ARC Unofficial Manual
to the
Solvent Dispensing System

Designed by
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Credits:

Design and Construction of System.....J. C. Meyer

Manual Preparation and Photography.....Matthew E. Hart

I. Introduction:

This manual gives a basic overview of the Solvent Dispensing System (SDS) to give the user a basic knowledge of the apparatus and explain some of its necessary operations. Since a group member is assigned to take care of the instrument, this manual is meant to assist him/her and others in the absence of the specified group member.

The SDS incorporates a vacuum pump and an argon supply and can be used to deliver dried solvent with a blanket of argon. In general, the reaction flask is evacuated with argon and placed under a vacuum. Then the solvent is sucked through the drying columns while also being pushed with argon out of the solvent reservoir (solvent kegs).

What follows is an explanation of the different parts of the SDS, including the argon supply, solvent kegs, columns and solvents, vacuum system, and system 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. Argon Supply:

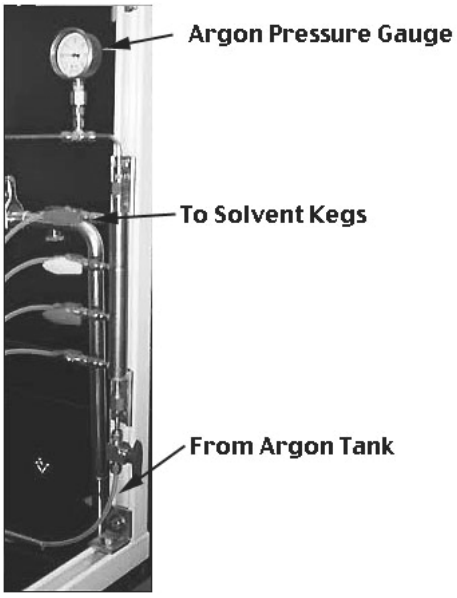
The SDS has an argon supply to keep the solvent under an inert atmosphere. The argon tanks used are ordered from Airgas. The tanks used are as follows:

Argon UHP Grade 99.999% Size 300, Cat. #: SGP015005606, cost $38.00

A blanket account has been setup with Airgas not to exceed $1000. The purchase order request form is filed in the lab information drawer under Airgas account. Name authorization has been given to several people in the lab. These people are the only ones that can order the tanks from Airgas. To contact Airgas call 1-800-2-AIRGAS

When the tanks are hooked up to the system the regulators should be set between 5 - 10 psi. Some regulators have a tendency to slowly

increase in pressure over time. Be sure to check the pressure on these gauges periodically.



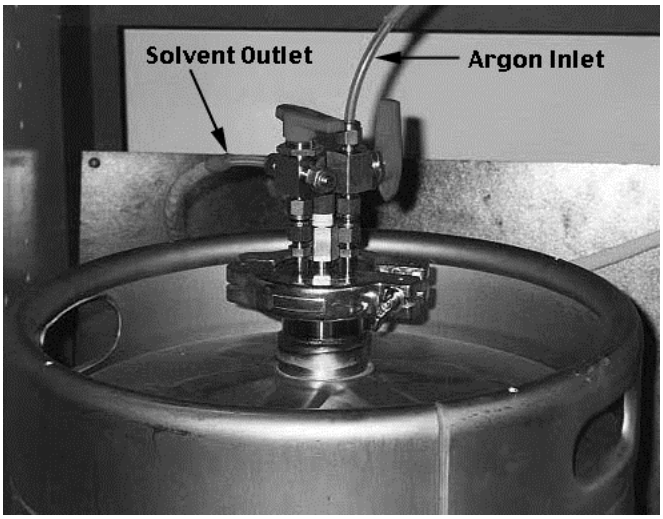
Another pressure gauge is present on the SDS itself on the right hand side. This gauge should also read 5 - 10 psi. Each solvent keg is supplied with a color coded on/off valve that controls the flow of argon. These valves should always be kept closed except for the solvent in use to prevent crossdiffusion.

In the event that the system does begin to overpressurize uncontrollably, the tubing should not rupture. A pressure release valve with a threshold of 50 psi is present on the solvent kegs (described below).

III. Solvent Kegs:

The solvent kegs can hold up to 50 liters of solvent. There are two control valves, one is an argon inlet and the other is the solvent outlet (under normal working conditions). The argon inlet is an on/off valve, while the solvent outlet allows flow in two different directions. One direction is covered by a brass cap. As stated above, the solvent kegs have a pressure release valve with a threshold of 50 psi. This ensures that the system does not become over pressurized.

The standard operation settings are shown in the illustration. The argon inlet is open and the solvent outlet goes into the stills. When not in use for extended periods the solvent outlet should be turned off. The argon inlet should remain closed except to pressurize the tank.



Adding New Solvent:

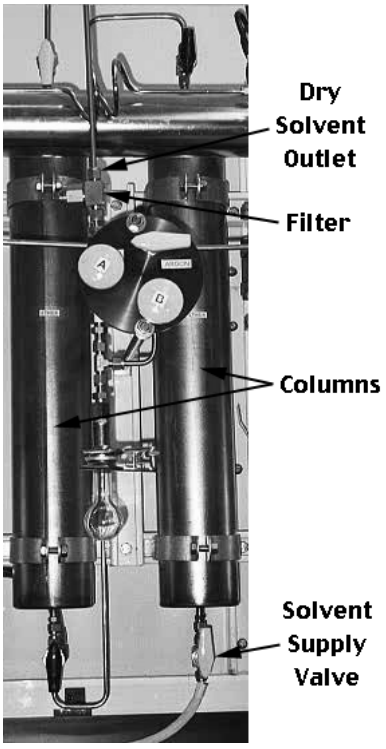
When new solvent needs to be added follow the following guidelines. Turn off the argon at the supply, not at the solvent keg. Next release the argon pressure by loosening the argon inlet. The top of the solvent keg can then be removed by loosening the thumb screw and the solvent is added. Fill to approximately 40 liters and replace the top.

Once the solvent is added, it's important to purge the solvent with argon. To do this the argon inlet is attached to the solvent outlet after removing the brass cap. An additional purge line is attached to the original argon inlet and vented into the hood.

The argon supply is turned on and the solvent is purged for 20 minutes. The argon supply is turned off and the solvent is allowed to settle for 10 minutes, then the solvent is purged for an additional 20 minutes. Once the purge is complete, the argon inlet is reattached to its original position and the valves are turned to the standard operating positions.

The solvents used in this SDS are as follows: Red = THF, Yellow = Ethyl Ether, Blue = Methylene Chloride, Black = Toluene. Note that all of the control valves are color coded for convenience.

IV. Columns:



The columns used in the SDS are intended to vigorously dry the solvents. The solvent is passed through two packed columns of alumina and through a 15 micron filter before being dispensed. In the case of toluene, one of the columns is alumina and the other is Q5, a copper(II) oxide oxygen scavenger.

The solvent supply valve can be turned off when the SDS is not going to be used for a while. In the case of methylene chloride, the solvent supply valve should be turned off if the solvent hasn't been used in a few days. This avoids the chance of back siphoning of the solvent into the solvent keg due to the solvent's specific

gravity.

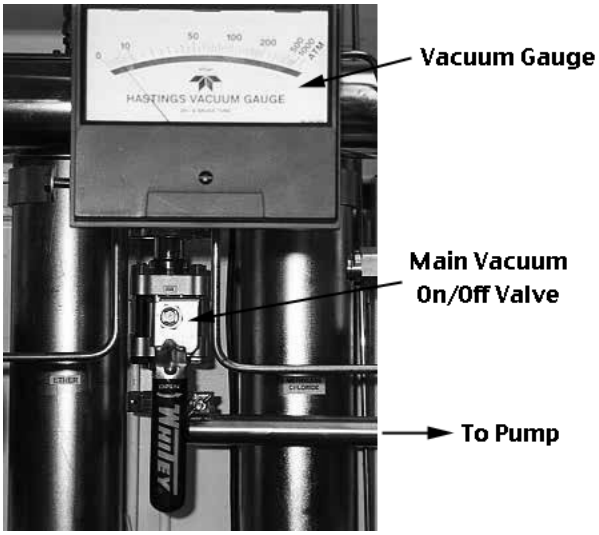
The 15 micron filter ensures that no alumina residue is dispensed with the solvent. The filters used by the SDS are Nupro TF series Tee-type sintered filters. Replacements are ordered from Orange Valve & Fitting Co. phone number (714) 634-0216. Use the following part number:

SS-4F-K4-15, 15 Micron Replacement Filters, $3.30 ea.

A small supply of these filters is kept with the SDS. Instructions for replacement of the filters can be found at the end of this manual. Alternatively, the filters can be sonicated to loosen the filtered alumina.

V. Vacuum System:

The vacuum system of the SDS is a crucial component to proper operation. It is important that the vacuum in running efficiently. The vacuum system consists of the vacuum pump, solvent trap, and the vacuum manifold. It is also important to note the main vacuum on/off valve, the vacuum release valve, and the vacuum gauge.

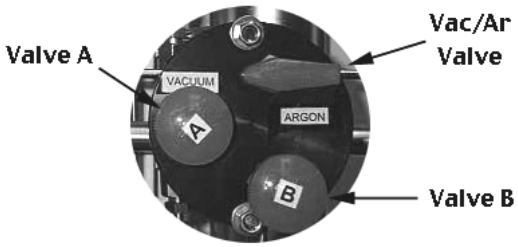


The solvent trap must be filled with liquid nitrogen everyday. If the SDS is not maintained daily, the solvent trap should be emptied and the vacuum pump turned off. This ensures that no solvent is sucked into the pump. Under standard operating conditions, the vacuum gauge should read under 10 milliTorr of pressure. If the pressure is significantly higher than this, it is likely that the solvent trap must be emptied. The pump oil should also be checked for contamination at this time.

To empty the solvent trap, the main vacuum on/off valve must be turned off. Then the green vacuum release valve is opened and the pump is turned off. The two Thomas clamps holding on the solvent trap are removed, and the trap is removed from the dewar. After the solvent melts, the trap is emptied. It is then replaced and clamped

into position. Care must be taken not to over tighten the Thomas clamps. Then the vacuum pump is started again and the vacuum release valve is closed. Finally, the main vacuum on/off valve is opened.

VI. Control Valves:



The solvent delivery controls consist of Valves A and B, the Vac/Ar valve, and a solvent recovery vessel. Valve A controls the delivery of desired solvent. Valve B controls the application of a 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 vacuum line, to the left, and the argon supply, to the right. Under standby conditions, Valve A and B are closed and the Vac/Ar valve is set to vacuum.

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 into the solvent trap and eventually into the pump. This is the most important warning to note!

VII. Dispensing Solvents:

The following is a handout that describes the general procedure for dispensing solvents. It notes some precautions to take before using the SDS. Everyone that uses the SDS should understand this section completely.

Before collecting solvents, check the following system settings:

1) The vacuum pump is operating and maintaining adequate vacuum.
2) Argon is flowing.
   - Check pressure gauge (should read 5-10 psi)
   - Argon valve should be opened for desired solvent
3) Check column and solvent keg valves.
   - All valves should be open

Precautions to take:

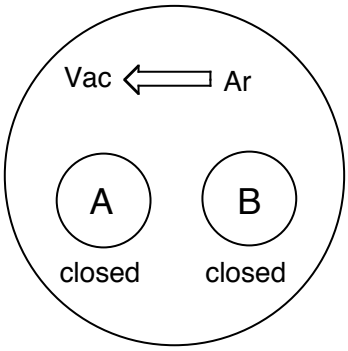
1) **Do Not** over tighten the valves! Finger tight is sufficient.
2) **Never** open valves A and B when the vacuum is open.

Identification of valves.

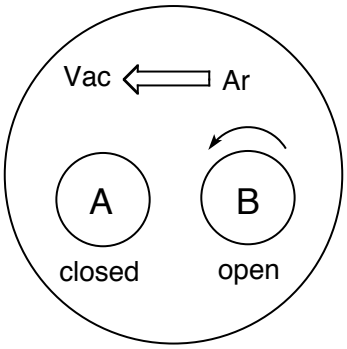
Vac/Ar Valve: Used to switch between the vacuum manifold or the Argon manifold.
Valve A: Used for the addition of solvent into the receiving flask.
Valve B: Used to apply the vacuum or Argon pressure, as dictated by the Vac/Ar valve.

Steps for delivery solvent:

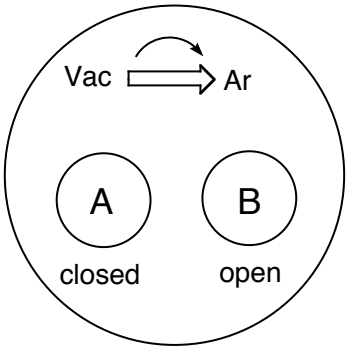
1) The system should be maintained under vacuum, with valve A closed, valve B closed and the Vac/Ar valve set to the vacuum position.



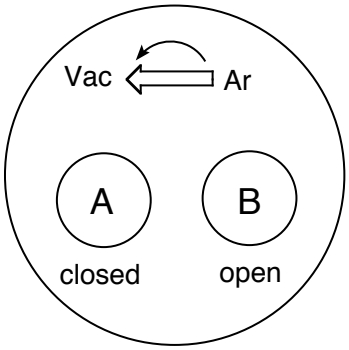
2) Remove the Thomas clamp and the standby flask. Clamp on the appropriate adapter and attach the oven dried receiving flask. Open valve B to apply a vacuum.



3) The receiving flask needs to be flushed with Argon several times. To achieve this, an adequate vacuum must be on the flask (approx. 200 millitorr), then the Vac/Ar valve is turned to Argon while valve B remains open to fill the flask. Once again the valve is returned to the vacuum position and an adequate vacuum is pulled (See I and II). Repeat this procedure as necessary (2-3 times).



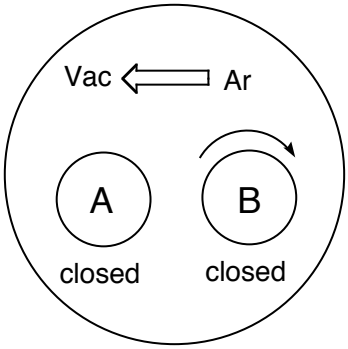
I. Fill with Argon



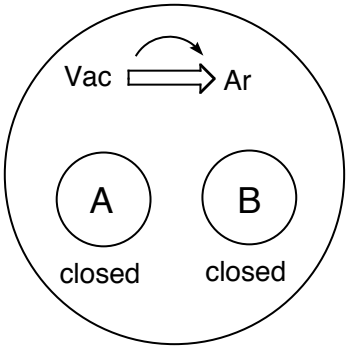
II. Apply Vacuum.

Now the flask has been purged with Argon and is ready to receive the solvent.

4a) With the Vac/Ar valve set to vacuum, valve B is closed (III). Then, the Vac/Ar valve is turned to Argon (IV).

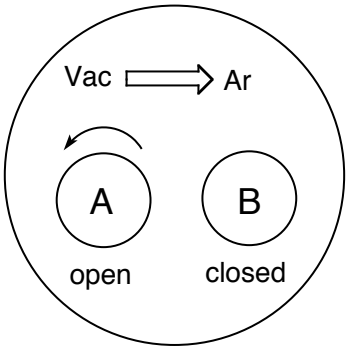


III.



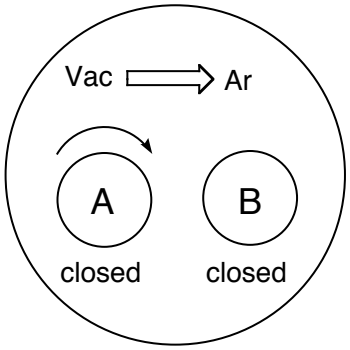
IV.

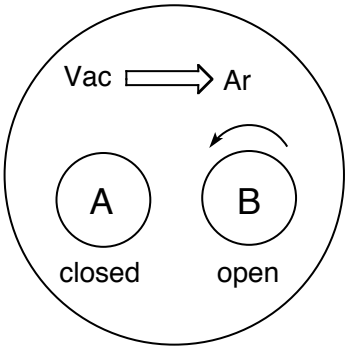
4b) To dispense the solvent into the receiving flask, open valve A. If solvent doesn't flow or only dribbles out, STOP, and close valve A. Check the valves on the column, tanks, and argon lines. Make sure valve B is closed and the Vac/Ar valve is set to Argon. If solvent is still not dispensed, consult the Trouble Shooting Guide or consult the solvent system manager.

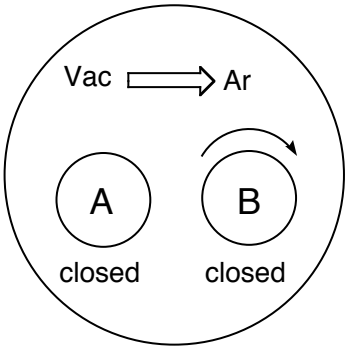


Note: **Never** open valve A while valve B is open and the Vac/Ar valve is set to vacuum! This will fill the pump with solvent.

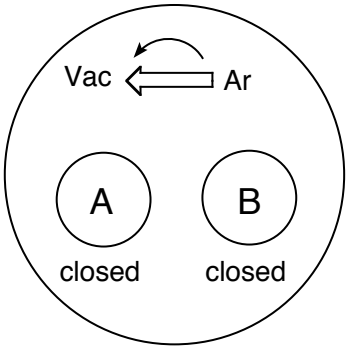
5) When you are finished dispensing the solvent, close valve A. Hold onto the receiving flask and open valve B. The Argon will blow the solvent remaining in the line into your flask and fill the flask. Finally, close valve B.







6) Remove the receiving flask and cap immediately. Replace the adapter with the standby flask. Then turn the Vac/Ar valve to vacuum. Be sure to replace the Thomas clamp. The system should now be ready for the next user.



VII. Trouble Shooting:

This section is a growing section where I will include important information about common problems associated with the daily use of the SDS. It will be as complete as possible. Other labs will be consulted for additional potential problems.

1) Maintain the argon regulator and service the vacuum pump. Most importantly, change the pump oil regularly.
2) Be careful not to kink the teflon tubing beyond its natural ability to bend.
3) Use the proper grade of solvent in the solvent kegs. Stabilized solvents should never be used.
4) A teflon grooving tool must be used when replacing any teflon fitting. This is necessary to ensure a complete seal.