TESCAN VEGA3
SCANNING ELECTRON MICROSCOPE
OPERATOR'S MANUAL

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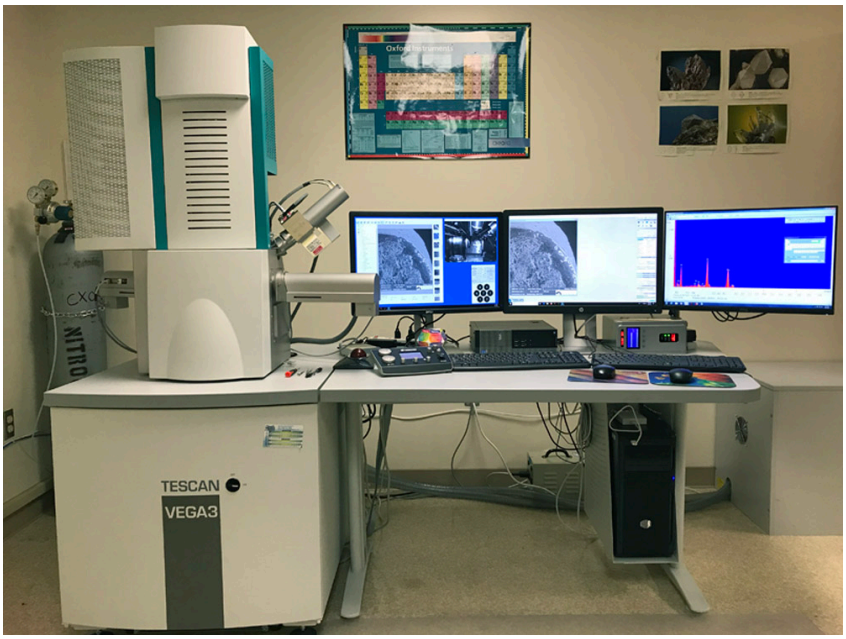

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High Vacuum Operation

GETTING STARTED

Note: for the following discussion, **BOLD CAPS** indicate the appropriate button or action to select; SMALL CAPS indicate the control panels or software windows. See Figure 1 for explanation of operating system window with the various panels described in this manual.

OPERATION IN HIGH VACUUM MODE

I. PRELIMINARY PROCEDURES

A. Handle all sample holders with gloves.
   1. Most holders are kept in the desiccator on the counter. Odd-shaped sample holders are in the Tescan accessary box in the second drawer.

B. Mount samples in the appropriate holder. All mounts use a 3 mm pin to secure the holder in the instrument.
   1. 12 mm Pin-stub mounts (mounted directly on the stage):
      a. Use carbon paint, carbon tape, hot glue, or Super glue to secure the sample to a sample stub.
      b. Make sure that there is a conductive path to ground from the sample surface (use carbon/copper tape or carbon paint).
   2. Rectangular thin sections:
      a. Secure 1 or 2 sections in "Geo-slides" holder with metal hold-down tabs.
      b. More sections (up to 6) may be arranged on the 10 cm aluminum disk and held with double-sided Scotch tape. Use copper tape to ground the top surface of the section to the aluminum.
   3. Polished epoxy mounts and 1" round thin sections:
      a. A single 1" thin section or polished round may be secured in the gold 1" round holder. That holder is then mounted in the single round pin-mounted holder.
      b. Multiple rounds may be placed in the 4-section holder.
   4. Several other holders are available for odd-shaped samples.

II. VENT INSTRUMENT AND LOAD SAMPLES

A. If necessary, log into Tescan computer on the left monitor.
   1. Login as "**SEMUser**" with the password "**Tescan**".

B. Start Tescan software from the  icon on the left-hand monitor.
   1. Select account name and enter password.
   2. Wait for system to go through self checks.

C. Vent the chamber
   1. Click on the **VENT** button in the VACUUM window.
      a. The "Column Pressure" bar in the VACUUM window will drop to zero and the sample icons on the STAGE CONTROL window will turn red as the chamber is backfilled.
      b. Wait until the chamber backfills (a crack may appear at the edge of the white door and/or you can hear the hissing of the N_2 gas into the chamber.)
      c. Swing the front door out and to the left.

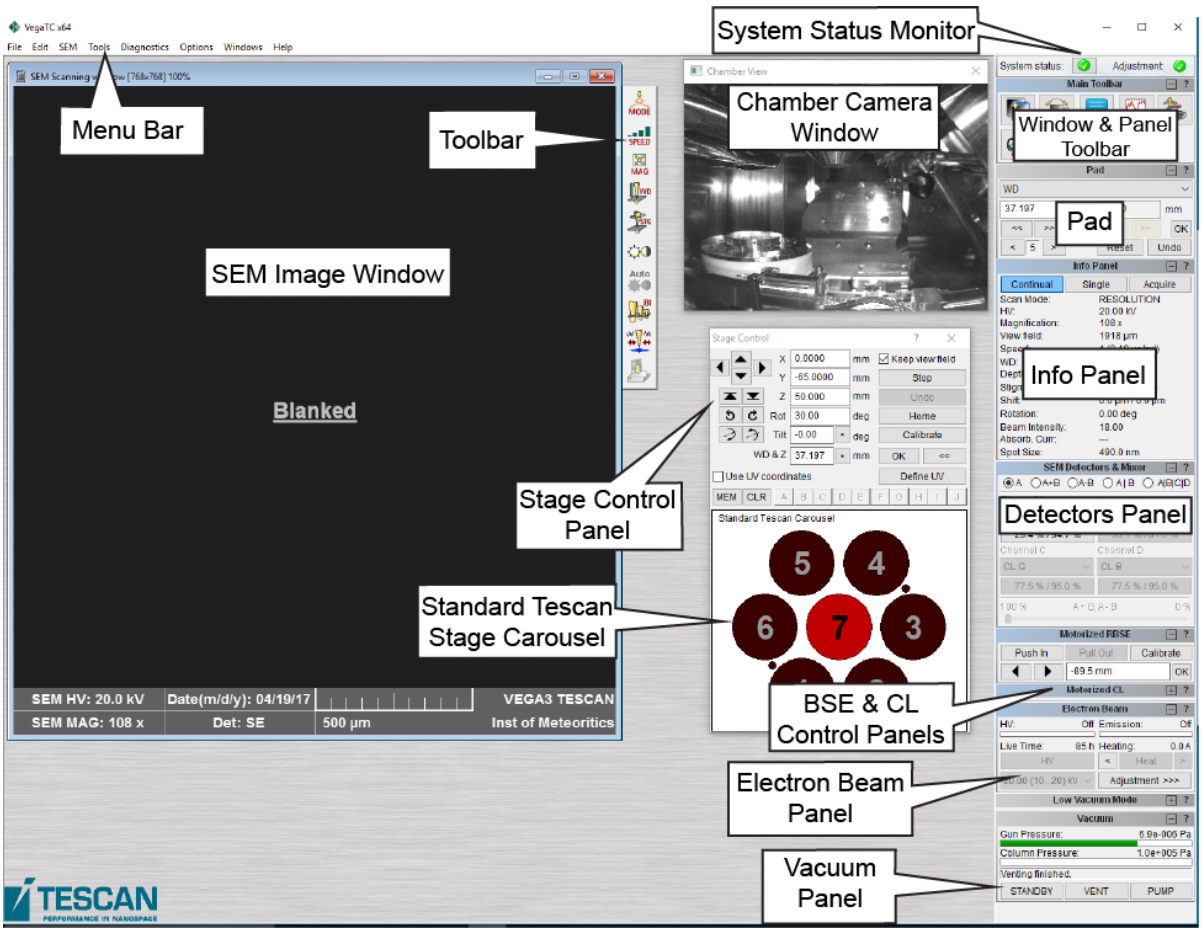


Figure 1. Tescan operation window.

2. Place the sample into the pin mount hole.
   a. To mount pin samples on the stage, click on the appropriate sample number in the STANDARD TESCAN CAROUSEL to rotate each toward the door. See Figure 2.
   b. To mount other holders, click on sample # 7 to rotate the set screw toward the door.
3. Secure the pin holder to the stage by tightening the set screw.
4. Gently swing the door closed.
5. Click on the **PUMP** button in the VACUUM window (Fig. 3).

III. SETUP INSTRUMENT

Proceed with setup while instrument is pumping down.

A. Move stage to center position.
   1. Enter "0" in the **Y** box on the STAGE CONTROL window and click **OK**,

B. Set accelerating voltage.
   1. In the INFO PANEL, check the HV setting. Some recommended values:
      a. Use 15-20 kV for most applications, including EDS analysis.
      b. For highest resolution, select 25-30 kV.
      c. For CL work, select 10-15 kV.
      d. For uncoated samples at low voltage, select 5 kV.

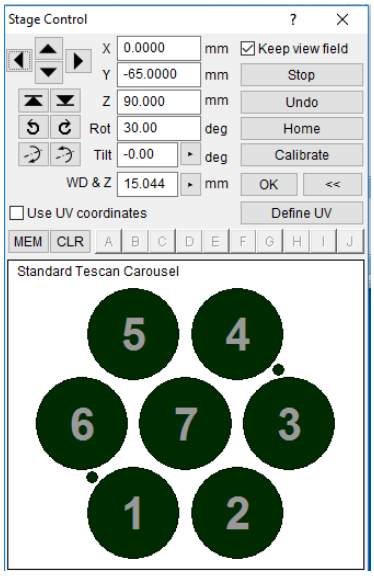


Figure 2. Stage control window.

2. To change accelerating voltage, in the ELECTRON BEAM panel, select the desired voltage range in the box under the HV button. See Fig. 4 below.
   a. Click on the ▼ and select the range.
   b. For a fractional value, then enter it in the HV colum in the INFO PANEL (e.g. for 15 kV, select the third range (10-20 kV), then enter 15 in the INFO PANEL).

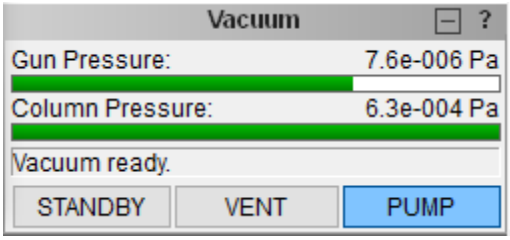


Figure 3. Vacuum control panel. Gun and column are in an operational state.

C. Start and align the gun.
   1. In the SEM DETECTORS & MIXER panel, make sure the secondary detector (SE**)** is activated. If not, click ▼ and select **SE**.
   2. In the Vacuum panel, both "Gun Pressure" and "Column Pressure" will turn green when chamber vacuum has been achieved.
   3. In the ELECTRON BEAM panel click on **HV** button. The gun will automatically heat over a period of about 1 minute.
   4. HV button will turn blue when the gun is ready and the beam is on.
   5. Perform an automatic gun alignment procedure:
      a. Move to any place on the aluminum sample holder and zoom to high magnification. Be sure there are no features (e.g. particles) in view.
      b. Click on the **Adjustment>>>** button in the ELECTRON BEAM panel.
      c. Select **Auto Gun Centering**.

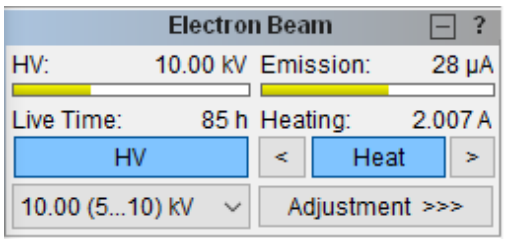


Figure 4. Electron beam panel with the gun operating.

D. Do an initial image focus.
   1. Right click on SEM IMAGE WINDOW and select "Minimum Magnification."
   2. Click on **Auto** contrast and brightness in the IMAGING TOOLBAR.

   3. Set scan speed by clicking on **Speed** in the IMAGING TOOLBAR. Use a relatively fast scan speed like 2 or 3. Hint: you can also roll the mouse thumb wheel to adjust speed.

   3. Use the WD knob on the desktop CONTROL PANEL to focus image. Hint: double click on image window to get a reduced scan area. Double click outside of box to return to full screen mode.
   4. Consider the desired stage height.
      a. For low magnifications or large samples, use a long working distance (25-50 mm). Use of Wide Field Mode will then allow full view of the sample holder at very low magnification
      b. For EDS analysis, the optimum distance is 15-17 mm.
      c. BSE will work at most WD but do not get closer than 8.5 mm.
      d. The CL detector cannot be inserted at WD less than 15 mm.
      e. For highest magnification in SE, a short working distance of 5 mm is optimum.
   5. If you have loaded samples other than pin mounts, you may need to use Wide Field Mode to find the position of your sample.
      a. Left click on **Mode** in the TOOLBAR at the right of the SEM IMAGE WINDOW and select **Wide Field**.

      b. Click on mouse thumb wheel and drag in the SEM image to move

      or

      c. Use Joystick to move the sample under the beam. Push the joystick in the direction that you want the sample to move.

6. Set the desired height.
   a. After initial focus, the WD & Z box in the STAGE CONTROL window displays the correct WD.
   b. To change working distance without defocusing the image, enter a value in the **WD & Z** box. A standard height such as 25 or 15 mm can be selected by clicking ▼.

E. Optimize and focus the image.
1. Consider the desired beam intensity.
   a. Use low beam intensity for high magnification and higher resolution (e.g. around 12 or below).
   b. Use higher beam intensity for EDS or backscattered imaging (12 or above).
2. Adjust beam intensity with the **BI** knob on the CONTROL PANEL or adjust from the BI button on the TOOLBOX.

3. Move to a visible feature on your sample. Adjust contrast & brightness if necessary.
4. Double click in the SEM IMAGE WINDOW to get a reduced area focus window.
   a. Move window with the left mouse button. Make it larger by dragging with the right mouse.
   b. Set scan speed slightly slower.
5. Increase the magnification significantly by turning the **MAG(nification)** knob on the Tescan CONTROL PANEL.
6. Focus carefully with the WD knob.
7. If image moves during focusing, you will need to check the Objective Centering.
   a. Click on the **Wobble** button in the imaging TOOLBAR.

   b. Click **WOB** and then **NEXT**.
   c. Use either the small knobs on the center of the CONTROL PANEL or the Turboball to adjust X & Y alignment until image remains centered while wobbling in and out of focus.
   d. Click **FINISH**.
8. Complete image optimization by checking stigmation.
   a. Click on the **Stig** button in the imaging TOOLBOX.

   b. In the PAD panel above the Info Panel, click **Reset** to set the stigmators to 0.
   c. If necessary, use the small knobs on the center of the desktop CONTROL PANEL sharpen the image as much as possible.

IV. START WORK ON YOUR SAMPLE

A. Saving an image.

1. Select the appropriate detector(s) from the SEM DETECTORS & MIXER panel.
2. Adjust the contrast & brightness either manually from the Tescan CONTROL PANEL or by clicking on the **Auto** contrast and brightness in the TOOLBAR.
3. Compose the image and note the magnification.
4. Focus the image at a higher magnification and then return to the desired magnification.
5 Select an appropriate scan speed by clicking the **Speed** button in the TOOLBAR. Scan speed should be slow enough to reduce or eliminate salt & pepper noise in the image. Speed 5 or 6 work well although 7 may be necessary for low current/low voltage applications.
6. If desired, set the image resolution and scan speed (you can set saved image resolution and scan speed be set to different from than the live scan image).
   a. Pull down the **SEM** menu from the MENU BAR.
   b. Select IMAGE PARAMETERS
      1) To set a constant scan speed, click on ▼ in the top **Acquisition** box and select an option such as "Set scan speed 6."
      2) To set a larger/smaller saved image resolution, click on ▼next to **Save** in the **Windows** box.
   c. Click on **Apply**, and then close the window.
7. Click on the photo button on the TOOLBAR to start the picture.

8. Enter info in the Header Save window. This is metadata, it's not required to enter anything. Click **OK**.
9. Select or create a folder, enter file name, and select file type. Click **Save**.

B. BSE imaging.

1. The BSE detector must be inserted under the polepiece before BSE imaging can be used.
   a. Expand the MOTORIZED RBSE panel if necessary.
   b. Click on **Push In** to insert the detector.
2. From the SEM DETECTORS & MIXER panel, click on ▼ in the "Channel A" box and select **LE-BSE**.
3. Click on **Auto** contrast and brightness in the TOOLBAR.
4. BSE imaging will generally require a slower scanning for imaging, so reduce scan speed (5 or 6).
5. When finished, click on **Pull Out** in the MOTORIZED RBSE panel.
6. Select **SE** from the SEM DETECTORS & MIXER panel.

C. Cathodoluminescence (CL) imaging

1. CL imaging generally works best with low kV (e.g. 10 kV) and high current (BI=12 or higher).
2. The CL detector must be inserted under the polepiece.
   a. Expand the MOTORIZED CL. Note: the system will not allow both the CL and BSE detectors to be pushed in at the same time. The RBSE must be pulled out.
   b. Click on **PUSH IN** to insert the detector.
2. From the SEM DETECTORS & MIXER panel, click on ▼ in the "Channel A" box and select **CL Color** or **CL P** (panchromatic).
3. To use **Auto** contrast and brightness, select CL P first.
4. Select a slow scan speed (6 or 7 or slower).
5. To view all 4 channels (Color+RGB or panchromatic +RGB), from the SEM DETECTORS & MIXER panel, click the radio button **A|B|C|D** and select the desired channels and order.
6. When finished, click on **Pull Out** in the MOTORIZED CL panel.
7. Select **SE** from the SEM DETECTORS & MIXER panel.

V. INSTRUMENT SHUTDOWN

The chamber can be vented immediately after turning off the gun.

A. Turn gun off and vent the chamber.
   1. In the ELECTRON BEAM panel, click on **HV** button.
   2. Click on the **VENT** button in the VACUUM window.

B. Return stage to exchange position.
   1. Wait until the icons in the STANDARD TESCAN CAROUSEL turn red.
   2. If you have loaded pin-mounted samples, click on the first sample (**1)** to move it and rotate it toward the door. For large holders (e.g. thin section holder), click on position **7**.

C. Remove samples.
   1. Wait until the chamber backfills (a crack may appear at the edge of the white door and/or you can hear the hissing of the N_2 gas into the chamber.)
      a. Swing the front door out and to the left.
      b. Loosen set screw for pin mount and remove sample (WEAR GLOVES).
      c. If necessary, click on the next sample number to rotate it to the door.
   2. Gently swing the door closed.
   3. Click on the **PUMP** button in the VACUUM window.

D. Secure the instrument.
   1. Close the software.
      a. Click on the **Exit only** button.

E. Fill out logbook

F. Clean up around instrument and sample prep counter.

TESCAN SOFTWARE

I. PANORAMA (STITCHED) IMAGE

Tescan's Image Snapper software will collect and stitch together a number of BSE maps into a single image. For X-ray maps, see Map Acquisition in the IXRF section.

A. Setup image area.
   1. For large map, select 2 diagonal corners.
      a. Move to and focus at upper left corner.
      b. Record position in STAGE CONTROL
         1) Click **MEM**, then button "**A**"
         2) Move to lower left and click **MEM**, then button "**B**"
   2. For small map, i.e. smaller than a view field, set magnification low enough to include area to be mapped.

B. Set up image stitching software
   1. Select TOOLS menu and **Image Snapper**
      a. A new window will appear. Click **Next**.
      b. In the EDIT SAMPLES window, select the **Rectangle** icon.
         1) In STAGE CONTROL, click button **A** to move to starting point.
         2) Click in **SEM Scan Window** to set starting point.
            a) For maps smaller than the view area, simply draw a rectangle in the **Scan Window**.
         3) Click on button **B** to move to end point.
         4) Click in **SEM Scan Window** to set ending point. A shaded rectangle should appear in the SEM Scan Window.
      b. In the EDIT SAMPLES window, enter a **Name** for stitched map in the first column.
      c. Click **NEXT**
   2. Set image conditions to desired values.
      a. MAGNIFICATION to desired value (higher magnification means more images).
      b. Set SCAN SPEED to get good clarity (BSE, usually speed 4 or 5).
   3. Click PROPERTIES button.
      a. Click top **READ** button to read field view size.
      b. Set "Overlap" between 10-20% (larger overlap is easier to match but takes more images).
      c. "Field" gives number of images that will be collected. Can adjust with magnification if too many.
      d. Enter **Image name** for images. All images will be saved with this name (e.g."name1-1", "name1-2", etc.).
      e. Click bottom **READ** button to read Z & WD from stage. (if sample is tilted, use image Field Mode for better depth of focus).
      f. Click **OK**.
   4. For multiple maps, add another map in the EDIT SAMPLES window and repeat.
   5. Click **NEXT** button.
   6. In the OPTIONS window:

a. Set "Autosignal" to **off** (or else set to "Before each sample"--don't use before each image).
b. Set "Autofocus" to **off**

7 Under "Panorama":
a. Select "S**imple Overlapping**" to start with or leave **Off** to do later.
b. Unselect "Shading Correction" box (can use for SE, don't need for BSE).

8. Under "Output":
a. Select either "**Live image size**" or larger format (e.g. 1024x1024).
b. Select TIF or BMP file format
c. Click on **...** box and provide the output path to a file folder in your account.
b. Set "Add Infobar to image" **off** (can add to stitched image later).
c. Click **NEXT**

9. In PROCESS window:
a. Under "After Finish," for long maps, select "**Turn emission off**" for overnight run. Don't use "Standby." Otherwise set to blank.
b. Click **START**. Map will begin. Progress can be observed in the monitor window and time in the lower box of the Image Snapper window.

C. When complete, stitch images into a single image
1. Select TOOLS menu and **Image Snapper**
a. Click **Stitch Offline.**
2. In INPUT IMAGE SET window:
a. Click **OPEN** button.
b. Navigate to saved files. Click on an image (e.g. "name1-1.tif").
c. Click **OPEN**.
d. Select "Stitching Method." Try "**Simple overlap**" first. If stitching artifacts are present, use "**Correlation matching**."
e. Click **START**
3. Save image

D. To add scale bar or infobar to final image
1. Open image from FILE menu and **Open Image.**
a. Select SEM TOOLS menu and **Image Processing**
b. Under "Select a category," use "Infobar manipulation" to add infobar to the whole panorama image.
c. Can make other adjustments by changing categories.
2. To add scale bar on image,
a. Click **Image Measurements** tool.
b. Click Scale Bar and place on image.
1) Select text box and drag larger to make text visible.
c. Save image.

II. X-POSITIONER (OVERLAY IMAGES)

Use this procedure to overlay a scanned image or photograph on the BSE/SE image and use that to control stage position, for example, from an optical photomicrograph.

A. Load and calibrate an external image (any image file format will work)
   1. Full thin section scans may be used by starting in lowest magnification. Use a stage Z position of 40 or 50 mm and "Field Mode" in the SEM scan window.
   2. Select **X-Positioner** from the TOOLS menu.
      a. Select **OPEN** button.
         1) Navigate to "Static" or external image file.
         2) Load image.
   3. Define 2 fiduciary points in the image (best to use 2 diagonal marks)
      a. Click on the **first** point in the static image.
         1) Move the stage to the same point in the live SEM image.
         2) Click on the same point in the live SEM image.
      b. Click on the **second** point in the static image.
         1) Move the stage to the same point in the live SEM image.
         2) Click on the same point in the live SEM image.
      c. Click **OK**.
   4. Calibrate the live SEM image to the static image.
      a. Click on **New Item** in the **Sets of Layers** tab in the X-Positioner window. The static image will be overlaid on the live image.
         1) To adjust overlay transparency, click **Options** tab.
         2) Move transparency slider (lower right) toward "Original" to increase static image transparency or toward "Overlay" to make it darker.
      b. To improve fit between images, click **Calibrate** tab.
         1) Use arrow keys to align the static image with the live SEM image.
         2) When satisfied with alignment, click on "Use Calibration."

B. Use static image to navigate the SEM stage and save positions on the static image.
   1. Double-click on the static image to move to any point.
   2. To save annotations on the static image, click on **Positions** in the X-Positioner window.
      a. Click on **Add Position** in the X-Positioner window. The position name will be overlaid on both the live and static images.
      b. Static window with marked positions can be saved.
         1) Make sure static window is selected by clicking on the window header bar.
         2) May need to adjust size of text in the static window.
         3) From the Vega TC menu bar, select Save Image and enter a file name.

IXRF SOFTWARE

I. EDS SPECTRAL ACQUISITION

A. Start **Iridium Ultra** software on the right-hand monitor.

B. Set acquisition parameters
   1. Set **Time Constant** (TC). Note that smaller time constants produce lower deadtime and more throughput but lower spectral resolution (wider peaks).
      a. Use TC 0.5 for most mapping
      b. Use TC 1.0 for spectral collection and quantitative spot analysis
      c. Use TC 2.0 for light element analysis
   2. Set **Preset** time to 30-60 sec (or more for more accurate analysis)
   3. Stage height (WD) on microscope should be between 20 and 15 mm, with maximum count rates obtained at 15 mm WD.

C. Collect spectra.
   1. For single spectrum acquisition, control acquisition position from the Tescan scan monitor. Either:
      a. zoom the microscope to highest magnification on the point of interest, or
      b. double click on scan window to get reduced area box. Shrink the box with the right mouse button to a small size and place on the point of interest.
      c. Click on **New Spectrum** icon in the IXRF tool bar.
   2. For multiple acquisitions on the same image, set appropriate magnification on the SEM and collect an image on the IXRF.
      a. Click on **Camera** icon in the tool bar to collect image.
      b. Spot Mode: click on cross hair at right side of tool bar and click on image.
         1) Other modes such as freehand area or rectangular area can also be selected on the image.

D. Quantify spectra
   1. Identify all peaks that will be analyzed. Coatings such as C, Au and Pd should not be selected. Otherwise they will be included in the analysis.
   2. In the TOOL BAR, click on the **Lightning Bolt** (or RIGHT CLICK->ANALYZE->QUANTIFY).
      a. To get oxide values, RIGHT CLICK->PROPERTIES. Click **Display** tab.
      b. Click **Components Table** and check the boxes "Component, Conc, & Units" ("Mole Conc" if desired).
      c. Now force oxide recalculation of Components. From **Properties** dialog window, click on **Advanced** button in "Quantitation" section.
      d. In the dialog box, click on **Convert to Oxides**, select elements, **OK** & **Close**.
      e. Force the program to recalculate the analysis with oxides by clicking on the **Lightning Bolt** again.

E. Save and export spectra and reports
   1. Save each spectrum and image if you wish to keep it or if you would like a name on the exported reports. FILE->SAVE AS. Save as type = spectrum (.xsp).
      a. If you want an image of the spectrum, export from the Save window by checking the EXPORT box at the left side and selecting file type (usually JPEG).
   2. All open spectra, images, and maps can be saved in a single file from FILE->SAVE AS EDS DATA SET.

3. To build a Word-formatted report, select FILE->REPORTS->CREATE WORD REPORT.
   a. A new dialog window will open. Transfer from the list of files on the left.
   b. If an image has been collected, select "Image" and then click **Add to Report**.
   c. Expand "Spectrum" folder. Clicking on the name will add both the spectrum and quantitation data, if quantitative analysis has been done.
   d. When finished, select FILE->SAVE AS in the dialog window.
4. To save quantitative analyses results as a text table (CSV format), select FILE->NEW.
   a. In the Dialog box, select **Spectrum Report**. An empty window will appear.
   b. Select DATA->ADD SPECTRA.
      1) Click on the left-hand box beside each spectrum to select it for saving or click on the upper left box to select all. The boxes will turn green when selected.
      2) Click OK. Only the elemental weight percent values will be saved, not the oxide values.
   c. Select FILE->EXPORT. Save as CSV text that can be opened in Excel.

IXRF File Tags

File Type	Tag
EDS Spectrum	.xsp
EDS Data Set	.eds
Image	.imx
Single X-ray Map (Fast Map)	.mpf, .mpx, .mps
Line Scan (Fast Line Scan)	.lnf, .lnx, .lns
Spectrum Report	.srp
Stage Scan	.sls
Stage Map	.sma
Stitched Image	.imxs
Stitched Map	.mpfs
VP-mode Spectrum	.vps

II. MAP ACQUISITION

Use this procedure for doing a single X-ray map.

A. Set up area to be mapped on the Tescan monitor, including desired magnification, BSE detector, and brightness/contrast.
   1. Maps require a high count rate. Typical microscope conditions include:
      a. Accelerating voltage = 20-25 kV
      b. Beam intensity = 15-20
      c. Working distance = 17-15 mm
   2. Set IXRF Time Constant to TC = 0.5 or 1.0

B. Set **Fast Map** on IXRF
   1. Select FILE->NEW from the Menu bar.
      a. In the Dialog box, select "Fast Map."
      b. Right-click on the blank map window and select **Properties**.
      c. Under the **Acquisition** tab, set the desired parameters. Typical values:
         1) Dwell time = 0.5-3 msec
         2) Multiple frames box checked
         3) Number of frames = 1-20
         4) Resolution = 256 or 512 pixels
      d. Under **Elements** tab, select elements desired. Note, elements can be added or subtracted at any time.

C. Start map
   1. Click on **Acquire** button at left side.
   2. Check status in the lower left corner of the IXRF window.
      a. The software may not use the right resolution or number of frames.
      b. If necessary, **Stop** the map, check the **Acquisition** parameters again, and start again.

D. Save and export maps
   1. Select FILE->SAVE AS. Save as type = fast map (.mpf).

III. STITCHED IMAGES AND MAPS

Use this procedure to acquire multiple X-Ray maps over a large area and stitch them together into a single image. Bear in mind when selecting numerous maps that IXRF stitching software may leave visible overlaps that will need to be hand corrected.

A. Determine corners of mapped area.
   1. It may be helpful to have a photo of the entire sample to help set up the map area.
      a. With polished sections or 1" thin sections, use **WIDE FIELD** mode and low magnification to collect image of entire sample.
      b. For rectangular thin sections or larger samples, take a high-resolution scan on a flat bed scanner before inserting the sample in the SEM.
   2. Import the image into ImageJ and set scale.
      a. Draw rectangle to define the map area.
         1) You will need to determine size of map area (even numbers helps!).
         2) Map will start in upper left and progress to lower right.
      b. Move stage to upper left corner and record stage coordinates.
      c. Repeat for lower right corner.

B. Set up instrument
1. For maps, set up for high current.
   a. KV: 20-25 kV
   b. BI: 16-20
   c. Z: 15 mm
2. Insert BSE and switch to BSE mode.
3. For both maps and stitched images, set contrast & brightness to higher (brighter) values than what normally looks good on SEM monitor.

C. Set up IXRF software
1. Select FILE->NEW->IMAGE. Click **OK.**
   a. Select **PROPERTIES**.
      1) Set VIDEO SOURCE to 2 for BSE image (make sure detector is in)
      2) Set AUTO IMAGE ADJUST to **OFF**.
   b. Select ACQUIRE->START.
   b. Adjust Contrast & Brightness on SEM if necessary.
2. Set EDS for high throughput
   a. In menu bar, set TC: 0.5 - 1.0
   b. Collect spectrum (new spectrum icon).
   c. Adjust BI on instrument so that Deadtime is high but not over 40%.
      1) Example: @25kV, BI=18, TC=1, DT is around 21%
3. Set up "Fast Map"
   a. Select FILE->NEW->FAST MAP
   b. Select **PROPERTIES** and the ACQUISITION tab.
      1) Set DWELL TIME and RESOLUTION to give reasonably fast map (1-2 minutes per frame), but more time may be needed to see minor elements.
      2) Example: 1 msec per pixel @ 256 resolution, frame time is 1 minute 15 sec per frame.
      3) Software supports a maximum of 4096 x 4096 pixels in the entire stitched map. (Example: @256 resolution, a maximum of 16 x 16 fields could be collected)
      4) Set MAX SCANS: 1 or more (if 2 or more scans are desired, make sure MULTISCAN box is checked).
   c. Select the ELEMENT SELECTION Tab
      1) Click on desired elements (Note: this need not be an exhaustive list as display elements will be selected later).
   d. Click on SET AS DEFAULT.

C. Set up image stitching software.
1. Select FILE->NEW->STITCHED MAPS. A blank screen will appear.
2. Select **ACQUIRE** and **START.**
3. A setup window will appear (Figure EDS1).

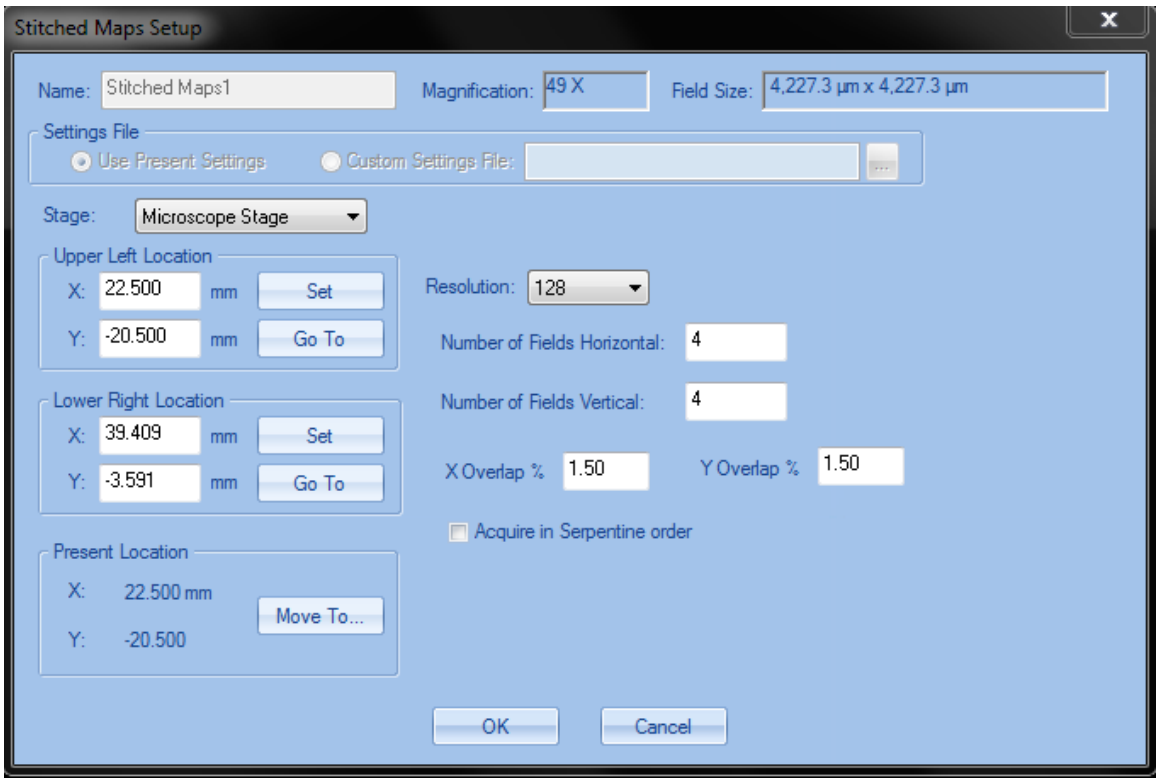


Figure EDS1. Stitched maps and images setup window.

a. You will adjust field size and number of fields to fill desired map area.
   1) Set **MAGNIFICATION** on SEM to give appropriate field size (2 mm or less). Field size is shown in upper right corner.
   2) Magnification of 100x or greater works best. Magnification lower than 100x (or greater than 2 mm field size) may cause darkening at edges.
b. Enter recorded upper left stage coordinates in UPPER LEFT LOCATION box. Click GO TO if not already there.
c. Enter recorded lower right stage coordinates in LOWER RIGHT LOCATION box.
d. Check number of fields in NUMBER OF FIELDS HORIZONTAL and NUMBER OF FIELDS VERTICAL boxes. You may need to play around with these numbers and field size (magnification).
e. **Note:** If number of fields is hand entered, check that LOWER RIGHT coordinates are correct. See Figure EDS2 or EDS3 below.
   1) Software may try to send stage in the wrong direction (e.g. moving to left rather than right).
   2) If so, enter NUMBER OF FIELDS HORIZONTAL as negative.
f. Select RESOLUTION and set most likely 128 or 256. Don't exceed 4096 x 4096 for total image pixels.
g. Set OVERLAP%: 0 - 1.
h. For large maps, select ACQUIRE IN SERPENTINE ORDER.
g. Click **OK**. Map will start.

4. After completion, maps should be saved and then exported.
   a. Select FILE->SAVE AS to save raw images.

b. Select FILE->EXPORT AS STITCHED to create single stitched BSE image.
c. Stitching may take some time, depending on number or fields.

5. After exporting stitched maps, a Map Window appears with the completed image and a blank spectrum.
   a. Right click on window and select **PROPERTIES**.
   b. Select ELEMENT SELECTION tab.
   c. Click on element to select a map.
      1) Check box SHOW MAP shows individual map windows.
      2) Check box ON SEM shows maps as overlays on SEM image.
   d. Save individual maps in "tiff8" format to manipulate in Photoshop or ImageJ.

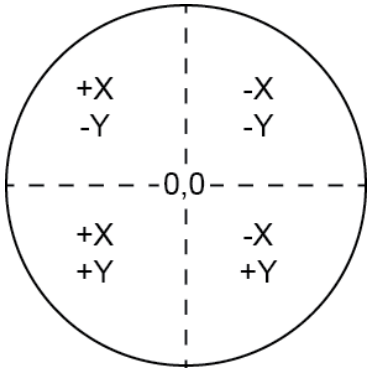


Figure EDS2. Stage X-Y coordinates

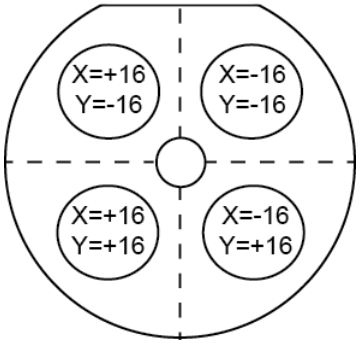
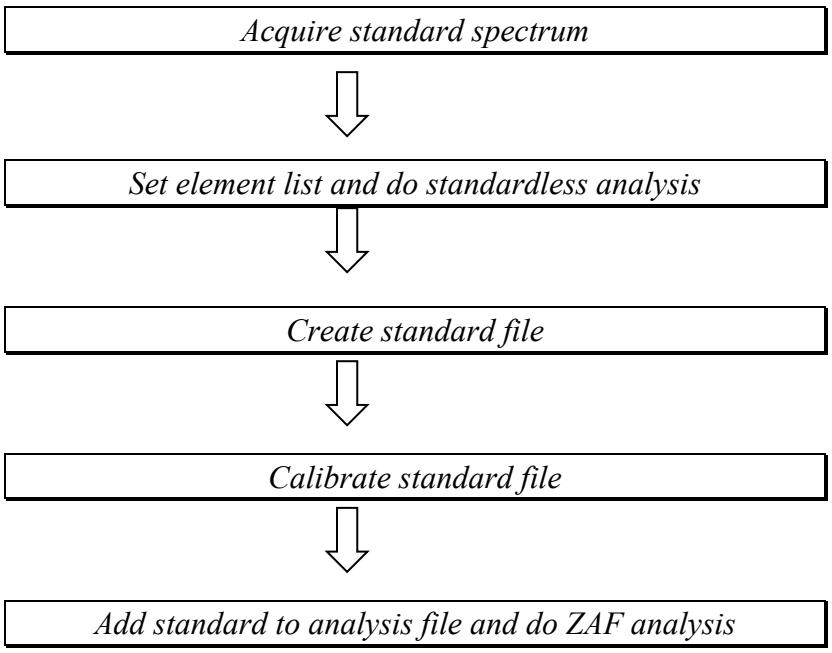


Figure EDS3. Stage X-Y coordinates for 4" round thin section holder.

IV. QUANTITATIVE ANALYSIS USING STANDARDS

A. Standards analysis flow sheet.



B. The following must be done to use standards analysis.

1. Acquire standard spectrum.
   a. Collect a spectrum on suitable sample/standard mineral.
   b. Note the excitation and geometry parameters such as Accelerating voltage, Time Constant, Z height.
   c. Save the spectrum (StandardName.xsp).
2. Setup element list and analyze the spectrum
   a. Ensure that spectrum processing is proper set (smoothing, strip Si Escape, background removal, deconvolution)
   b. Perform standardless analysis to obtain integrated counts
   c. Conditions can be saved using SPECTRUM PROPERTIES->SAVE SETTINGS.
3. Create standard file
   a. Select UTILITY->CREATE STANDARD
      1) Enter the correct concentration values from an independent analysis of the standard.
      2) Add any other elements present in the standard even though they may not be selected for calibration, such C or H.
   b. Click **Save** button to save the standard file. The same name as the spectrum should be used for later in the calibration step (StandardName.std).
4. Calibrate standard file
   a. Select UTILITY->QUANTITATION CALIBRATION
      1) Browse for or enter the standard file name that was just created.
      2) Click on **Calibrate** button to perform the ZAF calibration.
   b. When the SAVE dialog appears, enter an appropriate file name and click the **Save** button to to save the calibration file (StandardName. fpc).

1) Note that calibration files can be merged such that standard spectra from different standards can be combined in one file, e.g. Mg, Si, and Fe standards could be merged into an "Olivine" calibration file.

5. Use standard file in a ZAF analysis
   a. Acquire an unknown spectrum using the same conditions as that standard acquisition.
      1) Accelerating voltage, incident angle, Z height must be the same.
      2) Quantitation conditions can be loaded from SPECTRUM PROPERTIES->LOAD SETTINGS.
   b. Enter calibration file into analysis file for the unknown.
      1) Select SPECTRUM PROPERTIES->QUANTITATION-> ADVANCED.
      2) Click on the gray select button to the left of an element name. The button will turn green.
      3) Click on the "Calib File" cell and select the appropriate calibration file from the dialog window. Click **Open** button and the calibration file will appear in the "Quantitation Advanced Settings" window. See Fig. EDS4 below.
      4) Add additional standards and perform the ZAF analysis as usual.

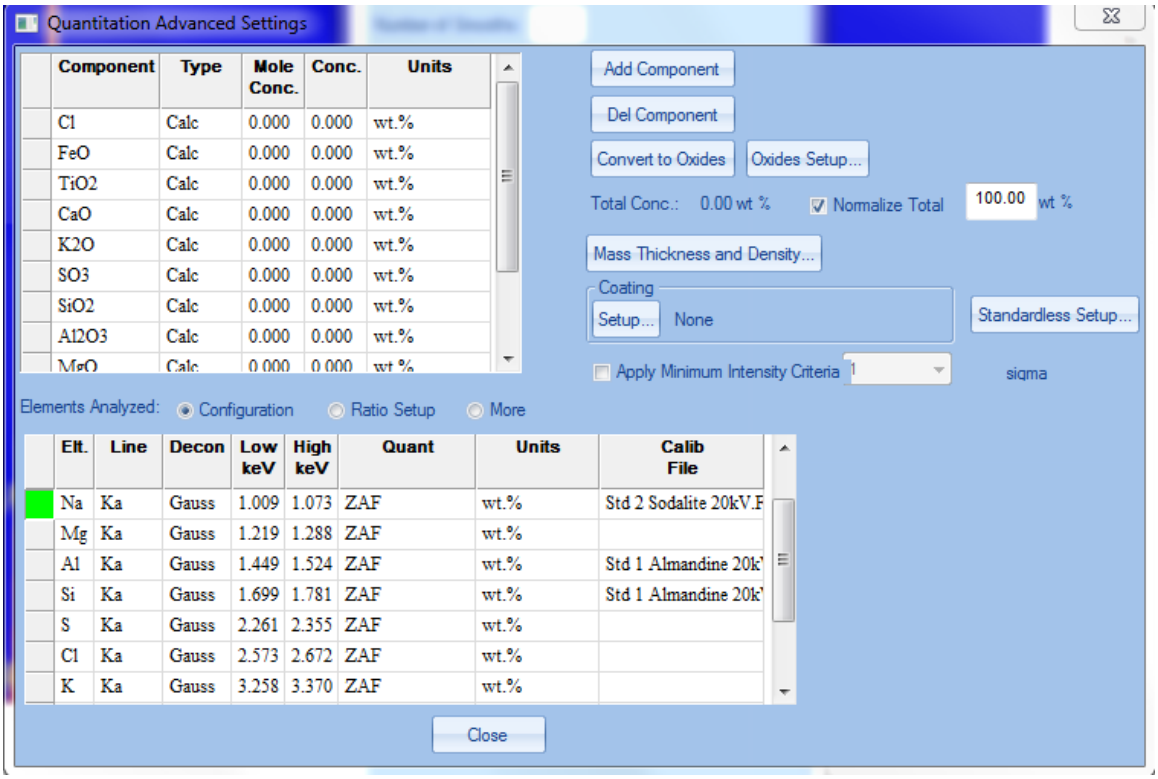


Figure EDS4. Quantitation Advanced Settings window. The "select" button for Na, now green, has been clicked and the file "Std2 Sodalite 20kV" has been enetered in the "Calib File" cell.

OPERATION IN LOW VACUUM MODE

I. SET UP INSTRUMENT

It is easiest to align the gun and column in high vacuum mode, then insert the low vacuum aperture and load your samples. Finally bring up the instrument in low vacuum mode.

A. Start Tescan software from the  icon on the left-hand monitor.
   1. Select user name and enter password (usually **SEMUser** with Password **Tescan**).
   2. Select an account at the bottom of the window (for example, Clients) and click **OK**

B. If the stage is in the exchange position near the door, move it to the center position.
   1. Enter "0" in the **Y** box on the STAGE CONTROL window and click **OK**,

C. Set accelerating voltage and beam intensity that you will use in low vacuum mode.
   1. In the INFO PANEL, check the HV setting.
      a. Use 10-20 kV for most low vacuum applications.
   2. To change accelerating voltage, click on the **HV** number in the INFO PANEL, then enter the new value in the PAD above (See Figure 1, Page 2).
   3 Set Beam Intensity to around 12 (read in the INFO PANEL)
      a. Adjust beam intensity with the **BI** knob on the desktop CONTROL PANEL.

D. Setup the gun.
   1. In the SEM DETECTORS & MIXER panel, make sure the secondary detector (SE**)** is activated. If not, click ▼ and select **SE**.
   2. In the Vacuum panel, both "Gun Pressure" and "Column Pressure" should be green to indicate that the chamber is under vacuum.
   3. In the ELECTRON BEAM panel click on **HV** button. The gun will automatically heat over a period of about 1 minute.
      a. **HV** and **Heat** buttons will both turn blue when the gun is ready.

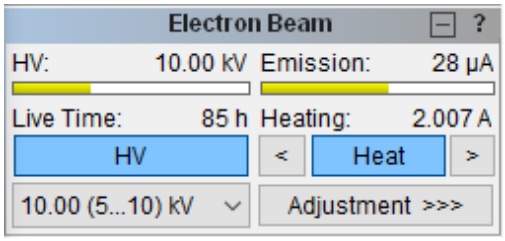


Figure LV1. Electron beam panel.

   4. Perform an automatic gun alignment:
      a. Move to any place on the aluminum stage.
      b. Click on the **Adjustment>>>** button in the ELECTRON BEAM panel.
      c. Select **Auto Gun Centering**.

E. Do an initial image focus on the stage.
   1. Right click on SEM SCANNING WINDOW and select "Minimum Magnification."
   2. Click on **Auto** contrast and brightness in the IMAGING TOOLBAR.

3. Set scan speed by clicking on **Speed** in the IMAGING TOOLBAR. Use a relatively fast scan speed like 1, 2 or 3.

3. Use the WD knob on the desktop CONTROL PANEL to initially focus the image.

F. Optimize the image.
1. Find a small feature such as a dust particle on the stage to focus on. Adjust contrast & brightness if necessary.
2. Increase the magnification significantly (e.g. 2000X) by turning the **MAG(nification)** knob on the Tescan CONTROL PANEL. Keep the small feature in view.
3. Focus carefully with the **WD** knob.
4. Set the desired height, usually between 25 and 15 mm for low vacuum work.
   a. After focusing, the **WD & Z** box in the STAGE CONTROL window displays the correct WD.
   b. To change working distance without defocusing the image, enter a value in the **WD & Z** box. A standard height such as 25 or 15 mm can be selected by clicking ▼ on the right side of the **WD & Z** box.

Always watch the infrared camera when raising the stage!
If the sample appears that it might, for whatever reason, contact the column or BSE detector, immediately press STOP in the STAGE CONTROL window to abort the move.

5. Check the Objective Lens Centering.
   a. Click on the **Wobble** button in the imaging TOOLBAR.

   b. A new window will appear. Click **WOB** and then **NEXT**.
   c. Use the small knobs on the center of the desktop CONTROL PANEL to adjust X & Y alignment until image remains centered while wobbling in and out of focus.
   d. Click **FINISH**.
6. Complete image optimization by checking stigmation.
   a. Click on the **Stig** button in the imaging TOOLBOX.

   b. In the PAD panel above the Info Panel, click **Reset** to set the stigmators to 0.
   c. If necessary, use the small knobs on the center of the desktop CONTROL PANEL sharpen the image as much as possible.

II. VENT INSTRUMENT AND INSTALL LOW VACUUM APERTURE

A. Turn off the electron gun.
1. Click on **Heat** button in the ELECTRON GUN panel to turn off the gun but leave the voltage on the LaB6 filament (See Fig. LV1).

B. Vent the chamber

1. Click on the **VENT** button in the VACUUM window.
   a. Wait until the chamber backfills.
   b. Swing the front door out and to the left.

C. Install the low vacuum aperture

1. Click on Position 1 in the STAGE CONTROL window to move the stage down and out to the exchange position at the door.
2. Enter "90" in the **Z** box on the STAGE CONTROL window and click **OK**.
3. Insert the aperture into the bottom of the column using the mounting screw that extends from the aperture. WEAR GLOVES. See Fig. LV2 below.
   a. Give the aperture body a slight twist as you insert it, pushing it up into the column.
   b. Remove the mounting screw and place in the plastic aperture storage box. Don't lay it on the counter--it will disappear.

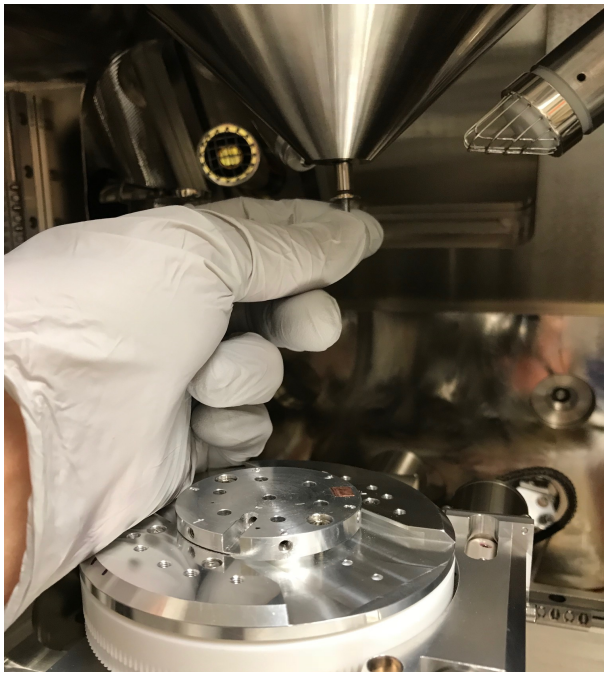


Figure LV2. Inserting the low vacuum aperture.

III. START THE INSTRUMENT IN LOW VACUUM MODE

A. Set the instrument to Low Vacuum mode.

1. Click on the **PUMP** button in the VACUUM window to start pumping.
2. Expand the LOW VACUUM MODE window by clicking in the + box on the right side of the title. The LOW VACUUM MODE window is shown in Figure LV3 below.
3. While the instrument is pumping, click on the **UniVac** button to start the low vacuum operational mode.
   a. Enter a value for the chamber pressure in the box to the right of the **UniVac** button.
   b. Pressures between 30 and 50 Pa work well for imaging. Start low and add pressure later if the image shows evidence of charging.

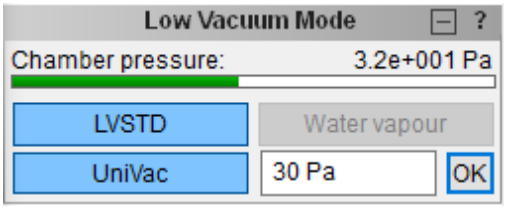


Figure LV3. Low Vacuum Mode panel.

B. Proceed with setup while instrument is pumping down.
   1. Move the stage back to the sample.
      a. Enter "0" in the **Y** box on the STAGE CONTROL window and click **OK**.

      OR

      b. If you have loaded pin-mounted samples, when the STANDARD TESCAN CAROUSEL turns green, click on the appropriate sample.
   2. Enter "50" in the **Z** box on the STAGE CONTROL window and click **OK**.
   3. Click on **LVSTD** button to start the low vacuum SE detector.
      a. In the SEM DETECTORS & MIXER panel, the low vacuum secondary electron detector (LVSTD**)** will automatically be selected
      b. In the LOW VACUUM MODE panel, the "Chamber Pressure" will be green and the LVSTD button will turn blue when the vacuum level has been achieved.
   4. In the ELECTRON BEAM panel click on the **Heat** button. The gun will automatically heat over a period of about 1 minute.
   5. **Heat** button will turn blue when the gun is ready and the beam is on.

C. Do an initial image focus.
   1. Reduce Magnification to minimum.
   2. Click on **Auto** contrast and brightness in the IMAGING TOOLBAR.

   3. Look for a pattern of several bright spots to appear in the image. You will need to use the IML Centering to "gather" the spots into a bright rectangle.

D. Use the IML (Intermediate Lens) to center the beam.
   1. In the PAD panel, click ▼ to get the list of selections.
      a. Look for and select **IML**, about half way down the list.
   2. Check that the desktop CONTROL PANEL says the **IML** is activated.
      a. Adjust the X and Y knobs on the CONTROL PANEL to force the beam into a skewed rectangle (see Figure LV4).

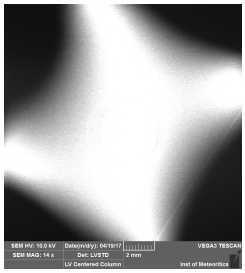


Figure LV4. Centered electron column in low vacuum mode.

b. Click on **Auto** contrast and brightness button again.

E. Find visible features on your sample and focus the image.

F. Raise the stage to the previous height used earlier to align the instrument

1. To change working distance without defocusing the image, enter a value in the **WD & Z** box. A standard height such as 25 or 17 mm can be selected by clicking ▼.

Always watch the infrared camera when raising the stage!

If the sample appears that it might, for whatever reason, contact the column or BSE detector, immediately press STOP in the STAGE CONTROL window to abort the move.

G. After raising stage, optimize focus.

1. If necessary, check IML centering again.

H. Start work on your sample.

IV. SHUT DOWN THE INSTRUMENT AND REMOVE THE APERTURE

A. Turn gun off and vent the chamber.

1. In the ELECTRON BEAM panel, click on **HV** button.
2. Turn off the **LVSTD** detector and allow it to spin down for a few minutes (listen for the high-pitched whine to decrease).
3. Click on the **VENT** button in the VACUUM window.
4. Click on the **UniVac** button to turn if off.

B. Return stage to exchange position.

1. Wait until the icons in the STANDARD TESCAN CAROUSEL turn red.
2. If you have loaded pin-mounted samples, click on the first sample (**1**) to move it and rotate it toward the door.

C. Remove your samples.

D. Remove the low vacuum aperture.

1. Enter "90" in the **Z** box on the STAGE CONTROL window and click **OK**.
2. Insert the mounting screw into aperture in the bottom of the column using the mounting screw that extends from the aperture. WEAR GLOVES. See Fig. LV2.
   a. Turn the screw into the aperture a few turns, then pull straight down to remove the aperture.
   b. Return the mounting screw and aperture place in the plastic storage box. Don't lay it on the counter--it may disappear.
3. Gently swing the door closed.
4. Click on the **PUMP** button in the VACUUM window.

D. Secure the instrument.

1. Close the software.
   a. Click on the **Exit only** button.

E. Fill out logbook

F. Clean up around instrument and sample prep counter.