High Pressure Reactors: Fisher-Porter Tubes

A Fisher-Porter tube or Fisher-Porter vessel is a glass pressure vessel that is commonly used for carrying out reactions at pressures ranging from approximately 1 atm (~ 15 psia) to approximately ~ 10-13 atm (~150-195 psia). The 3 oz. tubes used in the Tonks lab have a max. pressure rating of 225 psig at room temperature. The reaction vessel (Figure 1 below) consists of a heavy-walled borosilicate glass tube coated with a polymer sleeve for added safety in the event of an explosion. The mouth of the tube fits an O-ring lined metal coupling, which is clamped in place with a two piece screw cap, securing the tube to the upper valve assembly and allowing for a tight seal under pressure.

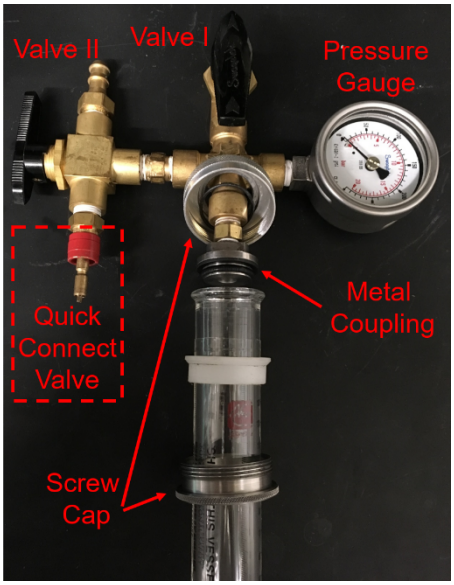

Figure 1. Overview of Tonks Group Fisher-Porter tubes.

A. General Safety Guidelines & PPE:

- Working with high pressure reactors such as a Fisher-Porter bottle 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 3 oz pressure reaction vessels that we have is 225 psig, and this **pressure rating will decrease at the rate of 0.25 psig per degree F rise above ambient temperature**. Close attention must also be given to any reactions that might release sudden surges of heat and pressure, and the safety valve should be properly set to account for these considerations.

2. The vessel must never be filled to more than three-fourths of its available free space, and in some cases the charge must be reduced even further for safe operation. Dangerous pressures can develop suddenly when a liquid is heated in a closed vessel, especially if the temperature is above the boiling point of the liquid.

Setting up your reaction:

3. Charge the Fisher-Porter tube with your reagents/solvent.

4. Secure the Fisher-Porter tube to the metal coupling by engaging both halves of the screw cap and tightening as tight as you can get it (Figure 2). Ensure that the O-ring situated between the top half of the screw cap and the top of the metal coupling is properly aligned and providing a proper seal. Once properly secured, the tube will still be able to rotate about the metal coupling.

5. Ensure that both valves are closed by turning them to perpendicular to the direction of gas flow.

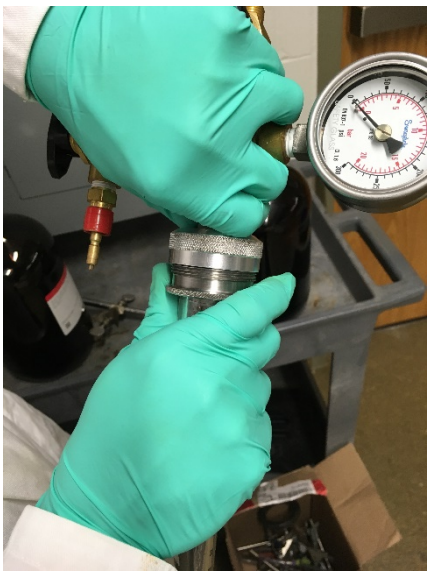

Figure 2. When properly secured, the tube can still rotate around the metal enclosure.

6. With the tube properly attached to the valve assembly via the metal coupling, slide a metal-mesh sleeve around the tube, and secure the apparatus by clamping the valve assembly to a ring stand (Figure 3).

Figure 3. Metal mesh provides additional blast protection.

7. Prepare to purge/pressurize the vessel by connecting the gas line to the Fisher-Porter apparatus via the quick connect attachment (Figure 4, left). Proper connection of the male and female ends of the quick connect requires some force, and a distinct clicking sound will be heard once they are properly connected. Note the noticeable gap highlighted in the dashed box shown in Figure 4 (middle), which disappears once both ends of the quick connect are properly attached (dashed box, Figure 4, right).

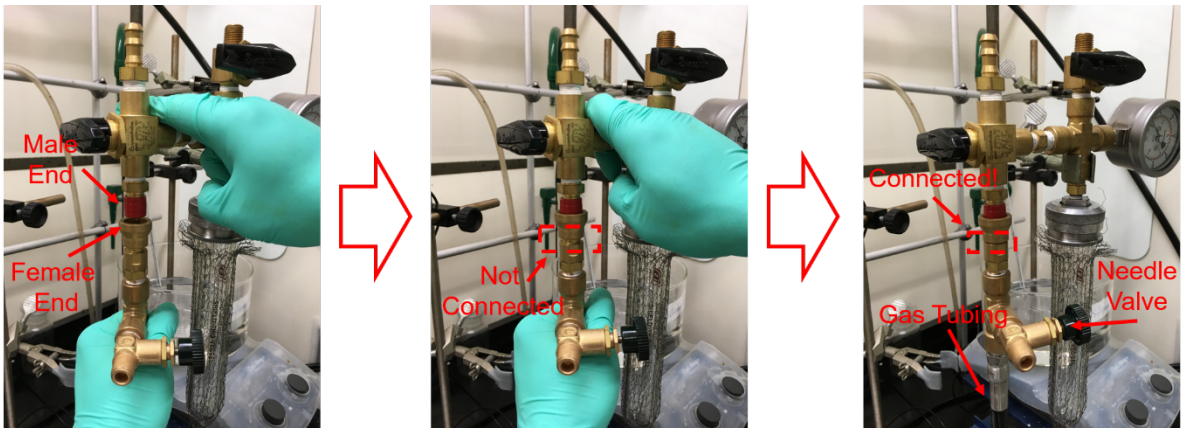

Figure 4. Quick-connect operation.

8. Before the tube can be purged/pressurized, the gas tank must be opened. Open the main valve on the gas cylinder all the way and crack open the needle valve on the gas regulator. NOTE: This SOP assumes that the regulator pressure has been appropriately set and that the user is properly trained to use high-pressure gas cylinders. Refer to gas cylinder SOP for additional assistance.

9. The vessel is now ready to be purged/pressurized. Open the Fisher-Porter tube to the gas line by turning “valve II” to the downward position (Figure 6, left). The pressure gauge on the tube should increase approximately to the pressure set on the regulator. Once pressurized, close “valve II” by turning it perpendicular to the quick connect attachment (Figure 6, right). Monitor the pressure gauge on the tube to ensure the apparatus is not leaking. If it is, the seal between the tube and the metal coupling will likely need to be re-established by disassembling and reassembling the apparatus.

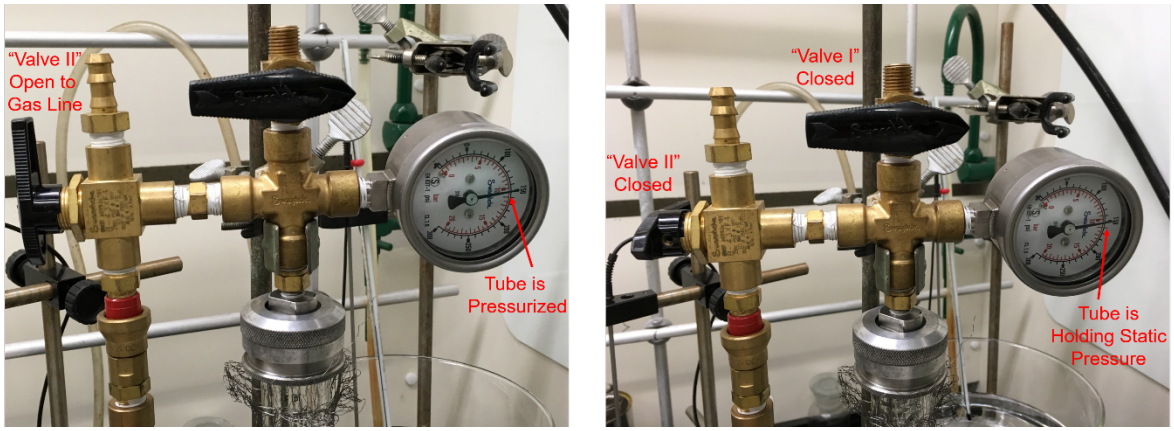

Figure 6. Pressurization of the tube.

10. It is often desirable to ensure that the atmosphere in the bottle is entirely composed of the gaseous reagent. To do this, pressurize and vent the reactor three times.

11. Once the desired atmosphere in the vessel has been established, the vessel can be finally pressurized to the desired pressure. To seal the vessel, close both valves, again monitoring the pressure gauge of the vessel to ensure no leakage.

12. Disconnect the gas tubing by applying upward pressure to the flanged half of the quick connect attachment, which should release the quick connect and allow the gas tubing to be removed.

13. Properly secure the blast shield and proper signage in front of the Fisher-Porter apparatus, and close the safety sash of the fume hood (Figure 7).

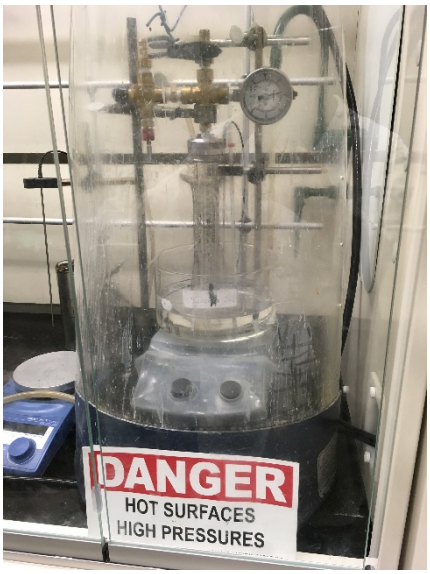

Figure 7. Use a blast shield and post proper signage indicating high pressure!

Proper Cleaning of the Fisher-Porter Tube

14. To clean the tube, rinse thoroughly with organic solvent, rinse with acetone, wash with soap and water, and finally rinse with deionized water. The tube must not be placed in the oven and is best air dried. The tube must never be stored in the base bath as this will degrade the polymer coating on the tube, and potentially weaken the glass. However, basic solution can be used to soak the inside of the tube if necessary for short periods of time. For removal of metal impurities, the inside of the tube can be soaked with an acid solution. As with other glassware, clean the tube ***immediately*** upon use.

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

Name: _____ Date: _____