



# UNIVERSITY of LOUISVILLE

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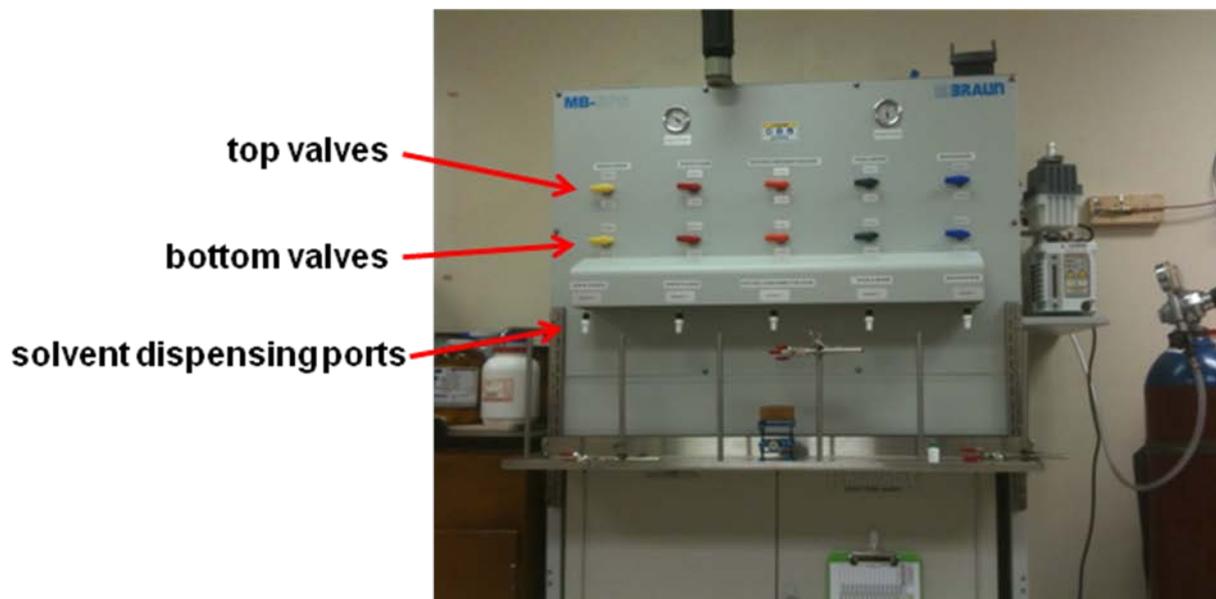
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## **Burns Group Solvent Purification System (SPS) Standard Operating Procedure (SOP)**

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mBraun Solvent Purification System (MB-SPS) is located in Chemistry 339.



### **Introduction to the MB-SPS**

MB-SPS is an instrument that allows removal of oxygen and moisture from up to 5 different organic solvents. It can be used as a stand-alone unit, as in our group, or it can be integrated into an inert atmosphere glovebox. Our system is set to purify (1) benzene, (2) toluene, (3) dichloromethane, (4) pentane, and (5) hexanes. The location of all of these solvents is clearly labeled on the MB-SPS.

The solvents are stored in 17 liter solvent cylinders in the lower solvent cabinet and are stored under nitrogen. The cylinders are connected to the MB-SPS under nitrogen and they remain under a low pressure of nitrogen throughout their operation. During dispensing, the solvent is forced via low nitrogen pressure from the tank through two gas-tight columns filled with anhydrous 3Å molecular sieves as the dessicant. The solvent is collected in a Schlenk flask which can be evacuated and back filled with nitrogen on the MB-SPS. Solvent can also be collected into pre-evacuated solvent storage flasks with the flask neck being evacuated and back filled with nitrogen on the MB-SPS.



Routine use of the MB-SPS is extremely simple, since the user only needs to operate the color-coded valves located on the front panel of the system. It is important to remember, though, **that all operations must be performed slowly and gradually to avoid rapid pressure changes.**

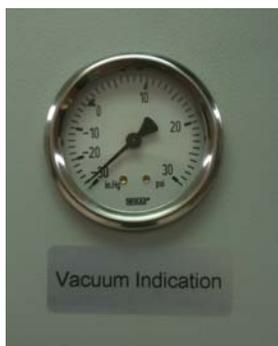
### Before Dispensing any solvent from the MB-SPS.....

- 1) Before you start - make sure to turn on the RV-5 vacuum pump (on right side of MB-SPS) and allow it to run for 30 minutes to properly evacuate the vacuum manifold of the MB-SPS. **When the MB-SPS is not being used the RV-5 vacuum pump should be turned off.** After 30 minutes, check the vacuum gauge located on the left of the front panel; it should read -29 inches mercury (in.Hg).



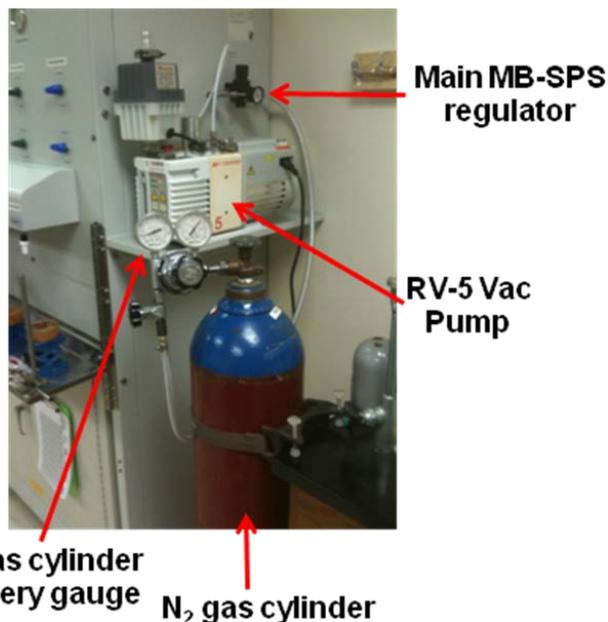
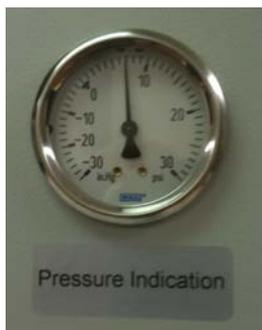
RV-5 Vacuum Pump

Vacuum gauge      Pressure gauge



- 2) The nitrogen used to push solvents from the storage cylinders through their respective drying columns is supplied from a nitrogen gas cylinder (12-14 psi delivery pressure) that is located to the right of the MB-SPS and is attached via 3/8" tygon tubing to the main MB-SPS regulator located above the vacuum pump on the right side of the MB-SPS.

Check the nitrogen pressure gauge located on the right side of the front panel; it should read between 6 and 7 psi. If the pressure is different tell the person in charge of the MB-SPS (currently – **Chris Burns**) and they will correct it by operating the valve located on the right hand side of the system.



## General Information

Dispensing solvent is performed using a **three step process**.

- The first step involves evacuating the collection vessel to create an anaerobic environment.
- The second step is the actual dispensing.
- Step three clears the line so solvent does not remain stored in the dispensing line while the system is not in use.

The flow of solvents during dispensing is regulated using the bottom valve on the front of the system (top figure page #1). The valves are color coded to correspond to the solvent reservoirs and cylinders (see pictures below).



**Solvent 5**  
**HEXANES**  
Yellow

**Solvent 4**  
**PENTANE**  
Red

**Solvent 3**  
**DICHLOROMETHANE**  
Orange

**Solvent 2**  
**TOLUENE**  
Green

**Solvent 1**  
**BENZENE**  
Blue

**NOTICE:** The flow rate of solvent during dispensing is regulated by the position the lower dispensing valve is put in. Maximum flow is achieved when the valve is put in the 6 o'clock (fully open) position. Flow will begin when the valve is put in approximately the 4 o'clock position. Always turn the valve handles using a slow steady motion. ***Turning valves too quickly may result in damage to the Burns group MB-SPS.***

## Dispensing a Solvent to a Schlenk Flask.

- 1) Confirm what size of ground glass joint (14/20 or 24/40) your Schlenk flask has. The Burns group MB-SPS has Teflon adapters for use on both joint sizes. Typically a 14/20 Teflon adapter (**black** top) should be attached to the dispensing port(s) when not in use, but if you need to use a 24/40 Teflon adapter (**green** top) simply disconnect the 14/20 Teflon adapter via its black pressure fitting and replace it with a 24/40 Teflon adapter that is stored in the grey bin in the lower solvent cabinet of the MB-SPS.



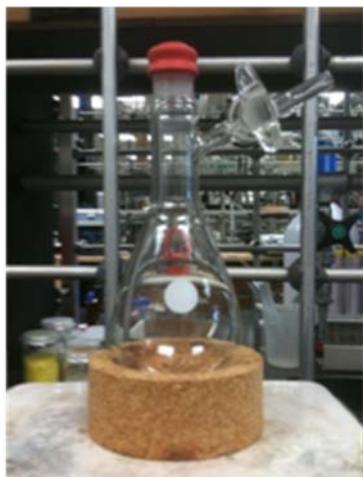
14/20 Teflon adapter



24/40 Teflon adapter

## Dispensing a Solvent to a Schlenk Flask – Evacuation of the Collection Vessel

- 2) Place a clean Schlenk flask that has been oven-dried and cooled (under a positive flow of nitrogen gas on your double manifold via the hose connection) to room temperature under the solvent dispenser. **Avoid the use of grease on the ground glass joint of your flask as this can lead to solvent contamination during collection and leaves residual grease on the Teflon adapter of the MB-SPS.** Use the lab-jack/cork ring set-up along with the 3-prong clamp (connected to the support post) to immobilize the Schlenk flask. *Schlenk flasks are the preferred collection vessel although round bottom flasks can also be used as long as they are clean, oven dried, and cooled with nitrogen gas to room temperature prior to solvent collection.*



oven dried, Schlenk flask  
@ 25 °C under N<sub>2</sub>



lab-jack/cork ring &  
3-prong clamp



attached & immobilized  
Schlenk flask under  
solvent dispenser

- 3) With the Schlenk flask connected, turn the bottom valve counterclockwise to **Evac/Refill** and the top valve clockwise to the **Vacuum** position. Leave the vessel for 2-3 minutes to evacuate. **Note:** *The bigger the vessel, the longer it will take to evacuate.* Check the vacuum gauge and make sure it reads -29 in.Hg after evacuating the flask for several minutes. **Never evacuate a glass collection vessel when there is any solvent present in the vessel.**



- 4) Refill the Schlenk flask with nitrogen by turning the top valve counterclockwise towards **Nitrogen** and hold it at the 1 o'clock position for 2-5 seconds. Turn the valve slowly to prevent a sudden burst of nitrogen into the Schlenk flask.



5) Repeat the evacuation and nitrogen backfill process of the attached Schlenk flask exactly as outlined in step 3 and 4 above.

6) Evacuate the Schlenk flask a third time for 2-3 minutes by turning the top valve clockwise to the **Vacuum** position. Make sure to confirm the vacuum gauge reads -29 in.Hg after the third evacuation of the Schlenk flask.



Turn the top valve counterclockwise to the horizontal, **Neutral** position. Make sure the top valve is horizontal and is not on **Vacuum** or **Nitrogen**. The bottom valve should remain in the **Evac/Refill** position. After the third evacuation process is complete, the solvent can be dispensed for use. **DO NOT** remove the collection vessel. Your Schlenk flask is now under a static vacuum and ready to have solvent dispensed to it.

### Dispensing a Solvent to a Schlenk Flask – Dispensing the Solvent

**CAUTION:** The collection vessel should never be filled to a level more than  $\frac{3}{4}$  of the actual capacity. Filling the vessel to a higher level may result in over pressurization which could cause the collection vessel to rupture (explode).

7) Make sure that the top valve is in the **Neutral** position. To start the solvent flow into the vessel, slowly turn the bottom lever clockwise from **Evac/Refill** to **Dispense**.

**Note:** The flow rate of solvent during dispensing is regulated by the position the valve is put in. Solvent flow will begin when the lower valve is put in approximately the 4 o'clock position. Maximum solvent flow is achieved when the valve is put in 6 o'clock (fully open) position. Keep bottom lever in this position until the desired amount of solvent has been dispensed and then turn the lever slowly to **Evac/Refill**.



8) After the desired amount of solvent has been dispensed to your Schlenk flask turn the bottom valve slowly counterclockwise to the **Evac/Refill** position.



Turn the top valve slowly counterclockwise towards **Nitrogen** and hold it at the 1 o'clock position for 2-5 seconds. This clears the line of solvent and backfills your Schlenk flask with nitrogen gas. Under a positive pressure nitrogen flow carefully remove your Schlenk flask and cap the ground glass joint with a septa.

9) Once your Schlenk flask has been removed from the solvent dispensing port and capped, stop the positive pressure nitrogen gas flow by turning the top valve clockwise back to the **Neutral** position. The bottom valve should remain in the **Evac/Refill** position.



## Dispensing a Solvent to a Schlenk Flask – Clearing the Solvent Dispensing Line

Follow the steps below to clear the solvent dispensing line of any residual solvent to aid in avoiding possible cross contamination.

- 10) Remove your capped Schlenk flask from the 3-prong clamp and place a waste collection vessel (i.e. a beaker) under the solvent dispenser port.



- 11) Make sure the bottom valve is still in the **Evac/Refill** position and turn the top valve counterclockwise to the **Nitrogen** position. Leave the valves in this position for 30 seconds to 1 minute – until no more solvent is dispensed.

**Note:** The times shown are minimum requirements. The valves should remain in these positions until no more solvent is dispensed.



- 12) Once no more solvent is dispensed, turn the top valve to the **Neutral** (horizontal) position. Turn the bottom valve to the **Neutral** (horizontal) position. Remove the waste collection vessel and dispose of the solvent accordingly.



- 13) Record the total amount of solvent just collected (in milliliters – left hand column) and update the running total of solvent collected (in Liters – right hand column) on the **SPS USER LOG** that is attached to a green clip board hanging on one of the doors of the lower solvent cabinet of the MB-SPS.

**IMPORTANT:** Once the running total of any solvent collected reaches 15 liters please notify Dr. Burns immediately so that replacement solvent can be ordered before the storage cylinder is completely empty.

## Dispensing a Solvent to a 500 mL Burns Solvent Storage Flask.

### - Evacuation of the Solvent Storage Flask Neck

- 1) The 500 mL solvent storage flasks used in the Burns group have 14/20 ground glass joints for the connection of the solvent storage flask to a solvent dispensing port on the MB-SPS. Confirm that a 14/20 Teflon adapter (**black top**) is attached to the dispensing port you wish to use.



14/20 Teflon adapter

- 2) Place a clean 500 mL solvent storage flask that has been oven-dried, cooled to room temperature under vacuum (on your double manifold via the hose connection of the flask), and sealed so the flask is under a static vacuum under the solvent dispenser port. As with collecting solvent in a Schlenk flask, **avoid the use of grease on the ground glass joint of your flask as this can lead to solvent contamination during collection and leaves residual grease on the Teflon adapter of the MB-SPS.** Use the lab-jack/cork ring set-up along with the 3-prong clamp (connected to the support post) to immobilize the 500 mL solvent storage flask.



oven dried, solvent storage flask @ 25 °C under vacuum



lab-jack/cork ring & 3-prong clamp

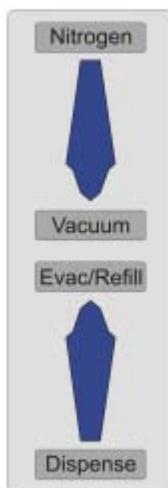


attached & immobilized solvent storage flask under dispenser – side view



attached & immobilized solvent storage flask under dispenser – top view

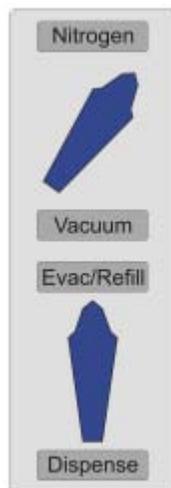
3) With the solvent storage flask connected, turn the bottom valve counterclockwise to **Evac/Refill** and the top valve clockwise to the **Vacuum** position to evacuate the flask's neck. Leave the neck under vacuum for 2-3 minutes. Check the vacuum gauge and make sure it reads -29 in.Hg after evacuating the flask for several minutes.



5) Repeat the evacuation and nitrogen backfill process of the attached solvent flask neck exactly as outlined in step 3 and 4 above

6) Evacuate the solvent flask neck a third time for 2-3 minutes by turning the top valve clockwise to the **Vacuum** position. Make sure to confirm the vacuum gauge reads -29 in.Hg after the third evacuation of the solvent flask neck.

4) Refill the solvent storage flask neck with nitrogen by turning the top valve counterclockwise towards **Nitrogen** and hold it at the 1 o'clock position for 2-5 seconds. Turn the valve slowly to prevent a sudden burst of nitrogen into the solvent flask neck.



Turn the top valve counterclockwise to the horizontal, **Neutral** position. Make sure the top valve is horizontal and is not on **Vacuum** or **Nitrogen**. The bottom valve should remain in the **Evac/Refill** position. After the third evacuation process is complete, the solvent can be dispensed for use. **DO NOT** remove the solvent storage flask.



## – Dispensing the Solvent to a 500 mL Solvent Storage Flask

**CAUTION:** The solvent storage flask should never be filled to a level more than 450 mL. Filling the flask to a higher level may result in over pressurization which could cause the solvent storage flask to rupture (explode).

- 7) Make sure that the top valve is in the **Neutral** position. To start the solvent flow into the flask neck, *slowly* turn the bottom lever clockwise from **Evac/Refill** to **Dispense**. Open the 0-4 mm Teflon plug of the solvent storage flask slightly to allow the static vacuum in the solvent storage flask to draw the solvent collected in the flask's neck into the 500 mL storage volume of the flask. Continue to draw the solvent into the solvent storage flask with vacuum until you have collected 450 mL of solvent.

**Note:** The flow rate of solvent during dispensing is regulated by the position the valve is put in. Solvent flow will begin when the lower valve is put in approximately the 4 o'clock position. Maximum solvent flow is achieved when the valve is put in 6 o'clock (fully open) position. Keep bottom lever in this position until the desired amount of solvent has been dispensed and then turn the lever to **Evac/Refill**.



- 8) After the desired amount of solvent has been dispensed to your solvent flask turn the bottom valve counterclockwise to the **Evac/Refill** position.

Turn the top valve slowly counterclockwise towards **Nitrogen** and hold it at the 1 o'clock position for 2-5 seconds. This clears the line of solvent and backfills your solvent flask with nitrogen gas. Under a positive pressure nitrogen flow close the solvent flask's 0-4 mm Teflon plug and carefully remove your sealed solvent flask from the dispensing port.



- 9) Once your solvent flask has been removed from the solvent dispensing port, stop the positive pressure nitrogen gas flow by turning the top valve clockwise back to the **Neutral** position. The bottom valve should remain in the **Evac/Refill** position.



## – Clearing the Solvent Dispensing Line

Follow the steps below to clear the solvent dispensing line of any residual solvent to aid in avoiding possible cross contamination.

- 10)** Remove your sealed solvent flask from the 3-prong clamp and place a waste collection vessel (i.e. a beaker) under the solvent dispenser port.



- 11)** Make sure the bottom valve is still in the **Evac/Refill** position and turn the top valve counterclockwise to the **Nitrogen** position. Leave the valves in this position for 30 seconds to 1 minute – until no more solvent is dispensed.

**Note:** The times shown are minimum requirements. The valves should remain in these positions until no more solvent is dispensed.



- 12)** Once no more solvent is dispensed, turn the top valve to the **Neutral** (horizontal) position. Turn the bottom valve to the **Neutral** (horizontal) position. Remove the waste collection vessel and dispose of the solvent accordingly.



- 13)** Record the total amount of solvent just collected (in milliliters – left hand column) and update the running total of solvent collected (in Liters – right hand column) on the **SPS USER LOG** that is attached to a green clip board hanging on one of the doors of the lower solvent cabinet of the MB-SPS.

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