Alumina Gel
    - Tap column to let gel settle
    - Use folded weighing paper to add gel
  - Clamp Column to lab jack
  - Position erlenmeyer beaker under pippette
  - Charge column with methanol
    - Slowly add about 2.0 mL of methanol to column, let drain
    - Use pipette bulb if it drains too slowly
  - When methanol reaches top of silica gel
    - Add aspirin extract to top of column
    - replace Erlenmeyer flask with new (empty) flask
    - Keep top of column wet (don't let run dry)
    - When extract reaches top chase it with 1 mL more of methanol.





Purity and Melting Point

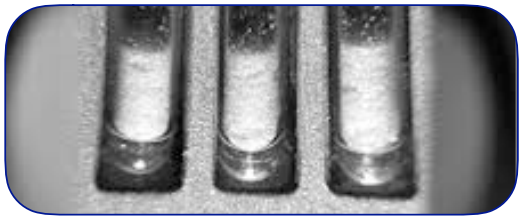
- Crystallization
  - Place half your collected methanol solution in a 5 mL conical flask.
  - Using the hot water batch concentrate it down to less than 1 mL.
  - Add the remaining methanol solution
  - Concentrate the collected methanol to less than 0.5 mL.
  - Place the vial in ice for about 10-15 minutes.
  - Collect the crystals by filtration.
    - Let crystals dry on funnel for about 5 minutes before collecting.
    - Label these crystals **sample C**.



Ex10

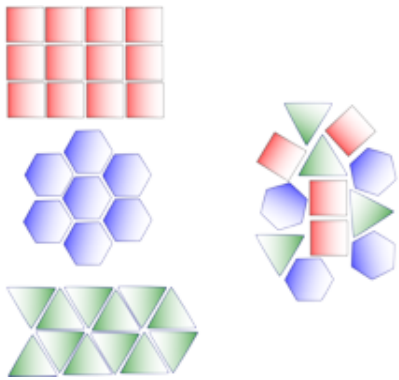
Purity and Melting Point

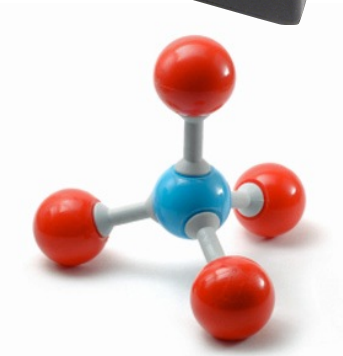
- Melting Point
  - Characterization
  - Purity & Melting Point Depression
  - Observations
  - Techniques



- The Experiment
  - A - Powder
    - sample A
  - A - Extraction
    - sample B
  - B - Chromatography
    - sample C
  - C - Collection
  - D - Analysis

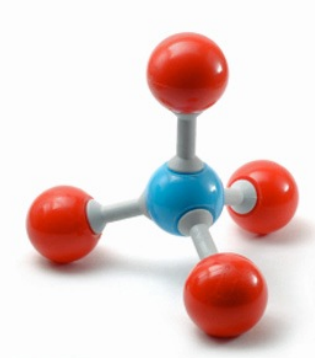
- For Next Week





Purity and Melting Point

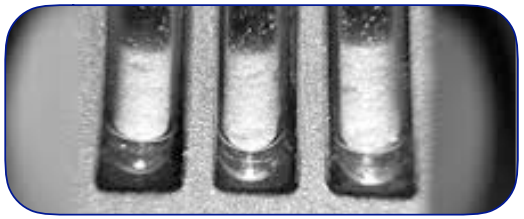
- Analysis:
  - Determine and report the melting point of…
    - Sample A (the powder)
    - Sample B (the extract)
    - Sample C (the column fraction)
  - In your report, comment on the purity of each.



Ex10

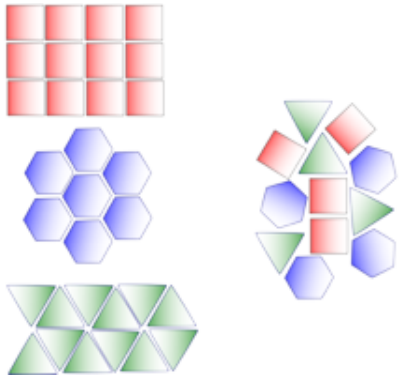
Purity and Melting Point

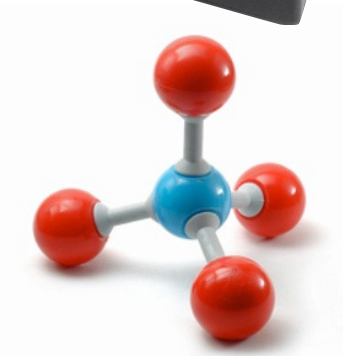
- Melting Point
  - Characterization
  - Purity & Melting Point Depression
  - Observations
  - Techniques



- The Experiment
  - A - Powder
    - sample A
  - A - Extraction
    - sample B
  - B - Chromatography
    - sample C
  - C - Collection
  - D - Analysis

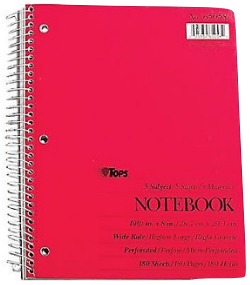
For Next Week

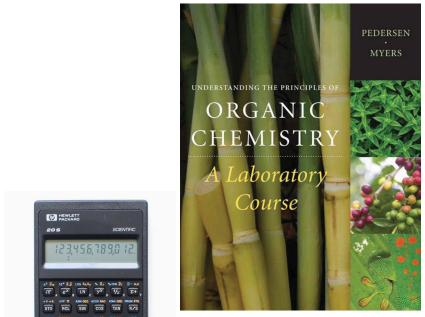


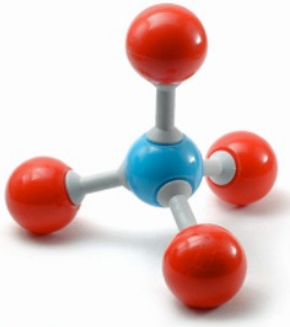


Next Meeting

- For next Meeting:
  - Bring to class:
    - Notebook
      - You will not be turning in notebooks, but this permanent record of your preparations, observations and notes will be essential to success in this class.
    - Textbook, calculator, pencils (yes, you can use pen)
    - Safety Glasses
      (you cannot participate without them)
  - Read through and take notes on:
    - Experiment 13 (p100)
    - Essay Caffeine (p96)
    - Sublimation Technique (p779)
  - Produce and bring to class:
    - Your pre-lab for exp 13 (p100)
    - Your procedure summary for exp 13







Questions?