Ball Lab Standard Operating Procedure 1

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Scope: This SOP details the procedure and safety considerations for the High Vacuum Manifold.

Kind of SOP: Apparatus.

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Introduction and Purpose:
The high vacuum manifold can be used to manipulate the environment inside a reaction vessel for reactions that require either inert and/or anhydrous environments.

Uses:
This apparatus can be used to a) create an inert and water-free environment in a desired reaction vessel and/or b) to remove excess high-boiling point solvents from desired products.

General Procedure:

Necessary Materials:

- Appropriately-sized reaction vessel (includes vials, Schlenk tubes, round-bottom flasks, etc.)
- Rubber caps
- Appropriate adapters
- Rubber tubing
- Support clamp for flask
- Gas source
- Enough Liquid Nitrogen to fill both dewars

Step-by-step setup: (Note: see picture on next page to see setup)

1. First set up the traps using the appropriate metal pinch clamps and O-rings. Attach your desired reaction vessel to the manifold using rubber tubing and the appropriate adapter/clamp. Make sure the manifold itself is well aligned and clamped, openings in the line will not allow for a proper vacuum to hold.

To use the Vacuum:

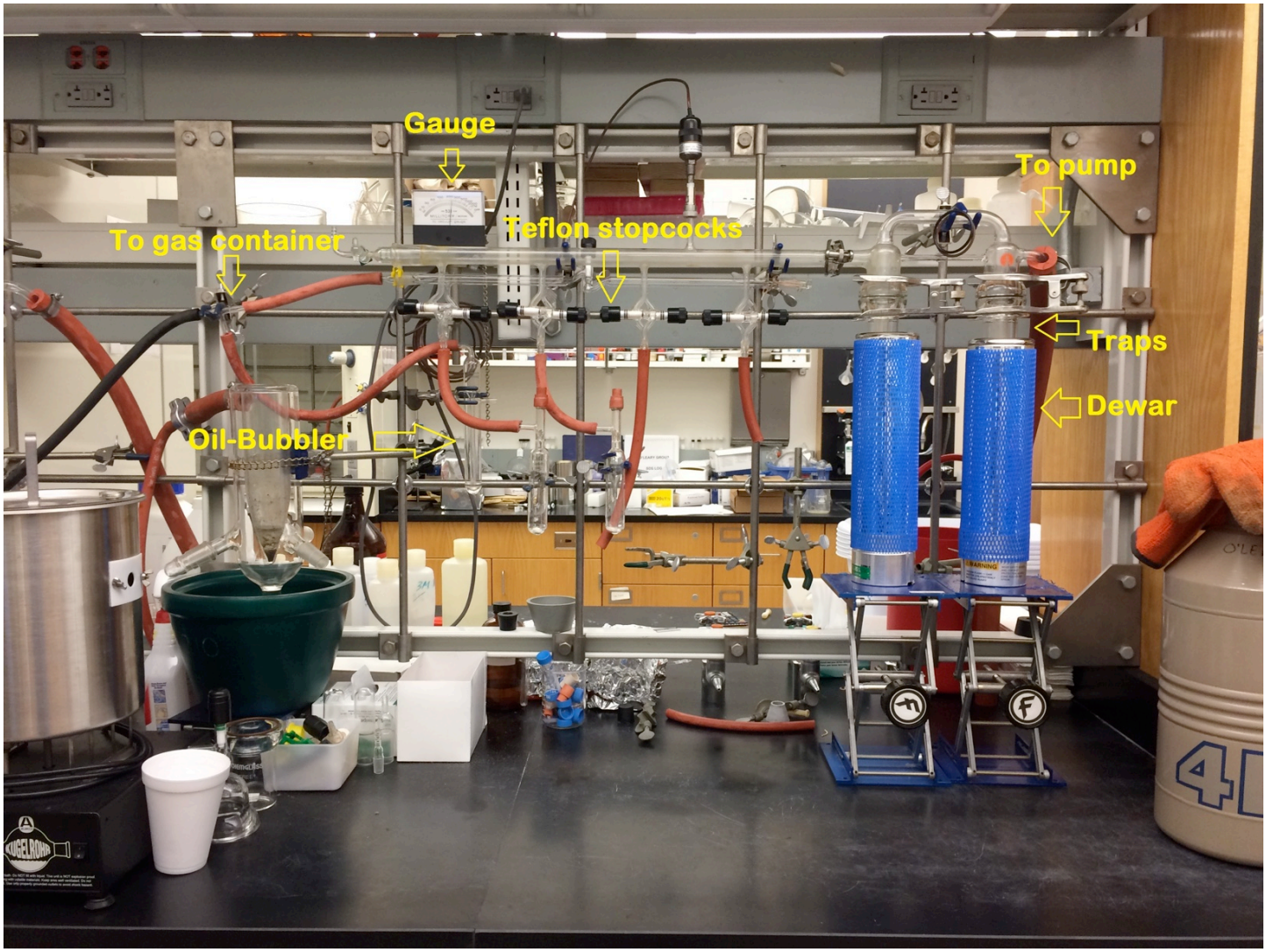
2. Make sure that all Teflon stopcocks are closed and that all vessels are tightly attached/sealed.
3. Turn on the vacuum pump using the switch on the bench located underneath the traps (not pictured). Listen for any abnormal noises, this includes loud gurgling or hissing, this could indicate opened stopcocks or breaks in the vacuum line.
4. Raise up the empty dewars and **FILL** them both with liquid nitrogen. Check the gauge to make sure the pressure is dropping. You will need to monitor the levels of liquid nitrogen every few hours and refill as necessary.
5. Open desired stopcock to the vacuum line and check that the gauge drops back down to an appropriate pressure. If the gauge does not drop back down or down as far, then the reaction vessel may not be attached or sealed correctly causing it to not hold a proper vacuum. Accumulated solvents in the vacuum pump line could also cause poor vacuum hold.

To use the Gas line:

6. In order to use the gas line, the vacuum line **cannot** be open simultaneously to that vessel. Close the desired stopcock to the vacuum line.
7. Turn on the gas source and check the oil-bubbler to make sure you have a steady flow of gas.
8. Open the desired stopcock to the gas line.

To take down:

9. Turn off gas source and close all stopcocks.
10. Drop the dewars and return liquid nitrogen to its container.
11. Turn off the vacuum pump and open one of the stopcocks to allow air to vent the system.
12. Allow the manifold to thaw at room temperature before removing the traps and disposing of any collected solvents.



Safety Considerations and Precautions:

1. **NEVER** have air running through the vacuum line when the traps are still up and filled with liquid nitrogen. **This could condense oxygen and result in an explosion**.
2. Make sure that gas source is closed when an inert atmosphere is not required.