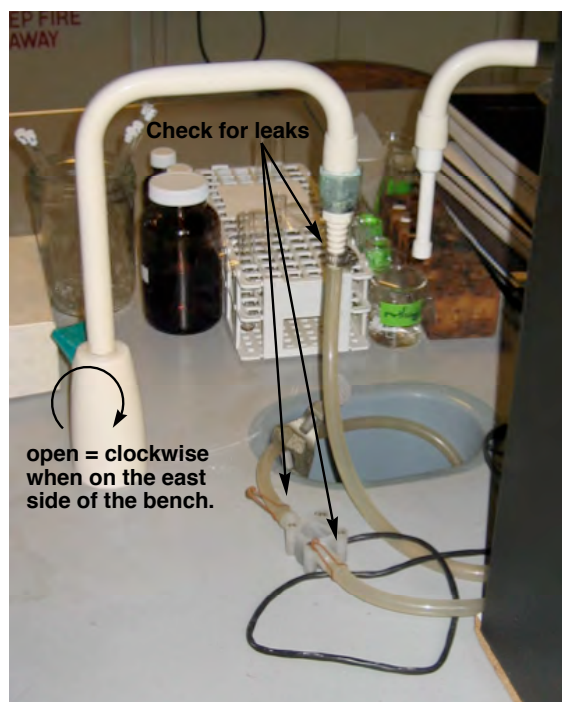


Instructions and checklist for using the Medium Pressure Mercury Lamp

- I. Cooling the Lamp.** Since the lamp may reach over 1000 °C, it is important to have adequate cooling throughout its operation. This is accomplished by placing the lamp in a quartz-jacketed condenser through which tap water can be circulated continuously. *Important: anytime you work with running water for cooling, it is imperative that you check all connections for leaks. A flood can cause major damage to the building. Floods can also initiate electrical fires and even electrocution.*

- ___ Turn the green knob of the faucet to the left of the photobox to allow water into the system.
- ___ Lift up the end of the tubing that goes into the drain to check the water flow. The flow should be fast enough to cool the lamp, but not too fast that it pops the tubing off, causes leaks, or overflows the sink.
- ___ Place the end of the tubing back down into the drain of the faucet. The metal clamp should be positioned as shown to keep the tubing from popping out of the drain.
- ___ Check all connections outside of the box for leaks (shown in the photo). *Note: if there are leaks, turn off the water, and either fix the leaks or inform Dr. Poon immediately.*



II. Turning on the emergency flow meter.

If the water stops reaching the condenser at any time, the lamp will make the interior of the box extremely hot. This could melt many of the items in the box and also cause a fire. A flowmeter has been installed so that the lamp will turn off if water flow is disrupted.

- ___ The flow meter is to the right and the rear of the photobox. Flick the switch on the right side of the box to “on” (upper half depressed).
- ___ The green light should come on and stay on. If it turns off, your flow is too low. If too low, turn the flow meter off before adjusting the flow, then turn the meter back on.
- ___ At this point, turn on the multi-outlet strip and the thermometer, too.



On/off switch

Thermometer

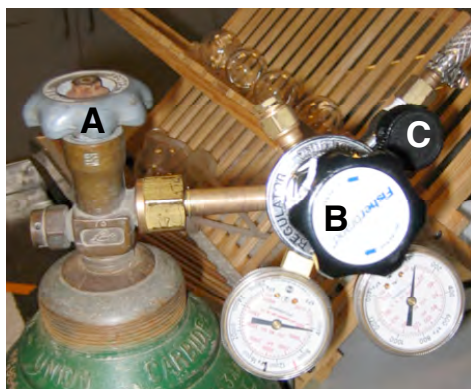
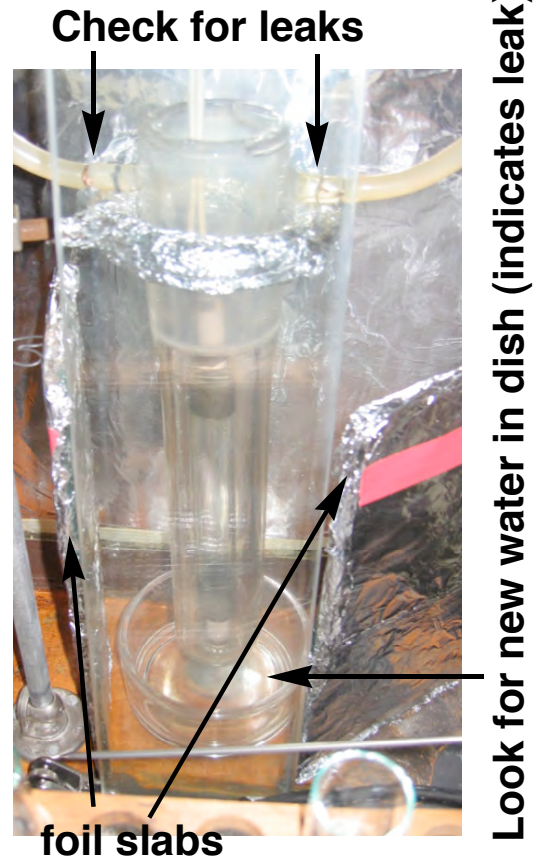
Power supply to the fan & stirrer.

III. Photobox safety checks. Check the lamp housing for leaks. If you spot any, contact Dr. Poon (do not attempt to fix leaks in the lamp housing).

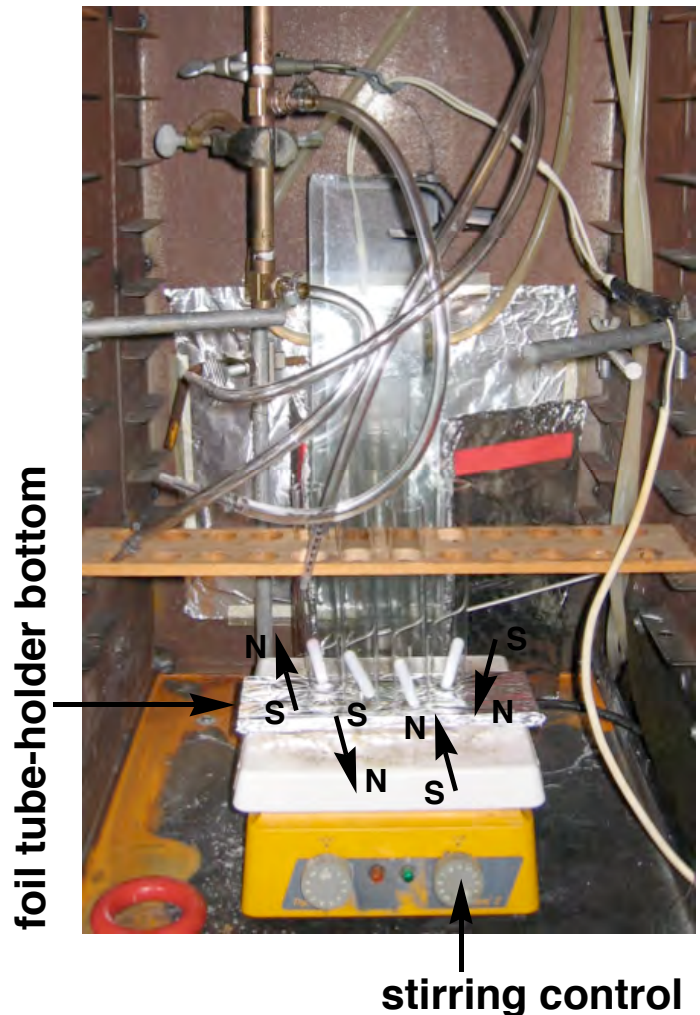
- ___ Check for leaks by observing whether drops of water form at the hose connections, or whether new water collects in the dish under the lamp.
- ___ Make sure there are no kinks in any of the tubing inside the photobox.
- ___ Make sure the foil slabs are resting in the positions shown. They block unwanted UV light from hitting our samples.

IV. Setting up your samples.

- ___ Add stir bars to your test tubes. Be sure that they alternate in polarity as shown.
- ___ Seal each test tube with a septum.
- ___ Add a vent needle to each septum.
- ___ Connect the long O₂ (or N₂) delivery needles to the manifold tubing. **DO NOT INSERT NEEDLES INTO SAMPLES YET.**
- ___ Place the samples into the wooden holder. Line up the tubes with the indentations in the foil tube-holder bottom.
- ___ Use the stirring control knob to activate the stir bars. Slide the stirring plate into the best position for stirring all the bars.
- ___ Turn off the stirring.
- ___ At the oxygen tank, follow the steps shown in the illustration for introducing O₂ (or N₂) to the needles.



- 1) make sure "C" is shut.
- 2) make sure "B" is shut (loose)
- 3) open "A"
- 4) turn "B" until 15 psi is reached
- 5) open "C" slightly check the flow coming out of a needle
- 6) adjust "C" to get slow-mod. flow



- ___ When the O₂ (or N₂) flow is satisfactory, insert the needles into the test tubes such that they are slightly above the stir bar. (IMPORTANT: Did you remember to insert the vent needles earlier?)
- ___ Make sure the flow is not too high (will cause too much solvent to evaporate) or too low (will clog up and stop working over time).
- ___ After needles have been inserted into all the samples. Recheck the O₂ (or N₂) flow.
- ___ Turn on the stirring. The spin rate should be just enough so that the zeolite powder rises at least half-way up the tube. If the spin rate is too high, then the powder will just get stuck to the upper sides of the test tube walls (bad news).
- ___ Push the test tubes down into their slots in the foil tub-holder bottom.
- ___ Close the door and engage the latch. Attach the hook to the screw.

V. Running the photolysis. It is now time to turn on the Hg lamp and irradiate your samples. NOTE: the light released by the lamp is high intensity UV light. Looking at it could result in severe eye damage. Exposure of skin to the light could cause severe sunburns. Therefore, it is important to always keep the box sealed while the lamp is on.

- ___ Make sure the felt cloth adequately covers all openings into the photobox.
- ___ Flick the on/off switch on the power supply to “on” (the up position).
- ___ Check the felt cloth for small openings where light can escape and adjust to cover these up.
- ___ Record the time when you first turn on the lamp. Wait 10 min and then record the temperature.



On/off switch

VI. Shutting down. These next steps will guide you through the procedure for shutting down the system and obtaining your samples. NOTE: the light released by the lamp is high intensity UV light. Looking at it could result in severe eye damage. Exposure of skin to the light could cause severe sunburns. Therefore, it is important to always keep the box sealed while the lamp is on.

- ___ Flick the on/off switch on the power supply to “off” (the down position).
- ___ Record the time when you turn off the lamp. Record the temperature as well.
- ___ Turn off the water at the faucet (rotate the knob counterclockwise if you are facing the faucet from the east side of the bench).

- ___ Lift up the tubing in the drain to check that the water no longer flows.
- ___ Turn off the water flow meter (which should be chirping by now).
- ___ Check to make sure the lamp in the box is not on. Then open the door to the photobox.
- ___ Observe whether stirring is still occurring in each tube, then turn off the stirring.
- ___ Allow the zeolites to settle, then observe whether bubbling is still occurring in each tube.
- ___ Remove the *long needle* from each test tube.
- ___ Turn off the O₂ (or N₂) at the tank using the following steps...



- 1) shut "C" (clockwise turn).**
- 2) disengage "B" (counterclockwise turn until the knob is loose).**
- 3) shut "A" (clockwise turn).**

- ___ Turn off the multi-outlet strip.
- ___ Place the long needles in the sonicator to clean for 10 min., then rinse with water, then squirt acetone through each one. Place them to dry in the 100 degree oven.
- ___ Remove the vent needles from your samples. Place their plastic sheaths back on and throw them into the sharps can.

VII. Working-up your zeolite reactions.

- ___ Centrifuge your samples.
- ___ Remove septa and save for reuse if they didn't get dirty or too full of holes from the needles.
- ___ Carefully pour off the supernatant (save just in case) being careful not to let the stir bar move too much or fall out.
- ___ Add acetonitrile to the usual level.
- ___ Cover tubes with Seal film (do not use parafilm)
- ___ Stir overnight to extract your sample.

The next day...

- ___ Remove the stir bar.
- ___ Centrifuge samples
- ___ Pour extract solvent into 30-50 mL luer-lock syringe fitted with a 0.2 μm filter.
- ___ Filter solvent into a **clean** round bottom flask. Bake in oven to remove grease if necessary.
- ___ Rotovap.
- ___ Use minimal amount of clean acetonitrile to get the product residue out of each round bottom and combine similar samples into an amber glass vial.

— You might want to place 2 drops of this into a GC/MS vial, dilute with acetonitrile, and run a GC/MS (ask Nicole for help) to analyze the products.