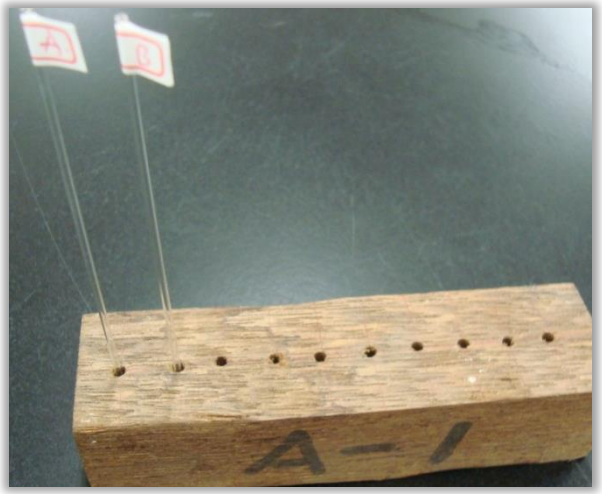
C26 Recrystallization and Melting Point

(2017/04/24)

Collect:

- Melt-Temp device
- Capillary tube
- Filter paper



Prepare:

- Thermometer
- Capillary tube stand
- Hollow glass tube
- Hot plate
- Erlenmeyer flask (50 mL, 2)
- Hirsch funnel
- Filtering tube
- Rubber stopper
- Water aspirator

C26 Recrystallization and Melting Point

- **Objective:**

  To learn the techniques of recrystallization and to determine the melting point

- **Technique:**
  - Electric balance, magnetic stirrer and hot plate
  - Recrystallization
  - Suction filtration
  - Packing capillary tube and m.p. determination

- **Flowchart:**

  Part I: m.p. determination of pure substances

  Part II: recrystallization of benzoic acid

  Part II: m.p. determination of crude and purified compd.

Principle

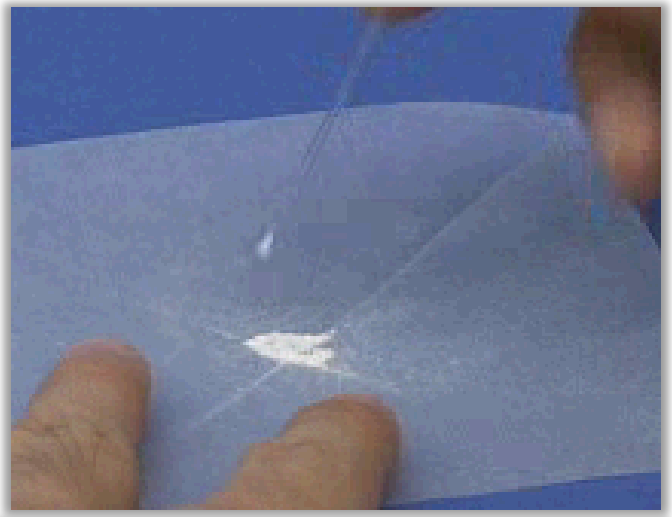
- **Melting point**
- The temperature at which it changes from solid to liquid at atmospheric pressure
- When a pure crystalline substance melts, the melting point range should not exceed 1 ^{o}C. An impure substance shows a larger melting point range than a pure substance
- The melting point can be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

Principle

- **Recrystallization**
- Recrystallization consists of dissolving the solid substance in a minimal amount of a suitable hot solvent. Cooling the solution causes the formation of a supersaturated solution, and the pure solid will be crystallized out.
- Ideally, the concentration of the impurity will not reach the saturation level; hence, it will not be crystallized out.
- During recrystallization, the sample adheres to the lattice in an orderly manner, without any impurities included that increases the purity.

Part I Melting Point – Packing Capillary Tube

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



- Transfer a dry and finely powdered sample onto a piece of weighing paper
- Insert the open-end of a capillary tube into the stack of the sample.

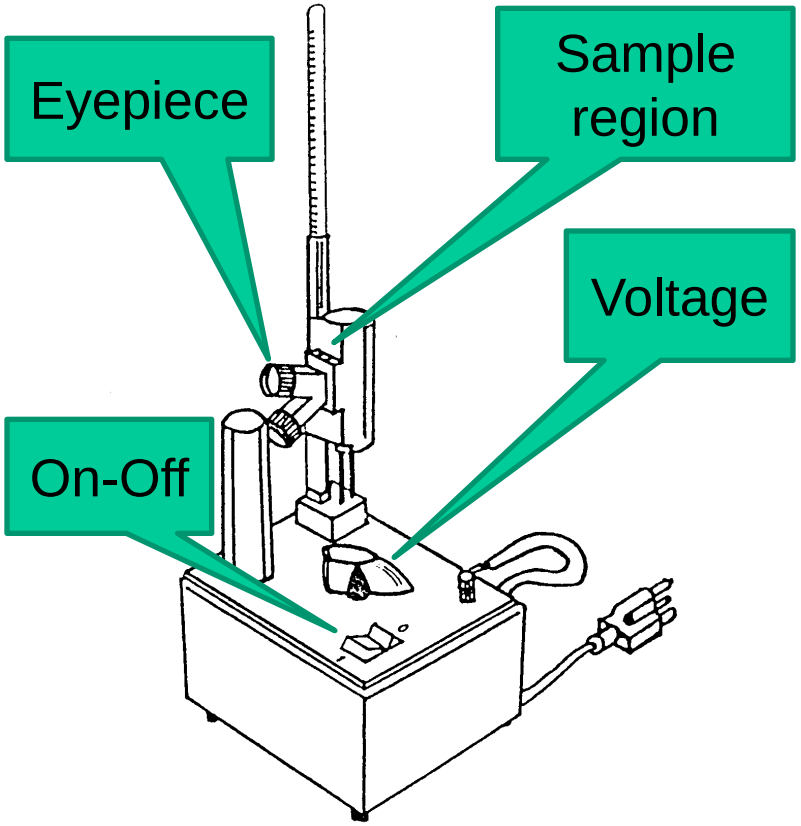
- Knock the closed-end of the capillary tube on the bench top 3-4 times

Keep capillary tube open-end up

- The sample is ca. 2-3 mm height

Part I Melting Point – Determine the Melting Range

- Adjust the voltage to zero, then turn on the power
- Carry out a crude melting point measurement for 3 samples in a faster heating
* **Start with the lowest m.p. and end with the one with the highest m.p.**
- Cool the Melt-Temp device to lower than the crude m.p. about 15 ^{o}C
- Replace the capillary tubes and slowly heat the samples at a rate of 2 ^{o}C/min. to determine the m.p.
- Record the m.p. range of the sample, i.e. the temperature range from starting to melt to completely melted



Heating rate
Initial: 10~15 ^{o}C/min
Lower than m.p. 10~20 ^{o}C: 2 ^{o}C/min
Lower than m.p. 2~4^{o}C: 1 ^{o}C/min

Part II Recrystallization – Dissolution

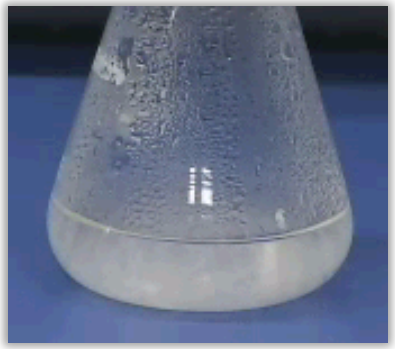
- Prepare 2 capillary tubes of crude benzoic acid sample

- Transfer the remaining sample to a 50 mL Erlenmeyer flask after weighing
- Add appropriate amount of hot water to the flask

* **Calculate the amount of hot water needed base on solubility**

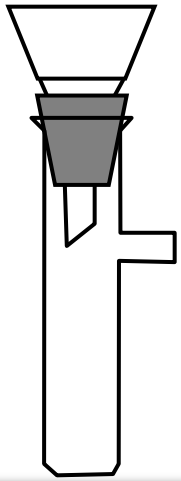
- Heat and swirl the solution gently to dissolve

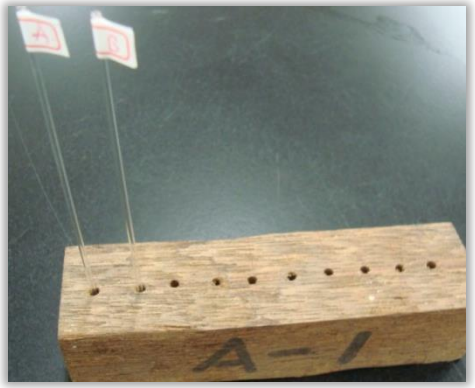
* **As water is the solvent, heat the solution on hot plate directly instead of water bath**



- Cool soln slowly at room temp for 15 ~ 20 min. to grow crystals

Part II Recrystallization – Crystallization





- Cool the flask in an ice-water bath to decrease the temp. and increase the yield

- Suction filtration to collect crystals
- Wash the crystals with small amount 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 m.p.

(1) crude benzoic acid

(2) recrystallized benzoic acid

(3) crude acetanilide

Manipulation of Suction Filtration

- Fill water in water pump that come in from bottom and flow out from top

- Fix water-trap bottle and filtering tube with extension clamp
- Place a rubber stopper between filtering tube and Hirsch funnel
- Cut the filter paper to cover all of the holes in the bottom of the Hirsch funnel but it must not extend up the sides
- Moisten the filter paper with small portions of solvent
- Close the two-way valve, then start and test the suction
- Start filtering when the filter paper is tightly stuck on the funnel
- Release the pressure before turn off the water aspirator

Notice

- **Melting point determination**
  - The sample inside the capillary tube should be 2~3 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 range of sample
  - Replace the capillary tubes in the second run
- **Recrystallization**
  - Use the Erlenmeyer flask to dissolve the sample
  - **Use water bath to heat the organic solvents that are flammable**
  - Use the least amount of hot solvent to dissolve the sample to increase the yield
  - Cool the solution slowly at room temp. to grow crystals that increases the purity
- **Recycle:** the benzoic acid, capillary tubes to designated waste bins