



University of Pittsburgh

Nanoscale Fabrication & Characterization Facility

Hitachi H-9500 E-TEM Gas Delivery Users Guide



The Gas Delivery System for the Hitachi H-9500 E-TEM is designed to delivery up to 2 process gasses controlled in parallel and one evaporation liquid source. It has dry pump rough vacuum and built in nitrogen purge for fast process gas switching.

NOTE: Before using any of the gasses on the E-TEM, contact NCF staff to discuss and plan your experiment. Any required gas species changes and or system re-configuration requires at least 24 hour notice in order to purge the systems and make the changes that are required.

Operation Procedure:

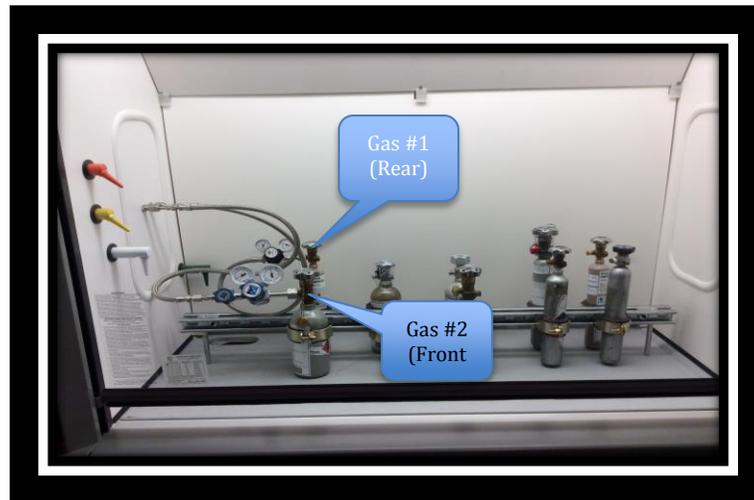
- 1) Once your sample is at the desired temperature proceed by verifying that all three blue delivery valves are closed.



- 2) Verify that the evacuation valve is open and the pressure is below 7×10^{-2} Pa. This is measured on the Varian gauge controller "AUX1".

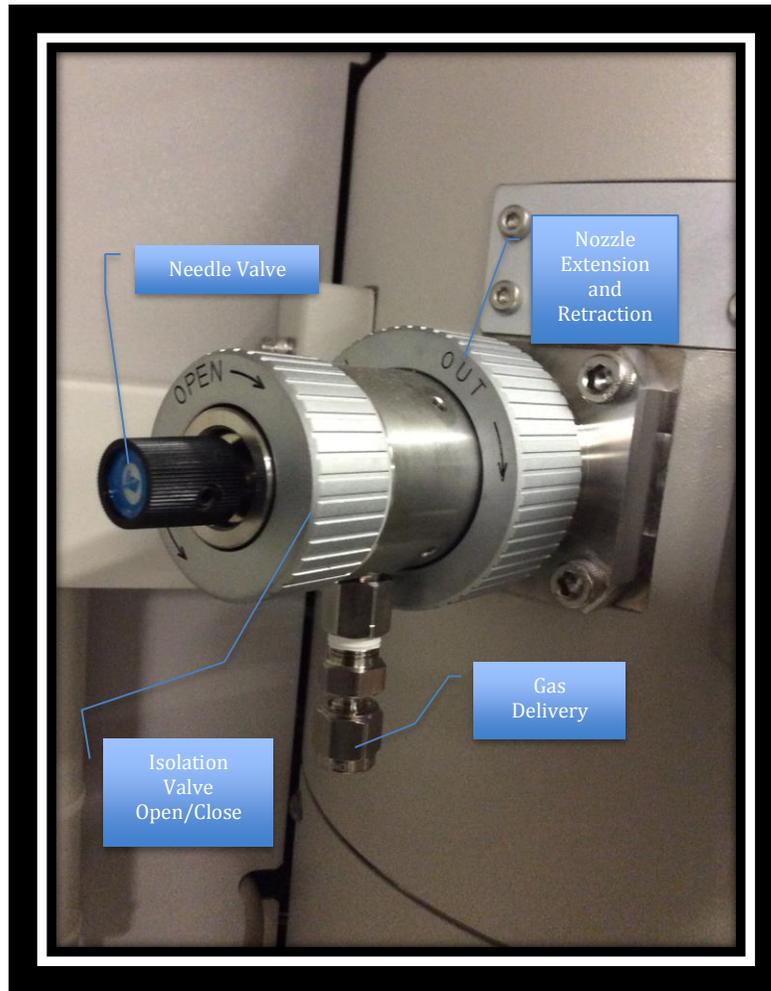


- 3) Open desired gas bottle or bottles in the fume hood. The two gasses with regulators on the left-hand side of the fume hood are the current gasses in use. The rear gas is considered gas#1 and the front gas is considered gas #2.



- 4) Make certain the pneumatic valve line is attached to the gas delivery line that you want. This connection is located to the right of the Alicat flow controller that you are using. If it is not on the correct valve, follow the procedure below:
- Turn off the valve at the regulator on the N2 purge bottle in the back right corner of the lab.
 - Press the pneumatic line in, hold the ring seal and pull the line out.
 - Press the line back into the valve you require.
 - Open the valve to the regulator for the N2 that you closed earlier.
- 5) Check and verify that the Alicat flow controller you are using is set at a set-point of 0 sccm.
- 6) Open the blue process delivery valve for the gas that you are going to use in your experiment.
- 7) Watch that the pressure at "AUX1" drops back below $7e-2$ Pa.
- 8) Close the vacuum valve.

- 9) Extend the gas delivery nozzle on the side of the H-9500 by turning it fully toward the "in" direction.
- 10) Open the isolation valve on the side of the H-9500 by turning it toward the "open" direction. Note: the needle valve on the H-9500 should be left open.



- 11) Wait for the pressure to stabilize in the specimen chamber.
- 12) Set the flow set point on the Alicat controller to the desired flow using the screen prompts. Remember to monitor the pressure and never let the specimen chamber pressure rise above $1e-1$ Pa.
- 13) Also monitor the turbo temperature and make certain it does not go above 55 degrees C.

- 14) Once processing is complete, set the Alicat controller to 0 sccm.
- 15) Allow the pressure to drop to 5 Pa in the specimen chamber.
- 16) Close the isolation valve on the side of the H-9500.
- 17) Retract the gas delivery nozzle on the side of the H-9500.
- 18) Open the green evacuation valve and verify that the pressure drops to near $5 \times 10^{-2} \text{ Pa}$ in the gas system.
- 19) Close the blue process delivery valve.
- 20) If the pressure is not dropping satisfactorily, cycle purge with the green N₂ purge valve. Close the vacuum, open the purge --- Close the purge and open the vacuum. Repeat 5-10 times and check the vacuum level again.
- 21) If you are finished processing, leave the valve to the vacuum open and close the process gas bottles that you used.
- 22) If you are switching process gasses, start over at step 1.

