

Solvent System Walkthrough

The Contour Glass solvent system is designed to give you air-free anhydrous solvent with minimal effort. However, the system is only as good as its users so please read this guide carefully. Refer to it or the stills operators (Jenna or Scott) whenever you have questions about proper use.

POLICY

Dry solvent is a privilege, not a right. The push stills have NO mechanism to wash out the bulb, so the previous user's behavior affects everyone else. Compliance with the following instructions is important and ensures the system is used properly. Failure to follow these rules will result in your loss of access to the solvent system. Have fun distilling your own ether/THF.

- **Always use personal protective equipment (safety glasses, gloves, lab coat) when using solvent system.**
- **Use the logbook and be as accurate as possible.** This enables the stills operators to know when the kegs of solvents are low and to find the source of problems with the system when they occur. By the time solvent does not come out, the columns have already been run dry and the operators will have to restart them, which is an exothermic and dangerous process.
- **Purge your needle and syringe with the argon supplied by the system.** If you forget to do this, DO NOT expel ANYTHING back into the collection bulb. If you do this, you risk introducing moisture and oxygen into the solvent and ruin it for other users.
- **Try not to distill more solvent than you need.**
- **Do not throw away the solvent.** It costs over \$1000 to load the THF keg. Check the logbook for when solvent was last distilled. If you think you need fresh solvent, please recycle the old stuff. See page 6 for instructions.
- **NEVER apply vacuum to a collection bulb that has ANY solvent in it.** If distilling new solvent, dry the collection bulb with a stream of argon first.
- **Don't use 16 gauge needles on the system, they totally destroy the septa.** If you need to use a big needle, replace the septum after use.
- **Close the stopcock!** Once pierced, the septum will not contain the argon and you will waste large amounts of expensive gas.
- If you encounter any problems with the system, talk to the stills operators (Jenna or Scott).

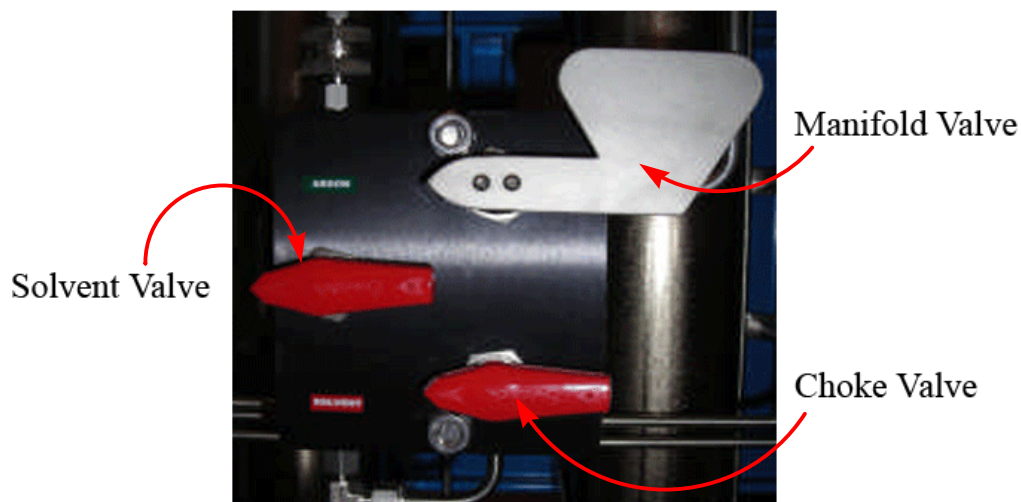
TRAINING

Because the solvent system is only as good as its users, all users are required to be trained *by the stills operators* (Jenna or Scott) and check out before they are allowed to use the system independently. This is important to ensure people understand how to use the system correctly.

YOU MAY NOT USE THE SYSTEM UNTIL YOU HAVE BEEN TRAINED!

HOW IT WORKS

The system works by pushing solvent from storage kegs through a drying tower. You will control the system with the valve plate located in front of the column. Note that everything is color coded and you should NEVER need to manipulate an uncolored valve. You should only manipulate the valves on the valve plate, which looks like this:



There are three valves, which all do different things.

Manifold Valve: This selects for argon or vacuum. The plate attached to it prevents opening the column up to vacuum, which results in solvent in the manifold and pump.

Solvent Valve: This opens the collection bulb to the column. If this is opened and argon pressure is applied, solvent will come out.
(Note: if the bulb is already at the same pressure as the column, nothing will happen).

Choke Valve: This closes off the collection bulb to the manifold. If the choke valve is closed, neither vacuum OR argon can be applied to the collection bulb.

A note on Swagelok valves: The valves are all designed to point in the direction of flow (or in the case of the manifold valve, the mode selected). If you are in doubt about what will happen when you open a valve, know that when the valve is lined up with tubing, that line is open. In the picture above, the argon manifold is open but the solvent and choke valves are both closed.

OBTAINING AND “DISTILLING” SOLVENT

This section of the guide is broken down into several usage scenarios.

Scenario 1: Obtaining solvent that is already in the bulb.

If the amount of solvent you need is already in the bulb, follow these directions:

1. Take a dry needle from an oven and assemble syringe like normal.
2. Check the valve panel corresponding to the solvent you are obtaining. It should look like this:



3. With the stopcock closed, pierce the septum.
4. Open the stopcock and insert the needle into the bulb. While opening the stopcock, hold on to your syringe plunger. The bulb is under ~10 psi argon pressure. The argon pressure will eject the plunger from the syringe unless you hold it!
5. Draw argon into your syringe.
6. Remove the needle from the bulb and expel the contents of the syringe outside of the system.
7. Repeat steps 4-6 twice more. (You should sparge your syringe 3 times.)
8. Insert your needle into the bulb.
9. Draw up the desired amount of solvent, then draw some argon into the syringe. **DO NOT** expel any solvent back into the collection bulb.
10. Remove the needle back through the stopcock, close the stopcock, and *then* remove the needle from the septum.
11. Check to make sure the stopcock is closed and that the septum is not totally destroyed. If the septum needs to be replaced, replace it. The new septa are next to the solvent system.
12. Sign the logbook with the date, time, your name, the solvent and amount used, and whether you distilled fresh solvent.

Scenario 2: Distilling solvent into an empty collection bulb

First you will evacuate and purge the collection bulb with argon **THREE** times.

1. Check to see if there is **ANY** solvent in the bulb, if there is, see the “draining solvent” section.
2. Flip the manifold valve so that it points to “vacuum”. The valve plate will look like this:



1. Watch the vacuum gauge on the top right of the system frame. When it reaches 20-25 torr, turn the manifold valve back to argon.
2. Repeat steps 1 and 2 **TWO** more times.
3. Repeat step 1, leaving the flask under vacuum by closing the choke valve after the vacuum gauge reads <25 torr.
4. *Check that the choke valve is closed* and then flip the manifold valve so that it is pointing at argon (see picture below).

The collection flask is now dry and degassed. The valve plate will look like this:



5. Hold the collection bulb and **SLOWLY** open the solvent valve. (Note: The solvent comes out **FAST**) Dispense only as much as you need. Be aware that it is hard to dispense less than ~10 mL.
6. Once you have dispensed your solvent, close the solvent valve and then open the choke valve. This will apply argon to the collection bulb.
7. See Scenario 1 for instructions on getting solvent from the bulb with a syringe.
8. Sign the logbook with the date, time, your name, the solvent and amount used, and whether you distilled fresh solvent.

Draining Solvent

If there is <3 mL left in the bulb but you need more solvent, you should empty out the bulb, dry it, and then use scenario 3 to distill fresh solvent. Here is how you remove the residual solvent so that you don't get solvent in the vacuum line.

1. Check that the bulb is open to the argon manifold. The valve plate should look like this:



2. Place the supplied solvent waste bottle underneath the drain stopcock.
3. Slowly unscrew the drain stopcock on the bottom of the flask. The residual solvent will be expelled with the argon in the bulb with some force, so be prepared.
4. When all the solvent has been removed, let the argon run for a little bit to push out any remaining drops of solvent. In the case of volatile solvents, this will also dry the bulb.
5. Close the drain stopcock by gently screwing it in. *Warning! If you apply too much pressure or tighten too much you will crack the collection bulb.*

The valve plate should now look like the picture above. If it does, proceed to scenario 3 to evacuate the collection bulb and distill fresh solvent.

Scenario 3: Obtaining MORE solvent than is already in the bulb (ghetto distillation)

For example, use this procedure if you need 50 mL of solvent and there is only 20 mL in the collection bulb. If there is <3 mL left in the bulb, drain the bulb and use scenario 2.

1. The bulb and the solvent kegs are at the same static pressure. This means you need to release some pressure from the bulb in order to push solvent through the columns.
2. Seal the collection bulb by closing the choke valve. The valve panel should now look like this:



3. Check the septum.
If it is old, just open the stopcock a little. You will hear a hissing sound. That is argon escaping from the bulb. When the hissing subsides, close the stopcock. You are no longer feeding gas into the bulb, so make sure you don't leave the stopcock open too long.
If the septum is new, open the stopcock and then loosen the septum cap slightly (i.e. a quarter turn). You will hear hissing. When the hissing subsides, tighten the cap and close the stopcock.
4. Hold the collection bulb and slowly open the solvent valve so that it is pointing to solvent. A large amount of ether will shoot into the bulb. The valve plate will look like this:



5. When you have as much solvent as you need, close the middle valve so that it looks like the picture at the top (all valves pointing left).
6. Follow scenario 1 to get the solvent from the collection bulb into your syringe.
7. Sign the logbook with the date, time, your name, the solvent and amount used, and whether you distilled fresh solvent.