Extraction

Last revised: 2023/06/06

Preparation

Collect the following items

- Filter paper, sticky labels
- Rubber stopper

From your personal equipment

- Separatory funnel
- Iron ring and support
- Filtering funnel
- Two 50 mL Erlenmeyer flasks
- One 100 mL round bottom flask
- Büchner funnel
- Suction filtering flask
- One large and one small test tube
- Water aspirator

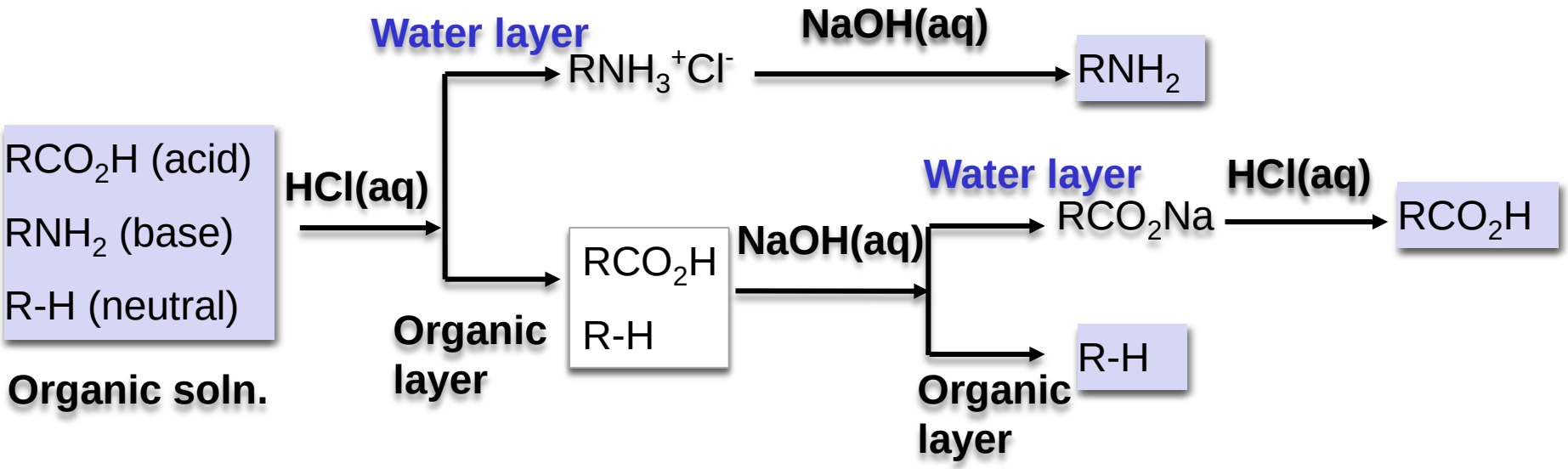
Objective and Principles

- **Objective:** use acid-base reaction to extract and separate the organic compounds
- **Lab techniques**
  - Extraction
  - Suction filtration
  - Gravity filtration
  - Rotary evaporator
- **Flowcharts**
  - Part I: Extraction by acid-base reaction
  - Part II: Separation of benzoic acid
  - Part III: Separation of acetanilide

Principle

- **Extraction**
  Use the solubility differences of substances in different solvents to transfer a specific component in the mixture to another solvent to achieve the purpose of separation

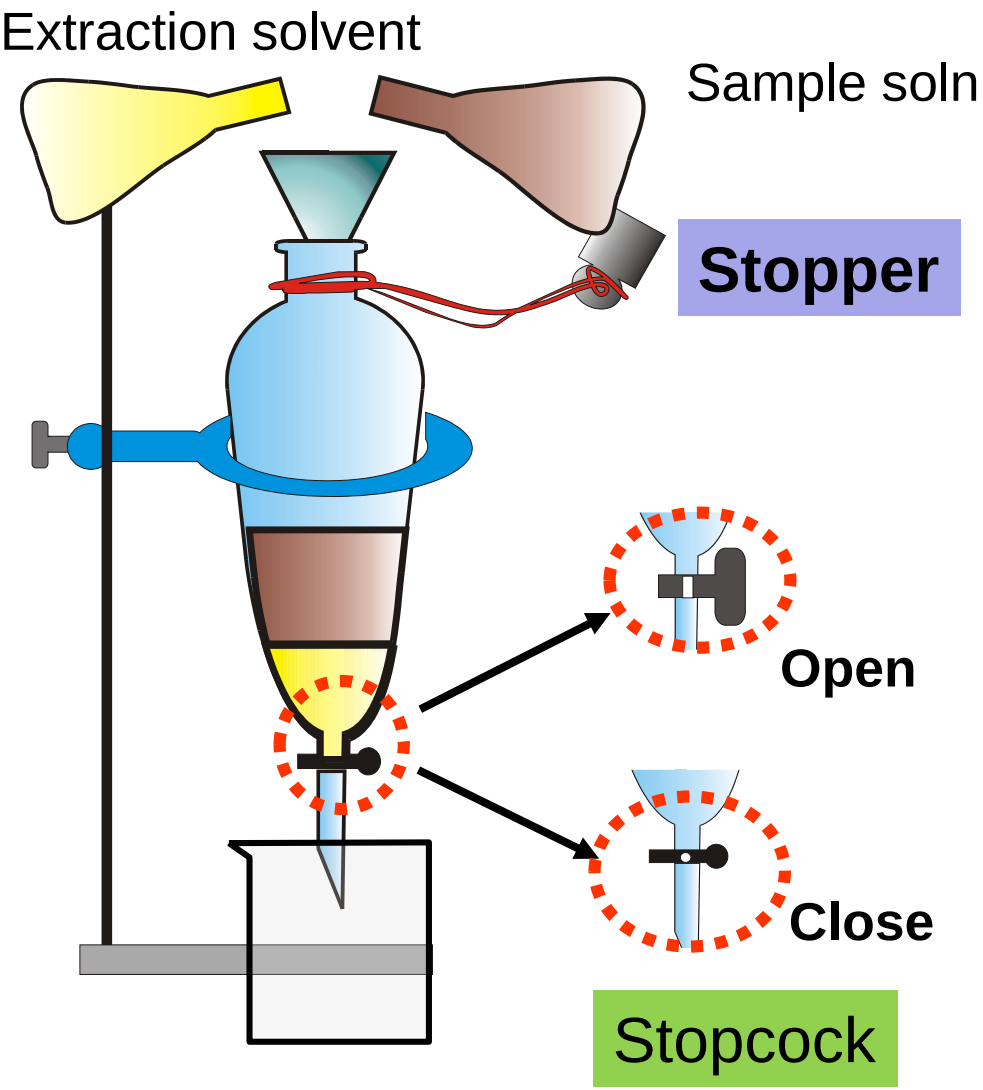
- **Extraction by acid-base reaction**



Preparation

Separatory funnel

- Check the stopper and stopcock to avoid leaking
- Support on an iron ring
- Place an empty beaker below the separatory funnel to collect liquid of accidental leakage
- Close the stopcock at the bottom
- Add sample soln through filtration funnel
- Rinse the sample flask twice
- Pour the extraction solvent, close with the stopper, and identify the layers



Shaking and Venting

- Hold the stopper securely in place with one hand, then invert the funnel
- Open the stopcock slowly with the other hand to relieve the pressure
- Close the stopcock and shake the funnel vigorously to mix two solvents
- To return the pressure in the funnel to normal, open the stopcock from time to time to release the vapor. Vent about once every five shakes
- Repeat venting and shaking until the “whoosh” is no longer audible

✓ Do not point the opening to the others

✓ Repeat venting as many times as possible

Separation

Separate two layers

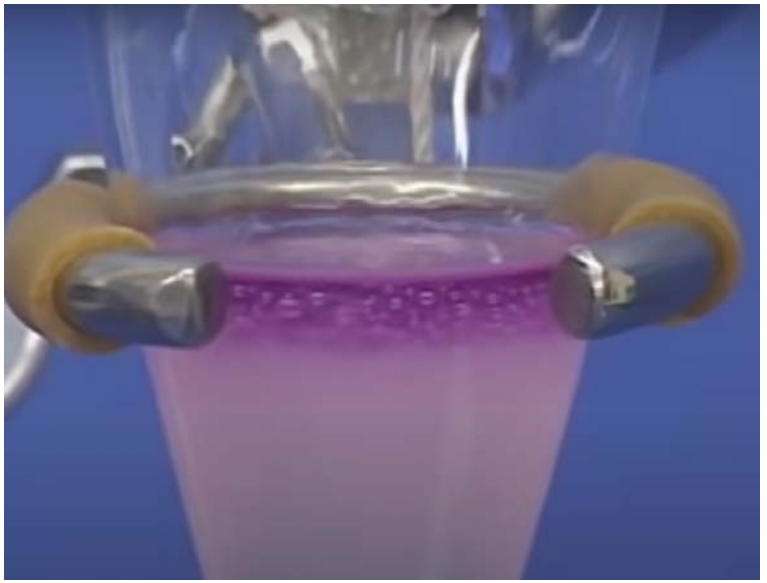
Drain the lower layer

Pour upper layer from top

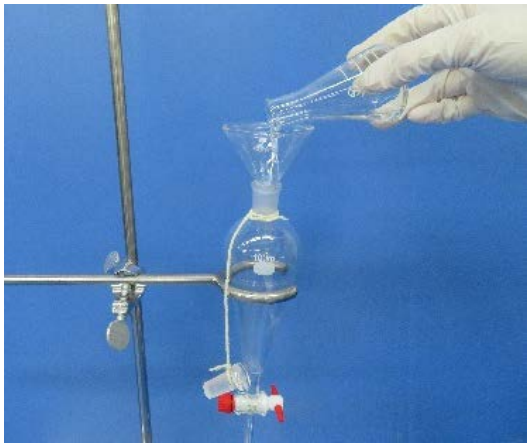
- Replace the funnel on iron ring, remove the stopper immediately
- Allow the two layers of liquid to separate
- Drain the lower layer through the stopcock
- Close the stopcock until the interface between the upper and lower phases just begins to enter the bore of the stopcock
- The remaining upper layer is removed by pouring it from the top opening

Emulsions

- **Emulsion**
  - Emulsion is a colloidal suspension of one liquid in another liquid
  - Minute droplets of an organic solvent often are held in suspension in an aqueous solution when the two are mixed vigorously
- **Break up emulsion**
  - Gently swirling
  - Allow the emulsion to stand for a time
  - Add a saturated NaCl(aq)



Step 1: Extract Benzoic acid by Acid-Base Reaction

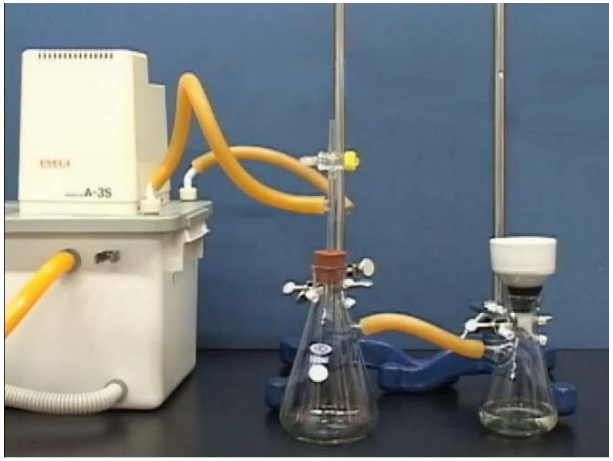


- Weigh 0.5 g benzoic acid and acetanilide
- Transfer to a 50 mL Erlenmeyer flask
- Add 10 mL EA to dissolve solid
- Pour the soln to separatory funnel
- Rinse the flask with 2 mL EA twice

- Use 5 mL 5%NaOH to extract benzoic acid
- Drain the lower NaOH aqueous layer to flask A
- Add 5 mL DI water to separatory funnel and extract again
- Combine the lower layer to flask A
- Pour the upper layer from top to another flask B

- Place flask A in ice-water bath
- Add 6 M HCl drop by drop till no more white ppt forms and pH < 3

Step 2: Separation of Benzoic Acid



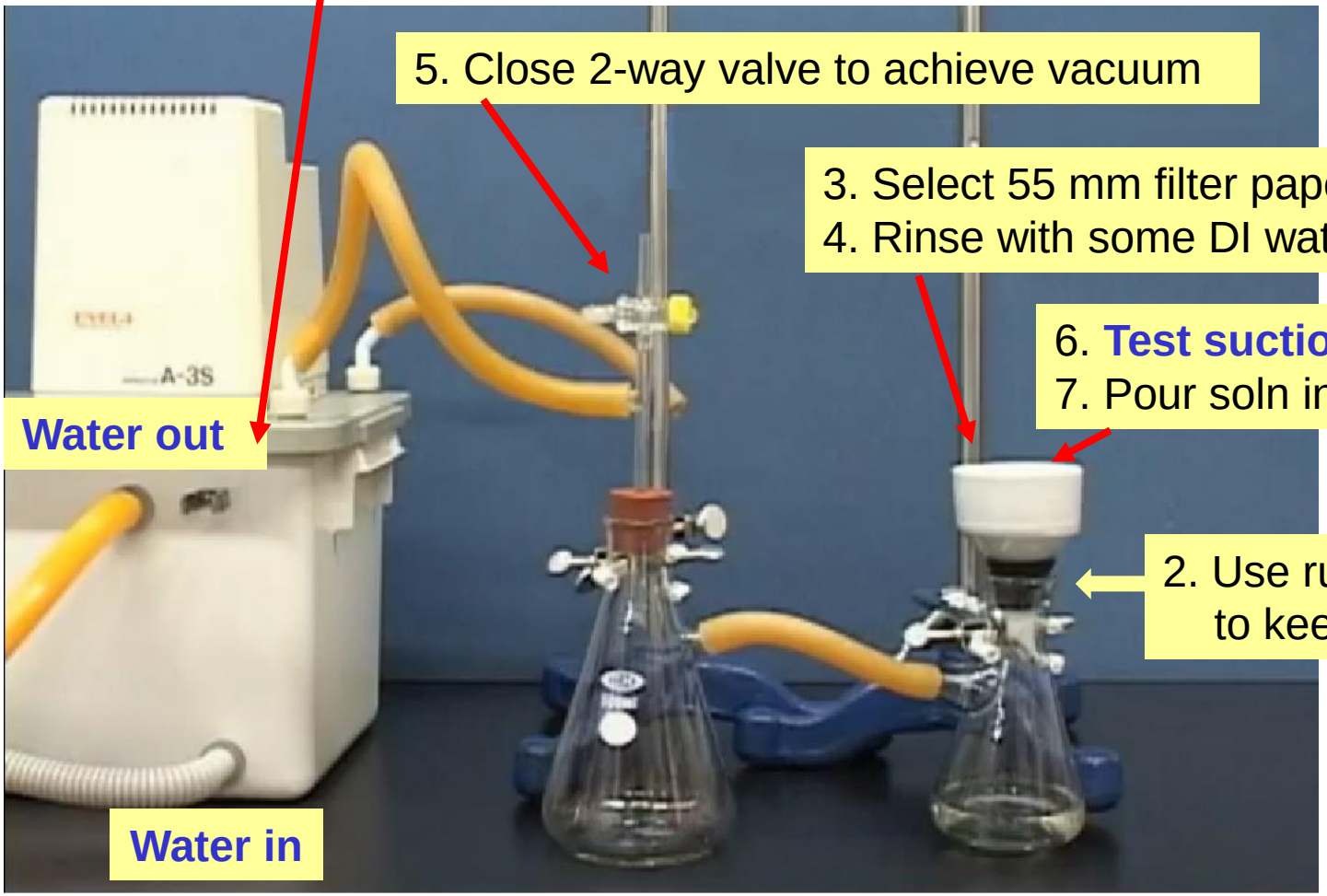
- Collect benzoic acid by suction filtration
- Suction dry for 10 min
- Collect product on a filter paper
- Press dry with filter paper; then air dry for 10 min

- Label and weigh the mass of the empty large test tube
- Transfer benzoic acid into the tube
- Weigh it again to calculate the percent yield

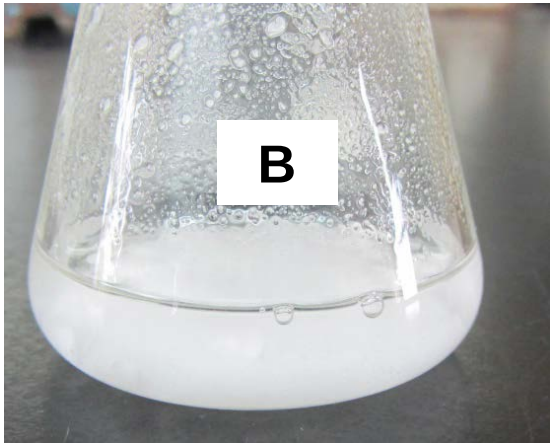
- Use a piece of aluminum foil to seal the tube
- Poke small holes to allow further drying
- Keep for the next experiment

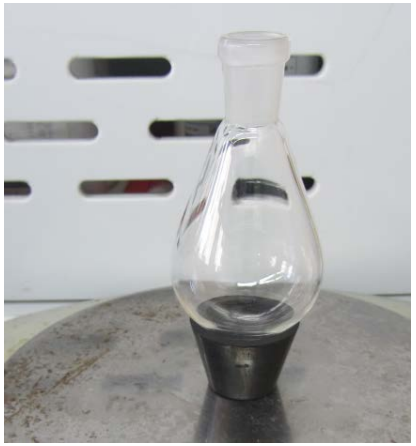
Setup of Vacuum Filtration

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



Step 3: Separation of Acetanilide





- Add proper amount of $MgSO_4$ (ca. 2 g) to flask B
- Swirl the flask while adding until $MgSO_4$ can move freely and does not stick to the bottom

- Filter the soln in flask B to remove $MgSO_4$ by **gravity filtration** to a weighed round bottom flask
- Rinse the flask B with ca. 2 mL EA, then filter and combine the filtrate into round bottom flask

- Evaporate EA and obtain acetanilide in round bottom flask by rotary evaporator
- Weigh and calculate the percent yield
- Transfer the product to a weighed small test tube
- Weigh the tube with product again and keep it for next experiment

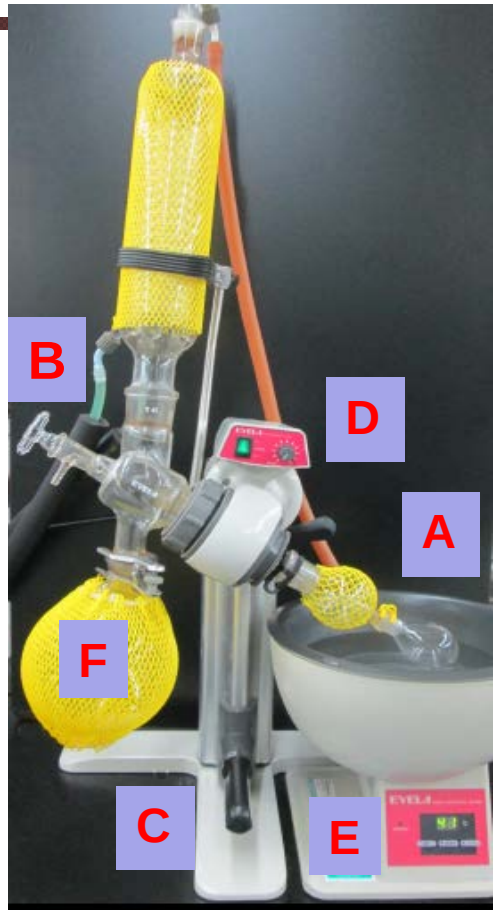
Rotary Evaporator (Rotavap)

Begin the operation

1. Turn on circular cooling system
2. Turn on and set the water bath to suitable temp. **(E)**
3. Place the flask on the rotavap **(A)** and secure the flask with a Keck clip
4. Balance the system with atmosphere **(B)**
5. Turn on the vacuum system
6. Lower the flask into the water bath **(C)**
7. Turn on the rotation to appropriate speed **(D)**
8. Turn the stopcock at the top of the condenser **(B)** to adjust the vacuum and avoid bumping
9. Start collecting solvent on the condenser and drip into the receiving flask **(F)**

End of the operation

1. Release vacuum **(B)** and discontinue spinning **(D)**
2. Raise the flask out of the water bath **(C)** and disconnect the flask **(A)**
3. Turn off water bath, water aspirator, and circular cooling system



Keck clip

Additional Notice

- Wear NBR gloves to keep from touching chemicals
- Place the separatory funnel on iron ring to avoid breaking it
- Carry out the extraction in fume hood when using organic solvent
- Organic solvents are usually flammable, so be careful not to have any heat source nearby
- Correctly identify the two layers
- Remove water out of the organic extract with a drying agent
- Handle the rotary evaporator with care. If there is any question, ask for help
- Save both layers until the end of the experiment to avoid accidentally discarding the desired one
- Keep the products to determine the m.p. next week

Clean-Up and Check-Out

- Recycle the organic waste 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