Short instructions for Quanta 650 –v14

Warnings

- ESC stopes the table!
- When inserting the holders make sure the samples are attached! Especially with powder samples. If it is not fixed it doesn't go in the SEM! Damage to the pump or image resolution.
- Be careful when moving the big table, use CCD to not damage the detectors
- It BD (Beam Deceleration) table is not mounted, do not turn it on! Dangerous when mounted in the station.
- Always remove Nav-Cam before closing the door.
- Always turn off the filament when switching vacuum modes!
- Use F1 manual. If you don't know ask CEMM.

COMPARISON JSM-7600F (x3.452)

JSM-7600F mag: 250x = 863x mag on Quanta 650
(xTM Preferences – Mag: Real screen size –Single image)

Image acquisition:

- Images are saved on (M-PC): Computer \ Shared Data (\\OXFORD... **USERS**\...
- Images collected (S-PC): D\Shared Data\USERS \ …
- EDS/S-PC can be stored in **USERS EDS**.
- Use "boksit" server and not USB sticks

Always save as TIFF (16 bit for B/W, 24 bit colors).
Photo F2 for one image, Shift+F2 multiple,
II unfreeze one, Shift +II unfreeze all.
Note: always have marked - data bar in ruler (a problem only with Center Cross turned on (Shift + F5)

Initial settings

- Green light ON on the microscope
- XT server on and UI on, a pop up window for log-in
- EDS inserted and cooled

SEM conditions: mag: 100x, 10 kV, spot 3, photo speed 30 µs (dwell time), square in MAG
Quads 1: ETD (SE), 2: CBSD (All), 3: Nav-Cam, 4: IR CCD (NAME: CEMM)

Scanning speeds: Scan – preferences – select Default (Photo 60µs) and press **Apply!**

Always check that »default« setting are on the digital post processing (digital brightness, contrast, and gamma).

SCAN for the manual:

Sample preparation

- Always clean the samples (air pump in the drawer).
- For easier work, all samples should be at the same height. Always link on the highest sample (CBSD!)

Starting (M-PC)

- Log in into the UI xT (U: user G: user)
- Inspect the CCD (IR), if anything wrong contact CEMM.
- Write the **vacuum**: XX·10^{-5} Pa (in the book)
- If any problems write to cemm@ijs.si

Inserting the samples in the chamber

- Vacuum green, beam off - press VENT on UI.
- Insert the samples in the table
- Gently close the door (do NOT press pump) set on 10 mm, for the Nav-Cam photo.
- Open the door.
- Rotate the arm (one finger!) of the Nav-Cam and wait for it to stabilize. Click to capture an image (turn off the light). When it finishes, rotate the Nav-Cam!! If it is too bright, correct the brightness.
- Alowly close the door (ONLY if the **Nav-Cam removed**) and make sure not to bump into any detectors!.
- Press PUMP on UI (caution on the pumping mode - mostly HiVac, if no detectors were installed! If the samples are not suitable for HiVac talk to the CEMM group.) Always **remove the samples from the objective lens,** if possible.

HiVac mode

Be careful, the large table is not intended for high tilt and min WD is 8 mm (NO PROTECTION for the CBSD!!). Max tilt is 60° but always inspect the CCD so no damage is made!

ETD image (HiVac)

- Wait until the pressure is < 1.8·10^{-3} Pa (green)
- click Beam ON (changes to yellow) – turns on the filament
- Auto Contrast Brightness (F9)
- Set the **FOKUS**
- Press »**link Z to focal plane**« (offset!) Attention: Always link the highest sample! When you link it turns the Z coordinate system!
  Alignment with set conditions (voltage, spotsize, WD)

Check that the beam shift is on zero (right click on the mouse)

- Set the **crossover (gun tilt)**
- Repeat until satisfactory (F9 - ACB):
  - FOKUS
  - STIG.

*There is only one fixed aperture, so there is NO WOBB centering.

- If necessary, change the conditions (0.2 – 30kV; Spot 1-10, WD min 8 mm (be careful CBSD!!).

Backscattered electron image

- Detectors: **ETD (set bias) or CBSD**
- CBSD has 4 rings that you can select.

Finishing in HiVac

- Turn of the beam e-, it changes back to gray.

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LoVac mode

Check that LFD and CBSD are inserted

TURN OFF THE FILAMENT when switching between vacuum modes!

Max pressure (recommended by a service technician) up to 120 Pa.

***Note: if the sample is vacuum compatible always pump first in HiVac and ten switch to LoVac. If it is not suitable for high vacuum you have to work in ESEM mode ***

- Check the amount of DI water, if low contact CEMM
- Always move the sample away from the lens! (Manually lower, if you have linked enter Z> 30 mm, if not linked 0 is the lowest value and enter.)
- Click Mode: Low Vacuum (selected pressure 70 Pa)
- The system asks you which PLA you have (No Accessory)
- The system tells you to open the valve!
- Start in the middle - 70 Pa pressure, you change the biasing LFD.
- You can do: Tools - Preferences - ESEM - Purge (Custom!)
- Raise the sample back to 10mm marker
- Be careful LFD is slow! Set the scan speed.
- Beam ON
- Change magnification
- AutoContrastBrightness (F9)
- Set FOCUS
- »**Link Z to focal plane**« (offset!) Attention: Always link the highest sample! When you link it turns with the Z coordinate system!

Beam alignment (voltage, spot, WD)

Check that the beam shift is on zero

- Set the **crossover**
- Repeat until satisfactory: FOKUS in STIG.
  If necessary, change the conditions (0.2 - 30kV; Spot 1-10, WD (* min height is 8 mm, watch out for CBSD), chamber pressure and bias on the LFD.

Ending in LoVac

- Turn off e- BEAM, it turns gray.
- If the sample is vacuum compatible, switch to HiVac (remove the sample from the lens!)
- The system tells you to close the EB valve
- Then wait a while (you see the pressure in the chamber drop)

Removing the sample

- Lower the height
- Press VENT
- Remove the samples
- Slowly close the door and press evenly
- Press PUMP in the HiVac mode
- Wait to see the vacuum ($1{,}8 \cdot 10^{-3}$ Pa < …< $2{,}31 \cdot 10^{-2}$ Pa)

End of the session

- **File – Log Off user (it turns off the CCD)**
- clean up after yourself, sign in the notebook
- if you turn off the monitor last day (right for both)

USEFULL keys *manual:
F12 = Compucentric rotation
Shift +F12 = scan rotation
Shift + F1 = image properties
Ctrl + Shift + S = save all

OBSERVATION tips:

»High resolution«
Mag > 50kx, Spot size 1,2 and min WD. Remember the source is W – not FEG!

Standard imaging
Spot size 3,4,5 (SE, CBSD, LFD, GSED)

EDS or more contrast on the BSE:
Spotsize >5, if the sample permits
Voltage from 5kV to 30kV, BD do 4kV (flat samples).

Working in ESEM mode*

If the sample cannot be in HiVac. Check the phase diagram to see the pressure and water vapor. Caution: you can damage the EDS!!

- Remove the EDS!!
- Always use clean gloves – no particles on the PLA
- Remove the CBSD (don't rotate - you can unscrew the lining tube!)
- Mount the PLA (HiRes or EDS) or take out the LFD and mount the GSED.
- Select "no purge"
- Check the water level.
- You insert the sample normally, away from the lens
- Set the mode (ESEM) and click PUMP (max 130Pa).
- The system asks you for a PLA - select the right one visible on the CCD.
- Open EBV when the window appears!
- Only then can you choose a higher pressure (up to approx. 2000 Pa) !!!
- Normal operation * (approx. 800 Pa)

- Ending – turn off the e- beam
- Remove the sample away from the objective lens
- Vent in ESEM mode (never switch to HiVac mode if there is a wet sample inside!)
- When you remove the samples, close the chamber
- Insert the proper detectors and remove the PLA (insert EDS at the end!)
- Click HiVac and PUMP
- The system tells you to close EBV

ESEM is done by prior arrangement with CEMM. Working in LoVac and ESEM is recommended in the afternoon to pump the system overnight.

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Working with EDXS

• Acceleration voltage 5-30 kV (for light elements: 5-10 kV and heavier: 10-20 kV, it is important that 2.5 is above the highest characteristic line)
• Current high (spot from 4 to 10 – if the sample allows)
• The sample should be on WD 10 mm
• At the bottom of the screen, check that the values are the same as on the microscope (e.g.: Mag) if not: CLICK Tidy UP!
• Read the manufacturer's instructions (on the desktop)
• **Cobalt standard for ESEM is in the drawer!**

Beginning:

- If the PC is not turned on (password: supervisor)
- Open AZtec (version 4.3), both lights are on (green - ON, blue - cooled) and EDS is inserted.
- Select "New Project" and a new window will open:

- write the name and select the location of the USERS EDS folder (always save the project, you can process the data again later).
Select YOUR or "Default profile". Do not change other profiles: Tools - User Profile… -Save As… (Export to your folder just in case – it can be deleted (error) - service technician informed).

EDS-SEM:

You can work within the (numbers follow usability):

- Analyzer (just EDS) -4)
- Point & ID (image and EDS): most useful -1)
- Linescan (line elements) -3)
- Map: second most useful -2)
- Optimize (semi or full quant) -5)

Qualitative or quantitative ("standardless")

1) **Point & ID** + quant and report

Step by step:

- **Describe Specimen**: describe the pattern and add tags, images… Select if coated – i.e. C.
- **AZtecLive**: without stopping the system, you move and search for the desired area. If you know what you are looking for skip it!
- **Scan Image**: you can scan up to two images (settings), make sure the BSE is in 1 QUADRANT (on ESEM), otherwise you get two SE images! The area will be marked on both. You can add text, tags, and measurements…
- **Acquire Spectra**: Sets the capture conditions (no need if you have your default settings). Otherwise you go to Settings, select the energy range, number of channels, processing time, spectrum capture time (auto, live time, counts), unchecked pulse pile up correction. Always check on the right: Mini View to see how many cps and dead time. Mark the area. You can mark more and work in sequence, you look at this in the Data View at the top right, you can also stop them there, delete them. You can add text and arrows to the recorded spectrum. You can export the image - settings you can set the exact dimensions of the image.
- **Confirm Elements:** If you want to look at the spectrum and peaks in more detail. You highlight the elements (selected ? and double-click on the top of the spectrum, it shows you overlaps / matches). Go to settings and check Show Fitted Spectrum. If these match then the elements are chosen correctly. This is especially important because the elements overlap.
- **Calculate composition** gives you quantitative results according to settings (normalized). Watch out if you save the items click Requantify! You have a lot of templates and you choose between wt% on at% (Be careful, if you have wt% it will give you this in the report, otherwise set at%). You can copy by highlighting and copying or going to report results.
- **Compare Spectra:** you can compare multiple spectra from different areas (site, this was not possible in INCI).
- **Report Results**: export in word / excel. You have several options, save everything to the project folder. If you want a special, consult the CEMM team to help you.
  If you are adding to the same word, click APPEND instead of save as (watch out, this only works on EDS in the hallway).

If you want to combine these steps, you can do a Custom overview, where the steps Scan Image, Acquire Spectra, Confirm Elements, Quant results are combined. But remember to always return to STANDARD settings!

2) **Mapping**

Step by step:

- **Describe Specimen**: describe the sample and add tags, pictures… S Select if coated – i.e. C. For mapping, it is great to set a spot above 5 if the sample allows. Then check the Ratemeter and set the correct process time so that the dead time is below 40% but more counts. Here the Count Rate of 100 kcps shows the true SDD power of a 40 mm^2 X-max detector.
- **AZtecLive**: without stopping the system, you move and search for the desired area. If you know what you are looking for skip it!
- **Scan Image**: you can take up to two images (settings), make sure the BSE is in 1 QUADRANT (on ESEM), otherwise you get two SE images! The area will be marked on both. You can add text, tags, measurements…
- **Acquire Map Data**: Highlights the desired area and starts working when you drop it. Color folders appear on the right. If you have overlaps with certain spectra, be careful because it gives the wrong contrast - here is the real power of TruMap! Fix this for the background and overlapping! The SEM image with the overlaid image can be exported by right-clicking on Export in settings.
- **Construct Map**: because we have the spectrum stored in a single pixel we can construct a spectrum from the individual area where we mapped. Spectrum reconstruction is stored in Data View. Then in MiniView we can select Compare and see the comparison in histogram or %.
- **Analyze Phases**: here you can enter phases or select the proposed ones to facilitate the presentation of the sample, where the difference in the composition of the elements is one phase…

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- **Report Results**: export in word / excel. You have several options, save everything to the project folder. If you want a special, consult the CEMM team to help you.

3) **Linescan**

Step by step:

- **Describe Specimen**: describe the sample and add tags, pictures… S Select if coated – i.e. C. For linescan, it is great to set a spot above 5 if the sample allows. Then check the Ratemeter and set the correct process time so that the dead time is below 50% but more counts. You can click on the Pre-defined Elements tab, because when you look at the line you are mostly only interested in certain elements and check the Auto ID. You can always add items later. Make sure you save this to your profile and leave the default settings!
- **AZtecLive**: without stopping the system, you move and search for the desired area. If you know what you are looking for skip it!
- **Scan Image**: you can scan up to two images (settings), make sure the BSE is in 1 QUADRANT (on ESEM), otherwise you get two SE images! The area will be marked on both. You can add text, tags, and measurements…
- **Acquire Line Data**: Highlight the desired area (click and drag) and click start. A snippet of the SEM image appears where the line is drawn and the elements (we selected) are in a line with a different color. If we have it set to record "until stop", we record so long that we still see an improvement in the statistics, then you stop. But you can set the time. Line profiles can be Stacked or Vertical Tiles. You can zoom in and right-click Export or reset scales
- **Construct Map**: because we have the spectrum stored in a single pixel we can construct a spectrum from the individual area where we mapped. Spectrum reconstruction is stored in Data View. Then in MiniView we can select Compare and see the comparison in histogram or %.
- **Report Results**: export in word / excel. You have several options, save everything to the project folder. If you want a special, consult the CEMM team to help you.

4) **Analyser**

Step by step:

- **Describe Specimen**: describe the pattern and add tags, images… Select if coated – i.e. C.
- **AZtecLive**: without stopping the system, you move and search for the desired area. If you know what you are looking for skip it!
- **Acquire Spectra**: Sets the capture conditions (no need if you have your default settings). Otherwise you go to Settings, select the energy range, number of channels, processing time, spectrum capture time (auto, live time, counts), unchecked pulse pile up correction. Always check on the right: Mini View to see how many cps and dead time. The EDS is from the whole picture on SEM! You can export the spectrum image - settings you can set the exact dimensions of the image.
- **Confirm Elements:** If you want to look at the spectrum and peaks in more detail. You highlight the elements (selected ? and double-click on the top of the spectrum, it shows you overlaps / matches). Go to settings and check Show Fitted Spectrum. If these match then the elements are chosen correctly. This is especially important because the elements overlap.
- **Calculate composition** gives you quantitative results according to settings (normalized). Watch out if you save the items click Requantify! You have a lot of templates and you choose between wt% on at% (Be careful, if you have wt% it will give you this in the report, otherwise set at%). You can copy by highlighting and copying or going to report results.
- **Compare Spectra:** you can compare multiple spectra from different areas (site, this was not possible in INCI).
- **Report Results**: export in word / excel. You have several options, save everything to the project folder. If you want a special, consult the CEMM team to help you.
  If you are adding to the same word, click APPEND instead of save as (watch out, this only works on EDS in the hallway).

Semi-quantitative EDS analysis

Make sure you have a standard! Set all the conditions for work and EDS and go to Optimize. CAUTION: be extremely careful not to run-over the original standards!!! Always return to the standards from Oxford! If you don't know what you're doing don't press it!

5) **Optimize** – semi-standard standardization

Step by step:

- **Calibrate**: select Beam Measurement (necessarily if you are doing NOT normalized) and select the element you have in the chamber (C, Si, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn). You click Start, watch it only work with the settings you have! So with each change (voltage, spot size) it is necessary to re-measure. When it finishes it tells you what% of the last value it is. You click Yes. It's good to do it again to see the stability of the flow!
  * You can also do Energy Calibration (only on standard!). Because the system is stable there is no need unless you see that the tops do not match. This can happen if the room temperature fluctuates. When it's done, he asks you if you save - Yes. MAKE SURE IT'S OK THE STANDARD!
  - **Standardize:** Quantitative analysis can be done without standard materials because the Oxford system is equipped with default standardization! In certain cases, however, this will show in the improvement of quantitative results. You have to have standards here and EXTREMELY be careful what you click so as not to erase Factory standards! Contact the CEMM team for the procedure.
  - **Pile Up Correction:** If several X-rays occur at the same time we can get the sum and the apparent dots of two photons. It is basically set to Oxford settings but you can correct it here.
  - **Report Results:** You can export recorded standards.

The end:

You always save the Project after work! Because you can still process and export everything!