Standard Operating Procedure

Task: High-pressure manifold (David Bowie) operation
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Background:
- David Bowie, the high-pressure manifold is utilized for reactions that require high temperatures and pressures. It allows for the safe and efficient pressurization of high-pressure autoclaves, including pressurization with gas mixtures. The line is equipped with a number of safety features including vent valves, pressure relief valves, and check valves. A Toxgard II CO monitor is located directly above the gas cylinders for detection of potentially toxic CO gas. The manifold is designed for short-term pressurization of reactors that operate as closed vessels. It is not meant for continuous flow pressurization.

Training Requirements:
- Lab safety training
- High-pressure manifold training
- Working with CO training if appropriate
- Changing gas cylinder training

Potential Hazards:
- Rupture of pressurized vessels/connections
- Release of flammable and/or toxic gases
- Burns by metal vessels at high temperatures

Special PPE Requirements:
- Personal CO detectors when working with CO

Materials Needed:
- High-pressure autoclave
- PTFE-coated stir bars
- Allen wrench or pinhook spanners of appropriate size for tightening autoclaves
- Reagents and solvents needed for desired reaction

Design and features of David Bowie:
- The manifold consists of three stainless steel lines that feed up to three gases into a single, main line via a four way Swagelok connector.
- Each line is equipped with a check valve to prevent cross-contamination between different gas cylinders.
- The catalysis center is equipped with two CGA-350 regulators with a maximum delivery of 3000 psi, and one CGA-580 regulator with a maximum delivery of 300 psi.
- Each line is labeled with gas flow direction stickers and gas content. A nitrogen line is labeled with green labels. The hydrogen line is labeled with red labels. The carbon monoxide line is labeled with blue labels. Note that when using syngas or any gas mixture containing CO, the “blue line” should be used. This line is fed directly from a ventilated cabinet to prevent potential exposure to CO in the event of a leak at the regulator/cylinder.
connection. The regulator can be manipulated using the window opening on the cabinet. An N₂ gas cylinder is permanently connected to the manifold to purge the reactors/manifold.

- Each line is equipped with pressure-relief valves inside the fumehood, pointing towards the back of the hood. This is a safety feature designed to provide a safe and controlled release of pressure in the event of over-pressurization. Note that these pressure-relief valves are set to rupture at different pressures as follows:
  - N₂ (green line): 1000 psi
  - H₂ (red line): 2750 psi
  - CO (blue line): 2750 psi

Caution: Do not open the N₂ line valve while operating high-pressures of gas in one of the other lines. A pressure of 1000 psi will set off the pressure-relief valve in the N₂ line.

- After the four-way Swagelok there is a single stainless steel line used to pressurize the autoclaves. This line is equipped with three vent valves. Two four-way Swagelok connectors feature pressure gauges and quick release adapters for rapid flexible hose attachment to the line. Valves separate the four-way Swageloks and the quick release adapters to allow or restrict gas flow. The pressure gauges on the line can read up to 3000 psi of pressure. The initial tick mark on the pressure gauges marks 100 psi. Note that any pressure below this (50 psi N₂ purge) will not be reflected in the pressure gauges.
Procedure: Pressurizing the line

- The pressurizing conditions will vary depending on the chemistry. Currently, the line is equipped with the following regulators, as mentioned above:
  - Two CGA-350 regulators for CO, H₂, ethylene. These regulators have a maximum delivery pressure of 3000 psi
  - One CGA-580 regulator for N₂, Argon. This regulator has a maximum delivery pressure of 300 psi
- **Caution:** When working with CO, a personal CO detector must be worn on the researcher's lab coat, in addition to the Toxgard II that sits above the gas cylinders.
- Ensure that the desired gas cylinder is properly restrained to the bench. If working with gas mixtures with CO, connect it to the line contained within the ventilated cabinet.
- Check the regulator for its CGA rating and make sure it matches the desired gas. Apply a fresh coat of LA-CO® high-density PTFE tape to threads of the working gas cylinder. Apply the tape in the direction in which the regulator will rotate. Attach the appropriate regulator to gas cylinder with a crescent wrench.
- Connect the high-pressure flexible hose from the corresponding gas line to the regulator via the quick release couple.
- **Check that all valves on the line are closed.**
- Connect the appropriate flexible gas-inlet hose from the manifold to your reactor or Parr manifold via the corresponding quick release couple. Ensure the inlet valve on your reactor/Parr manifold is closed. Open the gas delivery valve between the four-way Swagelok and the flexible tubing.
- Set the regulator dial to the lowest setting (all the way counter-clockwise).
Open the valve on the working gas line to allow flow to the main line. If using the Parr manifold, open the inlet valve to allow gas flow up to the sealed Parr vessels (See MRS5000 SOP).

Close the hood sash completely. Caution: Always keep the sash closed when vessels are pressurized!

Perform leak testing as follows:
- Ensure the needle valve on the regulator is 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 the needle valve on the regulator. Use the regulator dial to build a modest pressure (<100 psi). 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!

After ensuring there are no leaks, open the gas cylinder valve and pressurize the system (<100 psi).

Purge the manifold by slowly opening one of the vent valves on the line. 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 reactor(s) was loaded in the glovebox, pressurize the reactor by slowly opening the gas inlet valve. Once pressurized at the desired pressure, close the valve to the reactor/Parr vessels and watch for leaks by waiting a few minutes to look for a pressure drop. Wait for the leak check to begin heating.

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. 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 psi; close the gas inlet valve and open the reactor vent valve until the hissing stops (1 atm). Repeat three times.

Then use the regulator dial to adjust the pressure as desired. Caution: Ensure that the pressure limit imposed by the reactor used is not exceeded! The maximum operating pressure on the line is 2750 psi (limited by the pressure relief valve). When the desired pressure is achieved, close the gas inlet valve on the reactor.

Purging the manifold
- Once the reaction is set up, the manifold must be purged.
- Release the gases:
  - Close the working gas cylinder valve and reduce the delivery pressure by turning the dial on the regulator counter-clockwise (keep the N₂ cylinder valve open). The valve leading to the main line and the valve leading to the reactor/Parr manifold should still be open. Remember to have all CO detectors armed, if using CO.
o Slowly open one of the vent valves on the manifold. 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.

o After venting, check that the cylinder gauge, delivery gauge, and the gauges on the manifold all read 0 psi. If gas pressure remains in the regulator after the hissing stops, turn the delivery dial clockwise slowly to allow the last amounts of gas to flow out.

o After the full system is de-pressurized, open the needle valve on the N₂ regulator and the valve leading from the green line to pressurize the line with ~50 psi N₂. Open one of the vent valves on the manifold. Vent in three bursts of 30 seconds, then close the vent valve.

o Close the N₂ needle valve.

o Close all valves on the manifold and set the working gas regulator delivery pressure to its lowest setting.

Related SOPs
- Working with CO SOP
- Changing gas cylinder SOP
- Glovebox SOP
- MRS5000 Parr manifold SOP