

Note: This guide is NOT complete operation manual. It is a quick reference for daily operation.

### Remember:

1. **In emergency, close gun valve by pressing the “Beam” button at the upper-left corner of left control panel.**
2. Check conditions of the ARM-300.
  - o JEOL PC TEM Center (middle): Vacuum System (VAC), V1=OFF, V2=OFF, PIG1<35, Specimen Chamber (not ready) PIG4~255, PIG3<45, PIG5<65. **Specimen Vacuum <math>3 \times 10^{-5}</math>Pa** (JEOL-PC). It is OK if Column Pipe/ TMP Pipe are not ready.
  - o JEOL PC: Acc.= 300kV, TEM\_S, X=Y=Z=0, TX=0, TY=0.

### START-UP

1. Fill LN2, wait one minute till boiling then top off LN<sub>2</sub> again. (Take out ACD heater if you are the 1<sup>st</sup> user of the day)
2. Log in Access Control on NUCore.

### LOAD SPECIMEN

1. Put holder on sample loading station, with appropriate holder support lying underneath hold tip.
2. For single tilt holder, loosen two screws TWO turns, rotate the cover plate away and mount specimen with film side UP. For double tilt holder, loosen two screws TWO turns, rotate the two plate-clamping fingers and mount sample facing DOWN.  
**NEVER OVERTIGHTEN THE SCREWS!!!**
3. Check O-ring and clean with duster if necessary.
4. Align holder guide pin with the guide groove on the goniometer, push till it stops (**DO NOT TURN!**).
5. Keep pushing the holder in position, push the PUMP switch on goniometer. For double tilt holder, put the cable across the goniometer cover to prevent rotation of sample holder due to cable weight.
7. **Wait (DO NOT TURN!) until the green LED on goniometer turns on and the PIG4 reading is below 39.**
8. Turn holder clockwise and insert specimen holder into the goniometer. Try to insert smoothly and gently. Never use force sideways!
9. Check the column reading: wait till column pressure (read as *Specimen Vacuum*) is below  $4.5 \times 10^{-5}$ Pa.
10. JEOL PC: in “*Specimen*” window: select holder: single tilt holder, *Reinforced Be holder*, or other specific holders.

### TURN ON EMISSION (cold FEG)

1. Open “*Emission Controller*”, if you are the FIRST USER of the DAY, Click on Flash “High”; otherwise, click on Flash “Low” to flash the gun. When Flash is over, click on Emission ‘ON’ to turn on emission. Click on *HT* and *Emission Monitor*, to, write down A1, A2, emission current and high flash number on the sheet.

### FIND BEAM

1. Check emission current (~15 μA).
2. Open Gun Valve: press “*Beam*” button at the upper-left corner of left control panel.
  - o Dim or turn off room light. Remove cover of viewing chamber. Decrease **MAGNIFICATION** to see if beam appears.
  - o Press **LOW MAG**. If you see the beam, move a hole on your specimen to the center of the screen. Focus the beam to small spot through the hole on the sample; go back to **MAG** mode. Adjust sample position and BRIGHTNESS to find the beam.
  - o Call for help if you still cannot find the beam.

### TEM ALIGNMENT

1. Make sure the operation mode is TEM mode (TEM Center).
2. Check/Perform Gun tilt alignment. At 40xK, in “Wobbler Controller”, click **Anode**. In “Alignment Panel”, click “GUN ALIGN”. Adjust DEF-x and y to have an uniformly expanded beam.
3. Check/Perform Gun shift alignment. At 40xK, use spot selector, center beam at spot 1 by Gun Shift (“Alignment Panel”, click “GUN ALIGN”), and at spot 5 by Beam Shift (“Alignment Panel”, click “Beam ALIGN”).
4. CHECK Condenser lens (C2) Aperture centering:  
Change **BRIGHTNESS** largely back and forth to see how the beam image changes.  
If the beam spread and shrink like fig 3b and c.
  - a). Condense the beam to the cross-over, center it by using **Beam Shift**
  - b). Spread the beam out by turning **BRIGHTNESS** knob clockwise;
  - c). Select C1 and the arrow-buttons to center the beam.
5. Check condenser lens stigmatism: if beam is not round, press Cs-STIG (left panel) and use DEF-x and y to adjust (while adjusting brightness).
6. Find Specimen and set up eucentric height: **double-press** STD focus (right panel). Press the ‘Wobble-x’ (right panel) and adjust specimen height (Z+ and Z- buttons on the specimen consol) to minimize the movement of the image.
7. Beam-tilt alignment: Find a sharp edge of the sample, press the “HT Wobbler” (right panel) button, press “Beam ALIGN” and use the DEF-x and y to minimize the translation movement of the image.



### TEM imaging with the Gatan OneView camera

1. Gatan PC (Left): Start GMS software if they are not started. Right click Camera Icon and select ‘Insert’.
2. Select TEM-Imaging module, and image mode (Imaging or Diffraction can be selected).
3. Press “Screen” button (right panel) to lift up the screen;

4. Check the exposure time (start with shortest exposure time firstly) and binning, click on “View” to have the image in GMS. Adjust brightness and focusing if necessary.
5. Check objective lens stigmatism: find area of interest, adjust focus and stigmator. At higher mag (>100kx), you may use Process-Live- FFT to check focus and astigmatism.
6. To save image in dm4 or dm3 format, go to File menu – Save. You may temporarily save your file in MyDocuments\Users folder during experiment. After experiment please MOVE your files to TEMServer (Z:\) under folder with your own name. Any file saved in public folder will be deleted right away. MyDocuments\Users\ folder is also dumped regularly.
7. Check exposure time in Camera Acquire t and click Acquire. To use manual exposure, uncheck auto exposure. Drift correction option can be turned on.
8. To save image in DM3 format, go to File menu – Save As.
9. To save image in other formats, choose Save As.

### Selected Area Diffraction (SAD)

1. Center area of interest to center of screen. Spread beam with Brightness knob. You can use C1 Aperture 3 or 4 to reduce the beam brightness for electron diffraction.
2. Insert the SA aperture: press SA and desired size number (left panel).
3. Press SA DIFF button (right panel) and spread beam to have smallest transmitted spot.
4. Use MAG knob to adjust camera length.
5. If the transmitted spot is not at center of screen, press PLA button (left panel) and use DEF X and Y knobs to center it.

### STEM mode Alignment

1. Click **STEM** to go to the STEM mode, wait for lens relaxation.
  2. Adjust Condenser lens Stigm: click “Ronchigram” to freeze the beam; use small screen and binnacles to find the Ronchigram. Center ronchigram to center of screen with PLA DEF knobs. You may need to move sample around to locate an amorphous area and selected a large C1 aperture to see it. Set camera length to at least 80 cm (or 100 cm), use OneView camera to have a large view of the Ronchigram. Once you have a clear Ronchigram, adjust “Cond STIG” (left panel press-to-lit and use DEF-x and y) to make it as circular as possible.
- The other option is go the high magnification, by adjusting Stigm to make the image more clear and in focus, like what we do on a SEM.*
3. Center Ronchigram (to the center of small viewing screen) using PLA + DEF/STIG, correct astigmatism using Cond. STIG + DEF/STIG.
  4. Insert appropriate CL2 aperture (#2-3 typical) and center it to the center of Ronchigram.
  5. Select detector: in the STEM window, there are four detectors: “ADF1”- high angle annular dark field detector; “ADF2”- medium angle annular dark field detector; “BF”-bright field detector, “ABF”- annular bright-field detector.
  6. Start Scan to view a STEM image: use Digiscan in GMS, DigiScan – Search or Preview – Start, (or the STEM imaging in JEOL STEM Center). Adjust **Brightness** and **Contrast** knobs to have appropriate B/C. Select appropriate probe size (probe 8-10 for high resolution, probe 4-5 for EDS), and camera length (8 cm typical).
  7. Set up the Eucentric height: STEM has different Eucentric height than TEM; Find sample and Adjust Z-height to get a clear STEM image.
  8. Adjust fine focus (right panel) and click DigiScan – Record to record STEM image. If necessary, click DigiScan setup button at lower right corner of the tab to change parameters for search/preview/record and select different detector.
  9. Click TEM to go back to TEM mode after using STEM.

### EDS Spectra collection and mapping analysis

1. On the Oxford computer (right), open the software: AZtec; select your Project or set up a new one.
2. Find the EDS detector icon; Cool it down (it may take ~3 minutes). Position control: insert the detector.
3. Insert the EDS, HX aperture.
4. There are then several modulus in the Aztec: to acquire a spectrum, to do line-scan and mapping.
5. Drift correction: in setting, click Auto (default is off).
6. Save your data and copy the data to the TEM\_Server.
7. Take out the detector and stop cooling once you are done.

### Change specimen and shutdown

1. Remove any aperture that was used during your session except the CL aperture #2.
2. Go back to TEM mode.
3. Turn off Beam valve (left panel).
4. JEOL-PC: – Double click black **StageNeutral** button to neutralize sample position.
5. In **Emission** window, click on “Emission Off” to turn off emission.
6. Make sure sample shifts and tilts are zero, then remove specimen holder from the microscope.
7. Load new specimen if you want,
  - a. If you are not the last user of the day, follow this procedure: Refill LN2; Logoff your session in NUCore.
  - b. If you are the last user of the day, follow this procedure: 1-Plug in ACD Heater; 2-JEOLPC: TEM-Maintenance Menu – ACD and Bake to open ACD Heat window; 3-Turn ACD Heat On. 4-Logoff your session in NUCore.