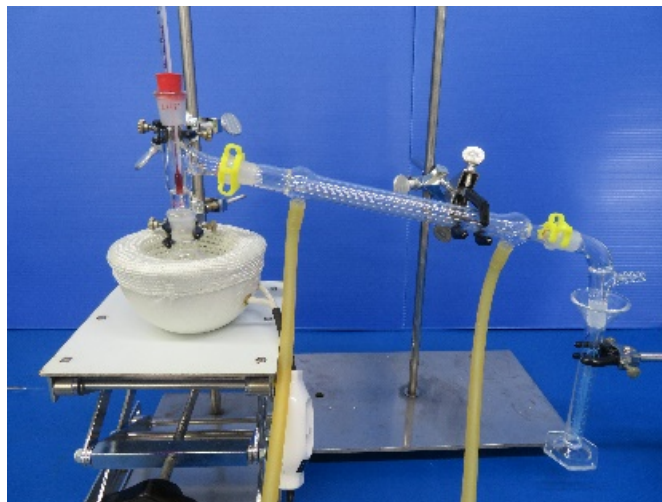




General Chemistry Laboratory

Distillation and Fractional Distillation





Preparation

Collect the following items

- Fractionating column
- Boiling chips
- Sea sand



From your personal equipment

- Lab jack, heating mantle and temperature controller
- Four extension clamps
- One 25 mL round bottom flask
- Condenser and two rubber tubes
- Distillation head
- Vacuum adaptor
- One-hole rubber stopper or septum stopper
- Thermometer
- Two 10 mL graduated cylinders
- Funnel
- Two keck clips



Objective and Principles

- **Objective:**

Learn to separate and purify substance by simple distillation and fractional distillation; then compare the differences

- **Principle:**

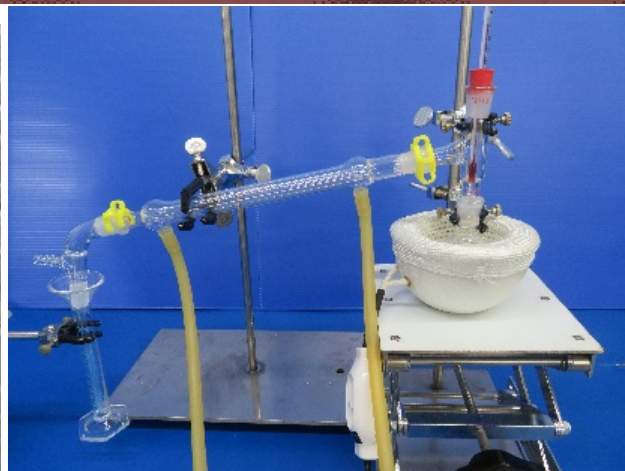
Use the difference in volatility, liquid acquires the needed thermal energy through heating to vaporize at its boiling point. Then, through the condenser, vapor is condensed into liquid and achieves the purpose of separation

- **Lab techniques:**

- Simple distillation
- Fractional distillation



Step 1: Simple Distillation

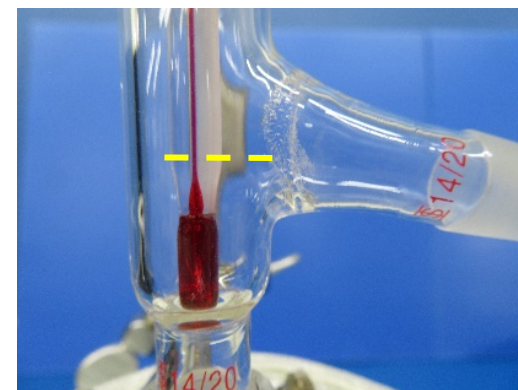
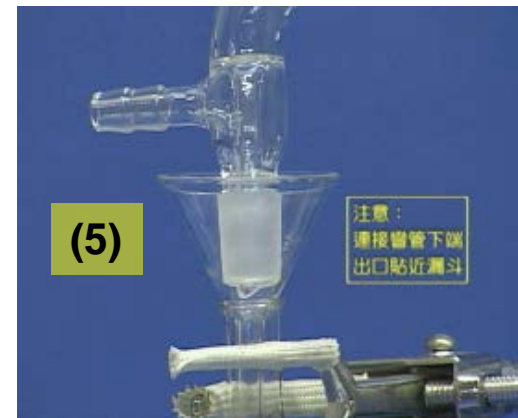
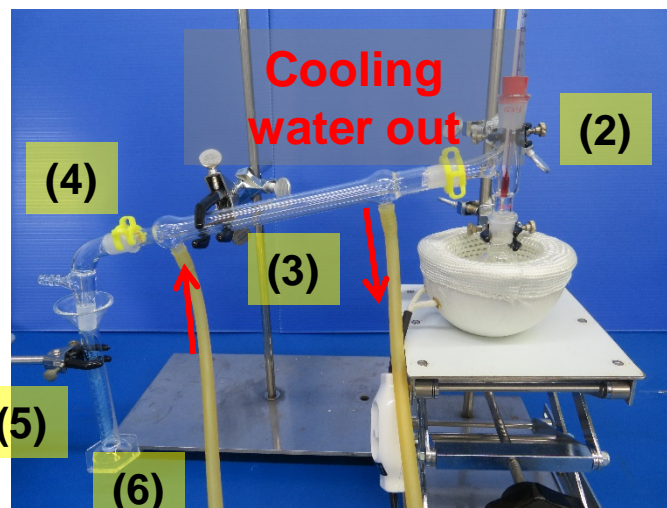
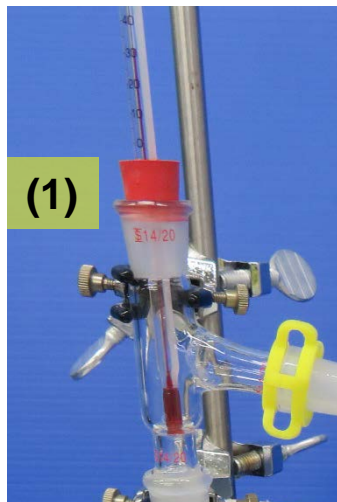


- Transfer 6 mL EA, 6 mL toluene, and 1-2 boiling chips to a 25 mL round bottom flask with the funnel
- Fix the round bottom flask with extension clamp
- Connect the heating mantle to the temp controller
- Raise the lab jack up to sit the flask in heating mantle
- Add sea sand to the height of liquid level

✓ Do not add too much sea sand that prolong the heating time



Step 2: Set up and Check of Simple Distillation

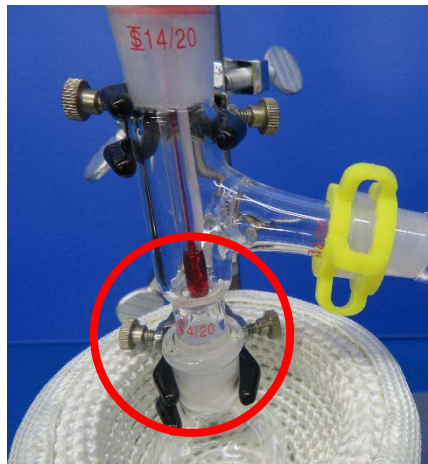


- (1) Connect distillation head and thermometer
- (2) Clamp the distillation head and condenser
- (3) Connect condenser with rubber tubes
- (4) Connect the vacuum adaptor
- (5) Connect the funnel and graduated cylinder as a receiver
- (6) Switch on the cooling water and adjust to suitable water flow. Water should flow from the lower inlet to the upper outlet.

✓ Check the position of thermometer



Step 3: Start Heating to Collect Distillate



✓ Check the set up by TA

- The entire assembly is upright and not tilted, and the ground glass joints are tightly connected
- The flask, condenser, and receiver should be clamped
- The thermometer bulb should be slightly below the sidearm opening of the flask
- Water flows from the lower inlet of condenser

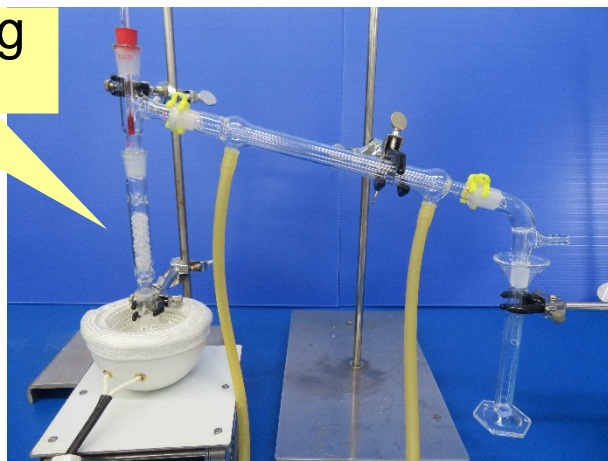
- Turn on heating
- Observe the refluxing and start recording the temp
- Record the temp once for every 0.5 mL of distillate collected until 10 mL of distillate collected
- ✓ Do not distill to dryness
- ✓ Adjust the heat input to a distillation rate of 1 drop/s

- Remove the flask out of heating mantle to avoid dryness
- Recycle the sea sand
- Record the atmospheric pressure



Step 4: Fractional Distillation

Fractionating column

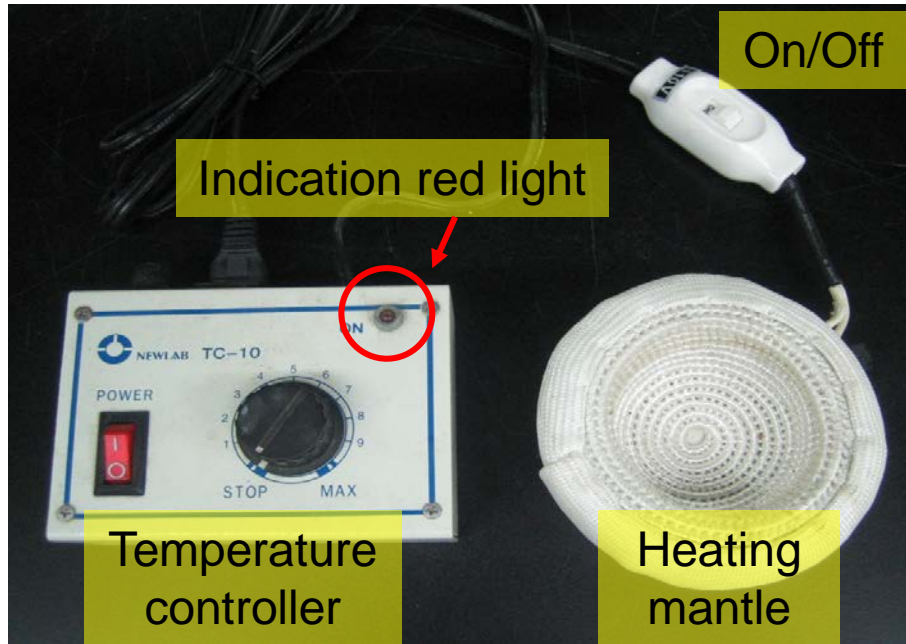


- Transfer the previously collected distillate to the round bottom flask and add new boiling chips
- Set up the fractional distillation devices and heat gently
- Record the temp when the first drop of distillate is collected
- Keep track of the temp. for every 0.5 mL distillate collected
- Record the total volume and temp. of the first component
- Raise the temp. of the heat source and keep recording the temp. until small portions of residue remains

✓ Wrap the column with aluminum foil to achieve the most efficient fractionation of distillate



Heating Mantle and Temperature Controller



- Connect the heating mantle to temp controller
- Turn the temp. controller on
- Turn the heating mantle on

Temperature Controller	
Appearance	Indication
Red light is blinking	<ul style="list-style-type: none">• Normal functioning
Red light is dark	<ul style="list-style-type: none">• Did not turn the temperature controller power on
Red light is always on	<ul style="list-style-type: none">• Did not turn the heating mantle power on or did not connect to controller

✓ Return the heating set to preparation room in case of malfunction



Additional Notice

- Operate the distillation in fume hood and turn the ventilation on
- Boiling chips should not be added during the heating process; new boiling chips should be added whenever distillation is restarted
- Set the distillation flask in the heating mantle then add sea sand
- All glass-to-glass connection should be tight and firm
- Be careful when installing the thermometer and avoid breaking it
- Set the distillation apparatus vertically
- The rubber tubing for cooling water should cover the nozzle in depth to secure the connection
- Cooling water flows in from the lower end of the condenser and flows out from the upper end
- At the end of distillation, remove the flask out of heating mantle quickly to avoid dryness



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 **Full Report** experiment, hand in the report next week
- Groups on duty shall stay and help clean up the lab

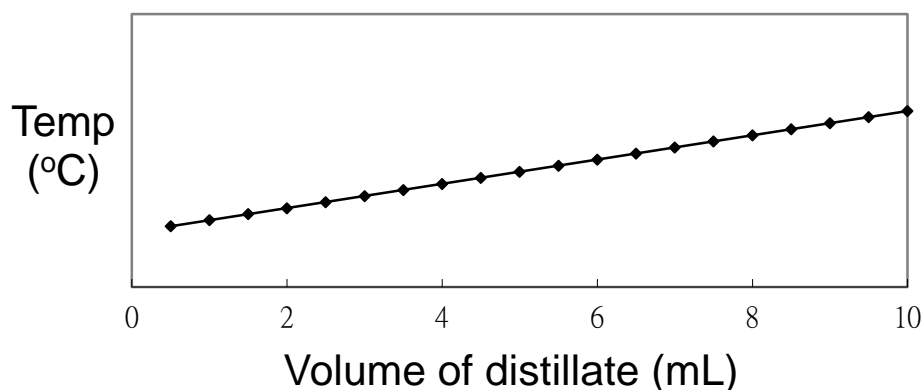




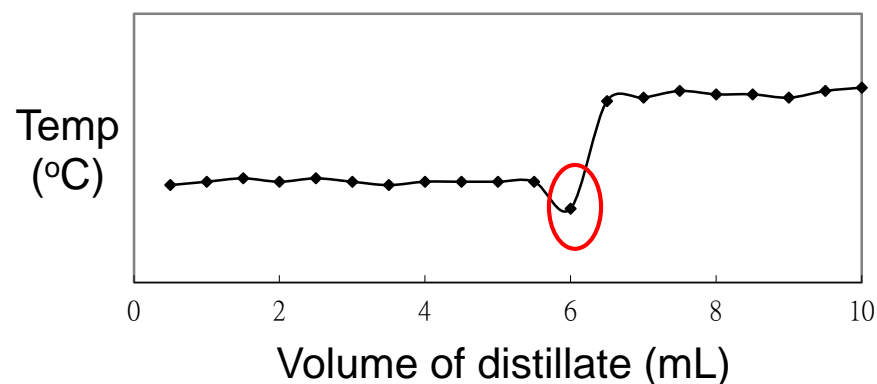
Report and Data Analysis

- Record the boiling point range, volume of distillate
- Indicate the height and materials packed in fractionating column
- Plot graphs with temperature as Y-axis and volume of distillate as X-axis

Simple distillation



Fractional distillation





Operation of Distillation

- Pour the liquid into the round bottom flask with a funnel
- Add 1-2 boiling chips to prevent bumping
- Clamp the flask
- Place heating mantle on lab jack
- Raise the lab jack to a suitable height
- Connect distillation head and thermometer
- Connect condenser and rubber tubes
- Connect vacuum adapter and receiver
- Add proper amount of sea sand; open the water valve for condenser cooling; water should flow from the lower inlet to the upper outlet
- Heat the round bottom flask until boiling begins
- Adjust the heat input to keep the rate of distillate 1 drop/s
- Collect the distillate in the receiver and record the temperature
- Continue distillation until only a small residue remains. Do not distill to dryness

