

Morris HPS-3210 Pressure Furnace

Standard Operating Procedure

1 PREPARING THE GROWTH

It is important that the ampules used in the furnace not be sealed shut. Due to the dimensions of the furnace quartz tubes of < 8 mm O.D. must be used. The length of the quartz tube for each growth should be long enough that it can be pulled out of the furnace with tweezers or needle-nose pliers, but also short enough to leave room for the cavity cap. Ideally, such a length is roughly 42cm, but the length should be checked each time to be sure.

2 LOADING THE GROWTH

- To open the furnace, the “unlock enabled” button must be held, for the furnace locking switch to be moved into the “release” position.
- Once the locking switch has been moved to the “release” position, the handle can be used to lift open the furnace lid. If the “quench” button is lit at any point, that means the lid of the heating element has come open and it must be shut before the start of any heating profile.
- When the furnace is not actively in use, the pressure chamber should be open, i.e. the cap to the chamber should be removed.
- To load the growth, first make sure all of the reactant materials are at the bottom of the quartz tube, then insert the tube – closed end first – into the pressure chamber, pushing it as far in as it can go (AFIACG).
 - AFIACG means either until the end of the tube hits the back of the pressure chamber, or until the tube can no longer easily be held by the tweezers. Ideally the first case should happen.
- After the quartz tube is inserted, the cap to the pressure cavity should be screwed on. The surfaces of the cap that go inside, or seal off the pressure cavity should be wiped down with ethanol every time before use. It is also often a good idea to make sure there is a thin film of vacuum grease on the o-ring of the cap. **BE CAREFUL NOT TO OVERTIGHTEN THE CAP.**
 - When screwing in the cap, only the threaded ring attached to the pressure chamber should move. The cap has multiple connected parts, and moving them too much can break their seals and open a leak.
- Once the cap has been screwed on, the lid to the furnace should be closed and the locking switch moved back to the “lock” position (no button needs to be pressed to lock the furnace).

3 FLUSHING THE FURNACE

Once the growth has been inserted into the pressure cavity and the cap has been sealed, the volume of the pressure cavity needs to be purged of air.

- Make sure the valve on the pressurized Argon bottle is open.
- Open the “fill” valve (farthest left handle on the furnace control panel) slowly until you see the reading on the digital pressure gauge (the left gauge).
- Let the pressure increase to > 90 bar.
- Close the “fill” valve. **BE CAREFUL NOT TO OVERTIGHTEN THE VALVE.**
- Only after the “fill” valve has been closed, open the “vent” valve slowly
- Let the pressure decrease back down, but not below 2 bar.
- **IF AT ANY POINT DURING THE VENTING PROCESS, YOU SMELL SELENIUM (A ROTTEN EGG SMELL, SIMILAR TO SULPHUR). CLOSE THE VENT VALVE IMMEDIATELY AND LEAVE THE GENERAL AREA AROUND THE PRESSURE FURNACE FOR AT LEAST 10 MINUTES.**
- Close the “vent” valve. **BE CAREFUL NOT TO OVERTIGHTEN THE VALVE.**
- Repeat the previous steps in this section (except checking the Argon bottle valve) at least 3 more times, for a total of 4 purges of the pressure cavity.
- Open the “fill” valve again, but only long enough for the pressure inside the cavity to reach the desired pressure for beginning the growth.
- Once the pressure has reached the desired value, close the “fill” valve and let the pressure furnace sit at that pressure for at least 10 minutes (recommended 30 minutes), to verify that there is no leak in the pressure chamber.
- Once it has been confirmed that the pressure chamber is not leaking (i.e. the pressure has not gone down significantly), the heating profile can be started.

4 PROGRAMMING THE FURNACE

The temperature controller is the digital panel to the far right on the control panel of the pressure furnace. It works like most temperature controllers, with a couple small differences.

- The programming menu items are **program ramp (Pr), ?? (Or), program level (PL), program dwell (Pd), ?? (Od)**.
 - The programming ramp (Pr) is in units of degrees per minute, while the programming dwell is in hours.
 - Programming dwell (Pd) is also just the dwell time for that step in the program, not total program time.

Other than those details, the temperature control program system works just like the programming systems for the other older Lindberg furnaces (furnaces 6 and 9).

IF THE HEATING BUTTON DOES NOT LIGHT UP WHEN YOU PRESS “RUN” FOR YOUR HEATING PROGRAM, THERE IS A PROBLEM.

- *There are many safeguards to make sure the pressure furnace does not explode and therefore if one of those safeguards is registering a problem, the furnace will not run even if the temperature controller is running the program.*
 - *Check to make sure the heat key is turned to the “on” setting.*
 - *Make sure the “quench” button is not lit*

- *Make sure the “reset required” and/or “overtemp event” buttons are not lit. If either is lit, press the “reset required” button.*
- *Make sure the locking knob is in the “locked” position*
- *If all of these things check out, only then make sure that the pressure chamber is sealed correctly.*

5 REMOVING THE GROWTH

Once the temperature profile has finished, wait until the furnace is below 200 °C before releasing the pressure. Ideally the furnace temperature should be all the way down to room temperature. Make sure you understand what possible elements or chemicals could be airborne inside the pressure chamber (Selenium Oxide, a dangerous compound, for example). **TAKE NECESSARY SAFETY PRECAUTIONS.** Slowly open the “vent” valve to release the pressure, letting it drop down to atmospheric (between 0.96 and 1.16 bar on the pressure gauge). Only then should you unlock and open the furnace, unscrew the cap, and remove the growth. Similar to when purging the pressure chamber: ***IF AT ANY POINT DURING THE PROCESS -- VENTING OR OPENING THE PRESSURE CHAMBER -- YOU SMELL SELENIUM (A ROTTEN EGG SMELL, SIMILAR TO SULPHUR). CLOSE THE VENT VALVE IMMEDIATELY AND LEAVE THE GENERAL AREA AROUND THE PRESSURE FURNACE FOR AT LEAST 30 MINUTES.***