

Standard Operating Procedure

Task: Parr MRS 5000

Created by: Javier A. Grajeda

Date: 7/18/14

Revision Date (Author): Lauren Gregor (8/5/16)

Background:

- The Parr MRS 5000 Multireactor is utilized for reactions that require high temperatures and pressures. The six individually controlled reactor vessels allow for variable temperatures and pressures to be run at the same time.

Training Requirements:

- Lab safety training
- Multireactor training
- Working with CO training
- Changing gas cylinder training
- Glovebox training

Potential Hazards:

- Rupture of pressurized vessels/connections
- Release of flammable and/or toxic gases
- Burns by stainless steel vessels at high temperatures

Special PPE Requirements:

- Two CO detectors (if needed)

Materials Needed:

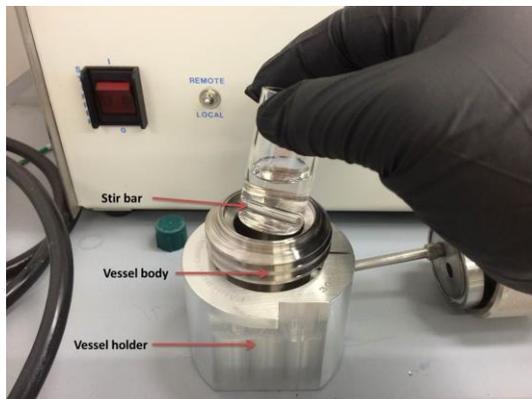
- Parr MRS5000 multi-reactor
- Teflon coated liners
- PTFE-coated stir bar
- Reactor vessel holder.
- Allen wrench of appropriate size for tightening vessel screws
- Reagents and solvents needed for desired reaction

Procedure:

Loading and sealing the vessel

- Inspect the reactors. Check that the liners and vessels are clean and dry. Check the state of the white PTFE O-ring on the vessel head. It should be clean and free of cracking or cuts. If the O-ring might be compromised, replace it. Check that the burst valve has not ruptured and that you are aware of the burst pressure. Check that all of the valves rotate freely, and that they are closed before charging the vessel.
- The vessels can be charged on the bench or in a glovebox, as required. The vessels can be brought into the glove boxes via the large antechambers. The vessel should be disassembled when brought into the glovebox, with the needle valves open.
- Slide Teflon liners into metal bodies. Avoid pinching gloves between the liner and metal body.

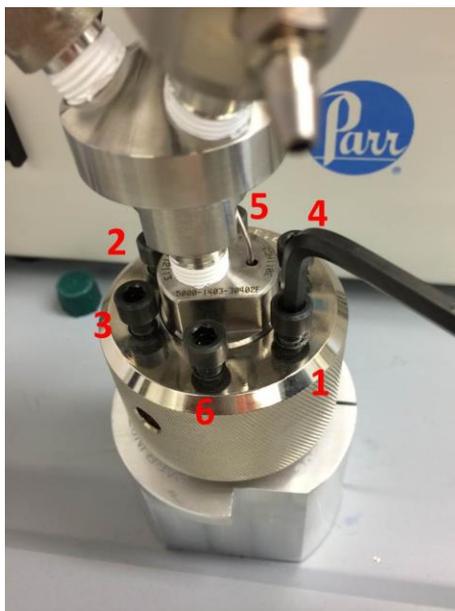
- Prepare and load your reaction mixture. A 10 mL volume is ideal. The minimum volume is 5 mL, which is required to provide good contact with the thermocouple insert on the reactor head. The maximum volume is 13 mL, to avoid filling the vessel to more than two-thirds of the total volume of ~20 mL.
- Add a magnetic stir bar. Glass-coated metal stir bars are optimized for stirring with the MRS 5000, but standard Teflon-coated stir bars can also be used.



- Place the vessel head on top of the vessel body, ensuring that the O-ring located on the vessel head, is resting on its counterpart in the vessel body.



- Hand-tighten the vessel head onto the vessel body by rotating clockwise.
- Use an Allen wrench to *gently* tighten one of the bolts on the vessel head. A good guideline is to initially tighten the bolts “pinkie tight”, meaning that the bolt should be tightened using the Allen wrench until your pinkie finger feels resistance. Tighten in a “star” pattern: after tightening one bolt, tighten the bolt on the opposite side of the reactor; then tighten the adjacent bolt to the right before tightening the bolt across from it; repeat this pattern until all six bolts have been tightened three times. Example tightening pattern: 1, 3, 5 then skip to 6, 2, and 4 (see figure). Tighten the bolts more aggressively each time, such that the last cycle is as tight as possible. Check that the bolts have been fully tightened once more by checking each bolt in a clockwise cycle.



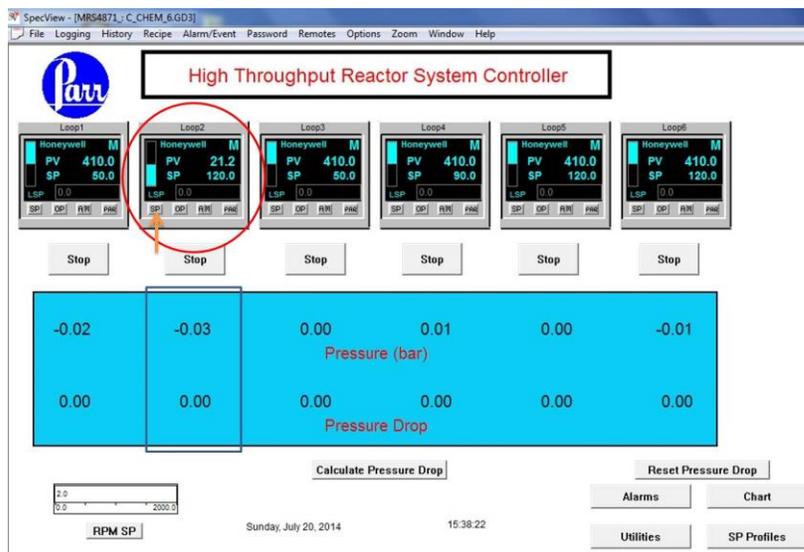
- Close the needle valves in the vessel head.

Placing the vessel in the reactor and setting up the software

- Check that the needle valves in the vessel head are closed.
- Place the vessel in one of the reactor sites (loops). Note the number: site 1 is at the far left, and the numbers increase until you reach site 6 at the far right.
- Connect the thermocouple, pressure transducer, and gas inlet to the reactor head, as shown below. The cables are color-coded by reactor site (loop). Ensure that the connections match the correct reactor site. **Caution:** the rupture disc should be oriented towards the left side of the hood.



- Power on the MRS 5000 (stirrer system) and the 4871 Process Controller.
- Open SpecView32



- The top of the screen shows information and settings for each reactor site (loop), labeled Loop1 through Loop6 in gray boxes filled with black. Each Loop window contains two values: Point Value (PV) and Set Point (SP). PV is the current temperature in the vessel as read by the thermocouple. Note that when not connected the temperature will read a value of 410 °C.
- SP is the requested reaction temperature. Change the target reaction temperature by clicking on the gray 'SP' box. Type the set point in the new window that pops up and click 'SEND'. The new value should appear in the corresponding Loop window. **The vessel will not heat until armed. Caution: maximum operating temperature is 300 °C!**
- If the reaction will not be pressurized, start heating: click on the 'A/M' button in the appropriate loop. Click on 'Auto' in the new window that appears. The letter "M" in the corresponding Loop# should switch to an "A" and the temperature will start to increase.
- Place a note (located in the drawer underneath the reactor computer), indicating that a reaction at high temperature/pressure is running, in a transparency taped on the hood sash. Also tape a safe operating card (SOC) to the sash detailing reaction conditions.

Pressurizing the vessel

- The pressure, as sensed by the transducer, appears in the blue box in the software. **Caution: the maximum operating pressure is 3000 psi!**
- The pressurization procedure will vary depending on the chemistry. Currently, the lab is equipped with the following high-pressure regulators, located in the vented cabinet next to Hood 5:
 - Two CGA-350 regulators for CO, H₂, ethylene. These regulators have a maximum delivery pressure of 2000 psi.
 - One CGA-320 regulator for CO₂. This regulator has a maximum delivery pressure of 500 psi. CO₂ tanks are pressurized to ~ 800 psi.
 - Note that your total pressure will be limited by (a) the total cylinder pressure, (b) the maximum delivery pressure that the installed regulator is capable of providing, and (c) the 3000 psi limit of the MRS 5000. Plan ahead and procure a high pressure regulator if needed.
- **Caution: When working with CO, at least two CO detectors must be used.** One detector is located next to the gas cylinder and one on the user's lab coat.

- The MRS 5000 reactor is served with compressed gas directly from a cylinder via the appropriate regulator. **Caution: every segment of the gas manifold must be suitable for high-pressure service!** Ensure that the desired gas cylinder is properly restrained in the cylinder rack. A braided stainless steel hose (rated to 3000 psi) is connected from the manifold to the regulator. Check the regulator for its CGA rating and make sure it matches the desired gas. If not, use the following instructions for changing the regulator connected to the steel hose.
 - **Caution: Do not rotate the hose as it will become strained and may suffer damage.** To remove the regulator, hold the brass end of the hose with a crescent wrench while carefully rotating the regulator with another crescent wrench. Once the connection is loosed enough unscrew the regulator by spinning it around, keeping the hose straight (this is much easier with two people).
 - Apply a fresh coat of Teflon tape to the appropriate regulator and connect it to the hose by rotating the regulator. After hand-tightening the regulator, hold the brass end of the hose with a crescent wrench while carefully rotating the regulator with another crescent wrench. Apply a fresh coat of Teflon tape to the gas cylinder and connect the regulator, tightening it with a crescent wrench.
- Check that all the gas delivery hoses in the reactor are properly seated on the wall holder or connected to a sealed vessel.
- Check that all vessel valves are closed.
- Close the hood sash completely. **Caution: Always keep the sash closed when vessels are pressurized!**
- Set the regulator dial to the lowest setting (all the way counter-clockwise).
- Perform leak testing as follows:
 - With the regulator delivery valve closed, open the cylinder valve. Make note of the tank pressure (the delivery pressure should be close to 0 psi) and close the cylinder valve. Watch for leaks by waiting a few minutes to look for pressure drop.
 - Open the cylinder valve and delivery valves. Use the regulator dial to build a modest pressure (~15 psig). Open the black valve leading to the MRS 5000 manifold. Make note of the pressure at the regulator. Close the cylinder valve and watch for leaks by waiting a few minutes to look for pressure drop.
 - If there is a pressure drop, close valves moving back towards the tank one at a time until the leaky segment of the manifold is discovered. Be cautious if you are using a toxic gas such as CO — keep a low pressure and only open the tank periodically to test for leaks!
 - A hissing sound in the MRS 5000 manifold may be indicative of a damaged O-ring in one of the gas hoses. Close the cylinder and notify the Parr Czar immediately.
- After ensuring there are no leaks, re-pressurize the system to 15 psig as before.
- Purge the manifold by slowly opening the black vent valve on the reactor. A loud hissing sound will be heard. Vent in three bursts of 30 seconds to displace all air in the manifold. Close the vent valve and allow the system to re-pressurize.
- If the vessel was loaded in the glovebox, proceed as follows:
 - Pressurize the vessel by slowly opening the gas inlet valve. The analog gauge should deflect slightly and a hissing sound or movement in the gas inlet hoses may be detected. There should be no sound or motion after this initial pressurization. Check the pressure reading of the Loop you are using in SpecView32. The pressure transducer (readout on the PC), analog gauge of the reactor, and the regulator should all match. Note: The software pressure is reported in units of bar. ***Know your desired working pressure in psi and bar ahead of time!*** Conversion

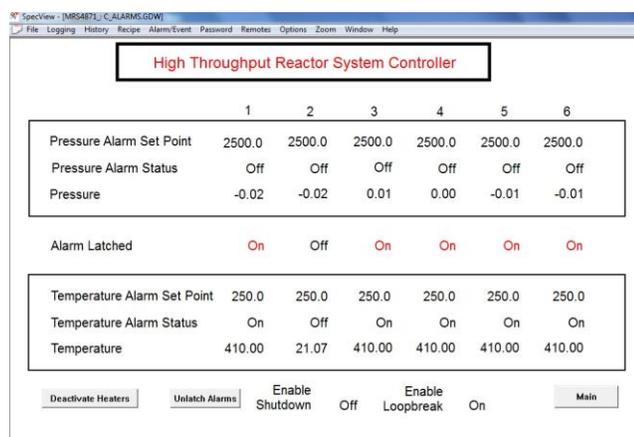
tables with approximate values are conveniently located next to the computer and on the wall by the gas cylinders.

- If the vessel was loaded under air, and the reaction does not require an inert atmosphere, follow the procedure above.
- If the vessel was loaded under air, and an inert atmosphere is required, proceed as follows:
 - Method 1 (Purge method): After pressurizing the vessel, slowly open the vent valve on the reactor head (not to be confused with the general vent valve for the MRS 5000). Vent in three bursts of 30 seconds to displace air in the reactor headspace.
 - Method 2 (Pressurize-vent method): After pressurizing the vessel, increase the pressure to ~100 psig; then open the vessel vent valve until the hissing stops (1 atm). Repeat three times.
- Use the regulator dial to adjust the pressure as desired. **Caution: maximum operating pressure is 3000 psi!** When the desired pressure is achieved, close the gas inlet valve on the reactor.



Finalizing software adjustments before initiating a reaction

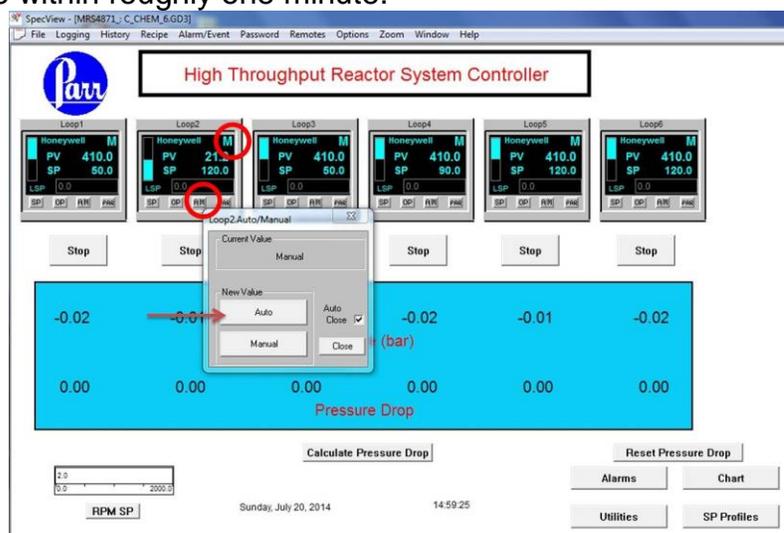
- Stirring: click on the 'RPM SP' button on the bottom-left corner of the software. A new window appears. Set the revolutions per minute at which to stir the reaction and click on SEND. The value will appear in a bar above the RPM SP button. It will also appear above the Tachometer reader in the stirrer system.
- Alarms: click on the 'Alarm' button on SpecView32. In the Alarm window, click on 'Unlatch Alarms'. Note that the alarms will only deactivate for the vessels for which the pressure transducer and thermocouple have been connected.



- Check that the Enable Shutdown option is off. The pressure and temperature alarm set points can be changed by clicking on the value specific to a loop, entering a new value, and clicking 'SEND'. Click on 'Main' to return to the main window.

Heating the Reaction

- Click on the 'SP' button in the appropriate Loop. In the new window, type the desired temperature (in ° C), and click 'SEND'.
- Click on the 'A/M' button in the appropriate Loop. In the new window, click on 'Auto'. The letter "M" in the corresponding Loop# should switch to an "A". The temperature should start to increase within roughly one minute.



Purging the manifold

- Once the reaction is set up, the manifold must be purged for the next use.
- Release the gases:
 - Close the cylinder valve. The delivery valve, SG1, and the black main inlet valve to the reactor should all still be open. Remember to have all CO detectors armed, if using CO.
 - Slowly open the black vent valve on the reactor. This will vent the pressure from the reactor all the way to the tank. Only open the hood the minimum amount needed to reach the vent valve. A hissing sound is normally heard.
 - After venting, check that the cylinder gauge and delivery gauge both read 0 psig. Toxic gases can be further removed by attaching a vacuum pump to the vent valve

- and evacuating the system. Do not use corrosive gases without consulting the Parr Czar. Thorough and prompt purging with N₂ will be required.
- Close all valves and set the regulator delivery pressure close to zero.
- Switch the tank as needed using standard procedures (see SOP: changing gas cylinders).

Ending the reaction

- Once the reaction is finished, stop heating by clicking on the 'A/M' button and clicking on 'Manual'. The letter "A" in the corresponding Loop should switch to an "M". Then click on 'Stop'.
- Allow the reaction mixture to cool close to room temperature before taking further action.
- For workup in air, proceed as follows.
 - Vent the vessel *into a hood* by slowly opening the needle valve in the vessel head. If potential products are volatile, the reactors may be cooled in an ice bath prior to venting. Note: if the gases are to be analyzed, a different procedure must be carried out. Consult the Parr Czar for advice on how to collect headspace samples or condense the volatiles using a vacuum line.
 - Disconnect the pressure transducer and the thermistor from the vessel head.
 - Disconnect the gas hose from the vessel head. Open the gas inlet valve to be sure that no pressure is built up during work-up.
 - Bring the reactors to a synthetic fume hood for work-up.
 - Loosen the bolts with the Allen wrench. Slightly open the pressure inlet valve to release any excess pressure built up.
 - Remove the reactor head. After high temperatures and/or pressures have been applied, the vessel head may be fused tightly to the vessel body. If the head does not release easily, a flat-head screwdriver can be used *gently* to separate the head from the body.
 - Remove the Teflon liner and work up the reaction as required.
- For workup under nitrogen, proceed as follows.
 - Attach the vessel to a bubbler and vent through the bubbler. Close the vent valve and keep all other valves closed.
 - Disconnect the pressure transducer and the thermistor from the vessel head.
 - Disconnect the gas hose from the vessel head. Open the gas inlet valve to be sure that no pressure is built up during work-up.
 - Pump into the glovebox. Check that all valves are closed before evacuation.
 - Loosen the bolts with the Allen wrench.
 - Remove the reactor head. After high temperatures and/or pressures have been applied, the vessel head may be fused tightly to the vessel body. If the head does not release easily, a flat-head screwdriver can be used *gently* to separate the head from the body.
 - Remove the glass or Teflon liner and work up the reaction as required.
- Close the software. Turn off the stirrer system and the 4871 Process Controller.

Cleaning the vessels

- The vessel body and the glass or Teflon liner can be cleaned with organic solvents. If needed, gently wipe the surfaces with a Kim Wipe or a paper towel. Do not place Teflon liners in the oven. **Never use aqua regia or other strong acids! Never place items in the base bath!**
- Liners may also be soaked overnight in 1 M HCl bath or in a 10 mM EDTA bath (adjusted to pH 14 with NaOH) to remove any excess salts used during the reaction.

- The reactor head is more intricate, and care should be taken to avoid contacting plastic parts and electrical contacts with organic solvent. Open the valves in the vessel head and run a slow stream of DI water through any ports where solvent may have condensed. Further rinse the ports with acetone. Run a gentle stream of air through all the ports to begin the drying process. Allow ample time for the vessel head to dry. **Do NOT place the reactor heads in the oven.**

Related SOPs

- Working with CO SOP
- Changing gas cylinder SOP
- Glovebox SOP