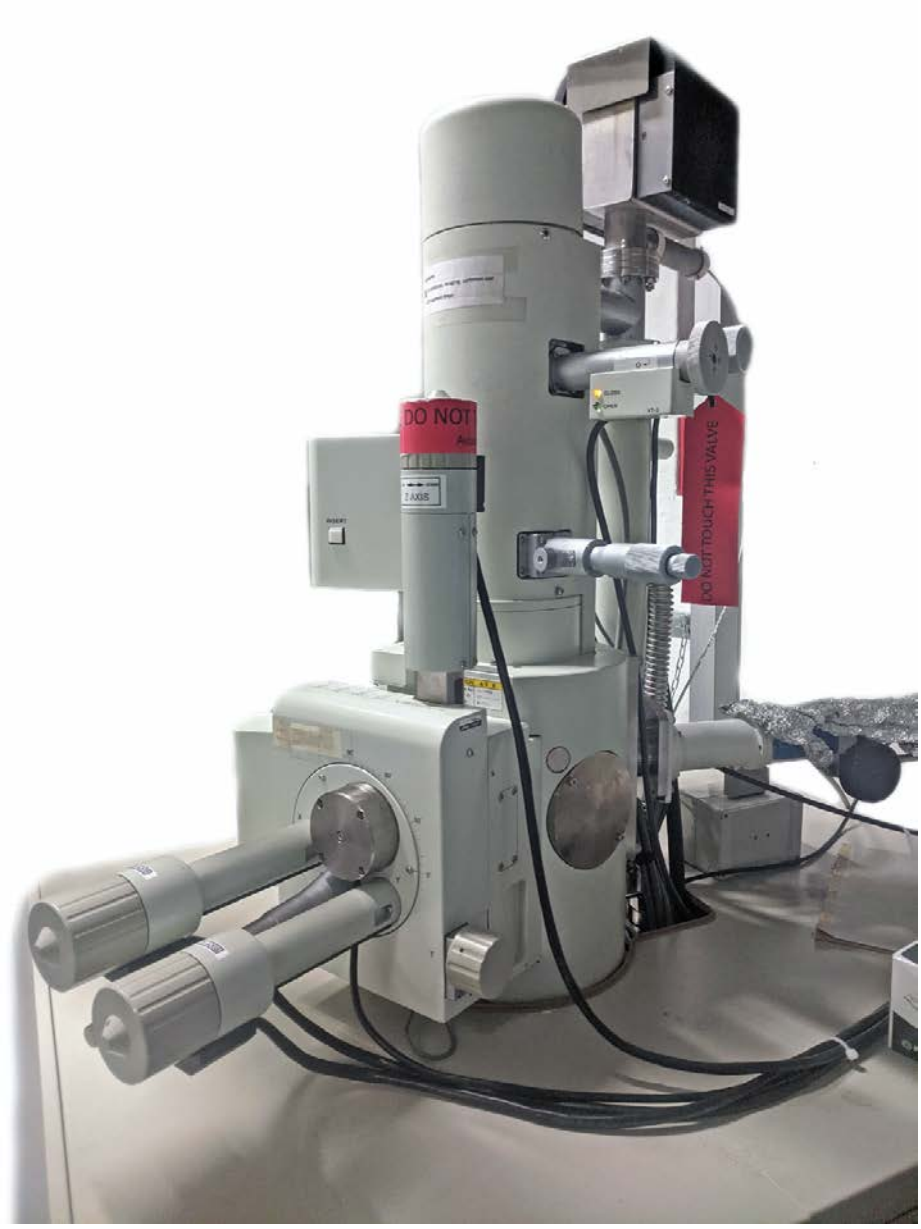
Scanning Electron Microscope (SEM) JEOL JSM-6390 LA

User's manual



Julien M. Allaz

May 6th, 2022

Version 2.0.2 (Pathfinder)

Visit the…

"Database of electron Microprobe Analysis (De⁻MA)"

…for a complete listing of
characteristic X-rays,
a list of standards used at ETH,
and more!

http://de-ma.ch

– Use the guest access "JEOL" –

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A) Generalities about the JEOL SEM @ IGP

A.1) Lab overview

The scanning electron microscope (SEM) at ETHZ D-ERDW / IGP has **SE** (secondary electron), **BSE** (backscattered electron) and **CL** (cathodoluminescence) **imaging** capabilities and can provide **accurate standard-based quantitative** analyses of major and some minor elements **by EDS** (energy dispersive spectrometry). See ***Figure 1***.

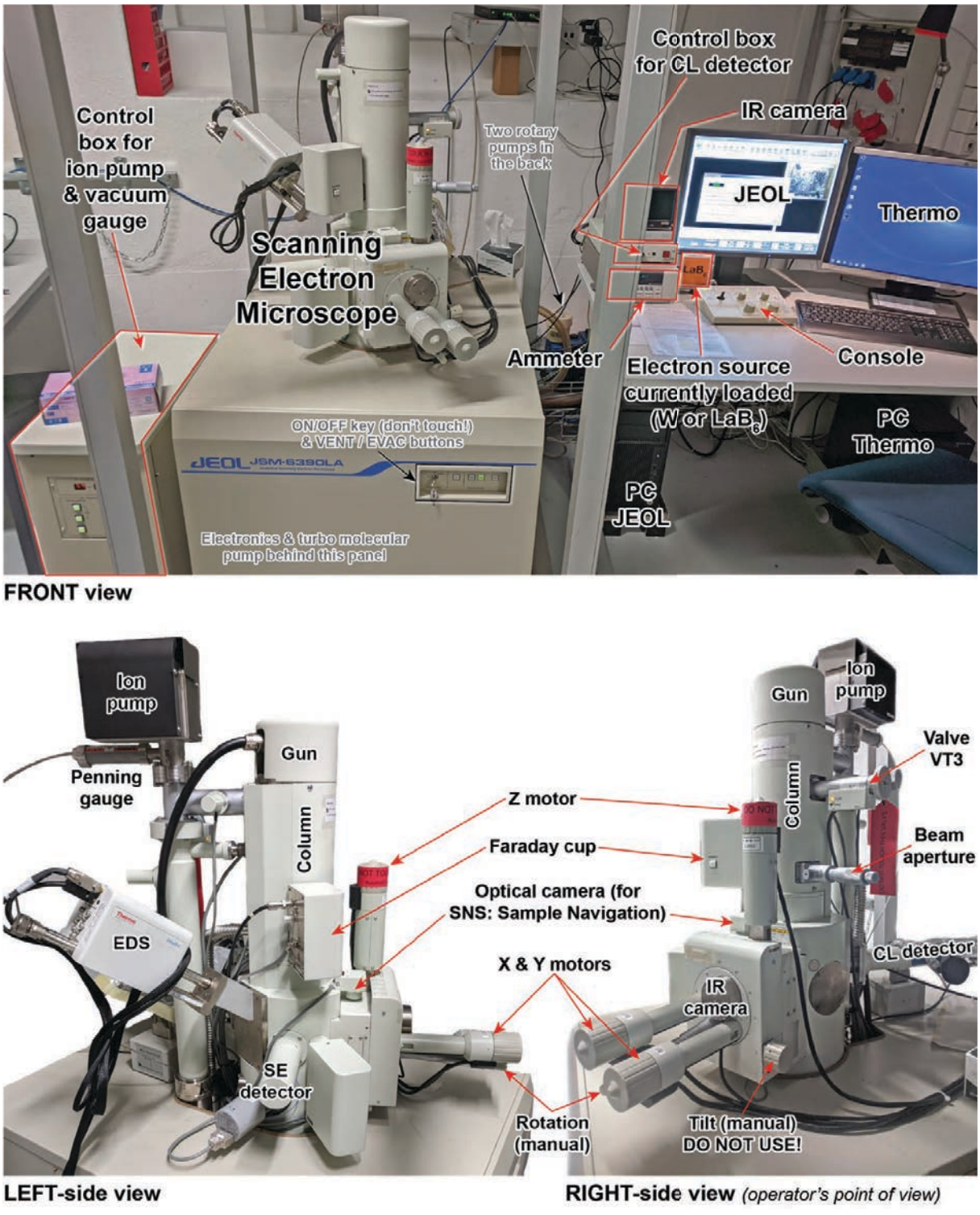


Figure 1. *Overview of the JEOL JSM-6390 at ETH Zürich, D-ERDW / IGP.*

A.2) Computers

The SEM is controlled by **two computers** that are **always ON:**

- **The LEFT computer** is the main **JEOL computer** controlling the instrument itself. It is used to control the electron source, to set up the instrument parameters, to perform the beam alignment, and to move the stage (X, Y, Z), among other…
- **The RIGHT computer** controls the **Thermo EDS system** through the program **"Pathfinder"** from Thermo. This program is used to ***acquire both images*** (SE, BSE, CL) ***and compositional data*** (e.g., quali- or quantitative analysis, element maps). This computer can be restarted anytime. Even if the Pathfinder program crashes, all your data are automatically saved, except for the analysis that was running when the program crashes. *The Windows login password is written on the computer.*
- **NEVER restart or shutdown the JEOL computer!**
- **NEVER exit the JEOL SEM program!**

A.3) First things to do when you arrive

When you first arrive in the lab (***Fig. 2***)…

1. **Turn ON the JEOL computer screens.** *The Thermo screen should be ON all the time.*
2. Check the **general status of the instrument:**
   - **Is it working and under high vacuum?** Check the vacuum controller (box on wheel on the LEFT side of the instrument with indicator on the top). **The vacuum should be in the mid to low 10^{-5} Pa range. (*)**
   - Is the **HT button** (high tension; top-left side of the JEOL PC) **green & ON?**
   - If something is wrong, call for assistance.
3. Check that nothing is running, on the Thermo computer; there could be some overnight map running. Check with the previous user if it's still running!
4. Activate the IR camera, and check if a sample is currently loaded.
5. **Which electron source is currently loaded?** Check the paper on the bottom of the left screen. *The starting procedure will depend on which source is currently loaded!*
   - **W** (tungsten filament – GREEN sign)
   - **LaB_6** (lanthanum hexaboride crystal – ORANGE sign)

*(*) **WARNING!** Exception applies when, due to some technical difficulties, the SEM is in "W-mode" with the ion pump OFF (no vacuum reading). See **Appendix A2** if you see the additional BLUE sign for "W-mode".*

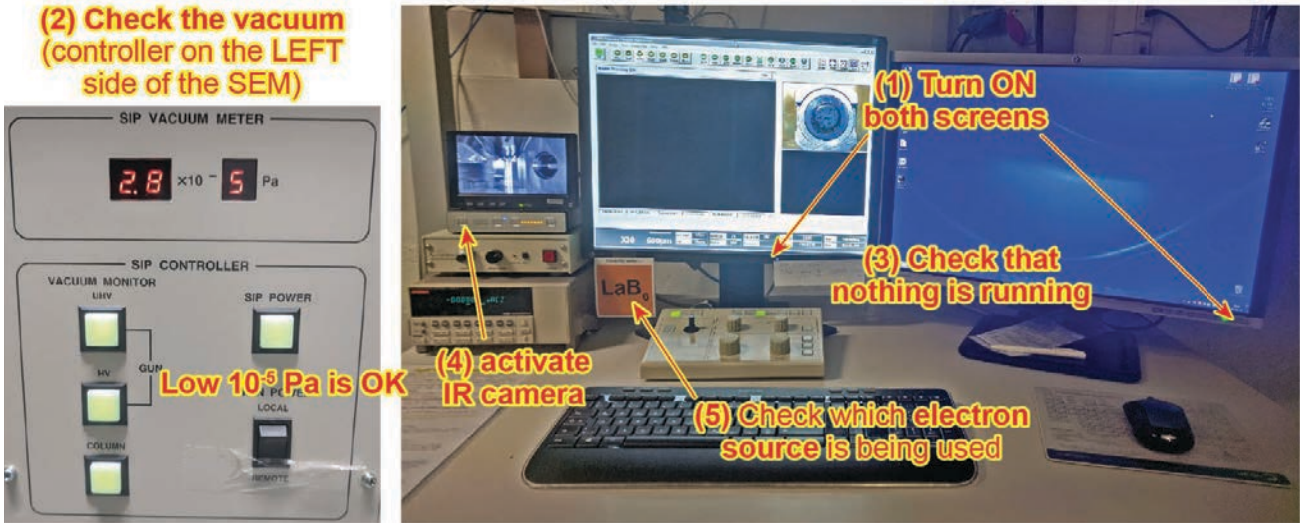


Figure 2. *Checking instrument status (in "LaB_6-mode" with the ion pump working).*

A.4) Overview of the JEOL console

The JEOL SEM has a **console** that is used to control the stage motion (X, Y), and the electron imaging capabilities (magnification, brightness & contrast, etc.; ***Fig. 3***):

- **The left side** of the console controls the stage motion. **Leave the "Stage" setting to "X/Y"!** You can then use the joystick to move along the X and/or Y axis.
- **The top-right side** of the console "Scanning mode" has several options for the different scanning rate. You will most likely use only **"Scan2"** (full-frame fast scan, ideal for navigating in the sample) or **"Scan1"** during the alignment (fast scan over a reduced area). Scan3 is a slow scan useful for checking image quality (especially with CL). Other scanning options (Scan4 and Photo) are not used; Pathfinder (***Section H***) is used for acquiring and saving high-quality images.
- **The middle-right side** controls either the brightness and contrast (when STIG is OFF) or the astigmatism correction (when STIG is ON). In any case, prefer the use of the buttons on the top of the electron image to adjust these parameters.
- **The bottom side** of the console has 2 similar knobs for magnification (left) and focus (right). ***Pay attention to which one you twist!***
    - ***Left-one*** is the **MAGNIFICATION knob**. It controls the field of view of the electron image, from 20-40x to 300,000x.
    - ***Right-one*** is the **ELECTRON BEAM FOCUS knob**. It controls the focus point of the electron beam through the objective lens and defines the ***working distance (WD)***. Activate the "coarse" button for large changes in the focus (e.g., rough focussing), and DE-activate this "coarse" button for fine tuning.

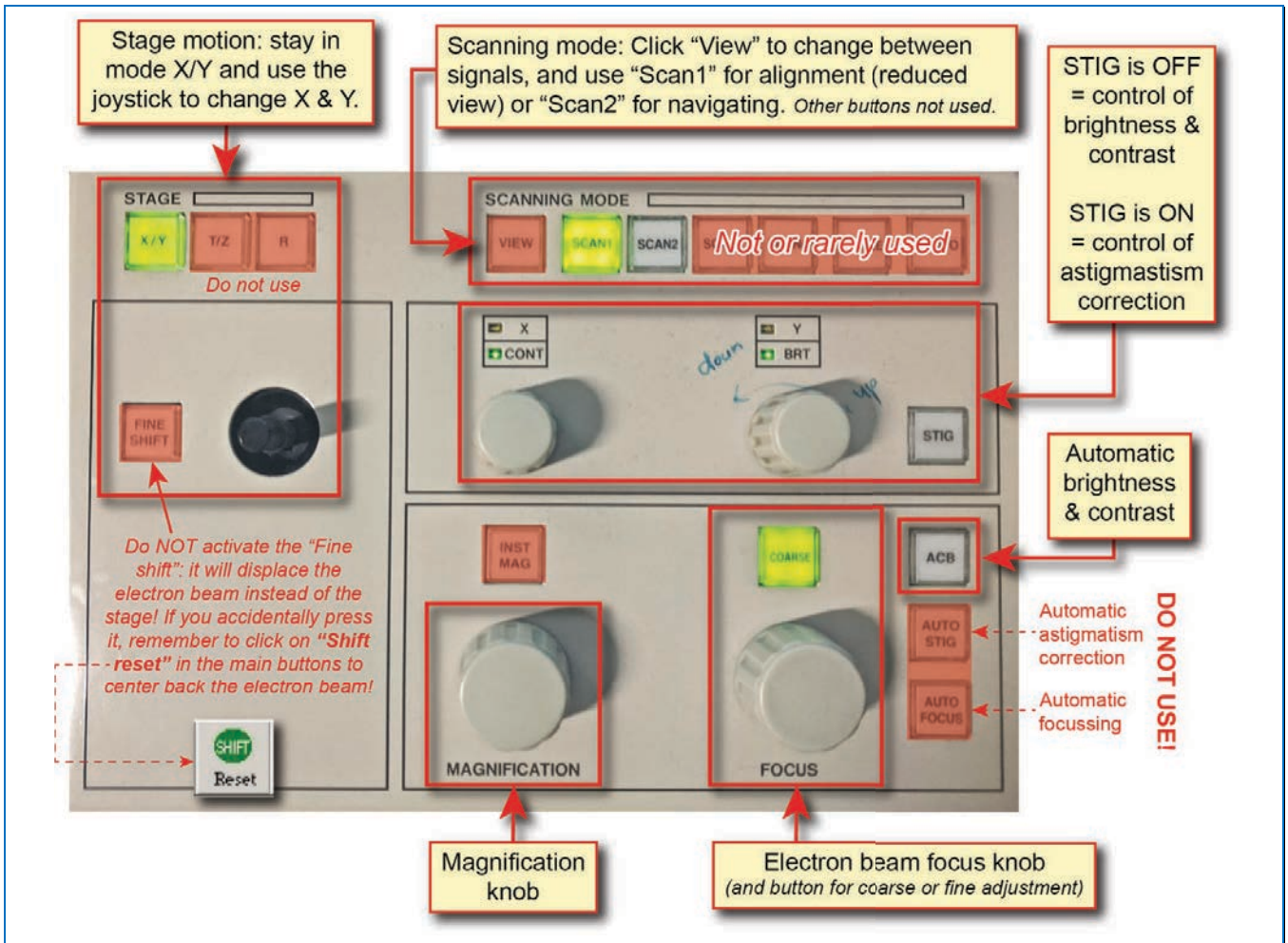


Figure 3. Overview of the SEM console.

A.5) Overview of the "JEOL Scanning Electron Microscope" program

On the LEFT computer, there is **a single program running constantly: "JEOL Scanning Electron Microscope"**. It is subdivided in three main parts as shown in ***Figure 4***:

- **Top section:** menu bar & main buttons.
- **Middle section:** electron image display with grey buttons used to control the image quality, and on the right side a snapshot image of the sample holder.
- **Bottom section:** instrument status (voltage, WD, spot size, etc.).

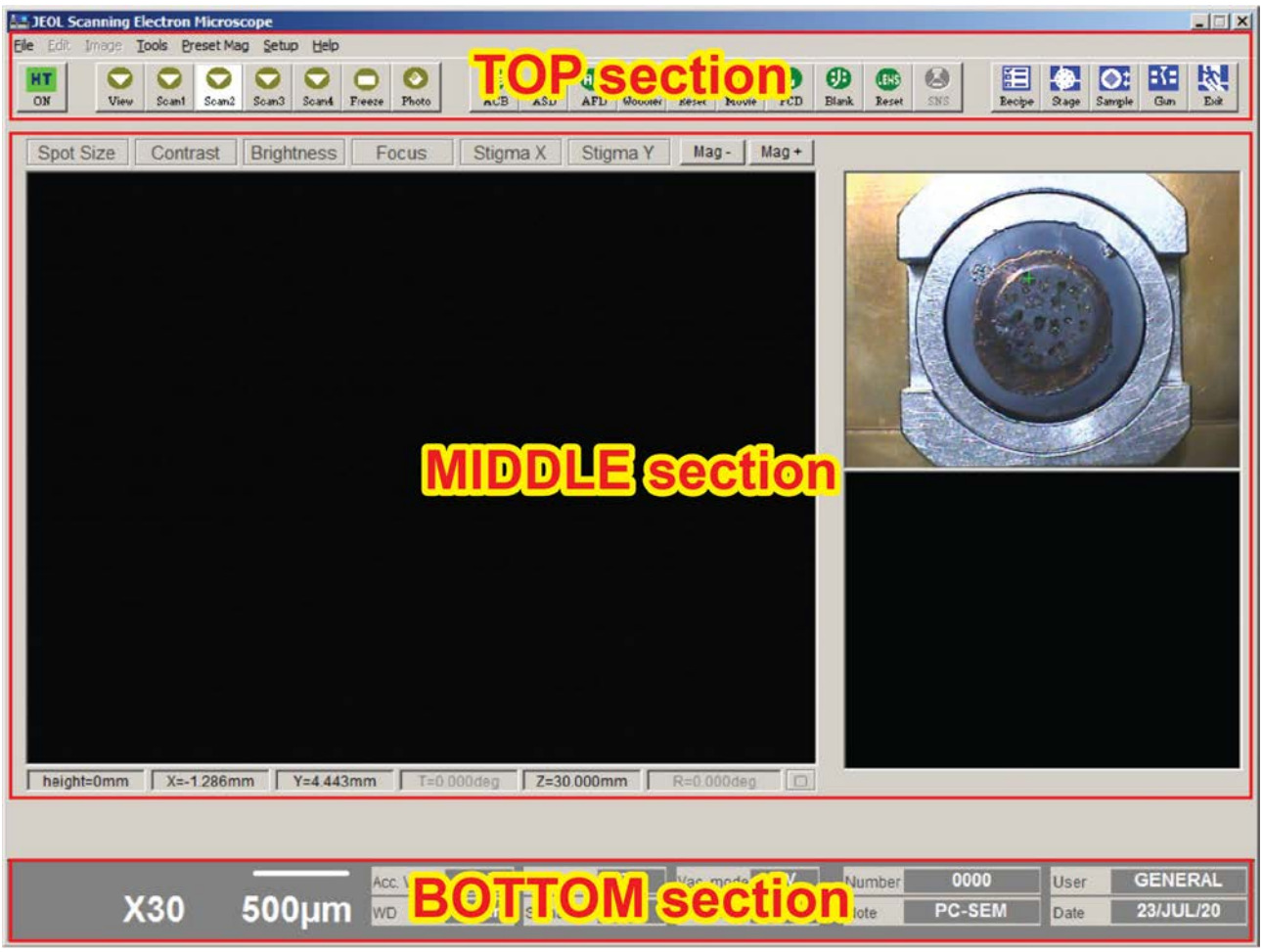


Figure 4. *Overview of the "JEOL Scanning Electron Microscope" program.*

The next ***Figures 5 to 7*** present an overview of the different sections of the JEOL SEM program.

Refer to the following ***Sections B to F*** for details on the important buttons in this program, and in ***Section G*** for the CL detector.

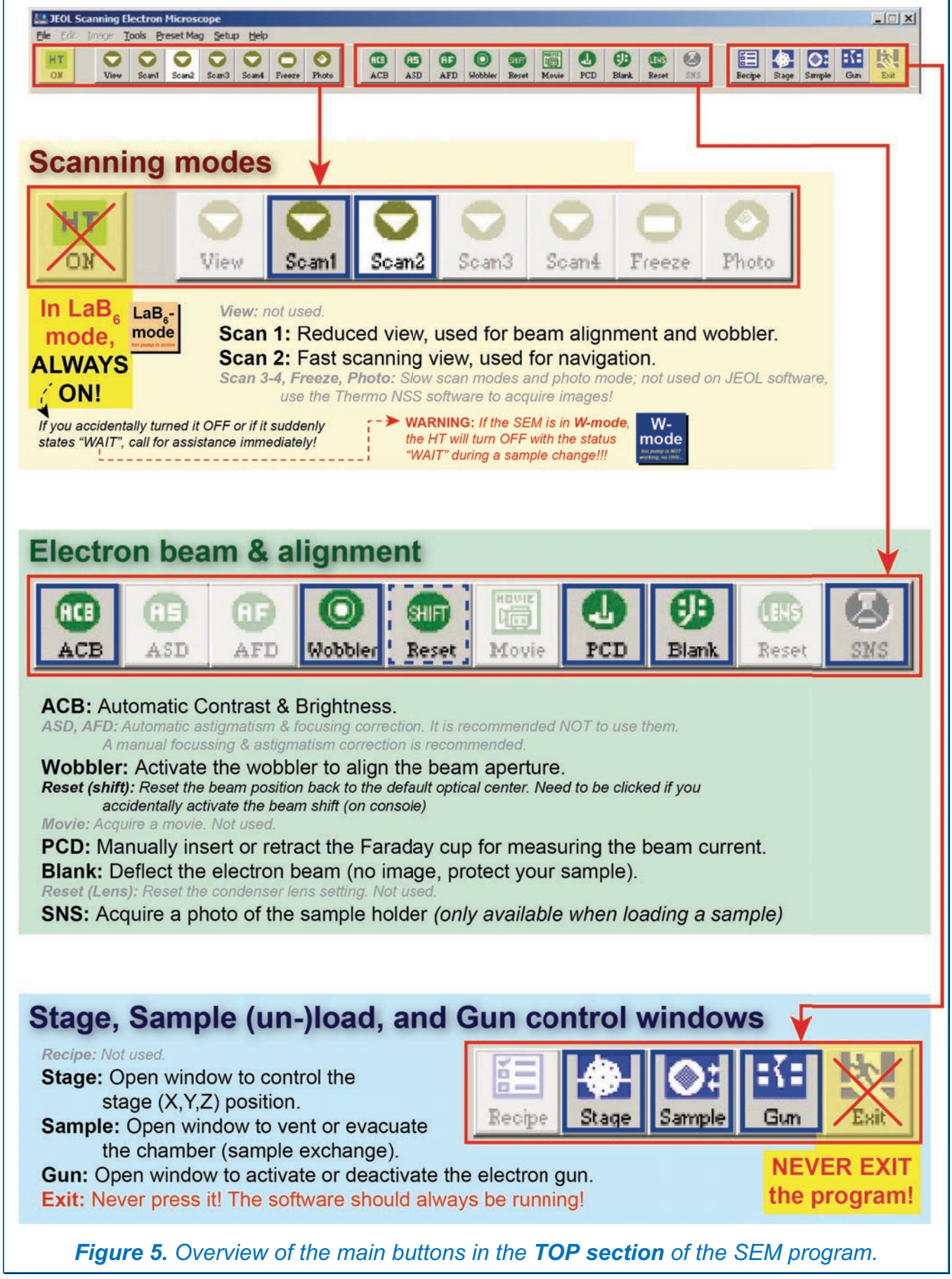


Figure 5. *Overview of the main buttons in the* ***TOP section*** *of the SEM program.*

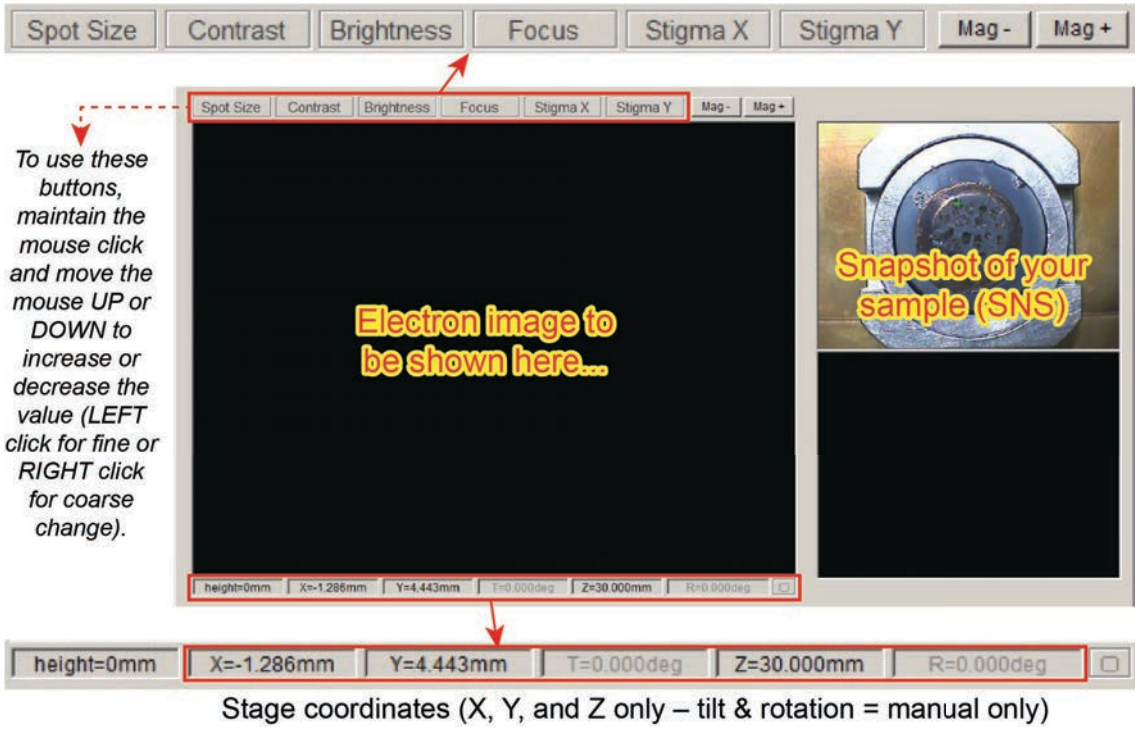


Figure 6. *Overview of the* ***MIDDLE section*** *of the SEM program.*

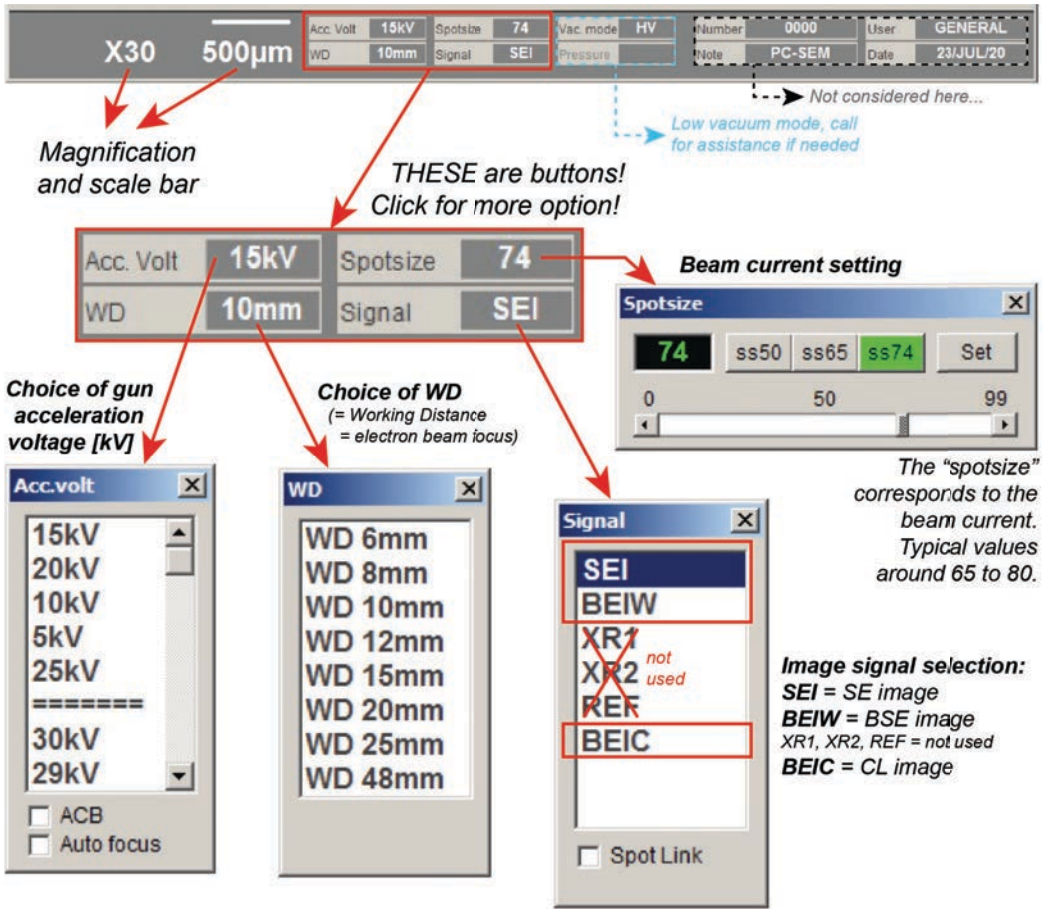


Figure 7. *Overview of the* ***BOTTOM section*** *of the SEM program.*

B) Starting your session

1. **Check if a notice with special instructions is on the table** (e.g., in case of an outage, a problem with the SEM or its detector, etc.).
2. Turn ON the JEOL computer screen.
3. Ensure that nothing is currently running. If an analysis is still running *(e.g., mosaic BSE or EDS mapping)*, contact the previous user, and see if you can wait for the completion or if you can cancel the analysis.
4. Turn ON the infrared camera ("Deben", small screen on the left; ***Fig. 8***).

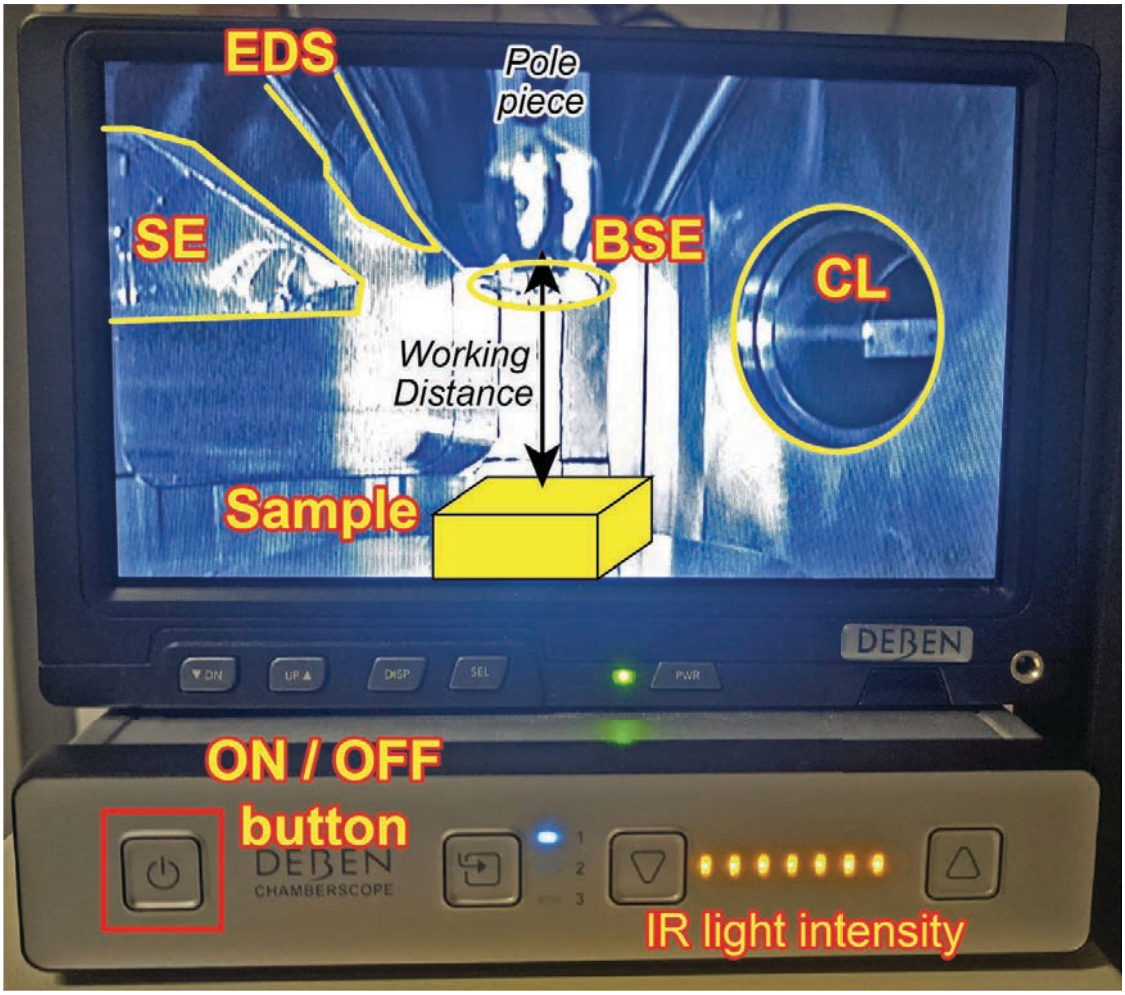


Figure 8. *Main SEM detectors and components visible on the Deben infrared camera.*

5. If a sample is currently loaded in the SEM, you will need to remove it (***Section C***).
6. Prepare your sample (***Fig. 9***): choose the thin-section or the 1-inch round mount.

WARNING! If your sample does not fit the thin section or the 1'' round holder, you MUST contact the lab manager or the assistant before continuing!

7. Proceed to the sample loading (see ***Section C***).

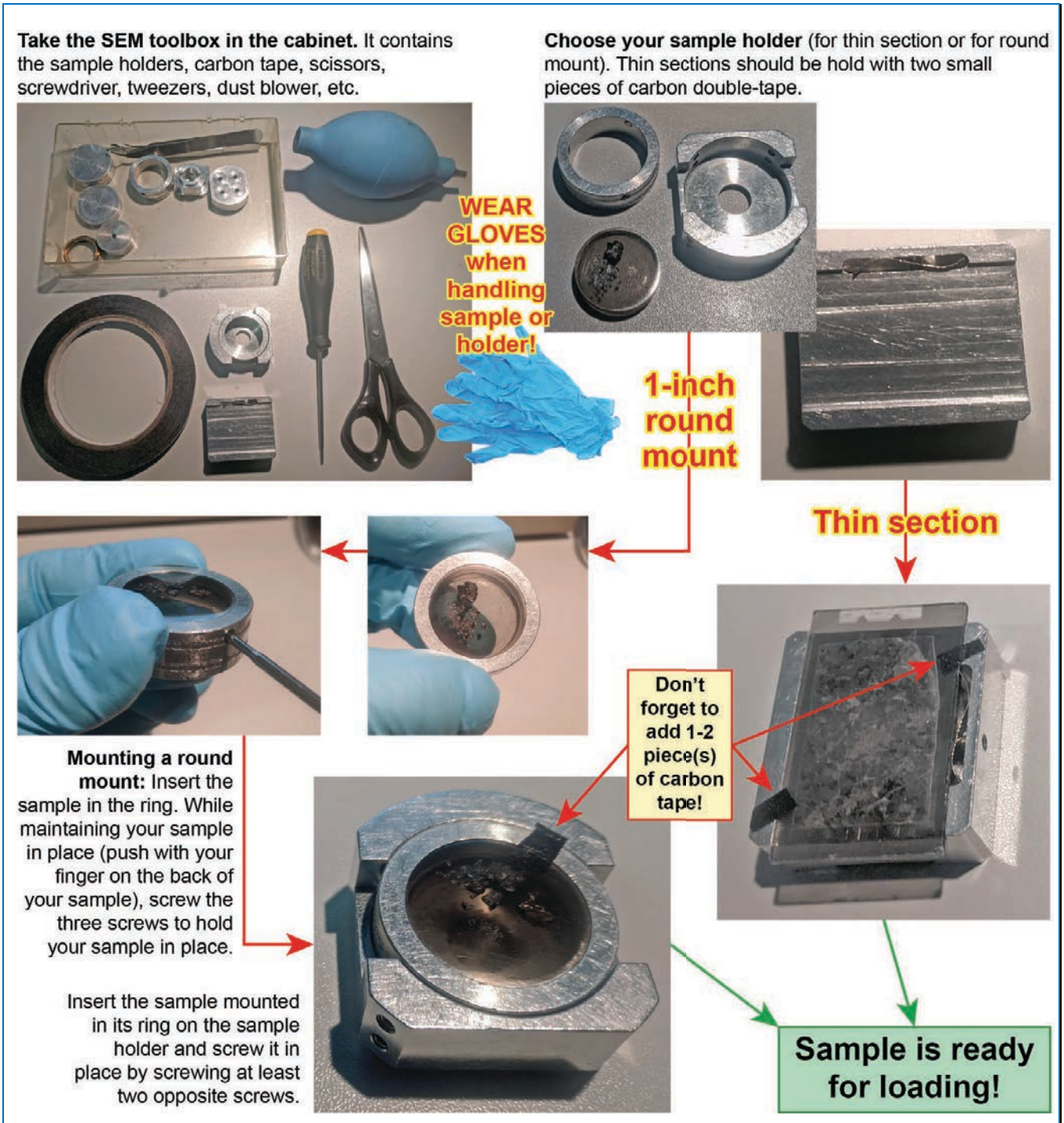


Figure 9. *Preparing the sample holder (for thin section or for 1'' round mount).*

C) Sample loading or unloading

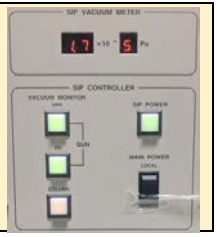
The sample change procedure is summarised below. Refer also to ***Figures 10 and 11a,b***.

C.1) Opening the sample chamber

1. In the JEOL SEM program, set the signal to SEI in the bottom section of the program.
2. **If the CL detector was in use, turn it OFF** (red button on the CL control box).
3. Turn ON the Deben infrared camera (if not already on).
4. Open the window "Stage" and move the stage to Z = 30 mm (absolute position).
5. **If the CL is inserted** (check on the IR camera!), you MUST remove the CL detector (see ***Figures 12 and 13***).
6. On the SEM column, close the valve VT-3 (tight, with the strength of two fingers).
7. Back to the JEOL SEM program, open the window "Sample" and click "Vent".

WARNING: When the SEM is in LaB_6 mode, check the vacuum gauge while venting (see on the left side of the SEM) to ensure you don't lose vacuum. Only the light "column" should turn orange, all other lights should stay green. **If you lose vacuum, ask immediately for assistance!**

This warning does NOT apply if the SEM is in W-mode *(see Appendix A2).*



8. When fully vented, open the sample chamber (see C.2 and ***Figure 11***).

C.2) Removing/Loading a sample & closing the chamber

1. Open the sample chamber up to the back stop (use two hands!).
2. Remove the sample and/or load your sample.
3. If you are loading a new sample, take a **snapshot with SNS:**
   a. Place your sample on the holder.
   b. Click the button "SNS" in the top section.
   c. Click "Snap". The stage will drive to the centre of the optical camera.
   d. Wait a few seconds until a snapshot of your sample appears in the SEM program. If you think the SNS image is not properly centred, refer to the calibration section to calibrate the SNS in ***Appendix A1***.
   e. **IMPORTANT!** Lower again the stage to Z = 30 mm after using SNS!
4. Close the sample chamber while looking simultaneously at the IR camera to avoid a disaster (e.g., sample holder touching a detector).
5. Open the window "Sample" and click "Evac", while pressing on the sample chamber door. Wait a few seconds until the vacuum catches up.
6. **Wait until the signal "Ready" appears.**

If this is the end of your session, you are done. Otherwise…

7. Open valve VT-3.
8. Remove the "Beam blank" and proceed with the gun activation (***Section E***) and the beam alignment (***Section F***).

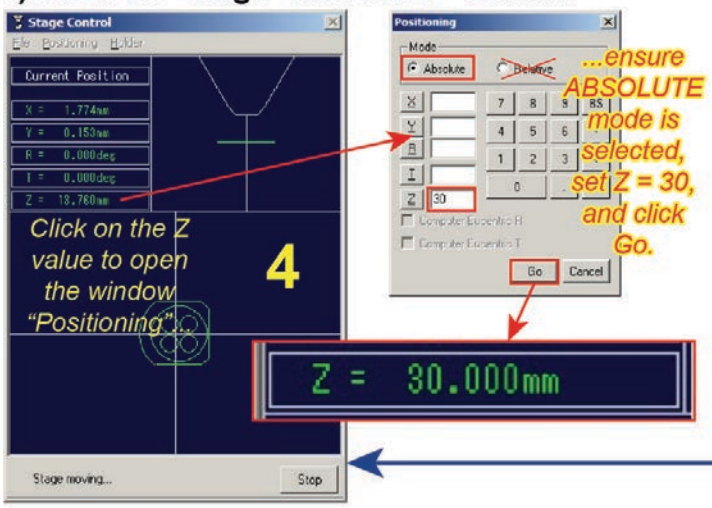
VENTING chamber (open & remove a sample)

1) Set the signal to "SEI".
Set the magnification to the minimum (~40x).

2) Turn OFF the CL detector if you were using it.

3) Turn ON the IR camera.

4) Click on "Stage" and set Z = 30 mm.



5) Remove CL detector *(if applicable)*

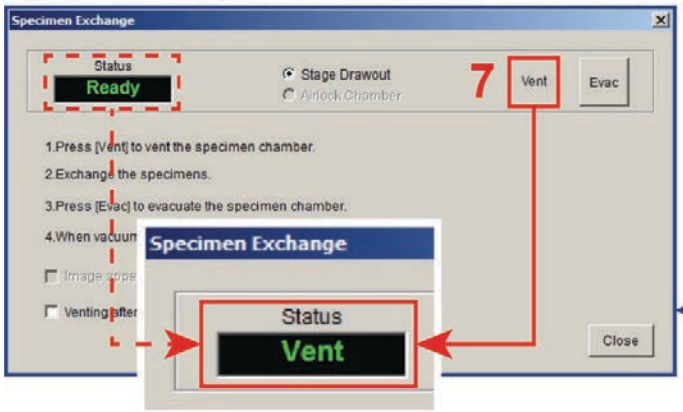
When in **LaB$_6$-mode** *Ion pump is active*

6) Close valve VT-3 *(tight, with two fingers)*

In ***W-mode****, it does not matter: you can leave VT-3 open...*

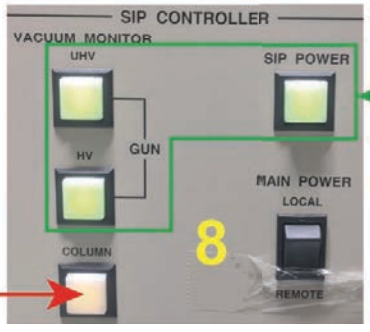
W-mode *Ion pump is NOT working, no UHV...*

7) Open "Sample" and click "Vent"



See ***Figure 11*** *for opening the chamber, (un-)loading sample, and closing it back...*

8) Check the vacuum gauge
(on the left side of the SEM)



All other lights remains green. ***If they turn orange, call for assistance immediately!***

W-mode *Ion pump is NOT working, no UHV...*

In W-mode, all lights will turn orange during the sample change.

Only the light "COLUMN" turns orange.

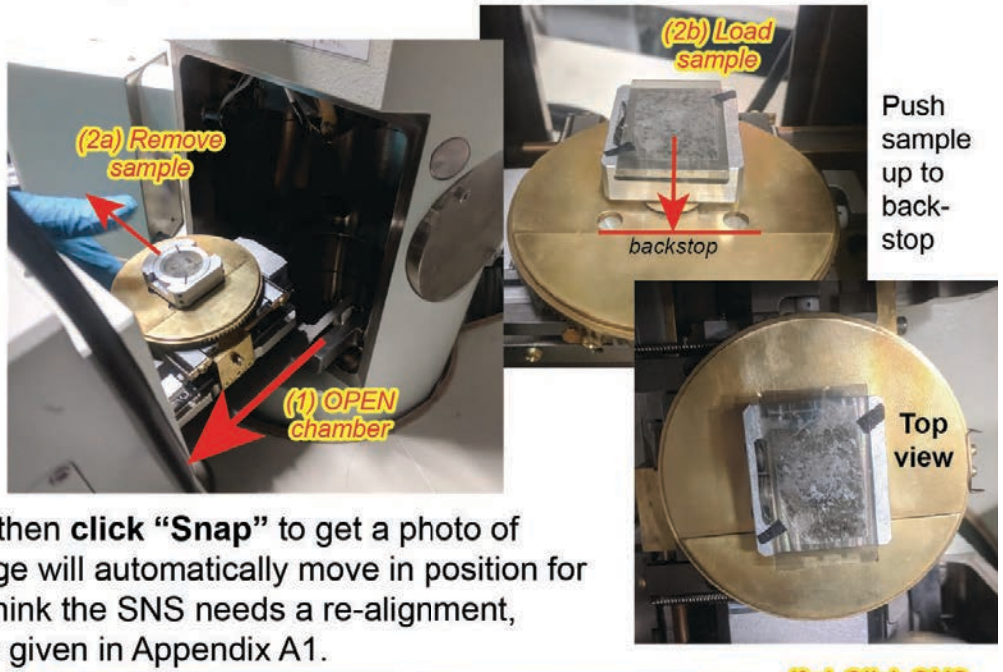
When in **LaB$_6$-mode** *Ion pump is active*

Figure 10. *Opening the sample chamber (to remove or place a sample).*

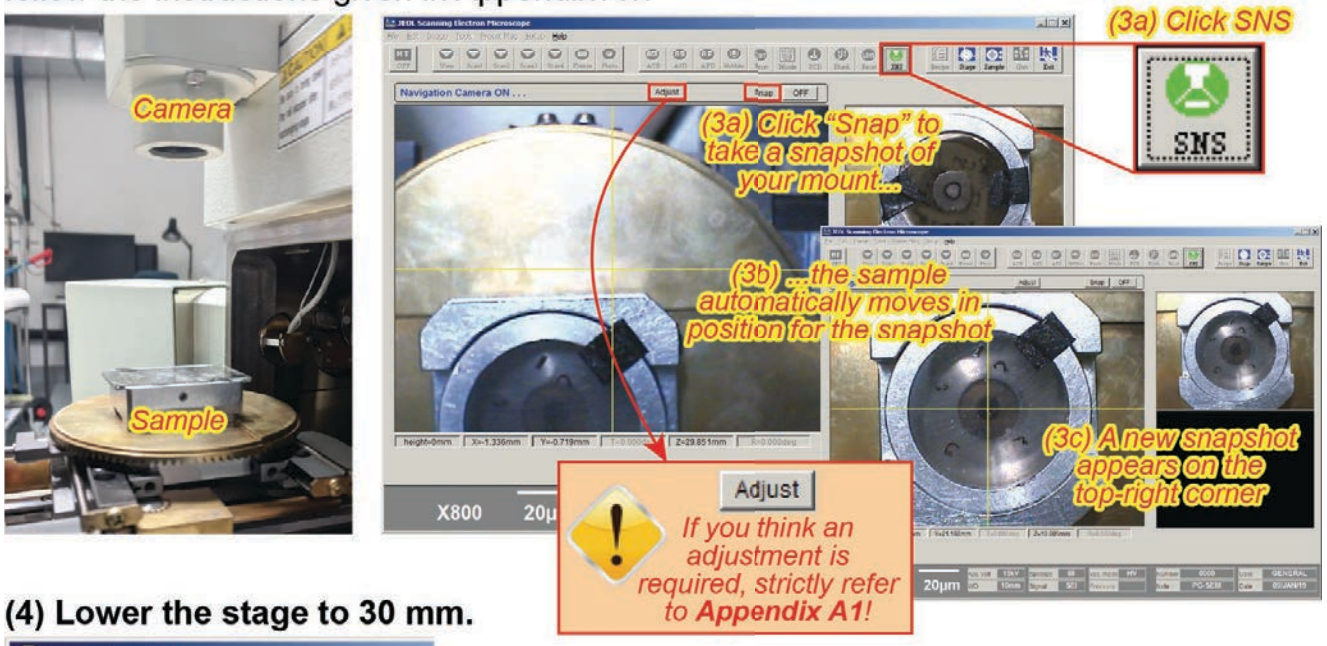
Open, (un-)load sample, and close the sample chamber

(1) Open the chamber fully (up to the back-stop)

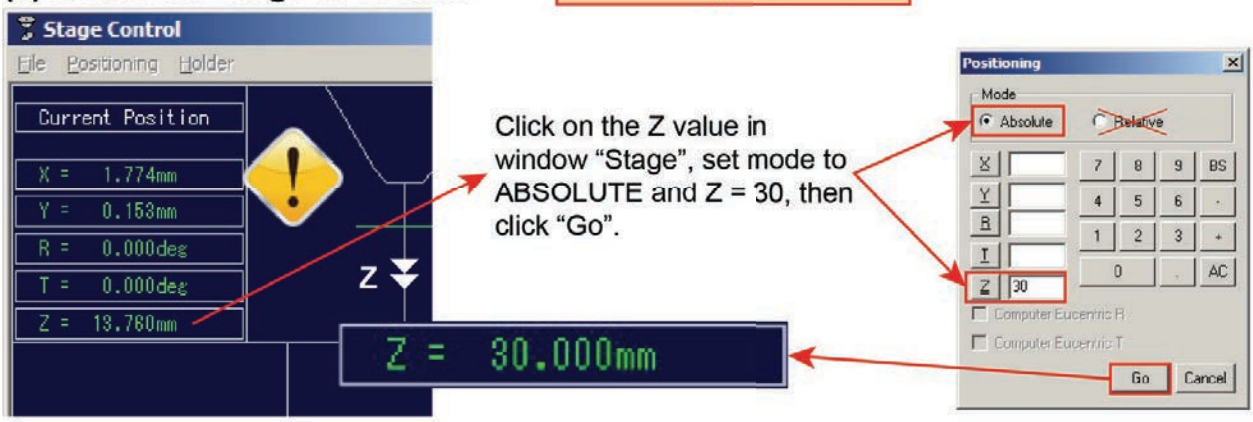
(2) Remove the sample and/or load your sample (thin section or one-inch round mount).



(3) Click "SNS" and then **click "Snap"** to get a photo of your sample. The stage will automatically move in position for the snapshot. If you think the SNS needs a re-alignment, follow the instructions given in Appendix A1.



(4) Lower the stage to 30 mm.



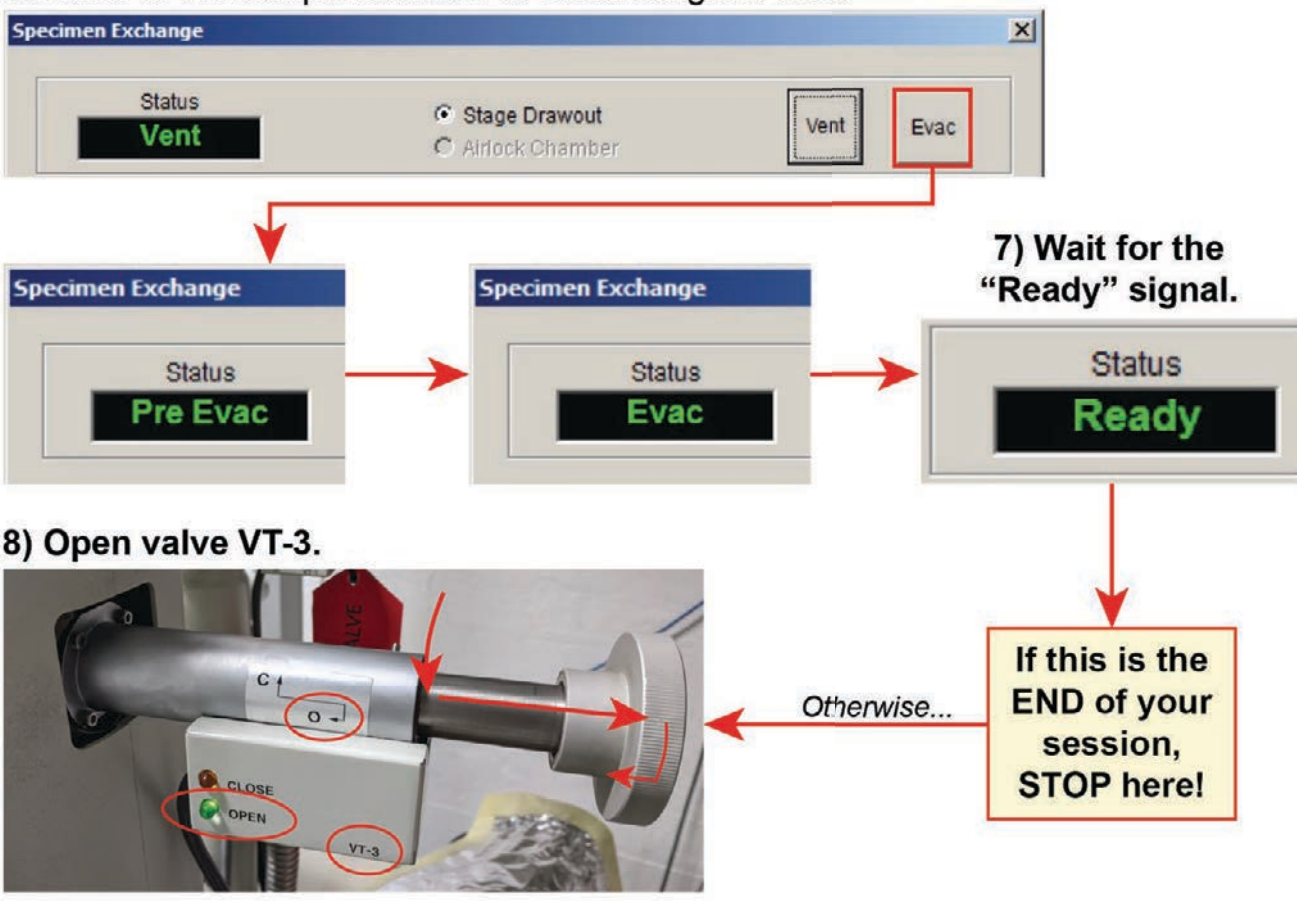
(5) Close the sample chamber; look at the IR camera to ensure you won't hit a detector!

Figure 11a. *Loading a new sample and taking an overview image of your sample (SNS).*

Pumping (EVAC) the sample chamber

1) to 5) see Fig. 11a

6) Open "Sample" and **click "Evac"**. Simultaneously, press on the door of the sample chamber to ensure a good seal.



8) Open valve VT-3.

Otherwise...

If this is the END of your session, STOP here!

9) Remove the "beam blanking" and proceed to the beam alignment.

Figure 11b. *Evacuating (pumping) the sample chamber after (un-)loading a sample.*

D) Stage motion

D.1) Horizontal displacement (X, Y)

There are several ways to navigate along the X- and Y-axis in your sample:

- ***Click-and-drag*** the mouse on the ***electron image***.
- ***Double-click*** on a feature on the ***electron image***.
- Use the ***joystick*** on the console (***in X/Y mode***).
- ***Double-click on the SNS*** image of your sample (inaccurate but close enough…).
- Use the ***window "Stage"*** and either…
    - Click on one of the coordinates and change the value in the window "Positioning". Ensure you are in **ABSOLUTE mode** and click "Go"!
    - Click on the map view (bottom section of window "Stage") and click "Go".
    - Note that in the Stage window, the "Go" button will change to "Stop" as soon as the stage is moving. Keep your mouse on this button so you are ready to stop the stage if it is approaching too dangerously from a detector!
    - *Other possibility (rarely used): Move the mouse along the X or Y axis of the map view. When the mouse pointer changes to a single or a double arrow, click to move by one step or maintain the click for a continuous motion. A single-arrow indicates a slow motion (micron-sized), a double-arrow indicates a faster motion (with acceleration feature).*

D.2) Vertical displacement (Z)

WARNING: Any time you significantly move the Z-axis (> 0.5 to 1 mm) you should ALWAYS turn ON the **infrared camera** and WATCH that IR camera to ensure that the stage does not bump into the BSE or the CL detector! ***Repairs can be costly!***

To move the sample up or down along the Z-axis:

- Open the window "Stage", and (two options)…
- ***(Safest)*** Use the cross-section view on the top-right of the "Stage" window:
    - Move the mouse over the vertical axis until the mouse pointer changes to a single-arrow (slow motion) or a double-arrow (fast motion).
    - Click a single time to move one step at a time or click and hold for a continuous drive (the stage will stop as soon as you release the click).
- To move to a fixed (absolute) value, click on the Z-stage position (top-left of the "Stage" window) and enter a new absolute Z position. BE CAREFUL not to set a Z-stage position that would drive the sample holder into the BSE or the CL detector!

D.3) Rotation (R)

The sample holder can be rotated manually using the small rotation knob situated between the X and Y stage motors on the front of the SEM door. It is best practice to rotate the sample on loading (i.e., before you take a snapshot using the button "SNS"), and then NOT to rotate the sample anymore, otherwise you won't be able to double-click on the SNS picture and locate a feature.

D.4) Tilting (T)

DO NOT USE IT! There is a high likelihood of damaging the detectors (SE, BSE, EDS, or CL) when you tilt your sample and move in along any direction (X, Y or Z). The tilting is manually controlled by the knob on the bottom-right side of the SEM door. **Leave it at 0° (horizontal)!** *If you absolutely need this option, contact the lab manager.*

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E) Activate the electron beam

WARNING! Different procedure to activate the electron beam if a W or a LaB_6 electron source is loaded (***Fig. 12***)! Check the note **W** or **LaB_6** below the computer screen!!!

If you see a BLUE sign W-mode, refer to ***Appendix A2*** for separate instructions.

E.1) Activating a LaB_6 electron source in "LaB_6 mode"

When a lanthanum hexaboride (LaB_6) electron source is loaded, it is ***left active at optimum filament heat*** all the time, and only the ***gun bias*** is adjusted to control the electron emission. LaB_6 offers a higher stability, a better image resolution, and a longer lifetime. However, it is fragile and expensive; it can be damaged when warmed up or cooled down too quickly. **Please, strictly respect the following to activate the electron beam:**

- Ensure that the window "Sample" indicates "Ready" (= high-vacuum condition).
- Check that VT-3 is opened and that the "Beam blank" is OFF.
- Open the window "Gun".
- Click on "Set Bias" and click "Preset". *This calls back the default bias value (~100 to 150).*
- ***Do NOT change anything else in the window "Gun Alignment"!*** **NEVER change the filament heat!** The optimum is set by the lab manager or the assistant.
- The "Filament heating" should read between 20 and 30 µA.
- An SE image should now be visible in the JEOL SEM program.
- Perform a beam alignment following the instructions in ***Section F***.

E.2) Activating a W electron source in "LaB_6 mode"

The tungsten (W) electron source is usually left in "standby" condition; the filament heat is just enough to keep the filament warm, but not enough to emit electron. You'll need to perform a ***filament saturation*** by setting the filament heat to its optimum:

- Ensure that the window "Sample" indicates "Ready" (= high-vacuum condition).
- Check that VT-3 is opened and that the "Beam blank" is OFF.
- ***Saturate the W-filament…***
    - Insert the Faraday cup by pressing the button "PCD".
    - Press the top-left button "LOCAL / CONFIGURATION" on the ammeter. This will activate the live beam current reading. If the wording "CONFIGURATION" appears on the ammeter display, press another time on the same button. If you still don't see a live reading of the beam current, press the button "TRIG".
    - The ammeter should indicate a varying value around zero.
    - Set the "Spotsize" around 74 to 78 (= medium to high beam current).
    - Open the window "Gun" and increase the filament heat until the emission current and the beam current increase. Continue increasing the heat, until the beam current decrease again (= "false peak"). Continue increasing the heat until the beam current increases again. You will reach the saturation point when the increase in beam current slows down or drops down slightly.
    - The emission current should read ~30 to 60 µA (or more), and the ammeter should read ~2 to 3 nA at the beam aperture #2.
    - Note that with a W filament, you can increase the heat rapidly. This saturation should not take you more than a minute…
    - When done, click on "PCD" to remove the Faraday cup.
- An SE image should now be visible in the JEOL SEM program.
- Perform a beam alignment following the instructions in ***Section F***.

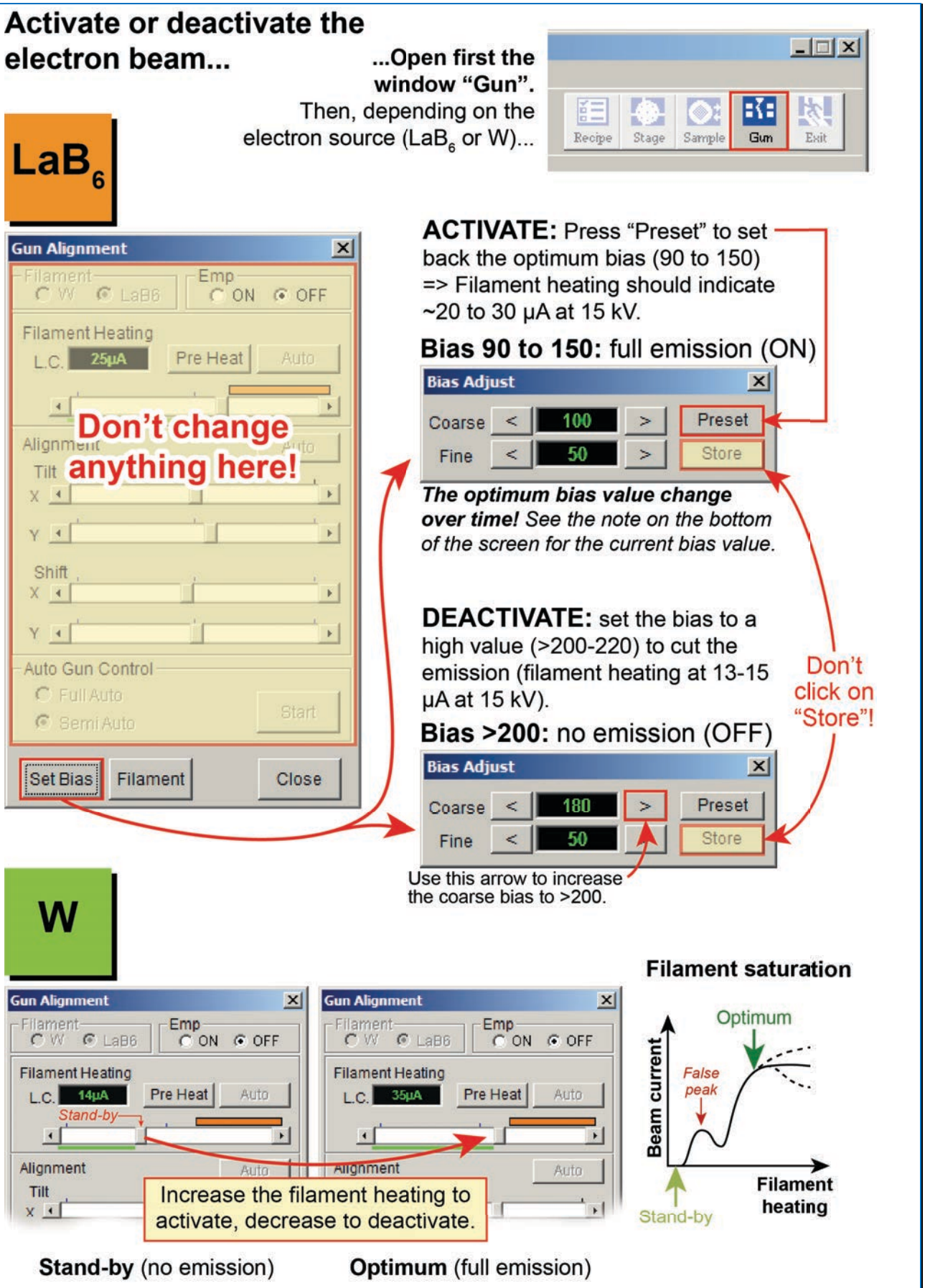

Figure 12. *Activation of the electron beam in LaB_6-mode. Two different processes depending on the electron source currently used: LaB_6 cathode or W filament.*

Refer to Appendix A2 if the SEM is in W-mode!

F) Beam alignment

Once the sample has been loaded and the electron gun has been activated, you must align the electron beam to obtain the ***best image quality***. Before you start the alignment, **set first manually the beam aperture number** you will need:

- Use aperture #2 for imaging and quantitative analysis.
- Use aperture #3 for element mapping.

NOTE: Aperture #1 is for high-resolution images with long depth-of-field at low current & high voltage (rarely used at D-ERDW).

Refer also to *Figures 13 to 16* in the following pages for more details. Always optimise the brightness and contrast to what you need to see (***Fig. 16***)!

WARNING: There are small differences in the alignment procedure if you are running… (a) quantitative analysis and SE/BSE imaging or (b) CL imaging!

F.1) Tilt & shift optimisation of the GUN

As for EPMA, the electron beam needs to be aligned at the gun level to ensure the beam of electron is going vertically down the middle of the column. This is done by adjusting the "tilt" and the "shift". When a LaB_6 electron source is in use, the tilt & shift adjustment is commonly minimal. For the W filament, it is recommended to check the tilt & shift alignment each time:

1. Insert the Faraday cup by pressing the button "PCD".
2. Activate the ammeter for continuous current reading *(button "CONFIG / LOCAL" or "TRIG")*.
3. Open the window "Gun".
4. Set a low current value under "Spotsize" (~60).
5. Increase or decrease the X-value of the **TILT** until the ***beam current*** is ***maximised***; repeat for the Y-value of the TILT.
6. Increase the current ("Spotsize" > 90) and adjust the **SHIFT** X & Y values.
7. Set back a low beam current ("Spotsize" ~60) and adjust again the **TILT**.
8. ***Repeat iteratively*** until the last changes you apply on the TILT are minimal.
9. Remove the Faraday cup by pressing the button "PCD".

F.2) Beam alignment for quantitative analysis or SE/BSE imaging

1. Ensure that the signal is set to "SEI", the magnification is low (~40x), and the "Beam Blank" is deactivated (you can see an image).
2. Set the acceleration voltage and beam current (default: 15 keV; spotsize ~70 to 75).
3. Move the stage (X, Y) position to the metal part of the sample holder.
4. Locate a particle or some visible (even blurry) feature intersecting at ~90°.
5. Adjust the brightness and contrast as necessary (***Fig. 16***).
6. **Set the Working Distance (WD) to 10 mm** by clicking on the WD value on the bottom of the JEOL SEM program, and double-clicking on 10 mm.
7. ***Do NOT change the electron beam focus anymore, as it will modify the WD!***
8. Bring the specimen up to Z-stage around 13 to 12 mm (if the specimen allows it: regular thin sections or 1'' round mount). As the stage goes up, ***monitor the IR camera*** to avoid bumping into the detector! The image quality will improve; get the best image quality possible. Adjust again the brightness & contrast if necessary.
9. Locate a smaller particle or feature and ***continue moving the Z-stage SLOWLY*** to lower values (down to 10-11 mm on thin section or 1'' mount) until the image is sharp.
10. Increase the magnification and repeat the Z-stage adjustment until the sharpest image is obtained. **PROCEED with CAUTION:** Do NOT bump into the BSE detector when moving the stage up (= at a lower absolute Z-value)!

11. Continue increasing the magnification up to at least 2000x to 4000x, relocate an even smaller particle (~1 µm-sized), readjust the brightness & contrast, and perform a final adjustment of the Z-stage position; final Z-position ≈ 10-11 mm (on TS / 1'' mount).
12. Activate the button "Wobbler" (in the top section) and adjust manually the X- and Y-axis of the beam aperture until the particle does not move sideways (adjustment knobs are on the SEM column). If you see the particle stretching along one diagonal and then along the other diagonal, this is a sign of astigmatism (see next).
13. Once properly adjusted, deactivate the "Wobbler".
14. Correct for the astigmatism using the "Stigma X" and "Stigma Y" buttons on the top of the electron image, first with "coarse" adjustments (= right-click + hold, then move up or down), then with fine adjustments (= left-click + hold, then up or down).
15. If necessary, iterate between the Z-stage position and the astigmatism correction to improve the image resolution, and check with the "Wobbler" that all is well aligned.

F.3) Beam alignment for CL imaging

1. Set the signal to "SEI", the magnification to ~40x, and deactivate the "Beam Blank".
2. Set the acceleration voltage and beam current (default: 15 keV; spotsize ~74).
3. Turn ON the infrared camera.
4. Set the stage to Z = 30 mm, and **insert the CL detector** (***Section G.1*** and ***Fig. 14***):
    a. Unlock the mechanism; lock is below the arm of the CL detector.
    b. Turn the black knob clockwise to insert the CL detector inside the SEM, and simultaneously watch the IR camera *(do not bump into detector or sample!)*.
    c. Continue inserting the detector until you reach the backstop.
    d. You should now see the hole of the CL detector on the SE image.
5. Move the stage to the metal part of the sample holder; increase the magnification to ~100 or 200x so you don't see the edge of the CL detector anymore.
6. Locate a small particle or some marks intersecting at ~90°.
7. Adjust the brightness and contrast as necessary.
8. While looking at the infrared camera, move the Z-stage position to around 16-17 mm. With normal thin section or round mount, you can move rapidly up to 19-18 mm, and then *move slowly* towards **17-16 mm** *(16.5 is fine for regular thin section or 1'' round mount)*. Don't go below 16 mm! Adjust again the brightness & contrast if necessary. **WARNING: Do NOT bump into the CL detector!!!** ***After this, do NOT change the Z-stage position anymore!***
9. Turn OFF the IR camera.
10. Observe the SE image and adjust the electron beam focus using one of the following:
    a. Knob "Focus" on the console (with the option "Coarse" activated);
    b. Button "Focus" on top of the electron image (right-click for coarse change).
11. When the image appears to be on focus, increase again the magnification to 200 or 500x, and adjust the focus in coarse then in fine mode. Continue increasing the magnification up to at least 2000 to 4000x, relocate an even smaller particle, and fine tune the focus. The WD should now be around 16 or 17 (= matching the Z).
12. Activate the button "Wobbler" (in the top section) and adjust manually the X- and Y-axis of the beam aperture (knobs on the SEM column) until the particle does not move sideways. If you see the particle stretching along one diagonal and then along the other diagonal, this is a sign of astigmatism (see next). Deactivate the "Wobbler".
13. Correct for the astigmatism using the "Stigma X" and "Stigma Y" buttons on the top of the electron image, first with "coarse" adjustments (= right-click + hold ➔ move up or down), then with fine adjustments (= left-click + hold).
14. If necessary, adjust again the focus and re-iterate the astigmatism correction to improve the image resolution, and check with the "Wobbler" that all is well aligned.

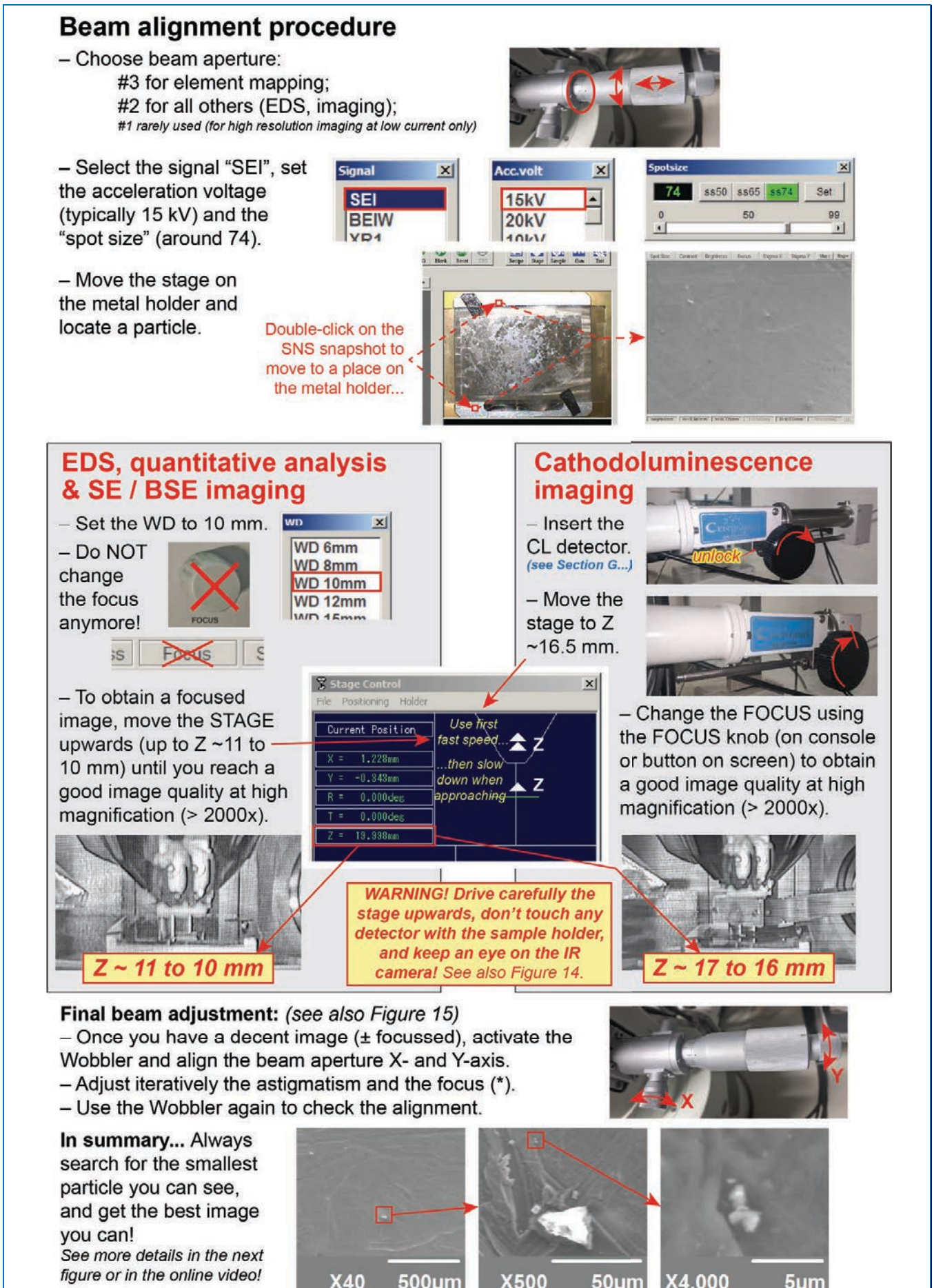

Figure 13. *Complete procedure for beam alignment.*

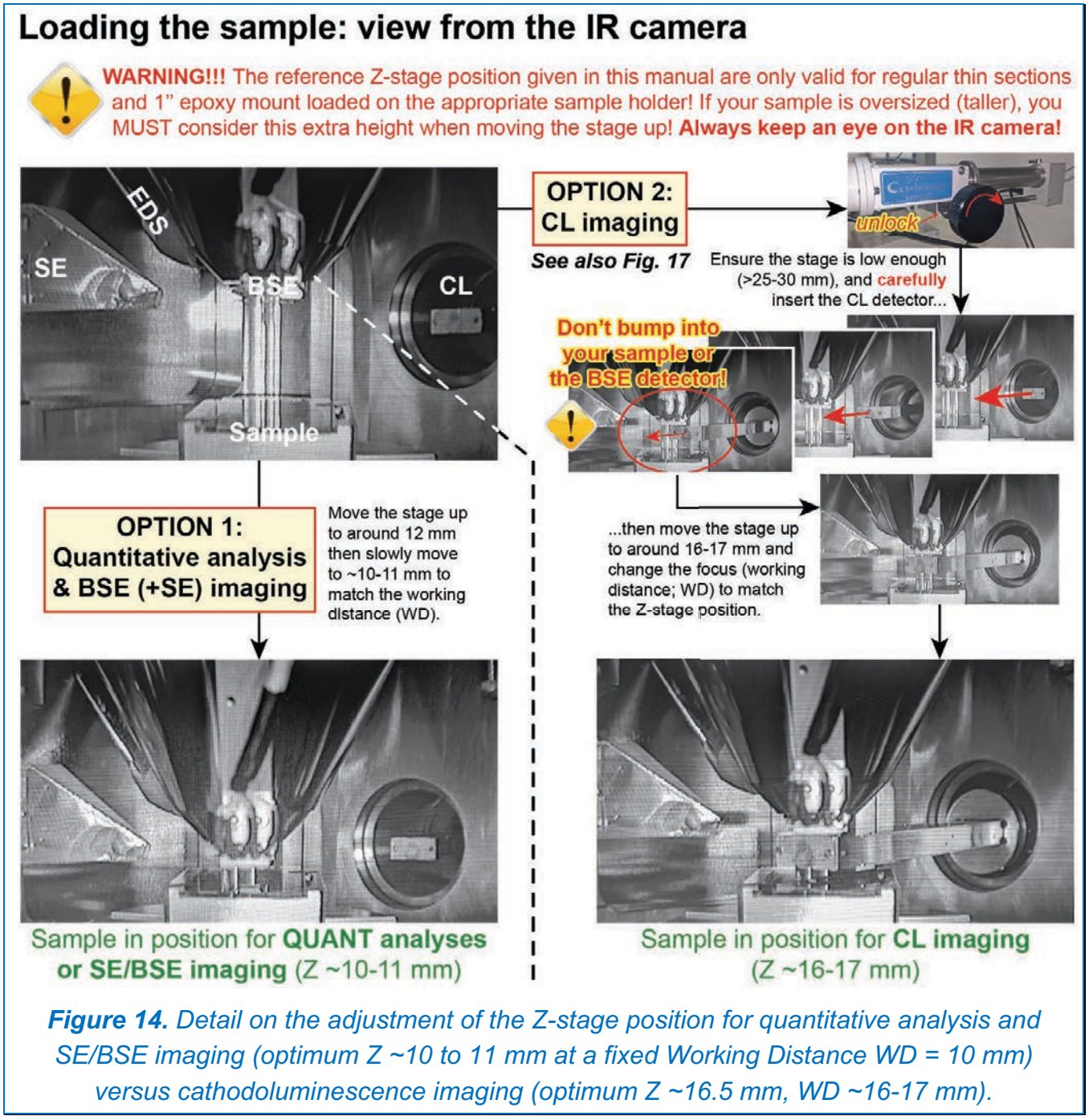

Figure 14. *Detail on the adjustment of the Z-stage position for quantitative analysis and SE/BSE imaging (optimum Z ~10 to 11 mm at a fixed Working Distance WD = 10 mm) versus cathodoluminescence imaging (optimum Z ~16.5 mm, WD ~16-17 mm).*

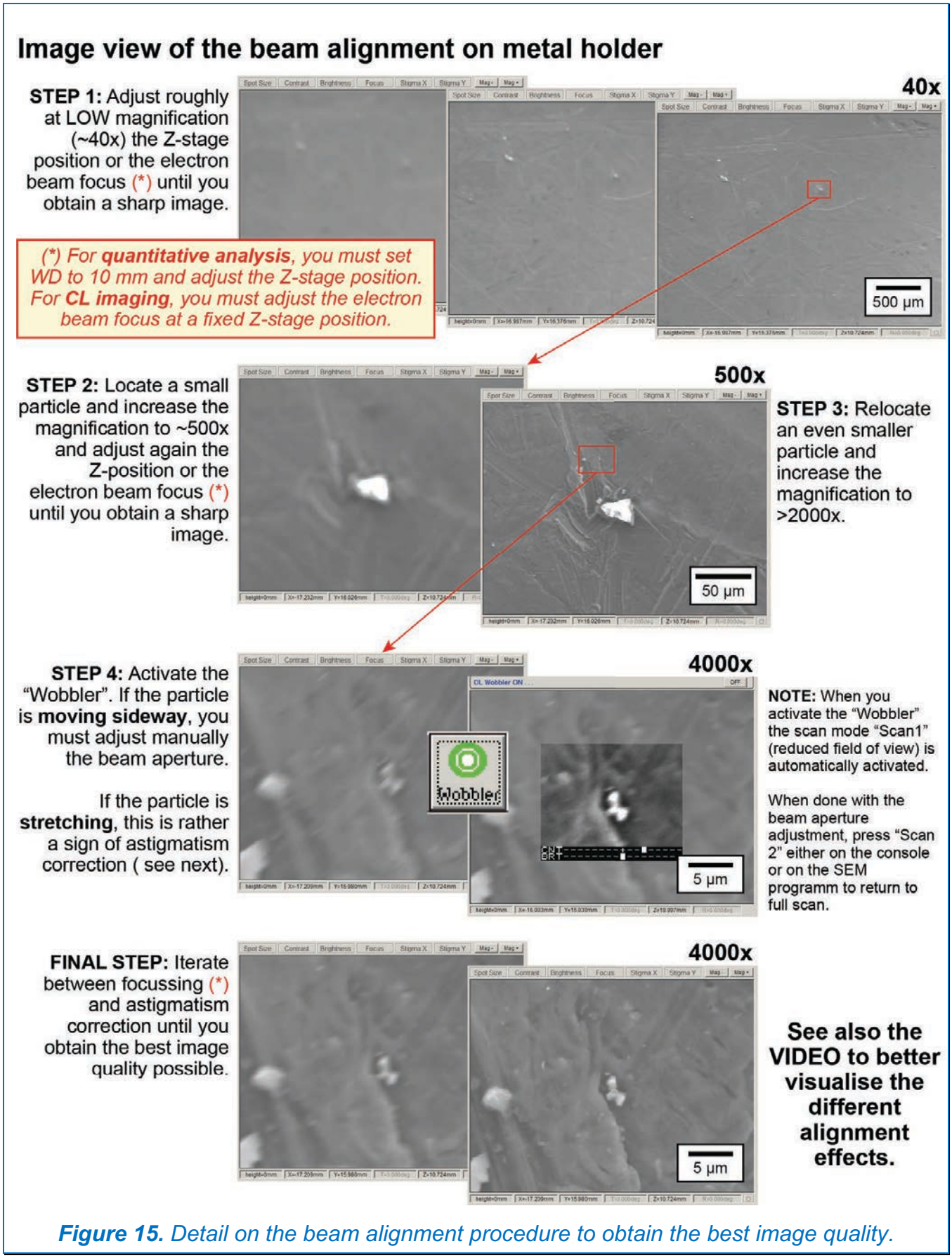

Figure 15. *Detail on the beam alignment procedure to obtain the best image quality.*

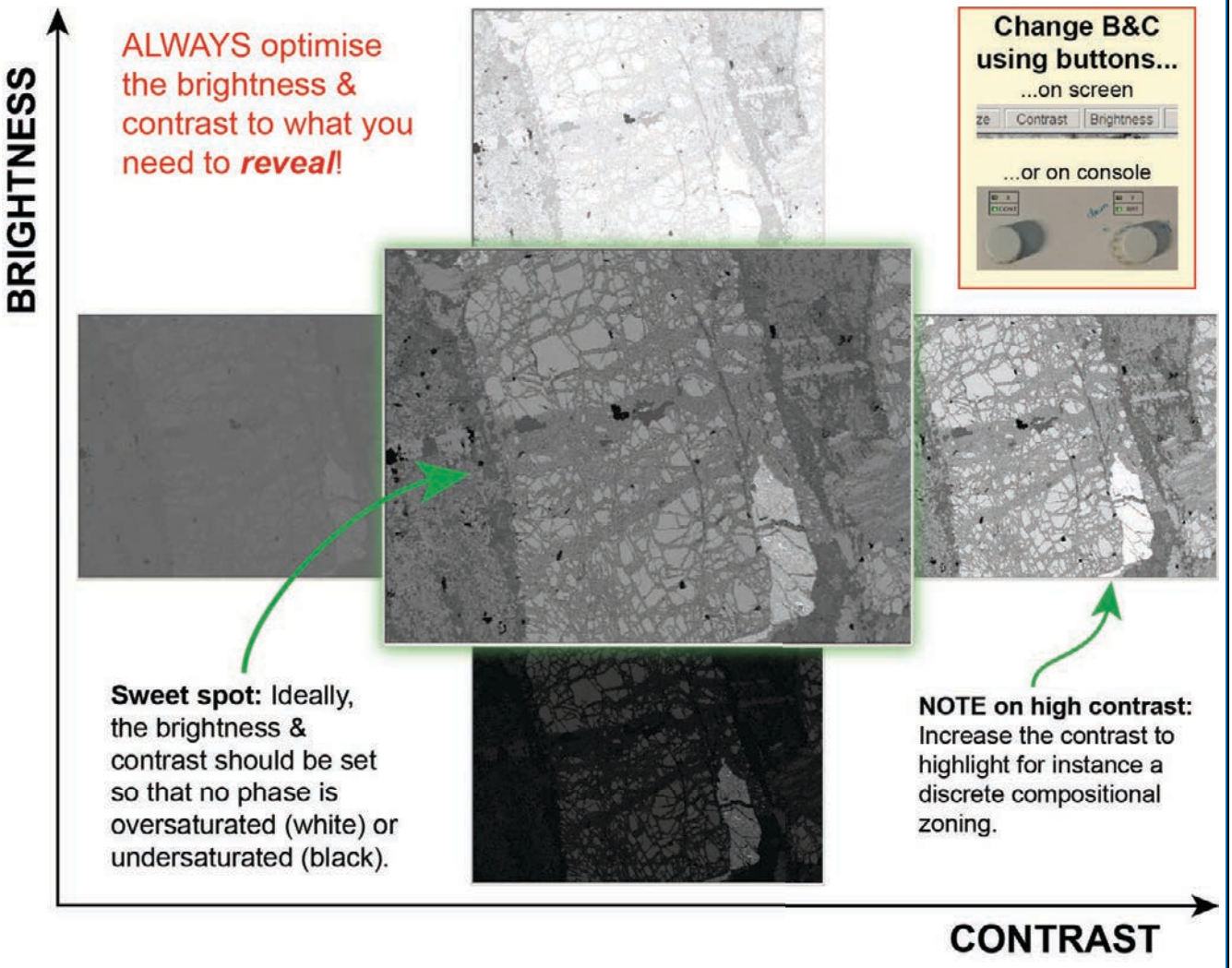

Figure 16. *Optimization of brightness & contrast. Constantly adjust B & C to reveal what you need to see. Avoid under- (black) or over-saturated (white) areas, unless a high contrast is required to reveal a small signal variation (e.g., discreet zoning, phase of similar density).*

G) Using the Cathodoluminescence (CL) detector

G.1) Inserting or removing the CL detector

WARNING! Whenever you ***insert*** or ***remove the CL detector***, ensure that the **Z stage position is at 30 mm**!

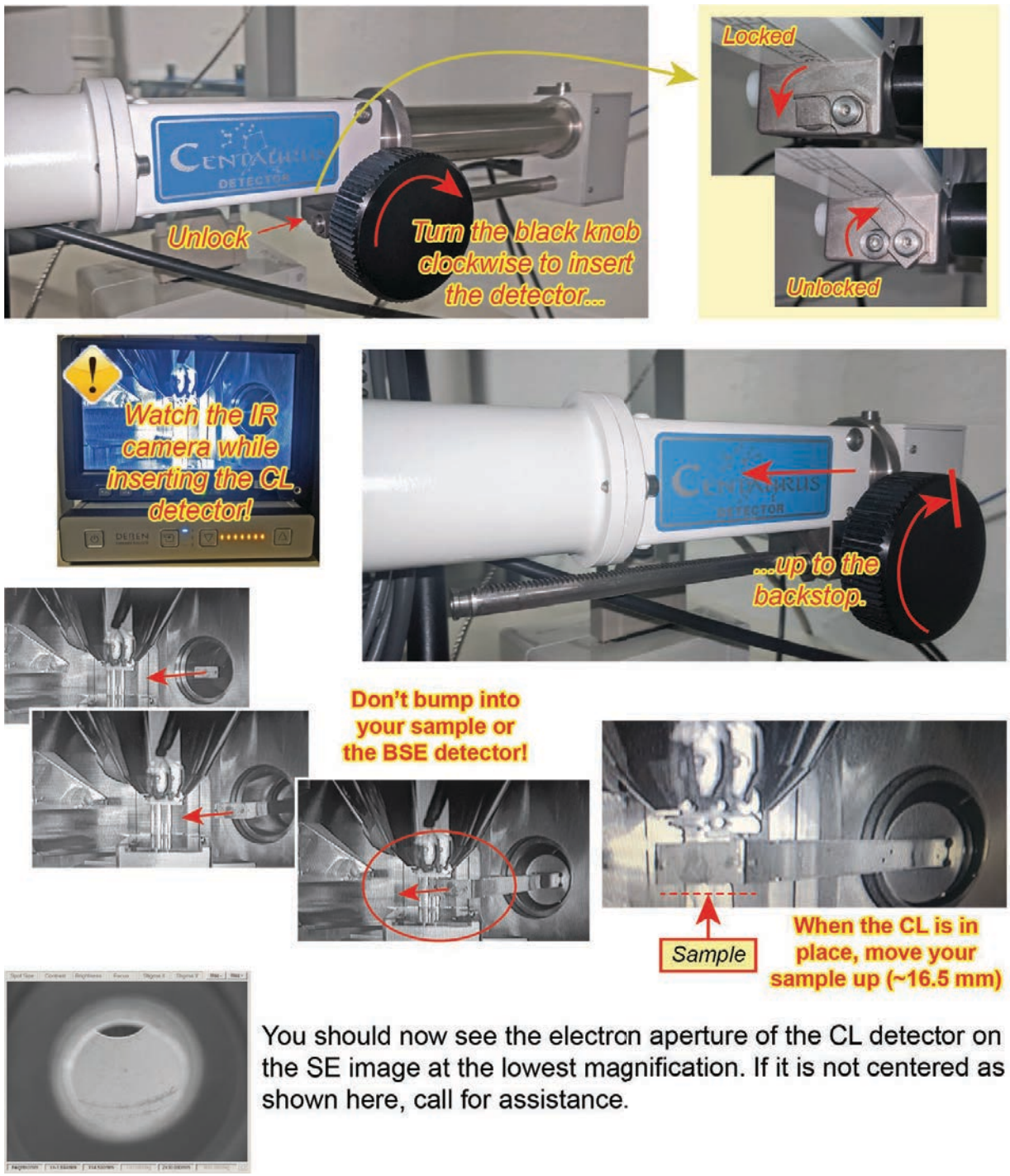

Figure 17a. *Inserting (or removing) the CL detector.*

WARNING! Ensure you have ***locked the CL detector*** when you are done!

G.2) Activating the CL detector

Once the CL detector arm is in place, proceed as following to activate the CL detector:

- Turn ON the IR camera.
- Move your sample close to the CL detector (usually between 16 and 17 mm for regular thin sections / round mounts). Always check the IR camera to avoid a disaster!
- Turn OFF the IR camera when the sample & detector are in place.
- Perform a beam alignment using the "SEI" mode (see ***Section F.3***).
- Change the signal to "BEIC" in JEOL SEM.
- Turn ON the CL detector (red button "POWER" on CL controller box).

IMPORTANT: Brightness & contrast (B&C) for CL image are first adjusted using the ***"Brightness" & "Contrast" buttons on the CL control box!*** Once the right B&C is found on the control box, then adjust the B&C of the JEOL SEM program.

Figure 17b. *Control box for the "Centaurus" CL detector.*

You can now start acquiring CL images with the Pathfinder software:

- To start Pathfinder follow the instructions given in ***Section H***.
- To acquire a single or mosaic image, follow the instructions given in ***Section I***.

WARNING! If the "OVERLOAD" button turns ON (Fig. 17b), it means that the detector is saturated with light. Either you forgot to turn OFF the IR camera or you activated the CL detector while the SEM is opened. Identify the source of this overload (turn OFF the IR camera, close and pump the sample chamber), and press the "RESET" button, or turn OFF the CL detector and ON again. If the problem persists, contact the lab manager.

G.3) Deactivating the CL detector

Any time you perform a **sample change or at the end of your session**, you **MUST deactivate the CL detector** and preferably **retract it** before removing your sample!

- Change the signal to "SEI" in JEOL SEM.
- Turn OFF the CL detector (red button "POWER" on CL controller box).
- Turn ON the IR camera and move the stage down to **Z = 30 mm (IMPORTANT!)**.
- Retract the CL detector (see ***Fig. 17***, just do it all backwards ☺).
- **IMPORTANT!** Lock the arm when the CL detector is fully retracted! If you cannot insert the lock, it is likely because the arm is not fully retracted.
- You can now safely do a sample change or remove your sample to end your session.

H) Pathfinder program

The Thermo "Pathfinder" program controls the EDS system and acquires electron images (SE, BSE, or CL). The JEOL SEM program can also acquire images, but it is not as easy to use and certainly not as versatile as the "Pathfinder" program.

Note that any data acquired by Pathfinder are stored in a specific "Project" folder on the "D:\" Data drive. Whenever you create a project, you must save it **IN YOUR OWN User Folder** such as ***"D:\LastName FirstName****Your project…".*

WARNING: Data backup is NOT guarantee. Data can be deleted at any time without any notice (especially old and heavy data: element maps, mosaic images, etc.)! **You MUST retrieve all your data and get a backup as soon as your analyses are over!**

H.1) Starting "Pathfinder"

Refer also to ***Figure 18***. It is assumed that…

- Your sample is loaded and set at the proper Z-stage position (***Section C***).
- The electron beam is active (***Section E***)
- The electron beam is aligned, and the optimum focus is reached (***Section F***).

- **Start the "Pathfinder" program** (if not already running). Shortcut on desktop: 
- When starting "Pathfinder", you will have to create or select a "Project" folder where all data will be automatically stored. It is recommended to **create a NEW project folder per analytical session**[1]…
  - If not existing, create a folder with your last name and first name…
    - Select the drive *"D:\"*;
    - Click on "New Folder" (yellow folder) to create a new sub-folder and name it with your *"LastName FirstName"*.
  - **Select your user data folder** *"D:\LastName FirstName"*.
  - Click on "Create New Project" to create a new "Project" folder (green folder).
  - Enter a name for your project, for instance "2022-04-19 PF test" or "Sample 79", click OK to create this project, and select it.
  - **IMPORTANT!** Choose the **project template "silicate calibrated analysis"** or a templated appropriate to your work! It will set some default parameters for your EDS & imaging setups, ideal for most situations. *If you forgot to set the project template, you must (a) re-open the "Project Explorer", (b) select your folder, (c) choose the correct project template, (d) press "Reset", and (e) press "Open Project".*
  - Click "Open Project".

- ***To OPEN an existing project*** (to append data to it or to re-process data)…
  - Click on button "Project Explorer"  if it is not opened.
  - Select the "Project" folder to use (green folder).
  - Click "Open Project".

[1] Some researchers such as experimentalists might consider creating a unique "Project" folder per experimental run. Just be aware that a lot of data stacked up in a single folder is not good when it comes time to find back something specific.

Otherwise, when creating a "per session" project folder, it can be useful to add the date when you acquire the data to the name of your project folder so you can link it to your notes in your laboratory book.

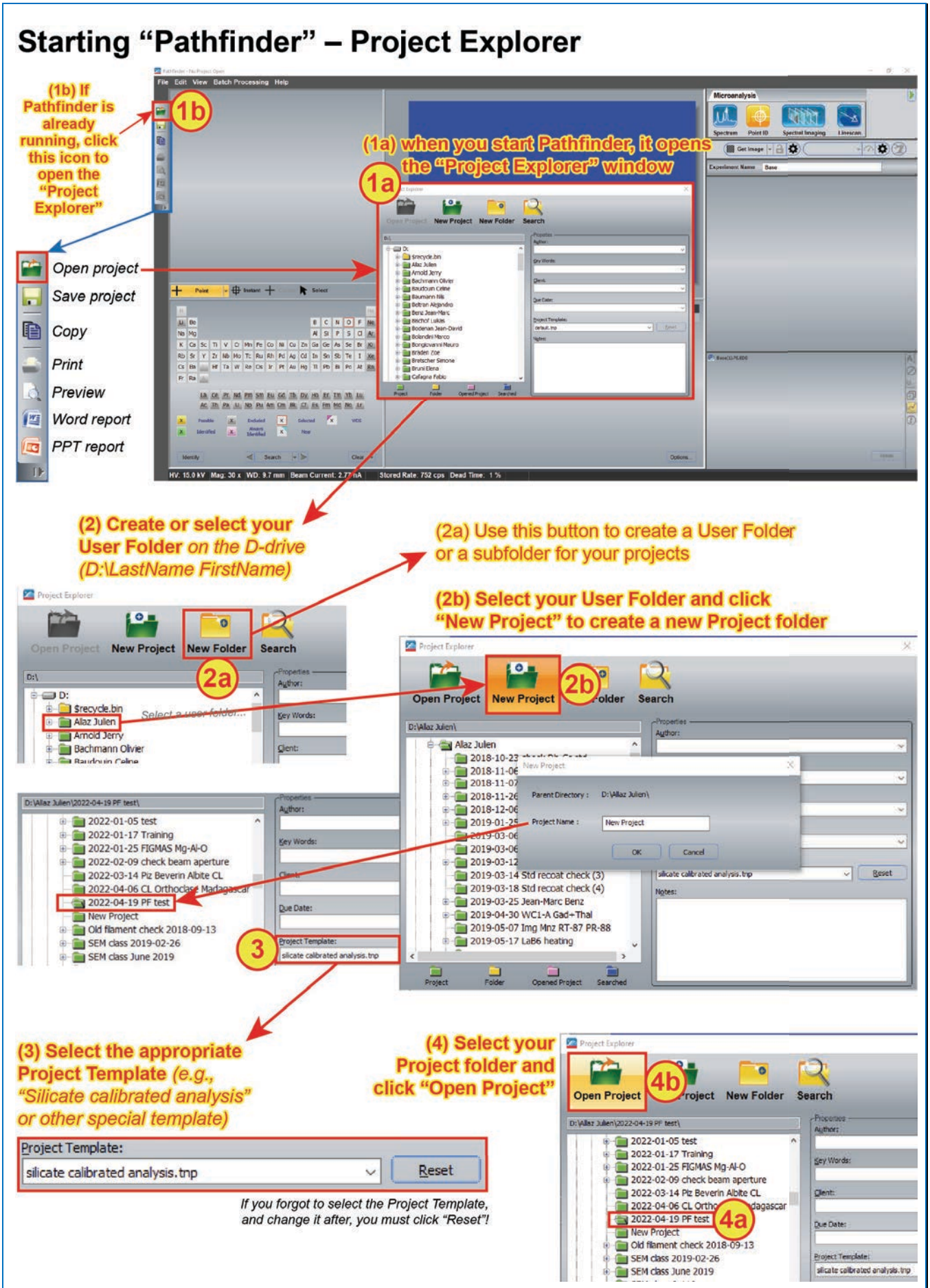

Figure 18. *Creating a new Pathfinder project (or opening an existing one).*

H.2) Overview of Pathfinder and the different analysis modes

The program Pathfinder consists of several panels described in ***Figure 19***. The present manual only covers the essential functionalities of the modes “Electron Imaging”, “Spectrum”, “Point ID”, and “Spectral Imaging”. For more details, ask for assistance.

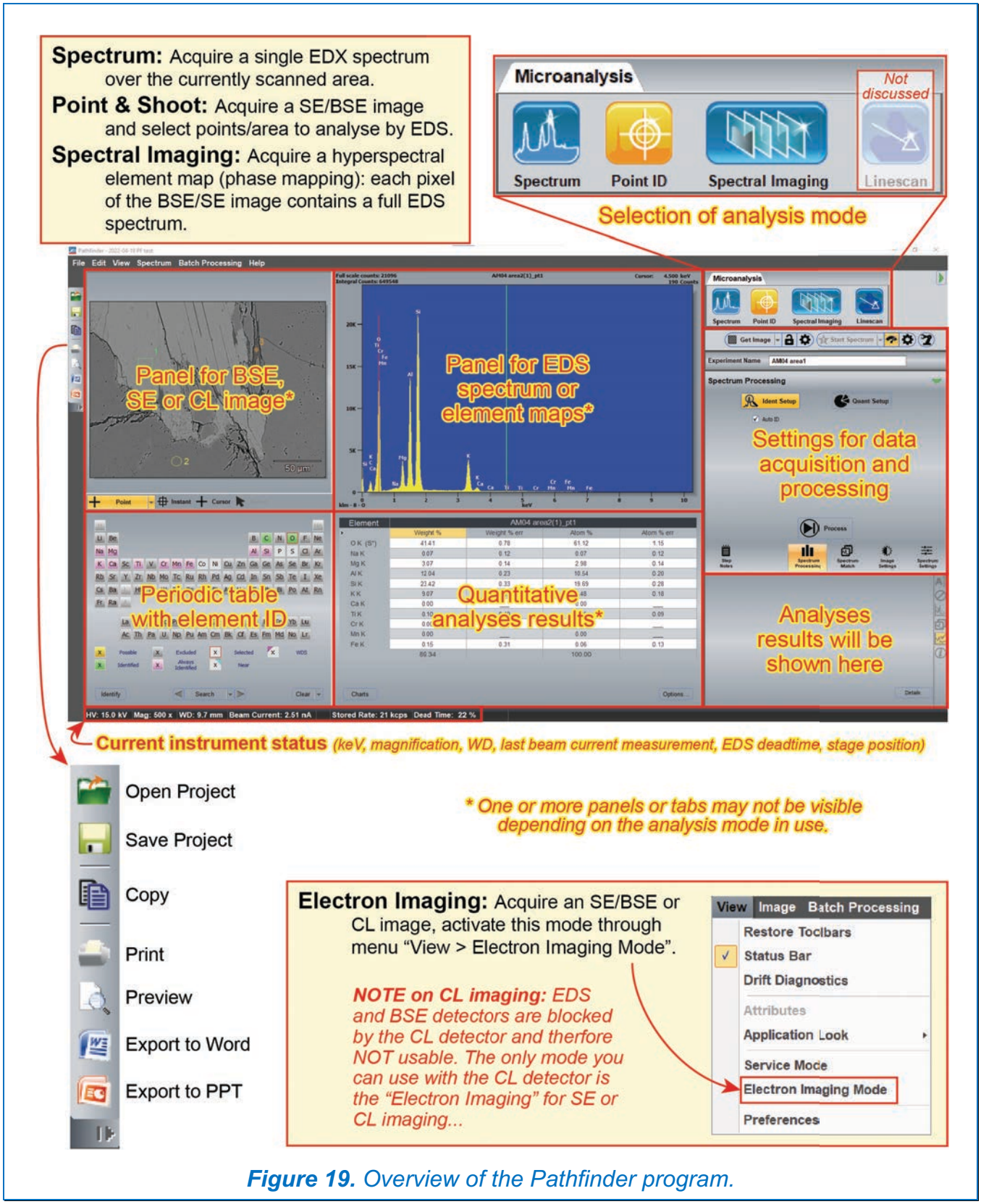

Figure 19. *Overview of the Pathfinder program.*

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I) SE, BSE, or CL imaging with Pathfinder

In the following, it is assumed that…

- The Thermo "Pathfinder" program is running.
- A Project folder is opened (see ***Section H.1***).

I.1) Acquiring an SE, BSE, or CL image

Adjust the **signal & field-of-view on the JEOL SEM program**…

- Select the desired electron signal: **SEI** for secondary electron, **BEIW** for backscattered electron (BSE), or **BEIC**[2] for cathodoluminescence.
  NOTE: If you choose the BEIW signal, select also the mode:
  - ***COMPO*** *(most common, compositional effect).*
  - ***TOPO*** *(topography ~ similar to SEI but lower quality).*
  - ***SHADOW*** *(mix of topography and compositional effect).*
- Adjust the stage position and set the magnification to the desired field of view. Avoid low magnification (< 70x) as you will have edge effects (shadow & deformation).
- Adjust the Z-stage position (for SE, BSE images at fixed WD) or the focus (for CL images) to get the sharpest image possible.
- Adjust the brightness and contrast (***Fig. 16***). For CL image, you must adjust the brightness and contrast directly on the CL controller box (***Fig. 17b***).

To acquire a single SE, BSE, or CL image (see also ***Fig. 20***)…

1. In Pathfinder, select **menu "View > Electron Imaging Mode"**.
2. Click on the gear button **"Electron Image Setup"**.
3. Enter a **name for your image** under ***"Experiment Name"***.
4. Set the image acquisition time (default: **2 frames, 10 s each**) and the image size (recommended: **1024 x 768**).
5. Click on the button **"Get Image"** to start the acquisition.
6. Optional: click on "Image Settings" to change the brightness and contrast.

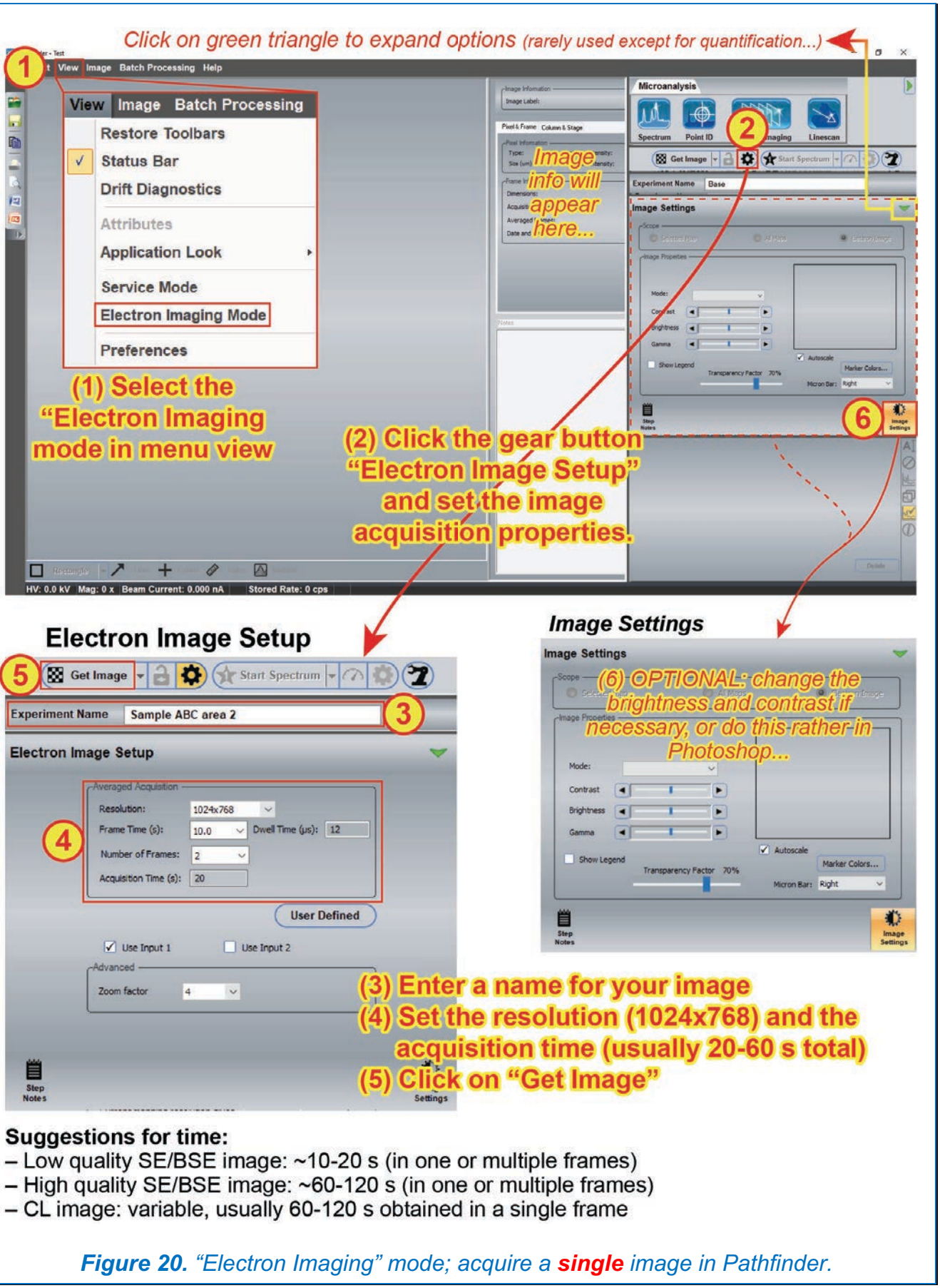
The image is automatically stored at the end of the acquisition, and an entry will be created in the bottom-right panel. You can then at any time click on one of the saved images to select and display it. If necessary, click on to rename the file or on to delete it.

Image information (including stage coordinate) are found in the middle panel.

Continue with the acquisition of the next image: move the stage in position, adjust the brightness & contrast as necessary, change the "Experiment Name" and click again "Get Image", etc.

At the **end** of your session, don't forget to reserve time to ***export your images*** with a scale bar by selecting menu "File > Export > Export as Full-Res TIF". If you have tens of images, this can take some time…

[2] *BEIC requires first that you insert and activate the CL detector! Refer to* ***Section G.1*** *for details.*



Suggestions for time:

– Low quality SE/BSE image: ~10-20 s (in one or multiple frames)
– High quality SE/BSE image: ~60-120 s (in one or multiple frames)
– CL image: variable, usually 60-120 s obtained in a single frame

Figure 20. *"Electron Imaging" mode; acquire a* ***single*** *image in Pathfinder.*

I.2) SE, BSE, or CL mosaic imaging

The mosaic imaging is a grid of X * Y individual images stitched together for imaging large areas (> 2 mm). In the following, ***it is assumed that you have set the image setting*** (resolution & acquisition time, ***see I.1 and Fig. 20***).

On JEOL SEM program:

- Select the desired electron signal: **SEI** for secondary electron, **BEIW** for backscattered electron (BSE), or **BEIC**[3] for cathodoluminescence.
  NOTE: If you choose the BEIW signal, also select the mode: ***COMPO*** *(most common, compositional effect),* ***TOPO*** *(topography, like SEI but lower quality), or* ***SHADOW*** *(mix of TOPO + COMPO).*
- Set the **magnification** (e.g., 70-150x for large areas, higher mag for more details).
- Adjust the Z-stage position (for SE, BSE images [at fixed WD]) or the focus (for CL images) to get the clearest and sharpest image possible.
- Adjust the brightness and contrast (***Fig. 16***). For CL image, remember to adjust the brightness and contrast directly on the CL controller box (below the IR camera).

To acquire a mosaic image in Pathfinder (Fig. 21):

1. In "**Electron Imaging mode**", click  to enter the "Automation" mode.
2. Enter an "Experiment Name" such as sample name with area ID, etc.
3. Select **"Grid"** under the ***"Acquisition Type"***.
4. Click on **"Add (Change) Location"** and follow the instructions:
   a. Move the stage to the ***upper-left corner*** of the mosaic image to acquire and adjust the Z-stage position (don't change the electron focus, only the Z-stage position if necessary!).
   b. Move the stage to the ***lower-right corner*** of the mosaic image to acquire and adjust the Z-stage position.
   c. Optional: Move the stage to ***another corner*** *and adjust the Z-stage position.*

   This will create an entry in the list below.
5. Click on **"Edit Location"** to modify it (e.g., adding an overlap).
6. In **"Edit Positions"** window you can…
   a. Set an overlap: 10% recommended to avoid black squares with no data.
   b. Update the magnification (e.g., after opting for a different magnification).
   c. Modify the grid label name, yet the default "Grid" name is usually fine (it will be appended to your "Experiment Name").
   d. Modify the two reference positions (upper-left and lower-right corners)
   e. Click "OK" to validate any modification to this grid acquisition.
7. Press **"Start Automation"** to launch the image acquisition.

GOOD NEWS! ***Compared to NSS, it is now possible to define more than one grid!*** It should automatically rename your second grid entry "grid_1", then "grid_2", etc.

WARNING: NEVER check the box "Beam OFF"!

[3] *BEIC requires first that you insert and activate the CL detector! Refer to* ***Section G.1*** *for details.*

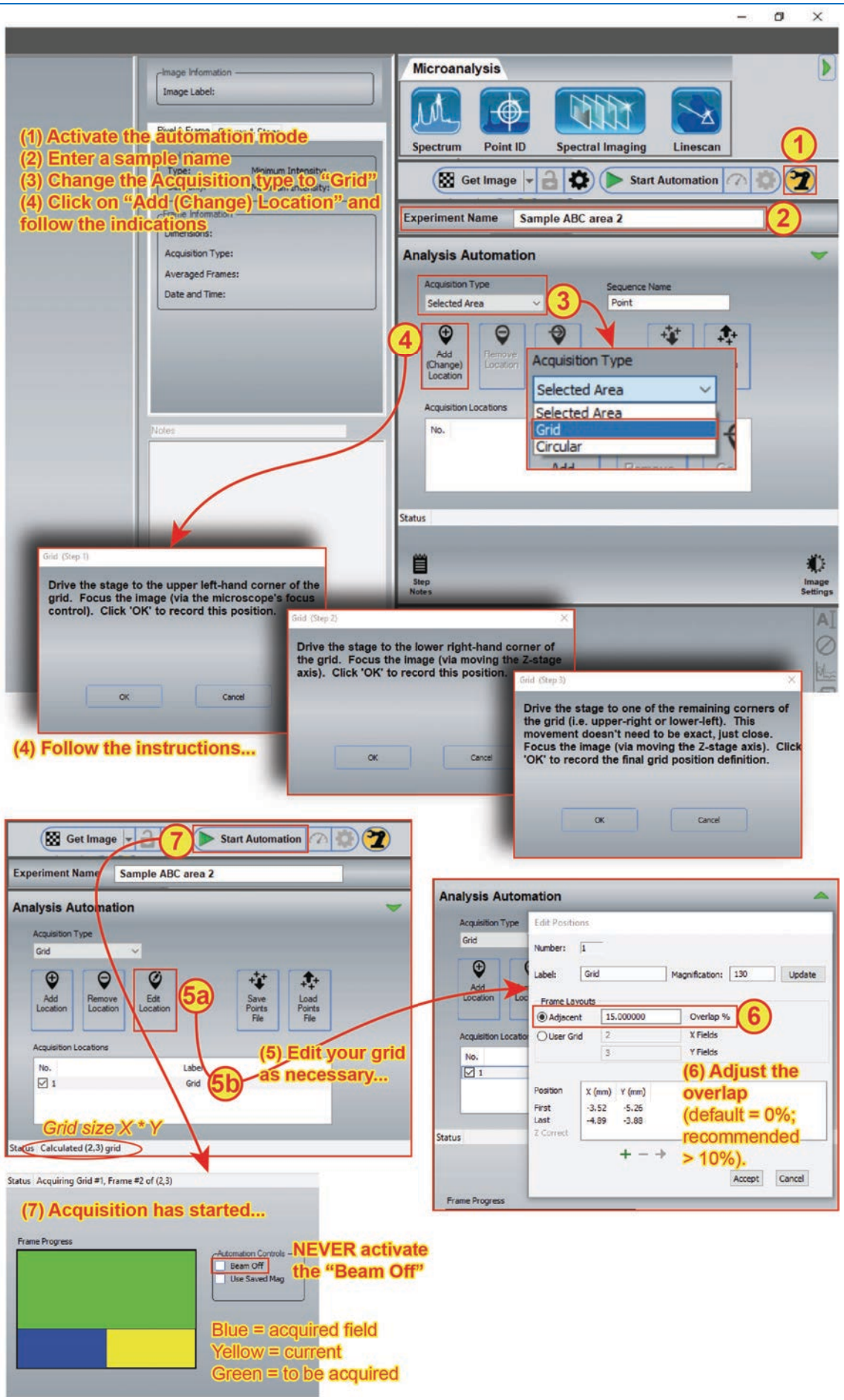


Figure 21. *"Electron Imaging" mode; acquire a* ***mosaic*** *image in Pathfinder.*

I.3) Tips on image acquisition time & magnification

- Always use aperture 2 *(or 1)* when acquiring images.
- You can try to increase/decrease the current and adjust the counting time. A **medium current** is recommended for most applications (spotsize 70-80) as it gives a ***strong signal*** and a ***decent sub-micron imaging resolution***. However:
    - A **lower current** (spotsize 50-70) will yield a *weaker signal* (➔ longer imaging time required) and provide a *higher spatial resolution* (<< 1 µm).
    - A **higher current** (spotsize >80) will *significantly increase the signal* but will yield a *lower spatial resolution* and is *NOT recommended for imaging*.
    - *NOTE: If a super-high spatial resolution in SE mode is required (e.g., for imaging foraminifera, small 3D-image of crystal), use a higher voltage (20 or 25 keV) and the beam aperture 1!*
- Prefer a **medium to high magnification** (> 100x) to avoid problems deformation at the edges or shadow effect from the BSE or CL detector or from the beam aperture.
- For mosaic images, **select a magnification sufficient to reveal all the feature to highlight** (i.e., consider the true pixel size in micrometre). Check the ***Appendix A4*** for determining the pixel size of one individual image. Do not choose a too high magnification that would require to acquire way too many images! For instance, a magnification between ~70 and 150x is commonly used for a mosaic image of a full thin section with large grains (> 50-100 µm), but a higher magnification and most likely smaller area would be required to reveal micron-sized phases in smaller areas.

I.4) Exporting images with scale bar

Images are stored immediately when the acquisition is over. However, you will still need to ***export individual images*** to get an image with a scale bar:

- Select the image (or Point ID) to export.
- Click on menu “File > Export > Export as FullRes TIF…”.

For mosaic images, you will first need to MERGE all the files by selecting the menu “Batch Processing > Create Montage”. Export then the resulting merged image as a full resolution TIF image (menu “File > Export as FullRes TIF…” as described above).

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J) Quantitative *(or qualitative)* EDS analysis

In the following, it is assumed that…

- The Thermo "Pathfinder" program is running.
- A Project folder is opened (see ***Section H.1***).

EDS acquisition can be considered as (a) "qualitative" for instance to check the presence or absence of some major to minor elements, or (b) "quantitative" providing you consider a standard-based quantification (which is what we do in our lab). The following assumes your goal is to get **standard-based quantitative** analysis:

- **"Spectrum":** acquire a single EDX spectrum of the currently scanned area.
- **"Point ID":** acquire an image and select point(s) or area(s) to be analysed by EDS in this image. This is most likely what you need…

WARNING: If your goal is to perform **quantitative analyses**, you MUST…

(1) Load the ***project template "Silicate calibrated analysis"*** if you want to run quantitative analyses (see ***Section H.1, Fig. 18***)! If you haven't set this template when creating a new project, click again on the "Project Explorer"  , select "silicate calibrated analysis", press the "Reset" button aside, and confirm.

(2) Ensure that you have set the **Working Distance (WD) to 10 mm**. If this is correct, Pathfinder will indicate in the footnotes ***WD 9.7 (at 15 keV)*** **or** ***9.8 (at 20+ keV)***.

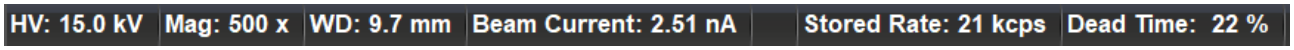


Figure 22. *SEM & EDS information found at the footnote of the Pathfinder program:* ***acceleration voltage*** *(HV),* ***magnification*** *(Mag),* ***working distance*** *(WD),* ***last beam current measurement****,* ***stored count rate****, and* ***dead time****.*

J.1) Load standard data

If your goal is to obtain **accurate quantitative data**, you must load the appropriate standard data to obtain standard-based quantitative analysis (see also ***Fig. 23***):

1. Select your analysis mode (e.g., "Spectrum" or "Point ID").
2. Click on "Spectrum Processing".
3. Click on "Quant Setup".
4. Click on "Standard Manager…".
5. Click on "Load" to load a set of standards. Alternatively (5a), you can select an individual standard in the list and click "Add selected". (5b) To remove a standard from your list, select it and click "Remove selected".
6. Select the latest standardisation for your type of analyses (e.g., silicate, carbonate) and click "Load".
7. Validate your choice of standard.
8. Your choice of standards will be displayed in the "Quant Setup" and elements will be highlighted in purple in the periodic table (i.e., always quantified, even if not present).
9. If you already have acquired data, press the "Process" button to re-quantify the data.

Verify this list is correct. You can also look at the periodic table in the bottom-left panel: all elements that have a standard defined are highlighted in purple (see ***Fig. 23***).

WARNINGS on the choice of standards / elements:

- *You MUST select standards acquired at the same VOLTAGE as your analyses (usually 15 kV)!*
- *If multiple standards are available such as Fe_2O_3 and FeS_2 for Fe at the same kV, choose the one the most similar to the type of phase to be analysed.*
- *The standardization on the EDS holds well for months and it is tested periodically. However, if you think it needs a new standardization, ask for assistance.*
- *It is better to **select your standards BEFORE acquiring any quantitative data!** If you have already acquired data and you change the list of standards, you will have to reprocess all your data, and this can be time consuming…*

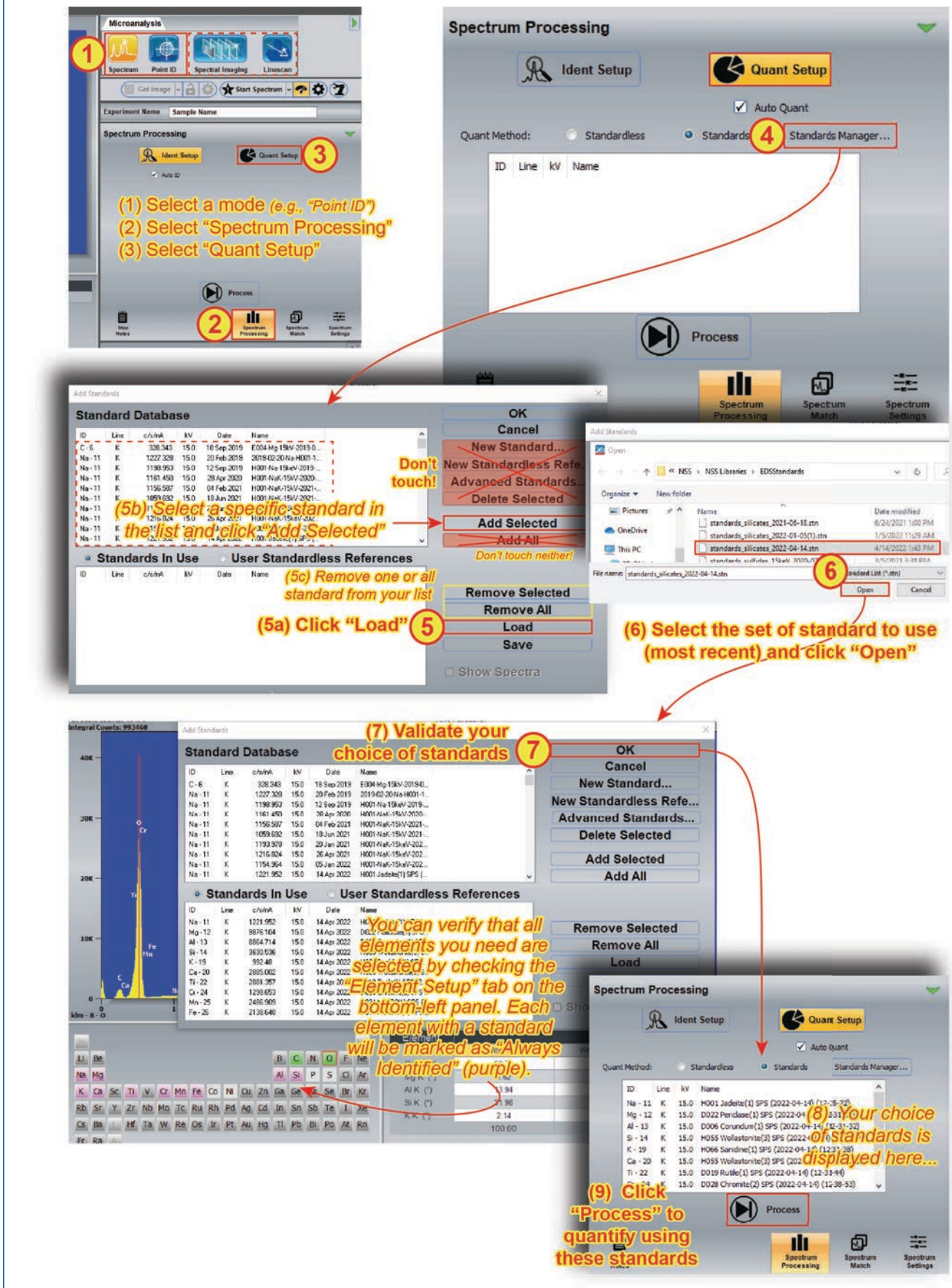


Figure 23. Loading standards into your project for quantitative EDS analysis.

J.2) Single Spectrum

J.2.1) Acquiring a single EDX spectrum

To acquire a single EDX spectrum (***Fig. 24***)…

1. Choose the analysis mode “Spectrum”.
2. Click on the right gear button to set the EDS acquisition conditions:
    a. Recommended “Live Time Limit”:
        i. 5 to 10 s for quick ID.
        ii. 30 to 60 s for quantitative analysis (30 s = default).
        iii. Considering a longer acquisition time? ➔ EPMA analysis / ask for help.
    b. Leave the default values for the Energy Range (0 to 20 keV), unless you are working above 20 keV (then choose “40 keV” for the high energy cutoff).
    c. **IMPORTANT for QUANTITATIVE analysis: “Time Constant”** must be set to **“Rate 2”** as all standards are acquired using this time constant.
    d. Ensure button **“Beam current”** is active.
3. Move the stage in position for the analysis, fine tune the Z-stage position, and increase the magnification (usually > 1000x) to scan only over the phase of interest.
4. Adjust the current (spotsize) to reach a deadtime (DT) of 20-30%, which is shown in the footnote of Pathfinder (***Fig. 22***). Decreasing the current will reduce the amount of X-ray produced, and thus the DT.
5. Enter an “Experiment Name” for your analysis, e.g., “SampleABC area # phase XX”.
6. Click on “Start Spectrum”. The remaining acquisition time will be indicated in a green progress bar on the top of the program window. Remember that the “real” acquisition time is the sum of the “live” time (when the instrument is processing the signal, as defined in 2 above) and the deadtime (DT, when the instrument cannot process the signal).
7. Process your data by clicking on “Spectrum Processing”.

See details for quantitative analysis in the next *Section J.3* about Point ID.

J.2.2) Navigating in the Spectrum panel

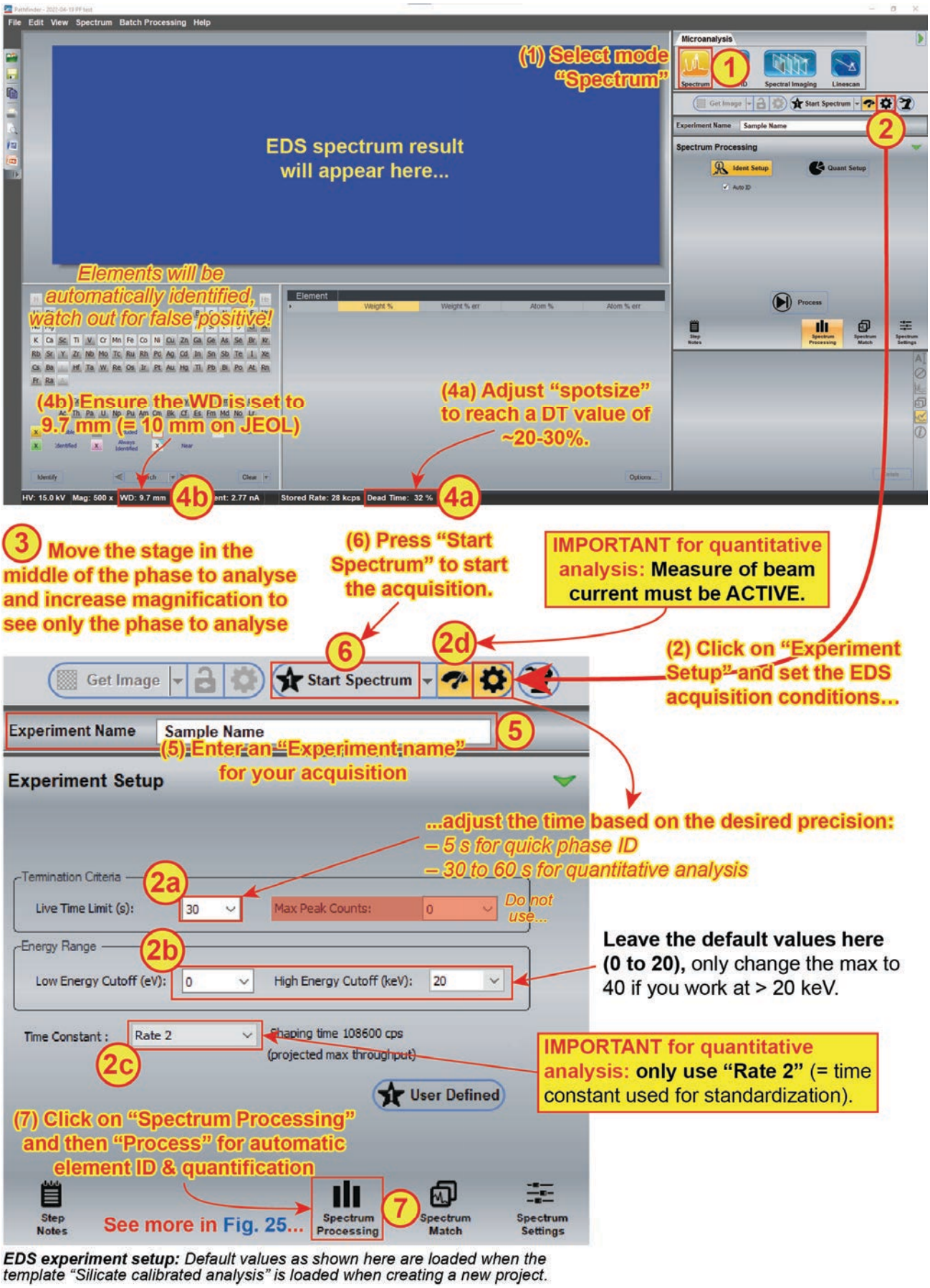
EDX spectrum acquisition is shown in one panel. To “navigate” in this spectrum…

- **Click-and-drag** horizontally (or vertically) to change the energy range (or the intensity range). If you reach the minimum, the spectrum will be expanded (zoom).
- **Double-click** on the spectrum to ***return to the default*** full view.
- ***To zoom-in and zoom-out*** on a specific energy position, place the cursor over the energy range of interest and use the **mouse-wheel**.

J.2.3) Exporting quantitative results for one or more single spectrum

To export quantitative analysis from a single spectrum:

- Click on menu “Batch Processing > Spectrum Analysis…”.
- Select all EMSA files to be treated, using CTRL to select individual files or SHIFT to select continuous series of files, and click “OK”.
- Choose a filename and location for the result file to be created.
- Click “Save”. A CSV file with all quantitative data for the selected EMSA file is created.



EDS experiment setup: *Default values as shown here are loaded when the template "Silicate calibrated analysis" is loaded when creating a new project.*

Figure 24. *"Spectrum": Acquiring a single EDS spectrum over the currently scanned area.*

J.3) Point ID

J.3.1) Acquiring Point ID analyses

To acquire multiple EDS analyses in a given area with a BSE image (***Fig. 25***)…

1. Select "Point ID" on the analysis mode panel.
2. Click on the left-gear **"Electron Image Setup"** to set the imaging conditions (see ***Section I.1; Fig. 20***), usually 2 frames of 10 s each at 1024 x 768 pixels resolution.
3. Click on the right-gear **"Experiment Setup"** to set the EDS acquisition conditions (see ***Section J.2; Fig. 24***), e.g., 60 s acquisition time at time constant ***"Rate 2"***.
4. If you are running ***quantitative analyses***, ensure the required **standards** are loaded:
    a. Select "Spectrum Processing" and "Quant Setup".
    b. Click on the green arrow to expand the setting options.
    c. Check the list of standard and modify it if necessary (see ***Fig. 23***).
    d. Ensure the options ***"Filter Fit"*** and the ***"Proza"*** correction method are set.
    e. **If analysing oxide:** Check the box ***"Calculate all elements as compounds"***, and set the ***number of oxygen atoms*** for a cation normalisation. Otherwise, uncheck it.
5. Move to an area of interest and set the magnification ≥ 500x. At lower magnification, you will get inaccurate results on points acquired near the edges.
6. Ensure WD is 9.7 mm (at 15 keV), and adjust the spotsize to reach 20-30% DT.
7. Enter a name for your analysis under "Experiment Name".
8. Acquire an image by clicking on "Get Image".
9. Set one or more points, rectangle, spot, or polygon areas on the BSE image:
    a. Ensure the "Instant" option is deactivated.
    b. Select the shape you need (rectangle, spot, polygon…).
    c. Draw shape(s) to be analysed on the BSE image.
    d. **WARNINGS: (1)** For accurate results, ***avoid setting points to close to a phase boundary*** or a crack. Keep also in mind that the actual analysed volume is probably a sphere of 1-2 µm diameter! **(2)** For ***beam sensitive phases*** (hydrous, carbonate, alkali-rich, etc.), it is best practice to analyse a "large" area (➔ 2-3 µm-sized rectangle or circle). **(3)** Always set your shape in a ***homogeneous domain***. Accuracy is NOT guaranteed for inhomogeneous domain.
10. When ready, click "Start Spectrum". Progress of EDS analysis will be indicated by the green bar on the top of the program window.
11. Quantitative results will be displayed in the bottom-middle panel (see ***Fig. 26***).

J.3.2) Reprocessing Point ID data

To review the acquired results (***Fig. 26***)…

1. Select the point of analysis in the list of results or use the "Select" tool.
2. Quantitative results for the selected point are displayed. Click on "Options…" to choose the data to be displayed (see ***Section J.3.3*** for details on each option).
3. You can activate the "Comparison mode" to compare two or more spectra. Choose to normalize the data to the live time or to a specific element. You can also activate the "calculated spectra" for synthetic, background, or residual spectrum.
4. To export in a CSV file your quantitative analysis, select menu "Batch Processing > Point ID Analysis", and select all Point ID results to export. Validate your choice and enter a name for the CSV file.
5. You can also choose the Word or PPT export (on the far left-side of Pathfinder window) to generate a report with the EDS spectra.

During the analysis or if you changed the list of standards linked to your project, you will need to force reprocessing the data manually:

- Click on "Identify Setup" under "Spectrum Processing" and click "Process". This will force re-identifying all X-ray peaks / elements.
- Click on "Quant Setup" and click "Process" to re-quantify the data.

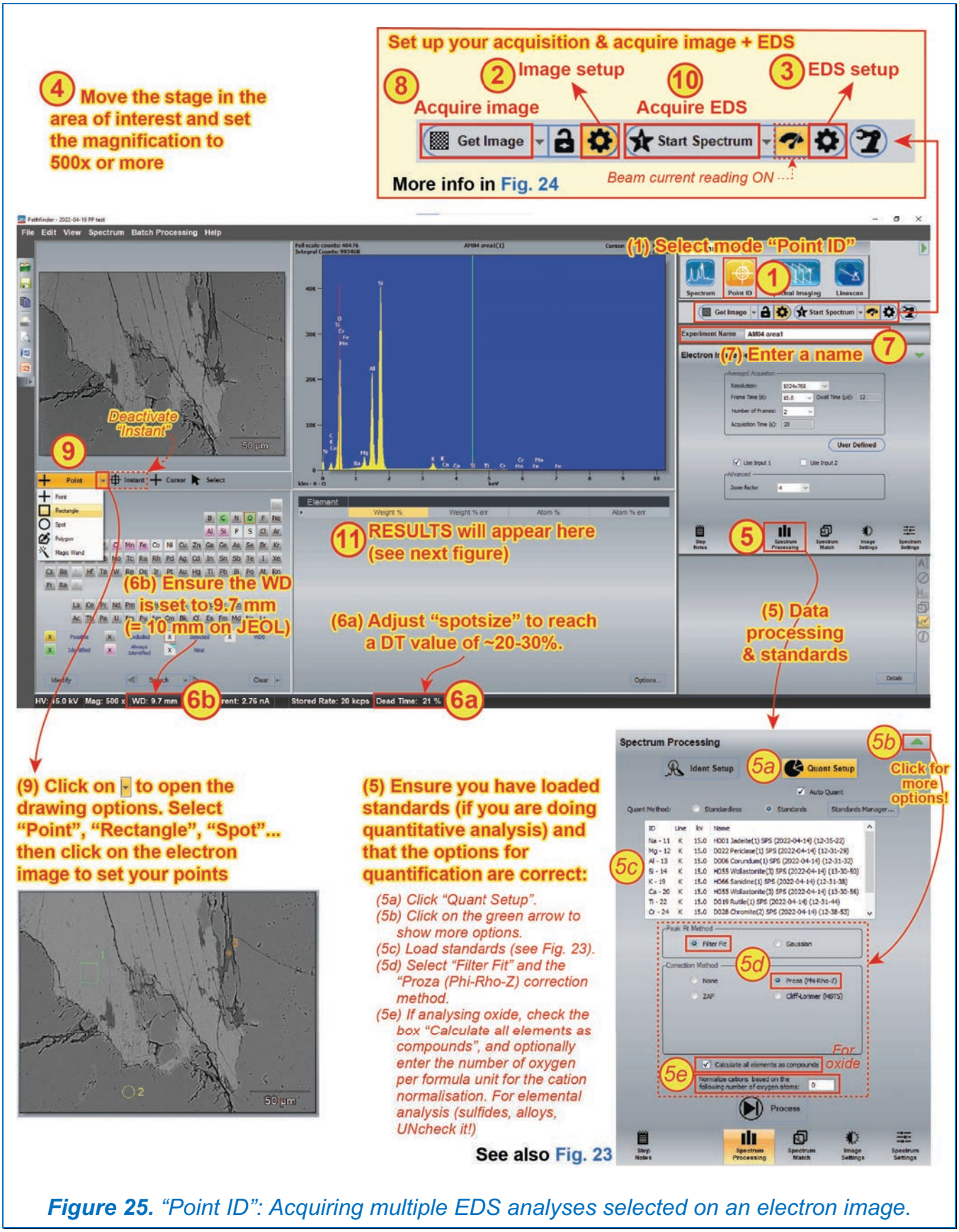


Figure 25. *"Point ID": Acquiring multiple EDS analyses selected on an electron image.*

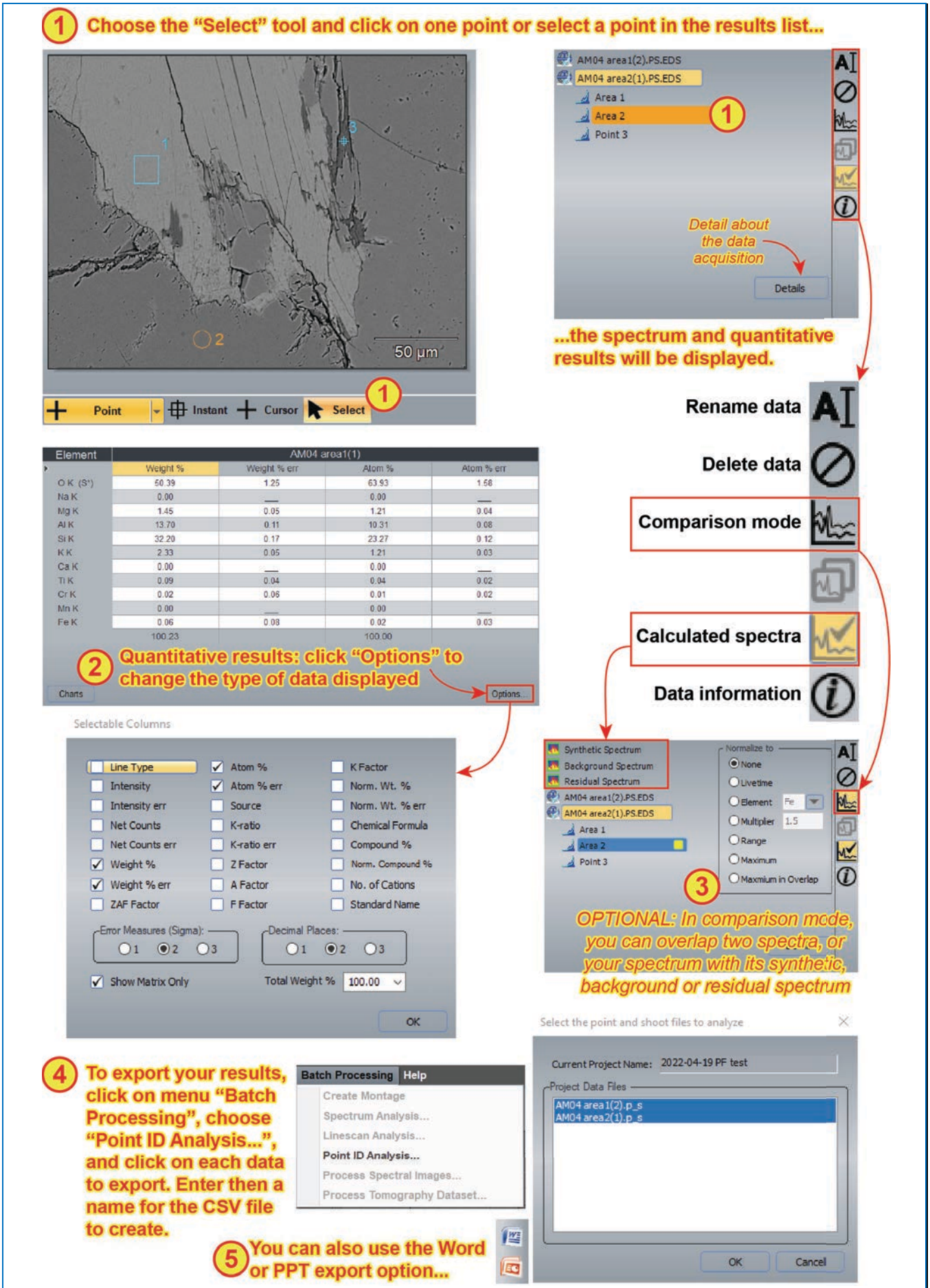


Figure 26. *Processing and exporting results from "Point ID".*

J.3.3) Choice of data to display and to export

You can choose the data to be displayed or exported by clicking on “Options…” in the quantitative results panel. The following entries are available (in **bold are the most important entries**, and *in small italic are the rarely used ones*):

- *Line Type*
- *Intensity, Intensity Error: Total peak intensity (peak and background, with error).*
- *Net Counts, Net Error: Background corrected peak intensity (with error).*
- **Weight-%, Weight-% Error:** Results expressed as element weight-%.
- *Z, A, F, and ZAF factor: matrix correction factors.*
- **Atom %, Atom % Error:** atomic proportion assuming a total of 100 atoms.
- *Source*
- *K-ratio, K-ratio Error: Ratio of measured net intensity over standard net intensity.*
- *K-factor: Calculated K-ratio assuming a pure element standard.*
- *Norm. Wt %, Norm. Wt % err, Norm. Compound %: Data normalized to 100%.*
- **Chemical Formula:** List of the compounds name (= oxide name).
- **Compound %:** Results expressed as oxide weight-%.
- **No. of Cations:** Atomic proportion from the oxygen normalisation (see ***Fig. 25, 5e***).
- *Standard Name: Name of the standard file used (usually written in your lab notes).*

You can also set the **uncertainty confidence level** (usually set to 2 sigma) and the **number of decimal places** (usually left at 1 or 2).

J.3.4) Probelab ReImager

Probelab ReImager is designed to read your project folder and export in a few clicks all your Point ID images with scale bar & EDS analysis points to a TIF file. You will need a 64-bit Windows or MacOS platform.

Get more info about it and download it here: https://reimager.probelab.net/

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K) EDS element mapping

WARNING: For element mapping, **use the beam aperture #3** (for high current)!

The **"Spectral Imaging"** option of Pathfinder (***Figs. 27 to 30***) is used you to acquire element maps and to perform a phase analysis. It is recommended for determining the modal proportions of minerals over a small area (50 – 1000 µm) for complex or fine-grained material, for a phase map over a larger area (> 1 mm). The following describes only the basics for acquiring and treating a spectral image using the capabilities of Pathfinder. It is recommended to **use the package "iSpectra" written for "IgorPro" by Dr. Christian Liebske** to treat your element maps.

Mapping a large area: You must use the analysis automation option to obtain a **mosaic element mapping**. The way you set this up is very similar to a regular mosaic imaging: set up first your element mapping conditions (see ***Section K.1***) and use the "Automation" tool to prepare the grid for your mosaic mapping (see ***Section I.2***).

K.1) Acquiring an element map with the mode "Spectral Imaging"

To acquire a single element map (see ***Fig. 27***)…

1. Select the mode "Spectral Imaging" and enter a name for you acquisition.
   OPTIONAL: *In the bottom-left panel select "Element Setup" and mark your element of interest as "Always identified" (purple-coloured). If you have standards loaded, the related elements will be already marked as "Always identified".*
2. Move to the area of interest, adjust the Z-stage focus, and set a magnification ≥ 70x.
3. Set the analysis conditions:
   a. **"Electron Image Setup":** Set the condition for the base BSE (or SE) image (e.g., 1024x768 pixels, 2 frames, 10 s each; see ***Section I.1, Fig. 20***).
   b. **"Spectral Image Setup":** Set the time constant between Rate 5 and Rate 7. For a higher spectral resolution, opt for ***rate 5***. For a higher throughput (higher current, more X-rays), opt for ***rate 6 or 7***. Set the ***resolution of your element map***, the number of frames, and the time per frame. Refer to ***Appendices A4 & A5*** for choosing the optimum resolution & analysis time or ask for assistance. *Suggestion: 15 to 30 frames, 60 s per frame, 256 x 192 px.*
4. Enter a name for your element map under "Experiment Name".
5. Ensure WD is 9.7 mm (at 15 keV) and adjust the spotsize to reach 20-30% DT. At a rate of 5 to 7 (= medium time constant), you should reach a stored rate of ~50'000 to ~80'000 counts per second (= 50 to 80 kcps).
6. Click on "Get Image" to acquire a BSE (or SE) image.
7. When the image acquisition is over, click on "Start Map" to start the acquisition.
8. During the acquisition, the buttons "Stop Map", "Abort" and "Pause" appear:
   - "Stop Map" will stop the acquisition at the end of the current frame and save the map.
   - "Abort" will stop immediately and won't save anything.
   - "Pause" will temporarily pause the acquisition.

   The remaining acquisition time is displayed on the top. Elements present in the mapped area are automatically detected the using the full EDX spectrum. Element maps of the "Identified" (green) and "Always identified" (purple) elements are extracted. When the acquisition is finished, add/remove elements using the periodic table and request to reprocess the data to extract more elements (see ***Section K.2***).

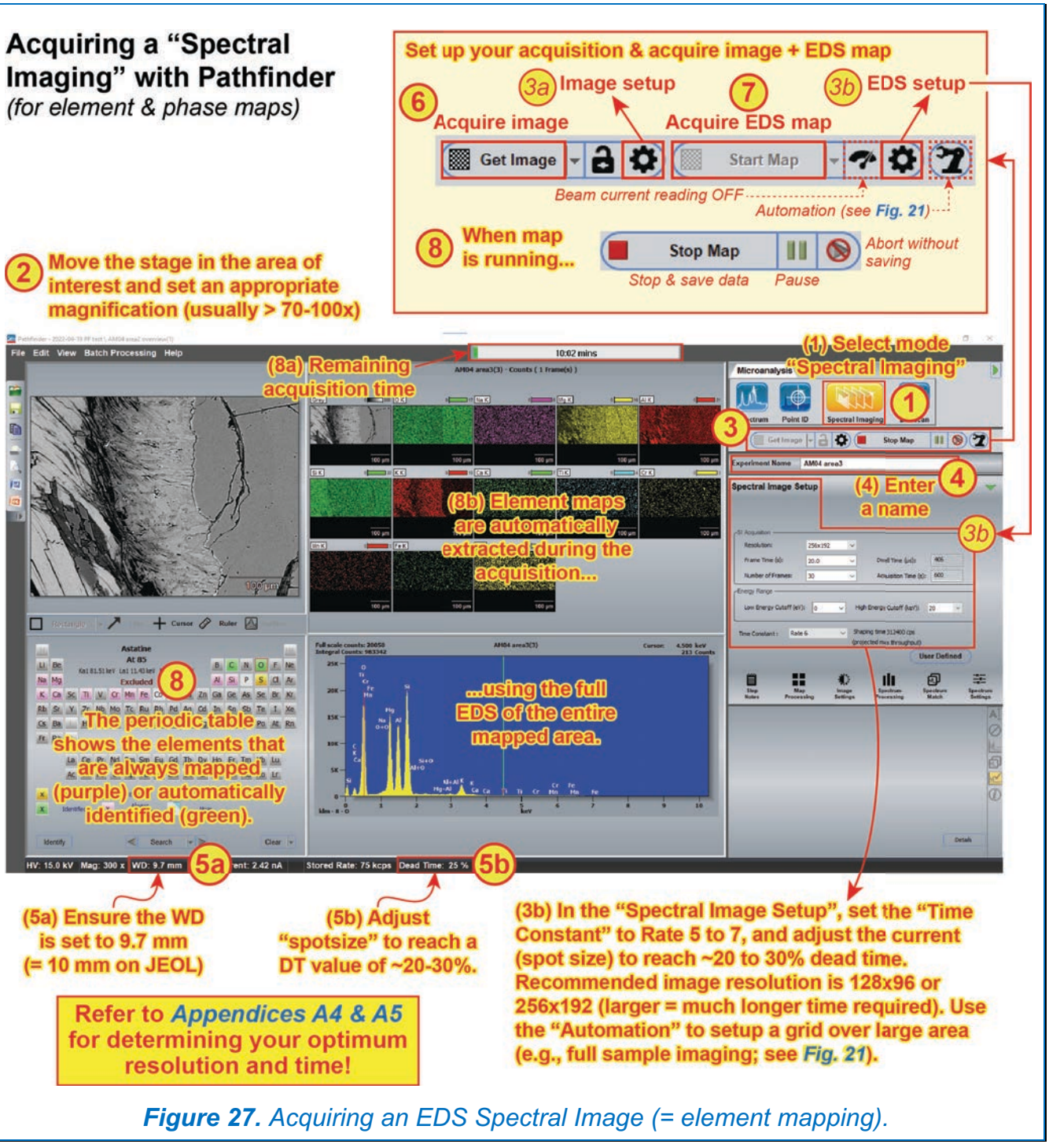


Figure 27. *Acquiring an EDS Spectral Image (= element mapping).*

K.2) Treating element or phase maps

K.2.1) Extracting "Element maps"

To extract element maps (see ***Fig. 28***)…

1. Select your Spectral Imaging acquisition in the bottom-right panel.
2. Mark the element you absolutely need for your research as "Always identified" to force their identification. Note that if you loaded some standards, the associated standard elements will be already marked as "Always identified".
3. Click on "Map Processing" (middle-right panel) to change the map treatment options.
4. Select "Quant" to display quantified data (watch out for low precision results!):
    a) Select in the list the type of data to export (atomic %, weight %, etc.).
    b) Change the "Output resolution" and "Processing Accuracy" to a higher quality setting if needed (longer processing time), or just use the default. To improve counting precision (to the cost of loss of spatial resolution), set the "Kernel Size" to 3x3 or higher (pixel averaging). This is recommended to smooth a map that would be too noisy (low counts per pixel).
    c) Click "Process", and wait for the calculation to complete.

See ***Section K.3*** for additional options once the element maps are extracted.

K.2.2) Extracting "Phase maps"

To extract phase maps (see ***Figs. 28 & 29***)…

1. Select your Spectral Imaging acquisition in the bottom-right panel.
2. Mark the element you absolutely need for your research as "Always identified" to force their identification. If you loaded some standards, the associated standard elements will be automatically marked as "Always identified". Watch out for false positive, your pixel statistics is very poor!
3. Click on "Map Processing" (middle-right panel) to change the map treatment options.
4. *….quant… see above*
5. To extract phase maps… (see also ***Fig. 29***)
    a) Select "COMPASS". If the "Phases" button is available continue to (b), otherwise click first on "Process" using the default options:
        i. Method set to "Area"
        ii. Background set to "Use Internal Model").
    b) Click on "Phases" and click on "Auto" using the default Phase Map Type (Maximum Intensity). The processing can take a few minutes depending on the options selected. See ***Figure 29*** for details on how to extract phase maps.
    c) Once the phase maps are generated, you can…
        i. Single-click on a phase name to activate or deactivate its overlay on the base BSE (or SE) image.
        ii. Double-click on a phase name to rename it (when identified).
        iii. Click on an image to change its setting (colour, brightness, contrast…)
        iv. Select "File > Export > Export Image to Full-Res TIF" to export the BSE/SE image with phase overlaps.

K.2.3) Extracting and quantifying a "Spectrum" from of an element map

To extract a spectrum, for instance, to properly identify a phase (see ***Fig. 30***)…

1. Activate the "Rectangle", "Spot", or "Polygon" option under the BSE image. With the rectangle option, move and resize the rectangle to the area of interest.
2. The sum of all EDX spectra from the selected pixels is shown as an "Extracted Spectrum" in the bottom panel, which should ease your phase identification.

3. *OPTIONAL: If you want to save this extracted spectrum as a single EMSA file…*
    a) Select the area with the rectangle / spot / polygon tool.
    b) Select the analysis mode “Spectrum”.
    c) The “Extracted Spectrum” should be displayed.
    d) Select menu “File > Save as…” and save the spectrum as an EMSA file.
    e) You can also quantify this spectrum (providing you are using standards) as you would quantify a point analysis (in “Spectrum” or “Point ID” mode). Be careful, the analytical precision is likely to be poor as there are very low counts on each pixel, even after pixel-averaging, unless you have hundreds of pixels included!

K.3) Additional tips on map treatment after extraction

- To overlay one or more map(s) on the BSE image, select or unselect an element or phase map by clicking on its name (background will turn black when selected).
- In “Phases maps” mode, click on any of the phase map to show the “Extracted Spectrum” of all pixels composing this phase. Be aware that this option will likely include pixels from other phases surrounding the phase of interest…
- Double-click on the name of a phase to rename it once you have properly identified it. Use the spectrum extraction tool to help with the identification (***Section K.2.3***)!
- To export the results after setting the overlay maps (elements or phases) on your BSE image, select menu “File > Export Image as Full Res TIF”.
- All (last) map treatment you perform (element or phase maps) will be automatically saved, and some of these files are already TIF files (images!). Look at ***Appendix A6*** for a listing of the file handling and signification of each file extension.

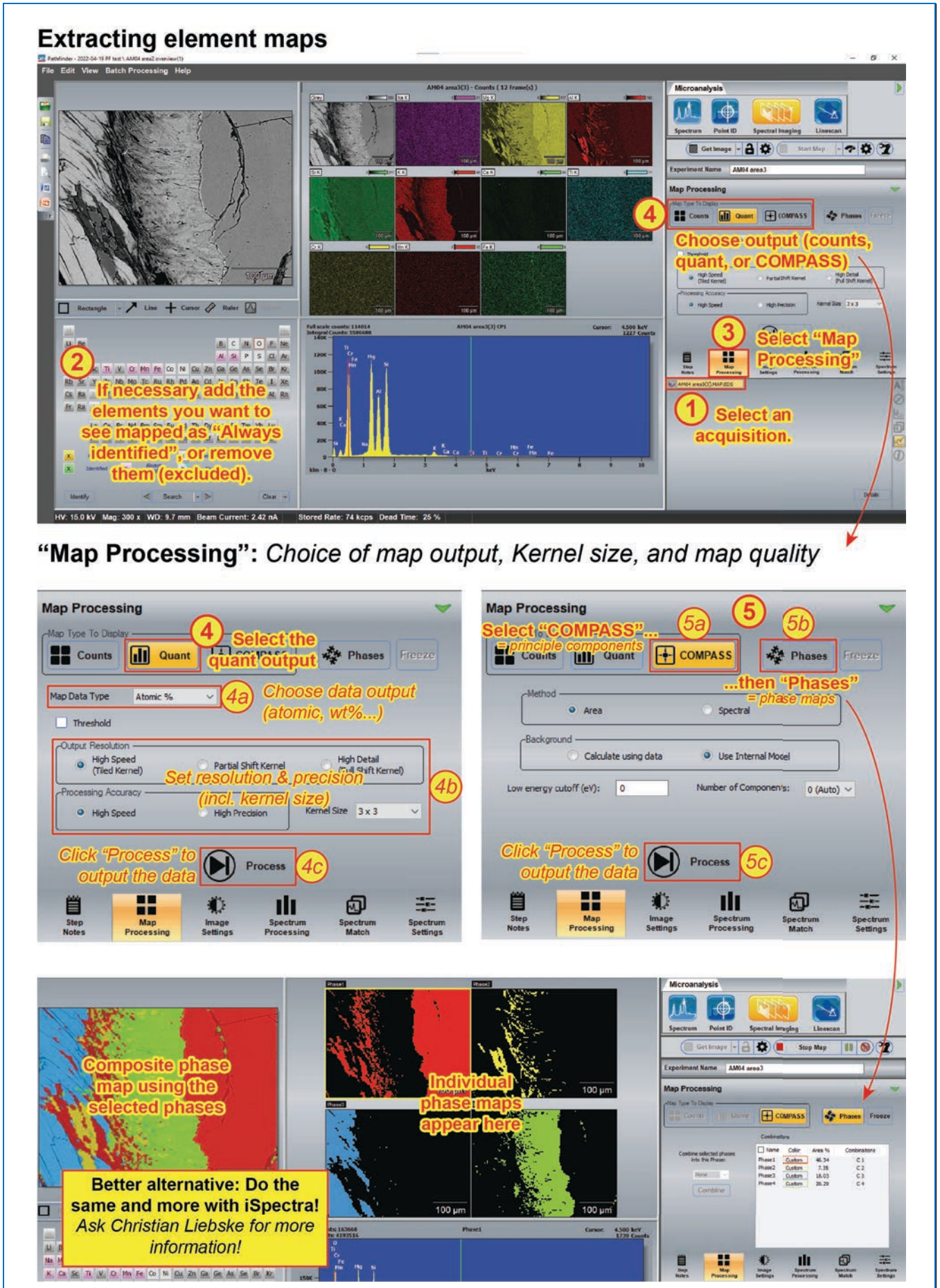


Figure 28. *Processing and extracting element maps.*

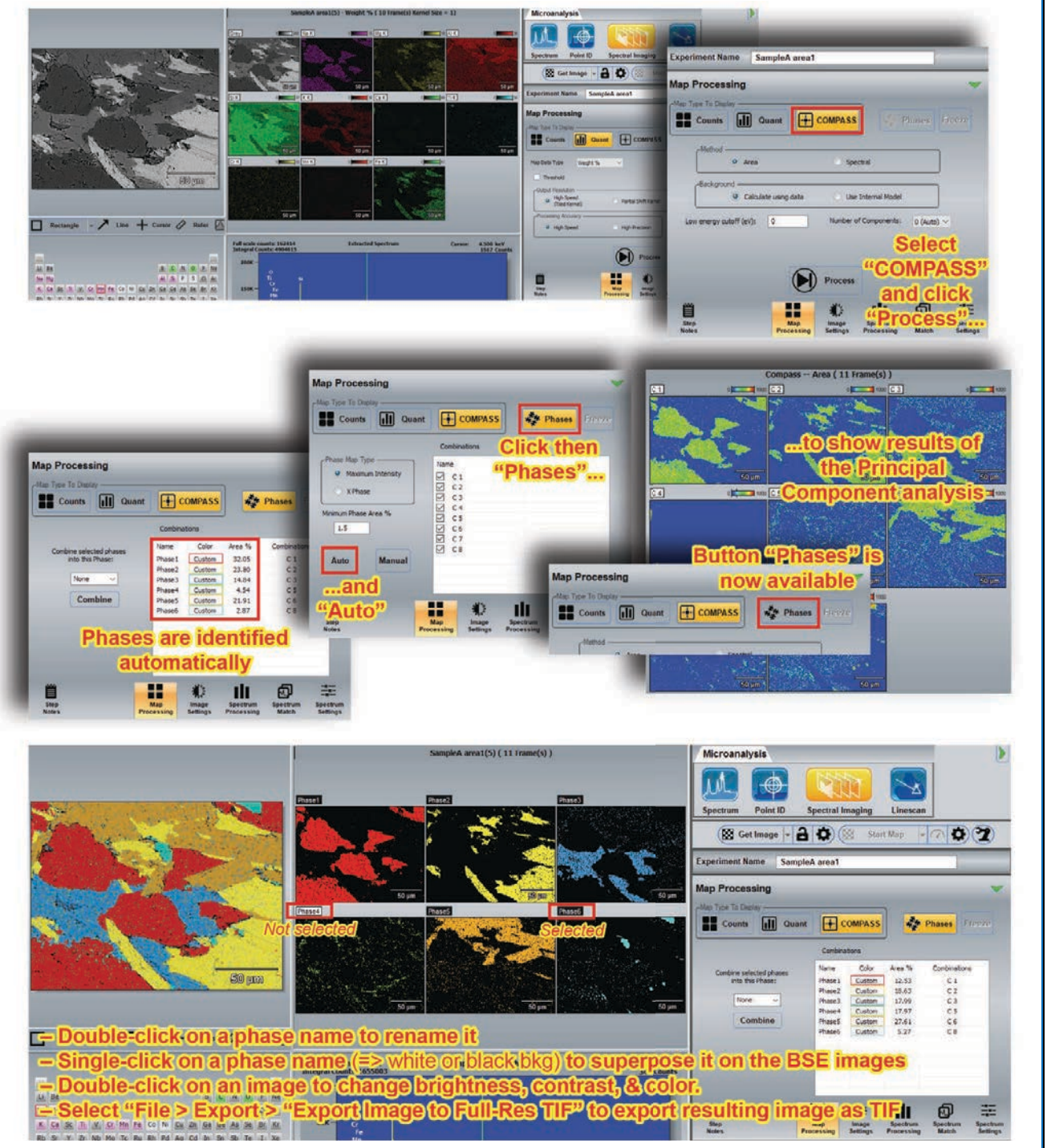


Figure 29. *Calculating and extracting phase maps.*

Extracting a spectrum from the element map

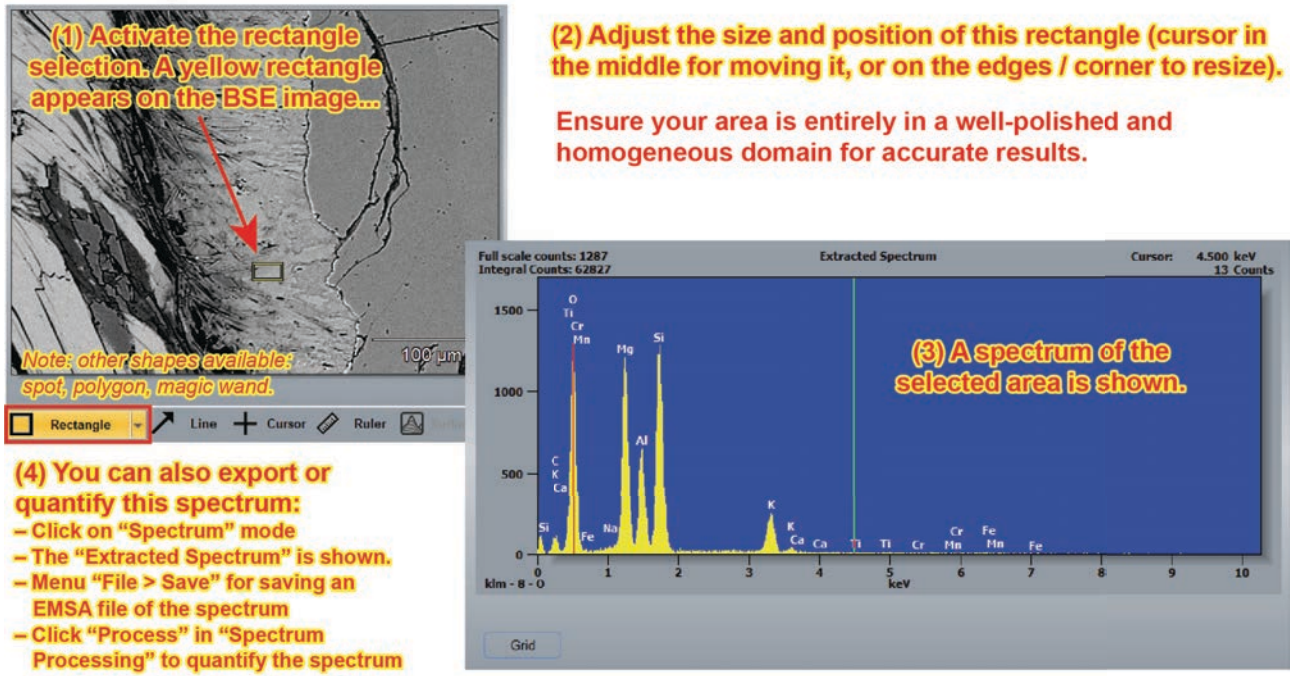


Figure 30. *Extracting a spectrum from an element map.*

Appendix

A1) Adjusting the SNS (Sample Navigation System)

The SNS image is not very accurate, yet it should drive the stage within a couple millimetres of the target. The calibration of this system is regularly checked by the manager. However, if you want to check and possibly adjust it (because someone screwed it up…), please, **strictly** follow this procedure:

- ***You MUST load the 32 mm sample holder*** *(for round mount)****. NEVER use the THIN SECTION holder****, as it will result in a wrong calibration of the SNS!*
- Ensure the ***sample chamber is fully opened***, up to the back stop.
- ***You MUST first press the button "Snap"*** *before "Adjust"!* It will move the stage and take a snapshot.
- **An adjustment is only required if the sample is not centred** (yellow line NOT in the middle of the stage holder). To perform the adjustment (see also ***Fig. A1***):
  - Press the button "Adjust". Ensure the Holder Size "32 mm" is selected.
  - Adjust the X and Y position of the stage using the button on the console (in X/Y mode) and recentre the holder (yellow line is in the exact middle of the 32 mm holder.
  - Move the horizontal and vertical green lines so that it matches the edges of the round mount.
  - Try rotating the sample using the rotation knob. If the adjustment is properly done, the sample should remain in the centre and the green lines should always touch the round mount edges.
  - Press "Set" when the holder is perfectly centred.
- Press again "Snap" to take another picture with the new adjustment, and then "OFF" to turn off SNS.
- ***Lower again the stage to Z = 30 mm before closing the chamber!***

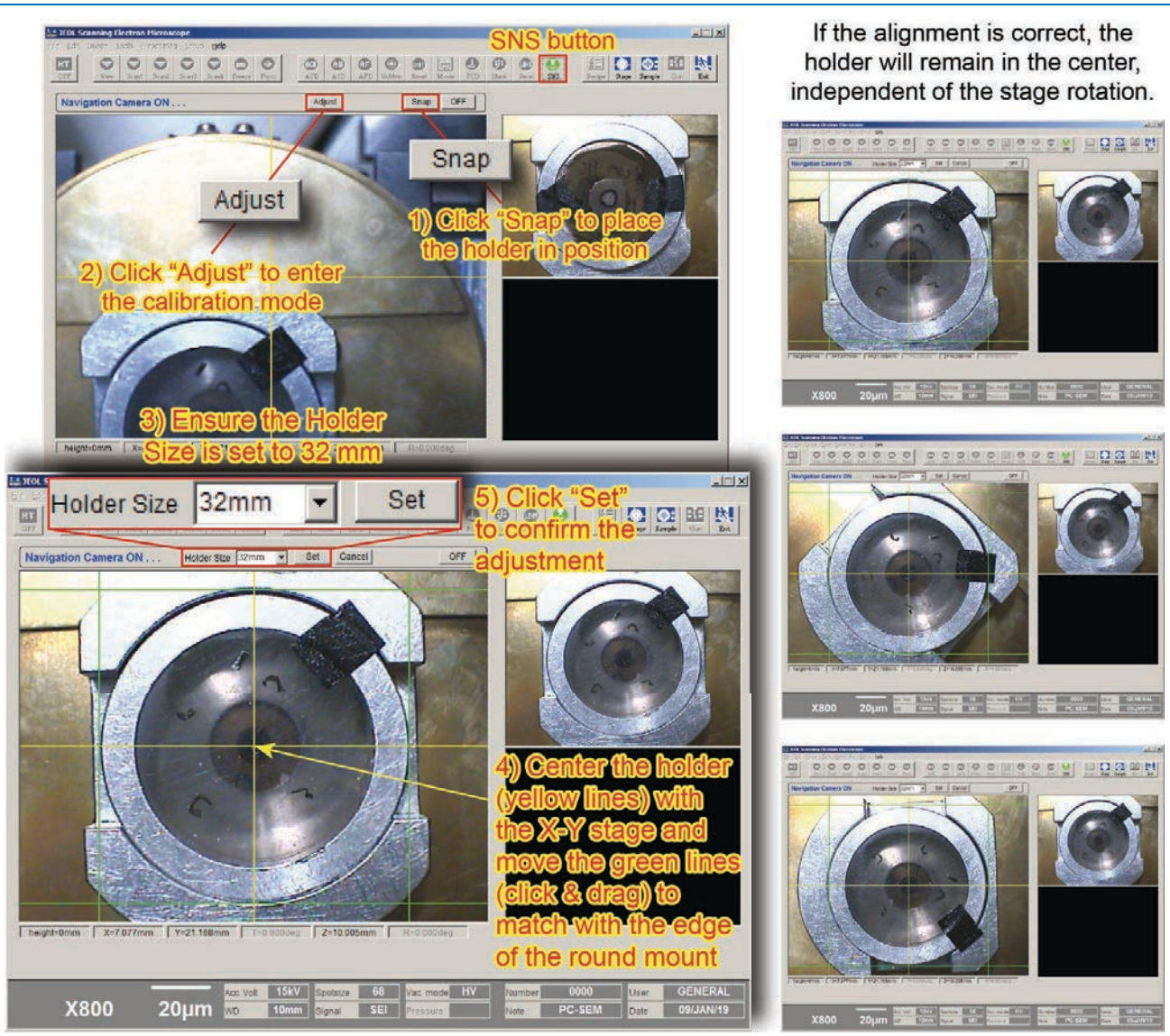


Figure A1. *Adjusting the Sample Navigation System (SNS).*

A2) Activating the SEM in W-mode

In a normal situation, the SEM is working under ultra-high vacuum (UHV) using an ion pump in the gun. However, some situation might force us to shut down the ion pump and to work only under "high vacuum" condition using only the rotary and the turbo pumps. Such an ***exceptional*** condition is commonly related to a vacuum leak in the gun or a problem with valve VT-1 or VT-3 and is only temporary. In this situation, only a W-filament can be used.

WARNING: Follow these instructions **ONLY** when…
(1) the SEM is loaded with a W-filament (green sign)
AND (2) when the SEM is in "W-mode" (blue sign).

The vacuum reading on the box on the left side of the SEM will be OFF. The light "UHV" will always remain orange, and both lights "Column" and "HV" will be either green when a sample is loaded (SEM under high vacuum) or orange when you open the sample chamber.

When the W-mode is in use, both valves VT-1 and VT-3 are OPEN. This implies that each time you vent the instrument, the vacuum is lost in the chamber and in the gun. You must therefore TURN OFF the filament heat each time you perform a sample change or at the end of your session. Follow these instructions to LOAD your sample:

- Lower the filament saturation to zero and turn OFF the electron gun (button HT OFF).
- Click on "Sample" and press "Vent". The HT button will turn grey and state "Wait…".
- Open the chamber and load your sample.
- Take a photo with "SNS" as usual. Then, lower the stage to 30 mm.
- Close the sample chamber (keep an eye on the IR camera!).
- Press the button "EVAC" to pump.
- Wait for the signal "READY".
- The HT button should now be blue. Press on it to turn HT button ON (green).

Perform then a ***filament saturation*** by setting the filament heat to its optimum (***Fig. A5***):

- Remove the "Beam blank".
- Insert the Faraday cup by pressing the button "PCD".
- Press the top-left button "CONFIG / LOCAL" on the ammeter. This will activate the live beam current reading. If the ammeter displays "CONFIGURATION", press another time on the "CONFIG / LOCAL" button. If you still don't see a live reading of the beam current, press the button "TRIG".
- At first, the ammeter should indicate a value around zero.
- Set the "Spotsize" to around 74-78 (= medium to high beam current).
- Open the window "Gun" and increase the filament heat until the emission current and the beam current increase. Continue increasing the heat, until the beam current start decreasing again as you are passing the "false peak". Continue increasing the heat until the beam current increases again. You will reach the saturation point when the increase in beam current slows down significantly or plateaued or drops down slightly (= saturation point). When you are at saturation ***lower*** by one or two clicks the heat.
- The emission current should read at least ~30 to 50 µA or more.
- The ammeter should read ~2 to 3 nA when the beam aperture #2 is in use.
- When done, click on "PCD" to remove the Faraday cup.
- You should now see an electron image on the screen.

Perform then a beam alignment following the instructions in ***Section F***.

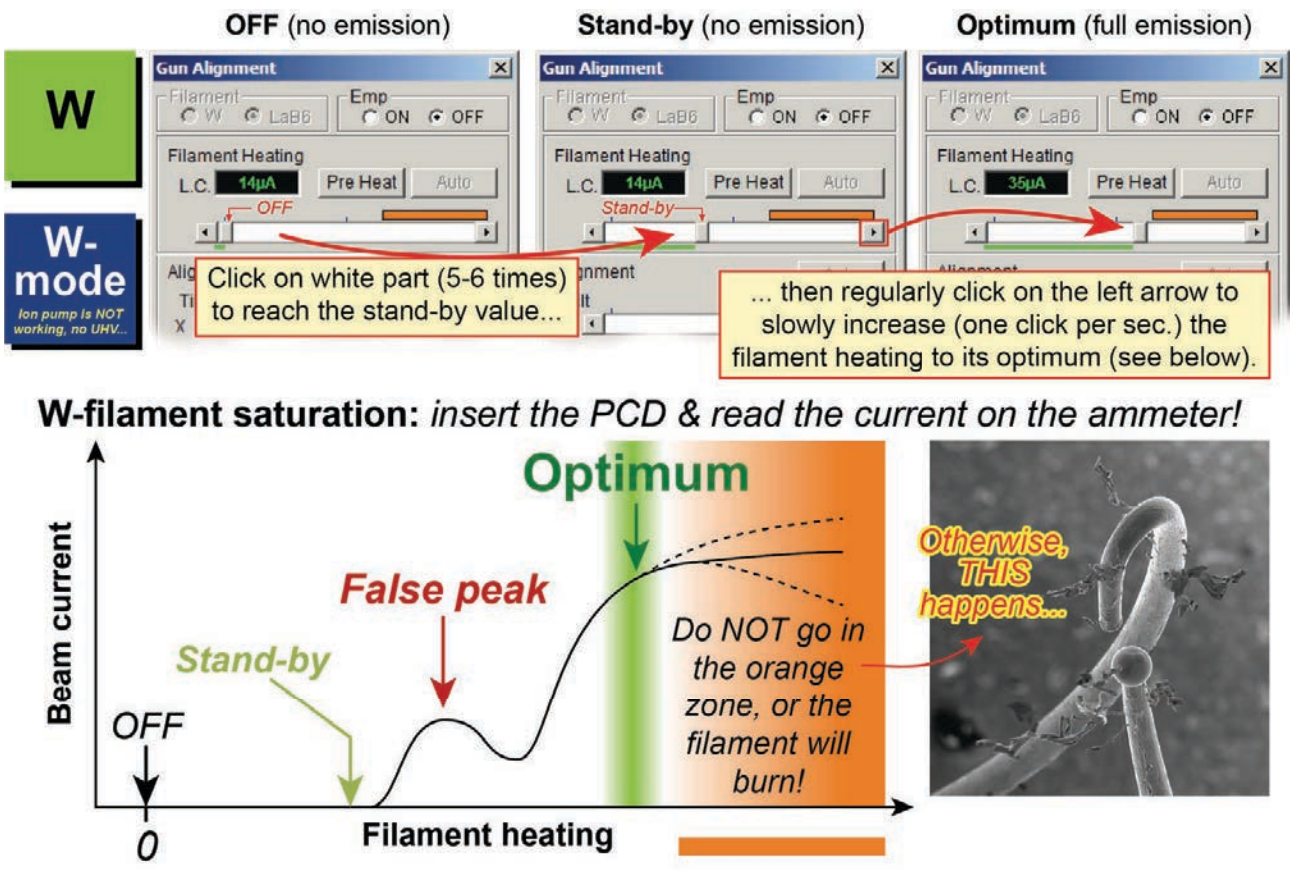


Figure A2. *Saturation of the W-filament in W-mode. When the optimum is reached, the emission current should read 30 to 50 µA (or higher).*

A3) Characteristic X-ray

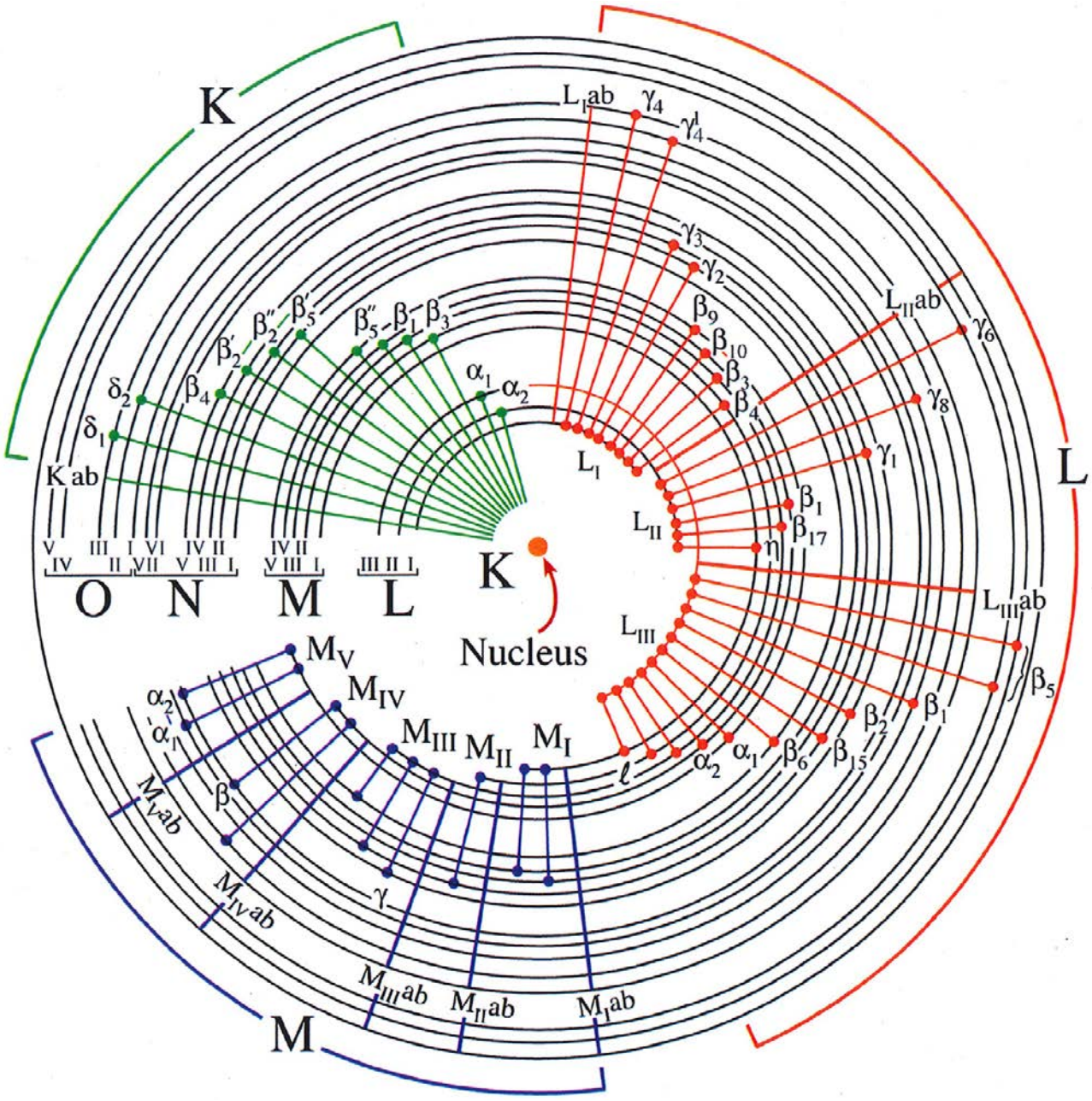


Figure A3. *All common electron transitions and characteristic X-ray generation.*

From Goldstein et al. (2003) "Scanning Electron Microscopy and X-Ray Microanalysis" (Fig. 6.9, p. 280). Original in Woldseth (1973) "X-Ray Energy Spectrometry".

A4) Image resolution

Relationship between the actual pixel resolution (expressed as micrometre) and the magnification & image resolution.

- **For regular SE / BSE images:** a resolution of 1024 x 768 pixels is recommended. Higher resolutions are possible with Pathfinder. However, keep in mind that if you double the width & height (2048 x 1536), you quadruple the area to scan and the total acquisition time (e.g., 2 x 40 sec instead of the regular 2 x 10 sec for good quality).
- **For EDS element mapping:** consider smaller images (e.g., 256 x 192 pixels), and select an image resolution and magnification such that the pixel size is equal or larger than the expected projected interaction area of the electron beam and the sample (typically 1-2 µm diameter for silicates at 15 to 20 keV, smaller for heavier phases). In any case, ***choosing a pixel resolution less than 0.5 µm is NOT recommended.***

SI Resolution in *µm/pixel* at 10 mm working distance

Mag.	128 x 96	256 x 192	512 x 384	1024 x 768
30	32.4	16.2	8.1	4.0
50	19.4	9.7	4.9	2.4
80	12.2	6.1	3.0	1.5
100	9.7	4.9	2.4	1.2
120	8.1	4.1	2.0	1.0
150	6.5	3.2	1.6	0.8
180	5.4	2.7	1.4	0.7
200	4.9	2.4	1.2	0.6
300	3.2	1.6	0.8	0.4
350	2.8	1.4	0.7	0.35
400	2.4	1.2	0.6	0.30
500	1.9	1.0	0.5	0.24
750	1.3	0.6	0.32	0.16
1000	1.0	0.5	0.24	0.12
1250	0.8	0.4	0.19	0.10
1500	0.6	0.32	0.16	0.08
2000	0.5	0.24	0.12	0.06
2500	0.4	0.20	0.10	0.05
3000	0.33	0.16	0.08	0.04
4000	0.24	0.12	0.06	0.03
5000	0.20	0.10	0.05	0.02

red: not recommended

Figure A4. *Table of actual pixel size in micrometre as a function of the image size in pixels and the magnification. Values are only valid at a working distance of 10 mm.*

A5) EDS mapping time & element map quality

For element mapping by EDS, you should consider the total X-ray counts received on each pixel. A minimum of ~1000 counts on each pixel is recommended. Above ~4000 there is no significant gain of extractable information for most applications, except maybe if you need to identify a significant zonation in a specific phase (e.g., Ca-Na in plagioclase). In this case, you should certainly first discuss with the lab manager...

Use ***Figures A5.1 and A5.2*** as a reference to define the minimum counting time per frame to reach the desired quality.

Avg counts per pixel in SI mode @85 kcps

Time constant: *Rate 6 or 7* **Storage Rate (SR):** *85000*

	Map resolutions [counts/pixel]			
Total time	128 x 96	256 x 192	512 x 384	1024 x 768
5	2075	519	130	32
10	4150	1038	259	65
15	6226	1556	389	97
20	8301	2075	519	130
30	12451	3113	778	195
40	16602	4150	1038	259
50	20752	5188	1297	324
60	24902	6226	1556	389
90	37354	9338	2335	584
120	49805	12451	3113	778
180	74707	18677	4669	1167
240	99609	24902	6226	1556
300	124512	31128	7782	1945
600	249023	62256	15564	3891

Avg counts per pixel in SI mode @50 kcps

Rate 5 or 6 **Storage Rate (SR):** *50000*

Map resolutions [counts/pixel]			
128 x 96	256 x 192	512 x 384	1024 x 768
1221	305	76	19
2441	610	153	38
3662	916	229	57
4883	1221	305	76
7324	1831	458	114
9766	2441	610	153
12207	3052	763	191
14648	3662	916	229
21973	5493	1373	343
29297	7324	1831	458
43945	10986	2747	687
58594	14648	3662	916
73242	18311	4578	1144
146484	36621	9155	2289

Quality	counts/px
poor	<700
ok	700 - 2000
Good	2000 - 4000
excellent	>4000

Figure A5.1. *Table of expected total number of counts received on each pixel of an element map as a function of the image size and counting time a minimum of 1000 counts per pixel is recommended. Average counts/px = SR * [Total time] / [Total number of pixel].*

Left table: Time constant of 6 to 7 and a storage rate (SR) of ~85'000 cps.
Right table: Time constant of 5 to 6 and a storage rate (SR) of ~50'000 cps.

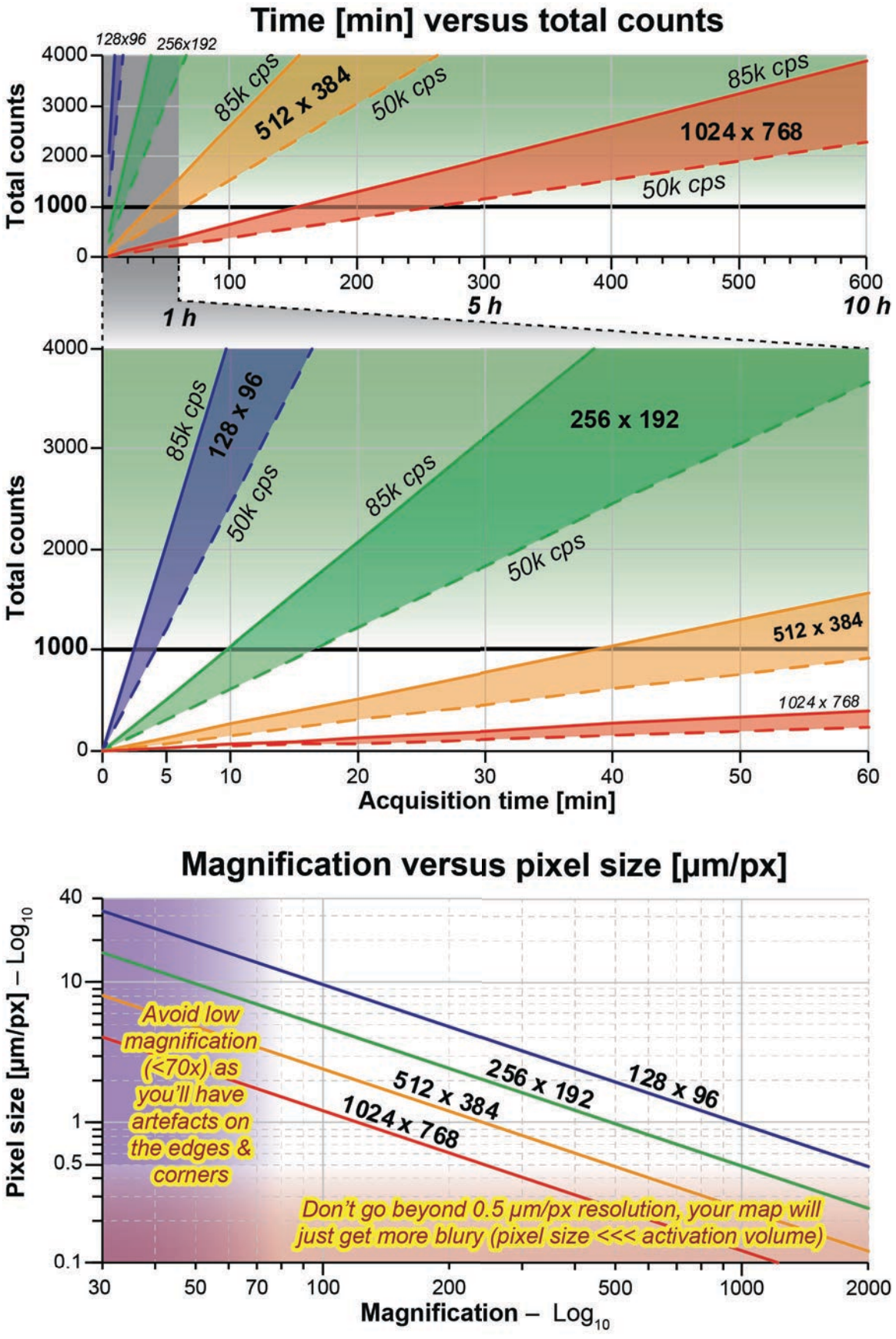


Figure A5.2 *Relationships between magnification, map pixel size, and counting time. Use these plots to estimate the best analytical conditions for your needs.*

A6) File handling in Pathfinder

Below is a list of the important files created by Pathfinder during EDS analysis.

A6.1) "Spectrum" mode

- EMSA [text file] EDS data including energy vs counts data (at the end).

A6.2) "Point ID" mode

- PSMSA [text file] EDS data including energy vs counts data (at the end).
- P_S [text file] Information on the point & area analysed.
- PSREF [TIF file] (*) Raw BSE/SE image (without scale bar).

A6.3) "Spectral Imaging" mode

- PCM [TIF file] (*) Image of a principal component (filename "xxx CP1", "xxx CP2", …).
- PCS [text file] EDS of a principal component (filename "xxx CP 1", "xxx CP 2", …).
- SITIF [TIF file] (*) Base image (BSE or SE) at the resolution of the spectral image.
- FZM [TIF file] (*) Image of a phase map (filename "xxx Phase1", "xxx Phase2"…).
- FZS [text file] EDS of a phase map (filename "xxx Phase 1", "xxx Phase 2"…).
- CSI [text file] Configuration file of the spectral image.
- SI [binary file] Compressed binary data of the spectral image.
- SIREF [TIF file] (*) Raw BSE or SE image at the full resolution (without scale bar).

A6.4) Export files generated by Thermo

- TIF [TIF file] Export of a BSE image (menu "File > Export Image as Full Res TIF").
- CSV [text file / XL] Export of quantitative analysis of selected EMSA file or Point ID.
- PPTX [PowerPoint] Report export in the PowerPoint format.
- DOCX [Word] Report export in the Word format.
- RAW [binary file] (**) Export of SI binary file using Lispix format (required for iSpectra).
- RPL [binary file] (**) Export of SI parameters using Lispix format (required for iSpectra).
- BMP [BMP file] Export of a BSE image (from menu "File > Export as Bitmap").

A6.5) Various parameter file (required by Pathfinder)

- AcqProps.tnp Setting for the acquisition properties.
- NSSProject.xml Information about your Pathfinder Project.

(*) These "hidden" TIF files can be opened with any imaging program (Paint, Photoshop, etc.). Just force opening the file with your favourite image editor.

(**) Use the **SIConvert.exe** tool to convert SI file into RAW and RPL files (see iSpectra manual). You will need to copy the SIConvert.exe file along with all .CSI and .SI files from your map in a single folder.

A7) "iSpectra" by Christian Liebske

iSpectra* is an open-source toolbox for the analysis of spectral images and provides the following features:

- RGB mapping tools,
- Principle component analysis,
- Transparent way of creating phase maps,
- Particle analysis,
- Batch processing to create large scale phase maps,
- Morphology operation to account for overlapping excitation volumes,
- User-adjustable image output,
- Ratio of two compounds in a single phase (e.g., Ca-Na in plagioclase),
- And more!

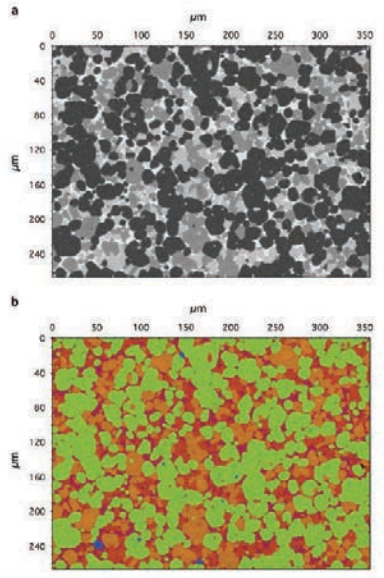


Figure 1. Displaying options to represent the 3D spectral image (SI). **a:** The default image-display is a "principle component map" associated with the highest variance (see text for details). **b:** Any element map that is extracted on SI import can be used as red, green, or blue channel of an RGB false color image; here an image of intensities at Si-, Mg-, and Al-Kα energies. Other possibilities include manually importing e.g. a back-scattered electron image that was recorded before or during SI acquisition or to use a sum-image. Any of these images can be used as a "base-image" on which groups of pixels are shown as colored overlays.

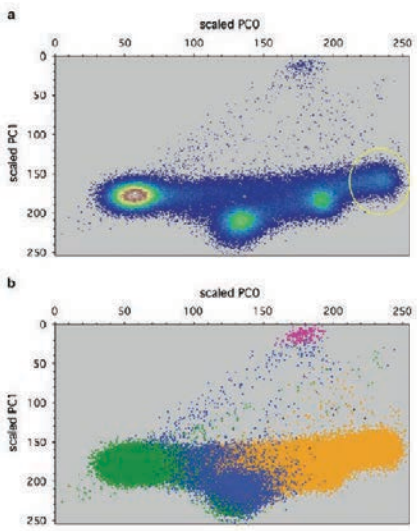


Figure 4. a: Density plot of the principle component maps (PCMs) associated with the largest variances across all elemental maps. Such a representation provides an objective way of visualizing populations with distinct chemical features. Pixels in the yellow region of interest (ROI) are back-traced for pixel segmentation (see text). For computational convenience PCMs are scaled to values from 0–255. **b:** Similar representation as above but displaying each individual pixel in its group color (see Figure 3). This shows that the orange group hosts two chemical features that need to be separated using ROI tools.

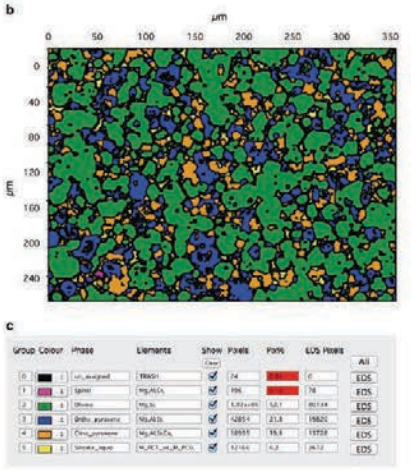
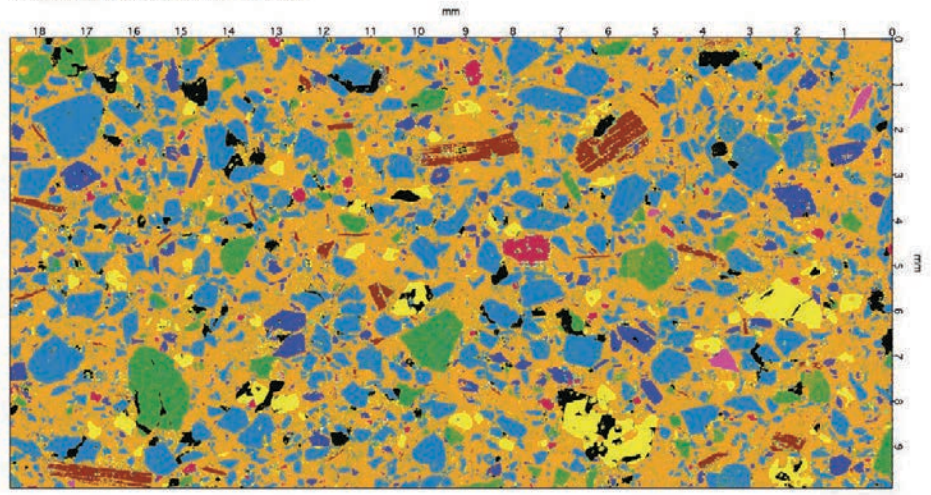


Figure 5. Final pixel assignment before (a) and after applying erosion to all groups. Eroded pixels, i.e. pixels discarded for forming cumulative phase spectra are blacked out (b). (c) Note that erosion significantly reduces the number of pixels used to form cumulative spectra as it can be seen in the columns "EDS Pixels" versus "Pixels" on the iSpectra group panel.

Figures from Liebske (2015)



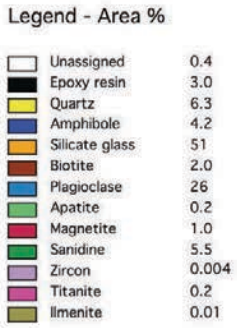


Figure A8. *Example taken from iSpectra (Liebske, 2015).*

*** For more information, contact Christian or see the iSpectra manual.** Liebske, C. (2015). ISpectra: An Open Source Toolbox For The Analysis of Spectral Images Recorded on Scanning Electron Microscopes. Microscopy and Microanalysis, 21(4), 1006-1016. doi:10.1017/S1431927615014336

A8) Troubleshooting for bad analysis & other known problems

If all elements are analysed, your standard-based quantitative analysis should yield between 98 and 102%, with occasional outliers slightly outside these limits. There are multiple reasons for obtaining odd totals. Check first the following common issues.

A8.1) SAMPLE related problem…

- **Are all elements analysed?** Do your sample contain non-analysed element (e.g., H [H_2O], C [CO_2], B, N, Li, etc.)? Have you forgotten some important element(s)?
- Is your sample **well-polished**? Are your analysis points correctly set on a **flat area**, **not too close to a phase edge** and **not over a crack**?
- Is your sample **mounted horizontally** (i.e., not tilted inside its mount)?
- Are you analysing homogeneous domain? If inhomogeneous (e.g., quenched glass with micro-crystals), you might "naturally" expect bad totals…
- **Problem of coating:** Is your sample **charging**? If you observe flashes or strong image deformation in SE imaging mode, this is likely a problem of charging. The charging can be small and may NOT induce (too much) imaging aberration. Therefore, you should also check the **Duane-Hunt limit**:
    - Acquire a long spectrum (ideally over 120 seconds);
    - During the acquisition, zoom in around the energy range of the acceleration voltage you are using (usually 15 keV). All X-rays (= mostly bremsstrahlung) should end very near to the acceleration voltage you are using (e.g., 15 keV mark) with a few abnormal values beyond (= uncorrected sum peaks).
    - At the end of the acquisition, check if you have a significant *(> 0.1-0.2 keV)* deviation from the acceleration voltage used (e.g., 15 keV). If so, the difference from the acceleration voltage used equals the negative charge (potential) on your sample. If this happens, you must remove your sample, repolish it to remove the coating, and re-coat it with 20 nm carbon. Ask for assistance if necessary.
- Are you analysing **"beam sensitive" material** (e.g., hydrated phase, Na- or K-rich phase, carbonate, phosphate, sulphate, etc.)? If so, it is possible you damaged the mineral during the analysis. In this case, do NOT use the "point" analysis, and rather draw a "rectangle" or a "circle" over a large area (several µm if possible).

A8.2) HARDWARE problem…

- Is the **dead time** (DT) between 20 and 30%?
- Have you set the **Time Constant to "Rate 2"** in the EDS acquisition setup?
- Is the **Working Distance (WD)** set at 10 mm on JEOL? If so, Pathfinder should indicate ***WD 9.7 mm in Pathfinder*** at 15 keV (or 9.8 mm at 20 keV and more).
- Have you **adjusted the Z-stage** to the optimum focus point (sharpest image)? It should be between 10 and 11 mm for quantitative analysis.
- Is the **beam current stable** and adequate (usually 2-3 nA)? Insert the PCD and check the ammeter for the actual value and its stability over the time of an analysis (usually 30 to 60s).
- Are you correctly analysing at **high magnification (≥ 500x)**? Off-centred analyses points will yield lower totals as they are not anymore in focus with the EDS detector.
- Check the **alignment of beam aperture**; if (strongly) misaligned it can partially block the electron beam and cause erroneous beam current reading.

If you are still getting trouble reaching good totals despite carefully checking all the above points, contact the lab manager or the assistant. An updated calibration might be required, or some other issue occurs.

A8.3) Other common problem

- If the ammeter is not responding, this is likely since someone turned it OFF… To reactivate it, start Pathfinder, and ask to run one random analysis. After a while, the ammeter should re-initialise itself. Try to get an analysis. It should now work…
- In imaging mode (SE, BSE, CL), you might sometime see a shadow on one corner or on one edge of the image. This is most likely due to a very bad alignment of the beam aperture. To fix this problem:
    - Lower the magnification to the minimum (30-40x)
    - Set the image signal to SEI
    - Move sequentially the X and the Y axes of the beam aperture to minimize the shadow effect and get the highest signal intensity without any shadow on either side.
    - Perform then a fine tuning of the beam aperture at high magnification (>2000x) as described in the beam alignment procedure in ***Section F***.

A8.4) Abnormal and known hardware-software bugs

The following list abnormal behaviour that will hopefully be fixed one day…

A8.4.1) Wrong EDS energy calibration

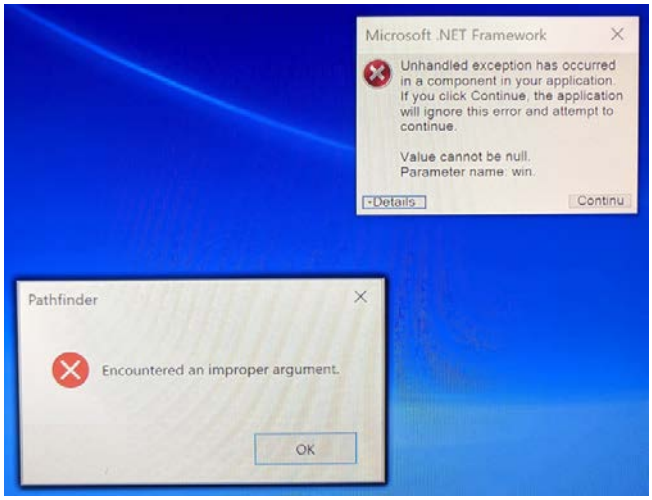
Starting in April 2019, we have observed a few times a shift in the X-ray peak position. This is due to the EDS hardware losing its calibration. This is NOT visible on low X-ray energy lines (e.g., O Kα or Si Kα) and should be observable at higher energy (> 5 keV). Check the position of the Fe Kα X-ray line (or any other line > 5 keV) and ensure it matches the theoretical position (Fe Kα = 6.40 keV; Fe Kβ = 7.06 keV).

If you observe a significant shift in energy (> 0.1 keV, see ***Fig. A9.3.1***), call for assistance, or perform the ***reset of the Pathfinder box*** as described below:

1) Select the acquisition mode “Spectrum” in the left menu.
2) Open the tab “Detector Status” on the bottom right panel.
3) Click on “Advanced Status”. It launches the program “FrontEnd Status”.
4) Close the software “Pathfinder”.
5) Click on the button “Reboot” in “FrontEnd Status”. The connection light turns red (disconnected). Locate the white box behind the Thermo PC (behind the table); wait 5-10 seconds until the indicator light on the top of this box turns green.
6) In “FrontEnd Status” click on “Reconnect”. The connection light should turn green in the software. If not, you have clicked too early (the hardware was not fully restarted). Wait and try again. If you still cannot connect, ask for assistance.
7) Restart the Pathfinder program.
8) Re-run an analysis and check the sum. Ask for assistance if you still observe a shift in the energy of a high-energy X-ray line.

A8.4.2) “Infinite” error message when closing Pathfinder

There is a non-reproducible (apparently random?) error sometime occuring when exiting Pathfinder. ***First, don’t worry, all your data are safe!*** See screenshot aside.



When this happens, use the keyboard connected physically to the left computer (usually above the left computer), and press CTRL + ALT + DEL. Open the Task Monitor and force closing the NSS / Pathfinder program running. This should automatically clear out all errors.

Acknowledgments: This manual would not be so complete without inputs from ***Dr. Christian Liebske*** regarding iSpectra and the former NSS Thermo program, numerous discussions with ***Dr. Eric Reusser***, and inspiration taken from the original 7-page SEM manual from ***Dr. Lukas Martin***.

Comments, suggestions, questions about this manual? Contact…

Julien M. Allaz
julien.allaz@erdw.ethz.ch
NW E 85
+41 44 632 37 20

J. M. Allaz © May 6th, 2022