

High Pressure Reactors: Parr Reactor

Parr reactors are sealed reaction vessels capable of carrying out reactions at pressures up to 3000 psig (~ 200 atm). The reactor in the Tonks group is a Parr 5500 Compact Micro Reactor (Figure 1, right), equipped with overhead stirring, a controlled heating block, and direct sampling capabilities. The vessel utilizes a flat PTFE gasket that is held in place by a split ring closure, allowing for a tight seal up to temperatures of 350 °C. While this is the max temperature allowable with this gasket, reduced operating temperatures will significantly improve the lifetime of the gasket. The vessel is constructed out of alloy C-276, a nickel-chromium-molybdenum alloy which has a broad general corrosion resistance.

The temperature and stirring capabilities are controlled with a Parr 4848 reactor controller (Figure 1, left). Realtime pressure monitoring is also possible; however our reactor setup does not include the required transducer necessary for pressure measurement.

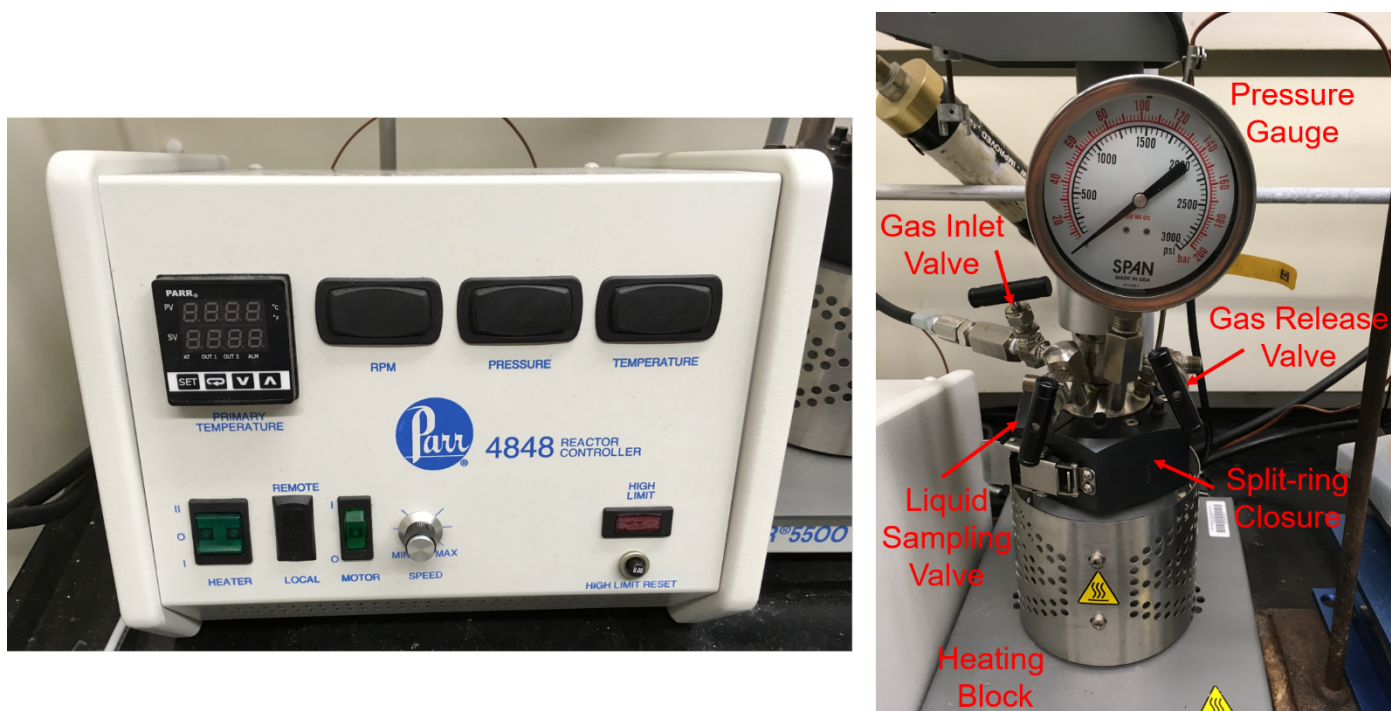


Figure 1. Overview of the Parr Reactor system.

A. General Safety Guidelines & PPE:

- Working with high pressure apparatuses such as the Parr reactor presents explosion dangers.
- Working with certain gases can pose additional fire/explosion dangers (e.g. hydrogen, oxygen) or acute toxicity dangers (e.g. CO, NO).

Full PPE (goggles or facemask, gloves, and labcoat) must be worn at all times, and manipulations should be carried out in a working fume hood with the safety sash providing a barrier between you and the vessel. High pressure gas cylinders must be properly secured, and the appropriate gas regulator must be properly attached and leak free (especially when working with toxic gases such as CO) prior to doing any work.

Work involving highly flammable gases such as hydrogen or oxygen must be carried out away from any flames or extreme sources of heat. Be aware of the location and use of fire safety equipment in the lab prior to using flammables.

When working with CO, a functioning CO detector should be secured on person or in the immediate vicinity (See CO detector SOP for more details).

All pressurized vessels should be secured behind a weighted blast shield. Proper signage notifying others of the potential danger should also be visible, and others working in lab should be notified that a high pressure reaction is being carried out.

B. Instructions:

Factors to consider prior to setting up your reaction:

1. The max pressure rating of the rupture disk installed on the Parr 5500 is 3000 psig \pm 5%. Other factors such as temperature or the nature of the reaction (e.g. gas evolving reactions, scale, etc) also need to be taken into consideration when planning reaction conditions and their effect on the pressure in the vessel. In general reactions should not be run at pressures above 2500 psig. A comprehensive discussion about operating pressure can be found in the manual.
2. The temperature controller is able to be programmed up to 800 °C, but must never exceed 350 °C as this is the max operating temperature allowable for the PTFE gasket. Also, the controller can be programmed in units of Celsius or Fahrenheit, and so it is important to ensure the correct temperature setting is selected. In order for the temperature to be properly regulated, the temperature probe must be properly submerged in the reaction solution. If it isn't, overheating can occur.

Setting up your reaction:

3. Charge the Parr reactor with your reagents/solvent.
4. Set the head on the cylinder (Figure 2).



Figure 2. Placing the reactor head on the reactor cylinder.

- Slide the two sections of the split-ring closure (Figure 3, left) into place and position them so that the shallow socket drilled in the outer surface of one of the ring sections is 180° from the gauge face. Raise the outer band into place around the ring sections and position the band so that the cone pointed screw enters the socket described above (Figure 3, middle); then tighten the screw lightly to hold the band in place (Figure 3, right).

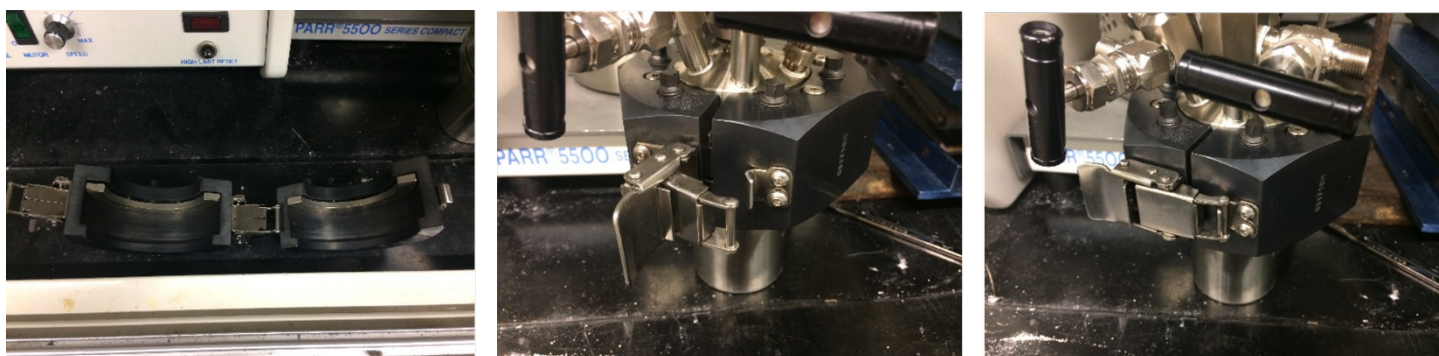


Figure 3. Utilizing the split-ring closure.

- Tighten each of the cap screws with the wrench included with the apparatus. Apply a firm pull to each screw. The manual recommends tightening each screw to a torque of ~ 15 ft-lbs., however the head can be deformed at torques greater than 25 ft-lbs. Tightening should proceed in a crisscross pattern rather than progressively around the circle.
- Place the assembled reactor on the heating block. A quick connect male joint has been connected to the gas inlet of the reactor with an A495HC pressure hose. Make certain the output pressure of the regulator on the gas tank is correctly set. With the inlet valve closed and the regulator valve closed, connect the quick connect. The gas release valve must also be closed unless the vessel is to be purged, or unless there is to be a continuous flow through the reactor during a run. Open the regulator valve then use the inlet valve to control the flow of gas into the vessel. After the desired pressure has been reached, close the tank valve, the needle valve on

the regulator, and the vessel inlet valve, and open the pressure release valve. Only disconnect the quick connect after the pressure in the tubing has been completely released (Figure 4).

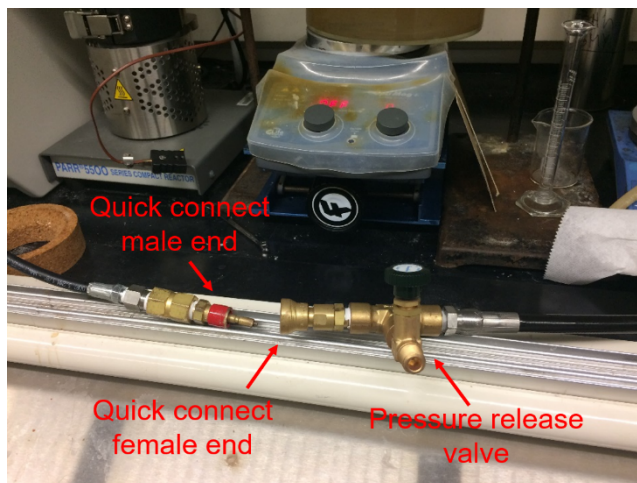


Figure 4. Quick connect gas fittings used on the Parr reactor setup.

8. Put the motor in place with the magnetic mechanical stirrer, and connect the thermocouple and extension wire (Figure 5).

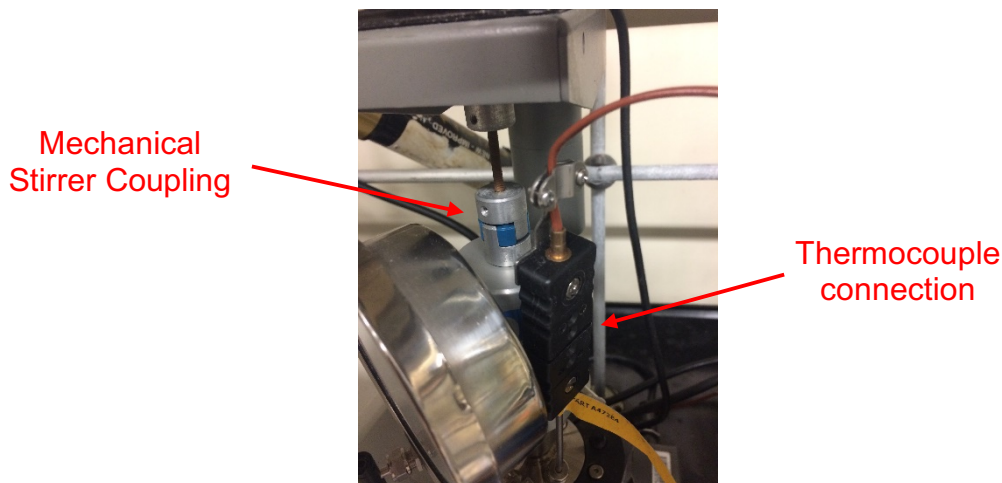


Figure 5. Close-up of the mechanical stirrer and thermocouple connections.

9. Set the controller to the target temperature. See manual for heating functions and programs. Turn on heating and stirring functions.

Proper Cleaning of the Parr Reactor:

10. In general, the reactor should be resistant to organic solvents and rinsing with acetone or methanol is always a good place to start, followed by washing with soap and water. Strongly acidic or oxidative solutions should not be used to clean the reactor.

By signing below, you indicate that you have read and understand the content of this document.

Name: _____

Date: _____