PPT Solvent Purification System Manual

Built by Pure Process Technology, LLC

Pure Process Technology

Delivering Pure Solutions On Demand

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Important Notes

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Important Safety Information

The PPT Solvent Purification system is designed to safely purify and dry solvents. It is the user's responsibility to follow safe laboratory procedures and practices when operating the solvent system. If the system will be re-installed to another location the user must take care that the system will be located and installed for safe use. Be aware that there may be requirements for local building codes, guidelines and regulations that apply. We highly recommend reviewing with EH&S or facilities group for questions in this area. Contact us if you would like to review moving procedures or to provide a quote for relocating a system.

This instruction manual contains information that should be followed for safe and correct operation of the solvent purification system. All personnel that will operate this system should read and understand the correct operation as well as safety precautions covered in this manual before using the system. The system contains warning labels that should be observed for safety and proper operation.

User Precautions

Always take precaution by using the appropriate personal safety gear for handling solvents. This includes gloves, safety glasses, protective outerwear and any other requirements for your lab.
This equipment **must** be grounded to avoid a potential spark hazard due to static electricity.
Follow proper operating procedures, safety guidelines and solvent handling instructions when using this equipment.
This equipment is designed to purify solvents. Any misuse or improper operation may pose a danger or hazard to the operator and damage the equipment. The manufacturer will not be responsible for any issues caused by improper use of the system.

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I. Introduction

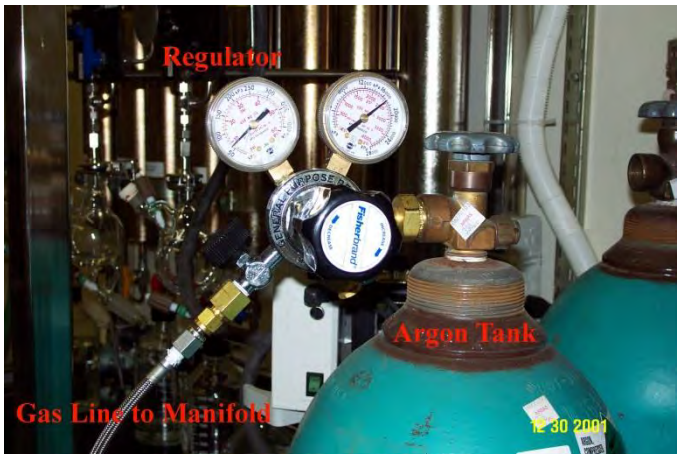
This manual gives a basic overview of the Solvent Drying System (SDS) to give the user a basic knowledge of the apparatus, and to explain some of its necessary operations. Since one or more group members are usually assigned to take care of the instrument, this manual is meant to assist individuals in the absence of the specified group member(s).

The SDS incorporates a high vacuum pump and an Argon supply, and can be used to deliver dried, degassed solvent under a blanket of Argon. The basics of its operation are as follows: (1) an oven-dried, solvent recovery flask is subjected to several vacuum "pump-down"/Argon "back-fill" cycles on the SDS, and finally left under vacuum. (2) The solvent is drawn through the drying columns by this static vacuum while being simultaneously pushed with a positive pressure of Argon out of the solvent kegs and collected in the recovery flask. (3) The flask is then either sealed and removed from the solvent system for personal use or the flask can remain attached to the SDS and be used by a group, in similar fashion to a collection bulb on a solvent still.

The following is an explanation of the different parts of the SDS, including the Argon supply, solvent kegs, columns, vacuum system, and solvent delivery controls. A short procedure has also been prepared that explains how to dispense the solvent. *Anyone using the SDS should be familiar with this procedure.*

II. Inert Gas Supply

The SDS has an ultra-high purity Argon supply to keep the solvent kegs, columns, and recovery flasks under an inert gas atmosphere. When the Argon tank is connected to the system, the regulator on the tank should be set between 8-12 psi.



(Note: some single stage gas regulators have a tendency to slowly increase in pressure over time.) Be sure to check the pressure on this gauge periodically. Do not exceed 15 psi or drop below 5 psi pressure. Please notify the SDS manager(s) immediately if the tank is nearing empty (at or below 500 psi) or if a leak is

suspected. Under normal operating conditions, an Argon tank should last for approximately 4-6 weeks.

Argon is introduced into the SDS system through an SST hose, which connects through a self-sealing Swagelok® Quick-Connect valve. After this valve, the Argon manifold splits it into separate Argon lines, one for each solvent. Argon flow to the lines is controlled through individual color-coded on/off Swagelok® valves. All of the valves controlling each solvent (on the Argon manifold, columns, and dispensing outlets) are uniquely color-coded (see picture). After the on/off valves, each Argon line is then split, with one line going to the solvent keg, and the other line going to the solvent dispensing outlet. Each of these lines is equipped with a one-way, check valve which prevents Argon back flow from the different solvents to mix and cross-contaminate each other. A pressure gauge is present on the SDS Argon manifold. The regulator on the Argon tank should be set so this gauge should read 8-12 psi.

Argon Manifold

Over Pressurization

In the event that the system does begin to over-pressurize with Argon uncontrollably, the stainless steel tubing is rated not to rupture up to high pressures (~800+ psi). The Quick-Connect valve on the Argon manifold allows the Argon tank to be disconnected, and pressure relief valves set at 30 psi are present on each solvent keg (covered with blue plastic caps). The pressure relief valves will reset automatically after the pressure drops below 30 psi.

III. Solvent Kegs

There are two control valves on each keg. The Argon inlet valve is green handled and the other is the solvent outlet (under normal working conditions). The solvent outlet valve is color-coded as to which solvent it corresponds. As stated above, the solvent kegs have a pressure relief valve (covered by a blue plastic cap) with a threshold of 30 psi. This ensures that the system does not become over-pressurized.

Refilling and Degassing Solvent Kegs:

When the volume of any keg has been reduced to **2** liters, new solvent should be added immediately. Please notify a group member in charge of the SDS at this time. When the volume of the keg has been reduced to approximately **2** liters, **STOP** using the keg, and notify the SDS manager (The SDS managers will take over at this point). Close the Argon inlet on the empty keg. Isolate the solvent columns from the keg under Argon by closing the solvent column inlet and outlet valves (colored handles). Finally, close the Argon inlet to the keg at the SDS Argon manifold. Disconnect the Argon line to the keg, and attach a Teflon line to the valve for pressure venting. Insert the opposite end of the line into a hood or exhaust line to contain the fumes. Slowly open the Argon valve to vent off the tank pressure. When the pressure is reduced to atmosphere, shut both the Argon/Vent valve and the solvent valve on the keg. Disconnect the solvent line from the valve quickly sealing it with a small SS cap provided to prevent air from entering the line. (Make SURE the solvent column inlet valve is closed AND the SS cap is placed at the end of the solvent line to prevent draining all of the solvent out of the column, introducing air into the column, and drying out the solvent packing!!).

The solvent keg is now ready for refilling, either by pouring more solvent into the keg, or vacuum transfer of solvent into the keg. In either case, the keg needs to be grounded before

transfer of solvent takes place. This can be done by attaching the clamp on the end of the system grounding line to the handle area of the keg.

If the pour method is being used, we recommend filling be done in a vented area or hood. Loosen and remove the clamp holding the valve assembly in place, and remove the valve stem assembly. Insert a funnel into the keg opening, and fill the keg to within 4 inches from the bottom of the opening. This will require about 16 liters of solvent. The open space at the top of the keg is needed for the degassing process. Replace the valve stem assembly, and tighten the clamp. See the degassing section that follows.

Vacuum transfer of solvent can be done using the vacuum transfer flange, or through the normal keg valve stem assembly. Using the valve stem assembly method requires one to have a good idea of how much solvent is remaining in the keg so the keg is not over filled. Again, it is important that the keg be grounded during transfer. If using the vacuum transfer flange, loosen and remove the clamp and the valve stem assembly. Place the Teflon gasket and vacuum flange on top of the keg opening and clamp in place. Attach a ¼" line from the fitting marked "Vacuum" on the flange to the three-way valve on the vacuum manifold. Attach a Teflon line from the fitting on the flange marked "Solvent" (this has the short length of SS tubing attached on the bottom side) long enough to reach to the bottom of the full solvent container to use as a dip tube. Turning the valve handle on the three-way vacuum manifold valve to the tubing position will then create a vacuum in the keg, and draw the solvent from the full container into the keg. You can remove the flange on a periodic basis to check the volume of solvent in the keg. Fill the keg to within 4" of the bottom keg opening as above. Return the vacuum manifold valve to the manifold position and remove the tubing. Remove the vacuum fill flange from the keg and re-install the valve stem assembly, securing tightly with the flange clamp. See the degassing section that follows.

Refilling the keg using the standard valve stem assembly is similar to filling using the vacuum flange. The advantage is the seal on the keg does not have to be opened, and the Argon valve on the keg can be used as a vacuum control valve. The disadvantage is you need to be aware of how much solvent is remaining in the keg, and how much you can add without over filling and sucking solvent into the vacuum manifold. It is better to under fill than over fill when using either vacuum transfer method. When using the standard valve stem assembly, attach the Teflon solvent fill line to the solvent valve and open the valve. Attach a line from the vacuum

manifold valve to the Argon valve on the keg stem assembly. With the Argon valve on the keg stem closed, turn the valve handle on the three-way vacuum manifold valve to the tubing position. You can now use the Argon valve on the stem assembly to turn the vacuum to the keg on and off. When transferring the solvent dip tube from bottle to bottle during the fill process, it is best to shut the vacuum to the keg off so you don't suck air into your solvent. This also cuts down on the amount of solvent fumes that transfer to the manifold and pump**. It is important not to overfill the keg**. If you see solvent in the vacuum line, shut off the Argon valve, and return the three-way vacuum manifold valve to the manifold position immediately. Again, you want to leave a space above the solvent in the keg for the degas procedure. When the keg is at the required fill level, shut the Argon/vacuum valve and the solvent valve on the tank. Return the three-way vacuum manifold valve to the manifold position. Disconnect the solvent fill line and the vacuum line at the keg and the vacuum manifold.

Degassing the solvent keg:

If prepackaged solvents are being used, the solvent has **NOT** been degassed when they arrive, and the column packing **DOES NOT** adequately degas the solvent. Therefore, before a fresh keg of solvent is attached to the SDS, it is important to purge the solvent with Argon in order to degas it. To do this, the Argon line is attached to the closed *solvent* outlet on the keg. An additional Teflon purge line is then attached to the original, closed *Argon* inlet and vented into a hood adjacent to the SDS, or a nearby exhaust line. This additional Teflon purge line should be stored in the vicinity of the SDS. (NOTE: the directionality of the Argon inlet and solvent outlet have been reversed - Argon will be introduced through the solvent outlet valve, and exit out of the Argon inlet valve.) This method takes advantage of the long "dip tube" in the keg to which the solvent outlet is attached, allowing Argon to bubble through the entire volume of solvent in the keg. Under normal operation, this "dip tube" facilitates efficient delivery of solvent from the keg to the columns.

The pressure of Argon should be approximately 8-12 psi. The Argon supply is turned on at the Argon manifold, and both the inlet and outlet valves on the keg are opened, and the solvent is purged for 30 minutes. (The Argon passing down through the "dip tube" and bubbling up through the solvent can be felt physically through the keg). Then, the outlet valve (Argon) is closed, pressure is allowed to build in the keg, and then the inlet valve (Solvent) is closed. The

solvent is allowed to equilibrate for 5 minutes. This purging procedure is then repeated one more time for 15 minutes by opening the inlet valve (Solvent) first, then the outlet (Argon) valve. Once the second purge is complete, the outlet valve (Argon) is shut allowing pressure to build, and then the inlet (Solvent) valve is closed. The vent line is disconnected from the Argon valve and the Argon inlet line is reattached to its original position (a low Argon flow through the line should be maintained on to purge the line). The SS plug is removed from the stainless flex tubing and the line is quickly reattached to the solvent valve outlet. The Argon valve is then opened, followed by the solvent valve returning them to the standard operating positions. The color-coded solvent valves on the bottom of the back column and top of the front column are now returned their open positions.

This procedure is also required of groups refilling their own kegs with bulk solvent (bottles, drums, etc.). Never use half empty bottles of suspicious origin or age and ONLY use recommended solvent grades.

Keg Solvent Levels

There are basically two ways to keep track of the solvent levels in the kegs. One is to keep a logbook of how much of each solvent is used. The other is to keep track of the weight of the keg. We recommend that you use both methods. An empty keg assembly weighs approx. 16 lbs. Use an inexpensive bathroom scale to weigh a full keg, subtract the keg weight, and you will be able to calculate your usage, and solvent remaining. This is important to know when calculating the amount of solvent to add using the vacuum fill method.

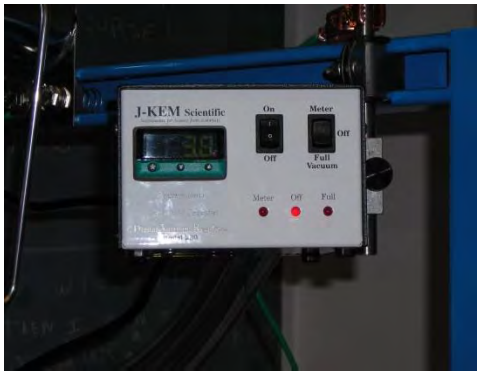
IV. Columns

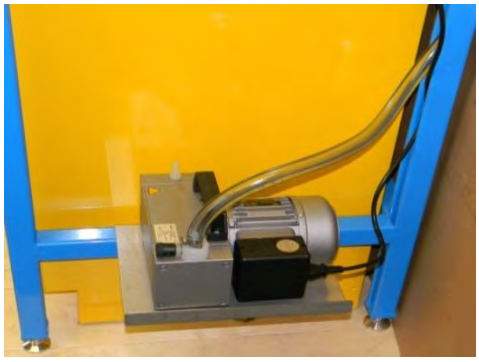
The columns used in the SDS are intended to vigorously dry the solvents. All solvents pass through two packed activated stainless steel columns. In the case of DMF, NMP, Triethylamine, and Isoproplyamine, a third column with Isosyanate scrubbing removes free amines and water not removed by the two standard columns. All of the solvents are passed through an in-line, 2 micron filter immediately before being dispensed. The 2 micron filter ensures that no packing material residue is dispensed with the solvent. The filters used by the SDS are Swagelok® FW series Tee-type sintered filters. Over time, these filters can become clogged with particulate and need to be cleaned or replaced. Clogged filters can be sonicated to

loosen the filtered material. Sonicate in the appropriate solvent and blow dry against direction of arrow using 20-30 psi of pressure.

IMPORTANT: The solvent column inlet valve should be turned off when a solvent is not going to be used for a while (1 week and longer). In the case of **DCM** and **Pentane,** the solvent column inlet valve should be turned off if the solvent will not been used for 2-3 days. This avoids the chance of back siphoning the solvent from the column into the solvent keg. If this happens you may need to re-prime the columns. In this case request column start-up directions.

V. Vacuum System

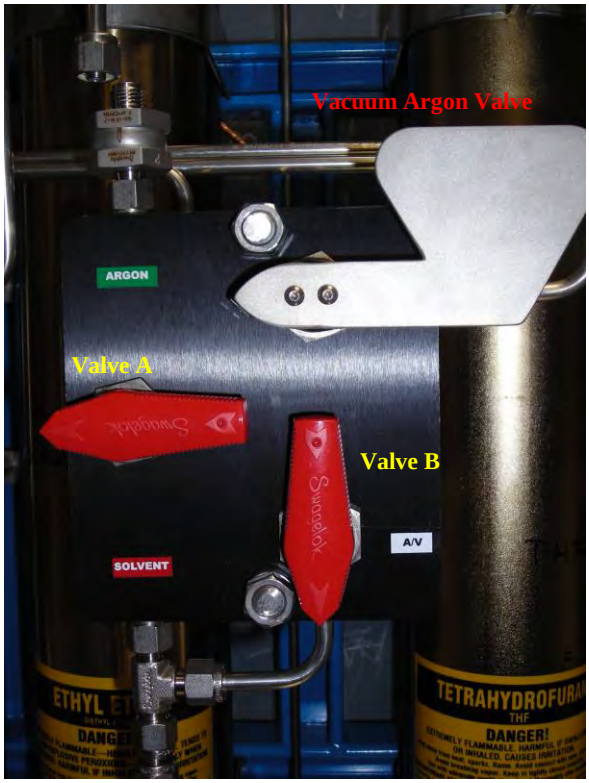




The vacuum system of the SDS is a crucial component to proper operation. It is important that the vacuum is running efficiently. A traditional vacuum system consists of the vacuum pump, solvent trap, and the vacuum manifold. However, our system operates using an oil-free diaphragm pump with or without a controller (see picture). The oil-free diaphragm pump is designed to handle solvents and solvent vapor without any effect, except for DMF, which with its very high vapor pressure, must be dispensed carefully so as not to get into the pump. In the event of a mishap, consult the pump manual for instructions on cleaning. Oil free diaphragm pumps normally pumps down to 5-10 Torr. The manual pump should be turned on when the system is in use. The pump with the controller automatically turns the pump on and off to maintain a preset vacuum level, which can be set by the user. Although the base pressure of diaphragm pumps is higher than the traditional oil pump with a LN2 solvent trap, they are much safer, and there is much less maintenance. By using the dilution principle of cycling between backfilling with Argon, and vacuum pump down of the recovery vessel, residual moisture in the vessel is effectively eliminated. Glass Contour Solvent Systems built by Pure Process Technology will only be supplied with Teflon diaphragm type pumps.

VI. Control Valves

The solvent delivery control panel consists of a vacuum/argon valve, a vacuum/argon on/off valve (valve B), and a solvent delivery valve (valve A). Valve B controls the application of vacuum or Argon pressure into the solvent recovery vessel, which is dictated by the Vac/Ar valve. The Vac/Ar valve is switched between the Argon supply, to the left, and the vacuum line, to the right. Under standby conditions with the recovery vessel in place, the Vac/Ar valve is left in the Argon position. Valve A is closed and Valve B is open so an Argon blanket is maintained on the solvent in the vessel.



NOTE: Never have valve A and B open at the same time when the Vac/Ar valve is set to the vacuum!! This will cause the solvent to be sucked out of the column, into the vacuum manifold, and eventually into the pump. This is the most important warning to note! The metal plate installed on the Vac/Ar valve is designed to prevent this happening when the Vac/Ar valve is switched to vacuum. This plate covers Valve A and inhibits its turning. Remember, always draw solvent into the recovery flask using STATIC vacuum, not DYNAMIC vacuum. **NOTE:** A dispensing valve configuration sheet is available from PPT if you would like to post it with the system.

VII. Dispensing Solvents

This section describes the general procedure for dispensing solvents. It highlights some precautions to take before using the SDS. Everyone that uses the SDS should understand this section completely. If you still do not feel comfortable using the system, *ask someone to help you.*

CHECK SYSTEM STATUS*: before collecting solvents, check the following system settings.*

1) The vacuum pump is operating and maintaining adequate vacuum. Check that the vacuum gauge reads at or around 10-12 Torr.
2) The Argon tank has sufficient Argon for use, and the Argon is flowing.
   -Check tank regulator and SDS pressure gauge (should read 8-12 psi)
3) Check column and solvent keg valves.
   -*All valves* should be open to the solvent keg and on the solvent column from which you wish to dispense (Argon inlet valve on Argon manifold, keg Argon inlet valve, keg solvent outlet valve, and the 4 valves on the solvent column).
   All *should be left open for routine use.*

Identification of valves:

Vac/Ar Valve: Used to switch between the vacuum manifold and the Argon manifold. This is an on/off Swagelok® valve.

Valve A: Used for the addition of solvent into the receiving flask. This is a color-coded on/off Swagelok® valve.

Valve B: Used to apply the vacuum or Argon pressure, as dictated by the Vac/Ar valve. This is a color-coded on/off Swagelok® valve.

Precautions to take:

Never open valves A and B when the vacuum is open.

Never use recovery flasks with star cracks or other defects!

Steps for solvent delivery:

Valve Position I

1) The solvent delivery outlet should be maintained with valve A closed, valve B closed and the Vac/Ar valve set to the Vac position (See Valve Position I). **IMPORTANT**: Inspect your recovery flask and make sure it has no star-cracks or defects that could cause the flask to implode/explode when placed under vacuum/Argon pressure.

Valve Position II

2) Clamp on the appropriate *clean, oven-dried*, receiving flask, securing the flask to the adapter with a Thompson clamp. Make sure that the Thomas clamp between the adapter and the outlet is NOT tightened ALL the way. This loose connection ensures there is an OUT upon back-filling with Argon. Open valve B to apply vacuum to the flask (See Valve Position II).

3) The receiving flask now needs to be evacuated with vacuum and back-filled with Argon several times. To achieve this, an adequate vacuum must be maintained on the flask (approx. 10-25 Torr). Then the Vac/Ar valve is turned to Argon while valve B remains open to fill the flask with Argon (See Valve Position III).

Valve Position III

Valve Position IV

Next, the Vac/Ar valve is returned to the vacuum position and an adequate vacuum is pulled (approx. 10-25 Torr) (See Valve Position IV). Repeat this pump-down/back-fill procedure 5 times. IMPORTANT: If you are filling a standard SDS solvent bulb, quickly open and close the vacuum stopcock to the 14/20 female joint (covered with a septum) in the middle of the Argon back-fill cycles to purge this port**. DO NOT** pump down on the inlet, as the septa are not airtight!! Leave this port **under Argon** at the end, and close the stopcock. **End with the recovery flask under vacuum.**

4) Now the flask is ready to receive solvent. With the Vac/Ar valve set to vacuum, valve B is closed (See Valve Position V). This leaves the recovery flask under STATIC vacuum. Then, the Vac/Ar valve is turned to Argon (See Valve Position VI).

Valve Position V

Valve Position VI

5) To dispense the solvent into the receiving flask, open valve A (See Valve Position VII). If solvent DOES NOT flow or only dribbles out, STOP, and close valve A. Check the valves on the column, keg, and Argon line, and make sure that they are all open. Make sure valve B is closed and the Vac/Ar valve is set to Argon.

Valve Position VII

If solvent is still not dispensed, consult the SDS manager(s). **Note: Never** open valve A while valve B is open and the Vac/Ar valve is set to vacuum! This will suck solvent directly out of the column and into the vacuum manifold and pump.

6) When you are finished dispensing the solvent, close valve A (See Valve Position VI). HOLD ONTO THE RECEIVING FLASK and carefully open valve B (See Valve Position VIII). The Argon will blow the solvent remaining in the line into your flask and fill the flask with a blanket of Argon (8-12 psi). Next, tighten the Thompson clamp to the adapter to prevent entry of air into the flask and the escape of Argon.

Valve Position VI

Valve Position VIII

7) To add more solvent to a bulb that has solvent in it and has been maintained under Argon pressure, close valve B. Loosen the septa nut, and open the vacuum stopcock to release the argon pressure in the bulb. Open valve A to add desired amount, and close. Reopen valve B, close the vacuum stopcock, and tighten the septa nut.

Routine use of solvent bulbs:

Solvent can be removed from the SDS solvent bulbs using the analogous procedure we use to remove solvent from still solvent bulbs. Here are some guidelines and instructions:

1) Make sure that the volume of solvent in the bulb will meet your needs. If not, dispense more solvent into a clean, oven-dried solvent bulb (see guidelines above). Clean bulbs and adapters can be stored in an oven near the SDS. Use clean stopcocks and new septa. **New**

septa (National Scientific P/N C4015-40) can be purchased through VWR (Cat # 66064-310) or Fisher (Cat # 03-377-107)

2) Always use a clean, oven-dried syringe needle.
3) The Teflon stopcock should be open already to allow the Nitrogen line cannula into the bulb. Insert the needle through the septum and into the inlet.
4) Purge the needle with the Argon/Nitrogen gas in the solvent bulb (3-4 times). HOLDING onto the solvent bulb, push the needle through the stopcock opening and into the bulb. Open the stopcock further if required to get your needle into the bulb. Remove the volume of solvent you want. DO NOT inject any solvent back into the bulb once you have removed it.
5) HOLDING the bulb, remove the needle from the bulb. Readjust the stopcock if you opened it further.
6) Larger volumes of solvent can also be removed using the additional outlet at the bottom of bulbs. Simply open the Teflon stopcock and dispense solvent. Please wipe up any spills you might have in the delivery process.
7) Out of courtesy for your co-workers, if you empty a bulb of a frequently used solvent, *please clean the used bulb, return it to the oven adjacent to the SDS, and refill a fresh solvent bulb*. Don't just leave the empty bulb on the SDS for someone else to clean and refill!

VIII. Trouble Shooting

This section is a growing section where we have included information about common problems associated with the daily use of the SDS. It will be as complete as possible. We will consult other labs with the same system for additional potential problems and solutions. If you would like to add items to this list, please let the SDS managers know or contact us. We suggest you keep a trouble-shooting notebook near the SDS for group members to record problems, and solutions, they have had with the instrument. *Please* make use of this notebook to maintain an up-to-date operational history of the system.

1) Maintain the Argon regulator and service the vacuum pump, if using a conventional pump. Most importantly, change the pump oil regularly.
2) Use the proper grade of solvent in the solvent kegs. **Stabilized THF solvent should NEVER** be used because the column packing material does not adequately remove the

stabilizer. **DCM** can be used with cyclohexane as a stabilizer but unstabilized is best. **Do not use DCM stabilized with Methanol or Amylene.** Contact us with any questions or to get a recommended solvent list.

3) If solvent only dribbles out or doesn't flow, and all the valves are open and the kegs are full, it is likely the filters need cleaning or replacement. DCM may have back siphoned because of its weight and low argon pressure.

4) Your system comes with a heavy duty Ilmvac brand vacuum pump. If your pump vacuum degrades, read the instruction manual for the proper remedy. The pump may need to be cleaned by spraying acetone into intake port and allowing pump to pump itself dry.

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