Pure Process Technology

Delivering Pure Solutions On Demand

Solvent Purification System

Installation Guide

This guide is to assist in the set-up and installation of a PPT Solvent Purification System

Solvent Purification Installation Guide

Important Notes

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Important Safety Information

The Glass Contour Solvent Purification system is designed to safely purify solvents. It is the user's responsibility to follow safe laboratory procedures and practices during the installation and operation of the solvent purification system. The user must also 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 this document with your EH&S or facilities group for questions in this area.

This installation manual along with the 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 these manuals 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 and protective outerwear as well 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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System Setup Overview:

The following outline is a guide to the steps you will take when setting up your new Solvent Purification System (SPS)
It is not meant as a substitute to reading through and understanding the entire installation text.

- Uncrate and place the Solvent Purification System into its location and level the SPS
- Place the solvent storage cabinet under the frame. Level the storage cabinet
- Attach grounding wires from the system to a nearby "**cold**" water pipe or a nearby Electrical Ground Terminal (contact your facilities department).
- Set up the system vacuum pump and controller
- Connect Argon hoses and solvent hoses between solvent kegs and purification columns
- Fill and degas solvent kegs
- Purge solvent purification columns ,hoses and piping
- Ready solvent collection vessels (flasks) for service

TOOLS NEEDED (Not Supplied)

· (1) ¾" WRENCH
· (1) 9/16" WRENCH
· (1) ADJUSTABLE WRENCH
· (1) FLAT BLADE SCREWDRIVER
· (1) TORPEDO LEVEL
· (1) ½" WRENCH
· (1) 5/8" WRENCH

MATERIALS (Not Supplied)

· SOLVENT (See preferred solvent list)
· ARGON or NITROGEN
· GLASSWARE (optionally purchased)
· NITRILE GLOVES
· SAFETY GLASSES
· GAS REGULATOR

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Introduction:

This manual provides a detailed overview for installation and set-up of your Solvent Purification System (SPS) and to give the installer / user a basic knowledge of the apparatus, explaining some of its necessary operations. What follows is an explanation of the different parts of the SPS, 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 SPS should be familiar with this procedure. Additional operating information is provided in the system instruction manual.*

Your SPS should arrive requiring only a small amount of set-up and assembly. Your system will arrive crated complete with the following items. Glassware if requested will ship separately to prevent damage from occurring.

1.) Frame assembly with columns, valve assemblies, vacuum manifold, argon manifold and spill tray, pump shelf, grounding wire and clamps. Solvent cabinet with empty, pressurized, solvent kegs.
2.) Hose and Tubing kit, (4) hoses per solvent, (1) 25' length Teflon tubing, (1) 4' length of SS covered Argon hose with connections. (1) ¼" SS male connector , (1) ¼" SS female connector, nuts and ferrules, (2) union connectors.
3.) Start-up kit, rubber stopper, polypropylene tubing, 3/8" x ¼" Coupling
4.) Vacuum System, (1) pump, (1) controller (if purchased), hose with clamps, controller mtg. hardware, power cords.
5.) Spare parts and supplies, ®Simriz O-rings for ball adapters, ®Krytox grease and SS plugs for hoses.

Items shown:

Vacuum Pump mounting shelf
Vacuum Hose
Polypropylene Tubing
Teflon Tubing

Items shown:

Installation Manual
Installation Manual (on disc)
Start-up kit w \ rubber stopper
SS Connectors male & female
Nuts, Ferrules
3/8" x ¼ coupling
Hose clamps
SS hose plugs
Simriz o-rings
® Krytox grease

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System Placement and Leveling:

- Locate where the system is to be placed for operation.

Allow no less than 3” clearance behind the system to allow for solvent hose connections to be made between the solvent cabinet and columns.

- Adjustable legs are installed on the frame. The legs can be adjusted using a ¾” wrench. Place a level on to the horizontal frame members between the legs and adjust the foot height until the system is level. Once the system is level, tighten the foot locking nut securely against the frame to prevent movement.
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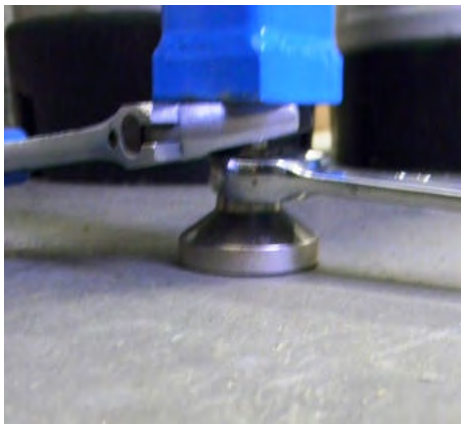
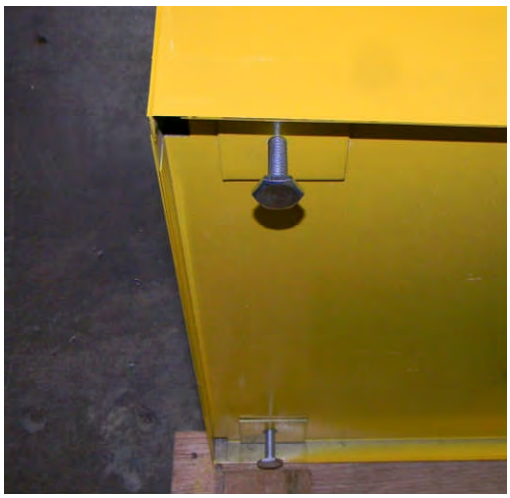


- Slide the solvent cabinet under the left side of the system. (Right side if the system has the Argon manifold on the right)



- Test the cabinet for level and rocking.
- Adjust the feet under each corner of the solvent cabinet by pulling it out, tilting it back or forward, adjusting the foot length and retesting for level. When properly installed, there should be a ⅛” – ¼” gap between the solvent cabinet and floor.

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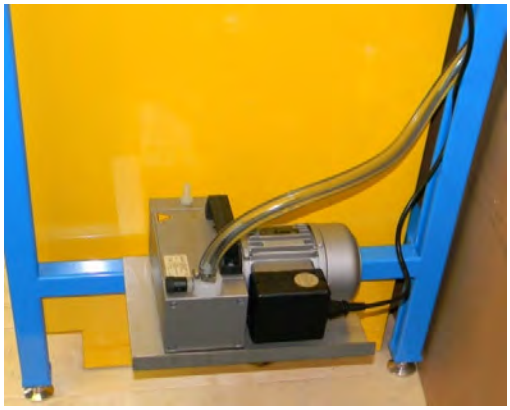
System Grounding:

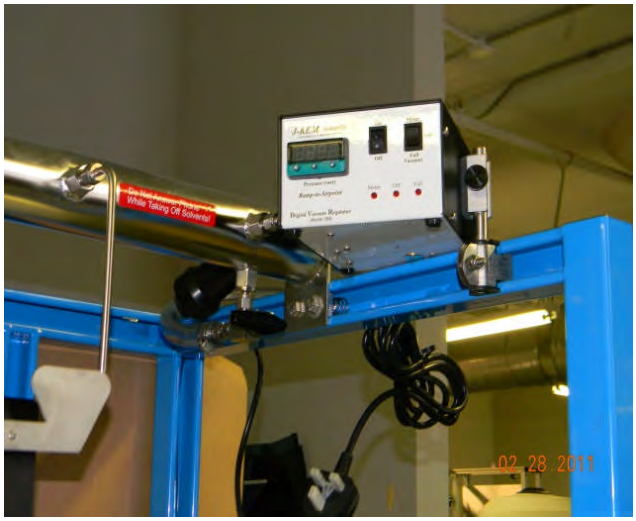
- It is imperative that your system be connected to a good earth ground. This is necessary to prevent any static electrical discharge when using the system. A separate green grounding wire with a copper clamp must be attached to the handle of any keg when filling them or de-gassing solvents. A heavy copper ground wire and green clamp wire must be attached to the system at one of the bolt points for the vacuum manifold. A pipe clamp has also been provided for mounting onto a nearby COLD water pipe for the attachment of the heavy copper wire. If not cold water pipe is available contact your facilities department for other grounding options.



Vacuum Pump and Controller Setup:

- Vacuum pump shelves are normally attached to the inside of the frame of the system on the right hand lower support rail. If there is insufficient room because of the solvent cabinet size, then it can be mounted from the inside of the rail to the outside. Remove the spring nuts from the bolts in the shelf, and insert them into the rail. Insert the bolts through the shelf holes and tighten them into the nuts with a 9/16" wrench.

- The vacuum controller is mounted to the rod extending from the upper right hand side of the system. Loosen the bolt on the clamp on the right side of the controller and slide it down over the rod.
- The clear heavy duty vacuum tubing should be run up from the vacuum pump inlet through the U channel at the shelf level and up to the controller inlet.
- Attach the hose to the pump, controller, and vacuum manifold using the clamps provided.



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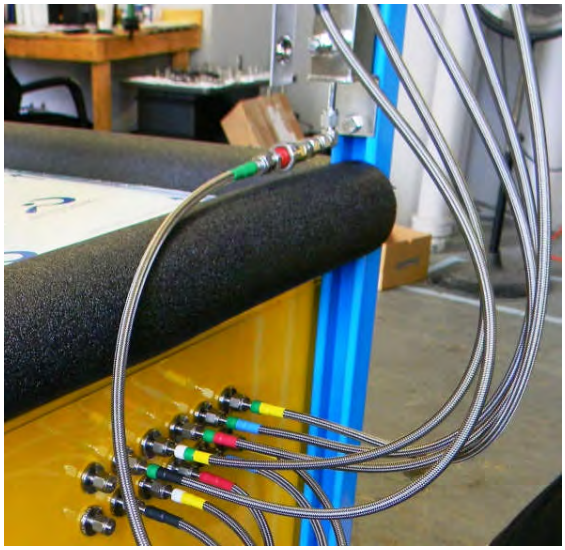

Hose Connections:

- All hoses are color coded for a specific solvent, or green banded in the case of the Argon Regulator to Argon manifold hookup hose. The Argon hookup hose is also equipped with a self-sealing quick connect for connection at the Argon manifold. Both a male and female ¼" NPT to Swagelok fitting have been provided for attachment of the Argon supply hose to the pressure regulator. Only one of these fittings will be needed depending on how your regulator outlet is configured.

- All hoses are color coded with bands located at each end. A "Green" band is always used for hoses carrying Argon, and used alone or in combination with another colored band representing a particular solvent. There are four hoses provided for each solvent. Two for each solvent keg located inside the solvent cabinet and two for use outside the solvent cabinet for attachment to the Argon manifold and solvent columns.
- Solvent hoses will only be banded with the solvent color. All hoses should come attached to the bulkhead fittings on the outside of the solvent cabinet.
- Attach the shorter Argon solvent hose on the outside of the cabinet to the check valve of the corresponding solvent on the Argon manifold using a 5/8" wrench on the check valve and a 9/16" wrench on the hose fitting. Securely tighten the hose nut, but do not over tighten the nut.

- Connect the longer solvent hose to the valve on the bottom of the corresponding back solvent column. Use an adjustable wrench to hold the valve and a 9/16" wrench to secure the hose nut. Again, tighten securely, but do not over tighten the hose nut.
- When all of the hoses have been attached to both the Argon manifold and the solvent columns, your solvent kegs will then need to be filled and de-gassed next.

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Inert Gas Supply:

The SPS requires ultra-high purity Argon or Nitrogen 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: A dual stage regulator is highly recommended, 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 SPS 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 8-12 weeks, depending on usage.

Argon Distribution:

Argon is introduced into the SPS system through a flexible 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 Swagelok® ball valves. All of the valves controlling each solvent (on the Argon manifold, columns, and dispensing outlets) are uniquely color-coded. After the Argon supply valves, each Argon line is then split, with one line going to the solvent keg, and the other line going to the solvent control plate. 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.

Over Pressurization:

The Quick- Connect valve on the Argon manifold allows the Argon tank to be disconnected and pressure relief valves set at 25 psi are installed on each solvent keg (covered with blue plastic caps.)

The pressure relief valves will reset automatically after the pressure drops below 20 psi.

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Solvent Kegs:

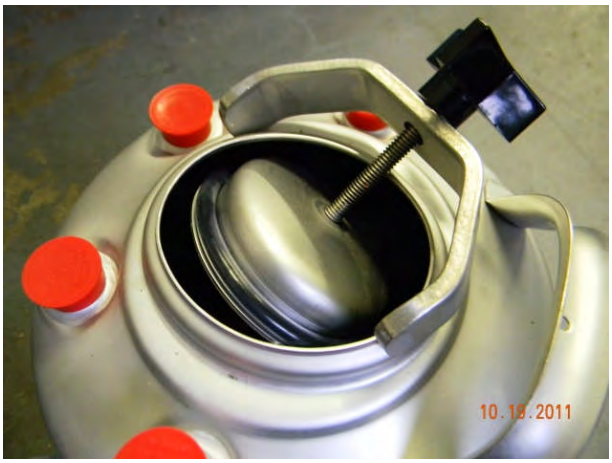
There are two control valves on each keg. The Argon inlet valve is green handled and the other is the solvent outlet (under normal working conditions). The solvent outlet valve is color-coded as to which solvent it corresponds. As stated above, the solvent kegs have a pressure relief valve (covered by a blue plastic cap) with a threshold of 25 psi. This ensures that the system does not become over-pressurized.

Note: Kegs are shipped under Argon pressure and must be vented before opening. Open the Argon valve on the keg to vent.

Caution!! Solvent kegs MUST be grounded before disconnecting, filling or de-gassing them.

Filling Solvent Kegs:

When filling kegs using the pour method, we recommend filling be done in a vented area or hood. Loosen the nut on the keg cap and remove the cap. Insert a funnel into the keg opening, and fill the keg to within 4 inches from the bottom of the opening. The open space at the top of the keg is needed for the degassing process. Replace the keg cap assembly, and tighten the cap nut. See the degassing section that follows.



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Degasing Solvent Kegs:

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 SPS, it is important to purge the solvent with Argon in order to degas it.

Caution!! Solvent kegs MUST be grounded before disconnecting, filling or de-gassing them.

- Attach an Argon supply to the valve on the keg labeled **SOLVENT**. Regulate the Argon supply to between 6 – 10 psi
- Attach a Teflon hose to the valve on the keg labeled **ARGON**, this hose will serve as a vent line during the process and should be connected to an exhaust line or ventilation hood.
- Open the keg valve labeled **SOLVENT**.
- Open the keg valve labeled **ARGON**.
- Argon should now be flowing through the keg **SOLVENT** valve and feeding down a long "dip tube" in the keg causing Argon to bubble through the entire volume of solvent.
- Allow the solvent to degas for 30 minutes.
- Close the vent (**ARGON**) valve, pressure will build in the keg, causing the bubbling to stop.
- Close the Argon supply (**SOLVENT**) valve.
- Allow the solvent to equilibrate for 5 minutes.
- Repeat the degassing process, this time for 15 minutes

Note: The Argon passing down through the "dip tube" and bubbling up through the solvent can be felt physically through the keg. A gentle purge is desired, not a violent keg shaking bubble rate, the bubble rate can be controlled by reducing the Argon flow. Repeat this process with each keg before proceeding with column start-up. Once the second purge is complete, the vent (**ARGON**) valve is shut allowing pressure to build, when the bubbling has stopped , then the Argon supply (**SOLVENT**) valve is closed. The vent and Argon supply lines can now be disconnected.

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Preparing Columns for use:

- Loosen the line to the valve on the bottom of the front column, slowly open the valve on the top of the back column and purge the line with Argon from the first column. Tighten the fitting while the gas is flowing and then shut the valve on the top of the first column. Make sure the fittings are Tight on both valves. Using both an adjustable & 9/16” wrench.
- Slightly open the valve on the bottom of the back column with the keg solvent supply line attached to it to purge this line with Argon from the column. Loosely attach it to the keg solvent valve with the Argon flowing and close the valve on the bottom of the first column. Tighten the line on the solvent valve with a 9/16” wrench and open the solvent supply valve on the keg.

- Open the valves on the top and bottom of the columns connected to the line between the two columns. This should equalize the pressure between the two columns.
- Slightly open the valve on the Argon manifold to purge the line that runs to the solvent keg and attach the Argon line to the keg while purging. Open the Argon manifold valve completely and the Argon supply valve on the keg.
- Attach a short section of Teflon hose to the end of the SS line that runs from the top of the second column to the filter on the control panel using a ¼” SS union. This hose should be long enough to run from the union down to the union atop of the rubber stopper. The stopper should be placed in a 1000ml Erlenmeyer flask on the spill tray with 1" of water in it. Attach a section of 3/8” polypropylene tubing to the other fitting on the stopper by pushing it in, then run it to a vent or exhaust hood.

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Preparing Columns for use: (continued)

- Slowly open the valve on the top of the front or second column about half way and observe the water in the flask for bubbles. Since the columns are shipped under 50-60 psi of Argon pressure, it is necessary to reduce this pressure to below the 25 psi set point of the solvent keg vent pressure relief valve before the solvent valve on the bottom of the first or back column is opened. Otherwise, the relief valve would open and the solvent fumes would be released into the cabinet. By adjusting the top valve position open or closed, you can see the bubble rate gradually decline and become less forceful. When it has slowed to a rate where you can see individual bubbles coming from the tube in the flask, fully open the solvent valve on the bottom of the first or the back column.
- Continue to monitor the bubble rate in the flask and adjust the valve on the top of the second or front column so you can almost see individual bubbles forming. The goal is to fill the columns with solvent at a constant, gentle rate and to purge the Argon out. Some solvent columns become warm when being filled. In some cases the filling process needs to be stopped to allow the column to cool to prevent solvent vapors from condensing in the flask exhaust line. Once the solvent starts to flow out of the column, purge about 500ml of it into the flask.
- Shut the valve on the top of the front column, disconnect the union and tube running into the flask from the SS line, and reconnect the SS line to the top of the final filter on the control panel**.**

Note:
See section below regarding optional Isocyanate Scrubber if ordered with solvent column set.
Caution:
The line will be filled with solvent. Ensure that all of the control panel valves are in the closed position and open the valve on the top of the column to the control panel. Your column should now be ready for use.

Isocyanate Scrubber:

Isocyanate scrubbers are installed after the columns are filled and flushed. The reason for this is to prevent any particulates from the columns entering the scrubber body. A second set of tubing is supplied with the scrubber.

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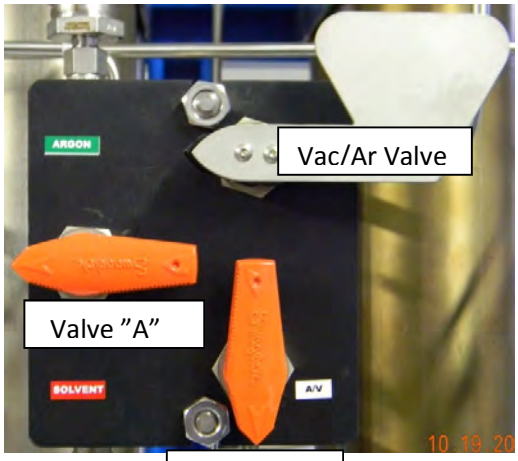
Isocyanate Scrubber: (***continued***)

Instead of re-connecting the tubing from the top of the second column to the final filter, remove it and install the scrubber with supplied tubing as pictured. After installation, open the valve on the top of the second column. The scrubber will need to be filled and saturated before solvent will flow from the control panel. Place or hold a beaker under the solvent outlet of the control panel. Open the solvent control valve on the panel for 5 seconds and then close it. Wait 10 seconds, and then reopen the control valve. Repeat this process until solvent flows from the control panel outlet. The column set is then ready for use just like the other columns.



Control Valves:

The solvent delivery control panel consists of

- A vacuum/argon valve shown set to "ARGON"
- A solvent delivery valve (valve A) Shown "CLOSED"
- An A/V on/off valve (valve B) Shown "OPEN"

Function of valves:

Vac/Ar Valve:	Used to switch between the vacuum manifold and the Argon manifold.
Valve A:	Used for the addition of solvent into the receiving flask.
Valve B:	Used to apply vacuum or Argon pressure to the recovery vessel, as dictated by the Vac/Ar valve.

Precautions: **Never use recovery flasks with star cracks or other defects!**

Never have valve A and B open at the same time.!!

Never have valve B open when the Vac/Ar valve is set to 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 into the system to prevent this from 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.

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Readying the Flask for Solvent Dispensing:

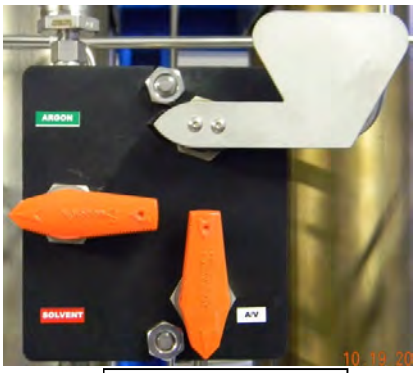
The solvent dispensing outlet should be maintained with valve A closed, valve B closed and the Vac/Ar valve set to the Vac position (See Valve Position I). Clamp on the appropriate *clean, oven-dried*, receiving flask, securing the flask to the adapter with a Thompson clamp. Make sure that the Thompson clamp between the adapter and the outlet is tight The receiving flask now needs to be evacuated with vacuum and back-filled with Argon several times (See valve position 2). An adequate vacuum must be achieved on the flask (approx. 15-20 Torr). Then the Vac/Ar valve is turned to Argon while valve B remains opened to fill the flask with Argon (See valve Position 3).

VALVE POSITION 1

VALVE POSITION 2

- Next, the Vac/Ar valve is returned to the vacuum position and an adequate vacuum is pulled (approx. 10-25 Torr) (See Valve Position 2). Repeat this pump-down/back-fill procedure 5 times. IMPORTANT: If you are filling a standard SPS solvent bulb, open and stopcock to the septum. **End with the recieving flask under vacuum.**

- Now the flask is ready to receive solvent. With the Vac/Ar valve set to vacuum, valve B is closed (See Valve Position 4). This leaves the recovery flask under STATIC vacuum. Then, the Vac/Ar valve is turned to Argon (See Valve Position 5).



Valve Position 3

Solvent Dispensing:

- To dispense the solvent into the receiving flask, open valve A (See Valve Position 4). When you are finished dispensing the solvent, close valve A (See Valve Position 5). HOLD ONTO THE RECEIVING FLASK and carefully open valve B (See Valve Position 3). 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).

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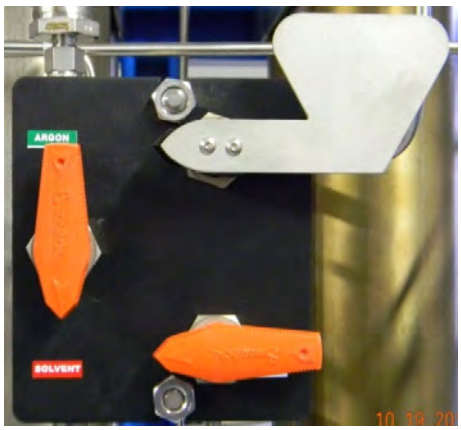
Solvent Dispensing: Continued



Valve Position 4

- 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.

Notes:

- If during the solvent dispensing process the 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. If solvent is still not dispensed, consult the SPS manager(s).
- **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.

Routine use of solvent bulbs:

Valve Position 5

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

- Make sure that the volume of solvent in the bulb will meet your needs. If not, dispense more solvent into the flask.
- Always use a clean, oven-dried syringe needle.
- 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. 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. HOLDING the bulb, remove the needle from the bulb. Readjust the stopcock to the closed position.

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Notes