

REACTOR CART STUDENT MANUAL

Ch En 345

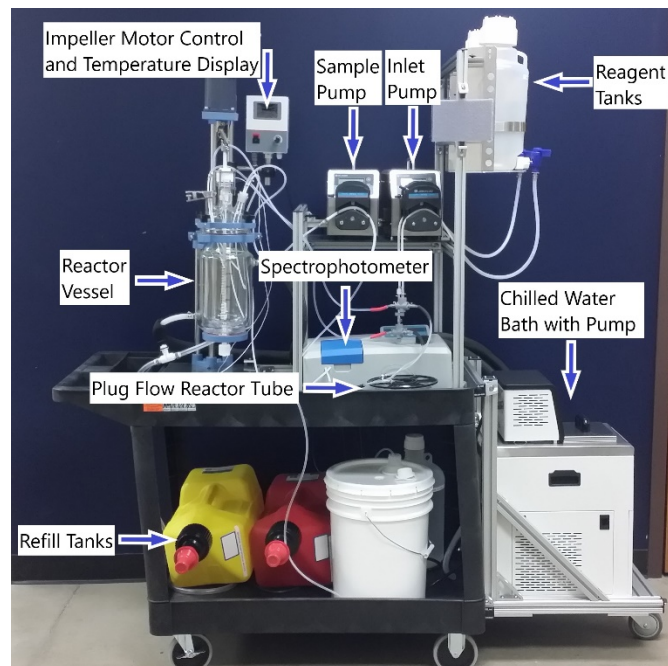


Figure 1 Reactor Cart Apparatus

Apparatus:

The reactor cart consists of the following Components:

- 2 Liter jacketed reactor vessel
- Impeller with motor controller and temperature display
- Thermowell with temperature probe
- Chilled water bath with pump
- Inlet pump
- Sample pump
- Reagent tanks
- Refill tanks
- Spectrophotometer
- Spool of tubing for Plug Flow Reactor
- Diverter valves for switching between stirred tank reactor and PFR

Procedures:

Using the spectrophotometer to measure absorbance of a sample:

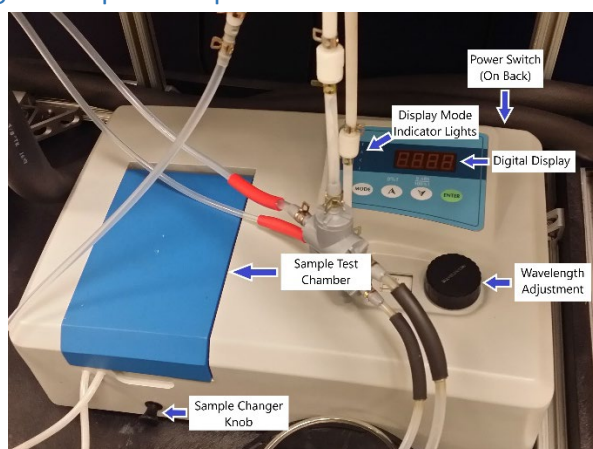


Figure 2 Spectrophotometer

- Start up the spectrophotometer by the power switch on the back of the device. Allow it to warm up for at least 20 minutes before taking any readings.

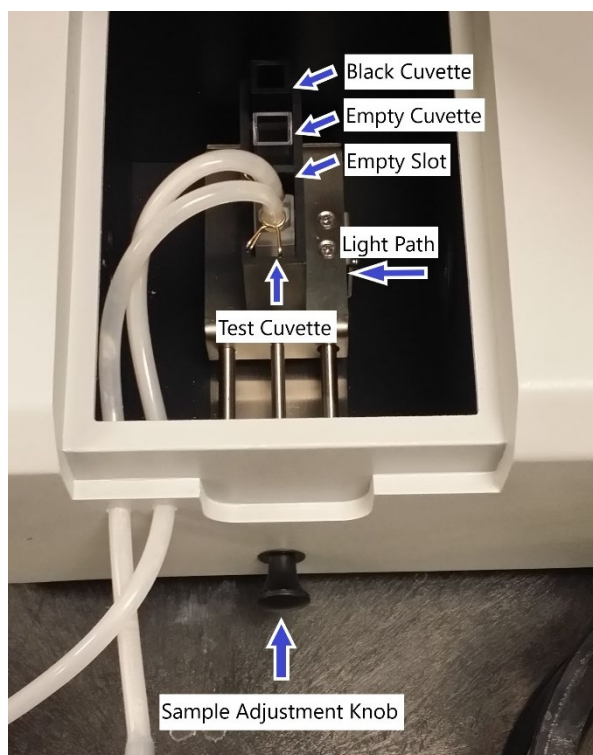


Figure 3 Spectrophotometer Test Chamber

- Open the cover of the spectrophotometer and fill the empty cuvette with RO water.
- Zero the spectrophotometer by pulling the sample adjustment knob on the front to position the black cuvette in the light path. Close the cover. Set the mode to "T" (Transmittance). Press and hold the button with 0%T above it until the display shows all zeroes. Use the sample adjustment knob to place the cuvette with RO water into the light path. Change the mode to "A" (Absorbance). Press and hold the button below $\frac{0 \text{ ABS}}{100\% \text{ T}}$ until the numbers displayed show all zeros.
- Open the cover and position the empty slot in the light path and place your sample cuvette in the slot.
- Close the cover and record the displayed absorbance.
- Open the cover to remove the sample cuvette and add the next sample.
- Repeat steps the previous two steps until all sample absorbances have been recorded.

Reactor Start Up:

General

- Fill the reagent tanks with the desired starting concentration of reagents from the refill tanks. To refill the reagent tanks, undo the clasp on the right side of the tank to release the metal strap holding it in place. Set the reagent tank on the edge of a countertop or table where the spout can hang over the edge. (You may need to unthread the tubing from the pump in order to get

enough slack.) Uncap the tank and pour in the desired liquid until the liquid level has reached the 5 Liter mark. Recap the tank and return it to its holder on the cart. (Reagent concentrations will be written with dry-erase marker on the tag on the reagent tanks)

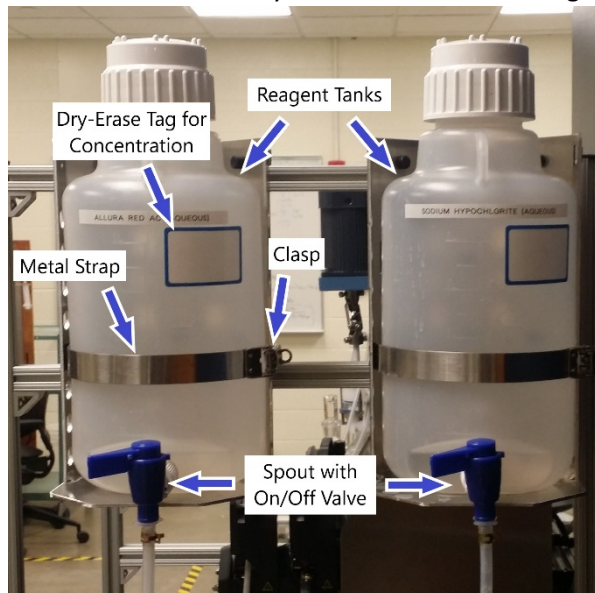


Figure 4 Reagent Tanks

- Plug in the main power bar to the wall outlet

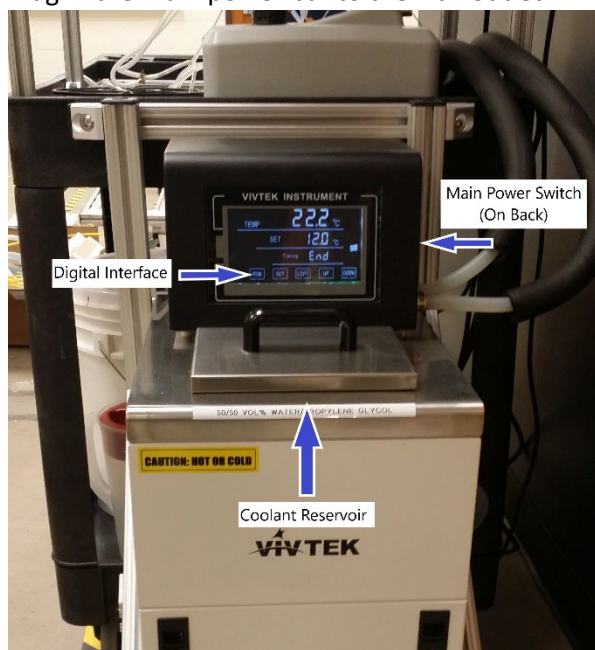


Figure 5 Cooling Bath

- Check the liquid coolant level in the cooling bath (Batch and CSTR only). Lift the lid on the coolant reservoir and make sure the coolant completely covers the silver-colored coils. If more is needed, notify lab staff and more will be added.

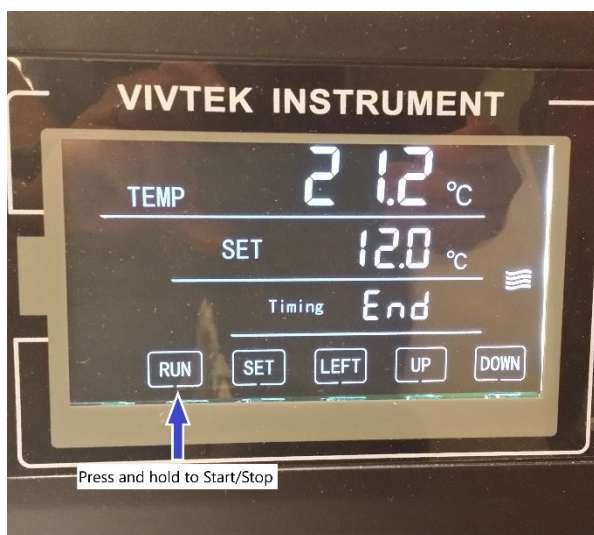


Figure 6 Cooling Bath Interface

- Start the cooling bath (Not for PFR). Turn on the Main Power Switch on the back of the device. When the display turns on, a touchscreen interface will be available. To start cooling, press and hold the RUN button until the cooler starts.
- Allow cooling water to reach set temperature (About 10-15 minutes)

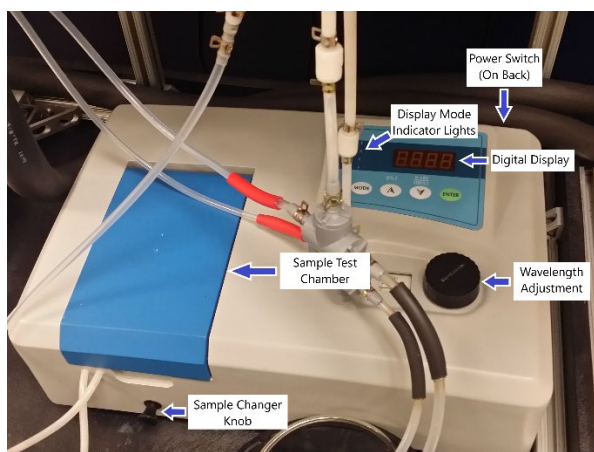


Figure 7 Spectrophotometer

- Start up the spectrophotometer by the power switch on the back of the device. Allow it to warm up for at least 20 minutes before taking any readings.

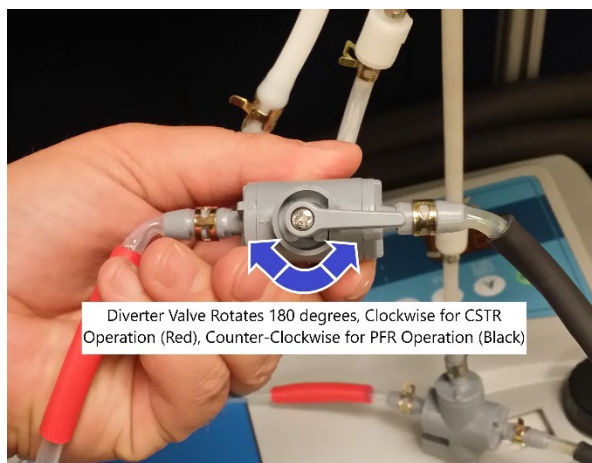


Figure 8 Diverter Valve

- Ensure that all diverter valves are set to the correct flow direction (red for batch/CSTR, black for PFR)

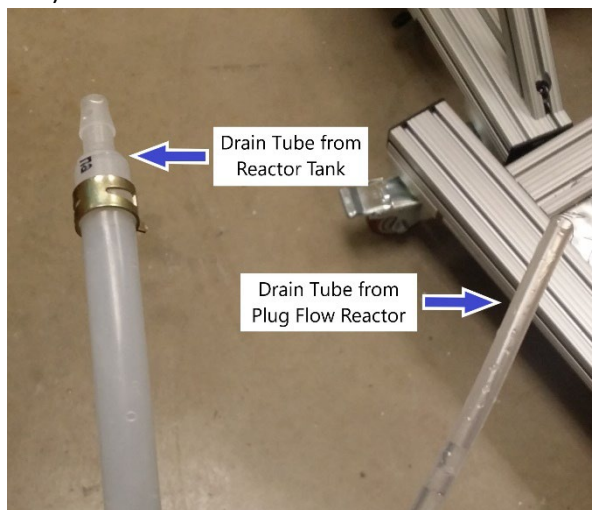


Figure 9 Drain Tubes

- Make sure the drain line is inserted into the lab floor drain

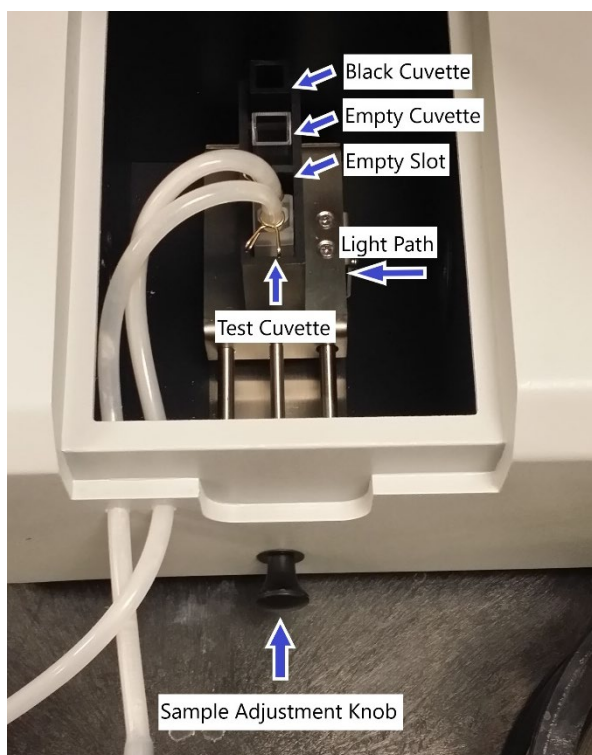


Figure 10 Spectrophotometer Test Chamber

- Open the cover of the spectrophotometer and fill the empty cuvette with RO water.
- Zero the spectrophotometer by pulling the sample adjustment knob on the front to position the black cuvette in the light path. Close the cover. Set the mode to "T" (Transmittance). Press and hold the button with 0%T above it until the display shows all zeroes. Use the sample adjustment knob to place the cuvette with distilled water into the light path. Change the mode to "A" (Absorbance). Press and hold the button below $\frac{0 \text{ ABS}}{100\% \text{ T}}$ until the numbers displayed show all zeros.
- Return the test cuvette to the light path. DO NOT adjust the wavelength knob, this will be done by a TA, if needed.

Batch

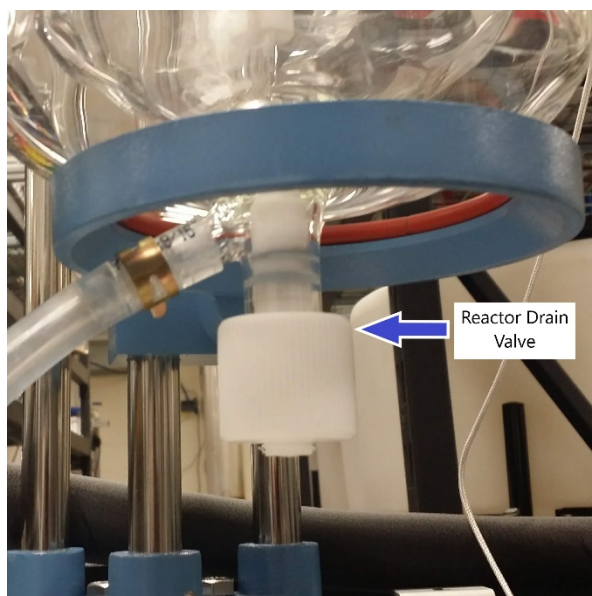


Figure 11 Reactor Drain

- Make sure the drain at the bottom of the reactor is closed by turning the knob counter-clockwise

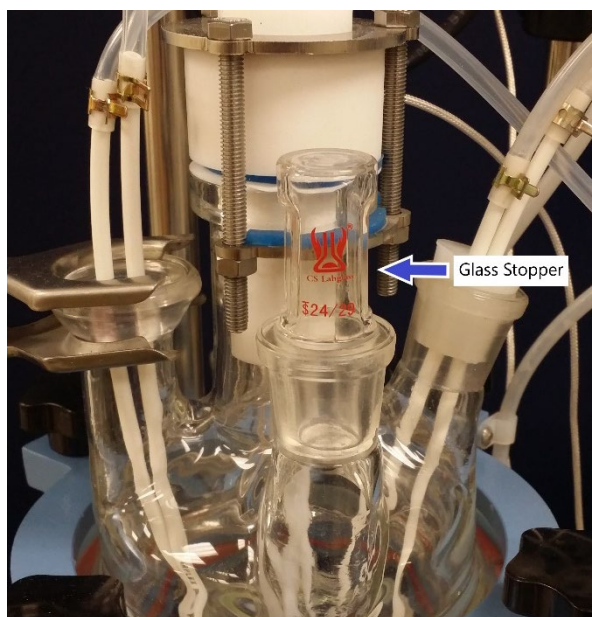


Figure 12 Top of the Reactor

- Remove the glass stopper in the top front of the reactor

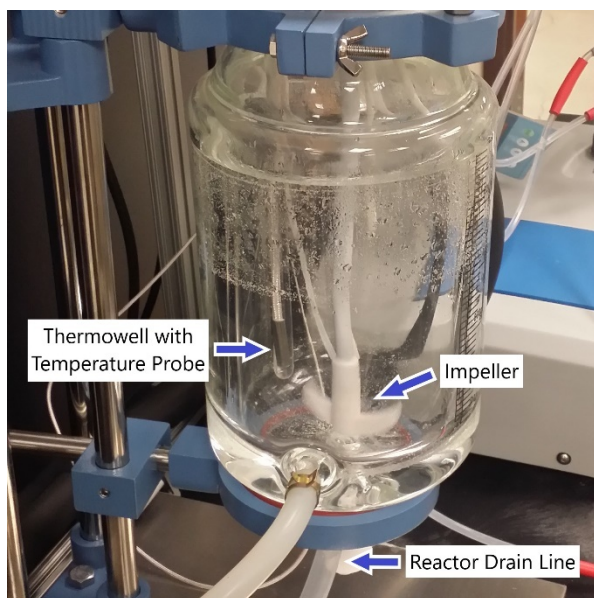


Figure 13 Reactor Tank Components

- Make sure the temperature probe is fully inserted into the thermowell

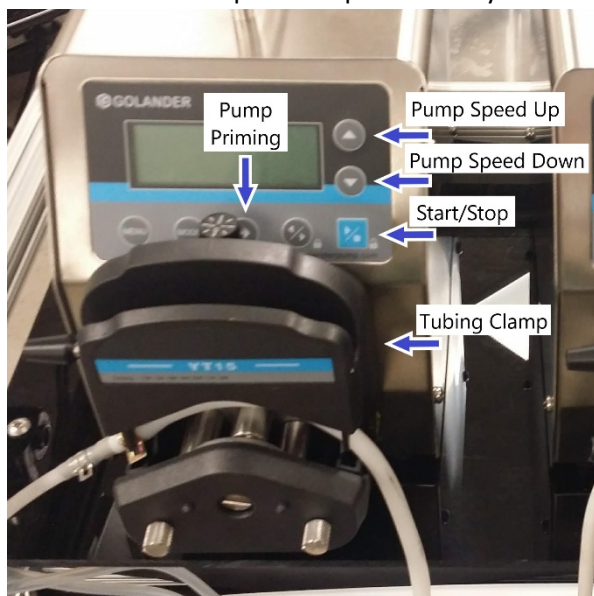


Figure 14 Pump Controls

- **Loading from the top of the reactor:**
- Insert a funnel into the top of the reactor where the stopper was taken from and gradually pour in the first reagent (the one with the greater volume), adding the desired volume

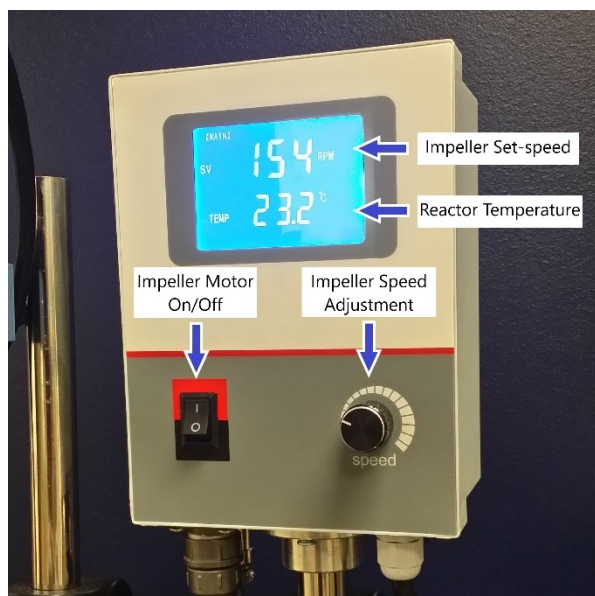


Figure 15 Impeller Motor Controls

- Turn on the impeller motor controller to read the reactor temperature
- Start the impeller motor at the controller above the reactor vessel. Set the motor speed by adjusting the knob on the controller (Usually between 175 – 250 rpm)
- Allow the reagent to cool (~10 mins)
- Close the clamp on the sample pump by pulling the black knob from left to right
- Make sure the pump is set to rotate clock-wise (pulling liquid from the bottom of the reactor)
- Start the sample pump
- Wait a few seconds to make sure the sample is flowing through the spectrophotometer. If there are any issues, get help from a TA
- Set the zero on the spectrophotometer
- Record the reactor temperature
- Add the second reagent quickly through the top of the reactor
- Replace the glass stopper
- Take readings from the spectrophotometer and temperature display once it reaches its maximum absorbance reading and every 10 or 30 seconds to monitor reaction progress

CSTR

- Open the valves on the reagent tanks
- Open the caps on top of the reagent tanks slightly to vent
- Turn on the impeller motor control and start the impeller motor at the controls above the reactor vessel
- Set the motor speed by adjusting the knob on the controller (Usually between 150 – 250 rpm)
- Make sure the drain at the bottom of the reactor is closed
- Remove the glass stopper in the top front of the reactor
- Close the tubing clamps on the inlet pump for both reactants and on the sample pump by sliding the black knob from left to right

- Start the inlet pump to load the reactants into the reactor vessel
- Fill the reactor to the desired level with reactants. Use the pump prime button to get maximum pump speed to fill the reactor (About 150 rpm) until the level in the reactor reaches between 800 and 1000 ml (Each mark on the reactor tank indicates 40 ml of fluid)
- At about the 300 – 400 ml level start the sample pump and make sure fluid is circulating between the reactor and spectrophotometer.
- Once the fluid in the reactor has reached the desired level press the pump prime button again and reduce the reactor inlet flow rate on the inlet pump (generally between about 50 – 75 rpm)
- Take readings from the spectrophotometer immediately and every 30 – 60 seconds to monitor reaction progress
- Open the drain at the bottom of the reactor and open the drain control valve about halfway
- Replace the glass stopper

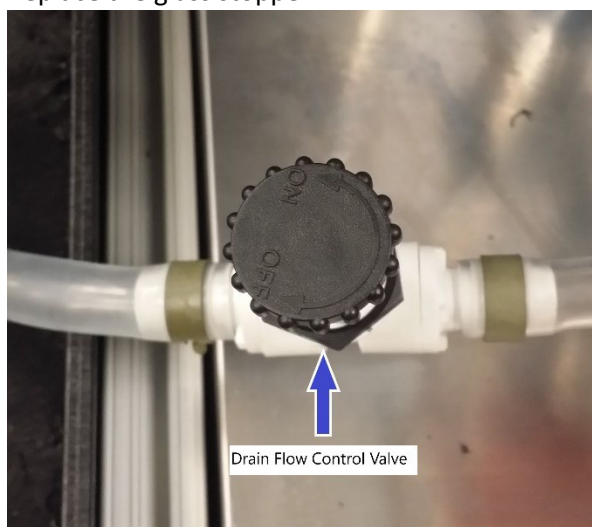


Figure 16 Drain Flow Rate Control Valve

- Maintain the liquid level with the drain control valve. Open (Counter-clockwise) is labeled “On”, closed (Clockwise) is labeled “Off”
- Adjust the reactor drain control valve as needed to maintain desired reactor level, keeping the reactor filled between 800 ml and 1200 ml
- Monitor the absorbance values until the values stop changing significantly over time. This will be your steady-state value

PFR

- Open the valves on the reagent tanks
- Open the caps on top of the reagent tanks slightly to vent
- No cooling is used
- Turn the diverter valves to the tubes marked with black
- Make sure the clamp is open on the sample pump
- Set the inlet pump rate and start the inlet pump
- Take readings from the spectrophotometer to determine outlet concentration of red dye
- Adjust the inlet pump rate to change the residence time in the PFR

Shut Down:

Batch

- Take final readings and turn off the spectrophotometer
- Stop the sample pump and open the tube clamp
- Turn off the impeller at the controller above the reactor
- Open the drain valve at the bottom of the reactor
- Remove the glass stopper in the top of the reactor
- Completely drain the reactants
- Close the drain valve
- Using Deionized or Reverse Osmosis water, rinse the reactor by filling it and draining it
- Leave the reactor drain valve open
- Replace the glass stopper
- Turn off the cooling bath

CSTR

- Stop the inlet pump and open the tube clamps
- Close the valves on the reactant tanks
- Take final readings and turn off the spectrophotometer
- Stop the sample pump and open the tube clamp
- Turn off the impeller at the controller above the reactor
- Remove the glass stopper at the top of the reactor
- Open the drain valve at the bottom of the reactor
- Drain the reactor completely
- Close the drain valve
- Rinse the reactor vessel using Deionized or Reverse Osmosis water
- Drain completely and leave the drain valve open
- Replace the glass stopper
- Turn off the cooling bath

PFR

- Stop the inlet pump and open the tube clamps on the pump
- Revert the diverter valves from black to red
- Take final reading and turn off the spectrophotometer
- Close the valves on the reactant tanks

Additional Notes:

If the sample pump is having difficulty drawing liquid, remove the glass stopper and tighten the tube clamp very slightly. To prime the sample pump, press the button with the two forward arrows, this will

cause the pump to run at max rpms. Once liquid has started flowing through the tubing, press the button again to return the pump to normal speed. If the previous procedure does not work, you may need to drain a small amount of fluid through the tubing until it reaches the pump by switching the diverter leading to the PFR drain-line from red to black. Once the sample line has started flowing, switch it back to red.

Draining the vessel will generate a vacuum within the reactor, so you may need to vent the reactor to drain it.

Be aware that, if the inlet pump is running while the drain is closed, the glass stopper must be off in order to prevent pressure buildup in the reactor. The pressure can build up enough to pop the stopper off violently, creating a potential hazard for people and equipments.

Be sure to turn off the spectrophotometer as soon as finished with it in order to prolong the life of its bulb.

Changing between reactor types will be performed by the TA.