Vacuum Distillation Instructions
(for purification of nopinone on an ca. 5 g scale)

1. Transfer your sample, which you have carefully checked in advance to ensure that it does not contain peroxides, to a tared 50 mL round bottom flask with a 14/20 neck.
2. (Carefully) Remove all excess extraction or transfer solvent on a rotary evaporator. This is an important step because residual solvent will often 'bump' during distillation, which will negatively impact the efficiency of the purification.
3. Measure the crude mass of your sample at this point.
4. Add a magnetic stirbar to the flask; the largest football shaped bar that readily will fit into the neck of the flask is desirable.
5. The vacuum distillation apparatus is stationed in the east hood. Ensure that the cold trap is charged with dry-ice and acetone or isopropanol (either solvent is fine).
6. Apply a light layer of silicone vacuum grease to the top half of all male joints on the all-in-one short path distillation head.
7. Attach the 50 mL flask, the distillation "pot," to the distillation head with a yellow Keck clamp and clamp the head itself to the three finger clamp that is situated above the silicone oil heating bath. This bath sits atop a magnetic stirrer. Do not add additional clamps to the setup. Lightly grease the top portion of the ground-glass joint on the standard taper thermometer and insert it into the distillation head.
8. Tare three 25 mL round bottom flasks (14/20 necks) as potential receiving flasks.
9. Attach one of the three 25 mL round bottom flasks to the short path setup with the aid of a yellow Keck clamp. Gently rotate all greased joints to ensure the grease is evenly spread to provide a good vacuum seal.
10. Turn on a **gentle** water flow through the condenser.
11. If it is not already attached, connect the vacuum hose to the vacuum sidearm on the distillation head.
12. Check your setup with a TA.
13. Place a safety/blast shield in front of the setup.
14. Turn on the magnetic stirrer to a gentle speed and turn on the vacuum pump to reduce the system pressure. Have the TA show you how to use the McLeod gauge to measure the internal pressure BEFORE beginning to heat your distillation pot (i.e., 50 mL flask). Two valuable rules of thumb are to i) always rotate the McLeod gauge slowly, both before and after taking your reading of the reduced pressure and ii) always leave it in the upside down position except for the 30 seconds or so when you are actually taking a pressure reading. Once you have recorded the pressure and slowly returned the gauge to its inverted position, isolate the McLeod gauge from the system by closing the stopcock and then carefully further rotating the stopcock to vent the gauge to atmospheric pressure.
15. If excessive bubbling and/or bumping is observed then turn off the vacuum pump, vent the system, and remove additional residual solvent on the rotary evaporator. (Check with a TA if your solvent bumped into the distillation head.)
16. Reassemble your apparatus and proceed.

17. Once all bubbling and/or bumping has subsided, lower the apparatus carefully into the ambient temperature oil bath or raise the bath to immerse the pot with the lab jack below the magnetic stirrer.
18. Submerge the flask as deeply as your clamp (and the paper clip stirrer in the oil bath) will permit.
19. Ask the TA to help you turn on the variac (a variable transformer) and adjust it to an appropriate setting; this provides current (and heat) to the oil bath. *Note: very small changes (<1 V) in the voltage setting can result in large changes in temperature.*
20. Monitor the temperature of the oil bath with the external hanging thermometer.
21. Watch for droplet formation in the condensing arm of the head.
22. Once the first one or two droplets are collected in the receiving flask, remove the apparatus from the heat source (most easily done by carefully lowering the scissoring lab jack supporting the oil bath), use the T-stopcock to vent the apparatus to atmospheric pressure (while leaving the vacuum pump isolated and under vacuum, replace the receiving flask with a second tared flask, reduce the pressure once more (stopcock change), and reapply heat by slowly re-immersing the distillation pot into the oil bath (by carefully raising the lab jack).
23. The liquid that begins to distill next should be your purified product; continue to apply heat and distill all contents of the pot into the receiver. You will likely find that the temperature of the internal thermometer (with which the boiling point is measured) will be 20-30 °C lower than that of the external oil bath. Record the temperature range of the internal thermometer while droplets are being collected. You can coax the last half mL or so of pot content over to the receiving flask by warming the glass joint between the pot and distillation head with a heat gun. Don't make is to hot that you melt the Keck clamp and don't point the gun downward into the oil bath, because that will spray hot oil.
24. Terminate the distillation just as you did when you changed the receiver flask. Record the mass of the distillate – your pure product.
25. Clean the outside of your distillation pot with a paper towel and hexanes to remove the silicone oil and remove the residue inside the pot flask with acetone (and into organic waste) before further washing it with soap and water.
26. Rinse all surfaces of the short path distillation head, inside and out, with acetone, catching the washing in a beaker, to ready it for the next user.
27. Be careful not to contaminate the silicone oil bath with any of the acetone wash solvent (or any other organic contaminants).

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