

University of Manchester Department of Chemistry
Procedures and Methods Sheet: 008B
Local Rules for the High Pressure Hydrogenation Equipment

Researcher signed:

date:

Supervisor signed:

date:

Departmental Instructor signed:

date:

This P&M sheet is 2 pages long.

Prior to carrying out any hydrogenation experiments the researcher must receive suitable training from the Departmental Instructor, Mrs Rehana Sung. Hydrogenation experiments are carried out in the Special Purpose Laboratory for which a booking procedure is in operation. Each individual experiment must have its own specific COSHH assessment, which must be displayed by the reaction. While the apparatus is situated in the basement, at least two people must be present while carrying out the procedure.

Hazards

1. Hydrogen is harmful, extremely flammable and explosive.
2. At high concentrations, hydrogen functions as a simple asphyxiant by displacing air. Symptoms may include head-ache, fatigue, increased breathing rate, dizziness, muscular un-coordination, nausea, vomiting and loss of consciousness. First aid - Fresh air or oxygen.
3. Hydrogen under pressure - DANGER : Flammable high pressure gas forms explosive mixtures in air.
4. MIXTURES OF HYDROGEN AND ORGANIC VAPOURS CAN IGNITE OR EXPLODE ON CONTACT WITH FINELY DIVIDED RANEY NICKEL, PALLADIUM, PLATINUM IN THE PRESENCE OF OXYGEN. In case of fire use sand to extinguish it. There is a sand bucket nearby.
5. Removal of catalysts/active metals by filtration: these finely divided metals are pyrophoric when dry, hence when being removed by filtration, the filter cake must be kept wet with solvent. After filtration is complete, the wet filter cake containing the active metal should be stored in a separate container, under water, and be returned to the supplier for refining.

Experimental Procedure

Instructions for filling up the Hydrogen Reservoir Tank. (See Figure 1.)

1. Close off the Bottle Valve and the Gas Release Valve on the left hand side of the apparatus before you start.
2. Connect the other end of the Gas Supply Hose to the valve of the hydrogen cylinder.
3. Open the Tank Filling Valve and fill the reservoir up with hydrogen up to a maximum of 60 psi. TAKE CARE.
4. Close the Tank Filling Valve. Do not disconnect the hose from the cylinder as more hydrogen will be required.

Procedure

1. Place the catalyst and sample in the reaction bottle (500 ml or 250 ml). Add the catalyst first to avoid possible vapour ignition by the dry catalyst. The total volume of the solution should not exceed two thirds of the capacity of the bottle.

2. Attach the stopper with the polypropylene tubing to the top of the reaction bottle and slide the bottle into the guard screen. Place the assembly into the bottle holder and tighten the knurled clamping nut.
3. Evacuate any air from the bottle by attaching the vacuum rubber tubing from the water pump to the Exhaust Nipple.
4. Open the Gas Release Valve and evacuate until the solvent starts to boil. Then close the valve.
5. Purge the bottle with hydrogen from the Reservoir (Gas Tank) by opening the Bottle Valve slowly. The Bottle Pressure Gauge reads the pressure inside the reaction bottle.
6. Repeat steps 3 to 5 a couple of times to completely purge the bottle of air with hydrogen. Finally fill the bottle up with hydrogen until both the Bottle Gauge and Tank Pressure Gauge read the same value.
7. Top up the Reservoir Tank (Gas Tank) with hydrogen from the hydrogen cylinder (you do not need to close the Bottle Valve) by opening up the Tank Filling Valve and allow the Reservoir to fill up to a maximum of 60 psi. **DO NOT EXCEED 60 PSI AS THE REACTION BOTTLE CAN ONLY WITHSTAND THIS PRESSURE SAFELY.**
8. The Reservoir Tank has been equipped with a safety valve at the back to withstand a pressure of 70 psi, any pressure higher than this will escape to the back of the tank.
9. Close the Tank Filling Valve and then the hydrogen valve. Release any excess hydrogen and then close the main valve of the hydrogen cylinder. There is a safety pin on the cylinder, when it is up, no hydrogen will be released; this is due to a high back pressure in the cylinder head. This has to be pressed down to access the gas.
10. Start the shaker and follow the reaction by observing the uptake of hydrogen via the Bottle Gauge.

At the end of the experiment

1. Stop the shaker, close the Bottle Valve and allow the catalyst to settle.
2. Remove any residual pressure in the bottle by opening the Gas Release Valve.
3. Do not empty the Reservoir Tank since the hydrogen can be used again.
4. Return the hydrogen cylinder to stores.

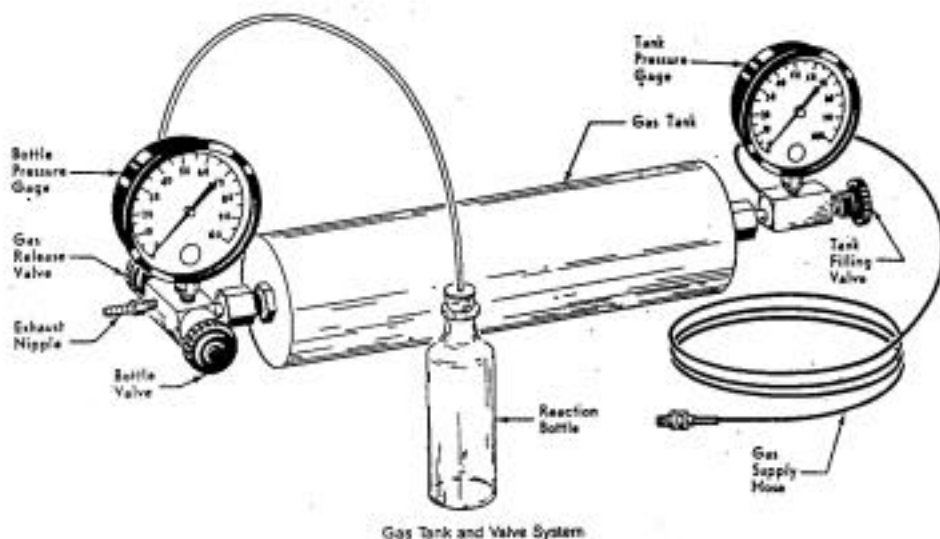


Figure 1. Parr 3119 High Pressure Hydrogenator