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1 INTRODUCTION

1.1 GENERAL

The Vacuum Atmospheres Solvent Purifier System (SPS) (patent pending) is a superior method of drying solvents compared to thermal distillation. It uses a combination of a sparging/degassing process and circulation between a solvent reservoir and purification cartridge to de-oxygenate and dry solvents without using heat or water reactive drying agents. The SPS allows for the dispensing of precise amounts of anhydrous solvents on demand.

The SPS circulates solvent through a factory conditioned purification cartridge and a stainless steel reservoir. The circulation repeatedly passes the solvent through the purification cartridge, allowing very low levels of moisture to be achieved in a short period of time. The purification cartridge is easily replaced through the use of drip free quick release connections. VAC’s SPS allows for quick and easy access to high purity anhydrous solvents.

The compact design of each unit (approx. 28” high x 10” wide x 15” deep) allows system to be mounted inside a fume hood, on a bench top, or inside a glovebox. The system is sized to be filled or refilled with a 4 liter solvent bottle. The smaller sized VAC system eliminates the need to store large quantities of unstable solvents and reduces potential waste of large amounts of solvent due to accumulation of impurities, such as peroxides. The reusable purification cartridge can be returned to VAC for recharging at a nominal cost.

This manual covers standard solvent purifiers as well as solvent purifiers that utilize glovebox dispensing units in addition to the standard dispensing apparatus. The startup and operation of the glovebox dispensing version is very similar to the standard solvent purifier.
1.2 MECHANICAL CONFIGURATION

The following figures display the general mechanical configuration of a typical Solvent Purification System.

![Solvent Purification System Front Isometric View]

Figure 1-1
Solvent Purification System Front Isometric View
Figure 1-2
Solvent Purification System Rear Isometric View
Figure 1-3
Installation Layout
Figure 1-4
Inert Gas Manifold
1. Sparge vent valve – vents gas during sparging
2. Circulation pump power switch
3. Sparge gas valve – starts gas flow during sparging
4. V1 source valve – selects vacuum or inert gas source
5. V2 dispensing valve – selects solvent or source valve

Figure 1-5
Operator Interface
(with sparge vent shown)
1. Dispensing valve – dispenses solvent when pointing down (towards spout)
2. Dispensing spout

Figure 1-6
Glovebox Dispenser Operator Interface
(shown with 5 solvent dispensers)
2 INSTALLATION

2.1 UNPACKING AND INSPECTION

Completely unpack the SPS. Carefully inspect for any sign of shipping damage. Call Vacuum Atmospheres immediately if any damage is noted.

CAUTION!!
The purifier cartridge is conditioned at the factory. DO NOT install the cartridge before purging the Solvent Purifier system with an inert gas or the charge will be contaminated, necessitating replacement of the purifier cartridge.

2.2 DETERMINING SYSTEM LOCATION

The SPS is designed to be placed in a fume hood or adjacent to a fume hood (within 10 ft). The area where the system is operated must be well ventilated. The SPS can also be installed in a glovebox, provided that the proper utilities (inert gas, vacuum, and a vent line) are brought into the glovebox through appropriate feed through ports. The units have a flanged base that can be used to secure the system to the structure it is resting upon.

The systems are designed as modular units that will fit closely together in the event that multiple units are installed. The right side of the housing is open and fits closely against the next unit on the right. Each unit is dedicated to a specific solvent.

VAC offers an accessory kit including a side cover panel with a 10 PSIG gas regular and manifold. If used, this would be installed on the last system on the right (Figures 1-3 and 1-4).

2.3 CUSTOMER FURNISHED ITEMS

The following items must be supplied for utility connections to Solvent Purification System.

1) Inert gas supply – nitrogen or argon, 99.995% minimum purity level, regulated to 35 PSIG.
2) Electrical power supply – single phase, 110 or 220 VAC, 60/50 Hz power.
3) Vacuum pump (corrosion-resistant, oil-free) – for evacuation of recovery flask. Requires a minimum flow of 0.64 CFM (18 L/min) and a minimum vacuum of 26 inches Hg.
4) 1/4 inch outside diameter tubing (copper or stainless steel) – to connect inert gas supply from source to Solvent Purifier regulator and from regulator manifold to Solvent Purifier unit(s).
5) 1/4 inch outside diameter tubing (of a solvent-resistant material) – for vacuum supply and sparge vent.
6) Low pressure gas regulator (0 – 10 PSIG) – for reducing gas supply pressure to usable levels (1 PSIG during normal operation, 5 PSIG during filling and sparging operations).
2.4 UTILITY CONNECTIONS

Refer to Section 1.2 Mechanical Configuration for figures called out in the following steps.

1) Before proceeding to the next step, verify that the circulation pump power switch (Figure 1-5) is in the OFF position.

2) **Electrical Power.** Connect the 24 volt power supply to the power supply connection on the SPS (Figure 1-2). The power supply is a universal (100-240VAC, 50/60Hz) unit. The appropriate power plug is determined when ordering the SPS. Connect this cord between the SPS power supply and the facility power outlet.

3) **Gas Connection.** Prepare an inert gas source and a low pressure regulator for attachment to the gas port connection. The configuration of this source may be bottles, cylinders or facility gas supply. The regulator must be able to set the gas pressure in increments of 1 PSIG. Typical inert gas sources include argon or nitrogen.

   Connect a ¼ inch outside diameter tubing line (use a non-diffusing material such as copper or stainless steel) from the inert gas supply to the low pressure gas regulator. Connect a ¼ inch outside diameter copper or stainless steel tubing line from the gas regulator to the gas port on the SPS. If more than one SPS is used, a manifold or “splitting” connection must be used to connect the regulator to each unit. Figures 1-3 and 1-4 show these connections.

   VAC offers an option that includes a side cover panel with a 10 PSIG gas regular and manifold that allows connection to multiple SPS units (see Figure 1-3, Figure 1-4, and Appendix A). If using this option, the inert gas supply will need to be regulated to 35 PSIG maximum.

4) **Vacuum Connection.** Prepare a vacuum source for attachment to the vacuum port connection. The system requires a corrosion-resistant, oil-free vacuum source with a minimum flow rate of .64 CFM (18 L/min) and a minimum vacuum of 26 inches Hg.

   Connect a ¼ inch outside diameter tubing line (use a solvent-resistant material, such as PTFE or stainless steel) from the vacuum supply to the vacuum port on the SPS (Figure 1-1).

   VAC offers an option that includes a dry diaphragm type vacuum pump with a manifold that allows connection to multiple Solvent Purification systems (see Appendix A).

   The vacuum pump outlet must be connected to a suitable ventilation system using a solvent-resistant tubing line.

   **Note:** The vacuum pump must be turned on at least 5 minutes prior to any activity requiring evacuation.

5) **Vent Connection.** Connect a tubing line (use a solvent-resistant material, such as PTFE or stainless steel) from a suitable ventilation system to the sparge vent valve on the SPS (Figure 1-1). It is necessary to use a fitting suitable to connect the 1/8 NPT threaded sparge vent outlet to the vent tubing.

6) **Leak Testing.** To ensure proper operation, perform a leak test of the inert gas supply lines and connections before starting the SPS. See Appendix C.
2.5 ELECTRICAL GROUNDING

The SPS must be electrically grounded. In the event multiple units are used, each unit must be grounded. On the rear of the SPS is a 10 ft. length of green grounding wire (Figure 1-2).

WARNING!!
The grounding wire must be secured to a proper grounding source prior to system operation. Static discharge in the presence of flammable solvents can cause fire or explosion.
3 START-UP

The following sections describe how to properly start-up the SPS. Closely follow the procedures to ensure safe lab operation and to avoid damage to the equipment. All manifolds, utilities, and grounding are to be connected prior to this procedure.

Note: If you have multiple Solvent Purification systems, the start-up procedures must be followed for each unit, from Section 3.3 (Purge Procedure) to Section 3.7 (Solvent Circulation), before moving on to the next unit.

Note: If the equipment is used in a manner not specified by VAC, the protection provided by the equipment may be impaired.

Glovebox Dispenser Note: Where directed, certain operations will be performed on the glovebox dispenser. The instructions always refer to the glovebox dispenser that is connected to the solvent purifier currently being operated.

3.1 GLOVEBOX PREPARATION

Follow this step if the glovebox dispenser option is present. Otherwise, skip to the next step.

Before performing any startup operations on the system, verify that an inert atmosphere has been established in the glovebox system. Refer to the glovebox system manual for startup and operation procedures. Do not begin startup of the SPS until the glovebox has an inert atmosphere established.

3.2 SOLVENT SELECTION

Choose a solvent of the appropriate purity level for your application. Vacuum Atmospheres recommends a solvent purity of 200 ppm or less of moisture. Keep in mind that solvents of lower purity will saturate the purification cartridge sooner than solvents of higher purity. This will require replacement of the purification cartridge more frequently.

Note: Solvents used with the VAC Solvent Purification System must be inhibitor-free.

The following is a list of the solvent purification cartridges available from Vacuum Atmospheres. The material in the purifier cartridge is optimized for each specific solvent. The VAC part numbers are specified for each solvent cartridge. Contact VAC if you wish to use a solvent not listed below.

VAC 104424 – Cartridge, Pentane, CAS 109-66-0
VAC 104425 – Cartridge, Hexane, CAS 110-54-3
VAC 104426 – Cartridge, Methanol, CAS 67-56-1
VAC 104427 – Cartridge, Tetrahydrofuran, THF, CAS 109-99-9
VAC 104428 – Cartridge, Methyl Tert-Butyl Ether (MTBE), CAS 1634-04-4
VAC 104429 – Cartridge, Diethyl Ether, Ethyl Ether, Ether, CAS 60-29-7
VAC 104430 – Cartridge, Benzotrifluoride, CAS 98-08-8
VAC 104431 – Cartridge, Ethylene Glycol Dimethyl Ether, DME, CAS 110-71-4
VAC 104432 – Cartridge, Toluene, CAS 109-88-3
VAC 104433 – Cartridge, Triethylamine, CAS 121-44-8
VAC 104434 – Cartridge, Acetone, CAS 67-64-1
VAC 104435 – Cartridge, Acetonitrile, CAS 75-05-8
VAC 104436 – Cartridge, Benzene, CAS 71-43-2
VAC 104437 – Cartridge, Disopropylethylamine, CAS 7078-68-5
VAC 104438 – Cartridge, Dimethylformamide, DMF, CAS 68-12-2
VAC 104439 – Cartridge, Dimethyl Sulfoxide, DMSO, CAS 67-68-5
VAC 104440 – Cartridge, Chloroform, CAS 67-66-3
VAC 104441 – Cartridge, Dichloromethane, CAS 75-09-2
VAC 104442 – Cartridge, Dioxane, CAS 123-91-1
VAC 104443 – Cartridge, Pyridine, CAS 110-86-1
VAC 104444 – Cartridge, Ethanol, CAS 64-17-5
VAC 104446 – Cartridge, Dodecane, CAS 112-40-3
VAC 104448 – Cartridge, Diethylformamide, CAS 617-84-5
VAC 104450 – Cartridge, Butanol, CAS 71-36-3
VAC 104452 – Cartridge, Cyclohexane, CAS 11-82-7
VAC 104454 – Cartridge, Chlorobenzene, CAS 108-90-7
VAC 104456 – Cartridge, Propylene Carbonate, CAS 108-32-7
VAC 104458 – Cartridge, Dimethyl Acetamide (DMAC), CAS 127-19-5
VAC 104460 – Cartridge, Hexamethyldisiloxane (Bis(Trimethylsilyl)Ether, CAS 107-46-0
VAC 106320 – Cartridge, Heptane, CAS 142-82-5
VAC 107467 – Cartridge, Dichloroethane, CAS 107-06-2
3.3 PURGE PROCEDURE

The following section describes how to purge the moisture and oxygen from your SPS. Do not start this procedure until you are ready to fill the solvent reservoir.

3.3.1

**Objective:** Initial control settings.

**Settings:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve - CLOSED
- Circulation Pump – OFF
- Vacuum Pump – ON (turn on vacuum pump 5 minutes prior)
- Low Pressure Regulator Setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

3.3.2

**Objective:** To purge the air from the plumbing and reservoir.

**Action:** Turn sparge gas valve to the left (on position). Open vent valve.

**Settings:**
- V1 – OFF
- V2 - OFF
- Sparge Gas Valve – ON
- Vent Valve – OPEN (vertical)
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

3.3.3

**Objective:** To purge the air from the lines leading to reservoir and glovebox dispenser.

**Action:** Turn V1 to off. Turn V2 to point left (solvent position). Close sparge vent valve. Allow gas to purge through dispensing spout(s) for 5 minutes. 

**NOTE:** Flow will be very low, this is normal.

**Settings:**
- V1 – OFF
- V2 – POINT LEFT (solvent position)
- Sparge Gas Valve – ON
- Vent Valve – CLOSED (horizontal position)
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF (for glovebox solvent loop connection), ON (for one line glovebox connection)
- One Line Diverter Valve (if equipped) – SAMPLE
### 3.3.4

**Objective:** To purge air from the lines leading to the dispensing spout.

**Action:** Turn V1 to point right (inert gas position), turn V2 to point right, turn sparge gas valve off. Allow gas to purge through dispensing spout for 2 minutes.

**NOTE:** Flow will be very low, this is normal.

**Valve Positions:**
- V1 – INERT GAS (Pointing RIGHT)
- V2 – POINT RIGHT
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED (horizontal position)
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

### 3.3.5

**Objective:** Conclude the purge procedure.

**Action:** Turn V2 off. Turn V1 off.

The start-up procedure up to this point has eliminated the air from the interior of the system. The Solvent Purifier is now ready for filling with solvent.

**NOTE:** Glovebox dispenser spout will be dried out by the glovebox atmosphere.

**Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED (horizontal position)
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION
3.4 FILLING SOLVENT RESERVOIR

3.4.1 Objective: To prepare unit for addition of solvent.
Action: 1) Open vent valve. 2) Turn sparge gas valve on. 3) Remove vent flange clamp.
Valve Positions:
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – OPEN (vertical position)
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

3.4.2 Objective: To add solvent while purging through the same fill port the solvent is entering.
Action: 1) Remove vent valve and o-ring seal. 2) Insert a funnel into the flange and fix it in place, leaving space for gas to exit between the flange and the funnel. 3) Slowly pour 4 liters of solvent into the funnel.
Valve Positions:
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – REMOVED
- Circulation pump – OFF
- Vacuum pump – ON
- Gas pressure setting – 5 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

WARNING!!
Wear personal protective equipment as required while pouring solvent. Pour solvent inside a fume hood or have means of capturing fumes and directing to a fume system.
3.5 SPARGING (DEGASSING) ROUTINE

CAUTION!!
On a system with multiple solvent purifiers: Do not sparge more than ONE SPS unit at a time. Do not leave sparge gas valve on with vent valve closed unless solvent is being dispensed in the glovebox using the one line method (see Section 4.5). This prevents cross-contamination that can occur with some solvents.

3.5.1

Objective: To degas or sparge solvent to help remove dissolved oxygen while minimizing loss of solvent due to evaporation.

Action: 1) Reduce inert gas pressure to 1 PSIG. 2) Reattach vent valve with valve in OPEN position and secure with KF clamp. Sparging was occurring while solvent was pouring. Continue to allow sparge gas to flow for short period (you will hear a bubbling sound).

Valve Positions:
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – OPEN
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 1 PSIG (normal operation setting)
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

3.5.2

Objective: To complete degas/sparge operation, seal unit, and stop sparge gas flow.

Action: 1) Turn Sparge Valve to OFF position. 2) Then IMMEDIATELY close vent valve.

Valve Positions:
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION
3.6 ATTACHING PURIFIER CARTRIDGE

Action:
1) Orient the cartridge adjacent to the quick release receptacles located on the front of the housing.
2) Insert the top quick disconnect by applying steady pressure against the top receptacle. A distinct snapping sound will indicate that the connection has been made. Make sure the “barrel” portion of the quick disconnect moves forward against the nut of the mating end of the cartridge.
3) Orient the bottom quick disconnect adjacent to the bottom receptacle. There is some flexibility in the tubing line for ease of orientation. Apply pressure to snap the bottom connection into place.

NOTE: The purifier unit may need to be held in place so that it does not move backwards while attaching the cartridge.

3.7 SOLVENT CIRCULATION

Objective: Initiate solvent circulation to purify the solvent stored in the SPS.
Action: After the purifier cartridge is installed, turn circulation pump ON. New solvent must be circulated for 6 hours to attain maximum purity. Turn vacuum pump OFF while circulating, as it is not needed for this process. After 6 hours, switch the circulation pump OFF. Do not turn the pump on again until the reservoir has been refilled with 4 liters of new solvent.

Valve Positions:
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation pump – ON
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION
4 SOLVENT DISPENSING

NOTE: When new solvent is added to the system, VAC recommends circulating for 6 hours in order to attain maximum solvent purity. After 6 hours, turn the circulation pump OFF. Do not turn the pump ON again until the system will no longer dispense solvent and the reservoir has been refilled with new solvent.

4.1 GLASSWARE REQUIREMENTS

The dispensing spout of the SPS is terminated with a 3/8 inch diameter tubing spout. A universal inlet adapter with a 24/40 inner joint and a #12 thread is supplied with the system. Alternative adapters may be used as required.

The recovery flask glassware used to collect the solvent must be compatible with a 24/40 connection (if the standard inlet adapter is used). A suitable sealing grease or PTFE sleeve is recommended where the recovery flask interfaces with the inlet adapter.

The flask must also be equipped with a valve to isolate the solvent from air exposure. Another alternative is to have a septum located on the recovery flask to allow extraction of solvent via a syringe. The septum allows the flask to remain in place while a small amount of anhydrous solvent is extracted.

Glovebox Dispenser Note: Because the glovebox atmosphere is dry (typically <1 ppm moisture), the use of a sealed flask is not necessary.

4.2 GLASSWARE PREPARATION

The recovery flask glassware used to collect the solvent must be oven dried before attaching it to the inlet adapter. The recovery flask must be secured to the inlet adapter with the weight supported by a clamp or height adjustable support base.

After the recovery flask is attached to the inlet adapter, the moisture and oxygen trapped inside must be removed. This must be done before extracting solvent to prevent contamination of the newly purified solvent. This is done by running through a minimum of three evacuation and refill cycles as described in the next section. Once this is done, the recovery flask can remain in place indefinitely.

Glovebox Dispenser Note: If the glassware used for the glovebox dispenser is stored inside the glovebox, it will be dried by the low-moisture atmosphere. It does not need to be oven dried, but should be stored in the glovebox overnight before being used.
4.3 SOLVENT DISPENSING OPERATION - STANDARD

**WARNING!!**
To prevent solvent spills, the recovery flask must be secured to the inlet adapter with a clamp and/or supported by a height-adjustable support base.

**CAUTION!!**
On a system with multiple solvent purifiers: Dispense only ONE solvent at a time. Do not leave the V1 valve (vacuum or gas) connected to a filled recovery flask; keep V1 and V2 valves in the OFF position when not dispensing solvent. This prevents cross-contamination that can occur with some solvents.

### 4.3.1

**Objective:** Purge the dispensing spout and inlet adaptor prior to attaching the recovery flask.

**Action:**
1. Turn V1 valve to inert gas position.
2. Turn V2 valve so it is pointing right.
3. Allow gas to purge through the inlet adaptor for 2 minutes.
4. Attach the recovery flask.

**Valve Positions:**
- V1 – Inert gas – RIGHT
- V2 – Point RIGHT
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation Pump – OFF
- Vacuum pump – ON (allow to run for 5 minutes prior)
- Low Pressure Regulator Setting – 1 PSIG

### 4.3.2

**Objective:** Remove air containing moisture and oxygen from the recovery flask used to collect the solvent.

**Note:** The vacuum pump must be turned on 5 minutes prior to the evacuation of the glassware

**Note:** The capacity of the vacuum pump will affect the amount of time needed to evacuate the recovery flask. This procedure presumes that a vacuum pump with the minimum capacity is being used.

**Action:**
1. Turn V1 left to vacuum position and allow to evacuate for 2 minutes

**Valve Positions:**
- V1 – Vacuum (pointing LEFT)
- V2 – point RIGHT
- Sparge Gas Valve – OFF
- Vent Valve - CLOSED
- Circulation Pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 1 PSIG
4.3.3  

**Objective:** To refill the recovery flask with inert gas. Then repeat evacuation and refill 3 times.  

**Action:** Turn V1 valve right to inert gas position.  

**Valve Positions:**  
- V1 – Inert gas – RIGHT  
- V2 – Point RIGHT  
- Sparge Gas Valve – OFF  
- Vent Valve – CLOSED  
- Circulation Pump – OFF  
- Vacuum pump – ON  
- Low Pressure Regulator Setting – 1 PSIG

4.3.4  

Repeat the evacuation and refill steps (4.3.2 and 4.3.3) three times before moving to the next step.

4.3.5  

**Objective:** Evacuate recovery flask in preparation for dispensing solvent.  

**Action:** Turn V1 left to vacuum position and evacuate for 1 minute.  

**Valve Positions:**  
- V1 – Vacuum (pointing LEFT)  
- V2 – pointing RIGHT  
- Sparge Gas Valve – OFF  
- Vent Valve – CLOSED  
- Circulation Pump – OFF  
- Vacuum pump – ON  
- Low Pressure Regulator Setting – 1 PSIG

4.3.6  

**Objective:** To dispense solvent into flask in a slow controlled manner.  

**Action:** 1) Turn V1 to off position. 2) Turn V2 left to solvent position. 3) Turn V2 to OFF position once desired amount of solvent is dispensed.  

**Note:** If the solvent slows or stops before the desired amount is dispensed, briefly evacuate the flask again, then finish dispensing. Do not use pump to remove solvent.  

**Valve Positions:**  
- V1 – OFF (pointing down)  
- V2 – point LEFT (turn valve slowly), then OFF  
- Sparge Gas Valve – OFF  
- Vent Valve – CLOSED  
- Circulation Pump – OFF  
- Vacuum pump – ON  
- Low Pressure Regulator Setting – 1 PSIG
4.3.7

Objective: To degas solvent after dispensing into flask.
Action: Turn V1 to Vacuum, Turn V2 to RIGHT position

Valve Positions:
- V1 – Vacuum (pointing LEFT)
- V2 – pointing RIGHT
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation Pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 1 PSIG

4.3.8

Objective: To refill flask with inert gas then close the flask valve to allow removal from spout.
Action: 1) Turn V1 to Inert Gas. 2) Close valve on flask.
You can now remove the flask.

Valve Positions:
- V1 – Inert gas – RIGHT
- V2 – Point RIGHT
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation Pump – OFF
- Vacuum pump – ON
- Low Pressure Regulator Setting – 1 PSIG

4.3.9

Objective: Reset valves to initial control settings
Action: Turn V2 off. Turn V1 off. Turn the vacuum pump power off.

Valve Positions:
- V1 – Inert gas – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation Pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
4.4 SOLVENT DISPENSING OPERATION - GLOVEBOX DISPENSING USING SOLVENT CIRCULATION LOOP

Follow these instructions if the system has been configured with the inlet and outlet of the Solvent Purification System plumbed into the glovebox. See Section 4.5 if the system has been configured with a pressurized one line connection from the SPS to the glovebox.

4.4.1

<table>
<thead>
<tr>
<th>Objective:</th>
<th>Start solvent flow through the circulation loop into the glovebox.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Action:</td>
<td>Turn Circulation Pump switch ON.</td>
</tr>
<tr>
<td><strong>Solvent Purifier Valve Positions:</strong></td>
<td></td>
</tr>
<tr>
<td>V1 – OFF</td>
<td></td>
</tr>
<tr>
<td>V2 – OFF</td>
<td></td>
</tr>
<tr>
<td>Sparge Gas Valve – OFF</td>
<td></td>
</tr>
<tr>
<td>Vent Valve – CLOSED</td>
<td></td>
</tr>
<tr>
<td>Circulation Pump – ON</td>
<td></td>
</tr>
<tr>
<td>Vacuum pump – ON (allow to run for 5 minutes prior)</td>
<td></td>
</tr>
<tr>
<td>Low Pressure Regulator Setting – 1 PSIG</td>
<td></td>
</tr>
<tr>
<td>Glovebox Dispensing Valve (if equipped) – OFF</td>
<td></td>
</tr>
</tbody>
</table>

4.4.2

<table>
<thead>
<tr>
<th>Objective:</th>
<th>Dispense solvent in a slow and controlled manner.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Action:</td>
<td>1) Slowly rotate the dispensing valve until it is pointing down. If you are dispensing a small amount of solvent, rotate the valve until the solvent just barely flows from the dispensing spout.</td>
</tr>
<tr>
<td><strong>Glovebox Dispenser Valve Positions:</strong></td>
<td></td>
</tr>
<tr>
<td>Dispensing Valve – ON (pointing DOWN)</td>
<td></td>
</tr>
</tbody>
</table>

4.4.3

<table>
<thead>
<tr>
<th>Objective:</th>
<th>To stop dispensing solvent.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Action:</td>
<td>1) Rotate dispensing valve until it is pointing up.</td>
</tr>
<tr>
<td><strong>Glovebox Dispenser Valve Positions:</strong></td>
<td></td>
</tr>
<tr>
<td>Dispensing Valve – OFF (pointing UP)</td>
<td></td>
</tr>
</tbody>
</table>
4.4.4

Objective: Reset valves and switches to initial control settings

Action: 1) Turn Circulation Pump switch OFF.

Glovebox Dispenser Valve Positions:
- Dispensing Valve – OFF (pointing UP)

Solvent Purifier Switch Positions:
- Circulation Pump – OFF
4.5 SOLVENT DISPENSING OPERATION - GLOVEBOX DISPENSING USING PRESSURIZED ONE LINE CONNECTION

Follow these instructions if a circulation/sample diverter valve shown below has been installed on top of each of the solvent purifiers.

In this case, there is a single line connection from each SPS to the glovebox.

4.5.1

**Objective:** Open the one line connection from the SPS to the glovebox with pressurized solvent.

**Solvent Purifier Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – CLOSED
- Circulation Pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG MAXIMUM

**Caution:** pressures in excess of 1 PSIG may result in cross contamination in multiple SPS installations.
- Glovebox Dispensing Valve – OFF
- One Line Diverter Valve - SAMPLE

4.5.2

**Objective:** Dispense solvent in a slow and controlled manner.

**Action:** 1) Slowly rotate the dispensing valve counterclockwise. If you are dispensing a small amount of solvent, rotate the valve until the solvent just barely flows from the dispensing spout.

**Glovebox Dispenser Valve Positions:**
- Dispensing Valve – COUNTERCLOCKWISE
4.5.3

**Objective:** To stop dispensing solvent.

**Action:**
1) Rotate dispensing valve clockwise until it is closed.

**Glovebox Dispenser Valve Positions:**
- Dispensing Valve - CLOCKWISE

4.5.4

**Objective:** Close the one line connection from the SPS to the glovebox.

**Solvent Purifier Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation Pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG MAXIMUM

**Caution:** pressures in excess of 1 PSIG may result in cross contamination in multiple SPS installations.
- Glovebox Dispensing Valve – OFF
- One Line Diverter Valve - CIRCULATION
5 REFILLING SOLVENT RESERVOIR

When the purified solvent is depleted, it will be necessary to add additional solvent and circulate it through the purification cartridge. The following sections explain how the user will be notified of a low solvent level and how to add additional solvent to the Solvent Purifier.

CAUTION!!
To avoid cross-contamination, always fill the SPS with the same solvent that had been used previously. Even when the reservoir has been emptied, the charge in the purification cartridge will still contain small amounts of the previously used solvent.

5.1 LOW SOLVENT LEVEL

When the quantity of solvent in the reservoir has been reduced to approximately 1 liter, the purified solvent will no longer dispense. At this point the reservoir must be refilled with 4 liters of new solvent. See the next section for instructions on refilling the solvent reservoir.

CAUTION!!
When the system will no longer dispense solvent, do not use the pump to attempt to dispense the remainder of the solvent in the reservoir – damage to the pump may result.
5.2 REFILLING THE RESERVOIR

**WARNING!!**

Wear personal protective equipment as required while pouring solvent. Pour solvent inside a fume hood or have means of capturing fumes and directing to a ventilation system.

---

5.2.1

**Objective:** To prepare unit for addition of solvent

**Action:** 1) Turn circulation pump off. 2) Open vent valve. 3) Immediately turn sparge gas valve on. 4) Remove vent flange clamp.

**Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – OPEN (vertical position)
- Circulation pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

---

5.2.2

**Objective:** To add solvent while purging through the same fill port the solvent is entering.

**Action:** 1) Remove vent valve and o-ring seal. 2) Insert a funnel into the flange and fix it in place, leaving space for gas to exit between the flange and the funnel. 3) Slowly pour 4 liters of solvent into the funnel.

**Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – REMOVED
- Circulation pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

---

**WARNING**

Wear personal protective equipment as required while pouring solvent. Pour solvent inside a fume hood or have means of capturing fumes and directing to a ventilation system.
5.2.3

**Objective:** To degas or sparge solvent to help remove dissolved oxygen while minimizing loss of solvent due to evaporation.

**Action:** 1) Reattach vent valve with valve in OPEN position and secure with KF clamp. Sparging was occurring while solvent was pouring. Continue to allow sparge gas to flow for short period (you will hear a bubbling sound).

**Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – ON
- Vent Valve – OPEN
- Circulation pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

5.2.4

**Objective:** To complete degas/sparge operation, seal unit, and stop sparge gas flow.

**Action:** 1) Turn Sparge Valve to OFF position. 2) Then IMMEDIATELY close vent valve.

**Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation pump – OFF
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

5.2.5

**Objective:** Initiate solvent circulation to purify the solvent stored in the Solvent Purifier system.

**Action:** 1) Turn circulation pump ON. The new solvent must be circulated for 6 hours to attain maximum purity. After 6 hours, switch the pump OFF. Do not turn the pump ON again until the reservoir has been refilled with 4 liters of new solvent.

**Valve Positions:**
- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve – CLOSED
- Circulation pump – ON
- Vacuum pump – OFF
- Low Pressure Regulator Setting – 1 PSIG
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION
6 MAINTENANCE

6.1 GENERAL

Maintenance of the Solvent Purifier System is uncomplicated due to the relative simplicity of the unit’s design and construction. However, standard safe practices should always be observed during maintenance.

The following sections outline several maintenance areas for providing optimal system performance. Always observe standard safe practices during maintenance of this system.

6.2 VACUUM PUMP

Refer to the vacuum pump manufacturer’s instruction manual for maintenance procedures and specifications.

6.3 LINES AND CONNECTIONS

Inspect all lines, valves, fittings, and connections for mechanical integrity at 3-month intervals.

6.4 INLET ADAPTER

Periodically inspect the o-ring that seals the inlet adapter to the dispensing spout for solvent damage or degradation. Replace if necessary.

6.5 PURIFICATION CARTRIDGE

Eventually, the purification cartridge will become saturated and will no longer be able to remove moisture from the solvent. When this happens, the cartridge media must be replaced with new, pre-conditioned media from VAC. The purification media is available in cartridge recharge kits specific to the particular solvent used. See Appendix A for a list of recharge kits.

Refer to Section 6.5.3 for instructions and a list of items needed for replacing the cartridge media. Refer to Section 6.5.1 for removal and replacement of the purification cartridge.
6.5.1 Removal and Replacement of the Purification Cartridge

**WARNING!!**
Wear personal protective equipment as required while replacing the purifier cartridge.

1) Before proceeding to the next step, verify that the circulation pump power switch (Figure 1-5) is in the OFF position.

2) Grasp the purification cartridge with one hand.

3) Push the “barrel” portion of the bottom quick disconnect receptacle back, away from the mating fitting on the cartridge. The cartridge-side fitting should pop out about ½ inch.

4) Repeat the previous step for the top fitting and pull the cartridge away from the SPS.

5) Replace the media in the purification cartridge according to the instructions in Section 6.5.3.

6) While the cartridge is removed, replace the particulate filter located between the dispensing spout and the front panel of the SPS. See Section 6.5.2 for instructions.

7) Orient the newly charged cartridge adjacent to the quick release receptacles located on the front of the housing.

8) Insert the top quick disconnect by applying steady pressure against the top receptacle. A distinct snapping sound will indicate that the connection has been made. Make sure the “barrel” portion of the quick disconnect moves forward against the nut of the mating end of the cartridge.

9) Orient the bottom quick disconnect adjacent to the bottom receptacle. There is some flexibility in the tubing line for ease of orientation. Apply pressure to snap the bottom connection into place.

10) Switch the circulation pump power switch (Figure 1-5) to the ON position to begin circulating the solvent through the new cartridge.
6.5.2 Replacing the Dispensing Filter

Always replace the dispensing filter when changing the purifier cartridge. The filter is located between the dispensing spout and the front panel of the SPS.

**Tools and Materials Needed:**

1) Large adjustable wrench (able to open to 1-1/8”)
2) One inch open end wrench or small adjustable wrench

The filter replacement can be done either by opening the filter housing and replacing the element, or by removing the filter housing and replacing it with a new one. If using the second option, be sure to reseal the connections using ¼" wide PTFE tape making sure the first thread is free of tape.
6.5.3 Recharging the Purification Cartridge

WARNING!!
Wear personal protective equipment as required while recharging the purifier cartridge. The nature and extent of protection required depends on the nature of the solvent. The determination of the type and extent of precautions required is the sole responsibility of the user. Always discard contaminated material in a safe and responsible manner in accordance with federal, state, and local regulations.

CAUTION!!
Once the bag containing the new media has been opened, complete this process and seal the cartridge within 10-20 minutes. Exposing the media to air for longer periods will decrease the solvent purifier performance.

Tools and Materials Needed:

3) Strap Wrench
4) Vise or large adjustable wrench (able to open to 1-1/8”)
5) 9/16 inch open end wrench or small adjustable wrench
6) Funnel (with spout able to fit into a 7/8 inch diameter opening and an inside diameter (of spout) at least 3/8 inches.
7) PTFE (Teflon) tape
8) Purification cartridge recharge kit (see Appendix A for list of kits available)
Instructions:

1) Remove the purification cartridge by following the instructions in section 6.5.1 (Replacing the Purification Cartridge).

2) Loosen the Swagelok fitting on the bottom of the purifier cartridge. Remove the tubing and allow the solvent to drain out of the cartridge. This may take several minutes.

3) Use one of the following methods to open the cartridge:
   A. If a vise is available:
      1. Turn the cartridge upside-down and clamp the ¾ NPT fitting (on the top of the purification cartridge) in the vise.
      2. Use a strap-wrench or other suitable means to turn the cartridge counter-clockwise, loosening the ¾ NPT fitting. When the ¾ NPT fitting can be turned by hand, remove the cartridge from the vise.
   B. If no vise is available:
      1. Place the cartridge on its side on a bench and put the strap wrench on the cartridge body as if you were going to turn it clockwise.
      2. Use the large adjustable wrench to loosen the ¾ NPT fitting (turn counter-clockwise) until it can be turned by hand. Use the strap wrench to prevent the cartridge from turning. You may need an assistant to hold the cartridge in place.

4) Remove the ¾ NPT fitting the rest of the way by hand. Pour out and discard the old purification charge. NOTE: There may still be some liquid solvent in the cartridge.

5) If necessary, clean the empty cartridge with a suitable cleaning agent. Allow the cartridge to dry before adding the new media.

6) Thoroughly clean the remaining PTFE tape from the threads of the ¾ NPT fitting and the threads in the mating cartridge fitting. Make sure that there are no particles or old PTFE tape on the threads.

7) Apply new PTFE tape on the ¾ NPT fitting threads. Make sure to apply the PTFE tape in the direction of the threads. Cover all of the threads with the tape, making sure that the tape is 2 or 3 layers thick at the top of the threads (near the hex part of the fitting).

8) Cut off one of the corners of the bag containing the new purification media.

9) Using the funnel, pour the contents of the bag into the cartridge. You will need to shake the cartridge periodically to level the media. Leave a space of about ¼ inch between the top of the cartridge and the top level of the media.

10) Install the ¾ NPT fitting and tighten by hand. Use the reverse of the method used in step 3 above to tighten the fitting completely.

11) Replace the bottom tubing and hand-tighten the Swagelok fitting.

12) Visually line up the bottom quick-connect fitting with the top quick-connect fitting. Tighten the Swagelok fitting using the 9/16 inch wrench. Verify that the quick-connect fittings are still aligned. The flexibility of the ¼ inch tubing will allow some leeway in the fitting alignment.

13) Install the newly recharged cartridge onto the solvent purifier by following the instructions in section 6.5.1 (Replacing the Purification Cartridge).
### 6.6 TROUBLESHOOTING

The following chart lists some simple problems and their solutions. Contact VAC if you are experiencing any problems not shown.

<table>
<thead>
<tr>
<th>SYMPTOM</th>
<th>PROCEDURE</th>
</tr>
</thead>
</table>
| Circulation pump does not operate | 1) Verify that power supply plug is completely inserted into power supply connection (see Figure 1-2).  
2) Verify that the power cord connectors are completely connected to the power supply and the facility power outlet.  
3) Verify that the correct voltage (110 or 220 volts AC) is present at the facility power outlet.  
4) Verify that the two terminal connectors on the back of the pump power switch are completely connected. |
| Solvent purity does not meet expectations | 1) Verify that the solvent was allowed to circulate through the purification cartridge for 6 hours before dispensing.  
2) Verify that the solvent of recommended purity was used (see Section 3.1).  
3) Verify that high purity inert gas is connected to the gas regulator.  
4) Verify that vacuum pump is allowed to run for 5 minutes prior to dispensing solvent.  
5) Verify recovery flask was properly prepared prior to attaching it to the dispensing spout.  
6) Verify recovery flask was evacuated and refilled a minimum of 3 times for 2 minutes each time prior to dispensing solvent.  
7) Verify that there are no loose connections at any of the gas or vacuum lines.  
8) Verify that the startup routine was followed correctly. If the system was not properly purged prior to attaching the purifier cartridge, poor performance can result. |
| Solvent dispenses slowly or not at all | 1) Verify dispensing filter element is free of any debris (see Section 6.5.2). Replace filter if any material is found.  
2) Verify that vacuum pump is on and allowed to run for 5 minutes prior to dispensing solvent.  
3) Briefly open and close the sparge gas valve to refill the vacuum in the solvent reservoir.  
4) Turn the circulation pump on. If solvent dispenses now, there is 1 liter or less of solvent in the reservoir (see Section 5.1). |
7 SYSTEM SPECIFICATIONS

Electrical
Input Power:
100 – 240 Volts AC - Single phase connection, 1.5 amps, 50/60 Hz. Power plug type specified at time of order placement

Utility Connections
Inert Gas: ¼ inch tubing connection, regulated to 35 PSIG
Vacuum: ¼ inch tubing connection, minimum vacuum level - 26 inches Hg, minimum flow - 0.64 CFM
Sparge Vent: 1/8 female NPT connection

Environmental
Ambient Temperature: 59° – 90° F (15° – 32° C)
Ambient Humidity: 0–90% non-condensing
APPENDIX A – ACCESSORIES

VACUUM PUMPS

Vacuum Atmospheres offers a vacuum pump that can be used with up to seven Solvent Purifier units. This pump is available in several configurations for different input power specifications. The vacuum pump manifold connections are shown below.

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>VAC 107086</td>
<td>Vacuum Pump Kit – Single phase, 115VAC, 60Hz with North American power plug</td>
</tr>
<tr>
<td>VAC 107087</td>
<td>Vacuum Pump Kit – Single phase, 230VAC, 50Hz with Continental European power plug</td>
</tr>
<tr>
<td>VAC 107088</td>
<td>Vacuum Pump Kit – 100VAC, 50/60Hz, Japan power</td>
</tr>
</tbody>
</table>

ACCESSORIES

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>VAC 104423</td>
<td>Regulator Kit, 10 PSIG (gas regulator manifold/side cover assembly, Figures 1-3 and 1-4)</td>
</tr>
<tr>
<td>VAC 400837</td>
<td>Tubing, PTFE, 1/8 inch ID, 1/4 inch OD, 1/16 inch wall thickness – sold by the foot</td>
</tr>
<tr>
<td>VAC 400772</td>
<td>Universal Inlet Adapter, 24/40 connection to 3/8 inch diameter tubing</td>
</tr>
<tr>
<td>VAC 105966</td>
<td>Solvent Purifier Service Kit (used for draining and cleaning solvent purifier and cartridge)</td>
</tr>
</tbody>
</table>

PURIFICATION CARTRIDGES

A list of the purification cartridges available from Vacuum Atmospheres can be found in Section 3.2. Different solvents work best with different purification charge material, so it is important to use the purification cartridge specified for the specific solvent.
RECHARGE KITS FOR PURIFICATION CARTRIDGES

The following is a list of purification cartridge recharge kits available from VAC. These kits include pre-conditioned purification media stored in a moisture resistant barrier bag. The purification media in each kit has been selected to work best with the specified solvent listed in the first column (below). Using the incorrect purification media may produce unsatisfactory results such as reduced moisture removal capacity.

To select the correct purification cartridge recharge kit, locate the solvent that you are using in the first column and order the corresponding part number from VAC. In addition to the recharge kit, you will need several other items to replace the purification media. See Section 6.5.3, Recharging the Purification Cartridge, for details.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Part Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentane CAS 109-66-0</td>
<td>105640</td>
<td>Kit, Recharge, Purification Cartridge, Pentane</td>
</tr>
<tr>
<td>Hexane CAS 110-54-3</td>
<td>105641</td>
<td>Kit, Recharge, Purification Cartridge, Hexane</td>
</tr>
<tr>
<td>Methanol CAS 67-56-1</td>
<td>105642</td>
<td>Kit, Recharge, Purification Cartridge, Methanol</td>
</tr>
<tr>
<td>Tetrahydrofuran (THF) CAS 109-99-9</td>
<td>105643</td>
<td>Kit, Recharge, Purification Cartridge, Tetrahydrofuran (THF)</td>
</tr>
<tr>
<td>Diethyl Ether, Ethyl Ether, Ether CAS 60-29-7</td>
<td>105644</td>
<td>Kit, Recharge, Purification Cartridge, Ethyl Ether</td>
</tr>
<tr>
<td>Ethylene Glycol Dimethyl Ether (DME) CAS 110-71-4</td>
<td>105645</td>
<td>Kit, Recharge, Purification Cartridge, Ethylene Glycol Dimethyl Ether (DME)</td>
</tr>
<tr>
<td>Toluene CAS 108-88-3</td>
<td>105646</td>
<td>Kit, Recharge, Purification Cartridge, Toluene</td>
</tr>
<tr>
<td>Triethylamine CAS 121-44-8</td>
<td>105647</td>
<td>Kit, Recharge, Purification Cartridge, Triethylamine</td>
</tr>
<tr>
<td>Acetone CAS 67-64-1</td>
<td>105648</td>
<td>Kit, Recharge, Purification Cartridge, Acetone</td>
</tr>
<tr>
<td>Acetonitrile CAS 75-05-8</td>
<td>105649</td>
<td>Kit, Recharge, Purification Cartridge, Acetonitrile</td>
</tr>
<tr>
<td>Benzene CAS 71-43-2</td>
<td>105650</td>
<td>Kit, Recharge, Purification Cartridge, Benzene</td>
</tr>
<tr>
<td>Diisopropylethylamine CAS 7078-68-5</td>
<td>105651</td>
<td>Kit, Recharge, Purification Cartridge, Diisopropylethylamine</td>
</tr>
<tr>
<td>Dimethylformamide (DMF) CAS 68-12-2</td>
<td>105652</td>
<td>Kit, Recharge, Purification Cartridge, Dimethylformamide (DMF)</td>
</tr>
<tr>
<td>Dimethyl Sulfoxide (DMSO) CAS 67-68-5</td>
<td>105653</td>
<td>Kit, Recharge, Purification Cartridge, Dimethyl Sulfoxide (DMSO)</td>
</tr>
<tr>
<td>Chloroform CAS 67-66-3</td>
<td>105654</td>
<td>Kit, Recharge, Purification Cartridge, Chloroform</td>
</tr>
<tr>
<td>Dichloromethane CAS 75-09-2</td>
<td>105655</td>
<td>Kit, Recharge, Purification Cartridge, Dichloromethane</td>
</tr>
<tr>
<td>Methyl Tert-Butyl Ether (MTBE) CAS 1634-04-4</td>
<td>106011</td>
<td>Kit, Recharge, Purification Cartridge, Methyl Tert-Butyl Ether (MTBE)</td>
</tr>
<tr>
<td>Benzotrifluoride CAS 98-08-8</td>
<td>106012</td>
<td>Kit, Recharge, Purification Cartridge, Benzotrifluoride</td>
</tr>
<tr>
<td>Dioxane CAS 123-91-1</td>
<td>106013</td>
<td>Kit, Recharge, Purification Cartridge, Dioxane</td>
</tr>
<tr>
<td>Pyridine CAS 110-86-1</td>
<td>106014</td>
<td>Kit, Recharge, Purification Cartridge, Pyridine</td>
</tr>
<tr>
<td>Solvent</td>
<td>Part Number</td>
<td>Description</td>
</tr>
<tr>
<td>---------------------------------</td>
<td>-------------</td>
<td>-------------------------------------------------------</td>
</tr>
<tr>
<td>Heptane CAS 142-82-5</td>
<td>106330</td>
<td>Kit, Recharge, Purification Cartridge, Heptane</td>
</tr>
<tr>
<td>Ethanol CAS 64-17-5</td>
<td>104445</td>
<td>Kit, Recharge, Purification Cartridge, Ethanol</td>
</tr>
<tr>
<td>Dodecane CAS 112-40-3</td>
<td>104447</td>
<td>Kit, Recharge, Purification Cartridge, Dodecane</td>
</tr>
<tr>
<td>Diethylformamide (DEF) CAS 617-84-5</td>
<td>104449</td>
<td>Kit, Recharge, Purification Cartridge, Diethylformamide (DEF)</td>
</tr>
<tr>
<td>Butanol CAS 71-36-3</td>
<td>104451</td>
<td>Kit, Recharge, Purification Cartridge, Butanol</td>
</tr>
<tr>
<td>Cyclohexane CAS 110-82-7</td>
<td>104453</td>
<td>Kit, Recharge, Purification Cartridge, Cyclohexane</td>
</tr>
<tr>
<td>Chlorobenzene CAS 108-90-7</td>
<td>104455</td>
<td>Kit, Recharge, Purification Cartridge, Chlorobenzene</td>
</tr>
<tr>
<td>Propylene Carbonate CAS 108-32-7</td>
<td>104457</td>
<td>Kit, Recharge, Purification Cartridge, Propylene Carbonate</td>
</tr>
<tr>
<td>Dimethyl Acetamide CAS 127-19-5</td>
<td>104459</td>
<td>Kit, Recharge, Purification Cartridge, Dimethyl Acetamide</td>
</tr>
<tr>
<td>Hexamethyldisiloxane CAS 107-46-0</td>
<td>105416</td>
<td>Kit, Recharge, Purification Cartridge, Hexamethyldisiloxane</td>
</tr>
<tr>
<td>Dichloroethane CAS 107-06-2</td>
<td>107468</td>
<td>Kit, Recharge, Purification Cartridge, Dichloroethane</td>
</tr>
<tr>
<td>N-Methyl-2-Pyrrolidone</td>
<td>109104</td>
<td>Kit, Recharge, Purification Cartridge, N-Methyl-2-Pyrrolidone</td>
</tr>
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<td>Hexene CAS 592-41-6</td>
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APPENDIX B – TECHNICAL INFORMATION

WIRING DIAGRAM
FLOW DIAGRAM (Systems with glovebox dispenser and solvent circulation loop)
FLOW DIAGRAM (Systems with glovebox dispenser and one line connection)
APPENDIX C – LEAK TESTING

The following sets of instructions describe the method for leak testing the SPS upon initial startup. The SPS should be leak tested before any solvent is added to the system.

**WARNING!!**
Perform the leak test BEFORE filling the SPS with solvent. Although the SPS has been leak tested at the factory, it is possible that unforeseen circumstances (such as damage during shipping) may have caused a leak. Performing the leak test after the reservoir has been filled could cause solvent to leak or spray out of any leaks that may be present.

There are two different methods for leak testing the SPS. The first method (CYLINDER GAS SUPPLY) describes the test used when the inert gas source is gas cylinders. The second method (FACILITY GAS SUPPLY) describes the test used when the inert gas source is a facility gas supply system. Use the method that applies to your setup.

**CYLINDER GAS SUPPLY**

1) Install the solvent purifier following the instructions in Section 2.
2) Connect the inert gas supply to the low pressure regulator.
3) Set the low pressure regulator to its lowest setting so that there is no pressure being supplied (on the VAC supplied regulator, turn the adjustment counter clockwise until it stops).
4) Set the inert gas supply regulator to 30 – 35 PSIG.
5) Set the solvent purifier controls as follows:

![Solvent Purifier Control Settings](image)

- V1 – OFF
- V2 – OFF
- Sparge Gas Valve – OFF
- Vent Valve - CLOSED
- Circulation Pump – OFF
- Vacuum Pump – OFF
- Glovebox Dispensing Valve (if equipped) – OFF
- One Line Diverter Valve (if equipped) - CIRCULATION

6) Close the cylinder regulator shut-off valve, then close the gas cylinder valve. This will test for leaks between the cylinder and the regulator. Watch the pressure on the cylinder regulator high-side gauge for 5 minutes. If the pressure drops, there is a leak. If there is no leak indicated, go to the next step.
7) Open the cylinder valve and the cylinder regulator shut-off valve. Allow the pressure on the regulator gauges to stabilize.
8) Close the cylinder valve. This will test for leaks in the gas line up to the low pressure regulator. Watch the pressure on the cylinder regulator high-side gauge for 5 minutes. If the pressure drops, there is a leak. If no leak is indicated, go to the next step.
9) Open the cylinder valve and set the low pressure regulator to 5 PSIG.

10) Close the cylinder regulator shut-off valve. This will test for leaks between the low pressure regulator and the solvent purifier(s). Watch the pressure on the low pressure regulator gauge for 5 minutes. If the gas pressure on the low pressure regulator gauge drops, there is a leak. If no leak is indicated, go to the next step.

NOTE: If you have a system with multiple solvent purifier units, perform steps 11 and 12 on the first unit, return its controls to the settings in step 5, then repeat steps 11 and 12 for the next unit in line.

11) Open the cylinder regulator shut-off valve. Set the SPARGE GAS valve to the ON position and set the V1 valve to the INERT GAS position.

12) Close the cylinder regulator shut-off valve. This will test for leaks in the solvent purifier. Watch the pressure on the low pressure regulator gauge for 5 minutes. If the pressure on the low pressure regulator drops, there is a leak. If no leak is indicated, open the regulator shut-off valve and continue with the installation.

FACILITY GAS SUPPLY

1) Install the solvent purifier following the instructions in section 2.

2) Connect the inert gas supply to the low pressure regulator.

3) Set the inert gas supply to 30 – 35 PSIG.

4) Set the solvent purifier controls as follows:

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<td>V2 – OFF</td>
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<td>Sparge Gas Valve</td>
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<td>Vent Valve</td>
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<td>Circulation Pump</td>
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<tr>
<td>Vacuum Pump</td>
<td>OFF</td>
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<tr>
<td>Glovebox Dispensing Valve (if equipped)</td>
<td>OFF</td>
</tr>
<tr>
<td>One Line Diverter Valve (if equipped)</td>
<td>CIRCULATION</td>
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5) Set the low pressure regulator to 5 PSIG.

6) Shut off the inert gas supply. Watch the pressure on the low pressure regulator gauge for 5 minutes. If the pressure on the low pressure regulator drops, there is a leak either in the gas line between the gas supply regulator and the low pressure regulator, or between the low pressure regulator and the solvent purifier. If no leak is indicated, go to the next step.

NOTE: If you have a system with multiple solvent purifier units, perform steps 7 and 8 on the first unit, return its controls to the settings in step 5, then repeat steps 7 and 8 for the next unit in line.

7) Turn on the inert gas supply and allow the pressure to stabilize. Set the SPARGE GAS valve to the ON position and set the V1 valve to the INERT GAS position. Wait for the pressure on the regulator to stabilize.

8) Shut off the inert gas supply. This will test for leaks in the solvent purifier. Watch the pressure on the low pressure regulator gauge for 5 minutes. If the pressure on the low pressure regulator drops, there is a leak. If no leak is indicated, open the regulator shut-off valve and continue with the installation.
APPENDIX D – SOLVENT REMOVAL AND SYSTEM CLEANING

This procedure explains the method for removing solvent from the solvent purifier system and cartridge. It also explains the method for cleaning the system in preparation for use with a new solvent.

WARNING!!
Protective masks, gloves, clothing, etc., may be required when handling and/or breathing the fumes of solvents or chemicals. The nature and extent of protection required depends on the nature of the chemical present in the solvent purifier. The determination of the type and extent of precautions required is the sole responsibility of the user. Always discard contaminated material in a safe and responsible manner in accordance with federal, state, and local regulations.

CARTRIDGE

Tools needed:

- Cartridge Drain Adapter (short “L” shaped piece of 1/4” diameter tubing connected to a female quick-disconnect fitting) – part of the Solvent Purifier Service Kit (VAC 105966)
- Strap Wrench
- Vise or large adjustable wrench (capable of expanding to 1-1/8”)
- Two 9/16” open-end wrenches or small adjustable wrenches

1) Remove the purification cartridge from the SPS as explained in Section 6.5.1.
2) Turn the purification cartridge upside-down and fix it into position so that it can not be knocked over.
3) Connect the 1/4” tube spout on the cartridge drain adapter to a suitable container for the used solvent.
4) Connect the cartridge drain adapter to the quick-disconnect fitting at the top (now on the bottom) of the cartridge.
5) Loosen the Swagelok tube fitting at what is now the top of the cartridge. Be sure to use a backing wrench on the base of the Swagelok fitting so that it does not rotate. Remove the tube from the Swagelok fitting. This will allow the solvent to drain from the cartridge.
6) After the solvent has drained from the cartridge, remove the cartridge drain adapter and use one of the following methods to open the cartridge:

   A. If a vise is available:
      1. Turn the cartridge upside-down and clamp the ¾ NPT fitting (on the top of the purification cartridge) in the vise.
      2. Use a strap-wrench or other suitable means to turn the cartridge counterclockwise, loosening the ¾ NPT fitting. When the ¾ NPT fitting can be turned by hand, remove the cartridge from the vise.

   B. If no vise is available:
      1. Place the cartridge on its side on a bench and put the strap wrench on the cartridge body as if you were going to turn it clockwise.
      2. Use the large adjustable wrench to loosen the ¾ NPT fitting (turn counterclockwise) until it can be turned by hand. Use the strap wrench to prevent the cartridge from turning. You may need an assistant to hold the cartridge in place.
7) Remove the ¾ NPT fitting the rest of the way by hand. Pour out and discard the old purification charge. 
NOTE: There may still be some liquid solvent in the cartridge.

8) Clean out the cartridge and its now separate top and bottom fittings with a suitable cleaning solvent 
(such as Acetone or isopropyl alcohol) to remove all traces of the old solvent. Allow the cartridge and 
fittings to dry completely.

9) If the cartridge will be recharged with new purification media, follow the instructions in Section 6.5.3, 
starting from step 6.

10) TOOLS: Clean the cartridge drain adapter with a suitable cleaning solvent (such as Acetone or 
isopropyl alcohol) to remove all traces of the old solvent. Place in a safe area to dry, such as a fume 
hood.

**SOLVENT PURIFIER UNIT**

Tools needed:
- Solvent Purifier Drain Adapter (the long “L” shaped piece of 1/4” diameter tubing with a male quick-
disconnect fitting at the long end) – part of the Solvent Purifier Service Kit (VAC 105966)
- Cartridge Replacement Loop (the “C” shaped piece of 1/4” diameter tubing with male quick-disconnect 
fittings at the top and bottom) – part of the Solvent Purification Service Kit (VAC 105966)
- Set of wrenches (adjustable or fixed) of appropriate size to open or replace the particulate filter housing 
(see step 19)

1) If you have not already done so, remove the purification cartridge from the solvent purifier unit as 
explained in Section 6.5.1.

2) Set the controls as follows:
   A. VENT valve – closed
   B. PUMP switch – off
   C. SPARGE GAS valve – off
   D. V1 valve – off
   E. V2 valve – off

3) Connect the 1/4” tube spout on the solvent purifier drain adapter to a suitable container for the used 
solvent.

4) Connect the solvent purifier drain adapter to the quick-disconnect fitting at the bottom of the solvent 
purifier unit.

5) Open the VENT valve and turn the PUMP switch on. The solvent will now rapidly drain from the solvent 
purifier unit. Watch the solvent stream carefully. When it begins to thin or slow, turn the PUMP switch 
off.

6) Allow the solvent to completely drain from the unit. To speed up the process, you can close the VENT 
valve and open the SPARGE GAS valve to pressurize the system and force the remaining solvent out. 
NOTE: There will still be a small amount of solvent left on the bottom of the reservoir after this process 
is complete.

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**CAUTION!!**

The circulation pump should not be run dry (without any solvent in the system). Doing so for more than a couple of seconds can damage the circulation pump.
7) Reset the controls to their original positions:
   A. VENT valve – closed
   B. PUMP switch – off
   C. SPARGE GAS valve – off
   D. V1 valve – off
   E. V2 valve – off

8) Remove the solvent purifier drain adapter from the bottom quick-disconnect fitting.

9) Install the cartridge replacement loop into the quick-disconnect fittings on the solvent purifier.

10) Remove the vent flange cap from the solvent purifier unit and pour 4 liters of a suitable cleaning solvent (such as Acetone or isopropyl alcohol) into the reservoir. Replace the vent flange cap and tighten the clamp.

11) Connect the solvent purifier dispensing spout to a suitable container for the waste cleaning solvent.

12) Set the PUMP switch to ON. Allow the solvent to circulate for 5 minutes.

13) After the solvent has circulated for 5 minutes, set the V2 valve so that it is pointing left. This will allow the cleaning solvent to flow from the dispensing spout, cleaning the remaining lines that are normally exposed to solvent.

14) Allow solvent to flow from the dispensing spout until it appears clean, then set the V2 valve to the OFF position.

15) Set the PUMP switch to OFF.

16) Remove the cartridge replacement loop from the quick-disconnect fittings on the solvent purifier.

17) Connect the solvent purifier drain adapter to the quick-disconnect fitting at the bottom of the solvent purifier unit (verify that adapter is connected to a suitable drain) and repeat steps 5 through 8 of this procedure.

18) At this point, the solvent purifier is clean but will have a small amount of the cleaning solvent at the bottom of the reservoir. This can be removed through evaporation by one of two methods:
   A. Remove the vent flange cap and place the solvent purifier into a fume hood. Wait until the remaining solvent is completely evaporated from the bottom of the reservoir.
   OR
   B. Purge inert gas through the solvent purifier by following the sparging instructions in Section 3.5. Purge as long as necessary. When complete, remove the vent flange cap and visually inspect the reservoir to make sure that all of the remaining solvent has been evaporated.

19) When all cleaning is complete, replace the particulate filter located between the dispensing spout and the front panel of the solvent purifier unit. This can be done either by opening the filter housing and replacing the element, or by removing the filter housing and replacing it with a new one. If using the second option, be sure to reseal the connections using ¼” PTFE tape, making sure the first thread is free of tape.

CAUTION!!
Do not use sealant made from any material other than PTFE (Teflon) as it may react with the solvent and cause a leak in the solvent purifier.

20) At this point, the solvent purifier can be treated as a new unit and started up according the instructions in Section 3.

Note: If you will use a different solvent than the one previously used, obtain the appropriate solvent labels from VAC and replace the existing labels located on the solvent purifier unit and the cartridge.
21) TOOLS: Clean the **solvent purifier drain adapter** with a suitable cleaning solvent (such as Acetone or isopropyl alcohol) to remove all traces of the old solvent. Loosen the Swagelok connections to remove the quick-disconnect fittings from the “C” shaped tubing on the **cartridge replacement loop**. Clean the fittings and tubing with cleaning solvent. Place in a safe area to dry, such as a fume hood. After the parts are dry, reassemble the cartridge replacement loop.
APPENDIX E – CIRCULATION PUMP TROUBLESHOOTING AND REPLACEMENT

Follow this procedure in the event that the circulation pump does not run when the pump switch is turned ON. Refer to the diagrams at the end of this section for identification of components.

ELECTRICAL CHECKOUT

If the circulation pump is not running, it is likely that the problem is caused by a faulty electrical connection, rather than a defective circulation pump. Follow these instructions to find or eliminate basic connection problems.

1) Verify that the power supply connector is completely connected into the 24 VDC power jack on the back of the SPS.
2) Verify that the AC power cord is completely connected into the power supply and that it is plugged into a working power outlet.
3) Verify that the two wire connections on the back of the circulation pump power switch are completely connected.
4) If the items above are all correct, proceed to the next step.
5) Disconnect the AC power from the power outlet.
6) Disconnect the power supply from the 24 VDC power jack.
7) Remove the pump cover from the back of the solvent purifier. This is secured by two sets of nuts, lock washers and flat washers, and one screw and washer.
8) Examine the wiring on the terminal strip mounted inside the pump cover. Verify that there are no loose wires. Also verify that none of the terminals are crimped onto the wire’s insulation (instead of contacting the wire directly).
9) Verify that the wires are connected according to the wiring diagram shown in Appendix B.
10) If everything listed above is correct, and the circulation pump still does not run, then the problem is most likely a defective pump. See the instructions in the following sections for replacing the pump.

SOLVENT DRAINING

Before disconnecting the pump from the tubing, any solvent present must be removed from the system.

Special tools needed:

- Solvent Purifier Drain Adapter (a long “L” shaped piece of 1/4” diameter tubing with a male quick-disconnect fitting at the long end) – part of the Solvent Purification Service Kit (VAC 105966)

WARNING!!

Protective masks, gloves, clothing, etc., may be required when handling and/or breathing the fumes of solvents or chemicals. The nature and extent of protection required depends on the nature of the chemical present in the solvent purifier. The determination of the type and extent of precautions required is the sole responsibility of the user. Always discard contaminated material in a safe and responsible manner in accordance with federal, state, and local regulations.

1) Remove the purification cartridge from the solvent purifier unit as explained in Section 6.5.1.
2) Set the controls as follows:
A. VENT valve – closed  
B. PUMP switch – off  
C. SPARGE GAS valve – off  
D. V1 valve – off  
E. V2 valve – off

3) Connect the 1/4” tube spout on the solvent purifier drain adapter to a suitable container for the used solvent.

4) Connect the solvent purifier drain adapter to the quick-disconnect fitting at the bottom of the solvent purifier unit.

5) Open the VENT valve. The solvent will now drain from the solvent purifier unit. This may take several minutes.

6) Allow the solvent to completely drain from the unit. NOTE: There will still be a small amount of solvent left on the bottom of the reservoir after this process is complete. This can be removed through evaporation by one of two methods:
   
   F. Remove the vent flange cap and place the solvent purifier into a fume hood. Wait until the remaining solvent is completely evaporated from the bottom of the reservoir.
   
   OR
   
   G. Purge inert gas through the solvent purifier by following the sparging instructions in Section 3.5. Purge as long as necessary. When complete, remove the vent flange cap and visually inspect the reservoir to make sure that all of the remaining solvent has been evaporated.

7) TOOLS: Clean the solvent purifier drain adapter with a suitable cleaning solvent (such as Acetone) to remove all traces of the old solvent. Place in a safe area to dry, such as a fume hood.

CIRCULATION PUMP REPLACEMENT

Follow these instructions to replace a defective circulation pump. At this point, all solvent should be drained from the system.

1) If you have not already done so, disconnect the power supply from the 24 VDC power jack and remove the pump cover from the back of the solvent purifier. This is secured by two sets of nuts, lock washers, and flat washers and one screw and washer.

2) Disconnect the pump’s five electrical leads. Four (white, red, black, and the ground shield) are connected to the terminal strip and one (green) is connected to a ground stud.

3) Completely loosen the two tube fittings on the circulation pump. Use caution, as there may still be some solvent inside the pump.

4) Remove the four sets of mounting hardware securing the pump to the solvent purifier casing.

5) Carefully pull the outer tube away from the tube fitting and remove the pump.

6) Allow any remaining solvent to drain from the pump, then remove the tube fittings.

7) Clean any PTFE tape from the threads on the fittings.

8) Install the tube fittings onto the new circulation pump, using PTFE tape to seal the threads.

CAUTION!!

Do not use sealant made from any material other than PTFE (Teflon) as it may react with the solvent and cause a leak in the system.
9) Crimp spade terminals (22 AWG wire size, #6 screw size) to the white, red, and black pump wires as well as the motor ground shield. Crimp a ring terminal (22 AWG wire size, #10 screw size) to the green pump wire.

10) Place the circulation pump onto the mounting studs. Place the tubing into the fittings on the pump so that the ferrule completely seats into the fitting, then tighten the fitting nuts finger-tight.

11) Install and tighten the pump mounting hardware onto the mounting studs.

12) Tighten the tube fitting nuts by hand until an increase in resistance is felt, then tighten slightly with a wrench.

13) Connect the pump wiring to the terminal strip on the pump cover per the wiring diagram in Appendix B. Connect the green wire to the ground stud.

14) Replace the pump cover and install its mounting hardware.

The pump replacement is now complete. To ensure safe operation, perform a leak test per the instructions in Appendix C. If a leak is indicated, use a liquid leak detector (such as Snoop) on the two tube fittings at the pump to determine the location of the leak. Tighten the fittings further if necessary.
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