Standard Operating Procedure

Task: Using the HEL CAT18 High Pressure Autoclave
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Background:

- HEL CAT18 ("Hell Cat") is a pressurizable reaction vessel that allows for high-throughput screening of small scale reactions. Inside the reactor are 18 holders for GC vials (2 mL). This SOP will explain in detail how to pressurize reactions in HEL CAT.

Training Requirements:

- Lab safety training
- High-pressure manifold training
- Working with CO training, if applicable
- 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:

- HEL CAT reactor
- Pinhook spanners for tightening HEL CAT
- Reagents and solvents for desired reaction
- PTFE-coated stir bars

Assembling the Reactor:

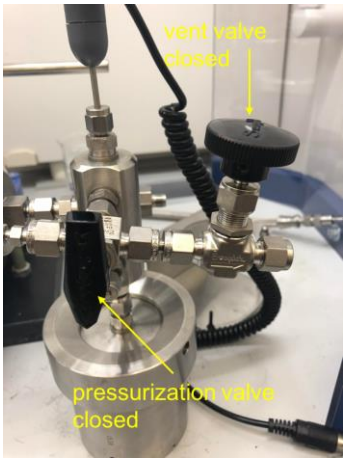
- For air- and moisture-sensitive studies, HEL CAT can be pumped into a glovebox, where reactions can be prepared and loaded into the vessel.
- The HEL CAT reactor consists of two main pieces. These pieces should be unattached when pumped into the glovebox, then attached inside the box after loading the vessel with reaction vials.
- The bottom half of the reactor has 18 holders for GC-sized vials. On the threads of the bottom half, there should be some brown grease. This is a copper-based grease that serves to protect the threads from damage due to tightening. If there does not appear to be any grease (if the metal of the threads is visibly bare or if there is friction upon tightening) then reapply a small amount of the copper-grease to the threads before bringing the reactor into the glovebox. Inside the bottom half of HEL CAT there is a rubber o-ring along the inside perimeter. Apply a small amount of silicone grease to the o-ring and spread evenly around. This improves the seal of the o-ring connection.

- The upper half of HEL CAT consists of the gas inlet/outlet ports, a permanently attached thermocouple, and the “cap” to the vessel that screws onto the bottom half. The whole upper half of the vessel fits into the lower half via two guiding rods and the tip of the thermocouple. *Note: the upper half is awkward to handle. Try to hold it as close to the base as possible, and not by one of the protruding arms toward the top.*

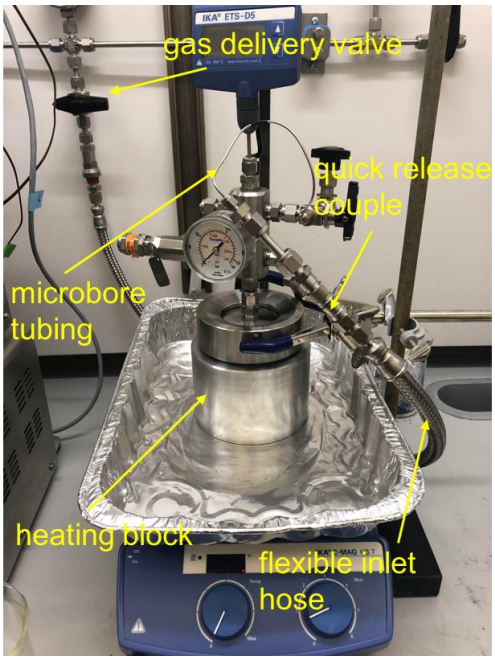
- Once the desired reactions have been loaded in the vial holders, the upper half can be attached by fitting the guiding rods and thermometer tip into the corresponding holes in the bottom half. *Note: aligning all three points with the holes can be tricky. When done properly, the top unit will “sink” nicely into the bottom unit, and the connection should look perfectly even, 360º around.*

Pressurizing the Reactor

- Before bringing HEL CAT out of the glovebox, be sure the round vent valve is closed and the pressurization valve is facing downwards (pictured below).



- Once HEL CAT has been brought out of the box and properly tightened, it can be brought to the fume hood. There is a cylindrical piece of aluminum that fits snugly around the bottom of HEL CAT. This is used to ensure even heat distribution. Clamp the vessel into place on the center of the stir plate, on top of an aluminum foil pan. The foil pan can be filled with ice and water after the reaction to expedite the otherwise long cooling process.
- HEL CAT can now be hooked up to the high pressure gas manifold via the flexible gas-inlet hose. This connects to the reactor via a quick release couple (depicted below). The microbore tubing slows down pressurization to help prevent vial rupture. *Note: Be gentle with the microbore tubing. Try not to bend it too far from its natural, resting position.*
- The complete setup should look like this:



- Next, open the gas delivery valve to the flexible hose.

- The entire manifold should be leak tested (up to the downward facing pressurization valve on HEL CAT) and purged with the desired gas or gas mixture. The line should be pressurized and leak tested according to the David Bowie SOP. Once the line has been leak tested at a modest pressure (100 psi), reopen the cylinder valve and purge the manifold with the desired gas or gas mixture. To do this, simply open and close one of the vent valves on the manifold three times for 10 seconds each. *Note: line purges are most effective when the rightmost vent valve is used.* Close the vent valve.
- Now that the manifold has been leak tested and purged, use the regulator dial on the gas cylinder to set the desired reaction pressure.
- On HEL CAT, turn the downward facing valve *toward* the gas source to initiate pressurization. The pressure reading on HEL CAT's gauge will start increasing slowly. *Note: it might take a few minutes to reach the desired pressure.*
- *Close the hood sash completely*. ***Caution**: **Always keep the sash closed when vessels are pressurized!***
- Once the pressure gauge on HEL CAT shows the desired pressure, check the vessel for leaks by turning the pressurization valve back to the downward-facing position and leave like this for 3 minutes. When 3 minutes have passed, reopen the valve (pointing *toward* the gas inlet hose). If no change is observed on the HEL CAT pressure gauge, then it has passed the leak test. Close the valve again (downward position).
- The gas manifold can now be vented and purged with N_2, as described in the David Bowie SOP. Once this is complete, the quick release couple that connects the gas inlet hose to the reactor can be disconnected
- Once HEL CAT has been pressurized and checked for leaks, the vessel can be heated. Set the thermocouple to the desired temperature, and turn the temperature dial on the hot plate to its highest setting. *Note: the plate will heat to the temperature that is set on the thermocouple, not on the plate's temperature dial.* Stirring can be adjusted using the dial on the right.

Depressurizing the Reactor

- Once the reaction is complete, turn the heat off and let HEL CAT cool to room temperature (add ice and water to the foil pan to accelerate this process).
- Once the vessel has cooled, turn the pressurization valve *away* from the gas source and *toward* the vent valve.
- Open the vent valve as slightly as possible (a hiss should be heard). As the hiss grows fainter, continue opening the valve until all pressure has been vented.
- *Inside the fume hood* , use the spanners or your hands to open HEL CAT
- With the vent valve open and the pressurization valve pointed towards it, purge the top half of HEL CAT with either nitrogen or compressed air (from the fume hood's house supply)
- Examine the reaction vials in the bottom half of HEL CAT. If any of the vial caps have ruptured, rinse the bottom half of HEL CAT and the gas inlet/outlet channels of the top half with a small amount of acetone before purging with nitrogen or compressed air.

Related SOPs

- David Bowie SOP
- Working with CO SOP
- Changing gas cylinder SOP
- Glovebox SOP
- MRS5000 Parr manifold SOP