



SOP: DISTILLATION

Distillation is the process of heating a liquid until it boils, then condensing and collecting the resultant hot vapours.

In the modern organic chemistry laboratory, distillation is a powerful tool, both for the identification and the purification of organic compounds. Distillation is used to purify a compound by separating it from a non-volatile or less-volatile material. The boiling point of a compound is one of the physical properties used to identify it. When different compounds in a mixture have different boiling points, they separate into individual components when the mixture is carefully distilled.

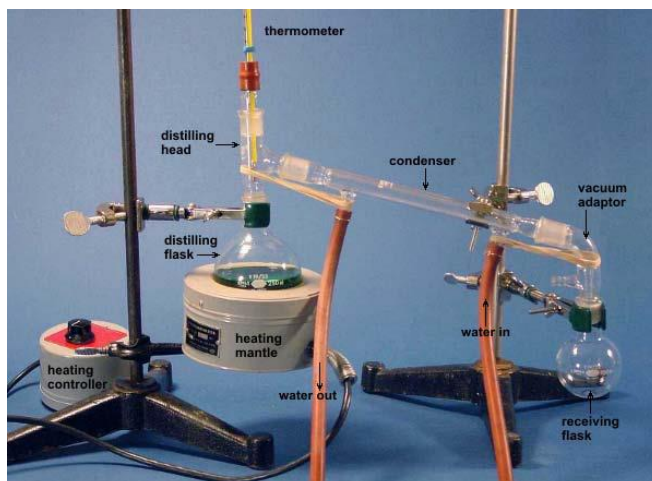
Simple distillations are used frequently in the following circumstances:

- the liquid is relatively pure to begin with (e.g., no more than 10% liquid contaminants)
- the liquid has a non-volatile component, for example, a solid contaminant
- the liquid is contaminated by a liquid with a boiling point that differs by at least 70°C

The more volatile liquid (the liquid with the lower boiling point) will typically evaporate first and the vapour will pass into a condensing column, where it can revert into a liquid (condense) on the cool glass where it trickles into a collection flask. Heating further will cause the less volatile liquids to evaporate and distil at higher temperatures. The two main kinds of distillation are *simple distillation* and *fractional distillation*, and both are used widely.

What is the difference between Evaporation and Distillation?

- In the distillation method, vaporization takes place at the boiling point whereas, in evaporation, vaporization takes place below the boiling point.
- Distillation is taking place from the whole liquid mass. In contrast, evaporation takes only from the surface of the liquid.
- At the boiling point of the distillation process, the liquid forms bubbles and there is no bubble formation in evaporation.
- Distillation is a separation or purifying technique, but evaporation is not necessarily so.
- In distillation, heat energy should be supplied to liquid molecules to go in to the vapour state but, in evaporation, an external heat is not supplied the pressure is lowered. Rather, molecules get energy when they collide with each other, and that energy is used to escape to the vapour state.
- In distillation, the vaporization happens rapidly, whereas the evaporation is a slow process.

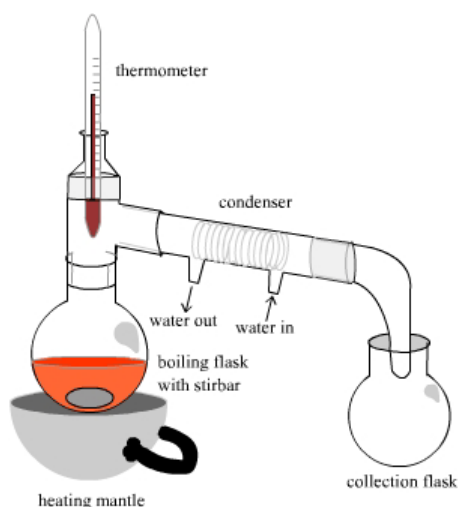




Simple Distillation

This technique is used to separate liquids with boiling points $>70^{\circ}\text{C}$ or separation of liquids from solid material. A simple distillation apparatus consists of a boiling flask (round-bottom flask) attached to a three-way adapter (stillhead) that is holding a thermometer (to determine the boiling temperature of the liquid). This adapter connected to a condenser (usually a Liebig condenser) into which cold water is constantly passed through. The condenser leads into a collection flask for the purified liquid.

The water supply (inlet) is connected on the bottom end, while the outlet goes either back to the sink or to the inlet of another water-jacketed condenser. An oil bath or a hotplate or heating mantle or block is used as heat source. The setup has to have a pressure release opening (usually the vacuum adapter). This set-up can be used under nitrogen or with a vacuum attached.



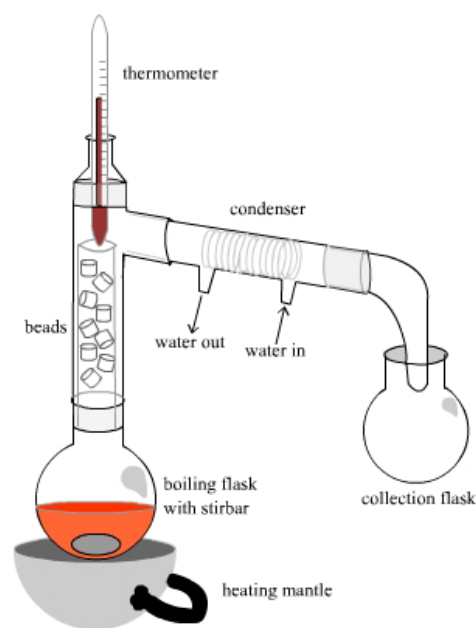
Steam distillation

This technique used when an organic compound co-distills with water. It is used to isolate organic compounds with high boiling points.

Fractional distillation

Fractional distillation is essentially the same as simple distillation except that a fractionating column is placed between the boiling flask and the condenser. This technique relies on increased number of liquid-to-vapour equilibria. It is used to separate 2 liquids with boiling points close to 25°C . The fractionating column is usually filled with glass or plastic beads or by using an Allihn condenser. Beads improve the separation between the liquids being distilled. The reason that fractional distillation gives better separation between the liquids is because the glass beads in the fractionating column provide "theoretical plates" on which the refluxing liquid can condense, re-evaporate, and condense again, essentially distilling the compound over and over. The more volatile liquids will tend to push towards the top of the fractionating column, while lower boiling liquids will stay towards the bottom, giving a better separation between the liquids.

Of course, the more theoretical plates that you add to a column (the more surfaces or beads), the longer the distillation will take (typically), and the more energy required to keep re-evaporating liquid in the fractionating column (this is more of a concern in industrial distillations than in an academic lab where energy cost is not a major cause for worry).





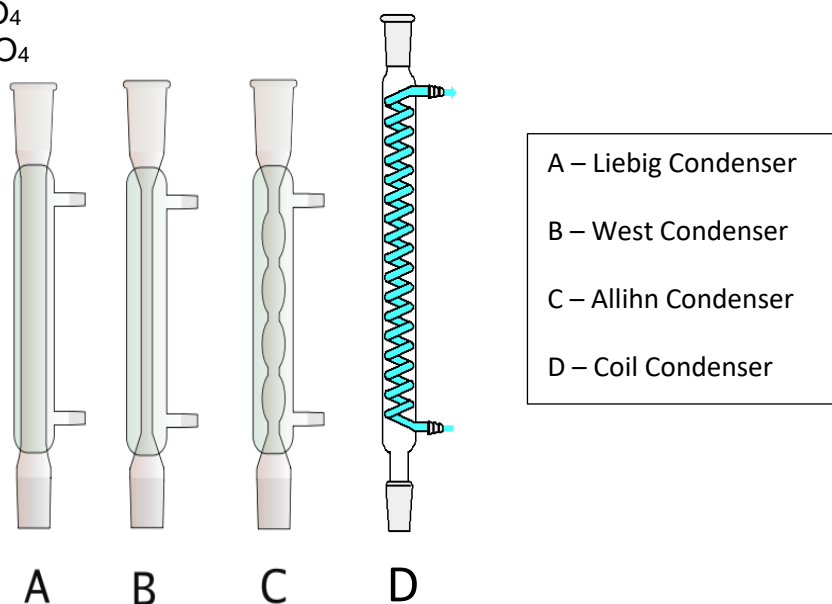
SETUP

1. Whenever possible do distillation in a fume hood. See Fume Hood Usage SOP for proper use.
2. Obtain the appropriate sized heating mantel (round bottom flask should fit snug).
3. Attach the metal rotating clamp to the support stand rod.
4. One can wet or heat tips of tubing in warm water to make it slide more easily over the in- & outlets. This assists not to break the condenser and helps to check if tubing is the proper length.
5. Attach the condenser to the support rod using the clamp.
6. Adjust the height of the condenser so that it is level with the boiling flask.
7. Apply lab grease at 2 points at top of all joints (not too close to the edge as it may cause contamination) 180° apart. This prevents leakage and freezing of heated glassware.
8. Turn joints to ensure grease is smoothed out over joint service.
9. Place the 3-way adapter into the boiling flask, and then connect it to the condenser tube.
10. Place the collection flask in the position to collect the fraction from the end of the condenser tube.
11. Attach silicone tubing to the bottom attachment or inlet of the condenser (where collection flask is) which is for the water supply and another tube to the top attachment or inlet of the condenser (on the sample with thermometer side) which is the water outlet of water to a drain.
12. Grease the thermometer joint as indicated above.
13. Suspend a thermometer in the boiling flask so that the top of the bulb is in line with the joint of the side arm to the condenser.
14. Lower the distil setup until solvent is in heating mantle or over heating source. Ensure heat is not directly applied to glass. Thus whatever heat source is used there must be a barrier between the flask and heat supply.
15. The apparatus is ready for use.
16. Turn on the water to the condensing column, and adjust the flow so that there are no bubbles visible inside the condenser tube.
17. Switch on the heating mantel. This should be connected to a temperature controller and indicator so that you can double check that the right temperature is applied.
18. The solution/sample will eventually start to boil. **DO NOT** let the solvent boil too vigorously.
19. A liquid boils when you observe a reflux ring going up the neck of your Hickman head and not when it starts to bubble. Often times, liquids has gases dissolved that are released prior reaching the boiling point.
20. Monitor the temperature on the thermometer. The temperature will not rise initially until the heated vapour reaches the bulb. Then the temperature will shoot up very rapidly and settle at the distillate's boiling point.
21. The temperature will be a good indication if you are distilling the correct solvent as each solvent has its own specific boiling points at the various pressures. This can be found on the web or boiling and vapour pressure tables.
22. Once the distillate's vapour reaches the condenser it will cool off in condenser and drip into the second collection round bottom or Erlenmeyer flask on the other side.
23. A good distillate rate is 1 drop / 1-3 sec. If faster it is a good idea to turn the heat down slightly or vice versa.



One can place a drying agent in the solvent solution, but you may have to purify your distillate afterwards. Only place 1 to 2 stones per 100ml solvent to distil. Drying agents include:

- a) CaCl_2
- b) MgSO_4
- c) Na_2SO_4



Example: Distillation of Ethanol

1. Set up the apparatus as described in "Setup."
2. Adjust the apparatus so that all the connections are tight.
3. Use an at least 400ml round bottom flask and place 100mL of ethanol and 100mL of distilled water in the flask. Solvent preferably should not be more than 50% of flask volume.
4. Place the boiling flask on the heating mantel large enough so that flask fits snug.
5. Set the heating mantel to about 80°C. (Ethanol boils at 78°C.)
6. Turn on the water to the condensing column, and adjust the flow so that there are no bubbles visible inside the condenser tube.
7. Closely monitor the heating liquid. When bubbles begin to form, turn the temperature up to 85°C.
8. As the ethanol begins to boil, the vapours slowly make their way into the 3-way adapter. At this point some of the vapour condenses and runs back into the boiling flask.
9. As the temperature of the vapour increases, the vapour begins to enter the condensing column and drops of the distillate collects in the Erlenmeyer or Round Bottom flask.
10. When the volume in the boiling flask is reduced to half the original volume, turn off the heating mantle and allow the apparatus to cool.
11. When the system is cool, remove the distillate flask.
12. This flask contains pure alcohol.

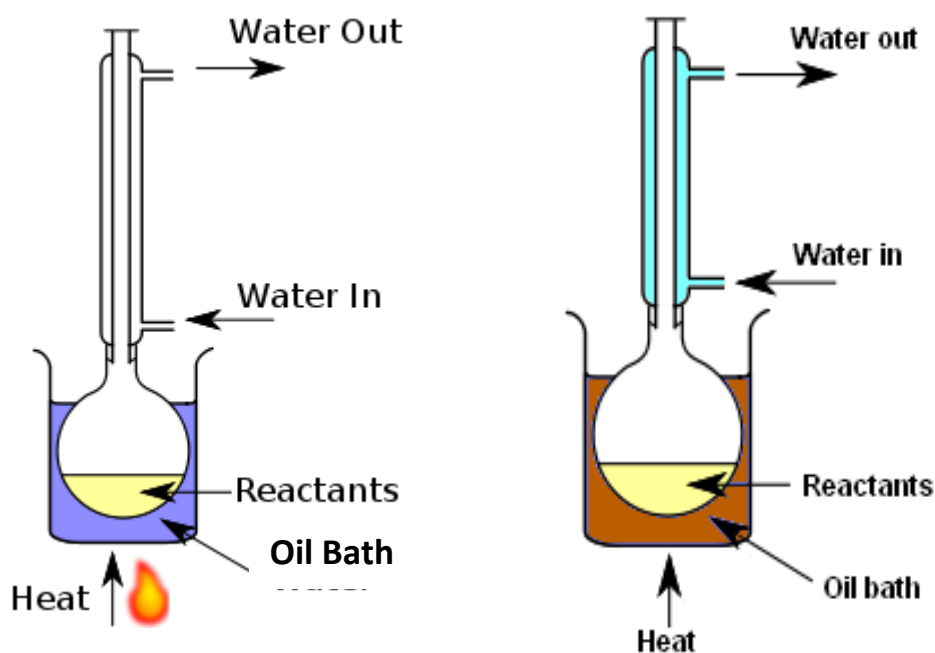


REFLUX TO SUPPLY HEAT TO REACTIONS

Reflux is a distillation technique involving the condensation of vapors and the return of this condensate to the system from which it originated. A liquid reaction mixture is placed in a vessel open only at the top. This vessel is connected to a Liebig or Vigreux condenser, such that any vapours given off are cooled back to liquid, and fall back into the reaction vessel. The vessel is then heated vigorously for the course of the reaction. The purpose is to thermally accelerate the reaction by conducting it at an elevated temperature (i.e. the solvent's boiling point).

The advantage of this technique is that it can be left for a long period of time without the need to add more solvent or fear of the reaction vessel boiling dry as any vapour is immediately condensed in the condenser. In addition, as a given solvent will always boil at a certain temperature, one can be sure that the reaction will proceed at a constant temperature.

The diagrams show a typical reflux apparatus for applying energy to chemical reactions. It includes an optional beaker of silicon oil or a heating mantle which heats the reactants at a constant temperature.





DRYING OF SOLVENTS

The process of synthesizing and isolating an organic compound often results in an organic compound or solution contaminated with traces of water. For instance, in aqueous extractions some water will be transferred into the organic phase because of the partial miscibility of the organic phase and water. Also, many reactions themselves are performed in an aqueous solution. This water must be removed before the required compound can be properly characterized. DO THIS IN A HOOD!!

Drying agents:

Alumina, Al_2O_3

Barium oxide, BaO

Boric anhydride, B_2O_3

Calcium chloride, CaCl_2

Calcium hydride, CaH_2

Calcium sulphate (Drierite), CaSO_4

Lithium aluminium hydride (LAH), LiAlH_4

Magnesium, Mg

Magnesium sulphate, MgSO_4

Molecular sieves

Phosphorus pentoxide, P_2O_5

Potassium hydroxide, KOH

Sodium, Na

Sodium sulphate, Na_2SO_4

1. Remove as much impurities from the glassware as possible beforehand with a proper washing technique.
2. Afterwards wash your already cleaned glassware with acetone to remove any water residue.
3. Use pure diethyl ether to remove any crud from the acetone.
4. Select the solvent and identify the ideal way of drying it.
5. Use a still to distil this solvent.
6. Set up your equipment so that the solvent will stay dry e.g. once dry and cool put the glassware under argon.
7. It is also a good idea to grease the farthest part of all joints lightly with silica grease to ensure an airtight seal.
8. Add the solvent and your drying agent to the solvent.
9. Dissolve the benzophenone in THF/solvent, then add the sodium.
10. Begin your distillation at the required temperature making sure you have a sufficient cooling system in place (i.e. if your solvent boils at lower temperatures then cool it well with ice water, but if it boils high or freezes at low temps then water at room temperature is not a problem).
11. Do not boil the still dry, this is dangerous, simply leave enough solvent to cover the drying agent completely
12. Once the distillation is complete, disconnect your collection flask ensuring that it is kept under argon, place a stopper (using a small amount of grease at the top of the neck) and place to one side.



DRYING OF SOLVENTS

Dispose of your waste in an appropriate waste container.

The use of activated molecular sieves or some other innocuous dried material (e.g. KOH for TEA) can be placed in your dry collection flask to prevent the solvent becoming wet over time.

Quenching: is scary stuff if you do not do it right. Initially to quench something using CaH_2 or Na you want to use something that will react with the agent but in a controlled way, for these two agents Isopropyl alcohol is a good choice as it will not react quickly with the agent.

First, cool the remaining solution on ice. Next slowly add Isopropyl alcohol (add a decent amount, usually around 50-100mL) and allow to stir for 15min or so. Now you can add Ethanol or Methanol (DO NOT ADD WATER) and continue to stir it on ice. Eventually, you can start to add water dropwise to complete the quenching of the solution but be mindful of the temperature of the solution and the development of bubbles. This is a warning that the quenching is occurring quite rapidly.



HEALTH & SAFETY ISSUES

1. **DO NOT** fill the sample round bottom flask more than half in order to leave enough room for the liquid to boil.
2. **DO NOT** forget to add a spin bar or a boiling stones (not both) to avoid bumping.
3. Ensure the glassware is secure by using e.g. clamps on stander and Keck clips at joints.
4. To avoid breaking the condenser, ensure the condenser is securely fastened and the stand assembly is stable.
5. Make sure that you have a good seal between the three-way distilling head and the water-jacketed condenser; otherwise you will experience leakage there and dripping on the heat source that may cause a fire.
6. Avoid heating by direct flame. Keep in mind that other heating forms can also cause problems. Be careful not to sustain burns at heat source or from boiling solvent or distillate. Let system cool off before removing components.
7. If possible know the boiling point of your sample and gradually turn up the heat source until the thermometer indicates a temperature just higher than your sought after boiling point – **DO NOT** start at a very high temperature unless it is automatically controlled by the heat source.
8. It is imperative to remove the drying agent after you distil the liquid.
9. Select your distillation glassware with care: **DO NOT** use any that are cracked or scratched.
10. It is advisable **NEVER** to use a flat-bottomed flask in any form on the heated side of the distillation.
11. **DO NOT** use a water-cooled condenser if the distillate entering the condenser is above 160°C.
12. Nitrogen is preferable to air on all capillary bleeds since many organic substances tend to oxidise at elevated temperatures.
13. Use a Quick fit capillary fitting, it is not good practice to use rubber stoppers in distillation equipment since they tend to soften with heat and may be pulled into the distillation flask.
14. Never subject the setup to rapid changes in pressure.
15. At the end of distillation under pressure - allow it to cool off before slowly admitting air.
16. Clean glassware right after finishing with distillation, especially when there are solids attached. Follow the Glassware Cleaning SOP found on Chemistry Health & Safety on Vula.
17. Always monitor a distillation to ensure it does not boil dry:
 - a. Can crack glass
 - b. Impurities in flask start to concentrate and chemicals such as peroxide forming ethers can peroxide and cause an explosion or fire. Thus always check ethers beforehand for any peroxides present and distil under nitrogen gas.