Standard Operating Procedure II: EDS (Bruker Flat-Quad)
> **FOLLOW the SOP strictly** to keep the instrument in good condition. Any violation will lead to user account suspension.
> **NEVER** use your own USB drive on instrument computer. Data can be transferred with the Jump Drive provided by the Core.
> **NEVER** surf the web on the instrument computer to minimize the risk of the computer being hacked.
> **NEVER** allow other users to get access to instrument computer on your reservation.
> **REPORT** any issues to Core director immediately so they can be fixed on time.
# Table of Contents

1. Introduction............................................................................................................................. 1
2. Sample Preparation ................................................................................................................. 2
3. Starting SEM Instrument ........................................................................................................ 2
4. SEM System Status Check .................................................................................................... 2
5. EDS Sample loading ............................................................................................................... 2
6. EDS setup on SEM computer .............................................................................................. 2
7. EDS setup on EDS computer ............................................................................................... 4
8. EDS measurement ............................................................................................................... 7
9. Closing EDS detection: ....................................................................................................... 13
Energy Dispersive Spectroscopy (EDS) Standard Operating Procedure

1 Introduction

1) Instrument features:
   > Cold field emission (CFE) e-beam source: high resolution on conductive surfaces
     (0.8 nm on Au clusters/magnetic tape)
   > Sliding-in annular Energy Dispersive Spectroscopy (EDS) detector: high elemental
     mapping resolution
   > Sliding-in annular Photo Diode PD-BSE detector: high signal intensity from
     backscattered electron
   > Scanning Transmission Electron Microscopy (STEM) detector: high resolution
     compositional contrast imaging, ideal for EDS mapping

2) Location
   Materials Characterization Core
   Room A119
   810 West Campus Drive
   West Haven, CT 06516

3) Primary Staff Contact
   Dr. Min Li
   Tel: 203-737-8270
   Email: min.li@yale.edu
   Office: ESC II, Room A119D

The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

**Warning:** Please follow strictly the SOP to keep the facility at good condition. We **DO NOT** recommend user explorations on program unless endorsed by core director.
2 Sample Preparation

**Note:** TEM samples can also be used with a dedicated TEM sample holder for EDS. The EDS mapping resolution can reach below 100 nm with TEM samples using STEM-EDS mode.

1) **Always wear gloves** for vacuum sample preparation! **Change gloves** if touched computer keyboard and mouse.

2) Follow instructions from SEM SOP to prepare samples. Use copper tapes to ground the sample surface with the holder to reduce the surface charging.

3) **Align sample surface strictly with the height gauge** tip as shown below. **Warning:** Samples mounted above Height Gauge tip will hit the EDS detector and the repair fee ($40,000) will be charged to PI’s account. **Note:** no need to use height gauge if TEM sample holder is used.

4) **Clean sample holder:** bring the specimen stub inside the fume hood and blow off loose particles on the sample surface using the N₂ gun.

3 Starting SEM Instrument

Follow Section 3 Starting Instrument in the separate SEM SOP.

4 SEM System Status Check

Follow Section 4 System Status Check in the separate SEM SOP.

5 EDS Sample loading

1) Follow Section 5 Loading the Specimen in the separate SEM SOP and leave the sample at exchange position

2) **Turn off** the small LCD from the back (top left) to avoid EDS detector damage and EDS spectra distortion. **Warning:** The LCD unit also provides power to the infrared camera inside main chamber. If it is left on during SEM/EDS scan, the camera will continuously emit infrared light to flood the EDS detector even at fully extracted position and shorten its lifetime quickly.

6 EDS setup on SEM computer

1) In PC_SEM program on the SEM computer (left side), click **HOME** button to move the sample holder to **HOME** position

2) **Turn off** the small LCD by pressing the switch in the back top-left corner of the monitor. **Note:** This step is crucial, otherwise **the EDS detector will be flooded by ambient**
signals leading to fat peaks in spectra.

3) Click Set button in the Stage tab in the popup window below:

4) Check the FQ-EDX box in the popup window below and hit OK.

5) The stage Z position should move to the default height at 14 mm as highlighted in the window below:

Warning: Never change the Z position < 11 mm. This will cause the sample holder crashing into the EDS detector. This severe SOP violation will lead to user account suspension and charge on PI’s account.

6) Check the working distance (W.D.) in PC_SEM below and make sure it is set at 15 mm.

If not, click on the top menu bar to switch imaging mode from LM (low magnification) to high magnification, then change W.D. in the window below. Switch back to LM.
7) Change Vacc (accelerating voltage in PC-SEM window) as highlighted below

![Image showing Vacc setting](image)

with the instructions listed below:

a) Check the EDS element table and choose a set of characteristic X-ray emission lines for intended sample elements that have least energy overlapping.

b) Choose the Vacc at least 2x the highest X-ray line in matrix elements. **NEVER** choose e-beam voltage above 20 keV!! The EDS detector will be burned!!

Tip: smaller beam voltage means higher EDS special/mapping resolution.

7 EDS setup on EDS computer

1) Log into EDS computer through FOM Screen Locker.
   a) The EDS software Esprit window should be always kept **ON**.
   b) If the Esprit program was closed and the EDS computer was logged off, select the profile PC-SEM with password hitachi

2) Clear **Yes** button on the popup INFORMATION window below to connect to microscope (SEM)

![Information popup](image)

3) Check EDS detector window position on the detector highlighted on the pictures below and make sure it was set by previous user at 20 kV. If not, report to manager immediately.

   **Warning:** It is the serious SOP violation if forgot to switch the detector window back to 20 kV after use. This may lead to detector damage and repair charge will be applied to user’s PI account.

![Detector window](image)
4) **Check to make sure** the Specimen Chamber SC pressure is at **LE-4 Pascal**.

![Image of LE-4 Pascal pressure control](image1.png)

5) Switch **EDS** detector in operation mode and set up **detector configuration parameters**:
   a) In **EDS Esprit** operating program, click the triangle in the **EDS** tab at the bottom left corner as indicated below:

   ![EDS Esprit control panel](image2.png)

   b) In the **EDS DETECTOR CONFIGURATION** popup window below:
      > Select **130 kcps** in **Pulse throughput** and **20 keV** in **Maximum energy**.
      > Make sure the **Thermostat** is checked in **Cooling** setting.
      > Select **Normal operation** in **Mode** setting. Select **Yes** in the popup **INFORMATION** window.
c) Close both INFORMATION and EDS DETECTOR CONFIGURATION window above.
d) The EDS detector will be cooled to the operating temperature of \(-20 \pm 0.5 \text{ ºC}\) in about 5 minutes.

6) **EDS detector window setting at different overvoltage/e-beam voltage:**
   a) **If the beam voltage \(\leq 6 \text{ kV}\):**

   > Enter Esprit System window by clicking button on the bottom left of the side menu and clicking tab as shown below. Click button to open Detector Data window and select “1µm Mylar” from dropdown list.

   > Go back to EDS detector head on SEM chamber as shown below, switch the detector window from 20 kV to **6 kV**.

   b) **If the beam voltage is between 6 kV and 12 kV:**

   > Enter Esprit System window by clicking button on the left side menu
and clicking as shown below. Click to open Detector Data window and select “1+2 µm Mylar” from dropdown list.

> Go back to EDS detector head on SEM chamber, switch the detector window from 20 kV to 12 kV.

c) **If the beam voltage is larger than 12 kV (Never change voltage to above 20 kV to damage the detector):**

> Enter Esprit System window by clicking button on the left side menu and clicking as shown below. Click to open Detector Data window and select “1+6 µm Mylar” from dropdown list.

> Go back to EDS detector head on SEM chamber, make sure the detector window switch is set at 20 kV.

d) **If higher Vacc is needed** in the middle of EDS scan, switch the EDS detector window to higher voltages first to protect the EDS detector.

e) **If lower Vacc is needed** in the middle of EDS scan, change the Vacc to lower voltages first to protection the EDS detector.

8 EDS measurement

1) **During EDS detector cooling**, turn on e-beam in PC_SEM program and find the area of interest.

2) Check EDS detector and **make sure** it has reached the operation temperature -20 ± 0.5 ºC

3) Check the Microscope tab below and make sure the HV (beam voltage Vacc in PC_SEM computer), Magn. (Magnification) and WD (W.D.) follows the numbers in PC_SEM program.

![Microscope tab](image)

4) **If HV, Magn and ED failed to match SEM program:**

    > clicking button on the **bottom left** of the side menu and clicking tab, then click button. If no error popup window appears, wait for several minutes, and monitor HV, Magn and WD changes on the Microscope tab. Restart Esprit program if needed.

    > If the following **ERROR** popup window appears,
Click **OK** button and follow the steps below:

a) Turn off beam voltage (Vacc) and close PC_SEM program and Esprit program,
b) Restart PC_SEM computer first,
c) Wait till the windows system is back on SEM computer, restart EDS computer,
d) Start PC_SEM program and wait until the program is completely back,
e) Start EDS program.

5) Click [ ] button in **EDS** tab in **Esprit** program and click **OK** on **DETECTOR POSITION** window to move the detector to the acquisition position right above sample.

6) If the user decide to run EDS in the middle of SEM imaging, make sure to check **FQ-EDS** mode in **PC_SEM** program **Stage > Set** setting window to lower the sample stage to required EDS stage height (14 mm). Please check Section 6 from Step 1-4) on page 3.

**Warning:** Failure to check FQ-EDS box will lead to EDS detector crashing into sample stage. The repair cost will be charged on PI’s account.

7) **EDS dead time** adjustment (Read steps below and move to Step 7) Spectrum Acquisition mode and follow steps a)-c)):

> As shown in the **EDS** tab below, the **Dead time** in percentage denotes the signal processing capability of the EDS controller. The higher number indicates the higher signal counts and the possibility of the **signal pileup or false peak** appearance. The **Dead time** should be kept around 30% for Spectrum Acquisition Mode and 10% for Mapping, Objects and Line Scan Mode.
8) **Spectrum Acquisition Mode:**

Note: Make sure the imaging area for spectrum collection is homogeneous if using this mode. Consider **Objects mode** next for inhomogeneous surfaces.

a) Go back to **PC_SEM** window, decide the image magnification and briefly adjust focus and stigma to get a good SEM image. No need to adjust aperture alignment and stigma alignment.

b) In **EDS Esprit** window click on the left side menu to enter **Spectra** workspace.

c) Click **Acquire** button and check and adjust the **Dead time** within ~10 – 30%:
   
   > If the **Dead time** is below 10%, in **PC_SEM** window (do the opposite steps if **Dead time** is above 30%):
   
   > Hit **Acquire** again to stop the acquisition. Increase the e-beam emission current (maximum 30 µA). Click **Acquire** to resume acquisition and check the dead time.

   > Hit **Acquire** again to stop the acquisition. Choose **High Probe current**. Click **Acquire** to resume acquisition and check the dead time.

   > If the dead time is still below the required values, click **Acquire** again to stop the acquisition. Change **Cond. Lens 1** setting from “5” to “1” or
“2” in the window above, the smaller number the higher the signal intensity. Click to resume acquisition and check the dead time.

d) If the interested peaks appear smooth, click again to stop acquisition.

e) To add collected spectrum into project or report, hit the button on the top right corner of the spectral workspace.

f) To save the data in Bruker spectra format (*.spx) or export to *.txt or *.xlsx format, click the lower button.

9) Objects Mode:

   Note: this mode should be chosen for inhomogeneous surface spectral analysis.

a) Adjust Dead time within 10 – 30% in Spectrum Acquisition Mode above.

b) Hit on the left side menu to enter Object workspace.

c) In PC_SEM window adjust focus and stigma to get sharp SEM image.

d) Click button on Scan tab to activate image drift correction. Make sure the button is highlighted in red to enable drift correction.

e) Go back to EDS Esprit window and click button to capture SEM image.

f) Select the desired object type on the bottom menu bar below and click on captured image above to specify positions. As the EDS spatial resolution is e-beam voltage/overvoltage dependent and typically around 1 µm, draw lines well inside the interested objects.

   > Choose Point object mode only if the interested feature is too small.

g) Click to highlight all objects and click 

h) If the interested peaks look smooth, click again to stop acquisition. Allow longer collection time if the point or small square or circle are
used.

i) Click the  button on the top right corner of the workspace window to save object data.

j) To save the spectrum only, click  at the top right corner of the spectrum window.

10) Line Scan Mode:
   a) Adjust Dead time within ~ 10 – 30% in Spectrum Acquisition Mode
   b) Hit  on the left side menu to enter Line Scan workspace
   c) In PC_SEM window adjust focus and stigma to get sharp SEM image
   d) Click  button on Scan tab to activate image drift correction. Make sure the button is highlighted in red to enable drift correction.
   e) Go back to EDS Esprit window and click  button to capture SEM image.
   f) Highlight the line and drag and adjust the endpoints to the desired position
   g) Set Point count of the line scan and click
   h) Use the  icon to identify elements
   i) If the interested peaks look smooth, click  again to stop acquisition.
   j) To save line scan data, click the  button on the top right corner of the workspace window.
   k) To save the profile, click the  at the top right corner of the lower profile window.

11) Mapping Mode:
   a) Adjust Dead time to be just above 10 % in Spectrum Acquisition Mode
b) Click button on Scan tab to activate image drift correction. Make sure the button is highlighted in red to enable drift correction.

![Scan tab with drift correction button highlighted](image)

b) Click button on Scan tab to activate image drift correction. Make sure the button is highlighted in red to enable drift correction.

c) Hit on the left side menu to enter Mapping workspace

d) In PC_SEM window adjust focus and stigma to get sharp SEM image

e) Go back to EDS Esprit window and hit button to capture SEM image.

f) Click button to start Mapping. If a green box appears inside the SEM window, click the ▼ on the bottom right corner of the and check Full below:

![Mapping window](image)

f) Click button to start Mapping. If a green box appears inside the SEM window, click the ▼ on the bottom right corner of the and check Full below:

g) Use the icon to identify elements in spectrum window during mapping.

h) Before stop mapping:

   > Click Spectrum tab above the top right corner of Map window, check if the interested elemental peaks are smooth in the spectrum.

   > Check if the mapping resolution has not improved with time. Typical mapping collection time depends on signal intensity (~10 – 30 mins or even longer).

i) Click again to stop Mapping.

![Mapping window](image)

i) Click again to stop Mapping.

j) To save map data, click the button on the top right corner of the workspace window.

![Save data button](image)

j) To save map data, click the button on the top right corner of the workspace window.

k) To save the large combined map image, click the on the top right corner of the image window.

![Save image button](image)

k) To save the large combined map image, click the on the top right corner of the image window.

l) To save individual element image in the thumbnail on the bottom, Shift+ left click
9 Closing EDS detection:

1) In PC_SEM program, click the OFF button to turn off electron beam

2) Click Standby button in EDS DETECTOR CONFIGURATION window to switch the EDS detector to Standby Mode

3) In EDS Esprit window, go to Data tab, click button to open Detector Data window and select “1+6 µm Mylar” from dropdown list and click OK.

4) Check EDS detector window position and make sure it is switched back to 20 kV to avoid detector damage.

5) Fully retract EDS detector by clicking in EDS Esprit program and wait until the detector is fully retracted.
6) In **PC_SEM** window:
   > Change **Vacc** back to **10 kV** and the **Ie** back to **10 µA**
   > Change the **Probe current** back to **Norm**.

   ![Probe current setting image]

   > Change the **Cond. Lens1** back to “**5.0**” in the window below in both **High Magnification** mode and **LM** mode:

   ![Cond. Lens1 setting image]

   > In **LM** mode, click the **Freeze** button below:

   ![Freeze button image]

   > Click the **EXC** button to move the specimen stage to the exchange position.  
   **Note**: click **Home** button if continue to do SEM imaging.

   ![Stage control image]
> Click **Set** button in the **Stage** tab; uncheck **FG-EDX** box below.

7) Check the **SEM SOP** closing steps to make sure all parameters are resumed to default settings.

8) **DO NOT** log off FOM from SEM computer while waiting for the EDS detector warmup to above **20 °C** (~ **20 minutes, be patient!**). **Never start sample transfer** while EDS detector is still being cooled. This will cause detector contamination.

9) During EDS detector warmup:
   > Make sure sample holder is at exchange (**EXC**) position
   > Data transfer is complete

10) **Check to make sure the EDS detector temperature rises above 20 °C.**

11) Turn **ON** small LCD

12) Finish sample extraction from SEM chamber. Check SEM SOP if needed.

13) Turn **OFF** small LCD

14) Clean the sample holder and work bench.

15) Make sure both **PC-SEM** and **Esprit** program windows are not minimized.

16) Upload data to box.yale.edu.

17) Log off FOM on both **SEM** and **EDS** computers.

18) Sign off the logbook.