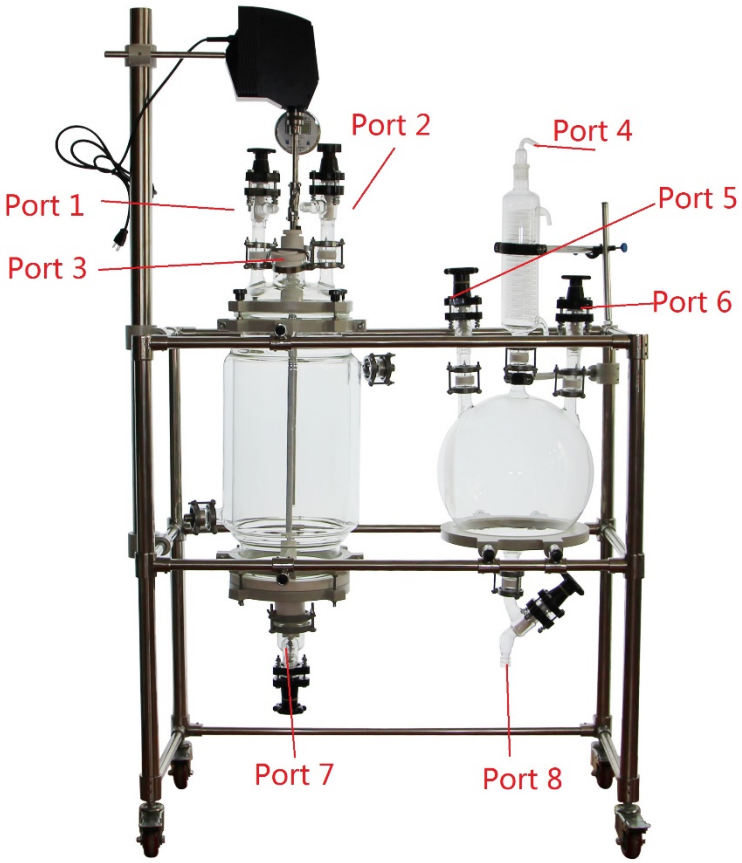
Setup and SOP for filter reactor (Crystallizer)

Set up your equipment with right tubing and connection.



Port 1: Connect it with PTFE tube, can be used to pump in your solvent, remain close during operation

Port 2: Connect it with Port 5, it transfer solvent vapor for condensing.

Port 3: It is a big opening, for adding your distillate in your reactor.

Port 4: Connect it with a vacuum pump, provide vacuum for your operation

Port 5: Connect it with Port 2.

Port 6: Connect it with Port 7, transfer mother liquid into collection chamber

Port 7: Connect it with port 6, transfer mother liquid to collection chamber after crystallization is complete.

Port 8: Collect condensed solvent or mother liquid.

Supporting Unit:

1: Heater/Chiller temperature control unit. Temperature range is -30C to 100C. Cooling capacity depends on the reactor size you have. For 20L unit, 1.6kw is good enough, for 50L, need 3kw. For 100L unit, need 4.5kw. Lower cooling capacity unit can be use, it just take longer to bring temperature down. Heating capacity: 2kw-4kw is good enough.

2: Chiller for cold trap: A small chiller is needed to maintain the cold trap at -20C or lower. Chilling capacity can be low, since there will only be a small amount of solvent evaporating.

3: Vacuum: A water vacuum pump is good enough.

Heat transfer liquid used in this operation:

The operation temperature for the jacket is from -30C to 80C. You can choose any heat transfer fluid that are stable at this range. The viscosity of this heat transfer fluid need to be low at this temperature range (around 50 cst). Recommended heat transfer fluid: Intercool P-300

Solvent and Ratio:

Pentane : Distillate ratio by volume: 2:1

This is a safe ratio for new user to get use to this machine and SOP. You

can decrease the amount of solvent when you are more familiar with this process.

Also pre-chill another bucket of pentane in your freezer for washing the crystal. If you using 10L pentane and 5L distillate for mixing, chill 10L pentane for wash. Chill it down to -10C if possible.

Crystallization:

1: Bring jacket temperature to 35C

2: Close Port 1,3,6,7,8. Port 4 is always connect to vacuum, no open/close option for Port 4

3: Port 2 and 5 are open, connecting reactor chamber and collection chamber

4: Turn on vacuum pump.

5: Start adding solvent by using Port 1, use marker on the reactor to check the volume of solvent you added in.

6: Close Port 1, start the motor and set mixing blade to 400 rmp.

7: Check thermometer reading, let solvent temperature come close to jacket temperature (jacket temperature is set on your heater/chiller unit). It will not be exactly the same, 5C difference is acceptable.

8: Warm your distillate up to 40C or higher, make it easy to transfer. Then add it into your reactor using Port 3. You can use a funnel. Vacuum will be break once you open Port 3.

9: Close Port 3 once all distillate is all added into the chamber. It will prevent pentane vapor escape and create potential hazard.

10: Let pentane and distillate mix well in reactor (20 mins), and make sure vacuum is pulled in this setup.

11: Start to lower the temperate of jacket, by 10C each adjustment.

12: After jacket is reach to each lower setting, wait 20 – 30 min for the liquid in the reactor to chill down before you lower the jacket temperature further.

13: When liquid temperature close to 0C, you will see crystal forming rapidly in reactor.

14: Set Chiller to -25C and wait inner temperature low down to -10C

Collect Mother Liquid and washing

15: Close Port 2 and 5

16: Open Port 6 and 7, vacuum will pull mother liquid from reactor into collection chamber.

17: Start adding pre-chill ethanol when mother liquid is close to all gone. Do not completely dry out the reactor. Your mixing blade will be stuck in the crystal if you completely dry the reactor. Let the mother liquid pull out when you adding fresh new pentane.

18: Once the liquid pulling from reactor doesn't look like mother liquid, Close Port 6 and 7, let fresh pentane mix with crystal for 5 min.

19: Close vacuum pump and open Port 8 to collect your mother liquid

20: Close Port 8, Pull vacuum, Stop the mixing motor

20: Open Port 6 and 7 and completely dry out your pentane.

21: Close vacuum pump and open Port 8 to collect your clean pentane

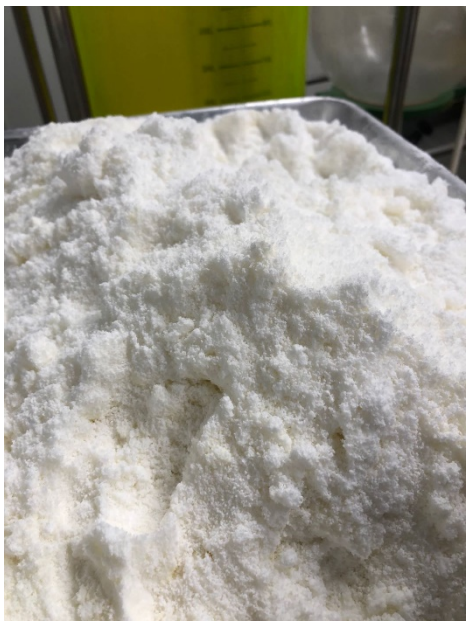
Collect CBD Crystal

22: Close Port 6,7,8, and open Port 2 and 5, Pull Vacuum, Set your heater/chiller to 60C, bring up the temperature to dry out pentane.

23: Wait 30 min after your jacketed reach setting temperature.

24: Close vacuum pump and open the bottom of the reactor to collect crystal. You need 2 people to complete this job. And place a large tray below the reactor is a good idea.

25: Close all supporting unit



Credit to Future4200 for providing perimeter for me to start with

Credit to Jon and Allan at Shyne Lab providing distillate, equipment and site for running this equipment.