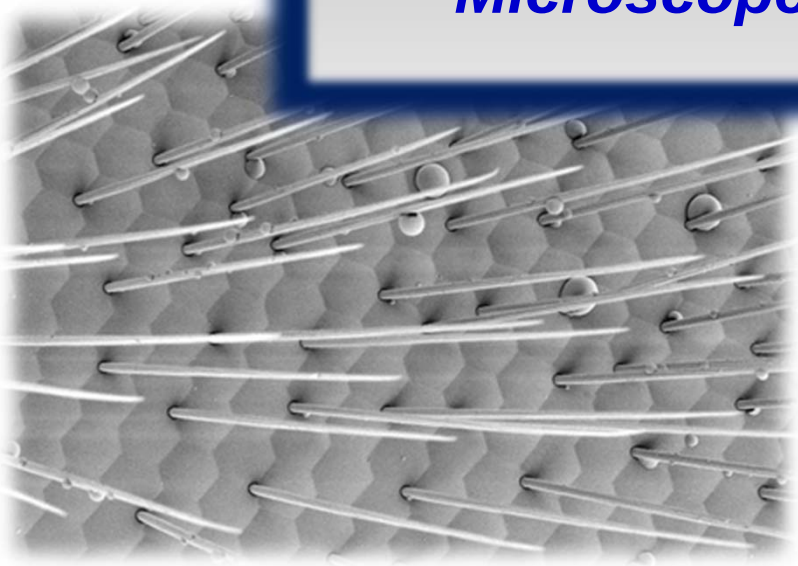
Operating Manual

Keck

Field Emission Scanning Electron Microscope



Contacts

Mick:	255-0650
Cornell Police:	255-5111
Emergency:	911
Phil Carubia:	255-6757

1.00

Laboratory Safety

Users are required to have passed Cornell's on-line laboratory safety training courses before they can use the Keck SEM.

Do not bring any chemicals or hazardous samples or materials into the lab.

Take specimens, stubs, and raw material with you when you leave. Do not store them in the bakeout chamber.

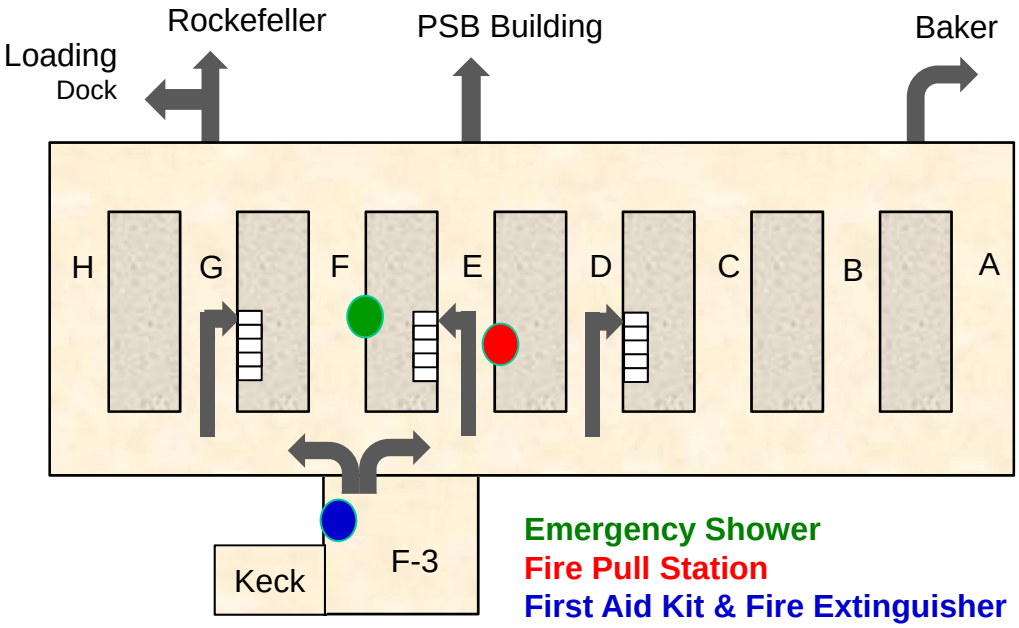
Isopropanol and Aero-Duster are supplied for your use. Wear safety glasses and read the appropriate MSDS before using them.

Nitrogen gas cylinders are changed by CCMR staff only.

Users failing to follow safety rules will be denied access to the lab.

Fire Safety

In case of fire leave immediately and close the door behind you. Do not use the elevators.



1.05

Laboratory Policies

If ANYTHING does not even SEEM right, leave everything as it is and get or call Mick (255-0650)

DO NOT attempt to repair ANY part of the microscope or remove panels, cables, or any part of the SEM.

DO NOT add any software or hardware to the computer

Flash Drives (Memory Sticks) are forbidden
Data MUST be removed by burning a CD or via Cornell DropBox

DO NOT change the room temperature or bring food or drink into F-3 or the SEM room

Wear gloves when venting the SEM, handling samples, or using the pumping station

If you sign up and then cannot use your time, cancel your time via Coral or contact Mick at least one weekday before your scheduled time.

Failure to show up without canceling, unless due to illness or other emergency, can result in use charges

If you are more than 30 minutes late then the microscope becomes available to other users.

If you feel sick, please reschedule your session. You will not be charged if you stay home due to illness.

1.10 Data Storage

Data storage and safety is NOT guaranteed! - users are responsible for their data, and must copy their data in a timely fashion.

Data will only be kept on the hard drive for one year. Periodically the hard drive will be erased, leaving only one years' data there.

1.20 How to Acknowledge CCMR

When research is published using data from this microscope, the facility and the grant number must be acknowledged:

This work made use of the electron microscopy facility of the Cornell Center for Materials Research (CCMR) with support from the National Science Foundation Materials Research Science and Engineering Centers (MRSEC) program (DMR 1120296).

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2.00 Access

User Status	Signup	Room Entry	Access
Novice (< 2 sessions)	Email	Manager	M-F 8am – 5pm
Trained (>2 sessions)	Coral	Card Swipe	M-F 8am – 5pm
Experienced (>4 sessions)	Coral	Card Swipe	24/7

DO NOT use your card to let others into the SEM room

DO NOT lend your card to other users.

About this manual (Version 1.0)

This is a ***limited use manual*** intended to help new users get started on the Keck SEM (Zeiss 1550). It provides information on basic imaging, column setup, and saving and annotating images. It is not intended to be exhaustive, and there are many principles about scanning electron microscopy and features of this microscope that are not covered by this manual. This manual will be updated and expanded as needed.

3.00

How to start the software:

Logon to Coral in outer room

Logon to SmartSEM

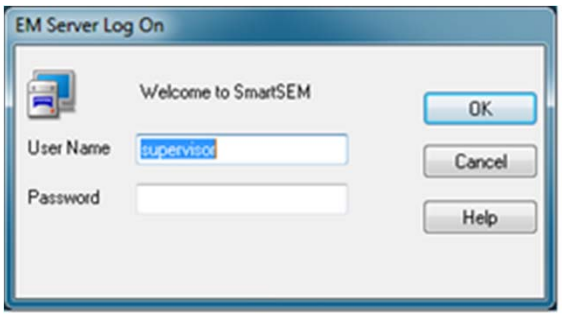
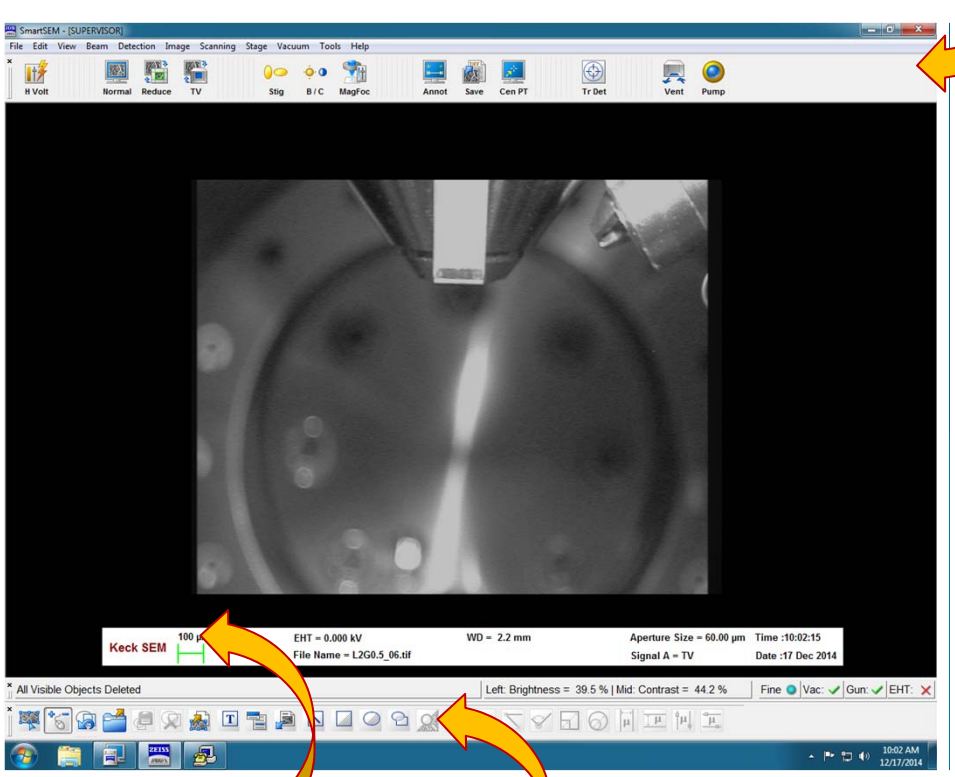


Image Control Toolbar



Data Zone

Annotation Control Toolbar

3.10

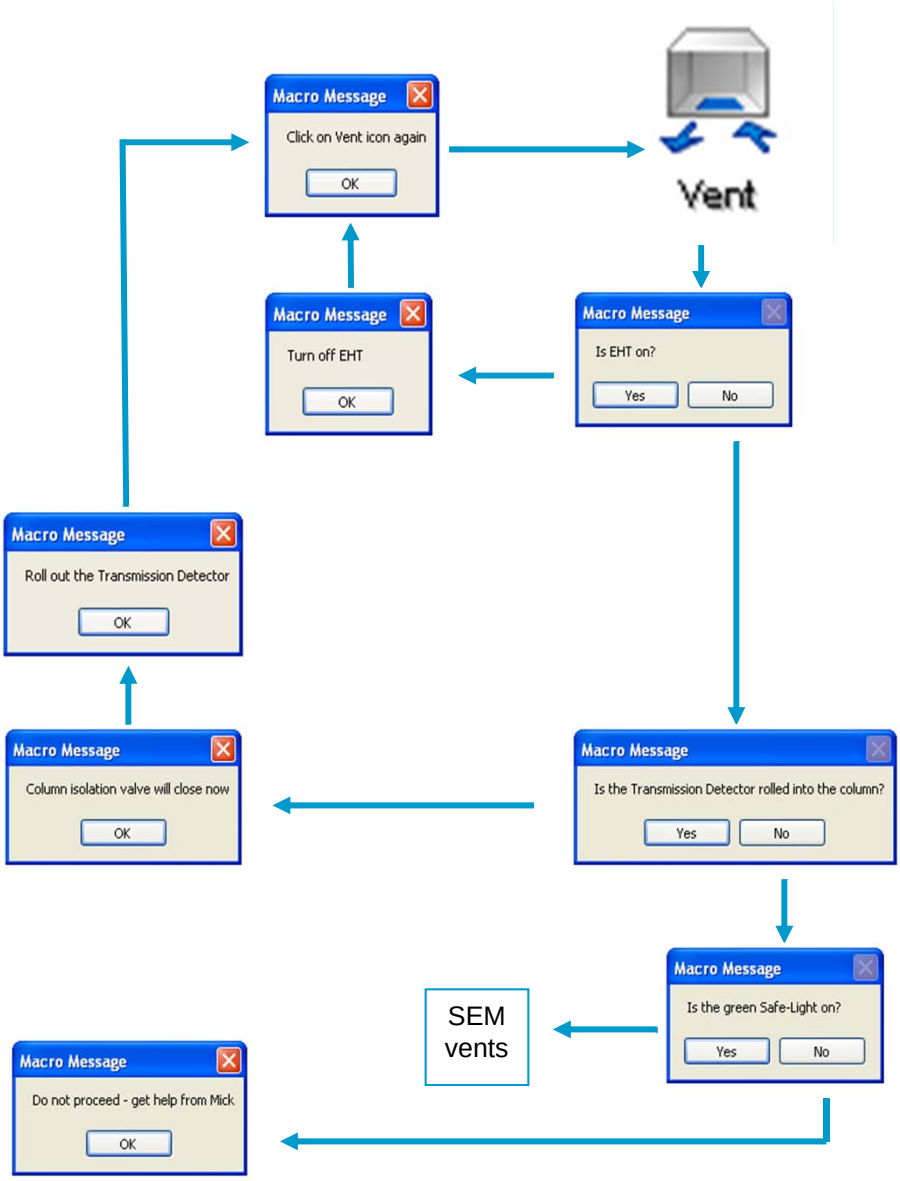
How to load the sample:

Click on the Vent Icon

Answer the questions in the dialogue windows

The stage will automatically go to the correct position

The column will vent



3.10

How to load the sample (continued)

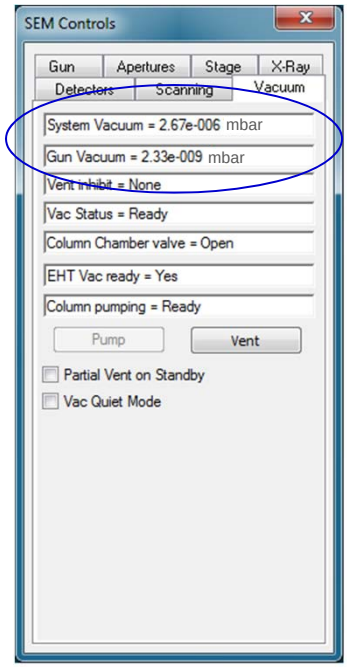
After venting, ensure that there is a gap between the motor and the chamber wall and the support bar and the chamber wall

Load the stage so that the flat part of the sample holder is flush with the flat part of the stage.

3.20 *How to pump down the chamber:*

Squeeze the chamber door shut with your left hand and click on Pump

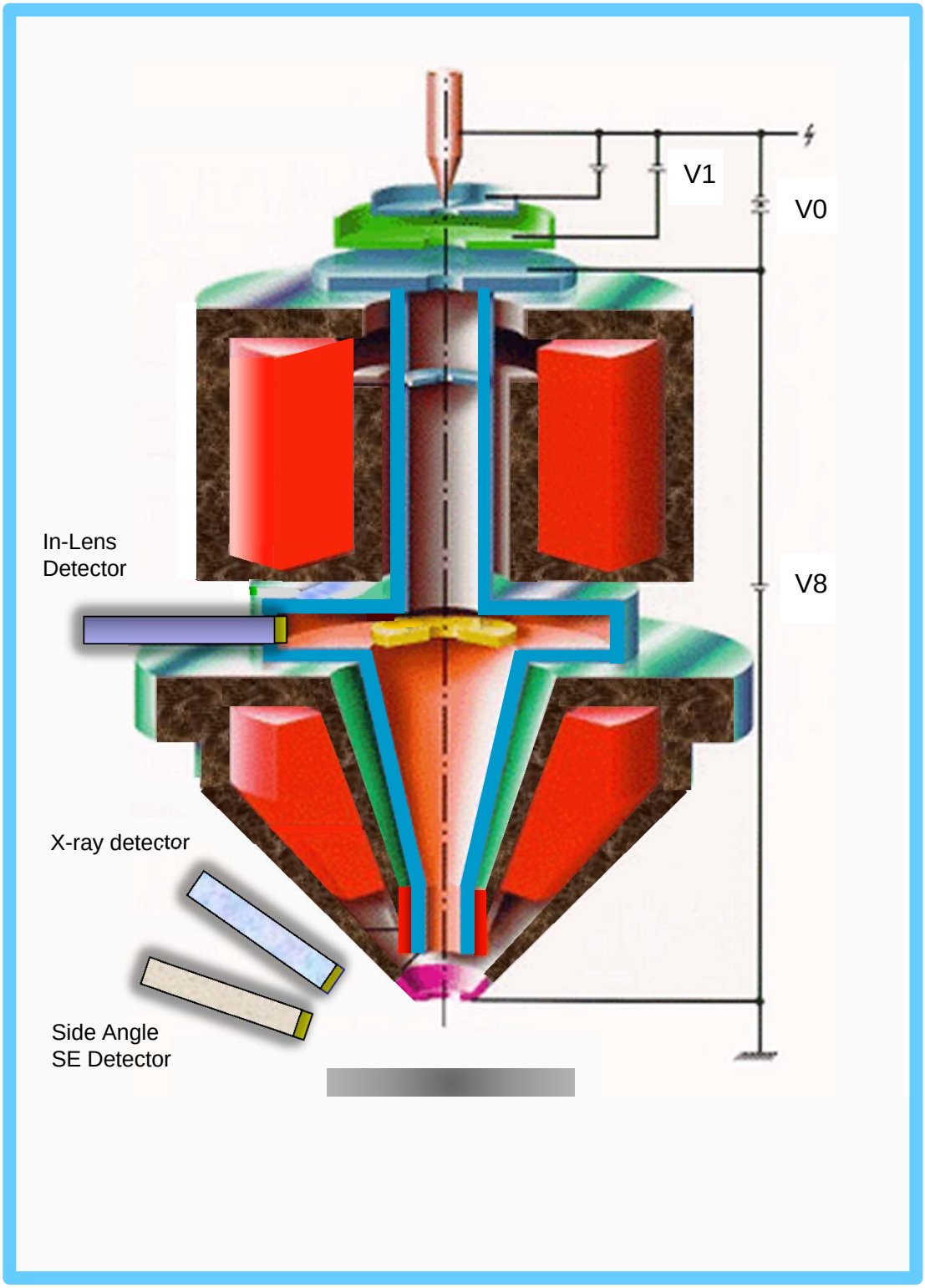
3.25 *Vacuum requirements before starting:*



System Vacuum < $2.0X10^{-5}$ mbar
Gun Vacuum < $5X10^{-9}$ mbar

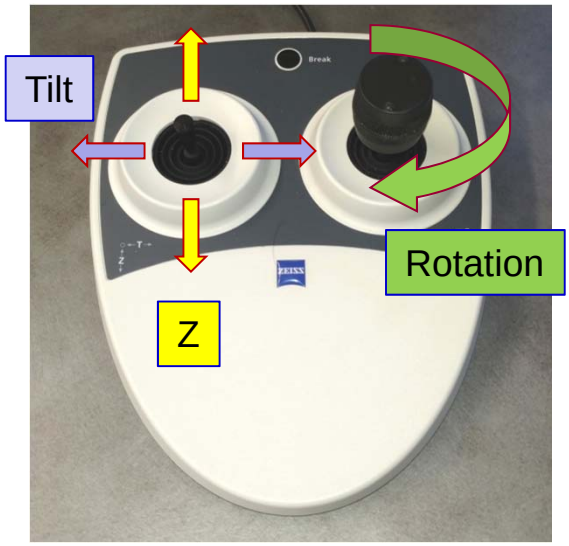
3.30

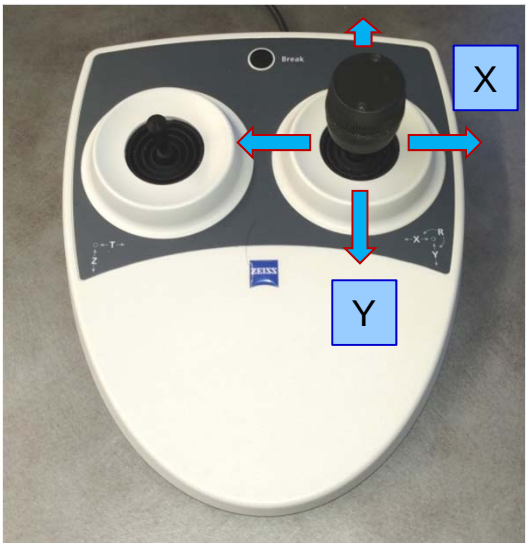
Cross-section of the microscope column:



3.35

How to mechanically move the stage:





How to move the stage with software:

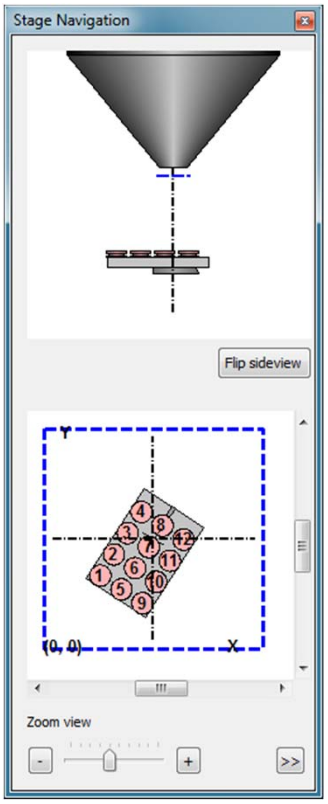
Click on **CenPt** – put cursor over area of interest

You can save positions so that you can come back to a previous position quickly.

Give the location a name

On the Stage Points List click on **Add**

3.37 *How to position your sample for imaging:*



Move the sample in Z-direction with the joystick to about 5mm from the end of the pole piece as shown below.

Do not trust the cartoon at left showing the gap between the sample and pole piece – your sample height will likely be different!

If you are using the 12-holder stage then select the sample you want to examine by clicking on the number.

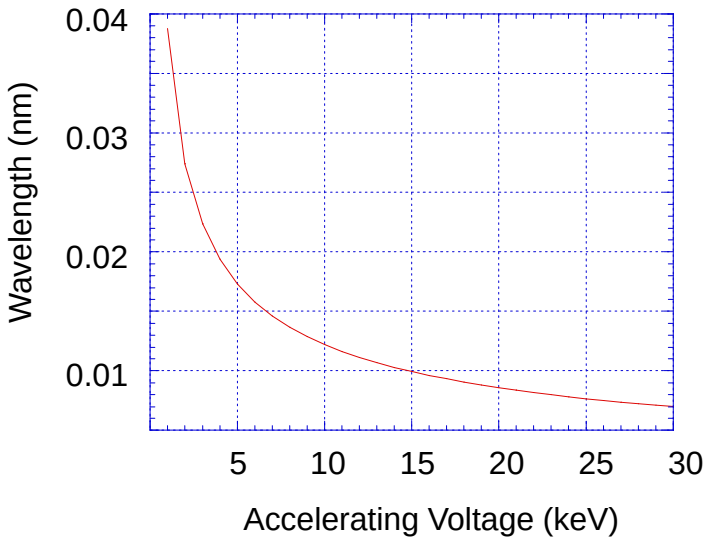
Otherwise move the sample under the pole piece using the joystick.

3.40 How to set the high voltage (EHT):

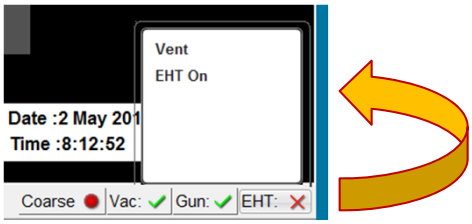
Click on the **H Volt** icon and type in a value in keV.

For imaging the EHT is generally set between 1keV and 5keV.

For x-ray analysis the EHT is generally set to about 3-4 times the energy of the edge you will analyze. For example, to analyze the K-alpha edge of titanium (~4.5keV) you want to set the voltage to about 18keV.

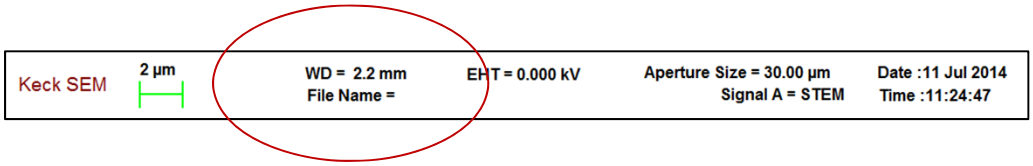


3.45 How to turn on the EHT



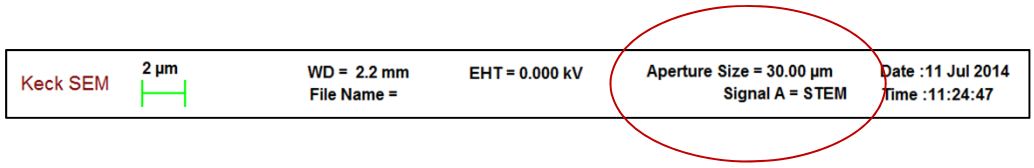
3.47 How to set the initial conditions:

If the sample distance looks similar to the previous page, set the Working Distance (WD) to about 5mm.



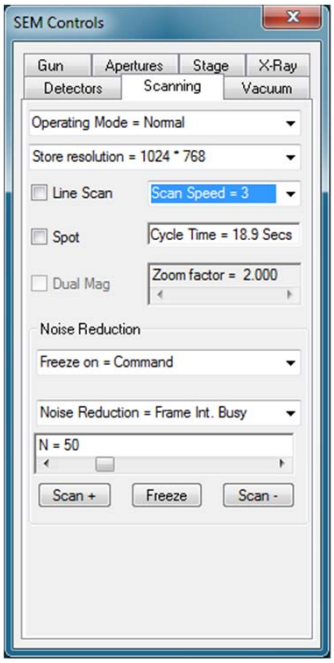
Set the aperture to 30um.

Set the Signal A to InLens



3.50 How to start imaging:

Click on **Normal**



Set the Scan Speed = 3
(Note the lower the number, the faster it scans)

Set Operation Mode to Normal

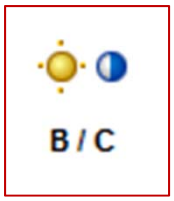
Set Store resolution to 1024 x 768

For continuous imaging:
Set the Noise Reduction = Pixel Avg

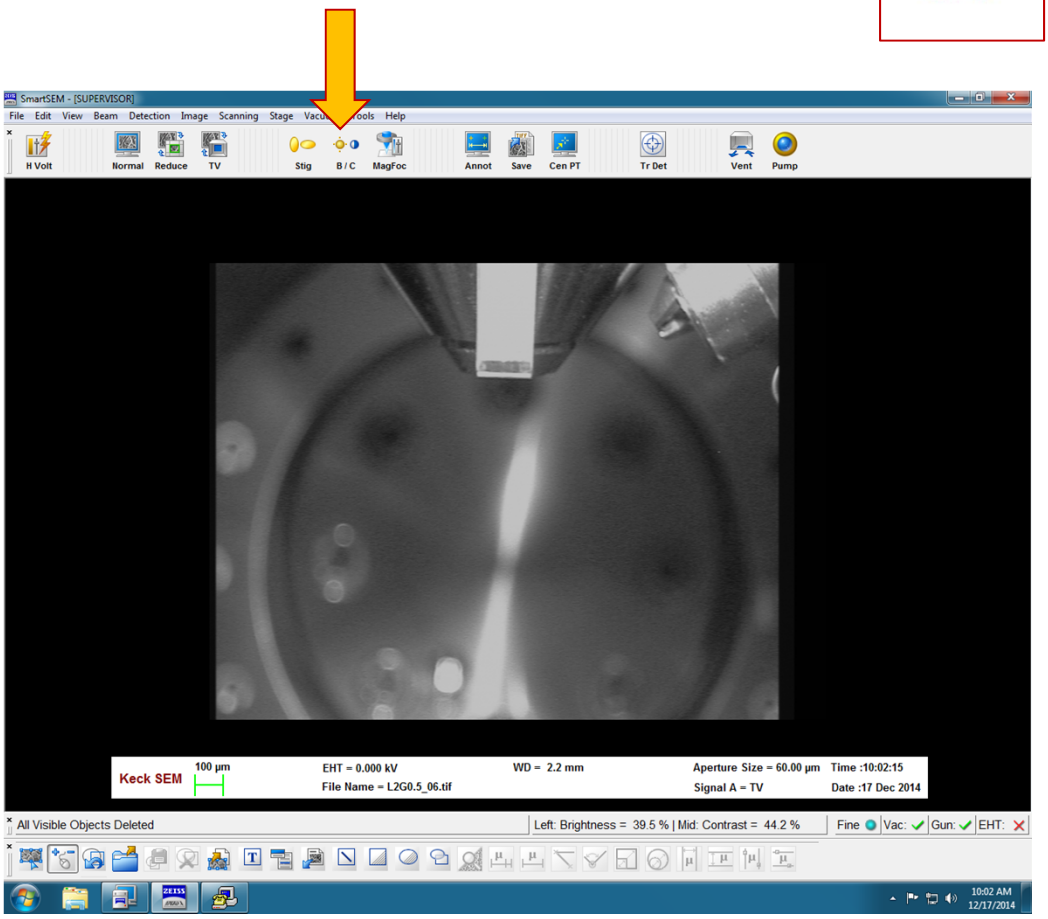
For recording images:
Set Noise Reduction = Line Int or Frame Int

3.53

How to set Brightness and Contrast

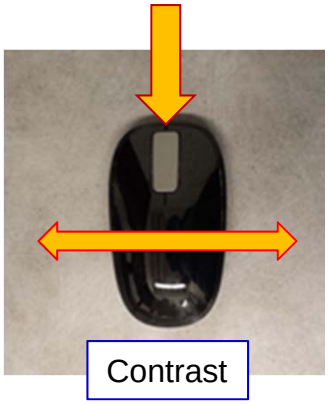


Click on the Brightness / Contrast icon



To adjust Brightness click on the left mouse button and move the mouse left or right.

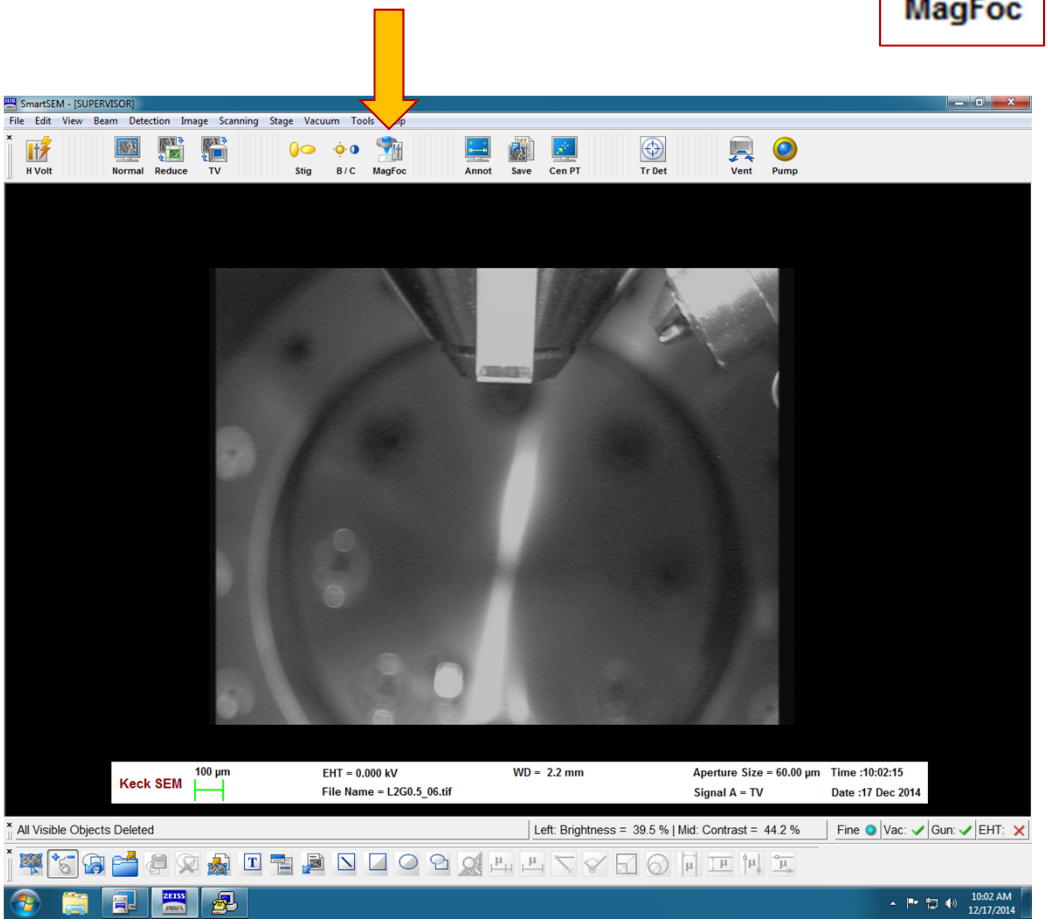
To adjust Contrast click on the middle mouse button and move the mouse left or right.



3.55

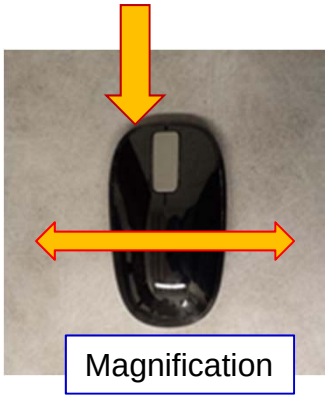
How to set Magnification and Focus

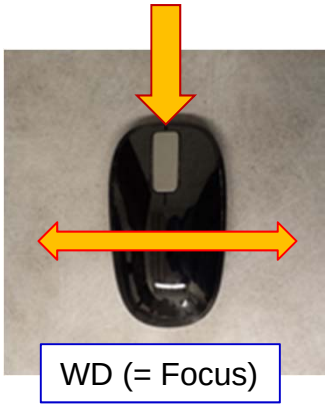
Click on the Magnification / Focus icon



To adjust Magnification click on the left mouse button and move the mouse left or right.

To adjust Focus click on the middle mouse button and move the mouse left or right.

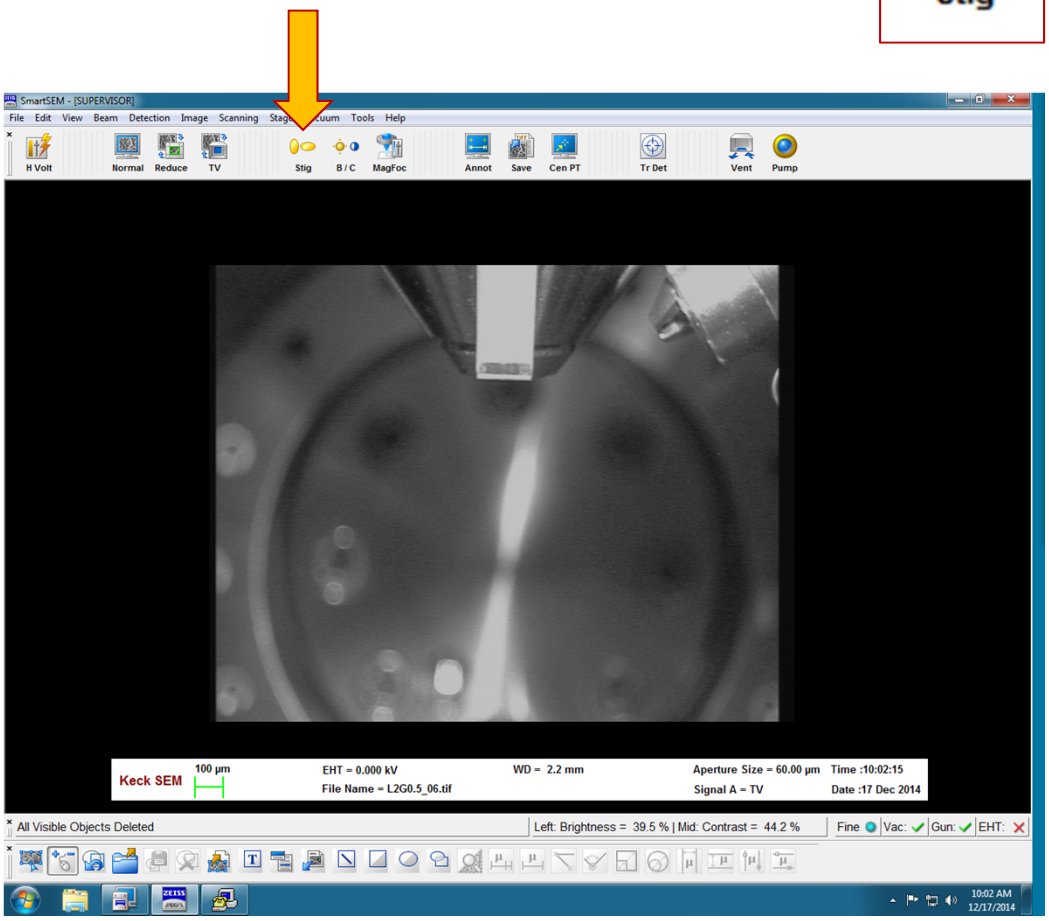




3.57

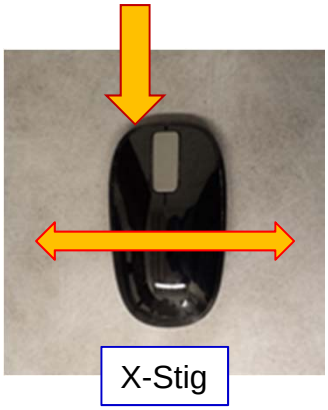
How to correct Stigmatism

Click on the Stigmator icon



To adjust stigmatism in the X-direction click on the left mouse button and move the mouse left or right.

To adjust stigmatism in the Y-direction click on the left mouse button and move the mouse up or down



WD (=Focus)

3.57

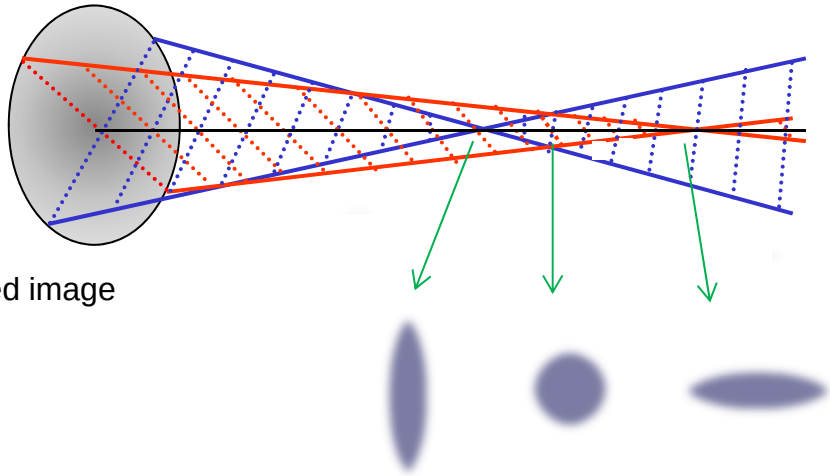
How to correct Stigmatism:

Use the middle button (focus) the go in and out of focus.

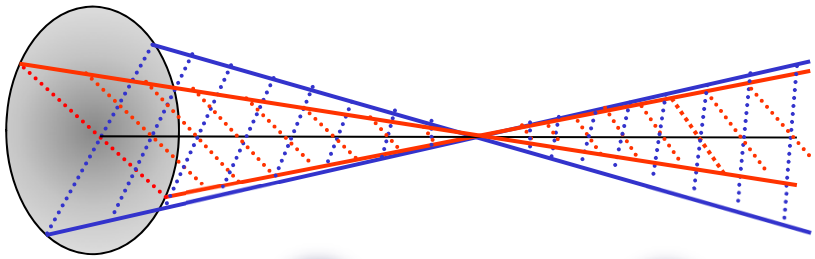
If the image stretches to a line on one side of the focal point and then stretches perpendicular to that line on the other side of the focal point, the image is stigmated.

Use the left button and move the mouse left and right and/or up and down to form a symmetrical (but not necessarily focused) image.

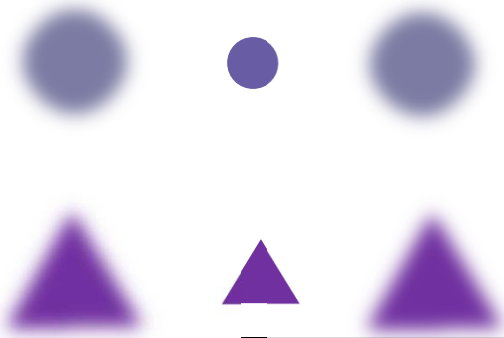
Refocus then check stigmation again.



Stigmated image

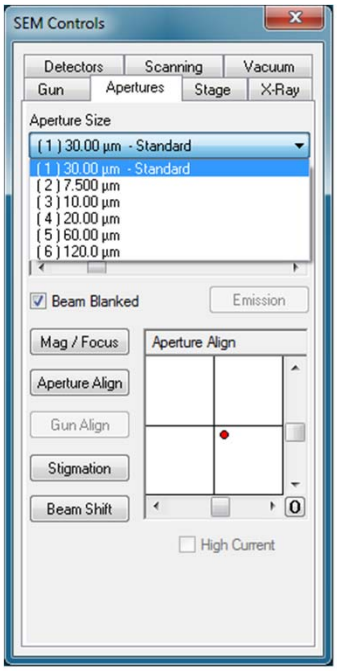


Corrected image.



3.60 How to choose and center an aperture

Click on the Aperture tab and select an aperture

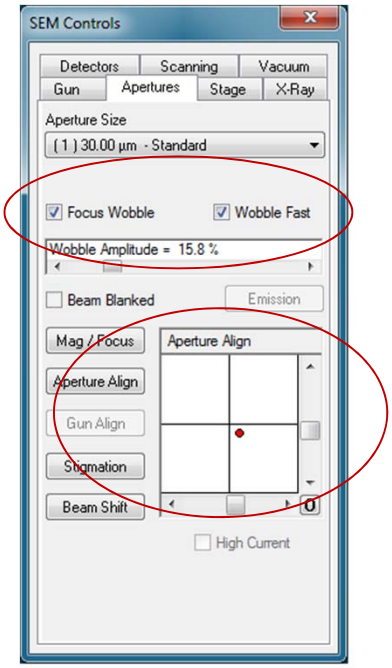


General aperture guidelines:

10um
Best for insulating samples, beam sensitive samples, or high depth of field:

20um
30um
Best for high resolution imaging and general purpose imaging

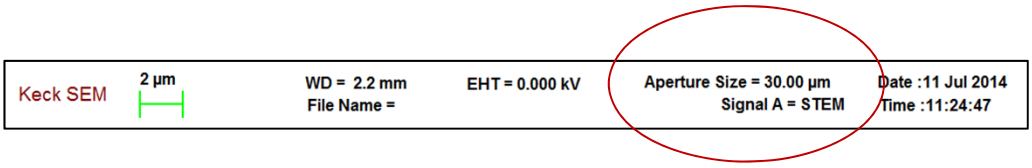
60um
120um
240um
Best for high current applications such as x-ray analysis:



Wobble-center your aperture by clicking on Wobble and using the graphic box to move your aperture to minimize wobble.

3.70 *How to choose a detector*

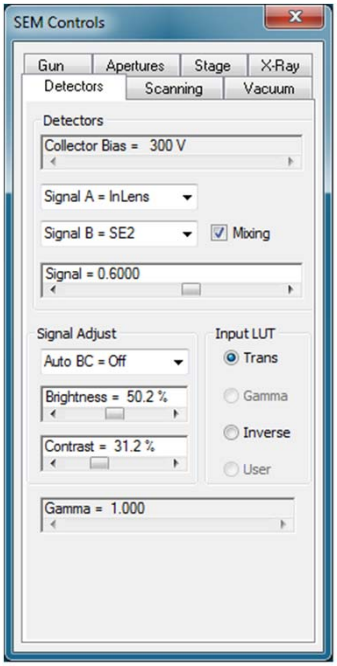
The detector is selected from the Data Zone by clicking on "**Signal A =**"



From the drop-down menu select the detector you want to use.

- The In-Lens detector is generally the best choice for high resolution imaging.
- The SE2 or Side-angle detector generally produces images that more accurately represent the 3-D surface of your sample than the In-Lens. However the SE2 signal to noise is lower than the In-Lens at short working distances.

Alternately, click on the Detector tab. Here a single detector can be selected (mixing un-checked) or a pair of detectors can be selected (mixing checked). An example is shown at left:



<u>Signal A = InLens</u>
The primary signal (A) is the InLens detector.

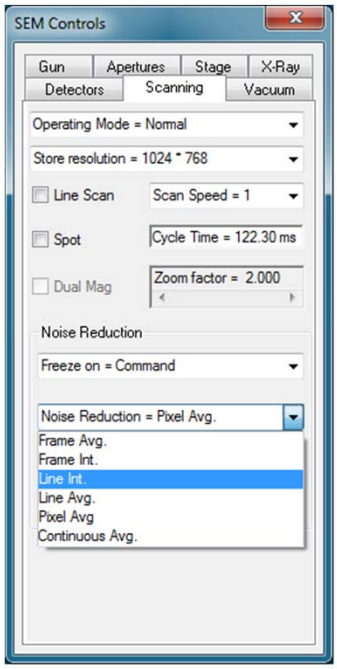
<u>Signal B = SE2</u>
The secondary signal (B) is the SE2 detector.

<u>Mixing</u>
If selected, this allows you to mix two signals.

<u>Signal = 0.6000</u>
This determines how much of the total signal comes from Signal A (balance comes from Signal B).

In the window at left, 60% of the total signal is from Signal A, 40% from Signal B

3.75 How to collect an image



Select either Line Int. or Frame Int. and select how many lines or frames you want to integrate (N value).

Noise Reduction Options

Frame Int Produces a final image by integrating N number of frames

Line Int Produces a final image by integrating N number of lines

3.80 How to make on-screen measurements

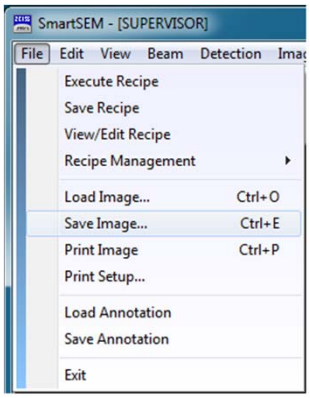
Click on this icon to enable on-screen measurements

3.85

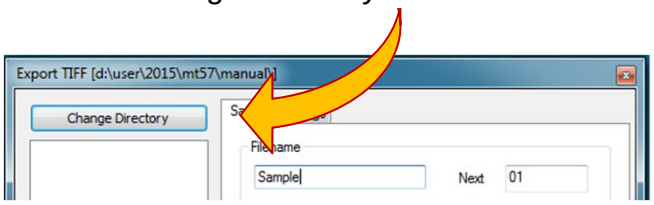
How to save an image

To save the first image from a sample:

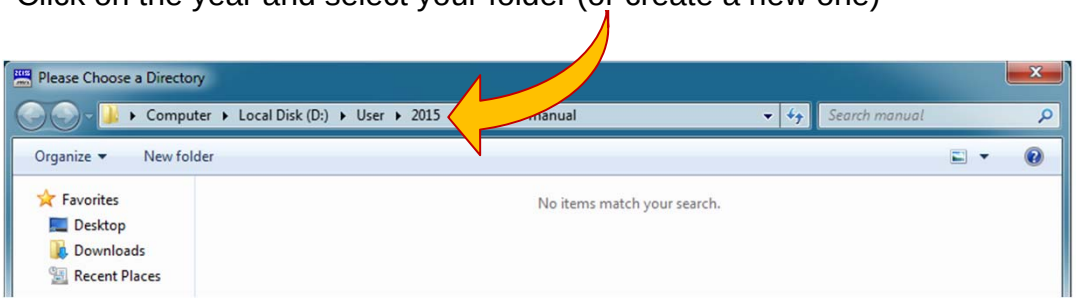
Click On File -> Save Image



Click on Change Directory



Click on the year and select your folder (or create a new one)

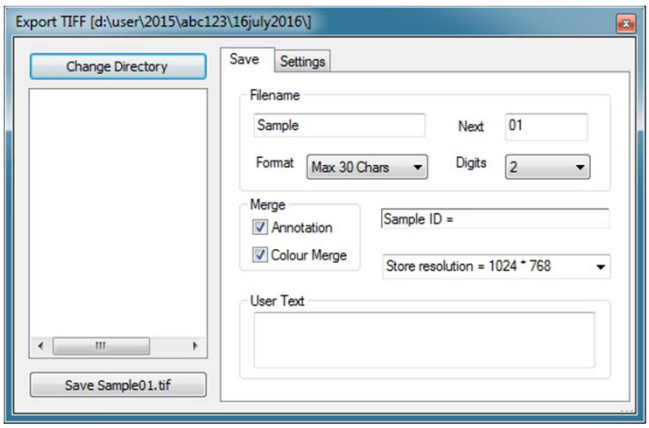


3.85

How to save an image (continued)

After selecting your folder click on **Select Folder** at the bottom of the window

The following window will appear:



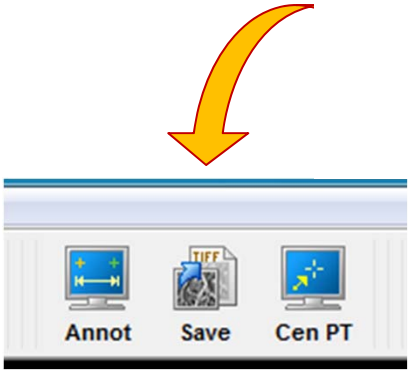
Click in the box below **Filename** and type in your new filename

Cilck on **Digits** and type “2” or “3”

Click in the box to the right of **Next** and type “01” or “001” as appropriate

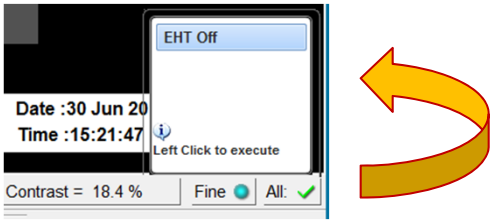
Click on **Save**

Foe subsequent images of the same sample simply click on **Save**



4.00 *How to turn off the SEM and end your session*

Turn off the EHT



Note the icon to turn off the EHT says "**All**" now, not "**EHT**"

Wait for the EHT to ramp down.

Click on the Vent Icon

Answer the questions in the dialogue windows

The stage will automatically go to the correct position

The column will vent

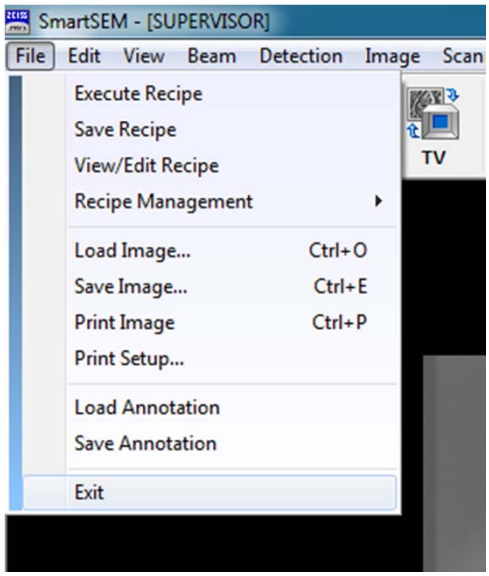
4.00

How to turn off the SEM and end your session

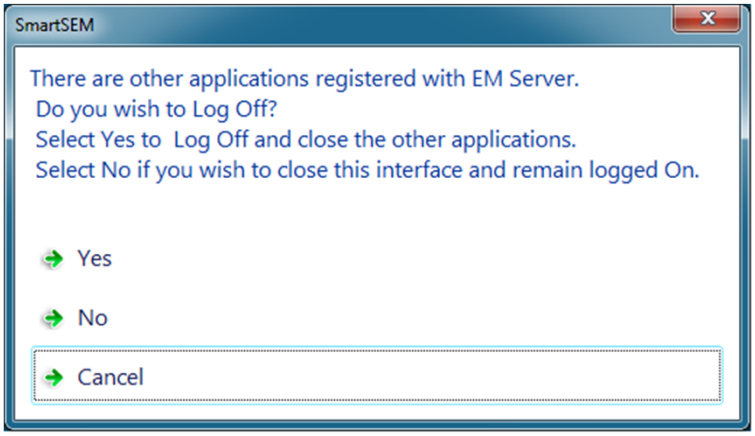
Critical – You MUST pump the chamber back down when you are finished – do not leave it up to air!

Hold the chamber door shut and click on Pump

AFTER the beeping has stopped AND the roughing pump has gotten quiet it is safe to exit SmartSEM.



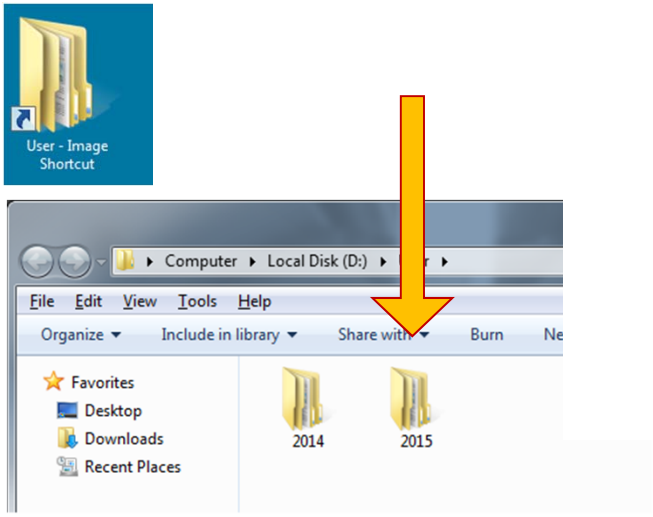
Click on File -> Exit



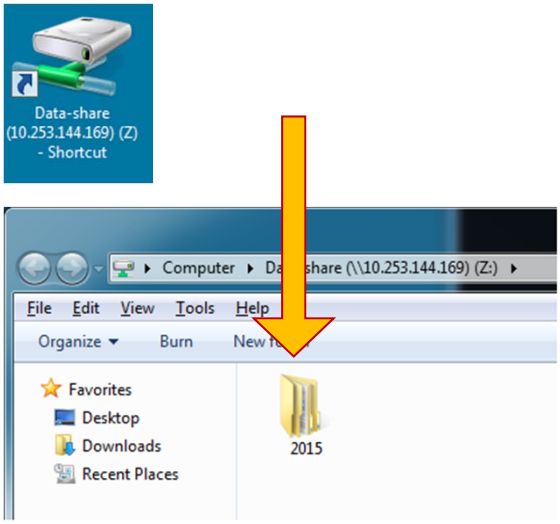
Click on Yes

5.00 *How to transfer your data*

Click on the **User-Image Shortcut** and select the appropriate folder



Click on the **Data-share Shortcut** and select the appropriate folder



Copy your data from the **User-Image** folder to the **Data-share** folder

Using **Cornell Drop-box** transfer your files

6.00 Shortcuts

<**CTRL + A**> Switches Annotation panel ON

<**CTRL + B**> Display Toolbar View dialog

<**CTRL + D**> Toggle Data Zone ON/OFF

<**CTRL + E**> Calls the Export TIFF dialog

<**CTRL + F**> Starts Auto Focus fine

<**CTRL + SHIFT + F**> Starts Auto Focus coarse

<**CTRL + G**> Switches SEM Control Panel ON

<**CTRL + I**> Switches SEM Status Panel ON

<**CTRL + M**> Switches to Annotation and inserts Point to Point Marker

<**CTRL + O**> Calls the Import TIFF dialog

<**CTRL + P**> Performs the Print Image function.

<**CTRL + S**> Performs Auto Astigmatism Correction

<**CTRL + SHIFT + S**> Performs Auto Astigmatism Correction with Auto Focus

<**CTRL + T**> Calls Text Annotation

<**CTRL + V**> Displays the Vacuum status data

Keypad <**+**> Faster Scan

Keypad <**-**> Slower Scan

<**SHIFT**> and double click performs Center Point function

6.00 Shortcuts

<**F2**> Toggles Tool Bar on/off

<**F2 + SHIFT**> Hysteresis removal

<**F3**> Closes all windows except the Tool Bar and Status Bar
<**F3 + SHIFT**> Toggles PC Plane ON/OFF

<**F4**> Step to next Magnification Table entry or Undo Center Feature Mag.

<**F4 + SHIFT**> Exit from Magnification Table mode.

<**F9**> Keys help (displays this information)

<**CTRL + 2**> Loads 2nd image window from display

<**TAB**> Toggle coarse/fine

<**CTRL + TAB**> Performs Center Point

<*> Performs Find Image function.

<**HOME**> Resets Beam Shift to zero.

<**PAUSE**> Causes currently executing macro to pause

<**SCROLL LOCK**> Toggles Freeze/Unfreeze

<**CTRL + SHIFT + TAB**> Performs Center Feature

<**F11**>, <**F11 + SHIFT**> User defined macros

<**F12**>, <**F12 + SHIFT**> Aborts Stage Movement.

<**F5**>, <**F5 + SHIFT**> User defined macros
<**F6**>, <**F6 + SHIFT**>
<**F7**>, <**F7 + SHIFT**>
<**F8**>, <**F8 + SHIFT**>

7.00 *Abreviated Instructions*

- Log into Coral then SmartSEM
- Vent the chamber
- Load samples
- Pump down chamber
- Raise stage to appropriate distance (about 5mm to start).
- When chamber vacuum is less than 2x10 -5 mbar and gun chamber is less than 5x10-9 mbar then turn on the EHT.
- Select an aperture
- Set magnification to its lowest value
- Adjust Brightness and Contrast to see something
- Focus on sample
- Wobble aperture to minimize motion in the image
- Zoom in and correct stigmatism.
- Take image
- Save image
- When finished turn off EHT
- When the EHT has ramped down go to TV mode and click Vent
- Remove sample(s) and pump SEM back down
- Log out of SmartSEM
- Burn/Transfer data
- Log out of Coral