

Hitachi H7800 Operating Instructions

HRTEM Standard Operating Procedure

BEFORE WORKING

1. Using a pen, log the appropriate date, account number, and any comments into the logbook while paying attention to the counter number from the previous user. This number should correspond to the number located in Filament/Bias Control panel on the computer screen. If this number does not match, please seek assistance.

2. **Fill the two (2) TEM dewars with liquid nitrogen (LN2). The large and small dewars are located in the rear and front of the TEM, respectively. If you are having trouble locating or filling the dewars please seek assistance. Once the dewars are filled, allow five (5) minutes before beginning so that the TEM may cool.**

3. While waiting for TEM to cool, turn on both computer monitors.

4. Assure that Objective Aperture is out. Located on the left control panel (LCP), the Aperture IN/OUT should be unlit. (**NOTE:** For the purposes of this document, “out” or “off” will be always be indicated by an unlit light/button).

BASIC PROCEDURE FOR ALL TEM USERS

I. INSERTING THE SAMPLE

1. Remove sample holder from specimen stage and place on specimen platform.

2. Using the tweezers provided at the TEM station, carefully pull back the screw along the top of the sample holder which releases the specimen cover. Use extra caution when handling the specimen cover. Carefully load the copper grid onto specimen holder. When seated properly, place specimen cover atop copper grid and slowly release the screw to lock the sample in place.

3. Perform the “Shake/Tap” test by rotating the sample holder while seated in the specimen platform and gently tap. If the sample is not seated properly within the sample holder the copper grid will dislodge.

4. Once sample has been properly loaded within sample holder, check the O-ring for any debris, cracks or other items that could cause a vacuum leak. If the O-ring has been compromised or has loose impediments or cracks please seek assistance.

5. If the O-ring is free of defects and debris, insert sample holder in specimen stage carefully observing the keyway. Once introduced into keyway, a micro-switch will activate. This will specify that the sample has now being housed in the second détente.

6. Flip the switch located on the specimen stage to EVAC. **WAIT UNTIL THE GREEN LIGHT TURNS ON!!!**

7. Immediately after green light has illuminated, turn sample holder clockwise (CW) 45° and allow the newly created vacuum to *pull* the sample holder to the first détente. This position is known as the standby position. If this step is not performed immediately, flip the switch from EVAC to AIR and repeat process.

8. Once in the standby position turn the sample holder counterclockwise (CCW) 45° to lock.

□ **II. AXIAL ALIGNMENT OF ILLUMINATION SYSTEM**

1. Check if High Voltage (HV) on left control panel (LCP) is “on” or “lit.” If not, ask for assistance.
2. Press PDS Icon located on the Hitachi Tool Bar. There are currently three (3) protocols that have been created for use on the TEM. Refer to the chart below for an explanation regarding the difference between each PDS file.

HC @ 100kV	0.7k – 200k magnification
HR @ 80kV	4k – 600k magnification (For use in beam sensitive samples)
HR @ 100kV	4k – 600k magnification

NOTE: HC = High Contrast, HR = High Resolution

3. Ensure that the lens mode on the left control panel (LCP) is in Zoom1 and that the alignment located on the right control panel (RCP) is in BH mode. Using a magnification of 4k or less, center the beam to the smallest spot using the Brightness Knob located on the LCP. If for some reason the beam is not present, open the BD Control Panel and click GH (Gun Horizontal) (DO NOT click the “check” box, but rather click on the icon itself). When GH has been enabled, use the X,Y adjustment knobs to locate the beam. While still in GH mode, again center the beam using X,Y knobs.
4. Select BH (Beam Horizontal) mode from RCP and center the beam using the X,Y knobs.
5. To maximize beam brightness, open the BD (Beam Detection) Control Panel and “click” GT (Gun Tilt) (again, not the check box, but the icon itself) and manipulate X,Y until maximum brightness has been achieved.
6. Return to BH mode by pressing BH on RCP and close BD Control Panel.
7. Bring beam to crossover with the Brightness control knob located on the LCP. During this step it is also necessary to re-center the beam. This is performed by using the alignment or X,Y knobs. (**NOTE:** Make sure that you have selected BH from the BD Control Panel).
8. If required, adjust the Condenser Lens Movable Aperture so that the beam spreads evenly to the edge of the screen.
9. Ideally, the beam should also be round. The roundness of the beam is most easily seen at approximately 4k magnification. If the beam is not round, “click” CS (Condenser Stigmation) on the RCP. Slowly manipulate the X,Y knobs in such a manner that the beam becomes round.
10. When beam is spherical, remember to re-center the smallest spot size of the beam using the X,Y knobs while in BH mode.
11. Once re-centered, “click” IA (Intermediate Aperture) on the BD Control Panel. Adjust from lowest magnification at 700k - 30k when in high contrast (HC) or 4 - 60k when in high resolution. Then, re-center beam to highest magnification using IA. When centered, return to BH mode and close BD Control Panel.
12. Move the sample holder which is currently in the standby position, the beam path by turning the sample holder counterclockwise (CCW) 45°. Locate an image by moving the stage knobs on both the LCP and RCP. (**NOTE:** In some cases one may need to change the lens mode to LOW MAG to easily locate the sample. However, this should only be

performed on a case by case basis).

13. Increase the magnification between 15k – 20k and press WOB on the RCP. In most cases this will cause the sample to pulsate on the screen.

14. From the LCP, select SPOT. Focus the beam on the spot mirror. Using the binoculars, cause the image to be motionless using the Z Control Knob which is located on the underside of the right assembly. Use the X,Y knobs on the LCP and RCP to keep the image on the spot screen and focus the image. Once aligned and focused, the image will no longer appear to pulsate. When aligned and focused, press WOB (Wobbler) to deactivate objective stigmatism (OS). (**NOTE:** The X,Y knobs are very sensitive to change when using WOB and/or MODU (Modulator). Make very small adjustments until aligned and focused. For novice users it may be beneficial to adjust X,Y knobs separately until comfortable with machine sensitivity until the sample image is the most stable on the screen).

15. Press MODU on RCP. Adjust modulator by using X,Y knobs. Once sample is focused and aligned turn MODU “off” and remove spot screen. When finished with this step also return to BH mode which is located on the RCP.

16. Fine focus the image using the Focus knob on the RCP.

17. Press IN/OUT button located on the LCP to insert the objective aperture. Doing so will cause an increased contrast.

18. Start AMT software which is located on the desktop labeled “AMT V540.” Once the software has initialized, “click” Camera Out to insert camera. Press the “Click for Live Image” icon located in the upper right quadrant of the AMT software.

19. Acquire a background. The software will prompt you with three (3) steps the first of those steps being retracting the specimen or finding an empty hole in the grid. The best method involves retracting the sample from the beam. This is performed by pulling the sample holder out and rotating it 45° clockwise (CW). This position is known as the standby position. Center the beam by adjusting the intensity to center the histogram. When centered place the sample holder back into the beam and “click” Proceed.

20. Find a suitable image. Adjust brightness.

21. When a suitable image has been discovered, “click” on Final Image.

22. Go to File/Save As and the appropriate caption, “click” Save with caption and select the proper folder to save file.

23. “Click” Live Image to continue.

III. CHANGING SPECIMEN SAMPLES

1. Move the specimen stage back to X,Y = 0,0 by opening the Stage Control Panel. This is performed by “clicking” Operate/Stage/List/Start. Close this window when finished moving stage back to X,Y = 0,0.

2. In the Filament/Bias Control window “click” Filament CURRENT which will turn the VOLTAGE OFF.

3. Pull sample holder straight out to first détente and turn clockwise (CW) 45° which is the standby position.

4. Pull sample holder out again to second détente and turn counterclockwise (CCW) 45°.

5. Flip switch to AIR. **WAIT FOR RED LIGHT TO TURN OFF!**

6. Once red light has turned off pull the sample holder out from specimen stage and exchange the specimen.

2. Using the tweezers provided at the TEM station, carefully pull back the screw along

the top of the sample holder which releases the specimen cover. Use extra caution when handling the specimen cover. Carefully load the copper grid onto specimen holder. When seated properly, place specimen cover atop copper grid and slowly release the screw to lock the sample in place.

3. Perform the “Shake/Tap” test by rotating the sample holder while seated in the specimen platform and gently tap. If the sample is not seated properly within the sample holder the copper grid will dislodge.
4. Once sample has been properly loaded within sample holder, check the O-ring for any debris, cracks or other items that may cause a vacuum leak. If the O-ring has been compromised or has loose impediments or cracks please seek assistance.
5. If the O-ring is free of defects and debris, insert sample holder in specimen stage carefully observing the keyway. Once introduced into keyway, a micro-switch will activate. This will specify that the sample has now being housed in the second détente.
6. Flip the switch located on the specimen stage to EVAC. **WAIT UNTIL THE GREEN LIGHT TURNS ON!**
7. Immediately after green light has illuminated, turn sample holder clockwise (CW) 45° and allow the newly created vacuum to *pull* the sample holder to the first détente. This position is known as the standby position. (**NOTE:** If this step is not performed within 10 seconds, flip the switch from EVAC to AIR and repeat process).
8. Once in the standby position turn the sample holder counterclockwise (CCW) 45° to lock.
9. Move the sample holder which is currently housed within the specimen stage into the beam path by turning the sample holder 45° counterclockwise (CCW).
10. Again, in the Filament/Bias Control window “click” Filament CURRENT which will now turn the VOLTAGE ON.
11. While the Lens Mode is in LOW MAG with Aperture IN/OUT decrease the brightness and locate sample.

IV. END OF SESSION

1. Ensure that the Aperture IN/OUT button is unlit.
2. Reset stage back to X,Y = 0,0 by “clicking” Operate/Stage/List/Start. Close this window when finished moving stage back to X,Y = 0,0.
3. Check to make sure that the specimen holder empty and in the standby position with the switch set to EVAC.
4. Magnification at x600k and enter total filament time located from the Filament/Bias Control window in the TEM logbook.

9/21/2007

Signature

Date

NEST Lab Signature

Date

