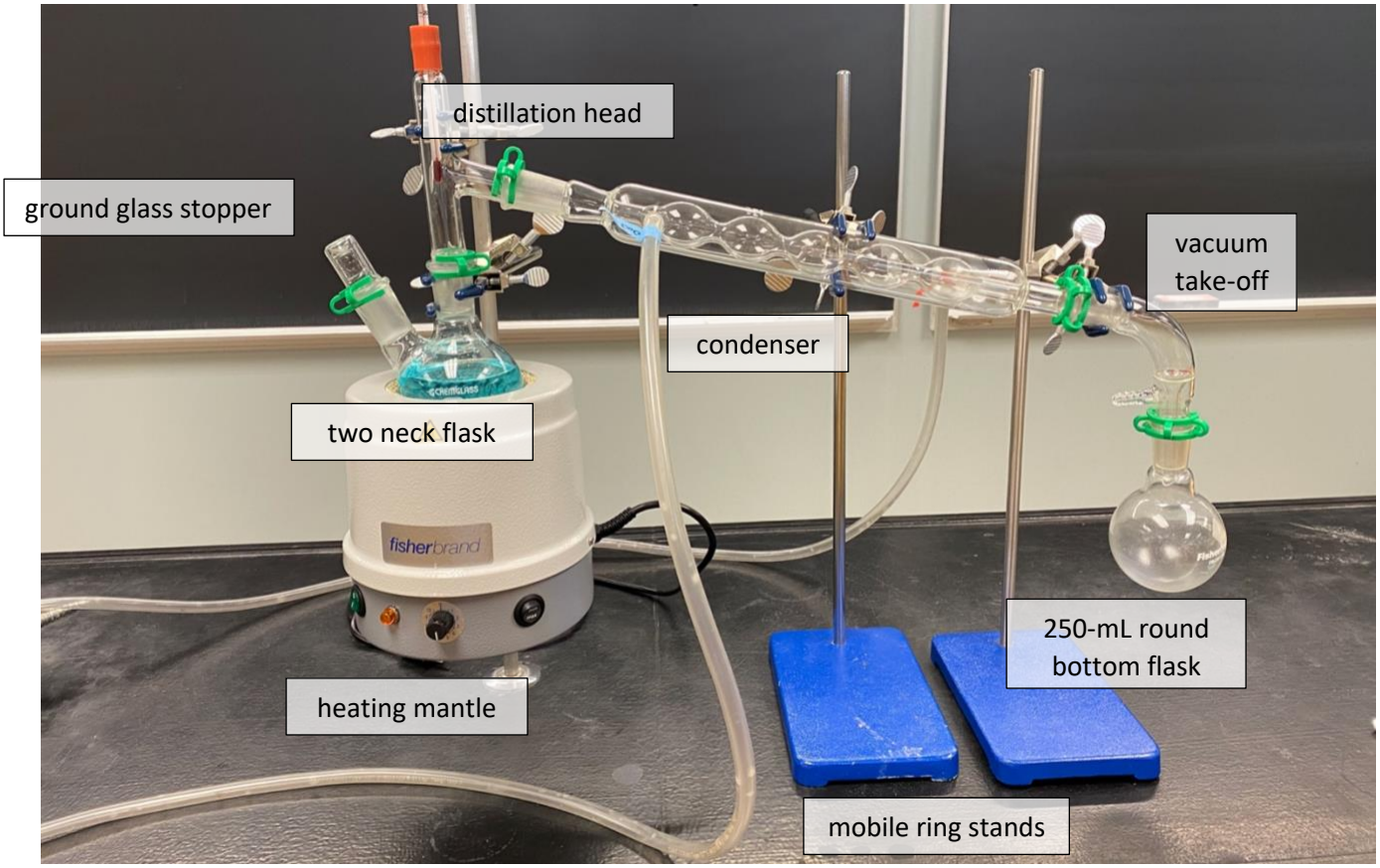
Updated: 8/27/2023

Experiment CPC Distillation Setup and Operating Procedures

General Statement

The distillation apparatus is composed of four main sections: the cooling water bucket, the heating mantle, the condenser, and the receiving flask. When properly assembled the apparatus should look like the one pictured below.



In non-renovated labs, the CPC distillation apparatus must be installed before the lab session starts and removed after the lab session has finished if the apparatus is not needed for the next lab session in the room. In renovated labs, the apparatus simply needs to be unplugged when it is not needed. Below we will review how to setup each of the four sections and how they are managed before, during, and after lab.

Safety Considerations

The distillation apparatus is hot and can cause thermal burns. Care should be taken to avoid touching hot components such as the heating mantle, the flask inside the heating mantle, and the distillation head. The substances used in the distillation apparatus (water and copper (II) chloride) can pose hazards to bare skin. A lab coat, goggles, and gloves should be worn when working with this equipment.

Distillation Apparatus Assembly

All equipment for the distillation setups in the non-renovated labs is stored in the blue-shelved cabinet in the 340/360 balance room. The key for this cabinet is the same as some of the spectrometer cabinets. The equipment for the renovated labs is stored in a cabinet in each room, near Hood A.

Setup should be done in a lab room to be sure all components connect appropriately. If the apparatus is not immediately needed in a non-reno lab, it can be removed and reconnected using the directions on page 7.

A. Cooling water bucket setup

Relevant Components

red bucket


Tubing


fountain pump

Note: This is usually done in the lab room when assembling the distillation apparatus for use.

1. Place the small red bucket in the sink.

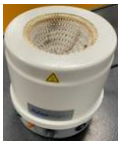
2. Use the suction cups on the bottom of a fountain pump to stick the pump to the inside bottom of the bucket. DO NOT PLUG THE PUMP IN.
   NOTE: Its ok if the pump is not completely stuck to the bottom of the bucket. The pump just needs to be in the bucket.

3. Attach the length of tubing labeled “supply” to the barbed connection on the top of the fountain pump.

4. Do not attach the “bucket end” of the tubing labeled “return” to anything. Be sure the end of this tubing is inside the bucket.

B. Heating mantle setup

Relevant Components


heating mantle


250 mL 2-neck flask


distillation head


thermometer


large rod


3-prong clamps (2)


keck clips (2)

grease


ground glass stopper


copper(II) chloride


0.5G scoop


glass beads

1. Place the large rod into the hole in the TA bench closest to the sink. In a new room, use a ring stand.

2. Slide the rod through the bracket on the back of the heating mantle. Use the thumb screw on the bracket to clamp the heating mantle about 6 inches above the benchtop.

3. Place the two-neck flask in the top of the heating mantle. Use a 3-prong clamp to clamp the vertical neck of the flask to the rod.

4. Add two scoops of copper(II) chloride and about 225-mL of distilled water to the two-neck flask.

5. Unclamp the flask and swirl to dissolve the solid. Add a few (3-4) glass beads to the flask.
   NOTE: a powder funnel can be used to fill the flask but is not included in the list of components.

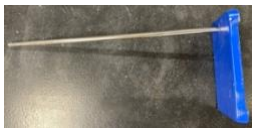
6. Place two small lines of grease (about the width of a grain of pea) on the ground glass stopper and insert it into the side neck; twist to spread grease and secure with a green keck clip.

7. Place two small beads of lines on the ground glass portion of the distillation head. Insert the distillation head into the vertical neck of the two-neck flask; twist to spread the grease and secure with a green keck clip. Use a 3-prong clamp to clamp the distillation head to the rod.

8. Carefully insert a glass thermometer into the top of the distillation head so that the bulb is aligned with the arm coming off the side of the distillation head (the bottom of the thermometer bulb is no lower than the bottom of the “armpit”). The scale on the thermometer should face the room.

C. Condenser setup

Relevant Components


mobile ring stand


condenser


keck clip


tubing


3-prong clamp

grease

1. Place two small lines of grease on the remaining ground glass joint of the distillation head. Insert into the condenser and rotate to spread the grease; secure with a green keck clip. Rotate the condenser so the water ports are parallel with the bench.

2. Secure the 3-prong clamp on the condenser to the ring stand.

3. Connect the supply tubing from the fountain pump to the lower port on the condenser. Connect the return tubing to the upper port on the condenser. The tubing should cover at least half the length of the port.

D. Receiving flask setup

Relevant Components


mobile ring stand


vacuum take-off


keck clips (2)


3-prong clamp


250-mL round bottom flask

grease

1. Place two small lines of grease on the ground glass portion of the condenser. Attach the vacuum take-off to the end of the condenser and rotate to spread the grease; secure with a keck clip.

2. Attach the 3-prong clamp to the mobile ring stand. The clamp should be attached to the vacuum take-off around the joint with the condenser. See photo below.

3. Place two small lines of grease on the bottom of the vacuum take-off. Attach the 250-mL round bottom flask to the bottom of the vacuum take-off and rotate to spread the grease; secure with a keck clip.
   NOTE: The flask will fall off of the vacuum take-off if not properly secured with a Keck clip.

Distillation Apparatus Start-Up, Maintenance, and Shut-Down

Start-Up

The distillation apparatus should be started 5-10 minutes before the start time of lab.

1. Fill the red bucket ¾ full with cold tap water. Fill to the lip with ice.
2. Double check the water lines are securely connected to the condenser. Plug in the fountain pump and the heating mantle.
3. Turn on the heating mantle (power switch glows green), set it to level 4 or 5 on high. The contents of the flask should begin to boil after about 15 minutes.

Maintenance

Every 30 minutes the distillation apparatus should be checked.

1. Replenish the ice in the bucket.
2. Allow students currently using the apparatus to finish.
3. Carefully remove the 250-mL flask from the vacuum take-off by removing the keck clip. Remove the ground glass stopper from the two-neck flask (may be hot). Pour the contents of the 250-ml round bottom flask into the two-neck flask.

Shut-Down

Both the fountain pump and the heating mantle should be unplugged after all students are done using the distillation apparatus. The contents of the 250-mL round bottom flask should be emptied into the two-neck flask.

Distillation Apparatus Setup & Removal Between Labs [old labs only]

Setup

1. Place the large rod into the hole in the TA bench closest to the sink.
2. Place the bucket containing the fountain pump into the sink.
3. Align the condenser with the distillation head, secure connection with a keck clip.
4. Align the vacuum take-off with the condenser, secure connection with a keck clip.
5. Connect the supply tubing from the condenser to the fountain pump. Place the return tubing inside the bucket.
6. Visually check all glassware for cracks or misalignment. Ensure the thermometer markings are facing the windows.

Removal

An aluminum support bar should be securely clamped to a large lab cart. The support bar has hole drilled into it that match the large rods used to hold the heating mantles.

1. Disconnect the water tubes from the pump and empty the bucket.
2. Disconnect the receiving flask portion of the apparatus from the condenser.
3. Disconnect the condenser from the distillation head.
4. Transfer the large rod with heating mantle and distillation head attached to the aluminum support bar.
5. Transfer the bucket, the condenser component, and receiving flask components to the cart.

Distillation Apparatus Teardown, Storage, & Inventory

Carefully disconnect all components and store them in their original packaging. Set aside, and notify the lab supervisor of, any discolored, broken, dirty, or cracked components. Use the table below to inventory all of the usable components at the end of each semester.

Item	Quantity	Initials
heating mantle		
250-mL two neck flask		
distillation head		
thermometer		
large rod		
ground glass stopper		
red bucket		
tubing		
fountain pump		
mobile ring stand		
condenser		
vacuum take-off		
250-mL round bottom flask		