Standard Operating Procedure
Inert Vacuum Line

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Description of Process

The inert vacuum lines located in the fume hoods one (Conjugated Polymer Synthesis: CPS) and two (ATRP polymer synthesis: ATRPPS) (Location FRNY 2182). These pieces of equipment are used to inert reaction vessels with nitrogen (N_2) or Argon (Ar) via flame dry purge/refilling, freeze-pump-thaw or vacuum distillation techniques.

The flame dry purge/refilling technique will inert the atmosphere in the vessel. During evacuation, running the butane flame gun over the glass aids in desorption of oxygen (O_2) as well as water (H_2O) from the interior surface of the glass.

The freeze-pump-thaw technique is often used after the flame dry purge/refilling technique to remove oxygen from injected liquids into the vessel prior to reaction.

The vacuum distillation technique is used for separating or transferring a liquid or solid from a more volatile liquid or solvent. This technique requires the use of the liquid nitrogen trap (LN_2) to condense vacuumed vapors to prevent cavitation in the vacuum pump.

Required Training

Purdue Department of Chemical Engineering Safety Orientation
P.O.W.E.R. Lab safety orientation (including fume hood induction)

Engineering and Ventilation Controls

The inert vacuum lines are located inside of fume hood one (conjugated polymer synthesis) and two (ATRP polymer synthesis) (Location FRNY 2182). Ensure that while working in the fume hoods (regardless of whether it contains a vacuum line), ***the sash must be at least at the minimum working height during experiments!*** This minimizes exposure to the chemicals as well as projectiles from explosion/implosion of vacuum line vessel in case of breach.

Personal Protective Equipment (while using fume hood/vacuum line)

EYE PROTECTION: Safety glasses or goggles (***mandatory!***) ***or face shield, if desired***

PROTECTIVE CLOTHING: Laboratory coat and gloves (nitrile/neoprene) (***mandatory!***)
Thermal insulated gloves for freeze

Use of Vacuum Line (for flame dry/evacuating/refill)

1. Ensure that the vacuum pump is on (switch on back) and the regulator supply pressure to the desired fume hood is ≤ 0.5 bar at all times (refer to **Appendix 2 & 3**).

2. If you wish to use the vacuum, ensure that it is not currently in use and its pressure is ≤ 0.3-0.5 torr (refer to **Appendix 4**). ***Ensure that during experiment AS WELL AS setup, sash is at or below the minimum safe working height!***

3. Ensure that the inert gas line is working properly by attaching a half cut syringe with sheathed needle into a vacant line. Ensure first that ALL other gas/vacuum supply valves on the manifold are closed (close **FINGER TIGHT CLOCKWISE**). Unsheathe the needle and open the BOTTOM valve on your connected syringe line and to test the inert gas flow. If you can feel gas flow from the needle 2-3cm away from your hand, the gas flow is sufficient. If you cannot feel the gas flow, check again to make all other gas/vacuum supply valves in the manifold are closed. If unsuccessful, adjust the inert gas supply line regulator (*in the fume hood*) to get flow. If this does not work, ***CLOSE*** the open gas supply valve ***AND*** the inert gas supply line regulator (*in the fume hood*) ***FIRST BEFORE*** you iteratively adjust the regulator supply pressure to the desired fume hood to ≤ 0.5 bar to get sufficient gas flow.

4. Connect the reaction flask using *thick* walled red rubber tubing to a vacant port. ***Once again, ensure that during experiment AS WELL AS setup, sash is at or below the minimum safe working height!*** (refer to **Appendix 1 & 4**).

5. To use the vacuum, ensure that all connections are on tight (use Keck clamps/rubber septums/vacuum greased stopcocks where required). Ensure first that ALL other gas/vacuum supply valves on the manifold are closed (close **FINGER TIGHT CLOCKWISE**). Open the TOP valve on your connected port to evacuate your reaction vessel. Note: Vacuum line pressure should steadily decrease to below 0.5 torr in a few minutes (approx. <0.1 torr after 5-10 minutes) depending on vessel size. If this does not happen, check your connections and try again.

6. To use the inert gas, ensure that all connections are on tight (use Keck clamps/rubber septums/vacuum greased stopcocks where required). Ensure first that ALL other gas/vacuum supply valves on the manifold are closed (close **FINGER TIGHT CLOCKWISE**). Open the BOTTOM valve (COUNTERCLOCKWISE ~90° on your connected port to fill your reaction vessel. If you are refilling a vessel, only open for 2-3 seconds! You MUST have a relief if you are purging the vessel.

7. While performing steps 5 of 6 (2-3 times), ensure that the line that you are using is isolated from all other reactions using the vacuum line.

8. To use a hotplate for your reaction, immerse the vessel in the silicone oil bath (operating temperatures <200°C). Temperature control can be achieved in one of two different ways. The first method is without the thermocouple. Upon removal of the temperature thermocouple ***AND*** reinsertion of the attached grey plug, temperature control is achieved via adjusting the temperature dial to the desired value via the LED display. The second method is with the thermocouple. Insert the thermocouple into the oil bath/medium you wish to heat. Ensuring that the thermocouple is plugged into the hotplate, adjust the ***thermocouple LED display*** to the ***desired temperature*** (***not*** on the hotplate!). After the desired value has been set, adjust the temperature dial on the hotplate from 250°C to 500°C. The temperature dial on the hotplate is the proportional heat control for the thermocouple controlled heating.

9. After evacuation/refill procedure is complete, ensure that you write your reaction and reactions conditions with contact details on the sash using a marker.

Use of Vacuum Line (Freeze-Pump-Thaw Technique)

1. Ensure that the vacuum pump is on (switch on back) and the regulator supply pressure to the desired fume hood is ≤ 0.5 bar at all times (refer to **Appendix 2 & 3**).

2. Follow the previous procedure outlined in "***Use of Vacuum Line (for flame dry/evacuating/refill***)" section to prepare the vessel for solid/liquid insertion.

3. For solid insertion, break the seal under an inert gas blanket flow and add. Reseal and repeat the "***Use of Vacuum Line (for flame dry/evacuating/refill***)" outlined above.

4. For liquid insertion, inject using a needle under a positive inert gas pressure using a separate syringe needle for pressure relief. Upon completion, remove the relief needle and close the inert supply stopcock to the vessel

5. Wearing thermal gloves, immerse the vessel in the fluid container and leave for five minutes. During this time, ensure that the LN_2 level does not fall below the liquid that you are to freeze. After 5 minutes or when the liquid is observed to be completely

frozen, open the TOP valve (COUNTERCLOCKWISE ~90° on your connected port to evacuate your reaction vessel and the vessel stopcock to vacuum.

6. Upon the LED vacuum gauge displaying less than 0.3 torr, CLOSE the vacuum and isolate your reaction vessel from vacuum. Lift the vessel from the LN_2 bath and immerse in warm water. Do not stir contents during thaw cycle.

7. Upon melting of contents in the vessel and ceasing of bubble formation, repeat steps five though seven two to three more times. Repeating this cycle multiple times will remove oxygen from the reaction vessel.

Use of Vacuum Line (Vacuum Distillation Technique)

1. Ensure that the vacuum pump is on (switch on back) and the regulator supply pressure to the desired fume hood is ≤ 0.5 bar at all times (refer to **Appendix 2 & 3**).

2. Wearing thermal gloves, remove and fill the liquid nitrogen vacuum trap to approx. ¾ full of LN_2 (refer to **Appendix 4**). During this time, ensure that the LN_2 level does not fall below the liquid that you are to freeze. *(Each complete fill of the liquid nitrogen vacuum last from approx. 9-12 hours. If you are using the vacuum line where liquids will be entrained for longer periods of time, you will need to organize to have the liquid nitrogen vacuum trap refilled as long as it is in active use).*

3. After waiting for approx. ten minutes, prepare the vacuum distill head for vacuum distillation, as well as multiple appropriately sized round bottom flasks (r.b.f.). Connect the liquid r.b.f. to be distilled to the evaporator and the collection flask to the side arm. Ensure connection is secure at all times.

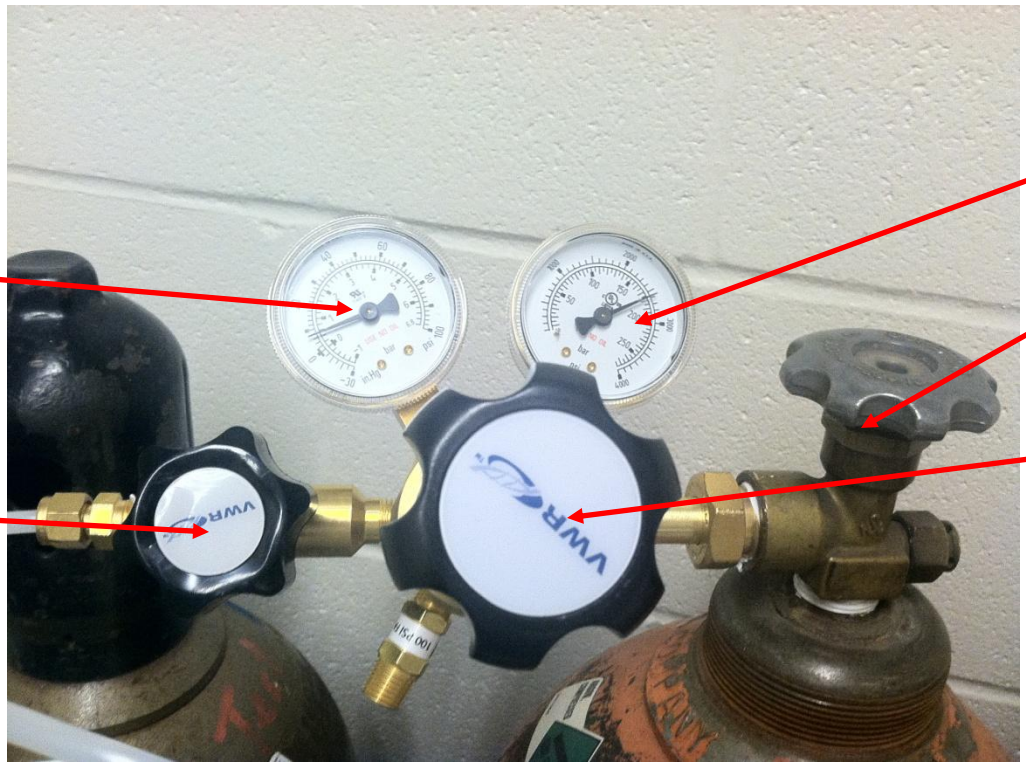
4. Ensure the condenser is off before securing the lines to the side arm of the vacuum head. Ensure to use ties to properly seal both the condenser and vacuum line. Vacuum grease the thermometer and hold into the vacuum head.

5. ***Quickly*** open and close the vacuum line and refill from the inert line to ensure distillation occurs under low concentrations of oxygen.

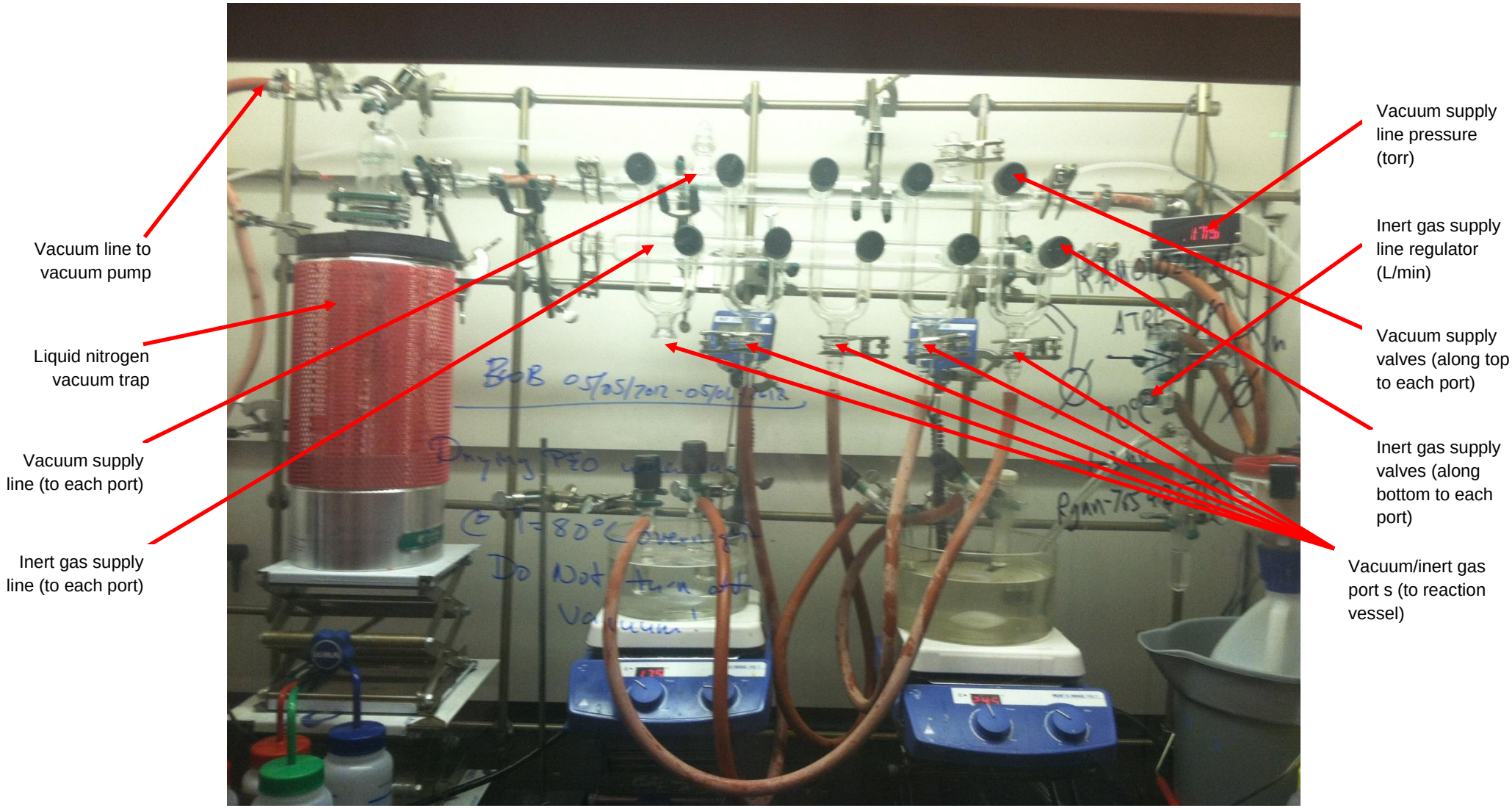
6. If under the presence of heat, immerse the liquid material into thermostat controlled oil bath while under vacuum to distil the required components at the required temperatures/pressures.

Appendices

Appendix 1: Fume hoods containing vacuum lines in FRNY 2182.



Appendix 3: N_2/Ar regulator for vacuum lines.



Appendix 4: Vacuum line setup.