Mini Pressure Reactor Standard Operating Procedure

THIS SOP IS NOT MEANT TO REPLACE HANDS-ON TRAINING FROM QUALIFIED PERSONNEL!

This SOP describes the basic procedure for using the minireactors, routine maintenance, common problems, and their solutions. There are two Autoclave Engineers (AE) Minireactors: the original, unlabelled reactor and the second, newer reactor labelled "T-Bone". They are nearly identical. Any differences in procedure between them will be described below.

General Comments

Treat the pressure reactors with respect at all times. If you are at all unsure of something, ask for help. NEVER WORK ON A PRESSURIZED SYSTEM!

The volume of the reactor vessel is 50 mL. Maximum reaction volume is 25 mL.

The reactors have a burst disc rated to approximately 1750 psi at room temperature. Maximum working pressure should be no more than 1400 psi. The usual operating pressure for the minireactors is 700 psi at room temperature. This translates to about 1000 psi at 200 °C. A pressure of 1100 psi at room temperature is about 1400 psi at 200 °C.

Hydrogenation experiments are ONLY performed under STATIC pressure. NEVER do a reaction under dynamic hydrogen flow. The lab is not equipped with the proper controls and safety features for such experiments.

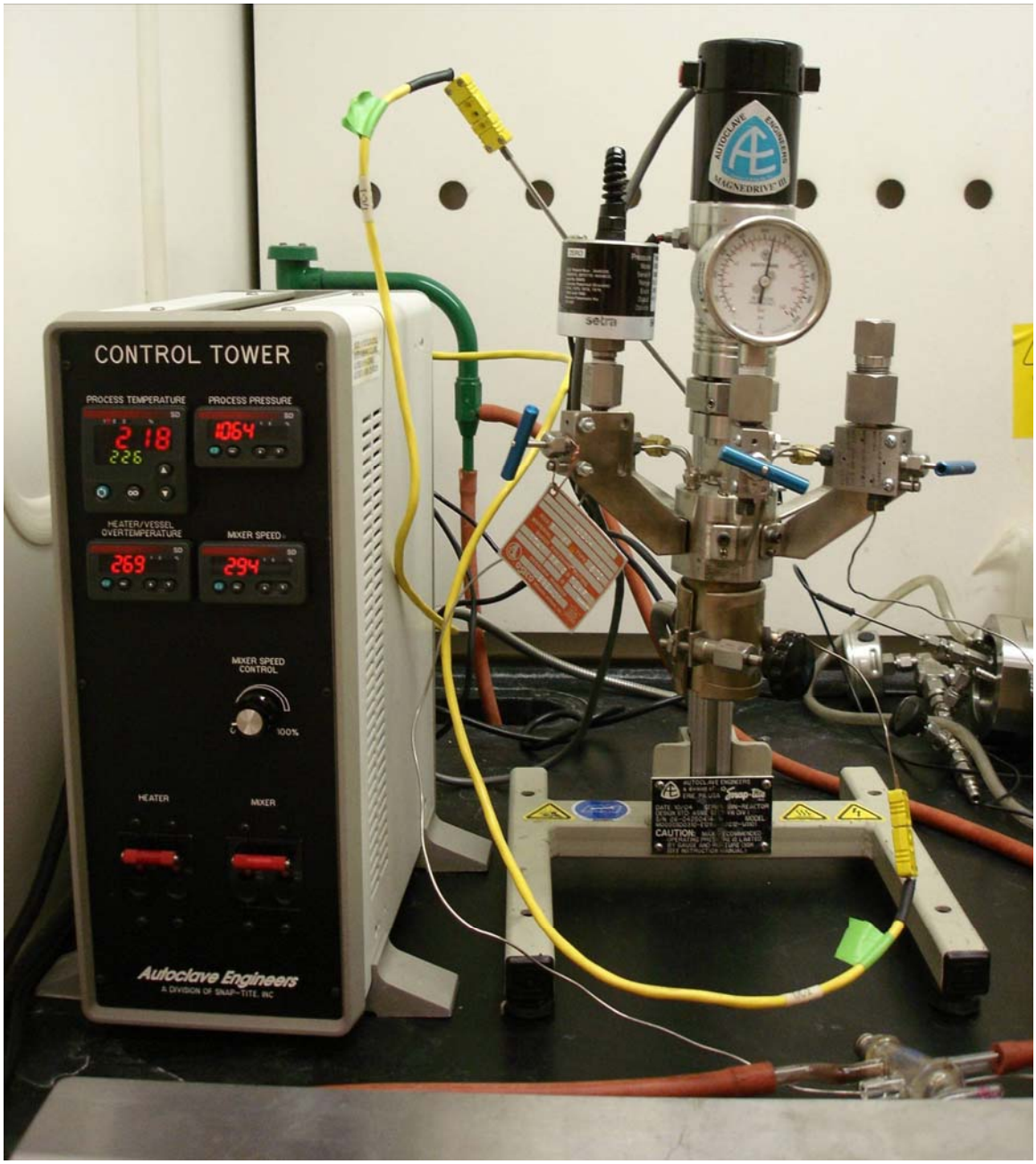
A single hydrogen tank is connected to both minireactors using a T-valve. The tank is generally regulated to 700 psi. Turn the T-valve towards the reactor to be pressurized. The T-valve should be in neutral, the main tank valve closed, and pressure bled from the regulator at all other times.

Familiarize yourself with the Pressure Reactor Checklist, posted in the hydrogenation lab.

Familiarize yourself with the minireactor documentation and the explosion drawings of the reactors. This information is contained in binders in the hydrogenation lab.

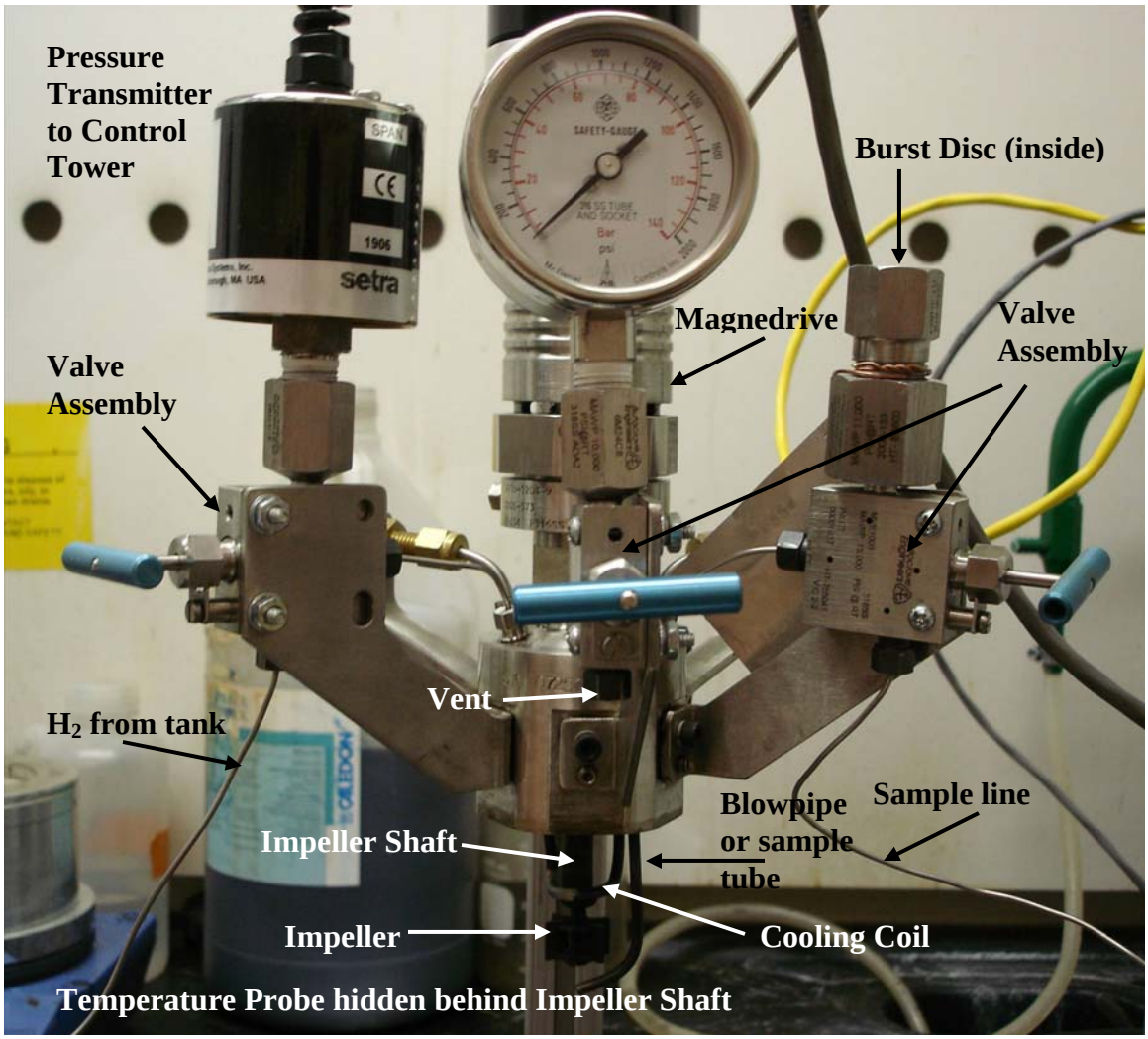
Spare parts (ferrules, nuts, O-rings, bearings, etc.) are stored in a small plastic box. Order more as needed from Autoclave Engineers. There should always be a supply of O-rings and bearings.

The Minireactors



Original Reactor, in Operation

“T-Bone” Reactor



Close-up of Minireactor

Setup

1. Flip the main power switch, located on the rear of the control tower. Flip the stirrer switch on the front of the control tower, but do not turn the stirrer control knob yet.

2. Combine the substrate solution and the catalyst in the reactor vessel. Screw into the reactor body and stir for several minutes. Stop the stirrer, open the reactor, remove 0.5 mL into a GC vial, and reseal the reactor. Reactions are stirred at about 200 rpm. The original reactor is equipped with a digital tachometer. The "T-Bone" reactor does not have a tachometer. Instead, a mark is drawn near the stirrer control knob on the "T-Bone" reactor to indicate approximately 200 rpm.

3. Evacuate the reactor for 2 minutes using the water aspirator (attach hose to vent). Close the valve, remove the hose, and turn the water off. Turning the water off before removing the hose can result in water being sucked into the reactor! Pressurize to 700

psi with H_2 gas, and let equilibrate for 2 minutes. The temperature will increase a few degrees with the rapid increase in pressure. The pressure will drop slightly as the temperature returns to ambient. A more rapid decrease in pressure indicates a leak in the system. LEAKS: Use Snoop to find. Close the main valve on the hydrogen tank. Vent the reactor. Tighten the fitting(s). Pressurize and check for leaks again. NEVER WORK ON A PRESSURIZED SYSTEM!

4. Repeat the evacuation/pressurization cycle twice more.

5. Check that the stirrer is on, and set the reaction temperature, high temperature shutdown (25 °C above reaction temperature), and heating mantle overtemperature (100 °C above reaction temperature). Slide the heating mantle onto the reactor vessel and tighten the screw clamp. Flip the heater switch on the front of the control tower to begin heating. The reaction temperature may need to be set a few degrees higher to achieve the desired temperature.

<u>Setting Temperatures, Original Reactor</u>

Reaction temperature (top left panel): Press up/down arrows to set. The set temperature is the number in green.

High temperature shutdown (top left panel): Press the green circle 4 times. ‘A2h1’ will be displayed in green. Press up/down arrows to set.

Heater overtemperature (bottom left panel): Press the green circle twice. ‘A1h1’ will be displayed in green. Press up/down arrows to set.

Both panels will return to the default display as shown here after 30 seconds. The actual reaction and heating mantle temperatures are in red.

Setting Temperature, "T-Bone" Reactor

Reaction temperature (top left panel): Press up/down arrows to set.

High temperature shutdown (top left panel): Press the 'square' button twice. 'AL LiST' will be displayed. Press the 'circular arrow' button once. '1FSH' will be displayed. Press up/down arrows to set.

Heater overtemperature (bottom left panel): Press the 'square' button twice. 'AL' will be displayed. Press the 'circular arrow' button once. '1FSH' will be displayed. Press up/down arrows to set.

Both panels will return to the default display after 30 seconds.

6. Wait until the set temperature has been reached. Record the pressure from the digital pressure transmitter.

7. BEFORE LEAVING THE HYDROGENATION LAB, ensure the main valve is closed on the hydrogen tank, the regulator is vented, the fumehood sash is lowered, and the safety chains are hooked.

8. For a 24 hour experiment with sampling, samples are typically taken at 1, 2, 4, and 8 hours from reaching the set operating temperature *via* the sample tube. Record the pressure, turn the stirrer off, and flush the sample line with about 0.5 mL of the reaction mixture into a waste vial to ensure cross-contamination from an earlier sample does not occur. Collect 0.5 mL in a GC vial for analysis. Turn the stirrer back on. Record the pressure again.

9. Lower the fumehood sash and hook the safety chains after every sample.

10. At the end of the reaction, record the pressure, turn the heater off, and remove the heating mantle. Place an ice bath under the reactor vessel for 30 minutes to condense any volatile products. Vent the reactor, open it, and take a final sample taken for GC. Transfer the remainder of the reaction solution to a large vial for storage. Vials can eventually be discarded, for example, upon degree completion.

11. Clean the reactor by rinsing it several times with methanol and wiping the vessel, stirrer shaft, reactor head, etc. with a Kimwipe. Rinse the sample tube and sample line by partially filling the reactor vessel with methanol, pressurizing to 200 psi, and

expelling the solution through the sample line. Repeat twice more. Pressurize the empty reactor to 200 psi and vent through the sample line to blow out the methanol. Remove the reactor vessel and allow the reactor assembly to air dry overnight. Turn all switches off. MAKE SURE THE HYDROGEN TANK IS CLOSED AND THE REGULATOR BLED OUT.

Routine Maintenance

1. Regrease the threads on the reactor vessel every 10 experiments or as needed using the Moly grease (molybdenum disulfide). A little goes a long away! More grease can be obtained from the machine shop.

2. Polish the reactor vessel, dip tube, stirrer shaft, and impeller every 10 experiments, or more often if excessive black deposits accumulate. The dip tube and stirrer shaft are polished by hand using fine sandpaper (located in the toolbox in the hydrogenation lab; more can be obtained from the machine shop). The vessel is polished by clamping it in a lathe (machine shop) and using an abrasive pad. The impeller is sandblasted (machine shop).

3. A Teflon O-ring (white) is used to seal the reactor vessel for all reactions up to and including 200 °C. Replace the O-ring every 10 experiments. Kalrez O-rings (black) are ONLY used above 200 °C. They are very expensive and should not be used for routine hydrogenation experiments. The reactor may be difficult to close the first time using a new O-ring. A wrench can be used to seal the vessel into the reactor, then it can be opened and closed by hand as usual. Record in the log book when the O-ring has been changed.

4. Inspect the bearings every 10 experiments, i.e., whenever the reactor has been disassembled for polishing. Replace the bearings every 50 experiments. The original reactor is equipped with Teflon bearings, as the stirrer shaft is slightly out of alignment. These bearings should not need replacing, but if they do the machine shop can make more. The “T-Bone” reactor is equipped with graphite bearings. The minireactor documentation thoroughly describes the procedure for replacing the bearings. Use the bearing tool to push the bearings out of the reactor body. Record in the log book when the bearings have been replaced.

5. Control experiments using no catalyst should be run periodically to determine the baseline activity of the reactor (typically < 2% hydrogenation). The stainless steel reactor contains nickel, a well-known hydrogenation catalyst.

Problems & Their Solutions

LEAKS: Use Snoop to find. Close the main valve on the hydrogen tank. Vent the reactor. Tighten the fitting(s). Pressurize and check for leaks again. NEVER WORK ON A PRESSURIZED SYSTEM!

CLOGS: Occasionally a piece of solid will get stuck either in the sample tube, the curved tubing connecting the reactor body to the sample valve, or in the sample line. First

remove the sample tube and sample line & attempt to flush methanol through manually. If that does not work, sonicate the tube and line in a beaker of methanol for at least 30 minutes. Force air through the tube and line. If solvent and air flow freely through the sample tube and line, then the clog is in the tubing between the reactor body and valve. Disconnect the tubing from the reactor body and the valve from the support arm. Remove the tubing from the valve. Force methanol through manually or sonicate repeatedly. If this does not clear the blockage, then replace the tubing.

REPLACING TUBING: Tubing is held in place by a combination of a ferrule and a screw. Once the ferrule has been compressed onto the tubing, it will not come off. The tubing can be unscrewed from and screwed into the reactor repeatedly. If a piece of tubing needs to be replaced, cut the tubing and remove the screw. Discard the ferrule and tubing. Cut a new piece and slide the screw and a new ferrule onto the tubing. Screw the tubing into the valve or reactor body. For a valve, it is easier to clamp it in the vise and then screw the tubing into it. Use a wrench to tighten the screw as much as possible. BE SURE TO PRESSURE TEST THE REACTOR AFTER REPLACING TUBING!

The machine shop has a supply of tubing. Use the 1/16 in. outer diameter × 0.014 in. wall thickness stainless steel tubing. Double-check that you have the right size by comparing the old to the new. They also have 1/16 in. × 0.020 in. tubing—do not use this, as the hole is too small and prone to clogs.

VALVES: The handles on the valves wear out after awhile. The valve needs to be replaced when it is difficult to completely close. Autoclave Engineers sells a replacement valve assembly, rather than valve parts individually.

BURST DISC: Have the machine shop replace the burst disc if it blows. Bring the valve with the burst disc attachment and a new burst disc from the plastic parts box to the machine shop. Check the reactor manuals for the required torque—the entry is highlighted. Replace the old tag on the reactor with the new one.

Commonly Ordered Parts from Autoclave Engineers

Part	Part Number	Quantity	Note
Valve Assembly	MVE-1003	5	
Teflon O-ring	55977	10	
Kalrez O-ring	64917	2	EXPENSIVE!
Graphite Spring Bearing	126C-6286	10	In reactor body.
Bearing	118B-3511	10	On top of impeller shaft.
Ferrule for 1/16 in. tubing	SSL10	10	Labelled 'Sleeve' on explosion drawing.
Set Screw	66229	2	Locks Magnedrive onto reactor body. Small, therefore easy to lose!
Blowpipe	102D-1781	1	
Burst disc	62204	2	

Other parts: Look at the explosion drawing to locate the part. Find the name and part number in the table on the explosion drawing.

Snoop can be ordered from Swagelok (part # MS-SNOOP-8OZ)