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Operation of high pressure reactor vessels

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Overview: The use of gases in the synthetic chemistry laboratory often require high pressures to ensure sufficient solubility of gases into the reactant solution. These reactions can be carried out using thick walled pressure vessels. The pressure vessel allows for operation at high pressure with appropriate safety concerns abated. The following document highlights the procedure for the safe operation of the high pressure reactor vessel.

Procedure:

The operation of the high pressure Parr reactor (or equivalent) can be roughly broken down into 3 steps.

1. Charging

1.1. Select an appropriate secondary reaction vessel based on the scale of the reaction. Test tubes, Erlenmeyer flasks, or round bottoms are some examples of appropriate reaction vessels. Make sure you keep enough head space above the solvent level in the flask as the solvent tends to bubble up during venting the pressure.

1.2. Place the reactants along with a stir bar in the reaction vessel and place it in the high pressure reactor.

1.3. Place the pressure gauge assembly on the top of the reaction vessel. Make sure the vent valve is completely closed. Turn it clockwise to finger tight. DO NOT OVERTIGHTEN IT.)

1.4. Assemble the split rings on the vessel.

1.5. Start tightening the diagonally opposite screws on the split rings, but do not tighten them all the way. This is to ensure that the pressure exerted by the gauge is even across the vessel.

1.6. Tighten all the screws completely.

1.7. Place the safety ring on the bench, and place the reactor in the ring.

1.8. Slide the ring up to the split rings, and align the screw with the dent on the side of the split ring.

1.9. Finger tighten the safety ring.

1.10. The vessel is now ready for the next stage.

2. Purging and Pressurizing

2.1. Attach the pressurized gas source to the reactor and turn on the main valve on the regulator.

2.2. Set the pressure to approximately 1/3rd of the final required pressure.

2.3. Slowly open the vent valve on the pressure gauge and pressurize the reactor.

2.4. Now close the main valve on the gas regulator.

2.5. Slowly loosen the pressure line going in to the reactor, so that the pressure in the reactor starts to fall. (Make sure the reactor is in a well-ventilated place when you do this)

2.6. Once the pressure falls back to zero, tighten the pressure line again and open the main valve on the gas regulator.

2.7. Adjust the pressure to 2/3rd of the final required pressure and repeat steps 3-6 above.

2.8. Now adjust the pressure on the regulator to the final desired value and pressurize the reactor.

2.9. Once the final pressure is reached, close the vent valve on the pressure gauge, and then close the main valve on the gas regulator.

2.10. Carefully loosen the pressure line, so that the gas in the line and the regulator is vented.

2.11. Always set the outlet pressure on the gas regulator back to zero (this usually means loosening the pressure control valve.) This ensures that gas would not leak even if someone turned on the main valve on the regulator by accident.

2.12. Now place the reactor in a hood and let the reaction run for the desired amount of time. The reactor can be heated if desired. Ensure the temperature is below the rated limit of the vessel.

3. Venting

3.1. Once the reaction time is over cool the reactor to room temperature (if necessary).

3.2. Now slowly open the vent valve on the gauge to vent the gas from the reactor. Do this as slow as possible so as to avoid the solvent from spilling over in the reactor. (MAKE SURE THE REACTOR IS IN A HOOD)

3.3. Once the pressure in the reactor drops to zero, loosen the safety ring and the screws on the split rings.

3.4. Disassemble the split rings and remove the gauge from the reactor.

3.5. Remove the reaction vessel from the reactor.

Once the reaction vessel is removed from the reactor, rinse the reactor with water and then acetone and leave it open to dry.

Summary: The manipulation of gases at high pressure can be done with the use of Parr reactor (or equivalent) vessels. Proper safety precautions should be observed while operating these vessels as they present an explosion hazard.