

Startup:

1. Turn computer on (or wake it up). Make sure both monitors are on.
2. Check the instrument log (Desktop > SEM user log.txt) and note the date of the most recent use of the SEM. *If the instrument was **not** used the previous day, you will need to warm up the turbo molecular pump (TMP). When in doubt (i.e., if you are not sure whether or not the instrument was used the previous day), run the sequence.* This warmup will take about 30 minutes. See step 6 below for details.
3. Turn power switch to on.
4. Press green "exchange" button to vent chamber.
5. Start TM-1000 software.
6. To warm up the TMP: in the TM-1000 software, select Maintenance > TMP running-in mode. If the instrument was used the previous day (see step 2 above), skip the warmup and move on to step 7.
7. Prepare your stub. You may wear gloves, but it is not necessary. Place a carbon tape dot on the stub and press or sprinkle your sample onto it. If your main interest is EDS, use a 22° angled stub, which directs more x-rays toward the EDS detector. Several other sample holders are also available: a plug holder for samples embedded in epoxy, a holder with set screws to hold odd-shaped samples at any angle, large stubs, graphite stubs (when analysis of Al content is of interest), adapters for holding one or four standard pin-mount stubs, etc. All stubs and supplies are in a labeled drawer opposite the instrument.
8. Screw your prepared sample stub on the sample holder, and place the sample holder into the hole in the guide (metal block with swinging arm). Adjust the height of your stub by loosening the nut at the base of the threaded rod, then screwing the threaded rod into or out of the base of the holder. When the desired height is attained (see step 9), retighten the nut to prevent vibration and poor grounding from interfering with your imaging.
9. Use the high-purity compressed air to blow any loose particles off of your stub.
10. Note: be sure that there is a minimum of 1mm between the swinging arm of the guide and the highest point of your sample. Nonconductive samples or those for which you desire a wide field of view at low magnification may be placed lower. For the 22° angle stubs, set the height of the stub so that the distance between the arm and the center of the sample (i.e., the working distance) is about 7mm.
11. Open the stage door and insert the sample holder into the hole on the stage. Make a note or sketch of the orientation of material on your stub.
12. Close the stage door and press the green **Exchange** button.
13. Listen to the roughing pump. If it doesn't quiet down within about 10 seconds, you have a small leak through the stage door. Press on the door to make a seal until you hear the noise of the pump change.

14. Turn on stage controller (rocker switch on back of controller) and EDS control box (toggle switch on left side of box near the wall).
15. Wait for green **Ready** light on instrument.
16. Wait an additional minute after **Ready** light appears. Press **Start** button in software to turn on electron beam.

General operation and imaging:

- Center your sample in the sample chamber by moving the “+” sign on the stage controller display to the center using the joystick.
- Check beam mode and vacuum level, and change if necessary.
 - ✓ Under Settings > Beam Emission, make sure the instrument is set to Mode 1. This setting prolongs filament life.
 - ✓ Under Settings > Observation Mode, start with the instrument in Standard Mode. This setting prolongs filament life, and provides better image resolution.
 - ✓ If you are working with non-conductive samples, or find that your samples are charging up (indicated by abrupt shifting of the image), change to Settings > Observation Mode > Charge-up Reduction Mode. If you see an “L” near the micron bar in the software, you are in charge-up reduction mode.
- **Tilt** and **Rotate** switches on stage controller are not active.
- Focus by holding down the mouse button and dragging to the right or left on the image. Autofocus is slow and doesn’t always work well.
- Sample name and image number, along with the scale bar and image information such as magnification, date and time, etc., are automatically included in images. To input your sample name and reset the auto-increment, go to File > Setting for save. Please do not change the “comment” field from its standard setting of “LacCore.”
- The cross line will be saved permanently in image if it is displayed while saving image. To turn cross line on or off, go to Setting > Cross line.
- For best focus on your image, zoom in to (much) higher than the desired magnification and focus. Then zoom out to the desired magnification. Press the “Auto B/C” button (auto brightness and contrast). Then press “Save” (not “quick save”).
- The displayed image will now be frozen. To return to live image mode, press Fast.

EDS elemental analysis operation:

- Open Quantax 50 software (on desktop).

- Choose Settings > Mode select > Analysis mode. All this actually does is change the buttons along the top of the main window. Choose "Area" to analyze a wider region (shown in reduced view) or "Spot" to focus on a very narrow region (nominally 0.2 μm , but note that the electron beam penetrates into the sample and so it actually interacts with a *volume* of sample; also, due to the relatively low vacuum, x-ray scattering due to collisions with air molecules means that you will always see some aluminum and carbon [stub and tape, respectively] in your analysis).
- Press "play" button (green arrow) to begin analysis. Counts per second are displayed at the bottom of the window; for best statistical results you need >1000 counts per second (1.0 kcps). For most materials, counts can be increased by using 22° angled stubs, or by increasing the amount of material on the stub. Even low counts can give surprisingly good compositional information. The program can be set to count for different lengths of time automatically, or as started and stopped by the user. 30 seconds is standard; the time countdown is near the cps display.
- Peaks are labeled automatically after some confidence threshold is reached. After the time expires, the program displays quantitative elemental data in several formats. You can click on the periodic table to see peak locations for a given element (e.g., in case its peaks are too small to be labeled); you can also identify unknown peaks by switching to the "Finder" tab above the periodic table and dragging the vertical line on the spectrum window to the peak.
- Several options for saving and exporting EDS results and spectra are available: click on the button with two arrows pointing opposite directions (upper right corner of Quantax window).

Sample exchange:

1. Turn off the electron beam (**Stop** button in software)
2. Allow the filament to cool for at least one minute
3. Vent the chamber by pressing the **Exchange** button and waiting for solid **Air** light

Shutdown:

1. Vent chamber and remove sample holder.
2. Remove your sample from the sample holder, peel all carbon tape off the stubs you were using and return them to the drawer (or fill out supplies form if you are keeping them), and place the sample holder in the guide block.
3. Close and evacuate chamber by pressing the **Exchange** button; wait for steady green **Ready** light.

4. Turn instrument power switch to off (roughing pump will stop but you will still hear TMP). Turn off stage control box. Turn off EDS control box.
5. Fill out or update log of supplies used and place on Amy's desk.
6. Make sure door to 672 is locked on your way out.