

# Biotage Endeavor Pressure Reactor



## 1 General Comments

1. Currently, the reactors 2, 3, and 4 are known to continuously leak. This is due to chips in the reactor and would require a physical replacement part.
2. For calibration, call Biotage. Do not calibrate pressure and/or temperature on your own.
3. Temperature range is room temperature to 200 °C.
4. Pressure range is 0 to 500 psi.
5. Each reaction vessel can be set up with its own unique temperature and pressure setting. However, stir rate cannot be different.
6. When both heat and pressure are required, program the software to heat first to the desired temperature, wait to reach the desired temperature, then pressurize to desired pressure. Command: heat, wait for, pressure.
7. Default venting in the software vents rapidly. This may lead to loss of majority of the reagents. So, program the venting at the end of the reaction to vent slowly, i.e: lower the pressure, wait, lower the pressure, wait, and continue until the pressure is down to 0 psi. In addition to programmed venting, perform manual vent prior to opening the vessel. This gets rid of residual pressure inside the reaction vessel. Even though LCD screen displays 0 psi pressure, there may be residual pressure left.
8. Use of two different reactive gases at a time is possible.
9. Solution or suspension of reagents can be injected during the reaction. Use blunt needles provided by Biotage for this purpose. This makes sure that the seal in reaction set up does not get punctured.

10. To order supplies for parallel pressure reactor, contact Brian Wagner to provide a quote for supplies:

(a) reaction vessel, part# 900409

(b) impellers, part# 900543

(c) O-rings, part# 105070

## 2 Running An Experiment

### 2.1 Preparing the Reactor

1. Add reagents and solvent to glass liners. Individual channels will only tolerate about a ~20 C difference in temperature between them. For example if 1 reaction is to be run at 60 and one at 120, you'd have to put the first one in a reactor, skip 2 spots, then put the second one in.

2. Place a glass liner tool on top of the reaction vessel.

3. Rotate the black top of glass liner tool clockwise until it holds the reaction vessel freely.

4. Push the reaction vessel down into the reactor vertically (not at an angle) until the grey part of the glass liner tool touches the reactor.

5. Rotate the black top of glass liner tool counterclockwise to loosen it from the glass line

6. Repeat the previous steps with up to 8 reaction vessels.

7. Place O-rings on the reactor for all 8 vessels.

8. Add impellers to the stirrer assembly. Make sure the impellers are held tightly.

9. Place the stirrer assembly onto the reactor unit with injection ports facing you. CAUTION: The stirrer assembly is heavy. Make sure the stirrer assembly seats on top of the reactor unit perfectly. Use the twist knob on the top right of assembly for a perfect fit. Also make sure that the injection ports are not bent and cannot overlap on the O-rings, as this will destroy them.

10. Fit the front and rear clamps. NOTE: Front and rear clamps are different. Front clamps have place holders for injection ports.

11. Use a hexagonal screwdriver with red top to tighten all the screws (8 total) in the order numbered on stirrer assembly. For the first round, tighten screws with a loose fit. Tighten screws down further in the second round until mild resistance is encountered. Repeat one more time. DO NOT OVERTIGHTEN.

12. Leak test the unit with inert gas before starting the run.
  - (a) Open Endeavor Advanced Software on Desktop
  - (b) In Reaction Vessels tab:
    - i. Click on Inert Gas valve.
    - ii. Open valves to reaction vessels 1-8.
    - iii. Close Inert Gas valve.
    - iv. Wait for 2 minutes to check pressure drop.

## 2.2 Programming the Run

1. Open Endeavor Advanced Software on Desktop.
2. Go to Programming tab.
3. Drag and drop the desired functions from left column to the spreadsheet on the right in the order desired.
4. RV volume must be filled in (1-5 mL) depending on the total reaction volume.
5. When both heat and pressure are required, program the run to heat first and then pressurize.
6. After the programmed vent, make sure to perform manual vent before opening the reactor. Manual vent can be performed using the reactor keypad.
7. Refer to Endeavor Catalyst Screening System Quick Cards guide for a sample macro and details on the functions.
8. Save the macros as an .ear file in an appropriate location.
9. Click Run. If this is successful then the connector port will blink blue.
10. Click Save when prompted to save the time stamped run file.

## 2.3 Monitoring the Run

1. On Endeavor Advanced Software, click Listen to start data collection and Stop Listening to stop data collection.
2. The sensor monitor data can also be helpful to show reaction progression (pressure, temperature) if the dosed gas is consumed throughout the reaction.
3. Refer to the Quick Cards guide for more details.

## Contacts

| Instrument                                  | Contacts  |
|---|---|
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