

ESEM Checklist

- 1) Sign into the log book.
- 2) Mount your sample on a sample stub. (*wear gloves*)
- 3) Load your sample (*wear gloves*)
 - a. Ensure the electron beam is off (the Beam On button should be grey in color)
 - b. Press Vent. Wait for the chamber pressure icon to turn grey in the main chamber. Once it does, *gently* try to open the door. It will take about 1 minute to vent the chamber, and it should not require force to open the door.
 - c. If you will need the Peltier stage:
 - i. Remove the normal sample stage and replace it with the Peltier stage
 - ii. Connect the Peltier stage cable
 - iii. Remove the water caps and connect the water lines
 - iv. Turn on the chiller power and ensure that the “Refrig” light is lit.
 - v. Turn on the flow box and press and hold the green “Start Flow” button until the lines are full of water. **Make sure there is no water leaking into the chamber.**
 - d. Decide which Vacuum Mode you will operate in and choose a detector from the table on the next page. Insert it if necessary.
 - e. Insert the low kV pressure limiting aperture (PLA) or the X-ray PLA if necessary.



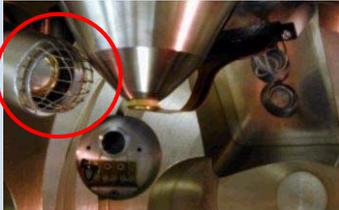
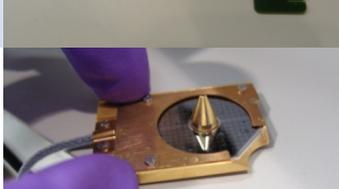
Low kV PLA



X-ray PLA

- f. Place your sample into the sample holder and gently tighten the set screw. Do not over tighten. Use the height gauge to make sure your sample is below the 10 mm mark.
 - g. When you insert a detector, the instrument will usually select a vacuum mode in the software for you. Make sure this selection is the mode you want to operate in. (HiVac: conductive samples; LoVac: nonconductive, mixed or contaminating samples; ESEM: wet samples)
 - h. If you are using LoVac or ESEM mode check the purge pressures (Beam → Preferences → ESEM menu) and set the final pressure.
 - i. *Gently* close the door, press Pump, and wait for the chamber pressure icon to turn green in the main chamber.
- 4) Make sure that the CCD camera window is active. Using the joystick raise the height of your sample to the 10 mm mark. **Do NOT move the 10 mm mark on the screen!**
 - 5) If you are using the Peltier stage, you can set the temperature or the humidity at this point. **Do not rotate the Peltier stage because of the cable and hoses that attach it to the chamber wall.**
 - 6) Turn on the electron beam by pressing Beam On under the Beam Control tab (the button should turn yellow when on).



Detector Name	Tag	Vacuum Mode ^a	Detected Signal	Maximum Pressure (Pa)	Picture
Everhart-Thornley	ETD	HiVac	SE or BSE (depending on detector voltage)	$3 \cdot 10^{-2}$ Pa ($2 \cdot 10^{-4}$ torr)	
Backscattered Electron	BSED	HiVac , LoVac	BSE	200 Pa (1.5 torr)	
Large Field	LFD	LoVac , ESEM	SE + BSE	200 Pa (1.5 torr)	
Gaseous Secondary Electron	GSED	LoVac, ESEM	SE	<ul style="list-style-type: none"> 100 μm aperture: 750 Pa (5.7 torr) 500 μm aperture: 2600 Pa (19.5 torr) 	
Gaseous Backscattered Electron	GBSD	LoVac, ESEM	SE, BSE	10 Pa (500 optimal) – 2600 Pa (optimal 3.75 – 19.5 torr)	
Gaseous Analytical	GAD	HiVac, LoVac, ESEM	BSE	2700 Pa (20.3 torr)	

^a Bold text indicates the preferred mode.

- 7) Focus your image. Do NOT adjust the height of the sample during this process.
- 8) Link your sample height (Z) to the working distance by pressing the Z-FWD button. After you have linked to an in-focus image, it is safe to change the height of the sample. 
- 9) In the Beam Control tab, press the Lens Align button, and use the Lens Align adjustment window to eliminate any movement in your E-beam image. Turn off the Lens Align when finished.
- 10) Check the beam position by pressing the Crossover button, and then use the Source Tilt adjustment to center the beam on the green “x”. Turn the Crossover off when finished.

- 11) Focus and stigmatize your image.
- 12) Adjust the contrast and brightness, and the scan parameters for image acquisition.
- 13) Take a photo (F2) or a snapshot  and save your data in “support-d8680/Shareddata on Support-d8680/Faculty Advisor Name/Your Name/”.
- 14) If you want to change vacuum mode:
 - a. Stop scanning and turn the electron beam off
 - b. Make sure you have a detector inserted that will image in the vacuum mode you want to switch to. If you don't, then you will need to follow the vent procedure below, install the correct detector and repump the system
 - c. Click on the new vacuum mode you want to use and wait for the vacuum mode to stabilize and for the chamber icon to turn green
 - d. Turn the electron beam back on, and continue imaging.
- 15) Shut down:
 - a. Turn the electron beam off by clicking on the yellow Beam On button in the Beam Control tab.
 - b. To remove your sample, press Vent. Wait for the chamber pressure icon to turn grey in the main chamber. Once it does, *gently* try to open the door. It will take about one minute to vent the chamber, and it should not require force to open the door.
 - c. Loosen the set screw, and remove your sample.
 - d. If you used the Peltier stage:
 - a) Stop the flow of water by pressing the red “Stop flow” button and turn the flow box off.
 - b) Disconnect the Outlet connection on the chiller.
 - c) Press and hold the white ring to enable flow. **Have a bucket ready to catch the excess water from the pump.**
 - d) Turn on the flow box and press and hold the green “Start flow” button and watch for bubbles in the water lines to indicate that the lines are empty. This may take a few minutes.
 - e) Release the “Start flow” button and turn the power off on the flow box.
 - f) Turn off the chiller.
 - g) Take the Peltier stage out of the system and replace it with the normal stage.
 - e. Set the system to HiVac mode.
 - f. *Gently* close the door, press Pump, and wait for the chamber pressure icon to turn green in the main chamber.
 - g. Leave the software running.
 - h. Sign out of the log book.

Shut down/Restart Procedure

- 1) Instrument Shut Down:
 - a. Turn off the electron beam.
 - b. Close the software (User Interface – UI)
 - c. Stop the server and wait for all of the lights at the bottom of the window to turn grey.
Exit from the server.
 - d. Restart the computer.
- 2) Instrument Start Up:
 - a. Log onto the microscope PC as “supervisor” with a password of “QuantaD8680”.
 - b. Open the FEI software. Once the four lights in the upper right hand corner have turned green, press the start button and wait for all of the lights in the bottom of the window to turn green.
 - c. The user interface should automatically restart.
 - d. Log on as “supervisor” with a password of “QuantaD8680”.
 - e. Run the Home Stage procedure if requested. **Do not do this if you have the Peltier stage inserted since it may rotate the stage!**

Common Instrument Settings

Parameter	E-Beam Setting
Vacuum Mode	HiVac: conductive samples LoVac: nonconductive, mixed or contaminating samples ESEM: wet samples (use H ₂ O gas medium, and cold stage if desired 100% relative humidity)
High Voltage	Select voltage relative to specimen type: <ul style="list-style-type: none">- Low voltage for surface imaging, beam-sensitive samples and slightly charging samples- High voltage for conductors, high resolution, compound info (BSE, x-ray)- Biological sample High Voltage = 1-10 kV- Metal sample High Voltage = 10-30 kV
Spot Size	HiVac and LoVac: 3 or 4 ESEM: 4
Pressure	HiVac: the lowest LoVac: 60 Pa (0.5 torr) ESEM: 600 Pa (3.7 torr)
Scan rate	HiVac: fast (dwell time about 0.1 – 0.3 μs) LoVac and ESEM: slow (Dwell time about 1 – 3 μs)
Working distance	Set the highest specimen point to approximately 10 mm (yellow mark in the optical quad)
Standard Detector	HiVac: ETD (SE) LoVac: LFD ESEM: GSED