CDD Standard Operation Procedures

Hydrogenation Parr Shaker

I. Prepare the reaction mixture

1. Dissolve the reaction substrate in a desired solvent in a pressure reaction vessel provided. Normally the solvent volume should not exceed one-third of the reaction vessel.

2. Flush the reaction vessel with argon.

3. Add catalyst under argon.

II. Assemble the reaction vessel

1. Assemble the reaction vessel on the Parr shaker. Make sure the reaction vessel is protected by the steel shield.

2. Seat the rubber stopper properly and then tighten the two screw clamps evenly.

3. Protect the Parr shaker with shields.

III. Purge the reaction vessel with hydrogen

1. Open the hydrogen cylinder valve.

2. Open the chamber valve (on the right) to fill the chamber with hydrogen at a proper pressure. The pressure should not exceed 60 psi.

3. Open the house vacuum.

4. Open the reaction vessel outlet valve (on the left, back) to evacuate the reaction vessel.

5. Close the reaction vessel outlet valve (on the left, back) and open the reaction vessel inlet valve (on the left, front) and fill the reaction vessel with hydrogen.

6. Close the reaction vessel inlet valve (on the left, front).

7. Repeat the above steps 4-6 several times.

8. After the reaction vessel is filled with hydrogen at a proper pressure, close the hydrogen cylinder valve and start the Parr shaker.
IV. Clean after the reaction

1. After the reaction is complete, close the reaction vessel inlet valve (on the left, front) and the chamber valve (on the right).

2. Open the reaction vessel outlet valve (on the left, back) to evacuate the reaction vessel.

3. Close the house vacuum and open the reaction vessel to air.

4. Remove the reaction vessel from the Parr shaker.

5. Close the reaction vessel outlet valve (on the left, back).

6. Clean the rubber stopper and inlet line with a proper solvent.

7. Make sure to maintain a positive pressure in the chamber.

V. Maintain a proper record in the log book