Philips XL 30 SEM Standard Operating Procedures

UC Davis PEK ver 7/7/11 6.7

1. **Check that vacuum in ON** at left panel below the microscope column:
   A. Red OFF button is lit.
   B. White Vac button is lit.
2. **Start Microscope control software:** Double click on the XL Microscope Control icon to start the software.
3. **Load Sample:**
   A. Open the N2 valve (CW to straight forward) behind the microscope.
   B. Click on "VENT" in upper right hand corner of screen (usually takes ~5 min to vent).
   C. Slide the chamber open and place the samples as desired.
   D. Check Z with the provided tool and adjust using the manual Z adjust knob on the chamber door.
   E. Close the chamber and click on "PUMP" in upper right hand corner of screen and wait for vacuum to reach the ~10^{-5}mbar range. This usually takes 3-5 minutes.
4. **Set Microscope Controls:**
   CAUTION: Do NOT change Z value until image is in focus. Damage to microscope will occur!
   A. X, Y and µm R: change to 0.0 and click on "Go To" button. NOTE: Stage will home to beginning (0) positions.
   B. On Main Menu Bar Settings:
      a. Beam > Choose kV (usually 10kV)
      b. Beam > Spot Mag. (3 usually works best for most work).
      c. Filter > Live and Hi def.
5. **Turn on high voltage and begin scanning sample as follows**:
   A. Press the white HT button on the console below the column of the microscope.
   B. Mouse click on the 10kV (or other chosen kV) in the upper right hand side of the screen.
      NOTE: Filament will saturate and image will begin to become visible.
   C. Adjust Contrast and Brightness with sliders on the right hand side of the screen as needed.
   D. In the log book, write down the kV used and resulting beam current (µA) in the appropriate column.
6. **Adjusting the Working Distance and Z:**
   A. **To focus**, hold down right mouse button and drag mouse left or right across the image. Focus image at >3,000x (type of specimen will determine magnification).
      a. To elicit the selected: In the main menu click on the Selected Area ( ) icon: Focusing aid.
         NOTE: This renders a small area (selected) thus giving a faster scan for fine focusing. Focusing box may be moved to desired area or resized for smaller or larger area. Click on icon to return to full scan (Options are also accessible through the main menu.)
   B. Check that WD (working distance), located at the bottom left hand side of the screen under WD, is the same number as the Z in the bottom right hand corner of the screen.
   C. There should never be more than a 0.5 difference between WD and Z. Check this every time magnification and/or location is changed.
   D. If there is a difference greater than 0.5 proceed as follows:
      a. Focus the image then press the **–Z<->FWD** button located at the bottom right of the screen.
      b. Press the Z drop down menu and select 10.
      c. Press the **GO-To** button located just right and down from the Z button.
7. **Optimizing the image:**
   A. Moving the Stage: There are four ways to move the stage:
      a. Using the X & Y knobs on the chamber door
         CAUTION: Do NOT move the Z by mistake!!!
      b. Click on the Track Mode icon ( ) in main menu. Click mouse in desired direction to move or use the arrow keys to move 1 field.
      c. Click on the Get Mode icon (+) in main menu: Place cross over the object to be centered and double clik the mouse left-hand button,
      d. Click on the Shift Mode icon ( )
   B. **Spot Size** (In main menu: Beam> Spot Mag)**:**
      NOTE: If a larger spot than needed is used, resolution is lost.
      < 2000x Use spot 5
      2000-4000x Use spot 4 (smaller spot)
      >5000x Use spot 3
   C. **Astigmatism Correction:** If the image "smears" in any direction when focusing, there is an astigmatism in the objective lens and must be corrected to insure a good image.
      a. Hold down the "Shift" key as you hold down right mouse button and drag mouse left or right across the image.
      b. Alternate between focusing and astigmatism correction until smearing is eliminated.

8. **Magnification:** There are 3 methods for changing magnification:
   a. Keyboard controls: "+" key doubles magnification; "-" Key halves magnification.
   b. Keyboard controls: Arrow keys move the specimen one page (screen)in the direction of chosen arrow.
   c. In the Main Menu: Go to Mag > select mag (if you go to "change" you may input desired magnification).
   d. Select Imaging Icon ( )
9. **Scanning Speeds**:
   a. TV or Scan Speed 1: Fastest scan rates, poorer image; Good for fast scan of sample.
   b. Slow Scan Speeds 2-Photo Scan : The slower the scan rate, the better the image but trade off speed.
10. **Saving Files:**
   A. If necessary, create folder: GoTo My Computer > C > XL > user ; create a new folder. (As many sub folders as necessary may be created). Path for saving files should be C:/xl/user/User's folder/User's file name.
   B. Set up Data Bar format as follows:
      a. GoTo In/Out > Data Bar (Data Bar Setup window opens).
      b. Type in desired information: User Name; Experiment Number (if desired).
      c. Display: OFF – Turns OFF Data Bar; Photo - Displays Data Bar on Photo; Real Time – Displays in real time.
      c. Select (check boxes) the information to be displayed on the image and print: **AccV** = Accelerating Voltage; **Spot** = Spot Size; **Magn** = Magnification; **WD** = Working Distance; **Exp** = Experiment number; **Background** = Dark background mask behind data display on image.
   C. To Freeze Image:
      a. GoTo SCAN >Slow Photo Scan.
      b. Immediately GoTo FILTER > Integrate 1.
      NOTE: The Snowflake Icon in the main menu turns yellow when ready to proceed.
      c. GoTo IN/OUT > Image (save dialog box opens).
      d. Direct the path for saving files (only needs to be done for the first image in session) by double clicking on the following: [..} > Previously named folder.
      e. Name file (8 characters + 3 file extenuation characters (i.e. yourfile.tif).
      f. Click on **Save.**
      g. Unfreeze by clicking on the Snowflake icon in the main menu.
      g. Change Scan Speed back: SCAN > Slow Scan 1.
11. **Shut down of microscope:**
   A. For a 10-15 minute break, click on the beam blanking icon in the main menu ( ).
   B. For a lunch break ,turn OFF the beam by click HT on the control panel.
   C. At the end of session, proceed as follows:
      a. Click the **HT** video button **OFF**.
      b. Press the **HT** button under column to **OFF** (button not lit).
      c. **Allow filament to cool 1 minute** before proceeding and insure that the N_2 tank is open.
      d. Press the **VENT** video button to vent the chamber. "Are you sure?", click "yes".
      e. Remove samples from chamber.
      f. Click on the **Pump On** to pump out chamber and wait until chamber is pumped out before leaving.
      g. Close the N_2 tank.
      h. Make entry into log book.
12. **Make CD of images:**
   A. Insert New CD and select "Roxio" icon on desktop and follow the instructions:
      a. Select Make a Data CD.S
      b. Find desired files: c: > xl > User > locate your files
      c. Click and drag files to window.
      d. When button to Burn is visible, press finish.
   B. Press the **BURN** button and wait until completed.
13. **Using the Backscatter Detector**
   A. Check that the Backscatter Detector is on the final lens (if not, check with lab staff).
   B. To switch to the Backscatter Detector, go to DETECTORS > BSE
      a. Adjust Contrast and Brightness as necessary
      b. If necessary, increase the signal by increasing the spot size; go to BEAM > SPOT (choose larger spot size-larger number).NOTE: Increasing the spot size will decrease resolution at higher magnifications
   C. To return to the secondary detector: Go to DETECTORS > SE.

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Short Version
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- Double click the XL Microscope Control icon to start the software.
- Open the N2 valve (CW to straight forward) behind the microscope.
- Click on "VENT" in upper right hand corner of screen (usually takes ~5 min to vent).
- Slide the chamber open and place the samples as desired.
- Close the chamber and click on "PUMP" in upper right hand corner of screen and wait for vacuum to reach approximately ~10-5mbar. This usually takes 3-5 minutes.
- Beam > Choose kV.
- Beam > Spot Mag. (3 usually works best for most work).
- Filter > Live and Hi def.
- Press the white HT button on the console below the column of the microscope.
- Mouse click on the chosen kV in the upper right hand side of the screen. Filament will saturate and image will begin to become visible.

  DO NOT CLICK "OK" IN BOX UNTIL YOU DO THE FOLLOWING:
  - Focus image then click on OK in box .
  - Press the Z drop down menu and select 10 then click on "Go To"
  - Adjust Contrast and Brightness with sliders on the right hand side of the screen as needed.
  - Focus, hold down right mouse button and drag mouse left or right across the image.
  - Check that WD (working distance), located at the bottom left hand side of the screen under WD, is the same number (+/-0.5mm) as the Z in the bottom right hand corner of the screen.

Saving Files:

- **Create folder**: YOU ARE LIMITED TO 8 Characters, no spaces, no special characters!
  - My Computer > C > XL > user ; create a new folder. (As many sub folders as necessary may be created). Path for saving files should be C:/xl/usr/User's folder/User's file name.
- **Data Bar**:
  - In/Out > Data Bar (Data Bar Setup window opens).
    Type in desired information
- **Freeze Image**:
  - SCAN >Photo Scan
  - FILTER > Integrate 1.
  - NOTE: The Snowflake Icon in the main menu turns yellow when ready to proceed.
  - IN/OUT > Image (save dialog box opens).
  - Direct the path for saving files (only needs to be done for the first image in session) by double clicking on the following: [..} > Previously named folder.
  - Name file (8 characters + 3 file extenuation characters (i.e. yourfile.tif).
  - Click on Save .
  - Unfreeze by clicking on the Snowflake icon in the main menu.
  - Change Scan Speed back: SCAN > Slow Scan 1.
- **At the end of session:**
  - Click the HT video button OFF.
  - Press the HT button under column to OFF (button not lit).
  - Allow filament to cool 1 minute
  - Press the VENT video button".
  - Remove samples from chamber.
  - Pump On to pump out chamber and wait until "VACUUM OK" **SS**
    DO NOT LEAVE UNTIL VACUUM OK IS DISPLAYED!!!
  - Close the N2 tank.
  - Make entry into log book.
- **Make CD of images:**
  - Insert CD and select "Roxio" CD creator
  - Select Make a Data CD
  - Find desired files: c: > xl > User > locate your files
  - Click and drag files to window. Follow instructions.