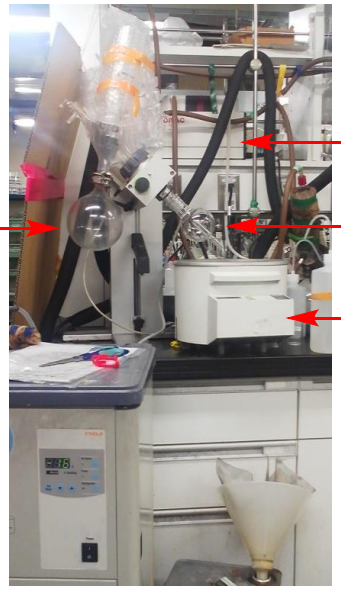
Rotary Evaporator



Operation

1. Turn on Cool Ace (all the day).
2. Remove remaining solvents in the solvent trap before use.
3. Attach a flask containing the solution to the bump trap with a joint clip.
   (Stick the stirrer bar to the upper side of the flask with a magnet in order to avoid bumping.)
   Bad-smelling solutions should be evaporated in the fume hood.
   KOH trap should be used when the solution contains acid.
4. Start the rotation and turn on the aspirator.
5. Heat the water bath if necessary.
6. When all solvent has been removed, turn off the vaccum and stop the rotation.
   Don't leave the flask for the long time when finished.

After use

1. Turn off the water bath after use.
2. Clean up bump trap, solvent trap and water bath if it gets dirty.

Glass vial

The vials can be connected to the bump trap with silicon adapter.
(Be careful not to drop the sample!)

5 mL　　10-20 mL　　50 mL

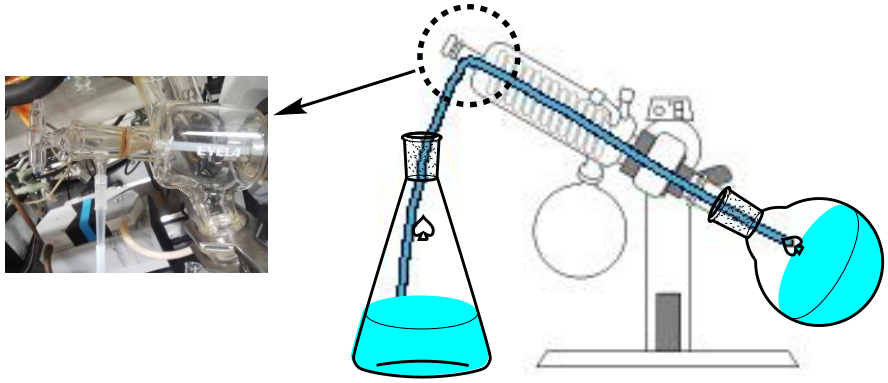
KOH trap

When the solution contains **Acid**, **Thiol** or **Sulfide**, **KOH trap** or **$HClO_2$ trap** shoud be used to prevent the damage to aspirator.

After using acid

1. Wash the inside of evaporator with acetone.
2. Keep the aspirator idling for more than 5 minutes.

Large scale evapolation



Principle

♦The solution in outer flask is drawn into inner flask under redused pressure.

♦Removal of solvents and injection of the solution are in equilibrium.

Attention

Large flasks (≥ 1 L) should be attached directly (without using the bump trap) and put in the water bath from the beginning not to damage the rotary axis.

Solvent containing powder

Block the bump trap with **cotton** when the solution contains powder such as silica gel.

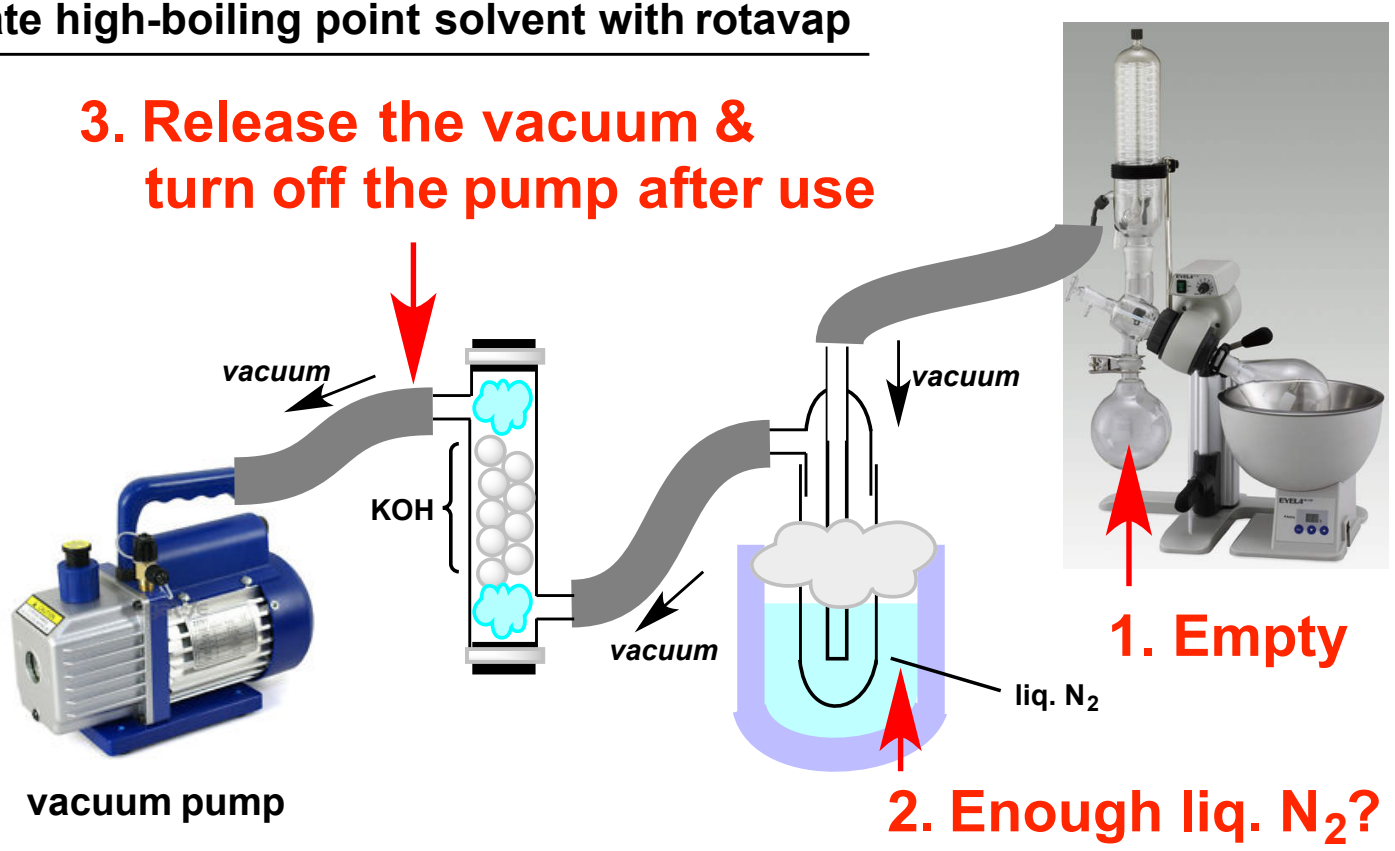
Azeotropic data

High boiling point solvent can be removed by mixing with another solvent which makes azeotropic mixture.

t_{az} = Azeotropic point, X_1 = mole fraction of component 1

Component 1	Component 2	t_{az}(°C)	X_1
Water	Toluene	84.1	0.44
	Benzene	69.3	0.30
	2-Propanol	80.4	0.35
	1-Butanol	92.7	0.75
	2-Butanol	87.0	0.60
Acetic acid	Hexane	68.3	0.13
	Heptane	91.7	0.45
Pyridine	Toluene	110.1	0.25
DMF	Heptane	97.0	0.08

Evaporate high-boiling point solvent with rotavap



Purpose of protocol

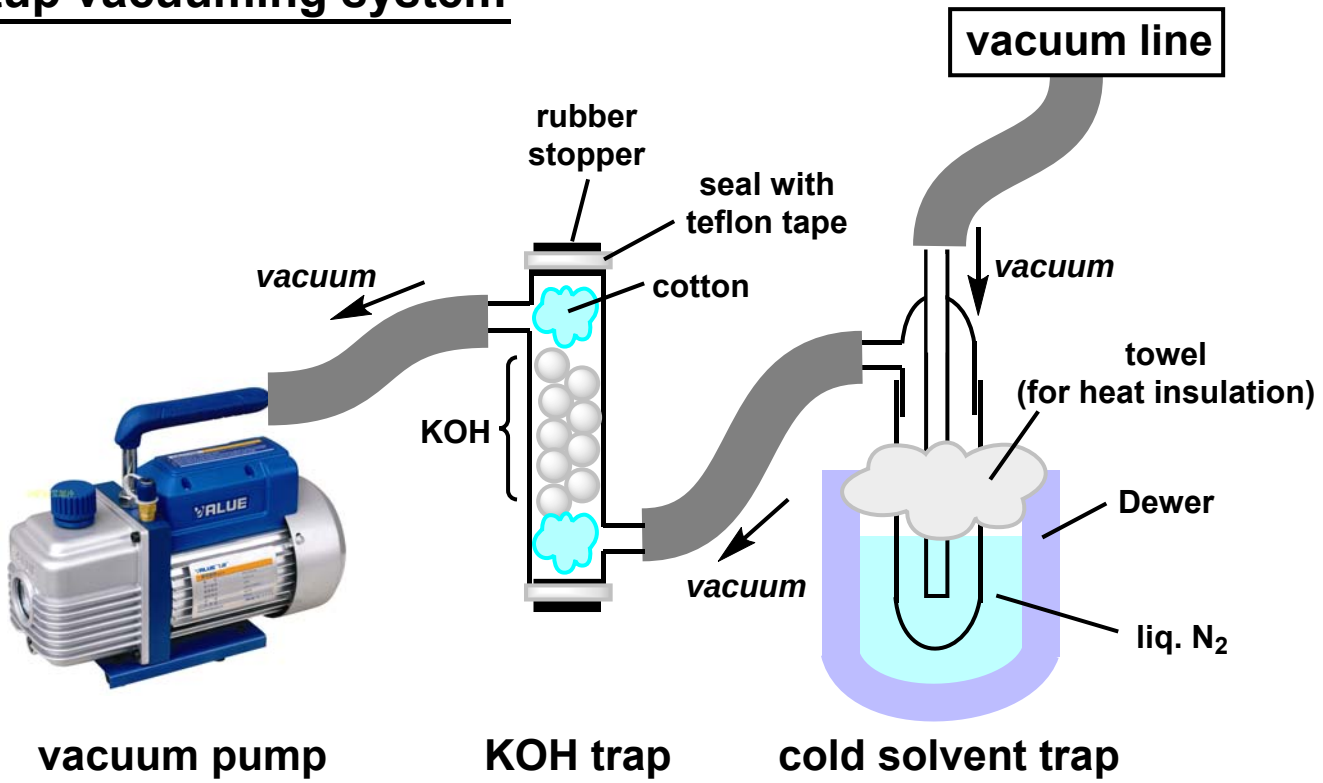
When you want to evaporate high-boiling point solvent, such as DMF, you may use a vacuum pump connected to the rotavap instead of a diaphragm pump.
Prevent damage on the vacuum pump by carefully trapping solvent vapor.

TIPS

1. Empty the 1st solvent trap to prevent vacuuming low-boiling point solvent, such as AcOEt.
2. Check whether there is enough liq. N_2 in the dewer.
3. Make sure to turn off the pump after you use, otherwise liq. N_2 will evaporate to eventually cause inflow of the trapped solvent vapor to the pump and damage it.

Temp. of water bath: For water, ~30 °C. For DMF, ~40 °C.

How to setup vacuuming system



Purpose of protocol

Cold solvent trap is used for capturing solvent vapor. KOH trap is used for capturing moisture and acidic volatiles.
They often damages vacuum pump severely if directly entered.
You ALWAYS have to connect both between pump and vacuum line in order to minimize the damage.

TIPS

You have to evacuate inside of the trap BEFORE cool down in liq N_2.
If you cool down before vacuum, explosive liquid O_2 will be generated inside.
You have to avoid such a very dangerous situation.

liquid O_2
(blue-colored)