STANDARD OPERATING PROCEDURES: ROMAG GALLIUM CLEANING SYSTEM

Ashna Aggarwal

aaggarwal01@ucla.edu

July 2, 2017

1 Purpose

The purpose of this SOP is to present RoMag's gallium cleaning system's design (Section 2), design of the fume hood in particular (Section 3), and methods of Operation (Section 4).

2 System Design

Gallium is one of the working fluids used in the convection tanks for experimentation. Storage and preparation of the metal is an important subsystem of RoMag.

*Note: All proper PPE, including gloves, a labcoat, and face mask, should be worn before cleaning gallium.

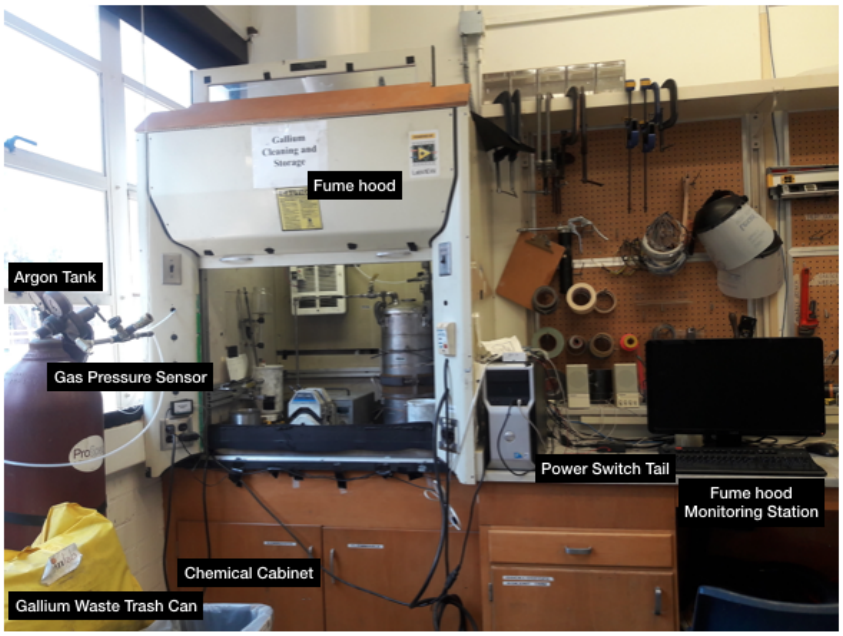

Figure 1: The main components of the gallium cleaning system, including a fume hood and argon tank (left), fume hood monitoring station (right), and chemical cabinet (below).

More specifically, the gallium cleaning system, shown in Fig. 2 allows the user to circulate gallium in order to remove gallium-oxide (which has different fundamental properties than pure gallium) that is generated when gallium contacts air. The major components of the

system are

- The Fume Hood, a confined and semi-sealed compartment where the gallium is stored and circulated
- The Fume Hood Monitoring system, where the temperature of the fume hood is monitored and the pressure (and thus height of gallium) of the cleaning tank is monitored.
- The Argon Tank, used to flush the pipes with argon gas
- A Vernier Gas Pressure Sensor (GPS), used to monitor the pressure (and thus the height) of the gallium in the cleaning tank
- A Power Switch Tail, a relay controlling the power to the Peristaltic Pump in the fume hood. If the Labview pressure monitoring system (based on the GPS) detects a level of gallium in the cleaning tank that is too high, the pump is shut off via SensorDaq analog output to prevent overflowing.
- A series of thermocouples to measure the temperature of the fume hood
- A Gallium Waste Trash Can, where all gallium related waste should be discarded.
- A Chemical Cabinet mainly used to store hydrochloric acid

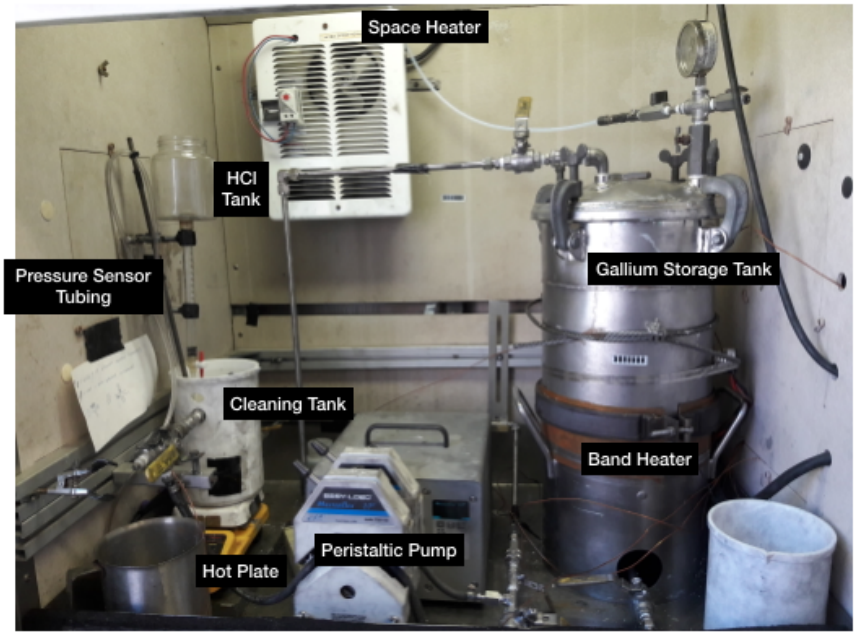

Figure 2: The major components of the fume hood are the gallium storage tank, peristaltic pump, cleaning and HCl tanks, hot plate, space and band heater, and the pressure sensor tubing.

3 Fume Hood

All of the cleaning process takes place in the fume hood. The major components of the fume hood are:

- A 20 liter Binks Stainless Steel storage tank. Stainless steel (SS 316L) was chosen since both copper and aluminum react with gallium. To open the tank, use the four clamps at the top of the tank. When not in use for experimentation, SPINlab's gallium supply should be stored in this tank. See Fig. 3.

Figure 3: When not in use, gallium is stored in this stainless steel tank.

- A Masterflex I/P Easyload Peristaltic Pump (model #:77601-00) that pumps gallium through the system when either (1) cleaning the gallium or (2) transferring the gallium from the fume hood to RoMag's convection tank. The speed of the motor is adjusted with the knob on the right side of the pump. For a photograph of the pump, see Fig. 4.

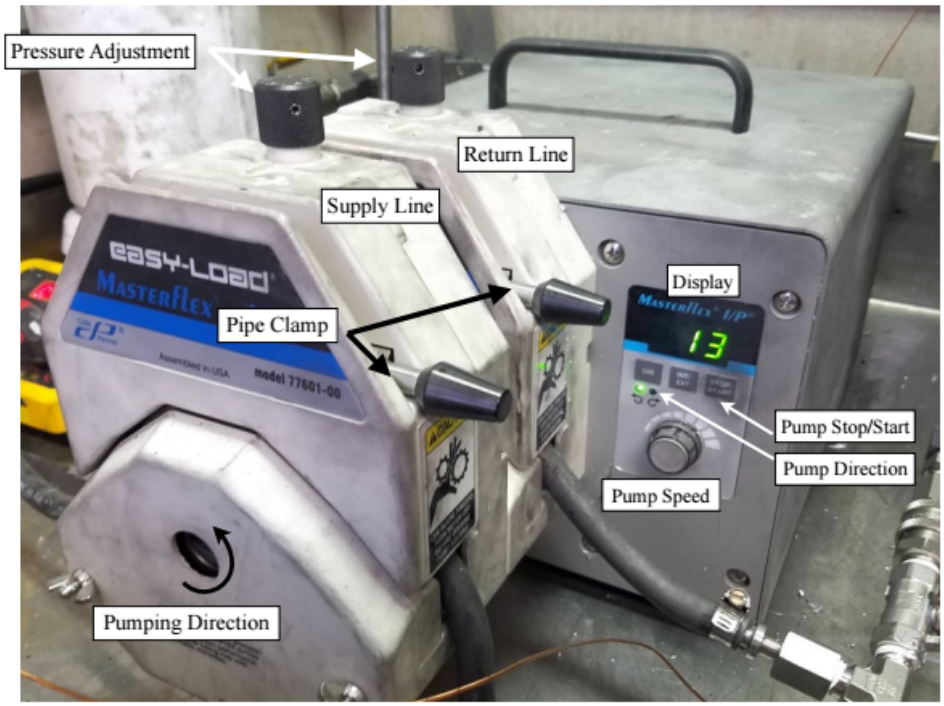

Figure 4: The Masterflex I/P Easyload Peristaltic Pump used to transfer gallium between separate tanks. Image and caption contributed by Alex Grannan

- Cleaning tank where the gallium and HCl-water mixture is combined and stirred with a hot plate
- The Vernier Gas Pressure Sensor Tygon tubing, zip-tied to a stainless steel rod for support
- A hot plate with magnetic stirrer used for stirring the gallium in the cleaning tank
- Space heater used to keep the fume hood warm and maintain the gallium in its liquid phase during cleaning

4 Operation

The following list is a series of phases and steps that should be taken to clean the gallium and maintain the cleaning system. The plumbing system should be assembled as shown in Fig. with all ball valves closed.

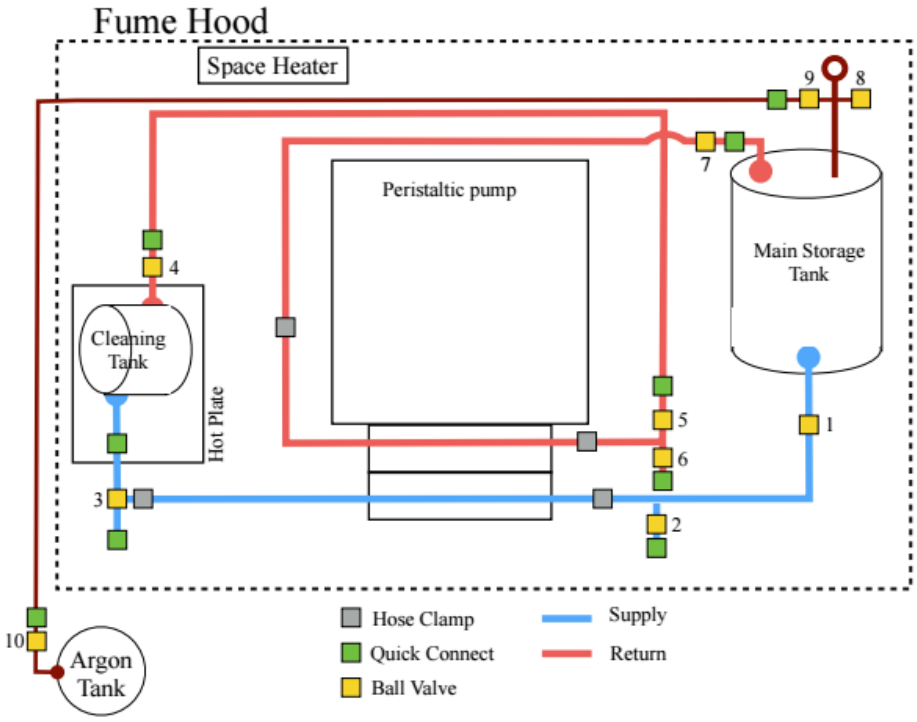

Figure 5: Schematic of the fume hood plumbing system. The supply line (blue) begins at the main storage tank, and ends at the cleaning tank. The return line (red) begins at the cleaning tank, and ends at the storage tank. The ball valves (yellow) can be open and closed, allowing the user to control the flow through the piping. Finally, the quick-connects (green) allow the user to disconnect and reconnect certain sections of the piping. Image and caption contributed by Alex Grannan

I outline the process in three phases: preparation - steps that should be taken to prepare the fume hood and monitoring programs for cleaning), cleaning - the main process of cir-

culating the gallium through the plumbing system, and flushing - using argon gas to 'flush' residual oxygen out of the system.

4.1 Preparation

1. Turn on the Labview temperature monitoring system. Navigate to a folder on the desktop labeled '*Ga_clean_temp*', and select the file labeled *Ga_cabine_tempmonitor_2.vi*. The front panel of the program is shown in Fig. 6

2. The band heater and space heater should be plugged into 208 VAC single phase outlets located to the right of the fume hood.

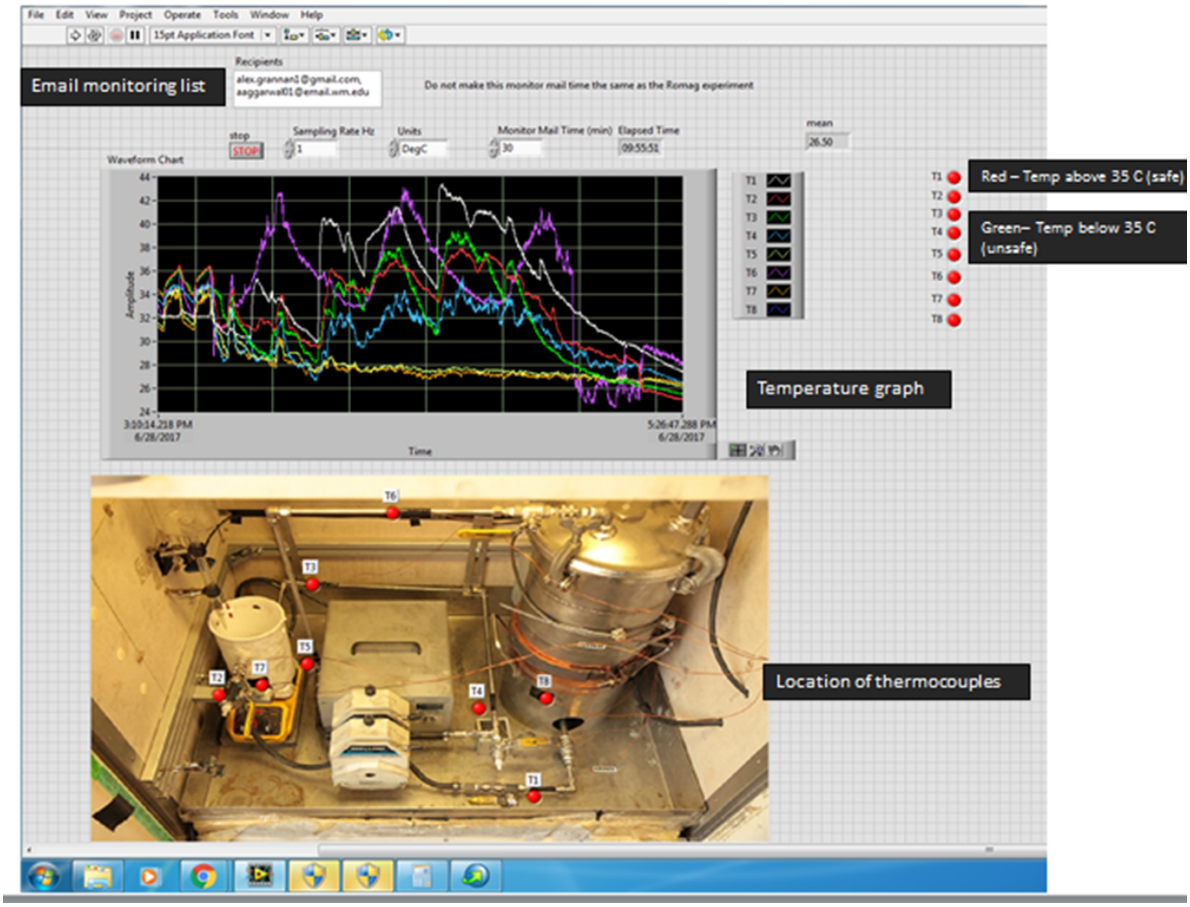

Figure 6: Temperature monitoring program in Labview. This should be turned on when the band and space heaters are turned on

*Sidenote: The band heater has a thermostat attached to the storage tank that activates at $41°C$. The space heater has a thermostat set at $80°C$. As the process of cleaning proceeds, the temperatures in the fume hood should reach steady state around $35-45°C$ before cleaning. The fan of the space heater is always on, however, the heater itself turns on and off based on the thermostat. The sash of the fume hood should remain closed for at least a day to maintain the internal temperature at about $30°C$, ensuring that the gallium melts for circulation.

4.2 Cleaning

1. Turn off the temperature monitoring program. Then, turn on the Labview pressure-based alarm system by navigating to a folder on the desktop labeled '*Ga_clean_pressure*', and selecting the file labeled *Ga_clean_pressure_alarm*. The front panel of the program is shown in Fig. 7

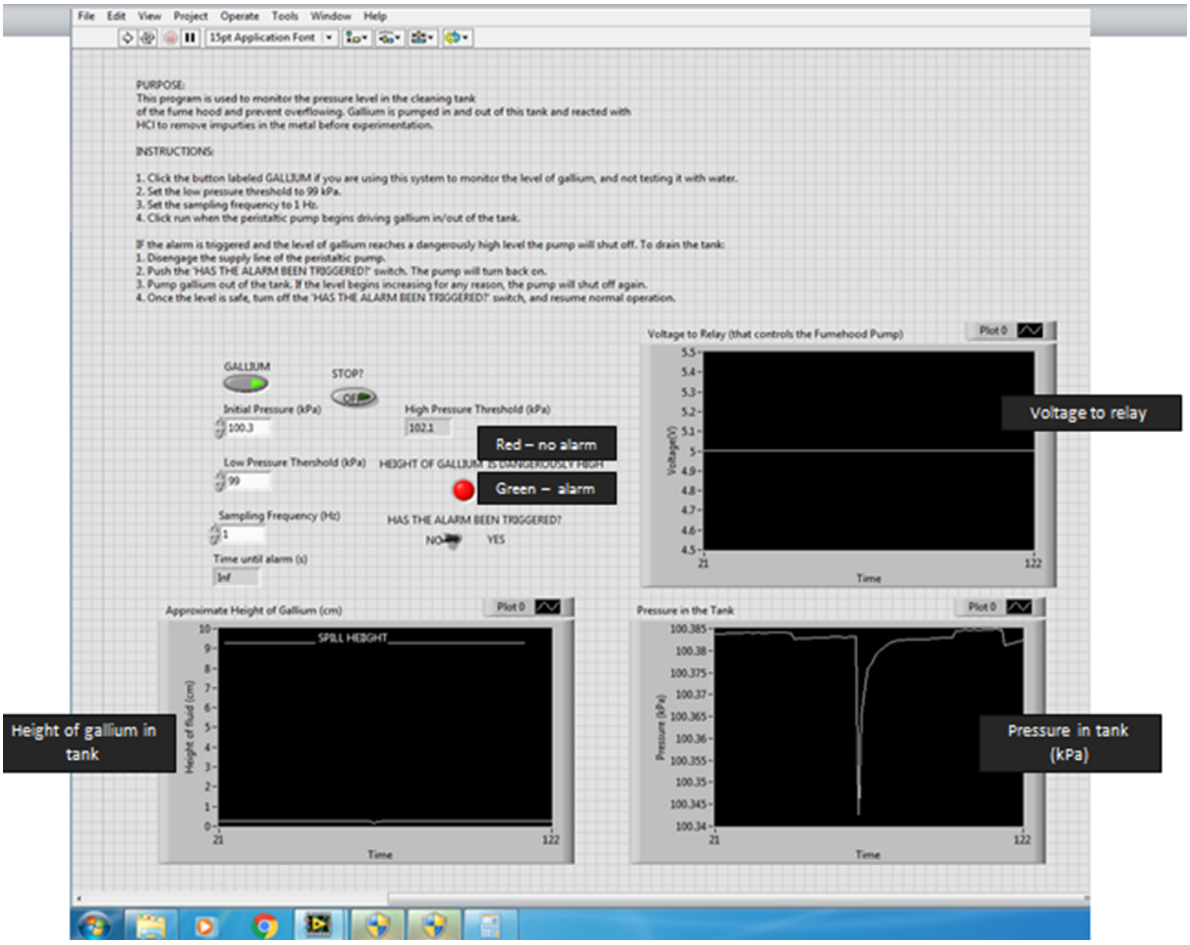

Figure 7: Pressure based alarm system for the cleaning tank. If the level of the gallium is too high, the voltage to the relay will be 0 and the pump will shut off. The red button will turn green to indicate an alarm and the speakers will begin to beep.

2. Plug in the Powerswitch Tail to a standard 120VAC outlet located near the fume hood

desktop which turns on the peristaltic pump. The LED display on the front of the pump should be lit.

3. Plug in the hot plate to a standard 120VAC outlet located on the left side of the fume hood. The LED display on the hot plate should be lit. Set the temperature of the hot plate to $45°C$ and the stirrer speed to 12.

4. The circulation of gallium begins by tightening and clamping the rubber supply line of the peristaltic pump. This activates the supply line of the pump.

5. Open Ball valve 1 and turn valve 3, labeled in Fig. 5 to a vertical position to direct the gallium into the cleaning tank.

6. Set the pump speed to 13 (see Fig. 4), and push the START button on the pump. Gallium will begin flowing into the cleaning tank. Check the Labview pressure program, and you should see the height level of the gallium slowly start to rise.

7. When the level of gallium in the cleaning tank is about 5cm, or the inlet pipe is submerged in gallium, the STOP button should be pushed on the pump. At this time, the user needs to prime the return line with gallium. To do this, release the quick connect at valve 7, and set up a separate tank in the corner of the fume hood. Generally, the final leg of the return line is vertical (solid line). To prime the return line with gallium, the leg should be placed horizontally (dashed line) into the extra tank. See Fig. 8 for an image of this process.

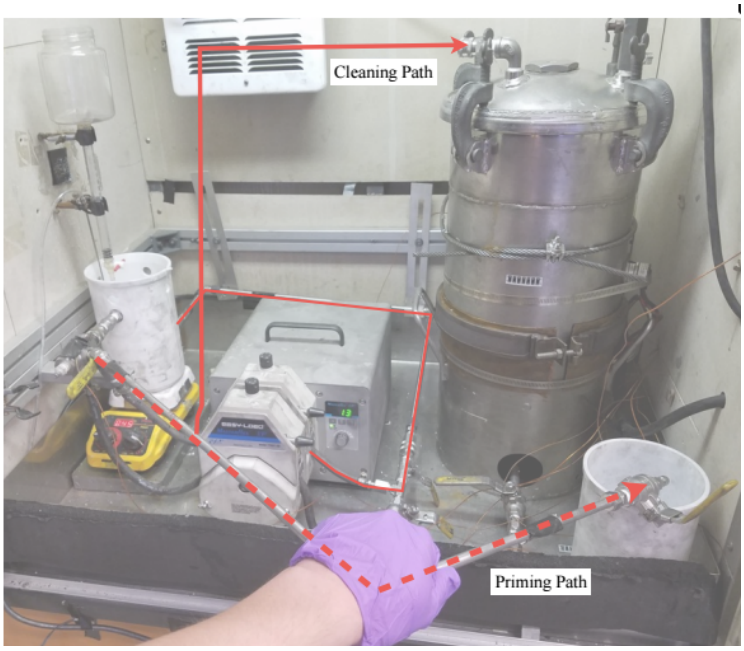

Figure 8: To prime the return line, the vertical return pipe should be disconnected and place horizontally to fill the line with gallium. Image and caption contributed by Alex Grannan

8. Clamp the rubber return line of the peristaltic pump. Open ball valves 4 and 5. Push the START button on the pump with valve 7 closed, allowing pressure to build up in the line. After several seconds, open valve 7. Gallium will flow into the extra tank for 5-10 seconds, verifying the flow is steady. Close valve 7 and stop the pump. Reconnect the pipe at the quick connect at valve 7, and re-open valve 7 to direct the return line into the storage tank.

9. Prepare the HCl-water mixture by locating the bottle of hydrochloric acid in the cabinet under the fume hood. Using a graduated cylinder, mix the HCl with water in a 1:10 ratio respectively, with about 10 mL of HCl. Place the solution into the holder suspended above the cleaning tank (see Fig. 2). Turn the drip valve slowly to allow a thin layer (0.5 cm) of the mixture to cover the gallium, then shut the drip valve off.

10. Recheck that ball valves 1, 3, 4, 5, 7, and 8 are open. Push START on the pump to

circulate the gallium.

11. Monitor the level of the gallium in the cleaning tank closely, both with the pressure-based alarm program and by eye. It is difficult to keep the level constant, but one can adjust the supply or return flow rates by turning the pressure adjustment knobs (see Fig. 4) the default values being 3. If level of gallium is rising steeply (AKA a sharp positive slope in the height graph), either decrease the supply flow rate or increase the return flow rate (see Fig. 9). If the level of gallium is falling sharply, vice versa.

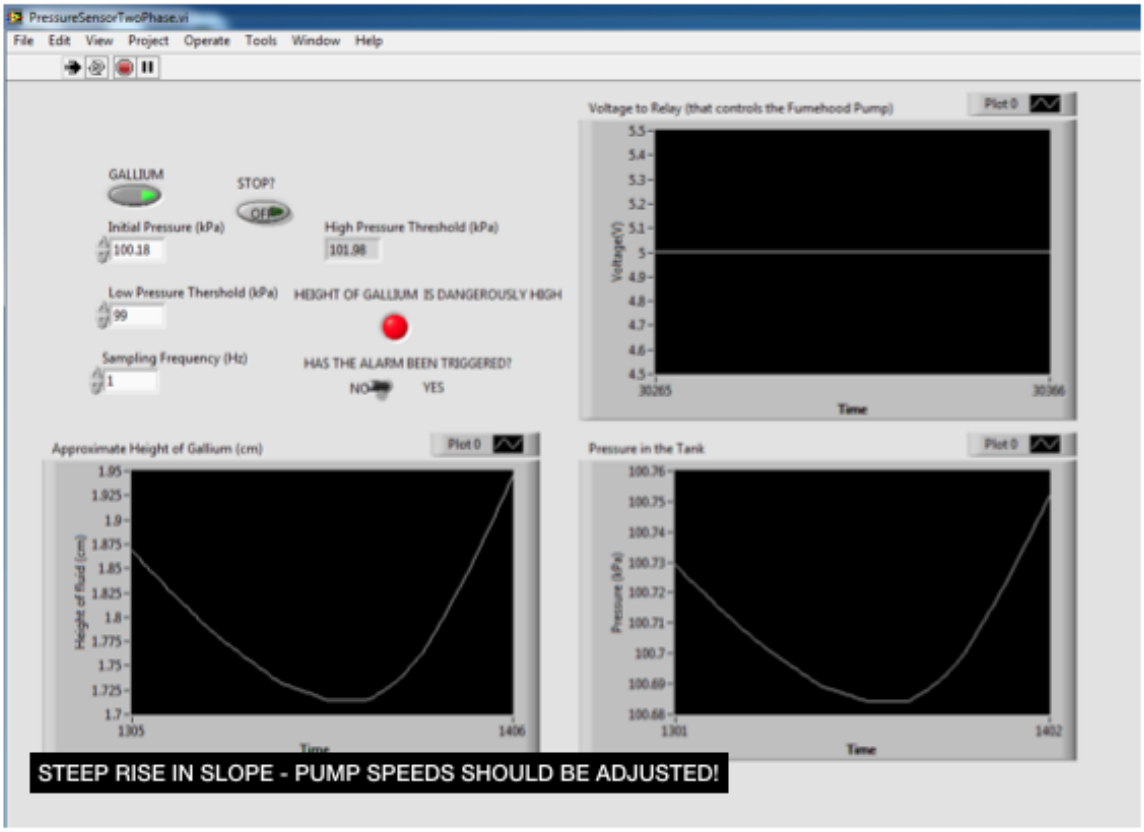

Figure 9: A sharp increase or decrease in the height indicates that the flow rates of the supply/return line need to be adjusted.

12. IF the level of gallium approaches the top of the cleaning tank (about 9cm), the pressure-based alarm system will trip the relay and shut the pump off (see Fig. 10). To drain

the cleaning tank to a safe level, first deactivate the rubber hosing for the supply line. After this is done, click the "HAS THE ALARM BEEN TRIGGERED" button to YES on the program. The pump will turn on and begin pumping, allowing the user to drain the cleaning tank to a safe level (the start button does not need to be pushed). If the height of the gallium increases (AKA the supply line is still activated for any reason), the pump will shut off again and the user should ensure that only the return line is clamped. Once the level is safe, the user should turn the switch back to no and continue normal operation.

*Note: Fig. 10 was taken by manually setting a low threshold level (about 6.5cm) to trip the alarm for the purposes of documentation.

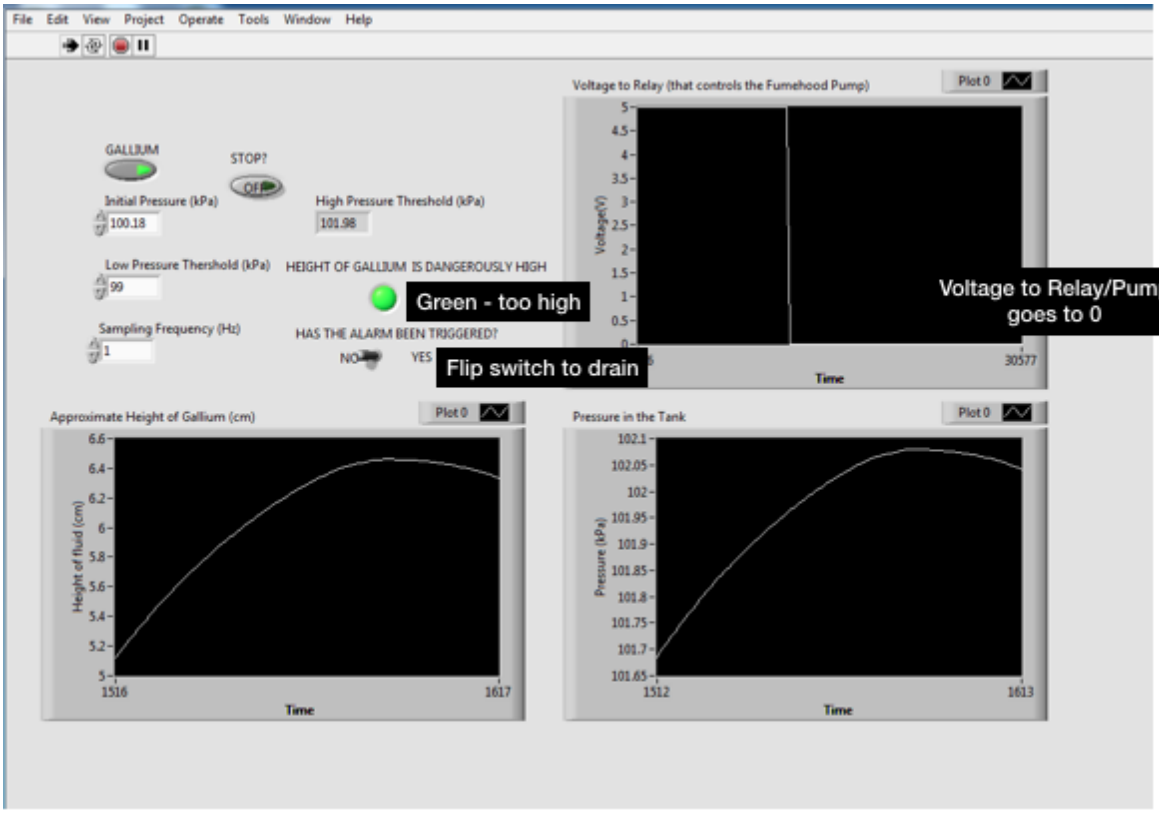

Figure 10: The voltage to the relay controlling the peristaltic pump will go to 0 if the level of gallium in the cleaning tank is around 9cm. To drain cleaning tank - disengage the supply pump and flip the switch under "HAS THE ALARM BEEN TRIGGERED?". The pump will turn on again and begin pumping only if the height of the gallium is decreasing

13. Generally, the user should check the gallium level in the tank every couple of minutes. Once the level is fairly constant, the user should still check every 10-15 minutes, with an ideal level being about 5-8cm (where the supply tube in the cleaning tank submerged and far from overflowing). A level of HCl should be maintained on the surface of the gallium. Circulation should be carried out in this manner for about 2 hours to clean the supply.

4.3 Flushing

1. Allow the HCl solution on the gallium to evaporate or dissolve before carrying out the next steps, since the mixture should be be poured into the storage tank. This should take approximately 2 hours if the layer of HCl on the gallium is thin.

2. Release the supply side pump, and close valve 1. Turn on the pump, and allow the cleaning tank to drain until the level is below the outlet.

3. Release the argon quick connect at valve 9 and place the line at the quick connect at valve 2. Use the valves on the argon gas tank to flush the gas through this line. Some gallium should be removed from the supply line into the cleaning tank.

4. Next, connect the argon gas line to the quick connect at valve 6. Close valve 4 and 5, and open valve 6. Open the return line pump clamp. Open the argon supply at valve 10, and flush argon through the return line going into the storage tank. You may hear the storage tank bubbling.

5. Finally, open valve 4 and 5 and flush the remaining parts of the line. The remainder of gallium in the cleaning tank might bubble.

6. Close all ball valves to trap argon in the pipes.

7. Unscrew the clamps to partially open the storage tank. Use the quick connects around the cleaning tank to disconnect it the cleaning tank. Pour the gallium into the storage tank with a funnel and tube.

8. Rinse the cleaning tank in a bucket with warm water, and reconnect it in its original location.

9. Finally, the storage tank needs to be flushed with argon. Connect the argon gas line to valve 9. Close valve 8 and use the knobs on the gas tank to release argon into the storage tank until the pressure in the tank is 5 PSI. Then, open valve 8 to release the argon. Repeat this 3-5 times. Close all valves.

At this point, the gallium is cleaned and storage. The band heater and space heater can be disconnected from the outlets and the fume hood should be allowed to cool. Turn off the Labview temperature and pressure monitoring systems.

References

[1] E. King (2009) An Investigation of Planetary Convection: The Role of Boundary Layers, P.h.D thesis, Dep. of Earth, Planetary, and Space Science, Univ. of California, Los Angeles, United States of America.

[2] A. Grannan (2017) Experimental and Numerical Studies of Mechanically and Convectively Driven Turbulence in Planetary Interiors, P.h.D thesis, Dep. of Earth, Planetary, and Space Science, Univ. of California, Los Angeles, United States of America.