JEOL JSM-IT500HR: Cathodoluminescence (CL) Imaging
Standard Operating Procedure (SOP)

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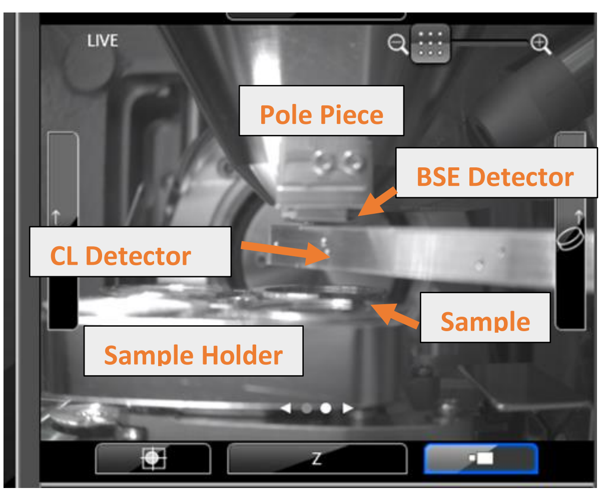
This document provides step-by-step instructions for obtaining cathodoluminescence images on the JEOL JSM-IT500HR scanning electron microscope (SEM). Prior to using the CL detector, users must be trained in basic imaging techniques. When exchanging samples, it is possible to cause a collision between the sample and the backscattered electron detector. In addition, the CL detector occupies the space between the backscattered electron detector and sample. Because of this, the stage and subsequent working distances are different when using the CL detector. ***Please read the section below prior to using this document.***

Sample height and specimen-detector collision

Once a sample is loaded onto the SEM stage, the sample must be moved to the proper location beneath the bottom of the electron column (i.e., the pole piece). Adjusting the X and Y coordinates is straightforward and poses very little risk on JSM-IT500HR. However, if the sample height is entered incorrectly, the sample and/or sample holder can collide with the backscattered electron detector mounted on the bottom of the pole piece or the CL detector (Figure 1). These collisions can destroy the detector and result in damages exceeding $15k.



When exchanging samples and/or inserting the CL detector, extreme caution must be exercised to ensure a collision does not occur. To prevent these collisions, it is critical to measure sample height correctly. In addition, while adjusting the sample/stage height it is crucial to use the side-mounted live camera and bring the bring the sample into position slowly in multiple steps. The methods for measuring sample height and adjusting the stage/sample height are discussed in step 13. Similar precautions arise when tilting samples. Please contact the laboratory manager if sample tilting is required.

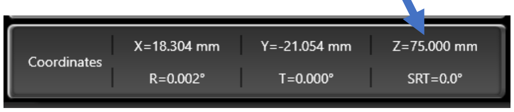
Step 1: Loading samples

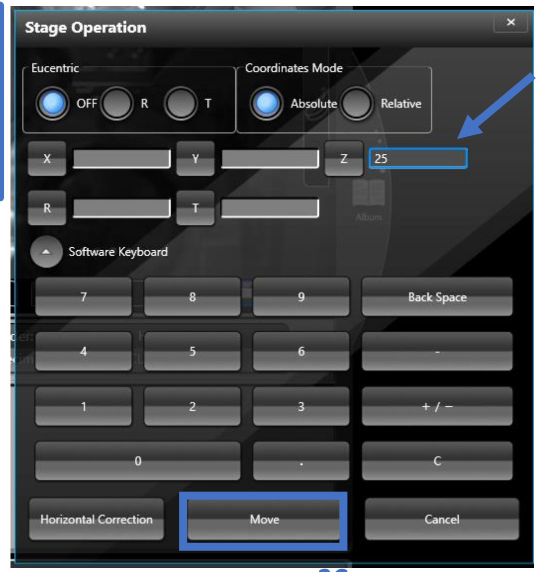
Follow steps 1-12 in the SEM Electron Imaging SOP.

Step 2. Once the system has pumped down and the live camera view is selected, **click on the Z value in the stage coordinates box** (2A) and move the sample surface to 25 mm beneath the pole piece. This can be done by **clicking on the z value box** (2B) in the Stage Operation window (pop-up window), entering a numeric value, and then **clicking the Move button** (2C).

At this step, we stop with the sample surface 25 mm away from the bottom of the pole piece. We will need the extra space to insert the CL detector in step 3.

2A

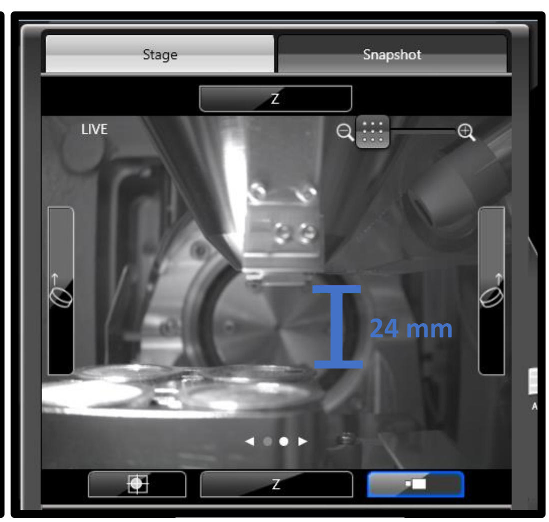




2B

2C

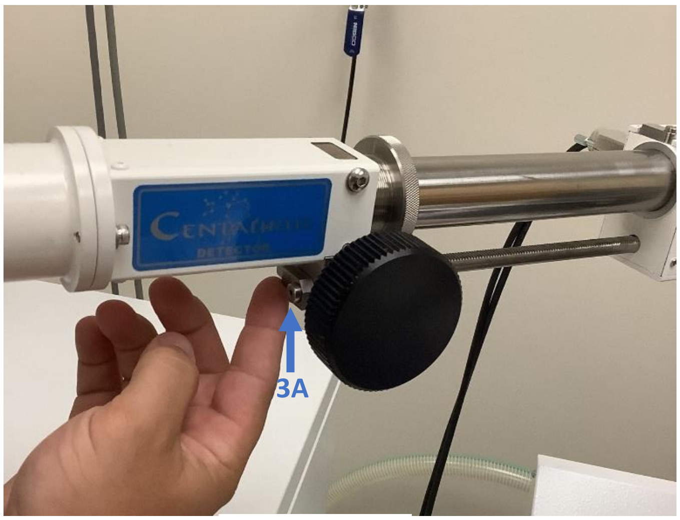
Stage at 75 mm



Stage at 25 mm

(sample is 1 mm tall)

Step 3: Push the locking mechanism up on the CL detector (rotating the handle counterclockwise helps be taking pressure off the lock) (3A). Once unlocked slowly **insert the CL spectrometer by rotating the handle clockwise** (3B). As you continue to rotate, the CL detector will come into view in the live camera (see images below). Be careful and go very slowly as the CL detector approaches the BSED and pol piece. If resistance increases, or the BSED shifts, stop inserting the CL detector immediately and contact Dr. Burns.



Push lock up

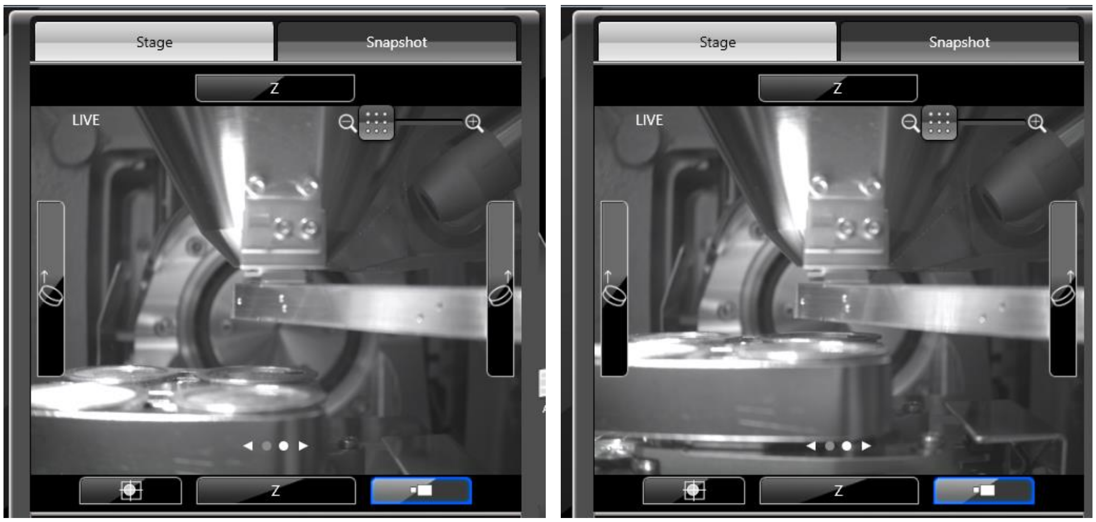
Rotate clockwise to insert detector

Detector out

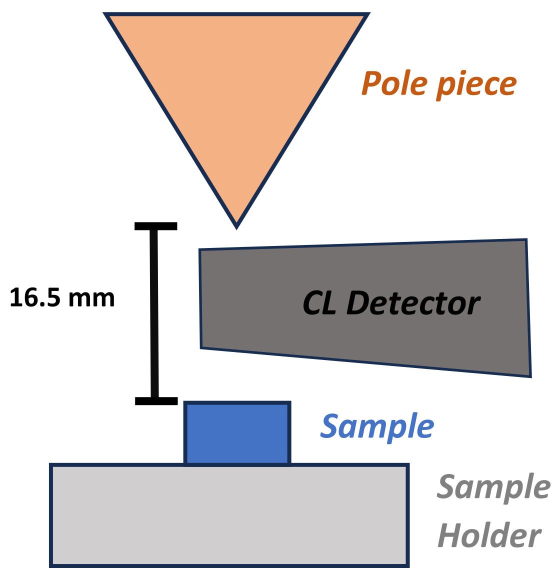
Detector partially inserted

Detector fully inserted

Step 3: Once the CL detector is inserted, adjust the z-axis until the surface of the sample is 16.5 – 17.0 mm from the bottom of the pole piece. Note that the height of the sample must be considered. For example, if the sample is 2 mm tall, the stage should be at 18.5 mm.

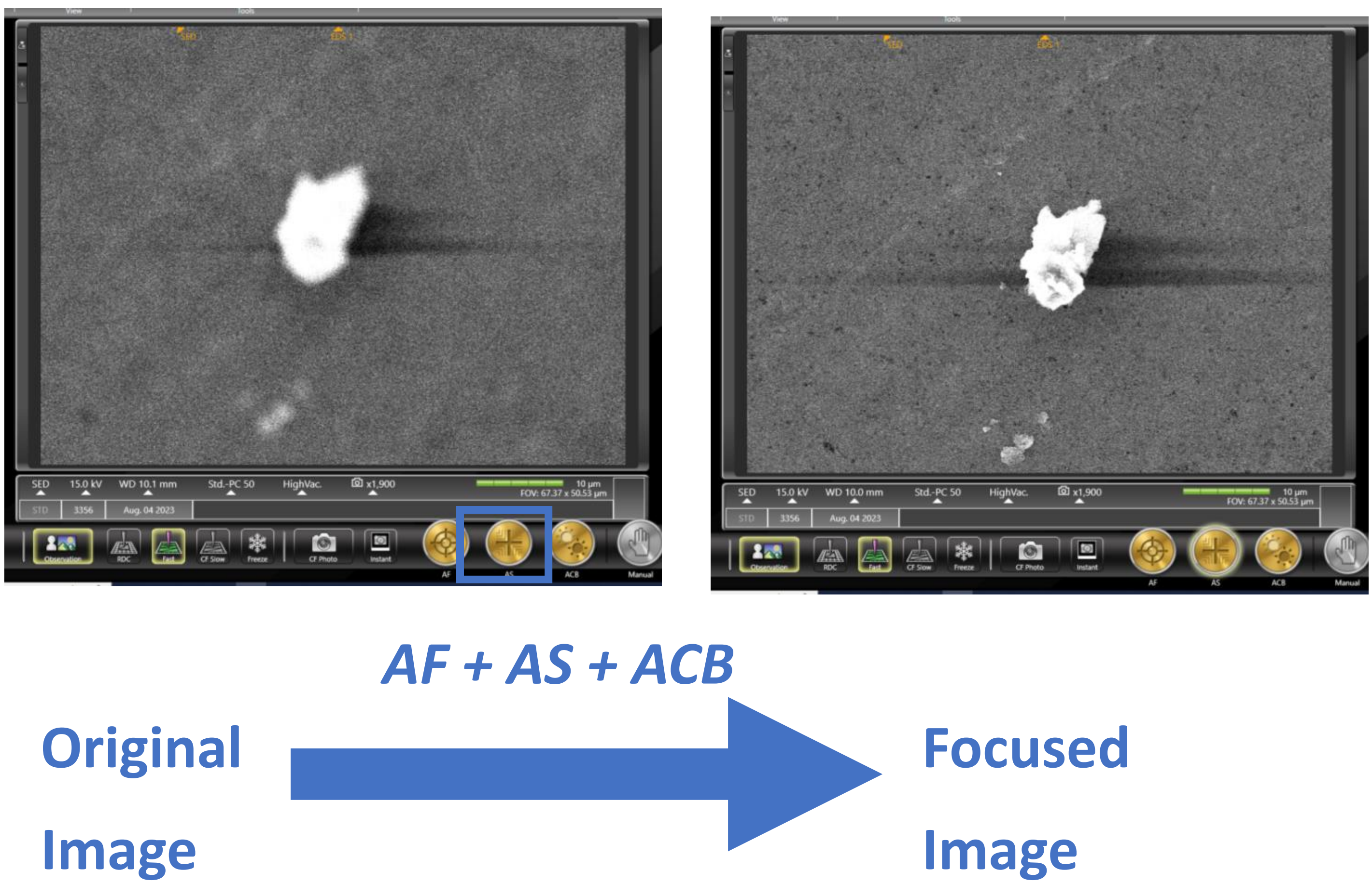


When adjusting the stage/sample height, it's useful to move in small steps (1-2 mm) and visually inspect the live image between steps. This ensures that a collision will not occur.



Step 4: Once the stage is in the correct position (see right image), turn on the accelerating voltage by **clicking on the Observation button** on the bottom left side of the screen. Once Observation is clicked, an electron image should appear (see lower photo). At low magnification the aperture in the CL detector will be visible (image below). Note that the BSE detector will not be accessible during CL imaging, as it is blocked by the CL detector.

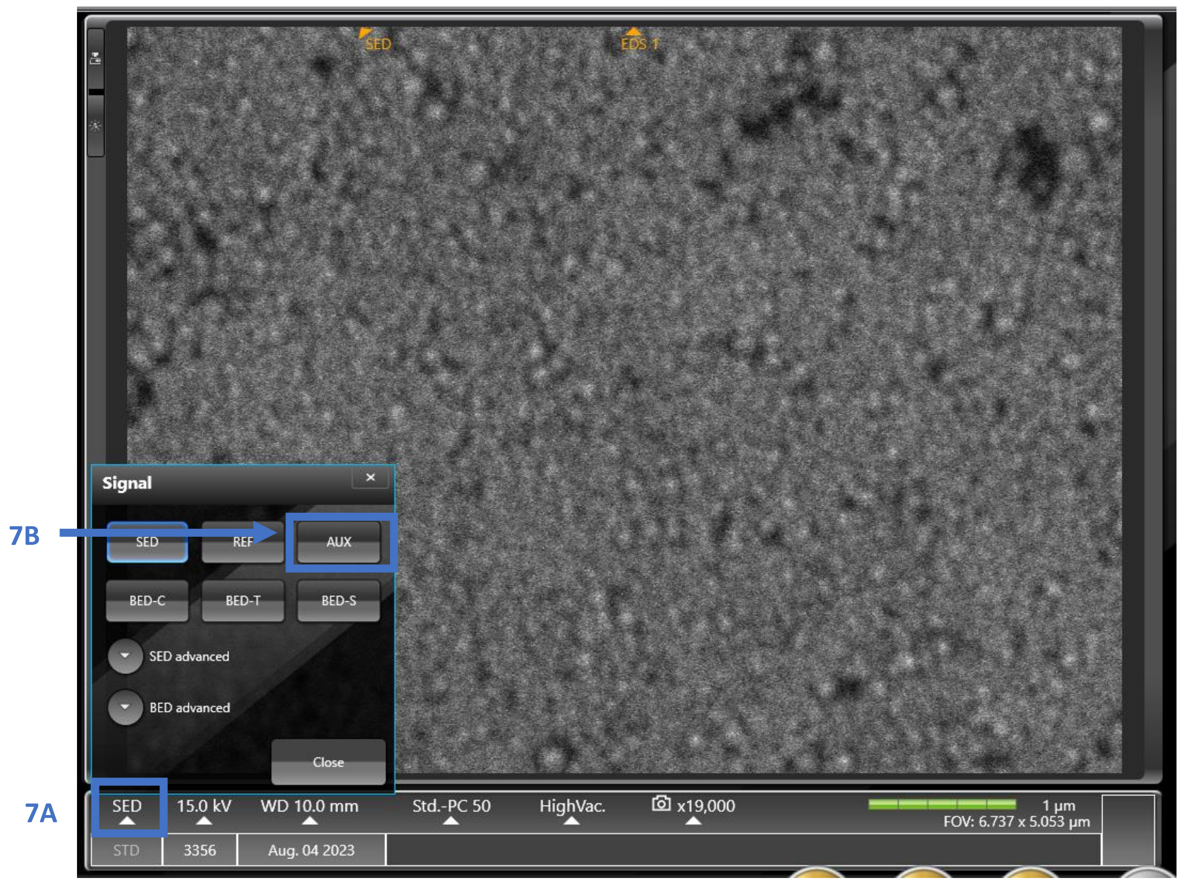
Step 5: In SED mode, zoom in on a small feature with relatively high contrast relative to the background. **Click the gold AS button** beneath the image. This will run automatic focus (AF), astigmatism (AS), and contrast and brightness (ACB) corrections (for details see next page). Once this sequence is complete, the image will be much sharper with better contrast and brightness. This may need to be done multiple times, particularly if a well defined feature is not used.



Notes: Focusing and astigmatism corrections are crucial to generating high quality SEM images. Electron microscopes consist of an electron source, where electrons are emitted, a cathode, and a set of electromagnetic lenses that condense the electrons into a beam. **Focusing** the electron beam requires optimizing the distance between the bottom of the column (i.e., final lens) and the sample surface to deliver the smallest possible primary beam diameter to the sample surface. Importantly, the spatial resolution of an electron image is directly controlled by the ability to focus the primary electron beam. In the case of the IT500HR, these corrections are done automatically through the AF function. In most cases, the auto function works very well. However, at high magnification, or with samples that have considerable topography, manual focusing may be required. This can easily be achieved using the focus knob on the operation console. The second major correction that can be critical to generating high quality SEM images is astigmatism. **Astigmatism** refers to stretching and/or distortion of the electron spot due to due to a lack of magnetic uniformity in the lens material. This is typically due to a lack of precision during machining and/or assembly of the lens. Like the AF function discussed above, the IT500HR has an automatic astigmatism correction feature. Generally, this feature works well at correction for astigmatism. However, at high magnification, an experienced user may choose to fine-tune the corrections manually using the operation console.

Step 6: Another step that is sometimes required to obtain a high-quality SEM image is to adjust the the beam alignment at the objective lens (OL). To check the alignment at the OL, zoom in on a small feature (2000-3000x) and click the Wobbler button (Home tab) in the upper left corner of the screen (16A). Once the wobbler is activated, look for movement in the X and/or Y direction (s). If there is notable movement in either direction, the alignment is off. If the alignment is off, click the ABA button (16B). This initiates an automatic OL beam alignment. If the alignment works properly, the feature will be pulsing in and out of focus but will not translate in the X or Y direction. If the alignment was considerably off, the screen may brighten significantly. If this occurs, the contrast and brightness can be adjusted with the ACB button or manually on the operation console. Once the alignment is adjusted, repeat step 15. It is worth noting that small, high contrast features that are roughly equant work the best for this alignment. Once the beam is aligned and focused, move forward to the next section.

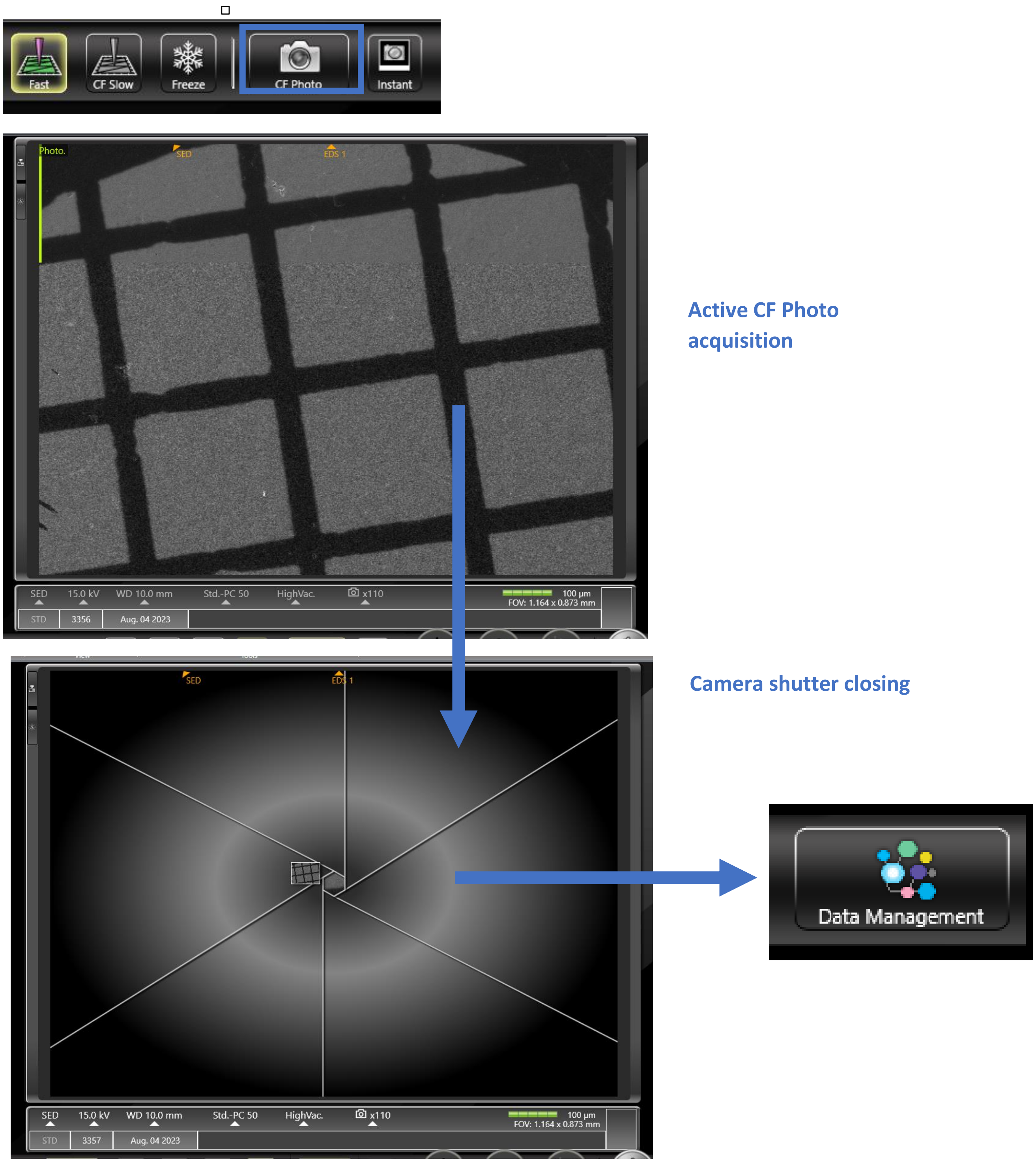
Step 7: Once the SED image is focused and the beam is aligned, switch to CL signal by **clicking on the small white triangle beneath SED** on the observation conditions panel beneath the image (7A). When the Signal box appears, **click AUX** (7B).



Step 8. Press the power button on the Centaurus Detector beneath the SEM-PC monitor (8A). **Turn the brightness knob clockwise** until the image on the screen is medium grey (8B). Now **turn the contrast knob clockwise** until a CL image appears (8C). Once an image is visible, the contrast and brightness can be adjusted to optimize the image quality. Make sure to try various scan resolutions and frame times. CL signal can often be low intensity and require long (>60s) frame times.

Step 9. Capturing images

To generate and save a high-resolution image, **right click on the CF Photo** button on the bottom of the screen. This will open a box displaying the available scan resolutions and frame times (see table below). The exact conditions will vary depending on user and the amount of signal needed to show the features/variations of interest. Recommended settings are provided below. After the scan resolution and frame time are selected, **left clicking on the CF Photo button** will initiate the scan. The SED image below shows a snapshot of an active photo scan (note the green progress bar). When the scan is finished, a graphic of a camera shutter will close, and an animation of a file moving into the Data Management tab in the lower right corner of the screen will play.



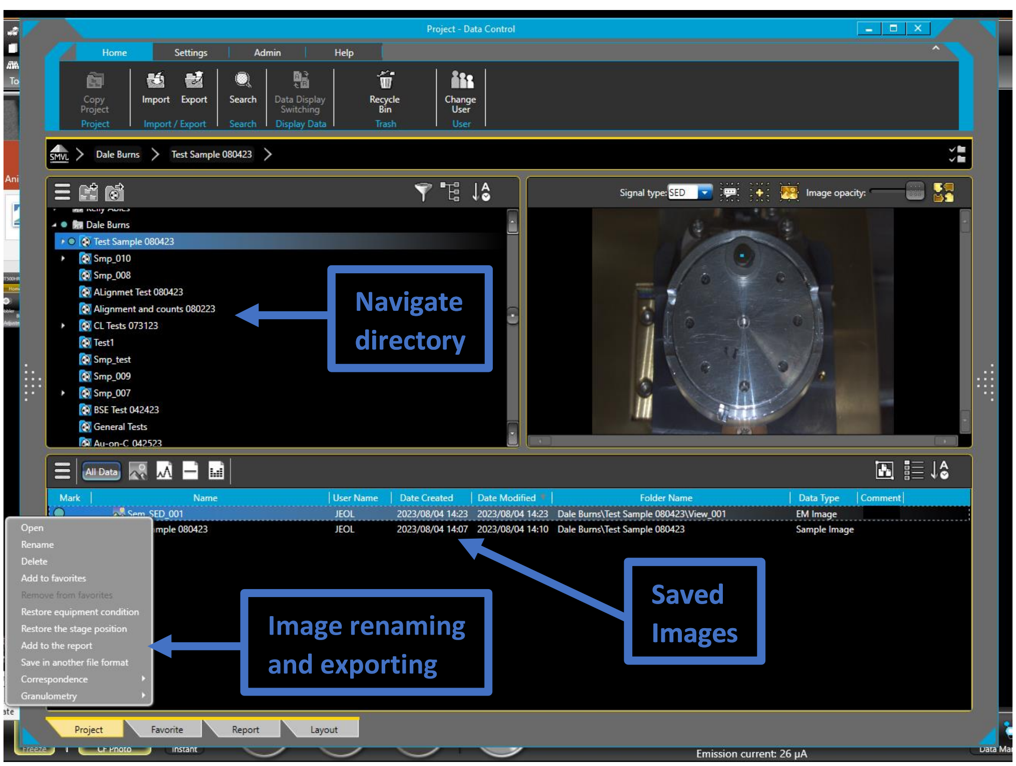
Scan Type	Resolution (pixels)	Frame Time (s)
Photo	1280 x 960	10
Photo	1280 x 960	20
Photo	1280 x 960	40
Photo	2560 x 1920	40
Photo	2560 x 1920	80
Photo	2560 x 1920	80
Photo	5120 x 3840	80
Photo	1280 x 960	160
Photo	2560 x 1920	160
Photo	5120 x 3840	160

Recommended standard CF Photo (2560 x 1920, 40 s)

Recommended high-resolution CF Photo (5120 x 3840, 160 s)

Step 10. Viewing and exporting images

To view your images, **click on the Data Management button** in the lower right corner of the screen. This will open a window to the user database. **Navigate to the appropriate username and project** in the lefthand screen. Once the correct project has been selected, the **saved images within the project will be visible in the lower portion of the window**. Note that there will be two types of images saved in the project directory: EM images and sample images. The EM images will have names that include the following: Sem_signal type_number (e.g., Sem_SED_001). These are the electron images. The other type of image, sample images, are images taken with the top-mounted camera during the sample exchange. **To change the name of an image, right click on the image and select Rename** in the drop-down menu. **To export images, right click on the image, or batch of images, and select the Save in another file format option**. This allows for the various types of images to be exported to physical or cloud storage. The system can also be set up to export images automatically during acquisition. See the note below for details.



Note: When starting an SEM session, the system can be set-up to automatically save the images in a user-defined location and format. This can be set by **clicking on the Settings tab** in the upper left corner of the screen, and then **clicking on the Image Setting button**. In the Image settings, file names, format, and export destination can be specified.