Recrystallization & Melting Point Determination

Last revised: 2025/02/11

Preparation

Collect the following items

- Melt-Temp (analog) or MP-2D (digital)
- Nine capillary tubes
- Hollow glass tube
- Büchner funnel
- Filter paper
- Your benzoic acid and acetanilide products

From your personal equipment

- Capillary tube stand
- Hot plate
- Two 50 mL Erlenmeyer flasks
- Suction flask
- Rubber adapter cone
- Water aspirator

Capillary tube stand

Objective and Principles

- **Objective:** Use recrystallization to purify compound and determine the melting point (mp)
- **Lab techniques**
  - Recrystallization
  - Suction filtration
  - Packing capillary tubes
  - Measuring melting points of organic compounds
- **Tasks**
  1. Recrystallization of the crude benzoic acid
  2. Measuring the mp of reference compounds
  3. Measuring the mp of crude and purified compounds

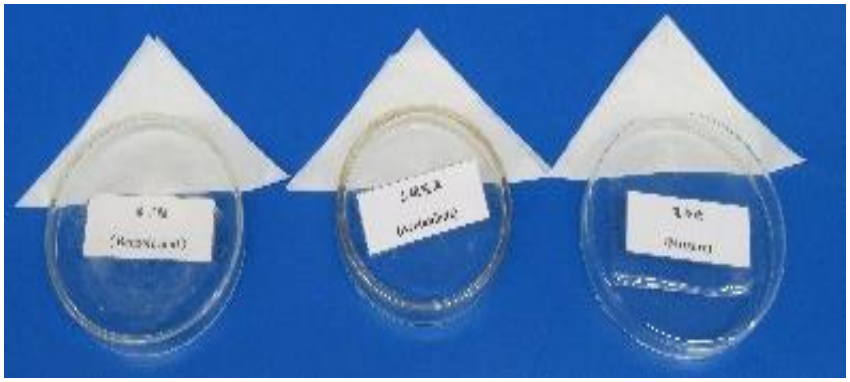
Principle

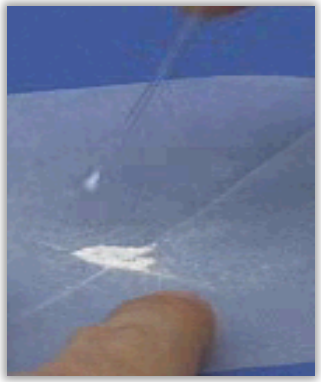
- **Melting point**
  - The temperature at which substance changes from solid to liquid at atmospheric pressure
  - When a pure crystalline substance melts, the melting point range should not exceed 1°C. An impure substance shows a larger melting point range than a pure substance
  - The melting point 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**
  - 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 (mp 122°C)
  (2) acetanilide (mp 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

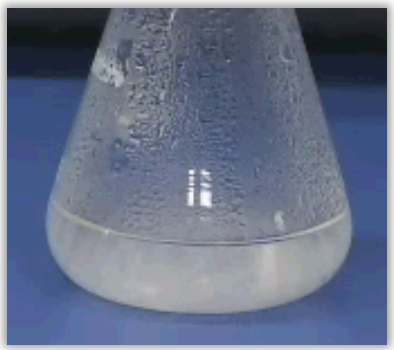
Step 2: Determine the Melting Point Range

Heating rate:
Initial: 10~15°C/min
Lower than mp 10~20°C: 2°C/min
Lower than mp 2~4°C: 1°C/min

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

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

Step 3: Recrystallization

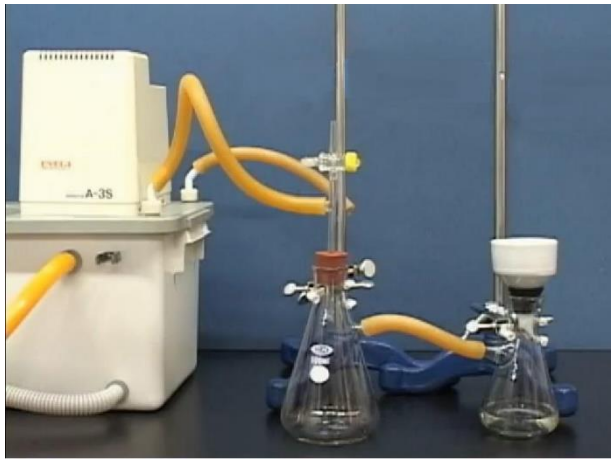


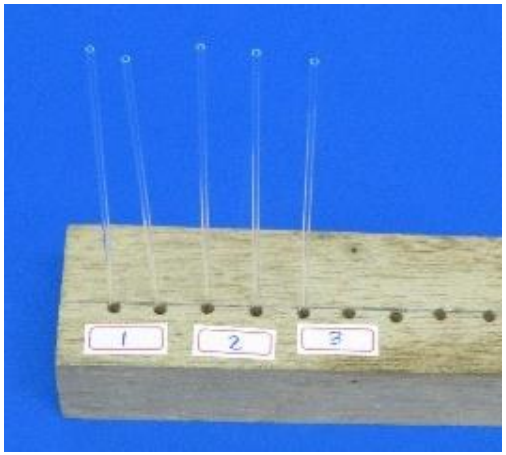
- Use a 50 mL Erlenmeyer flask to heat and boil some DI water
- Prepare 2 capillary tubes of crude benzoic acid for mp determination
- Transfer the remaining sample to a 50 mL Erlenmeyer flask after weighing
- Use dropper to add some hot DI water to the flask
- Heat and swirl the solution gently to dissolve 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

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 of 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