SOP: How to Run an Atmospheric-Pressure Hydrogenation Reaction

Hazards: Hydrogenation reactions pose a significant fire hazard due to the use of flammable reagents and solvents. Such reagents include palladium on carbon (Pd/C), which is highly flammable and can ignite solvents and hydrogen. It is especially dangerous after having been used for the hydrogenation. The presence of hydrogen gas increases the risk of explosion.

Special Precautions: Remove any excess clutter and any flammable solvents that are not needed from your fume hood for the reaction. Be prepared for the possibility of a small fire. Do not panic if this occurs, but simply cover the flask or funnel in which there is a fire with a watch glass and it will go out. *Have a suitable sized watch glass on hand.*

Recommended Apparatus: A three-necked flask equipped with a magnetic stirring bar, a nitrogen inlet adapter connected to a nitrogen/vacuum manifold, a glass stopper or rubber septum, and a gas inlet adapter with a stopcock and a balloon filled with hydrogen.

Procedure:

1. Put a weighed quantity of the catalyst in the flask.

2. Evacuate and back-fill the flask with nitrogen 3 times.

3. Add your solvent under a countercurrent of nitrogen. **CAUTION: Do not pour your solvent from a 4-liter bottle or a 1-liter bottle. Use a small Erlenmeyer flask (for example 125 mL) containing only the needed amount of solvent.**

4. Add your substrate to be hydrogenated to the flask.

5. Evacuate and back-fill the flask with hydrogen.

6. If needed, you may replace the balloon with a full one as needed during the reaction. Remember, it takes 3 molar equivalents of H_2 to hydrogenate a nitro group, which means that a 100 millimole scale reaction will require about three 2L balloons full of hydrogen.

7. If monitoring the reaction is needed, evacuate the flask and back-fill with nitrogen first. Then remove an aliquot under countercurrent of nitrogen. Then evacuate and back-fill with hydrogen.

8. When the reaction is done, evacuate and back-fill with nitrogen.

9. Under countercurrent of nitrogen, pour the contents of your flask into a filter funnel containing celite. Use solvent as needed to wash your flask and the celite. **CAUTION: Do not pour your solvent from a 4 liter bottle or a 1 liter bottle. Use a small Erlenmeyer flask (for example 125 mL) containing only the needed amount of solvent. (Methanol or ethanol are preferable to ether or THF for washing because the pose slightly less fire hazard.)**

10. As soon as you are done, wet the celite and Pd/C with water to reduce the fire hazard from the used catalyst and transfer to a small waste jar just for used catalyst. Keep the used catalyst wet with water and avoid adding flammable solvents.