GUIDELINES FOR CONDUCTING HYDROGENATION REACTIONS

1. Definition of Hydrogenation and Hydrogenolysis Reactions

Hydrogenation reactions are synthetic transformations involving hydrogen, metallic catalysts (such as nickel, platinum or palladium) and organic compounds. The reaction is performed to add hydrogen to sites of unsaturation in an organic compound or to reductively cleave bonds in the compound.

2. Categorization of Hydrogenation and Hydrogenolysis Reactions

Hydrogenation and Hydrogenolysis reactions (H_2 Rx) have been categorized by the University of Pittsburgh Chemistry Department and the Environmental Health and Safety Department, based on the following criteria:

2.1 Level 1 – H_2 Rx conducted under atmospheric pressure via a reaction balloon filled with hydrogen gas and the reaction vessel is a glass vial, flask or bottle; H_2 Rx conducted with a H-Cube ® Continuous-flow Hydrogenation Reactor.

2.2 Level 2 – H_2 Rx conducted at pressure levels not to exceed 60 psi; reaction vessel is a glass flask or bottle contained within a Parr ® shaker or comparable equipment.

2.3 Level 3 – H_2 Rx conducted at pressure levels above 60 psi and below 500 psi; reaction vessel is a sealed metal vessel contained within a Parr ® bomb or comparable equipment.

2.4 Level 4 – H_2 Rx conducted at pressure levels above 500 psi; reaction vessel is a sealed metal vessel contained within a Parr ® bomb or comparable equipment.

2.5 Hydrogenation and hydrogenolysis reactions should not be conducted with the use of Pyrophoric compounds.

3. Gas Cylinder Requirements for Conducting Hydrogenation and Hydrogenolysis Reactions at all Levels

The following gas cylinder safety requirements apply to all levels (1-4) of hydrogenation reactions. Additional safety measures are required as each H_2 Rx level increases.

3.1 All compressed gas cylinders must be stored and used according to University Guidelines for Compressed Gases (EH&S Guideline # 03-012) and includes the following:

3.1.1 All compressed gas cylinders (whether empty or full) must be secured in an upright position using an approved chain, strap, or floor device to prevent falling.

3.1.2 Approved chains or straps should be secured around the top 1/3 of the cylinders. Securing devices should never be around the neck, regulator, or bottom 1/3 of the cylinder.

3.1.3 All compressed gas cylinders must be fitted with a protective valve cap or guard while in storage.

3.2 Hydrogen gas cylinders must be stored and used according to University Guidelines for Flammable and Pyrophoric Gas (EH&S Guideline # 02-004) and includes the following:

3.2.1 Limit the number of hydrogen cylinders to approximately 200 ft^3 or one (1) "K" type cylinders in a laboratory or single fire area.

3.2.2 Adequate ventilation should be provided and maintained throughout the area where hydrogen cylinders are in use.

3.2.3 Provide 20 feet of separation from Class I, II and IIIA flammable liquids, oxidizing gases (such as oxygen) and readily combustible materials (such as cardboard).

3.3 Health hazard 3 or 4 gases (such as hydrogen or carbon monoxide) must be stored and used according to University Guidelines for Storage and Use of Health Hazard (HH) 3 and 4 (EH&S Guideline # 04-021) and guidance is available at the following link, http://www.ehs.pitt.edu/assets/docs/health3-4-gas.pdf.

4. Personal Protective Equipment Requirements for Conducting Hydrogenation and Hydrogenolysis Reactions

4.1 Flame retardant laboratory coat must be worn during all H_2 reaction work; including loading of vessels, entering blast rooms, and removing reaction vessels.

4.2 Safety goggles or safety glasses with side impact shields must be worn during all H_2 Rx work; including loading of vessels, entering blast rooms, and removing reaction vessels.

4.3 Chemical resistant gloves must be worn during all H_2 Rxs work; including loading of vessels, entering blast rooms, and removing reaction vessels.

5. Safety Guidelines for Hydrogenation and Hydrogenolysis Reactions at Level 1

Level 1 H_2 Rx is conducted under atmospheric pressure via a reaction balloon filled with hydrogen gas and the reaction vessel is a glass vial, flask or bottle.

5.1 Level 1 H_2 Rx must be conducted within a certified chemical fume hood.

5.2 Chemical fume hood sashes should remain closed at all times during the reaction process. Hood sashes should only be opened during set-up of the reaction, addition of chemical to the reaction vessel as necessary, and during work-up of the reaction mixture.

5.3 Hydrogen gas cylinder valve must be securely closed after filling of reaction balloon.

5.4 Glass vials, flasks or bottles must meet applicable standards for use at test pressures. Technical information sheets from glass manufacturers should be consulted to determine allowable pressures.

6. Safety Guidelines for Hydrogenation Reactions at Level 2

Level 2 H_2 Rx is conducted at pressure levels not to exceed 60 psi and the reaction vessel is a glass flask or bottle contained within a Parr ® shaker or comparable equipment.

6.1 Level 2 H_2 Rx must be conducted within a certified chemical fume hood; including the loading of all reaction vessels.

6.2 Chemical fume hood sashes should remain closed at all times during the reaction process. Hood sashes should only be opened during set-up of the reaction, addition of chemical to the reaction vessel as necessary, and during break down of the reaction.

6.3 An approved blast shield is recommended within the chemical fume hood, and placed between the reaction vessel and the fume hood sash.

6.4 Glass flasks or bottles must meet applicable standards for use at test pressures. Technical information sheets from glass manufacturers should be consulted to determine allowable pressures.

7. Safety Guidelines for Hydrogenation Reactions at Level 3

Level 3 H_2 Rx is conducted at pressure levels above 60 psi and below 500 psi and the reaction vessel is a sealed metal vessel contained within a Parr ® bomb or comparable equipment.

7.1 Reaction vessel should be equipped with pressure relief valve (PRV) or frangible disk (bursting disk) to allow venting of vessel.

7.2 Level 3 H_2 Rx must be conducted within a certified chemical fume hood; including the loading of all reaction vessels.

7.3 Reaction vessels should be securely closed and loaded within a certified chemical fume hood that is located in the same laboratory as the Parr ® bomb.

7.4 Chemical fume hood sashes should remain closed at all times during the reaction process. Hood sashes should only be opened during set-up of the reaction, addition of chemical to the reaction vessel as necessary, and during break down of the reaction.

7.5 An approved blast shield is required to be used within the chemical fume hood, and placed between the reaction vessel and the fume hood sash.

7.6 Reaction vessels should be opened and unloaded within a certified chemical fume hood that is located in the same laboratory as the Parr ® shaker or bomb after each test cycle.

7.7 Recommended that an approved means of leak detection and an emergency shutoff or excess flow control should be provided when using hydrogen gas in pressurized piping above a gauge pressure of 15 psi (103 kPa).

7.8 Hoses from hydrogen tank to the Parr bomb or equivalent device should be stainless steel braided with PTFE inner core (able to withstand medium to high pressure based on inner diameter of the hoses). Selection of hoses should be based on operating pressure.

8. Safety Guidelines for Hydrogenation Reactions at Level 4

Level 4 H_2 Rx is conducted at pressure levels above 500 psi and the reaction vessel is a sealed metal vessel contained within a Parr ® bomb, or comparable equipment.

8.1 Reaction vessel should be equipped with pressure relief valve (PRV) and frangible disk (bursting disk) to allow venting of vessel.

8.2 Reaction vessel should be securely loaded and closed within a certified chemical fume hood that is located in the same laboratory as the Parr ® or bomb.

8.3 Level 4 H_2 Rx must be conducted within a blast proof room meeting the following criteria:
- 8.3.1 All 4 walls and the ceiling and floor of the rooms utilized for a Level 4 H_2 Rx must be designed for pressure resistant construction.
- 8.3.2 Any openings or penetrations that have been made to the structure of the room should be reviewed for integrity and repaired as needed.
- 8.3.3 Pressure venting capabilities within the room, with the pressure vent directed away from interior walls.

8.4 The integrity of the blast relief wall panel should be reviewed to verify proper operation.
- 8.4.1 Blast panel should be sized based on calculations of the release of pressure from the maximum allowable chemical reaction.
- 8.4.2 If blast relief / shear bolts are utilized, these must be inspected to determine if their integrity for continued use meets applicable standards.
- 8.4.3 If blast relief / shear bolts are utilized, these must be inspected to verify that none of the devices have been replaced with standard bolts.
- 8.4.4 If a different method of release device has been used, this should be inspected for proper operation and functional control.
- 8.4.5 The blast relief panel must be tethered to prevent the panel from becoming a projectile hazard. The tether device should be inspected and verified for continued use.

8.5 Existing exhaust ventilation system should be inspected for proper function and flow rate.

8.6 All electrical equipment and wiring within the blast room should be inspected for proper classification, enclosure type, proper conduit, sealing, etc. for use within a hazardous location; all electric equipment and wiring within 15 feet of the ventilation should be Class1, Div 2, Group B.

8.7 Gas detection should be provided within the blast room and should meet required classification, enclosure type, proper conduit, sealing, etc. for use in a hazardous location.

8.7.1 Gas detection warning lights and alarms must be visible from both inside and outside of the blast room.

8.7.2 Gas detection alarms must meet applicable audible levels.

8.8 All observation ports within the blast room should be inspected to determine structural integrity.

8.9 Computer systems that are required for process control and/or reaction monitoring should be connected to data wiring ports within the blast room and routed outside the room so that the non-rated computer equipment is outside of the hazardous location.

8.10 If hydrogen gas is continuously feed into the reaction system, then an excess flow valve must be installed.

8.11 Personnel should only be in the blast room to load the reaction vessel into the test system and must exit the room after the loading process is complete. Observations of the reaction should be accomplished via the view ports and the external computer system.

8.12 Recommended that an approved means of leak detection and an emergency shutoff or excess flow control should be provided when using hydrogen gas in pressurized piping above a gauge pressure of 15 psi (103 kPa).

8.13 Hoses from hydrogen tank to the Parr bomb or equivalent device should be stainless steel braided with PTFE inner core (able to withstand medium to high pressure based on inner diameter of the hoses). Selection of hoses should be based on operating pressure.

9. Preventive Maintenance of Parr ® bomb

9.1 All bomb vessels should undergo annual routine maintenance including the replacing of all O-rings and valve seats

9.2 A socket and torque wrench should be utilized with the bomb vessel to perform weekly tightening of the packing nut while in use.

9.3 All bomb vessels should be inspected for damage and O-ring condition; any damaged equipment should be tagged out of service and maintenance should be immediately conducted.