



General Chemistry Laboratory

Extraction



Preparation

Collect the following items

- ☐ Filter paper (55-mm and 90-mm)
- ☐ Büchner funnel

From your personal equipment

- ☐ Set up an iron ring on an iron stand (or the stainless steel frame inside the fume hood)
- ☐ Place a 100 mL separatory funnel on the iron ring, and use water to check if the stopcock is leaking
- ☐ Set up a suction filtration system (Büchner funnel + suction flask)
- ☐ One 100 mL round bottom flask
- ☐ Two 50 mL Erlenmeyer flasks





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

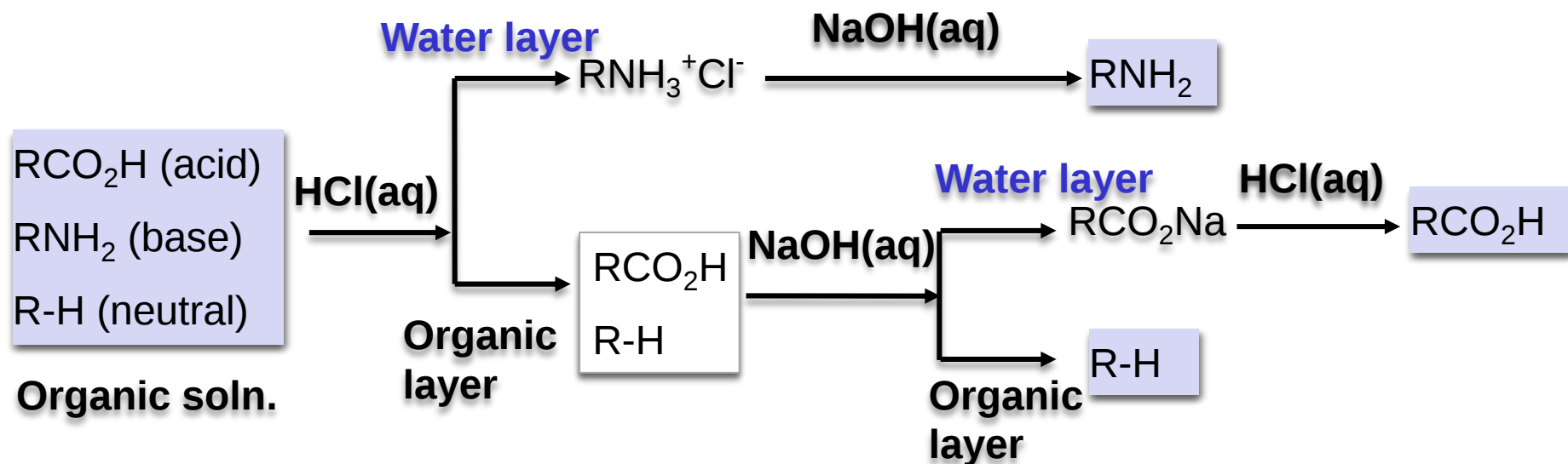


Rotary evaporator



Principle

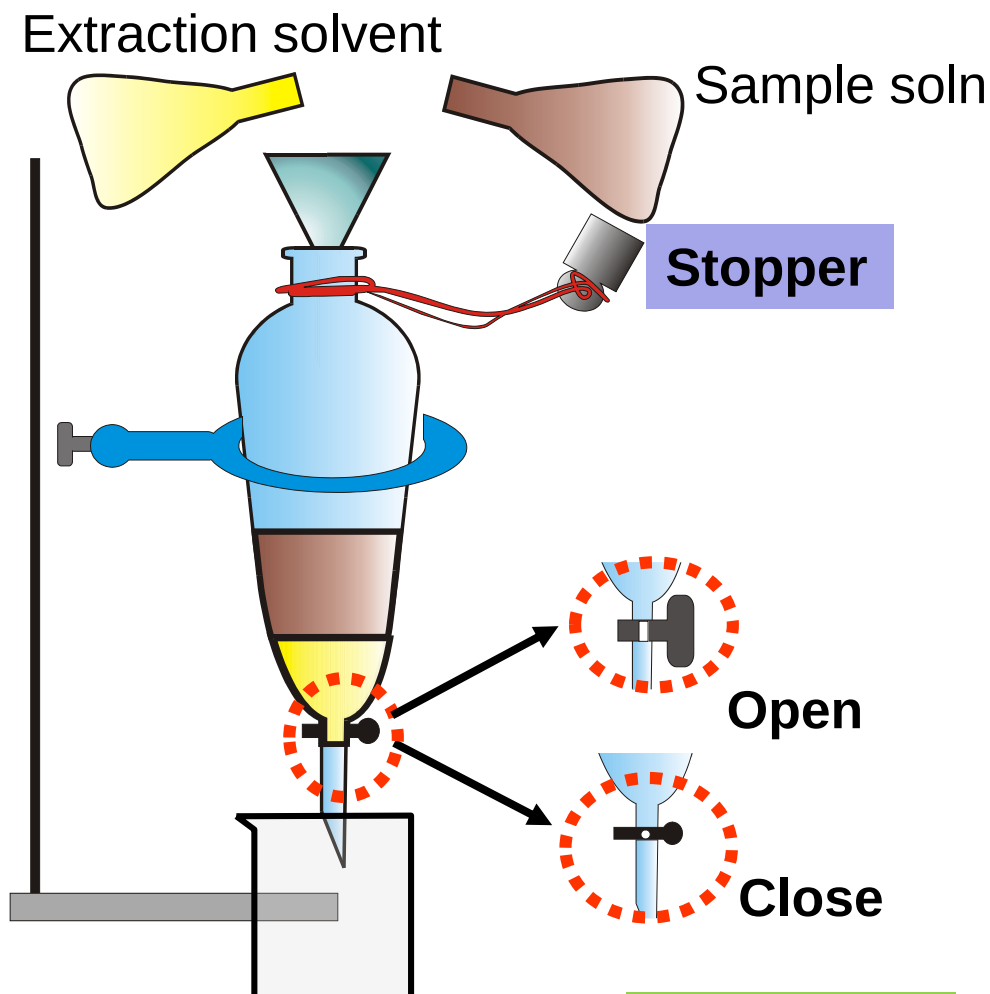
- **Extraction:** Transfer of a solute from one solvent to another by means of the distribution process
- **Extraction by acid-base reaction**





Operating a Separatory Funnel (1/3)

- Install the separatory funnel on a firmly fixed iron ring and close the stopcock
- Add in solution through a transfer funnel
- Use the same solvent to rinse the original container twice, then combine the wash solution into the separatory funnel
- Add the extractant, close the ground glass opening with a stopper



✓ Wear NBR/latex gloves

✓ The extraction process must be operated in a fume hood

Stopcock



Operating a Separatory Funnel (2/3)

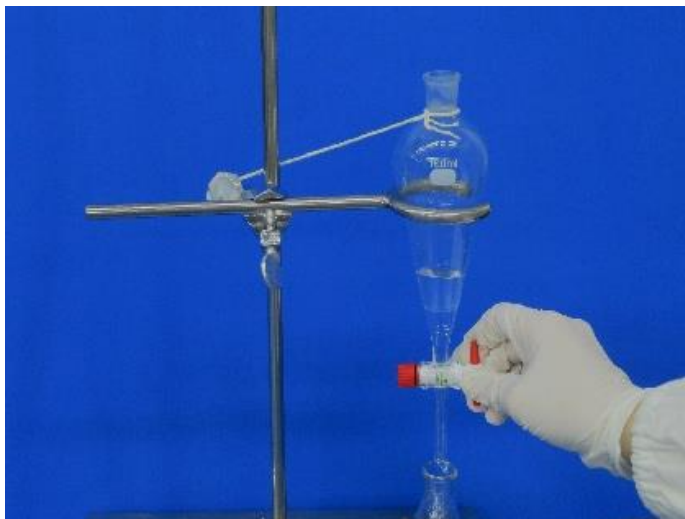


- ✓ Check the stopper and stopcock area for any sign of leaking
- ✓ Avoid pointing the opening of the separatory funnel toward the others

- Use one palm to hold the stopper securely in place while the other hand holds the stopcock, then invert the funnel
- Open the stopcock slowly to release the built-up vapor pressure
- Close the stopcock and shake the separatory funnel vigorously for 10-15 s to mix the two solvents
- Open the stopcock again to vent the pressure
- Repeat the shaking and venting steps until the “whoosh” sound becomes almost inaudible



Operating a Separatory Funnel (3/3)



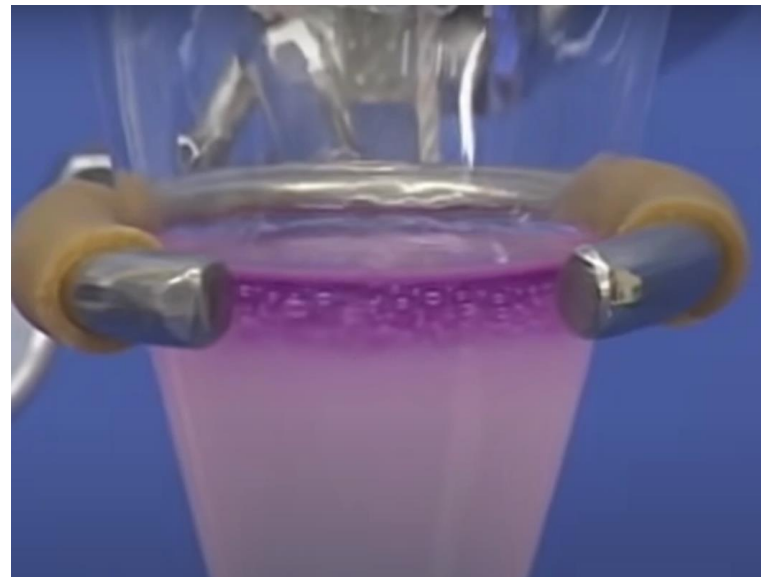
- Place the separatory funnel back on the iron ring, and remove the stopper immediately
- Allow the two layers of liquid to separate; a clear phase boundary should appear
- Slowly drain the **lower layer** through the stopcock until the phase boundary approaches the bore of the stopcock
- The remaining **upper layer** is transferred out via 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: Extraction 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



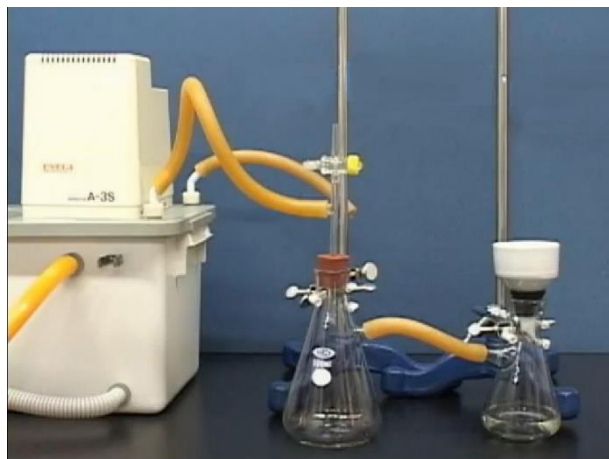
- Use 5 mL 5%NaOH to extract benzoic acid
- Drain the lower NaOH aqueous layer to flask A
- Add 5 mL DI water to separatory funnel and extract again
- Combine the lower layer to flask A
- Pour the upper layer from top to another flask B



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



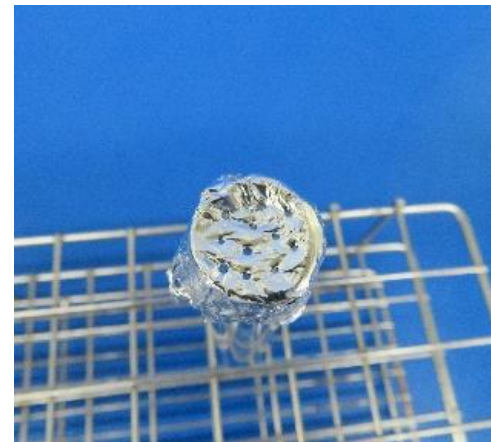
Step 2: Separation of Benzoic Acid



- 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

5. Close 2-way valve to achieve vacuum

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

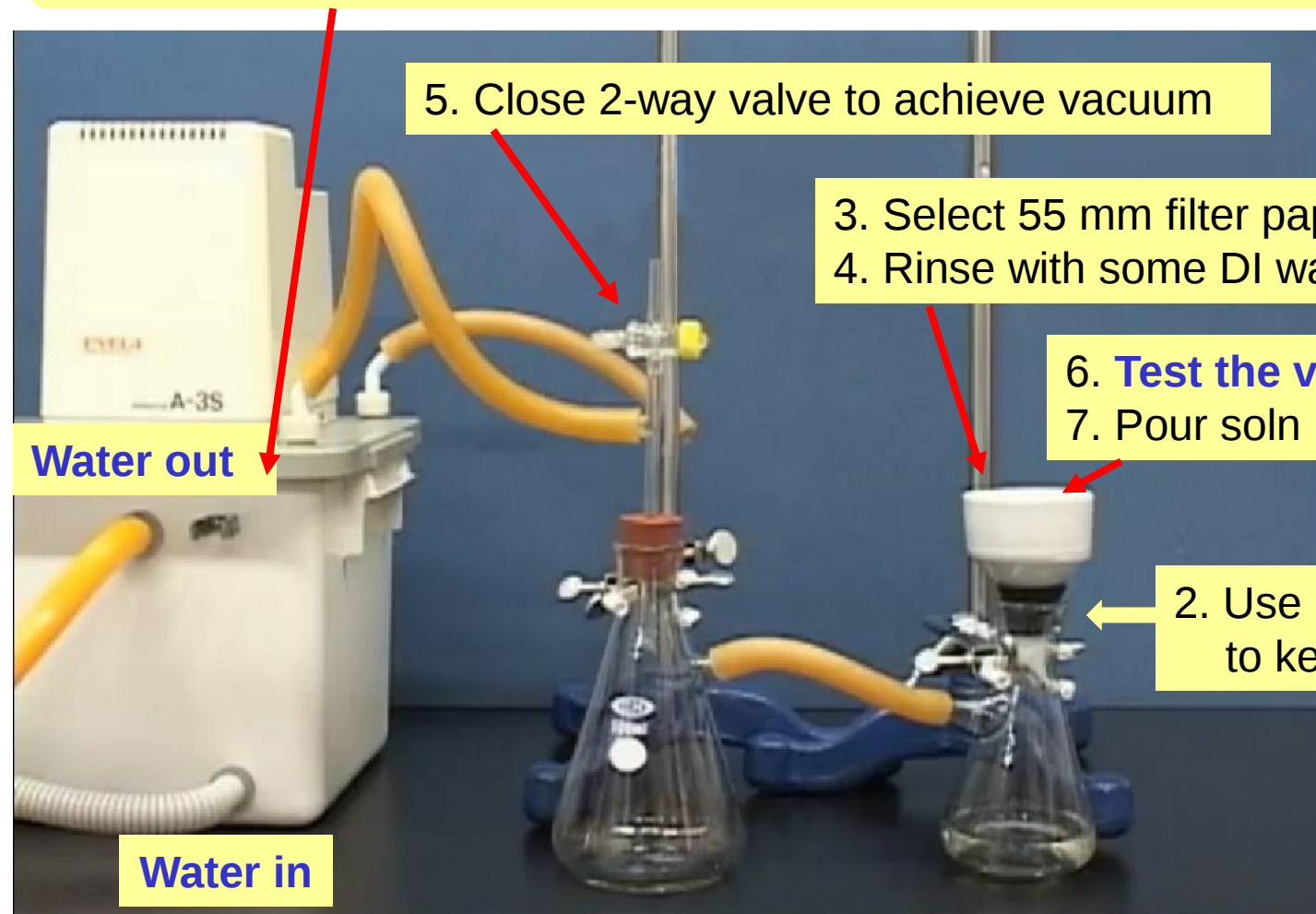
6. **Test the vacuum**

7. Pour soln into Büchner funnel

2. Use rubber stopper to keep air tightness

Water out

Water in



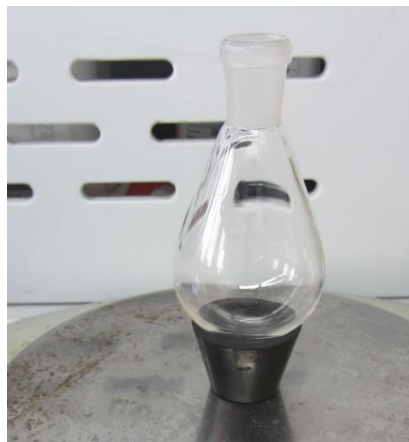
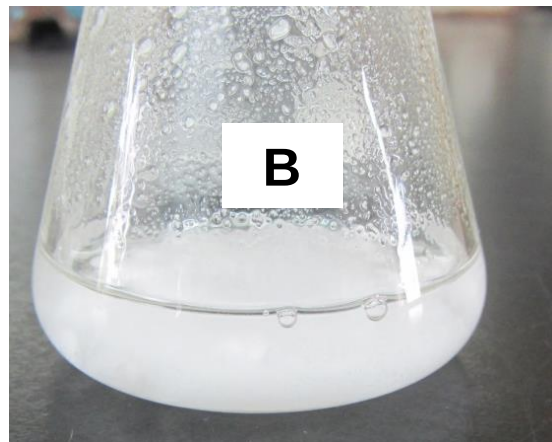
Aspirator pump

Safety trap

Suction Flask (fixed)



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



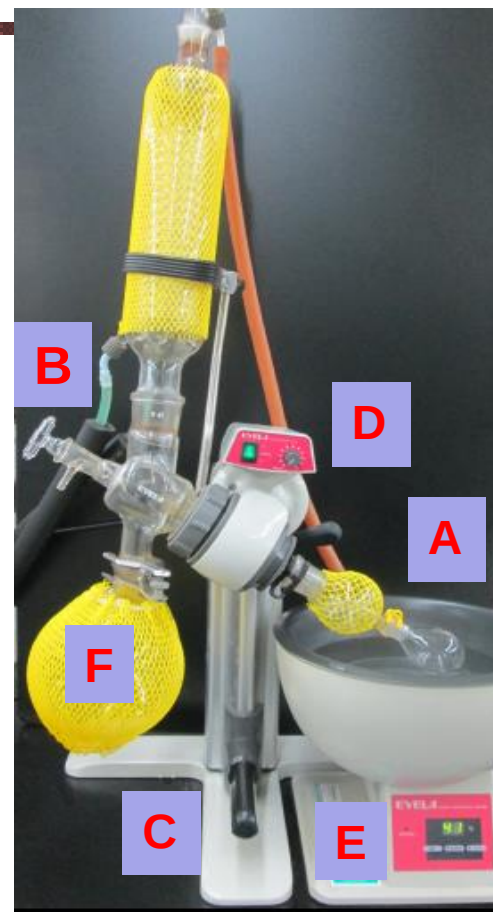
Rotary Evaporator (Rotavap)

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