U N I V E R S I T Y *of* H O U S T O N
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Solvent Purification System Use Guidelines

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mBraun Solvent Purification System (MB-SPS) is located in 48 Fleming.

Introduction to the MB-SPS

MB-SPS is an instrument that allows removal of oxygen and moisture from up to 6 different organic solvents. It can be used as a stand-alone unit—as in our group—or it can be integrated into the glovebox. Our system is set to purify (1) tetrahydrofuran, (2) diethyl ether, (3) N,N-dimethylformamide, (4) toluene, (5) acetonitrile, and (6) hexanes. The location of all of these solvents are clearly labeled on the MB-SPS.

The solvents are purchased at anhydrous grade in 18 L Pure-Pac tanks from Aldrich, and are packaged under nitrogen. The tanks are then connected to the MB-SPS under nitrogen and they remain under low positive pressure of nitrogen throughout their operation. During dispensing, the solvent is forced—under low nitrogen pressure—from the tank through two gas-tight columns filled with anhydrous alumina as the dessicant. The solvent is collected in a Schlenk flask which can be evacuated and back-filled with nitrogen on the MB-SPS.

Routine use of MB-SPS is extremely simple, since the user only needs to operate the color-coded valves located on the front panel of the system. It is important to remember, though, that all operations must be performed slowly and gradually to avoid rapid pressure changes.

Dispensing the Solvent

Before you start, check the nitrogen pressure meter located on the right of the front panel; it should read 10 psi. If the pressure is different, correct it by operating the valve located on the righthand side of the system, above the vacuum pump, or directly on the nitrogen cylinder.

Step 1: Evacuation of the Collection Vessel

1. Make sure that the vacuum pump is running.

2. Place a clean oven-dried vessel, equipped with a 24/40 joint, under the solvent dispenser. Use a lab-jack and plastic clips to immobilize the vessel. Schlenk-style vessels are preferred.
3. Turn bottom valve to Evac/Refill and the top valve to Vacuum position. Leave the vessel for few minutes to evacuate. The bigger the vessel, the longer it will take to evacuate.
4. Refill the vessel with nitrogen by turning the top valve to Nitrogen position. Do it slowly to avoid pushing the vessel out.
5. Repeat the evacuation and refilling step one more time.
6. Evacuate it third time and then turn the top valve to its horizontal, neutral position.

Step 2: Dispensing the Solvent

7. To start the solvent flow into the vessel, turn the bottom valve to Dispense position. You can regulate the flowrate through this valve. After you collect the desired amount of the solvent, turn the bottom valve to the Evac/Refill position.

Step 3: Clearing the Line

8. Turn the top valve to Nitrogen position to clear the line of the solvent and refill the vessel with nitrogen.
9. When you are finished, the top valve should stay in Neutral position and the bottom one in Evac/Refill.

Changing the Solvent Tanks

Solvents which are used in MB-SPS are delivered by Aldrich as dry and degassed, so they do not need any extra purification before connecting to the system. All of the fittings should be tightened first just with fingers and then slightly with the wrench. Forcing too much can damage them.

1. Before disconnecting the empty tank from the system, close the liquid outlet valve B (make sure it is pointing up) and turn the bottom color-coded valve into the dispensing position. This will empty the line and the column from the solvent and avoid spilling the liquid out of the liquid transfer hose. Make sure that you collect the solvent as it is being dispensed, since you can easily end up with up to 1 L of it.
2. Disconnect the old tank by unscrewing the nitrogen and liquid transfer hoses. Plug them with safety Swagelok caps (stored in a small box in the MB-SPS solvent cabinet) to isolate the system from air and moisture.
3. Place a new tank close to the MB-SPS. Open the cover and remove the seal from valve B. Turn the liquid outlet valve B to its horizontal, neutral position.
4. Remove the cap from the liquid transfer hose. Liquid transfer hose is the front hose in the MB-SPS solvent storage cabinet.
5. Remove plug C from the liquid outlet port A.
6. Connect the liquid outlet port A.with the liquid transfer hose using double-sided fitting.

7. Remove the cap from the nitrogen transfer hose. This is the back hose in the MB-SPS solvent cabinet.
8. Remove plug D from the nitrogen inlet port A.

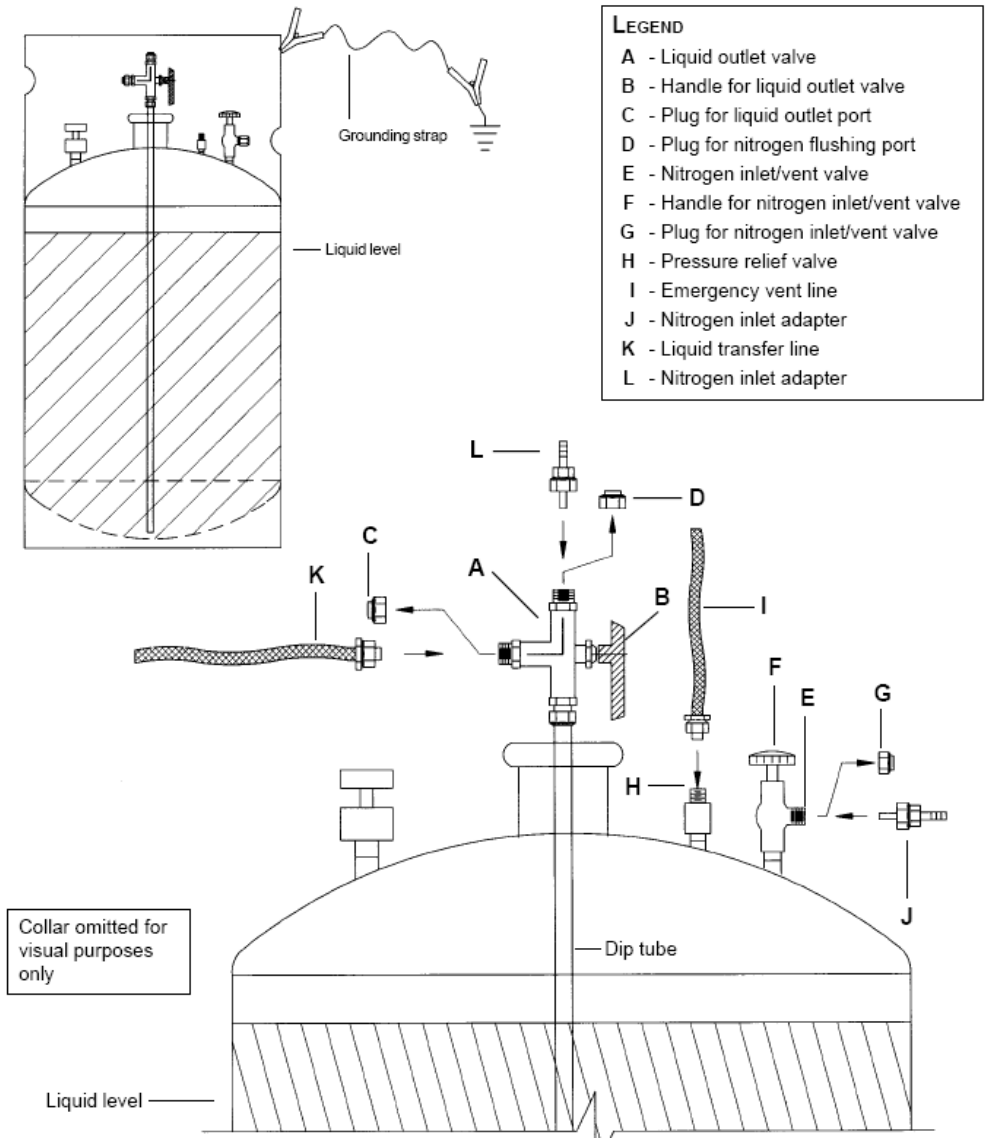

Figure. Schematic Connections to Aldrich Pure-Pac Solvent Tanks.

9. Connect the nitrogen transfer hose with the nitrogen inlet A.
10. Close the liquid outlet valve B (it should be in pointing up position).
11. Turn the bottom valve (operating when dispensing solvent) to Dispense position. The nitrogen stream should flow. Now the line and the column are purified from the oxygen and moisture which could have gotten there while you were disconnecting the system from the empty tank.

12. After one minute, turn the bottom valve (operating when dispensing solvent) and the liquid outlet valve B back to neutral position.
13. Disconnect the nitrogen transfer hose and replace it back with the plug.
14. Remove the plug G from nitrogen inlet valve E and connect it with the nitrogen transfer hose.
15. Turn on nitrogen inlet/vent valve E.
16. Place the tank into MB-SPS cabinet.
17. Dispense a portion of the solvent to fill up the line and the columns. It can take a little time before the solvent starts to flow.