



# General Chemistry Laboratory

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## Recrystallization and Melting Point



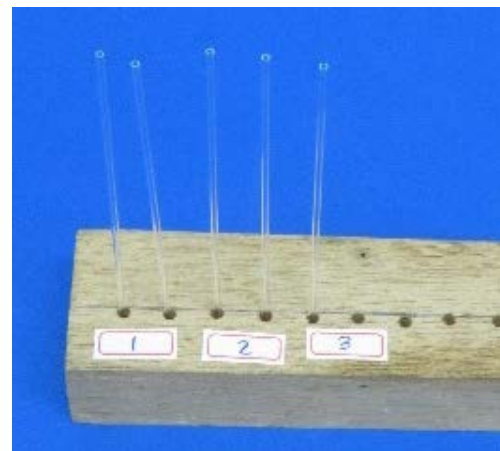
# Preparation

## Collect the following items

- Melt-Temp apparatus
- Capillary tube
- Filter paper and sticky labels
- Rubber stopper

## From your personal equipment

- Thermometer
- Two 50 mL Erlenmeyer flasks
- Capillary tube stand
- Hollow glass tube (~60 cm)
- Büchner funnel
- Suction filtering flask
- Water aspirator
- Hot plate



Capillary tube stand



# Objective and Principles

- **Objective:** Use recrystallization to purify compound and determine the melting point
- **Lab techniques**
  - Recrystallization
  - Suction filtration
  - Melting point (mp) determination
  - Electronic balance
  - Magnetic stirrer and hot plate
- **Tasks**
  - Determine the mp of pure compounds
  - Recrystallize the benzoic acid
  - Determine the mp of crude and purified compound



# Principle - Melting Point

## ■ Melting point

- The temperature at which substance changes from solid to liquid at atmospheric pressure
- When a pure crystalline substance melts, the melting point range should not exceed  $1^{\circ}\text{C}$ . An impure substance shows a larger melting point range than a pure substance
- The melting point can be used to
  - ✓ determine the identity of a known compound
  - ✓ define an unknown compound at later times
  - ✓ determine the purity of a substance



# Principle - Recrystallization

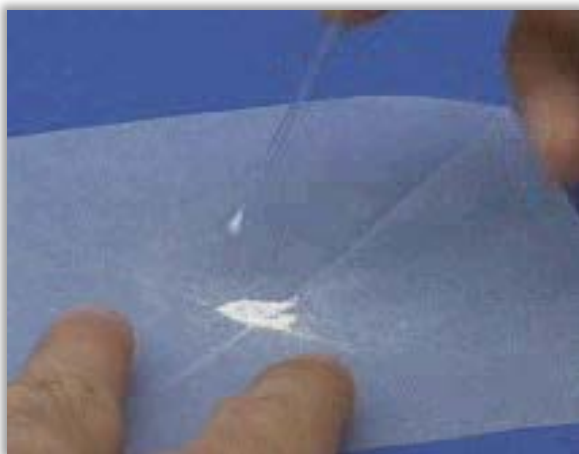
## ■ Recrystallization

- Dissolve the sample in a suitable hot solvent. After the solution is cooled, a supersaturated solution will be produced, and the solute will crystallize out
- During recrystallization, the sample adheres to the lattice in an orderly manner, without any impurities included that increases the purity
- Choose suitable solvent
  - has high solubility for the sample at high temperature, but low solubility at low temperature
  - has high solubility for the impurity; hence, it will not be crystallized out
  - Non-flammable, non-toxic, cheap, and volatile...



# Step 1: Packing Capillary Tube

- Pack 2 capillary tubes for each sample:
  - (1) benzoic acid (122°C)
  - (2) acetanilide (113°C)
  - (3) benzoic acid/acetanilide mixture (1:1)



Sealed end down and open end up

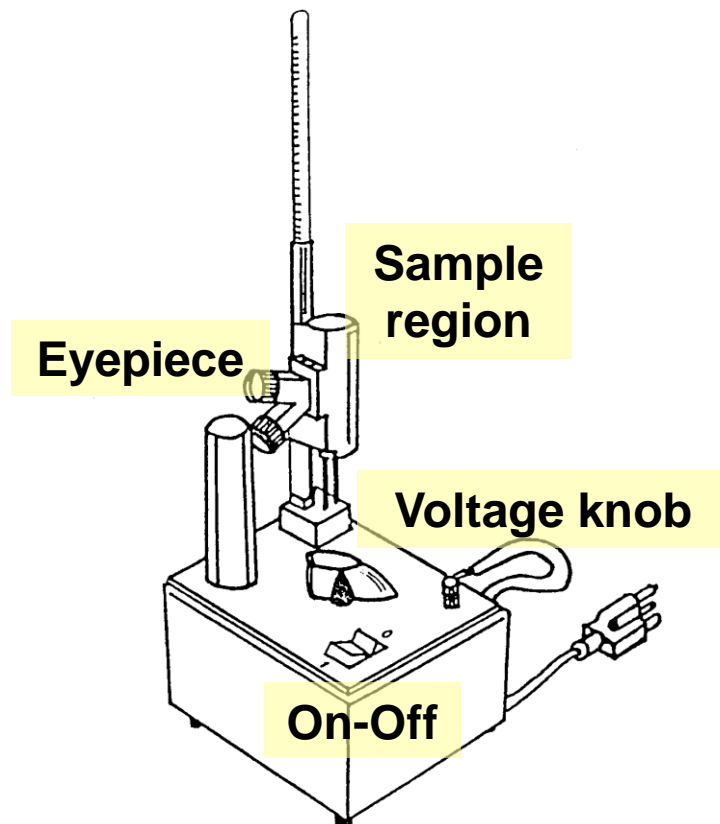
- Transfer a dry and finely powdered sample onto a piece of weighing paper
- Insert the open-end of a capillary tube into the pile of the sample
- Knock the closed-end of the capillary tube on the bench top 3-4 times
- The packed sample is ca. 2 mm height



# Step 2: Determine the Melting Point Range

- Set the voltage control knob to zero, then turn on the power
- To pre-measure a rough melting point range for three samples, apply a faster heating rate of about  $5^{\circ}\text{C}/\text{min}$
- Cool the Melt-Temp apparatus to lower than the approximate mp about  $15^{\circ}\text{C}$
- Replace the capillary tubes and slowly heat the samples at a rate of  $2^{\circ}\text{C}/\text{min}$  to determine the mp
- Record the mp range of the sample, i.e. the temperature range from starting to melt to completely melted

✓ Line up the samples from the one with lowest mp and end with the highest one



## Heating rate:

Initial:  $10\sim 15^{\circ}\text{C}/\text{min}$

Lower than mp  $10\sim 20^{\circ}\text{C}$ :  $2^{\circ}\text{C}/\text{min}$

Lower than mp  $2\sim 4^{\circ}\text{C}$ :  $1^{\circ}\text{C}/\text{min}$



## Step 3: Recrystallization



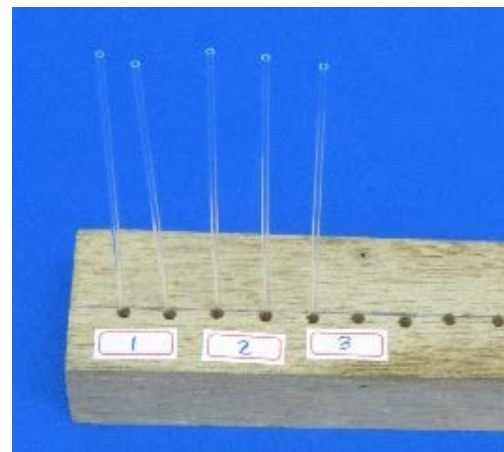
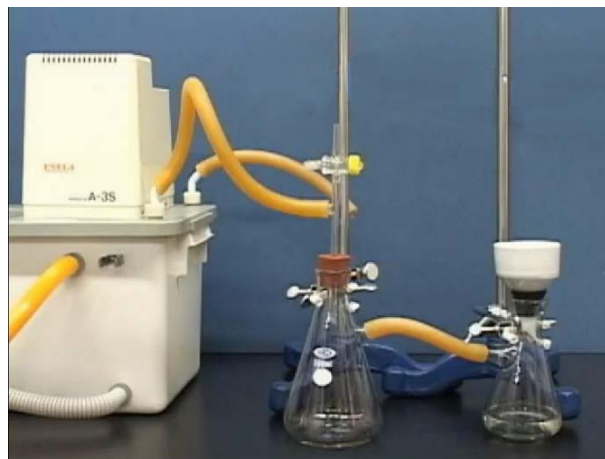
- Use a 50 mL Erlenmeyer flask to heat and boil some DI water
- Prepare 2 capillary tubes of crude benzoic acid for mp determination
- Transfer the remaining sample to a 50 mL Erlenmeyer flask after weighing
- Use dropper to add some hot DI water to the flask
- Heat and swirl the solution gently to dissolve sample
- Cool it down slowly to room temp., and let it stand to grow crystals

- ✓ Calculate the amount of hot water needed base on solubility
- ✓ As water is the solvent, heat the solution on hot plate directly instead of water bath





# Step 4: Collect Product and Determine the Melting Point



- Use an ice water bath to cool the mixture to a lower temperature and produce more crystals
- Use suction filtration to collect crystals
- Wash the crystals with small portion of cold-water
- Suction dry for 10 min.
- Collect the crystals on filter paper and press to dry
- Weigh the dried crystals and determine the yield
- Determine mp of (1) crude benzoic acid, (2) recrystallized benzoic acid, (3) crude acetanilide



# Setup of Vacuum Filtration

1. Fill the tank with water, maintain a slow overflow rate; turn on the power

5. Close 2-way valve to achieve vacuum

3. Select 55 mm filter paper  
4. Rinse with some DI water

6. **Test suction**  
7. Pour soln into Büchner funnel

2. Use rubber stopper to keep air tightness

Water out

Water in

Aspirator pump

Safety trap

Suction Flask (fixed)



# Additional Notice

## Melting point determination

- The sample inside the capillary tube should be 2 mm in height
- Glass tube for knocking the capillary tube should be kept clean and dry; wash and oven dry after class
- Record the melting point range of sample
- Replace the capillary tubes in the second run measurement

## Recrystallization

- Use an Erlenmeyer flask for recrystallization
- **Do not use flame or hot plate to directly heat flammable organic solvents. Use water bath instead**
- Leave the solution to cool to room temperature, and let it stand to grow crystals slowly that increases the purity



# Clean-Up and Check-Out

- Dispose benzoic acid, acetanilide, and capillary tubes to designated waste bins
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- This is a **Brief Report** experiment:
  - Hand in prelab/lab note/report together to the TA
- Groups on duty shall stay and help clean up the lab

