

MB-SP Series Solvent Purification System



Table of contents

1.	System overview	2
2.	Column activation	5
3.	System purging	6
4.	Glove box integration	7
5.	Solvent reservoir installation	8
6.	Solvent dispensing	9
7.	Servicing columns.....	10
	Figure 1 – Manifold Layout Drawing	11
	Figure 2 – Manifold Plate Controls.....	11

Edition Information

Edition December 2003

14 Marin Way • Stratham, NH 03885
Phone 603-773-9333 • Fax 603-773-0008
Email: info@mbraunusa.com • www.mbraunusa.com
Printed in the United States of America 2003

Title:

Operating Instructions for MBRAUN System
Manual Solvent Purification System

Technical Consultation:

B. Harrington, Production Department, M.Braun, Inc.
K. Prince, Programming, M.Braun, Inc.

Technical Assistance:

P. Cardone, Service Department, M.Braun, Inc.
J. Fairchild, Production Department, M.Braun, Inc.
S. Jewkes, Operations Department, M.Braun, Inc.

Layout:

B. Adams, Production Department, M.Braun, Inc.

Publication edited by means of:

Adobe Acrobat[®], Adobe Photoshop 7.0[®], Microsoft
Word[®]

Important Notes:

This technical documentation is not liable to any obligations on the part of the manufacturer. The manufacturer M.Braun, Inc. reserves the right for technical and optical modifications as well as functional modifications on the system or system's components described therein.

Any duplication of this documentation, even in the forms of excerpts, is only permitted after having obtained the manufacturer's information and concession.

1. MB-SP SERIES/SOLVENT PURIFICATION SYSTEM OVERVIEW

1.1 Description

The M.Braun Solvent Purification System removes oxygen and moisture from a wide range of organic and deuterated solvents quickly and safely. The system can purify up to 400 liters of solvent before requiring service. Purified solvents may be plumbed directly into a glove box or dispensed into receiving flasks. The addition of an optional manifold, facilitates distribution to receiving flasks and the glove box through a series of hand valves. Solvents properly purified by this method test free of oxygen and moisture to the PPM level.

1.2 Method

Degassed solvents are passed through two in-line purification columns via a 5-psi nitrogen push. Activated Alumina serves to remove moisture, while activated copper catalyst removes oxygen. The packing of columns is determined by solvent type. Ethereal solvents and Methylene chloride require the use of double Alumina columns. Activated copper catalyst in the second column allows for deoxygenating hydrocarbon solvents.

1.3 Operation

! NOTE !

Columns are pre-activated and ready for immediate use.

In the event the filter material in the columns needs to be repacked, it will also require activation. Follow the instructions provided in this manual for activating the columns.

A heating mantle is required for the activation process and can be purchased directly from M.Braun. Contact the Service Department for further information.

Activating the columns is necessary after the filter material has been repacked. Columns are heated via an optional heating mantle. Alumina columns are heated under a constant nitrogen purge (5-25 liter/min.), to a temperature of 400 degrees Celsius for six hours. Copper catalyst is heated to 250 degrees Celsius for three hours followed by a 25 liter/min forming gas purge, 5% hydrogen in nitrogen, for two hours. Both columns are then cooled under nitrogen flow and isolated from room atmosphere. The purifier columns are now activated and ready for use.

! CAUTION !

Failure to prevent water vapors in vent gas from condensing in the solvent lines could result in water re-entering the columns during and after activation.

? TECHNICAL SUPPORT ?

For technical assistance, please contact our service department.

☎ (603) 773-9333 ☎

Email: service@mbraunusa.com

1.4 Installing Solvent Reservoir

Prior to the introduction of solvents the entire system, columns and piping, must be purged with nitrogen. Hand valves provide venting outlets so the system may be purged in steps avoiding the introduction of any room air into the system. The solvent reservoirs are reverse purged for 20 minutes to degas the stored solvents. The Nitrogen supply is then switched to the inlet of the solvent reservoirs and a small amount of freshly degassed solvent is purged through the piping prior to the inlet of the first column. The tubing between the reservoir and column must be purged each time the reservoir is changed or refilled. Optional quick connect fittings ease removal and installation of reservoirs.

<i>! CAUTION !</i>	<i>! CAUTION !</i>	<i>! CAUTION !</i>
Reservoir inlet should be vented to a fume hood when degassing the solvents.	Alumina columns may become hot when solvent is first introduced due to heat of absorption.	The tubing between the reservoir and column must be purged each time the reservoir is changed or refilled.

1.5 Solvent Collection

Purified solvent can be drawn into a collection vessel or delivered directly into a glove box. Prior to filling a collection vessel with solvent it must be thoroughly purged of room air via the optional manifold. The collection vessel is evacuated and refilled three times. On the opposite side of the reservoir, a three-way hand valve is supplied with nitrogen and a vacuum source. After the vessel is attached to the outlet side of the purifier columns, it is drawn under vacuum by turning the hand valve to open the vacuum source, then refilled simply by turning the three way hand valve to the nitrogen supply. The vessel is then left in a static vacuum by turning the hand valve to the off position.

Hand valves are opened in sequence starting at the bottom of the first column. Solvent is dispensed into the vessel with a hand valve on the manifold. The vessel should be filled to three quarter capacity to avoid over-pressurization. When collection is complete, the vessel is to be backfilled with nitrogen to compensate for vacuum, then sealed and removed under nitrogen purge to blow out any remaining solvent in the collection tube. Collection tube connections should be properly sealed from room air and kept under vacuum.

<i>! CAUTION !</i>	<i>! CAUTION !</i>	<i>! CAUTION !</i>
Failure to properly purge the collection vessel will contaminate the sample of solvent.	Care should be taken to avoid over pressurization of glass collection vessels.	Failure to blow out collection tube may contaminate the vacuum source with solvent.

When dispensing into a glove box, the hand valve controlling purified solvent flow is turned from the collection tube to the glove box, thereby routing flow to a hand valve installed inside the glove box.

1.6 Servicing Your Solvent Purifier

When the performance of the purifier columns declines, the catalyst charge must be replaced. Reactivation of spent catalyst is not recommended due to the high temperature involved and the fact that removing all residual solvent is virtually impossible.

Begin by disconnecting the solvent reservoirs. Carefully drain each cylinder of residual solvent. Connect a nitrogen or compressed air source to the top of the cylinder and purge through the column into a fume hood to dry the catalyst. After the catalyst has been dried, removal of the bottom inlet valve facilitates disposal of spent catalyst. The columns should be thoroughly cleaned, washed and dried.

After disposal is completed, cleaned columns may then be repacked with the appropriate catalyst and activated as described in the operation section of this manual.

! DANGER !	! CAUTION !	! CAUTION !
Reactivation of used columns may result in personal injury.	Column unpacking should be performed in a fume hood while wearing protective clothing.	Keep away from spark sources and make sure equipment is properly grounded.

? TECHNICAL SUPPORT ?

For technical assistance, please contact our Service Department.
 ☎ (603) 773-9333 ☎
 Email: service@mbraunusa.com

2. Column Activation Procedure

2.1 Prepare Each Column:

1. Close all hand valves.
2. Disconnect all piping/filters from column and remove top hand valve.
3. Install heating mantle.
4. Install nitrogen supply to bottom hand valve.
5. Install vent tube on top of cylinder using ¼" swage stub and place in fume hood.

2.2 Alumina Columns:

1. Open bottom hand valves.
2. Set heating mantle to 400 degrees Celsius.
3. Start nitrogen purge between 5 and 25 liters per minute.
4. Continue purge for six hours.
5. Remove heating mantle.
6. Continue cooling purge for two hours, or until column is cool.
7. Install top hand valve in open position while still purging. Close top hand valve. Close bottom hand valve. Stop purging.
8. Reinstall all pipe work and filters.
9. For more information refer to section 3, **System Purging Procedure**.

2.3 Copper Catalyst Columns:

1. Open bottom hand valves.
2. Set heating mantle to 250 degrees Celsius.
3. Start nitrogen purge between 5 and 25 liters per minute.
4. Continue purge for three hours.
5. Replace nitrogen source with forming gas source (5% hydrogen in nitrogen) at 25 liters per minute.
6. Purge gas supply line of any room air.
7. Continue heating 250 degrees Celsius while purging for two hours.
8. Replace forming gas source with nitrogen source between 5 and 25 liters per minute.
9. Purge gas supply line of any room air.
10. Remove heating mantle.
11. Continue cooling purge for two hours, or until column is cool.
12. Install top hand valve in open position while still purging. Close top hand valve. Close bottom hand valve. Stop Purging.
13. Reinstall all pipe work and filters.
14. For more information refer to section 3, **System Purging Procedure**.

3. System Purging Procedure

- Be sure to keep all hand valves secured in closed position while handling newly activated columns.
 1. Connect all pipe work and filters.
 2. Connect nitrogen source (5 psi) to the bottom hand valve of the first column.
 3. Purge nitrogen supply line by loosening the connection at the bottom of the first cylinder and allowing fresh nitrogen to purge at fitting for one to two minutes, then retighten.
 4. Open the bottom hand valve of the first column by selecting the nitrogen side of the three-way valve.
 5. Allow time for the column to fill with nitrogen, as any additional cooling after the hand valves were closed during activation may have created a vacuum in the column.
 6. Disconnect the pipe work between the top of the first column and the bottom of the second column at the three-way hand valve on the bottom of the second column only.
 7. Open the hand valve on the top of the first column.
 8. Allow pipe to purge for two minutes.
 9. Reconnect pipe work to the three-way hand valve while still purging.
 10. Open the bottom hand valve of the second column by selecting the nitrogen side of the three-way valve.
 11. Allow time for the column to fill with nitrogen, as any additional cooling after the hand valves were closed during activation may have created a vacuum in the column.
 12. Open the top hand valve of the second column.

3.1 Systems Without Optional Manifold:

13. Allow pipe to purge for two minutes.
14. If dispensing into a collection vessel only, seal dispensing connection while purging.

If integrating directly to a glove box continue on to section 4, **Glove Box Integration Procedure**.

3.2 Systems With Optional Manifold:

13. Use the bottom hand valve to select dispensing of solvent to the collection vessel outlet.
14. Use the top hand valve to select collection vessel outlet.
15. Allow pipe to purge for two minutes.
16. Seal dispensing connection while purging.
17. Use the top hand valve to select the manifold setting.

3.2 Systems With Optional Manifold (cont.):

18. Use the manifold hand valve to select the vacuum source to evacuate the dispensing tube thereby removing all room air.
19. Set the top hand valve to the off position.
20. Set the bottom hand valve to the off position
21. If integrating directly to a glove box continue on to section 4, **Glove Box Integration Procedure**

4. Glove Box Integration Procedure

- Nitrogen source should still be hooked up to solvent purifier and all lines and columns purged up to the top of the second column.
 1. Open the hand valve on the top of the second column.

4.1 Systems Without Optional Manifold:

2. Allow pipe to purge for two minutes.
3. Glove box preparations: Turn off circulation.
4. Set the upper and lower pressure set points in a high positive pressure situation (eg. between 8 mbar and 12 mbar).
5. Remove cap from specific solvent feedthrough. (Normally 1/4" Swagelok® bulkhead type)
6. Open the valve inside the glove box to allow glove box atmosphere to purge out the inside line.
7. Allow pipe to purge for two minutes. Note: the glove box will appear to be leaking. This is normal.
8. Attach piping to feedthrough using Swagelok® nut and ferrules. Note: At this point the pressure in the glove box may rise due to the nitrogen being purged through the solvent purifier lines and into the glove box. This is normal.
9. Close the valve inside the glove box to allow glove box atmosphere to return to normal.
10. Reset the glove box system to normal operation as it was before purging the lines.
11. Circulation may now be resumed.
12. Set all hand valves to the off position.
13. Remove the nitrogen source from the bottom of the first column.
14. The solvent purifier is now activated and completely purged.
15. For more information refer to section 5, **Solvent Reservoir Installation Procedure**.

4.2 Systems With Optional Manifold:

2. Use the bottom hand valve to select dispensing of solvent to the glove box outlet.
3. Allow pipe to purge for two minutes.

4.2 Systems With Optional Manifold (cont.):

4. Glove box preparations: Turn off circulation.
5. Set the upper and lower pressure set points in a high positive pressure situation (eg. between 8 mbar and 12 mbar).
6. Remove cap from specific solvent feedthrough. (Normally 1/4" Swagelok® bulkhead type)
7. Open the valve inside the glove box to allow glove box atmosphere to purge out the inside line.
8. Allow pipe to purge for two minutes. Note: the glove box will appear to be leaking. This is normal.
9. Attach piping to feedthrough using Swagelok® nut and ferrules. Note: At this point the pressure in the glove box may rise due to the nitrogen being purged through the solvent purifier lines and into the glove box. This is normal.
10. Close the valve inside the glove box to allow glove box atmosphere to return to normal.
11. Reset the glove box system to normal operation as it was before purging the lines.
12. Circulation may now be resumed.
13. Set all hand valves to the off position.
14. Remove the nitrogen source from the bottom of the first column.
15. The solvent purifier is now activated and completely purged.
16. For more information refer to section 5, **Solvent Reservoir Installation Procedure**.

5. Solvent Reservoir Installation Procedure

1. Supply nitrogen at the reservoir outlet.
2. Open the reservoir inlet to atmosphere inside a fume hood.
3. Reverse purge like this for 20 minutes to degass the stored solvents.
4. Close inlet and outlet.
5. Switch nitrogen supply to the inlet side of reservoir.
6. Purge a small amount of solvent through the supply line.
7. Tighten piping to the bottom hand valve on the first column using Swagelok® nut and ferrules provided.
8. Open hand valves in the following order:
 - a. Select solvent supply on the three-way hand valve on the bottom of the first column.
 - b. Open the two-way hand valve on the top of the first column.
 - c. Select solvent supply on the three-way hand valve on the bottom of the second column.
 - d. Open the two-way hand valve on the top of the second column.
9. After initial setup, allow several hundred milliliters to flow through to remove powdered alumina dust.

5. Solvent Reservoir Installation Procedure (cont.)

10. For systems with optional manifold, refer to section 6, **Solvent Dispensing Procedure**, for more information.

6. Solvent Dispensing Procedure (systems with optional manifold)

1. Use the bottom hand valve to select dispensing of solvent to the collection vessel.
2. When not in use all dispensing connections should be capped and kept under a vacuum.
3. To remove the cap or standby flask, set the top hand valve to Nitrogen/Vacuum and select the nitrogen source with the manifold hand valve. Hold on to the cap when doing this, as it may fall and break when vacuum is relieved.
4. Install the collection vessel to the dispensing tube connection.
5. Switch the manifold hand valve to the vacuum source to evacuate the collection vessel.
6. Evacuate for one minute.
7. Switch the manifold hand valve to the nitrogen source to refill the collection vessel with fresh gas. Hold on to the collection vessel when doing this, as it may fall and break when vacuum is relieved.
8. Repeat steps five (5) through seven (7) twice more.
9. Switch the manifold hand valve to the vacuum source to evacuate the collection vessel.
10. Evacuate for one minute.
11. With the collection vessel under a vacuum, close the top hand valve and set the manifold hand valve to the nitrogen source. This will prevent the introduction of solvents to the vacuum pump.
12. Switch the top hand valve to Fill Collection Vessel and proceed to collect the solvent. Hold on to the collection vessel when doing this, as it may fall and break when vacuum is relieved.
13. Set the top hand valve to the off position.
14. To remove the collection vessel, first set the manifold hand valve to the nitrogen source, then set the top hand valve to Nitrogen/Vacuum.
15. Remove and isolate collection vessel from room air.
 - It is preferable to use a collection vessel with a plug valve in the neck so the vessel may be closed prior to removal from system.
 - If a collection vessel is used that has no plug valve in the neck, it must be removed and quickly stoppered as outlined in steps fourteen (14) and fifteen (15).
16. Allow the nitrogen to blow out any remaining solvent in the collection vessel dispensing tube. This will prevent the introduction of solvents to the vacuum pump.

6. Solvent Dispensing Procedure (systems with optional manifold) (cont.)

16. Replace the cap or standby flask and set the manifold hand valve to the vacuum source to evacuate the line.
17. The system is now set in standby mode.

7. Column Servicing Procedure

1. Connect a nitrogen source to open side of the three-way hand valve on the bottom of the first column.
2. Set the three-way hand valve to the nitrogen source.
3. Using the nitrogen source to push the solvent, dispense available solvents from the system.
4. Continue until only nitrogen purges through the system.
5. Close all hand valves on solvent purifier, solvent reservoirs, and manifold (if equipped).
6. Disconnect the solvent reservoirs from the bottom of the first columns.
7. Disconnect the piping at the top of the first column.
8. Set the bottom hand valve of the first columns to the open port where the solvent reservoirs had been connected.
9. Open the top hand valve to atmosphere to facilitate draining.
10. Drain the columns completely.
11. Connect a vent line between the fume hood and the top of the columns.
12. Set the three-way hand valve on the bottom of the first column to the nitrogen source.
13. Purge the first column for twenty minutes to dry the packing material.
14. Reconnect the piping at the top of the first column.
15. Purge the second column for twenty minutes to dry the packing material.
16. Remove the top and bottom fittings from the columns and extract the material.
17. Thoroughly wash and dry the columns.
18. Thoroughly wash and dry the hand valves, fittings, and filters.
19. Place glass wool inside bottom of columns to act as filter and to hold packing material in the columns.
20. Repack the columns with the appropriate packing material.
21. Rewrap the fittings with Teflon tape and reinstall them into the top and bottom of the columns.
22. Reinstall all piping.
23. Activate columns as outlined in the **Column Activation Procedure**, section 2 of this manual.

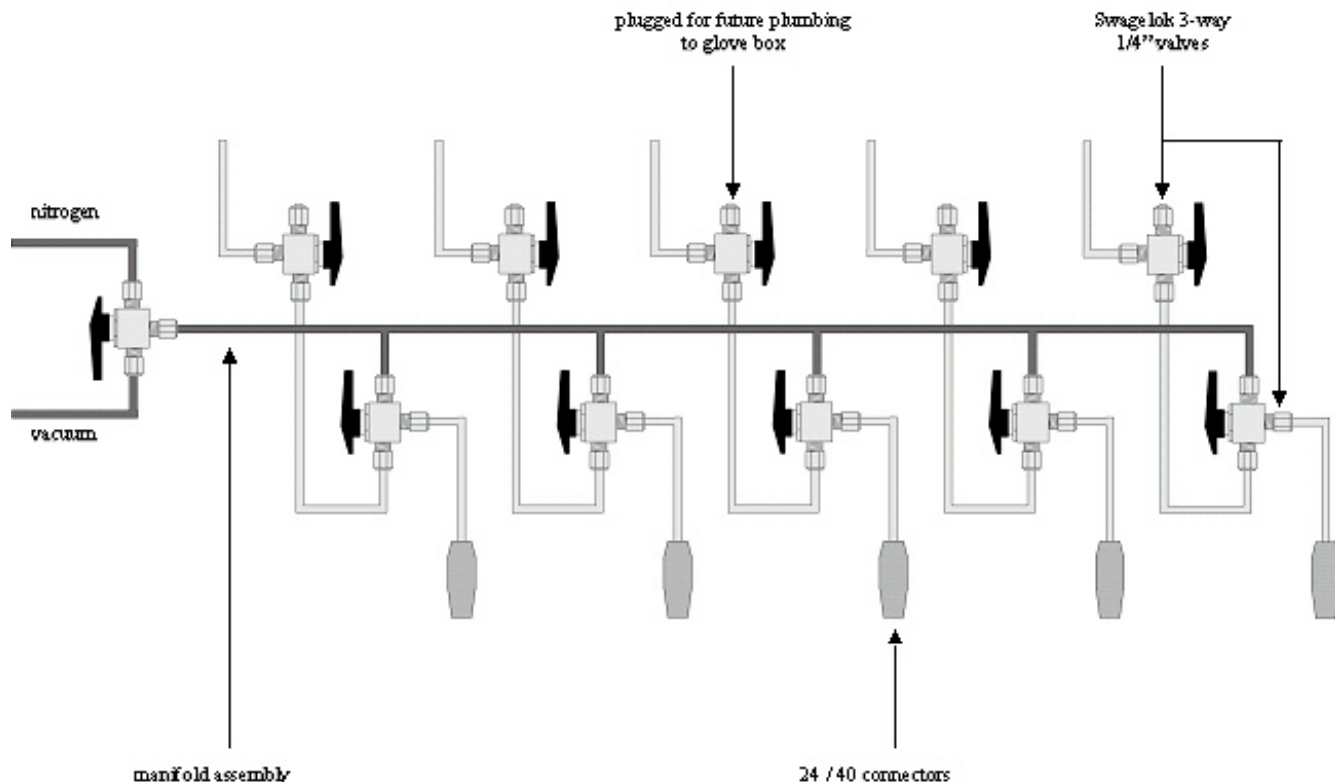


Figure 1 – Manifold Layout Drawing

Note: This diagram is for fluid and gas flow reference only. The actual item is plate mounted for stability.

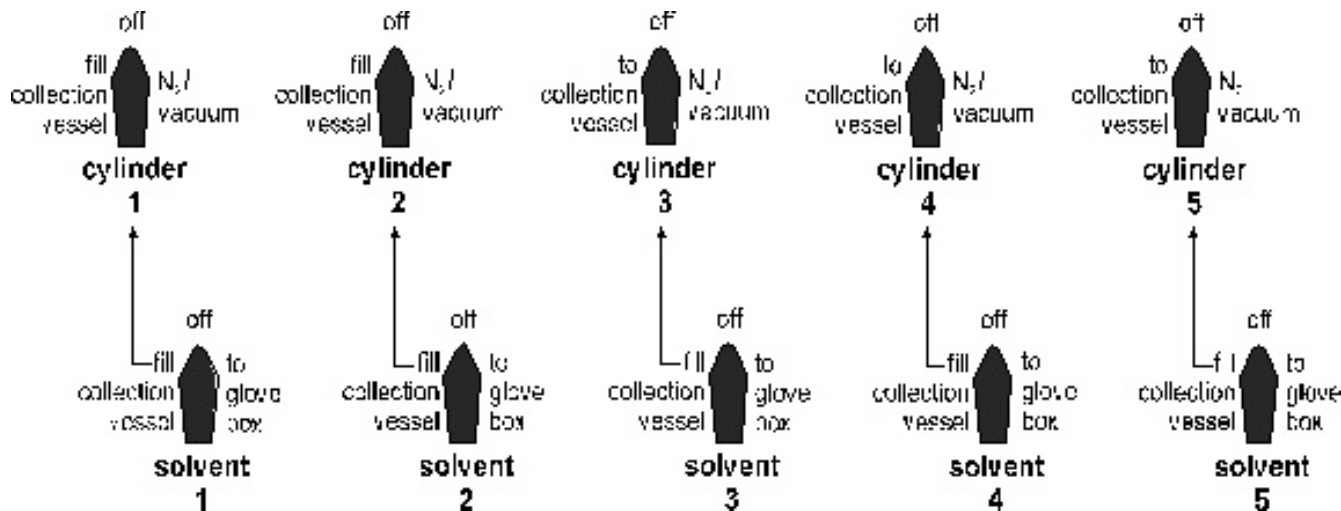


Figure 2 – Manifold Plate Controls

FILLING THE SOLVENT RESERVOIR

General Information

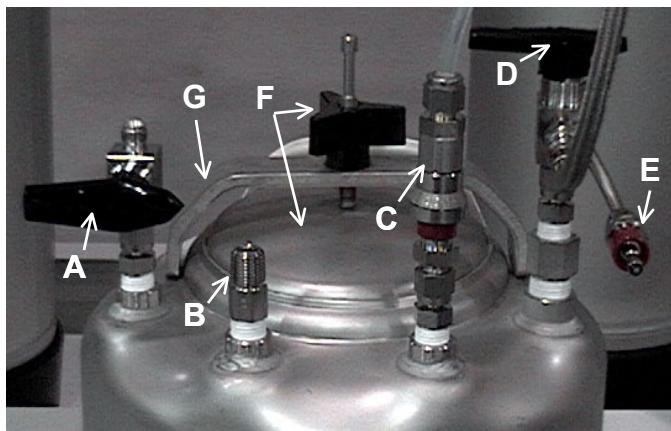
The following information provides instruction for filling the solvent reservoir(s) for a Manual or Automatic Solvent Purification System. Please contact the M.Braun Service Department with any questions regarding the instructions included below.

Valve and Line Types

Below is a picture of the solvent reservoir, one view shows the top and one view shows the front of the reservoir. Please refer to these pictures when following the instructions.



Top View



Front View

- A. Degas Valve
- B. Over Pressure Valve
- C. Nitrogen Push Line
- D. Dip Tube Hand Valve & Solvent Outlet Line
- E. Nitrogen Inlet Line
- F. Black Knob for removing and replacing the Reservoir Cover
- G. Silver Bar Handle for Cover

Hand Valve Position

The hand valves are shaped like arrows which indicate the open and closed positions. When the valve is turned in the direction of a line, it is open. When the valve is turned to the horizontal position it is closed.

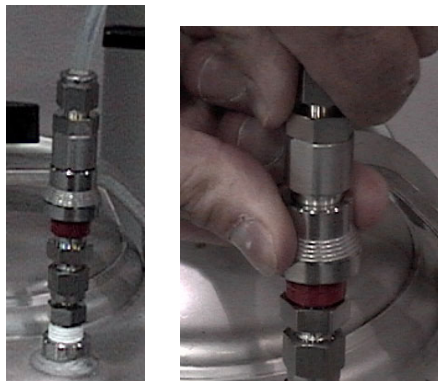
Filling the Reservoir

Close the Degas Valve (A) and Dip Tube Hand Valve (D).



Remove the Nitrogen Push Line (C) by pushing down until it releases and lift off.

Disregard this line until after the reservoir is filled with solvent.

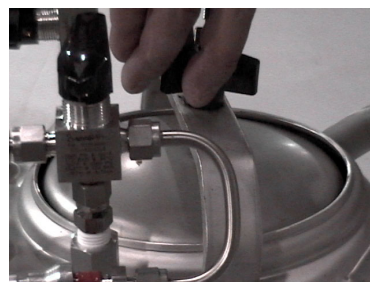


Open the Degas Valve (A) to vent out any pressure in the reservoir.



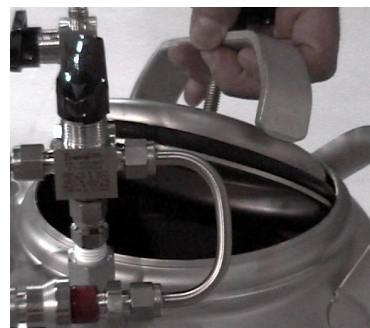
Unscrew the black knob (F) on the reservoir cover by turning it in a counterclockwise direction.

While unscrewing the handle, hold onto the silver bar (G) located directly under it on the cover.



Carefully remove the cover.

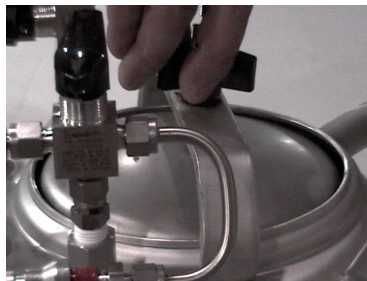
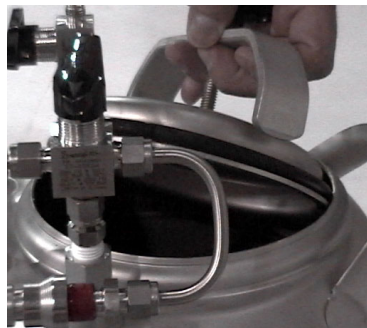
Carefully fill the reservoir with solvent.



Carefully replace the cover and tighten the black knob by turning in a clockwise motion.

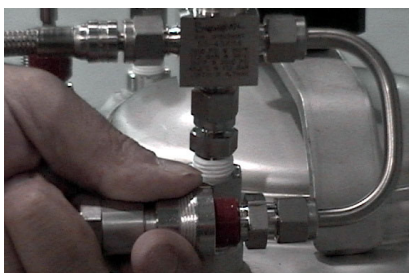
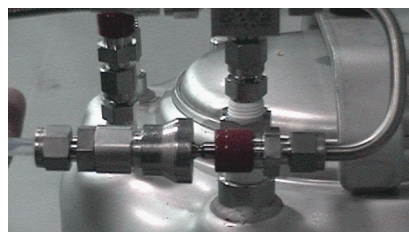
Pull up on the silver bar handle while tightening the knob.

The knob should be tightened hand tight.



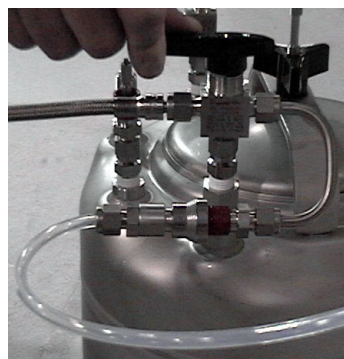
Place the Nitrogen Push Line on the Nitrogen Inlet Line (E).

Push on the valve and release to connect it.



Open the Dip Tube Hand Valve (D) facing the Nitrogen Inlet Line (E).

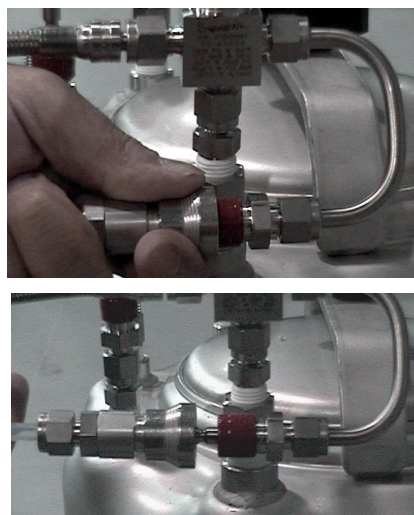
The reservoir will immediately begin filling with Nitrogen to degas the reservoir. This will remove as much Oxygen from the solvent as possible.



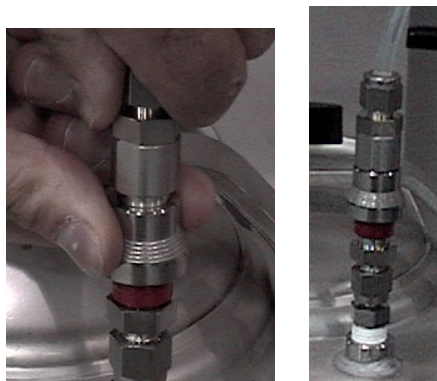
Close the Degas and Dip Tube Valves.



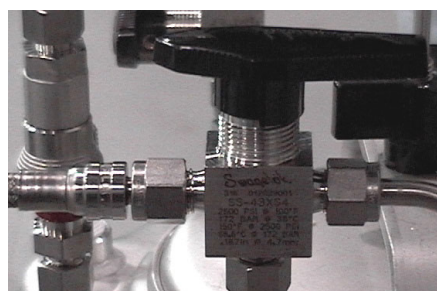
Remove the Nitrogen Push Line by pushing on it to release.

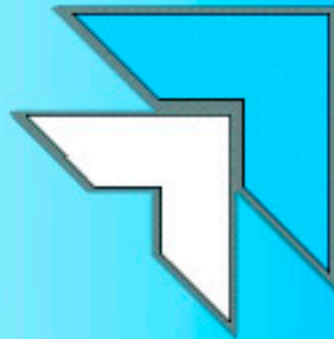


Place the Nitrogen Push Line back in the original position.



Open the Dip Tube Hand Valve towards the Solvent Outlet Line to commence normal operation.





A

MBRAUN

Appendix A
Installing the
Backflow Valve



INSTALLATION INSTRUCTIONS

Index of Appendix A	
Installing the Backflow Valve	
	General InformationA-1
	Tools and Materials Required. A-1
	InstallationA-1

APPENDIX A: INSTALLING THE BACKFLOW VALVE

General Information

The following information provides instructions for installing the backflow valve on the manifold of the Solvent Purification System. Please contact the M.Braun Service Department with any questions regarding the instructions included below.

Tools and Materials Required

Below you will find the listing of tools and materials required to install the backflow valve. None of the tools are supplied by M.Braun. All materials listed are supplied by M.Braun.

Tools:

- 9/16" Wrench

Material

**The materials listed below are supplied as a complete assembly. The list provides information regarding the parts included in the assembly. One assembly per solvent is required.*

- Kalraz Check Valve
- 1/4" Port Connector

Installation

Caution!



- Before beginning installation of the backflow valve:
 - Unplug any existing vacuum pump(s).
 - Unplug any existing heating mantle controllers.
 - Shut off gas supply to the system.

The steps listed below should be repeated in order for each solvent used in the system.



Note!

- The solvent reservoirs shown below may differ from the existing reservoirs. However, the connections are the same.

Locate the Nitrogen Push Line for the solvent (Figure A-1).

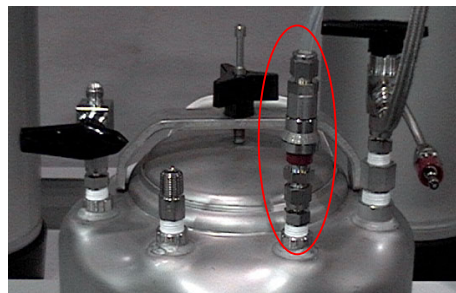


Figure A-1

Remove the Nitrogen Push Line by pushing down until it releases and lift off (Figures A-2 and A-3).



Figure A-2



Figure A-3

Unscrew the Swagelok® nut located on the nitrogen manifold (Figure A-4) using a 9/16" wrench.

Remove the nitrogen line from the fitting.



Figure A-4

Place the backflow valve assembly fitting onto the existing cross (Figure A-5).

Tighten the fitting onto the cross hand tight and make one extra 1/4" turn using a 9/16" wrench.



Figure A-5

Reattach the nitrogen line to the bottom of the fitting.

Tighten the Swagelok® nut on the line to the fitting (Figure A-6) using a 9/16" wrench.



Figure A-6

Place the Nitrogen Push Line back in the original position (Figures A-7 and A-8).



Figure A-7



Figure A-8

Reconnect any disconnected components.
Resume normal operation.