How to use Solvent Purification System
A brief manual

By V. Azev.

SPS (Figure 1) can be used to collect dry oxygen free solvents into two different types of containers. One of them is a syringe. Syringes are filled with a solvent in a usual manner using a septum port on the flask (Figure 2). The second type of container is a special flask equipped with a high vacuum valve (Figure 3). These flasks are used by a small population of the department, so their usage will not be discussed in this manual.

1. Sign in the log book. Data, name, solvent and volume taken are required fields.
2. Check out the pressure on the pressure gauge on the Argon tank. If the pressure is around zero then the system will not work at all.
3. Well, now you have to decide, would you trust the purity of the solvent, which remains in the flask, or you will have to use just the fresh one. It is quite hard to measure the desired amount of a solvent. Usually people collect an excess of it. And some of it remains in the flask unused. Solvent is kept under positive argon pressure, so it should remain quite dry and anaerobic. If your reaction is not so sensitive to these contaminants, use the solvent which remains in the flask and go to 5 (if you want to collect more) or 9.
4. In the case when you want a highly pure solvent, remove that remains. First of all, find a bottle with an appropriate solvent label, where you could pour the remaining material. Some of these bottles are in the SPS hoods and some are under the hood which is in the center of d’Alarcao lab. After you have found a bottle, open valve #7. ATTENTION You should turn it so that an arrow would point toward the flask. Then, carefully open valve #8  **HALFWAY WITH AN ARROW POINTING UP**. If you open it with an arrow down, the system will be connected to the air – the source of junk like water and oxygen (at least). If you would turn it ALL way, the huge 10 psi pressure of argon will flash out all the silicon oil from the bubbler. It will be a mess, which would have to be cleaned by you. The worst part of this mess is that the solvent could be contaminated with this ugly silicon oil. And as a consequence your product too. So, please, turn it approximately half way. The argon should flow through the
bubbler quite rapidly (~ 3-4 bubbles per second), but not crazy rapidly. Now put the bottle for the solvent under the flask outlet and open \textit{valve} \# 10 slowly (half of the turn). Allow the solvent to pour inside the bottle. As far as the system is under positive Argon pressure and the valve is open slightly, not so much air enter the system. After the flask has been emptied, close \textit{valve} \# 10 and \textit{valve} \# 8. Now we are ready to fill the flask with a fresh solvent.

5. Open \textit{valve} \# 1. It creates the driving force to transfer a solvent from a storage tank into a flask. Also, open \textit{valve} \# 2 (ARROW UP), \textit{valve} \# 3 (one way valve), \textit{valve} \# 4 (ARROW UP), \textit{valve} \# 5 (one way valve). There are two options at this point. \textit{Valve} \# 7 may be opened (if You did empty the flask) or not. If not – turn it with an arrow toward the flask!!!!

6. Slowly open metering \textit{valve} \# 6. You should be able to see, how the liquid flows. Try to take as precise as possible. Actually, collect approximately 10 mL less, then necessary.

7. Now, completely close \textit{valve} \# 6. Then, \textbf{carefully open \textit{valve} \# 8 \textbf{HALFWAY WITH AN ARROW POINTING UP}.} If you open it with an arrow down, the system will be connected to the air – the source of junk like water and oxygen (at least). If you would turn it ALL way, the huge 10 psi pressure of argon will flash out all the silicon oil from the bubbler with the outcomes described at 4. Solvent stuck in the tubing will come into the flask.

8. Close \textit{valves} \# 8, 7, 5, 4, 3, 2, 1. You may collect the solvent with your syringe now.

9. Before inserting a syringe needle inside the flask it is advisable to increase the bubbling slightly. To do this open \textit{valve} \# 9 a little bit CCW. A flow-rate of 1 bubble per second will suffice for this step.

10. After you have taken the desired amount of solvent \textbf{bring the flow-rate back to around 1 bubble per 2-3 seconds}, just turn \textit{valve} \# 9 back (CW).

11. \textbf{WHEN YOU LEAVE, MAKE SHURE THAT THE BUBBLING IS NOT CRAZY.}

12. Feel absolutely free to ask Slava Azev (M 302, 7-20-42) if you have questions.
Appendix

Figure 1. General view of one of the SPS units.
Figure 2. Collecting solvent with a syringe.
Figure 3. Solvent storage flask