

Technical Bulletin AL-258

Pure Solv™ Solvent Purification Systems

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About the Illustrations in this Bulletin

For clarity, some of the manipulations of the Pure Solv Micro valves and ports are photographed with the solvent reservoir outside the cabinet. In actual use, these manipulations would be made with the solvent reservoir installed inside the cabinet.



Installation

Required Materials

1. **Regulated inert gas supply, 15 psig**
- Nitrogen, Argon, or Helium (high purity)
2. **Tubing for inert gas connection**
- PTFE or stainless steel, ¼ in. o.d.
3. **Tubing for vacuum connection**
- Plastic or rubber, ⅜ in. i.d.
4. **Tubing for Port 3 vent line**
- Polypropylene or PTFE, ¼ in. o.d.
5. **Wrenches**
- 7/16 in., ½ in., 9/16 in., 5/8 in.

Action

1. Connect a regulated inert gas supply to Port 5 (bottom connection) using ¼ in. o.d. PTFE or stainless steel tubing. The regulator delivery pressure should be set to 15 psig at the gas cylinder. See the Accessories section of this bulletin for an inert gas cylinder pressure regulator if one is needed for the installation.



2. Connect a vacuum source to Port 6 (top connection) with ⅜ in. i.d. vacuum hose. There is a custom adapter available for connecting multiple Pure Solv™ Micro systems to a single vacuum source. Please contact Sigma-Aldrich Labware for information about these adapters, labware@sial.com.



3. Connect the grounding cable to a ground point by attaching the clamp to a metal cold water pipe or other earth ground. Consult a qualified electrician if in doubt about the earth ground.

Filling and Degassing Solvent

Important Notes:

1. All solvent placed in solvent reservoir should be sparged (bubble degassed) for at least 15 minutes to remove oxygen before running through solvent purification system.
2. The reservoir must be grounded when filling.
3. Tetrahydrofuran must not contain the inhibitor BHT.

Action

1. Make sure no inert gas is being delivered to the system and the reservoir is not under pressure.
 - a. Turn reservoir Valve 1 to the "closed" (middle) position.
 - b. Disconnect the inert gas supply from reservoir Port 3.
 - c. Connect ¼ in. o.d. tubing to the vent line's quick disconnect fitting. This tubing should be venting into a fume hood before inserting the fitting into reservoir Port 3.



2. Fill the reservoir with up to 4 L of HPLC grade solvent via Port 4. A safety funnel may be used to pour the solvent directly into the reservoir or the reservoir may be removed from the Pure Solv Micro cabinet for filling. **NOTE: The reservoir must ALWAYS be grounded for filling if removed from the cabinet. Connect a grounding cable to the reservoir and to a grounding point.** After filling, make sure the reservoir lid is replaced tightly and sealed.

Filling and Degassing Solvent, continued

3. Connect the inert gas supply fitting to reservoir Port 2.



4. Make sure the ¼ in. o.d. tubing from reservoir Port 3 is terminated into a fume hood during the degassing procedure.

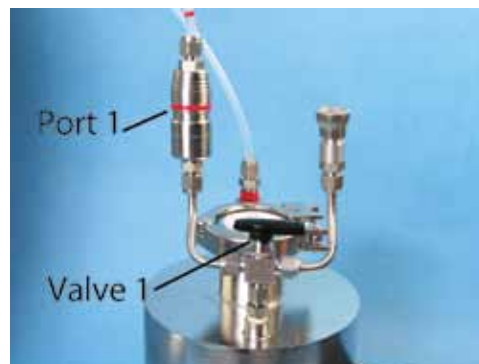


5. Turn reservoir Valve 1 toward Port 2.
6. Degas 4 L of solvent for a minimum of 15 minutes.
7. Turn reservoir Valve 1 to closed (middle) position.
8. Disconnect vent line from reservoir Port 3.
9. Disconnect inert gas line from reservoir Port 2.
10. Connect inert gas line to reservoir Port 3.

Priming the Purification Column

Action

1. Turn reservoir Valve 1 so it is pointing to Port 1.



2. Open column bottom Valve 3.
3. Turn column bottom Valve 5 so that it points towards front panel.
4. Open column top Valve 2.
5. Turn column top Valve 4 so that it points towards front panel.



Dispensing Solvent

1. Adjust the inert gas dispensing pressure to <5 psig from the small regulator located inside the Pure Solv cabinet. Unlock the regulator control knob by pulling outwards to expose an orange color band. Turn knob to set pressure <5 psig, then push knob back in to lock the setting.

NOTE: The Pure Solv Micro system is equipped with a pressure relief valve set to 7 psig to prevent over-pressurizing the collection flask. The inert gas dispensing pressure must be kept below 5 psig to avoid system pressure loss through the relief valve.



2. Check that a vacuum source is connected to Pure Solv Micro and the (pump) exhaust is vented to a fume hood.



3. Attach a suitably configured collection flask to the 24/40 dispensing joint. A septum-inlet flask, solvent storage flask, or storage/dispensing flask is recommended to enable manipulation of the dry solvent or removal of the flask for use at another location. European joint 24/29 flasks will fit provided there is no constriction in flask neck within 11 mm of the bottom of flask joint. Secure flask with a joint clip.

4. Turn valve on front panel to "Evacuate" position.
Note: follow direction of black arrow on valve label.



5. When the flask has been evacuated, turn the same valve to the "Refill" position to fill with inert gas. **Note:** follow direction of black arrow on valve label.
6. Repeat steps 4 and 5 three times ending with "Evacuate".
7. Turn valve to "Dispense" position and leave flask under vacuum condition.
8. Slowly open the (green handled) metering valve allowing solvent to dispense into the flask at the desired rate. Close the metering valve after dispensing the desired amount of solvent.
SAFETY NOTE: Never fill the collection flask more than 2/3 full.

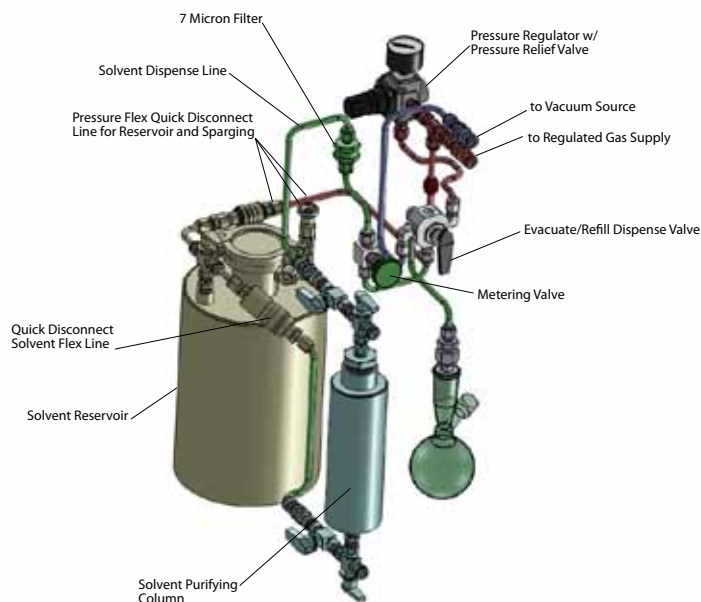


9. Turn valve on front panel to the "Refill" position to back fill the collection flask with inert gas, also clearing dispensing line of solvent and potential dripping after flask removal.
10. Turn valve on front panel to "Closed" position.

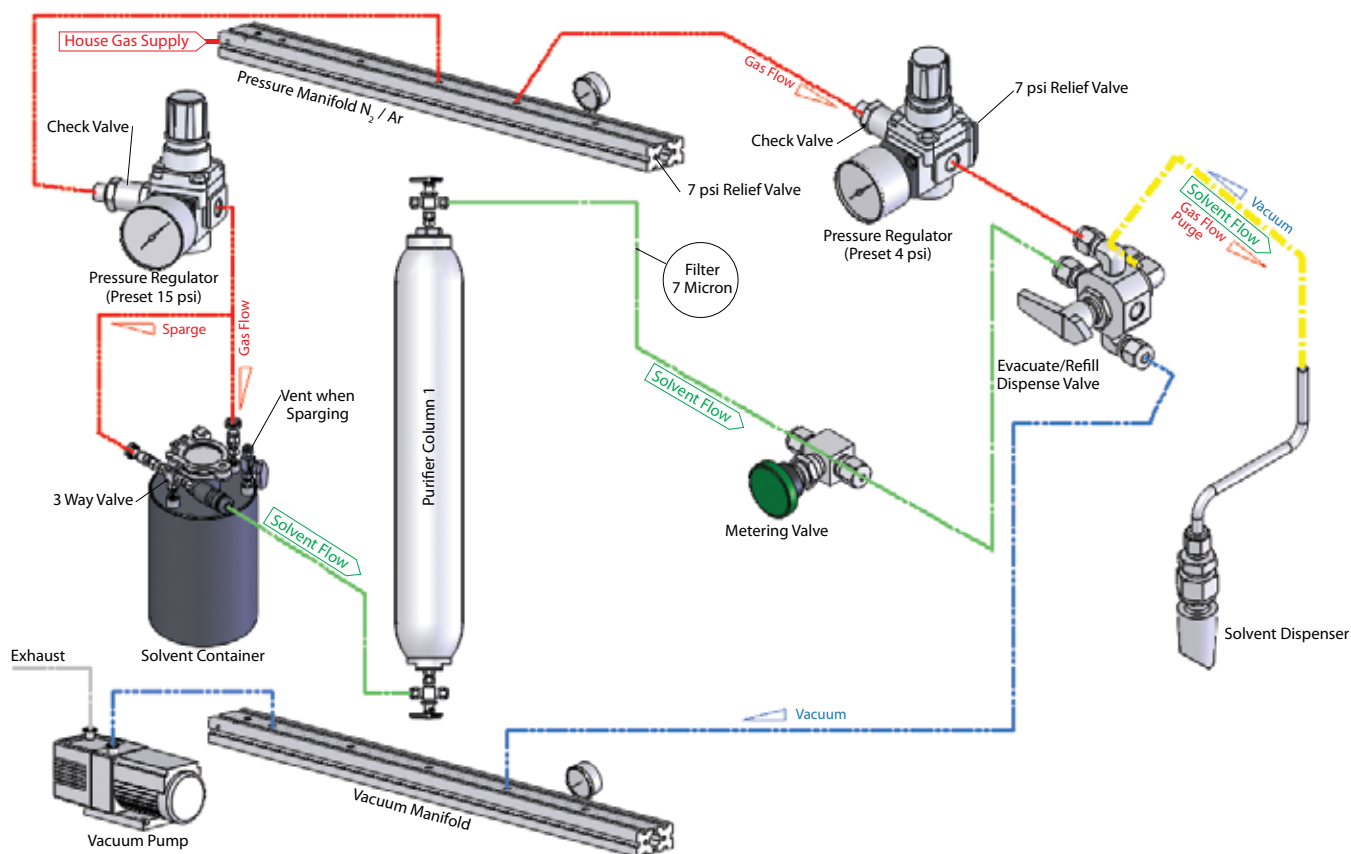
For detailed information about transfer techniques for air and moisture-sensitive reagents and solvents refer to [Aldrich Technical Bulletin AL-134](#).

Pure Solv Micro Specifications:

Item	Description
Frame material	Anodized aluminum
Tubing	Type 304 stainless steel
Valves and fittings	Swagelok®, stainless steel
Leak rates	No detectable leaks with helium mass spectrometer (sensitivity of 5×10^{-6} std cc/sec)
Dispenser fitting	Stainless steel, joint size 24/40
Inert gas dispense pressure	<5 psig
Column packing	Choice of activated alumina, molecular sieves, or Pure Solv media
Column life	~ 100 L HPLC grade solvent
Scrubbing capacity	5 weight % water absorption
Attainable purity	Low ppm H ₂ O and O ₂



Flow Diagram



Choose HPLC grade solvents and appropriate purification systems using our web-based Pure Solv Selection Guide.

For assistance in combining up to four systems together to share vacuum and gas lines, contact your local Sigma-Aldrich sales representative by email or go to sigma-aldrich.com/labware.

Accessories for Use with Pure Solv Micro

These are links to products on the Sigma-Aldrich website which are recommended for use with the Pure Solv Micro systems and for handling air and moisture sensitive reagents and solvents.

Air-Sensitive Products Guide

Bubblers

Chem-Flex Transfer Lines

Glassware Joint Clips

Inert Gas Cylinder Pressure Regulator

Rubber Septa

Schlenk Type Glassware

Septum-Inlet Adapters

Solvent Purification

Spill Tray

Storage Vessels – including Sure/Stor™, Solvent Storage, Septum-inlet Flasks and Storage bottles

NEW! Solvent Storage/Dispensing Flask, Septum-Inlet, with PTFE Inlet Valve

Syringes, Needles, Fittings

Glass Syringes

Non-Coring Syringe Needles

Double-Tipped Syringe Needles

Syringe Fittings & Connectors

Vacuum Equipment

Vacuum Gauges and Manifolds

Vacuum Pumps, KNF Laboport® with PTFE heads

Vacuum Pump/Inert Gas Tee Connector

Vacuum Traps