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.
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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
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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.
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.
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**.

Starting SEM Instrument

Follow Section 3 Starting Instrument in the separate SEM SOP.

SEM System Status Check

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

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.

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.

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1 **Always wear gloves** for your sample preparation in your own lab or in MCC! **Warnings** will be given for violations and the **user account will be revoked** after three warnings with notice to PI. Further training at PI’s expense will be required to resume the account.
2 **This step is crucial to keep the SEM chamber vacuum at good pressure**, which in turn improves the imaging resolution with less surface contamination and keeps the SEM lens system at good condition.
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) Make sure Vacc (e-beam accelerating voltage) is OFF (check in PC_SEM program)
Note: it is OK to leave the e-beam ON if decide to do EDS in the middle of SEM measurements.

8) E-beam parameter setup for EDS:
   a) E-beam voltage (the overvoltage to excite EDS X-ray emission lines) setup:
      > Check the EDS periodic table and choose a set of characteristic X-ray emission lines for intended sample elements that have least energy overlapping.
      > The chosen e-beam voltage, the overvoltage should be at least 1.5x the highest X-ray line in matrix elements and below 20 kV.
         Tip: smaller beam voltage means higher EDS special/mapping resolution.
         Warning: NEVER choose e-beam voltage above 20 keV!! The EDS detector will be burned!! The user’s EDS access will be suspended and the cost ($70k) will be charged on PI’s account.
   
b) E-beam current setup:
      > Switch to High Magnification (HM) mode, set the Probe current to High in the Operation condition window below.
      > For chosen overvoltage smaller than 12 kV, consider increasing the e-beam current further by lowering the Cond. Lens1 in the window below: select smaller numbers than default “5.0”.

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) 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.
3) **Check to make sure** the Specimen Chamber SC pressure is at LE-4 Pascal.

![Image of Specimen Chamber pressure at LE-4 Pascal]

4) 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:

   ![Image of EDS tab with triangle]

   b) In the **EDS DETECTOR CONFIGURATION** popup window below:
      > Check **130 kcps** in Pulse throughput and **20 keV** in Maximum energy.
      > Make sure the **Thermostat** is checked in Cooling setting.
      > Check **Normal operation** in Mode setting. Select **Yes** in the popup INFORMATION window.

**Warning:** the EDS detector cooling should be started only when the Specimen Chamber pressure has reached LE-4 Pascal to minimize detector contamination.
c) Close both INFORMATION and EDS DETECTOR CONFIGURATION window above.
d) The EDS detector will be cooled to the operating temperature of \(-20 \pm 0.5\) °C in about 5 minutes.

5) **EDS** detector window setting at different overvoltage/e-beam voltage:
   a) **If the beam voltage ≤ 6 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 “\(1\mu\)m Mylar” from dropdown list.
   - Go back to EDS detector head on SEM chamber, 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 the **System** button on the left side menu and clicking as shown below. Click **Data** 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 the **System** button on the left side menu and clicking as shown below. Click **Data** to open **Detector Data** window and select “1+6 µm Mylar” from dropdown list.
> Go back to **EDS detector head** on **SEM** chamber, switch the detector window from 20 kV to **20 kV**.

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 °C**

3) 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.

**Warning:**

> **If users 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 bring down the sample stage to required EDS stage height (14 mm).** Please check **Section 6, Step 3** on page 3.
Failure to follow will lead to EDS detector crashing into sample stage. User’s SEM access will be suspended and repair cost will be charged on PI’s account.

4) **EDS dead time** adjustment (check steps in Spectrum Acquisition mode below):
   - 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%.
   
   **Tips:**
   - To improve EDS mapping resolution, the dead time should be kept just above 10%. The input count rate (ICR) should be at least one hundred of kcps to keep a good signal/noise ratio.
   - For spectral acquisition, the dead time can be kept at around 30%.

5) 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 PCSEM window, decide the image magnification and make an initial adjustment on focus and stigma to get a good SEM image.
     b) In EDS Esprit window click on the left side menu to enter Spectra workspace
     c) Click button and check and adjust the Dead time within \(10 - 30\%\):
        - If the Dead time is below 10%, in PCSEM window (do the opposite steps if Dead time is above 30%):
        - Increase the e-beam emission current (maximum 30 µA);
        - Choose High Probe current;
        - If necessary, change Cond. Lens 1 setting from “5” to “1” or “2”, the smaller number the higher the signal intensity.
d) Leave spectral acquisition on, go back to PC_SEM window, adjust focus and stigma to get sharp SEM image.

e) If a good signal/noise ratio has achieved on the spectrum, click again to stop acquisition.

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

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

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

e) Select the desired object type on the bottom menu bar and click on captured image above to specify positions.

Tips:

> The EDS spatial resolution is e-beam voltage/overvoltage dependent which is around 1 µm.

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

> Allow longer collection time if the point or small square or circle objects are chosen.

f) Click Select all to highlight all objects and click

g) If a good signal/noise ratio has achieved on the spectrum, click again to stop acquisition.

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

i) To save the spectrum, click the lower spectrum chart.

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

e) Highlight the line and drag and adjust the endpoints to the desired position

f) Set **Point count** of the line scan and click 

![Line scan icon](image)

line scan

<table>
<thead>
<tr>
<th>Point count</th>
<th>Distance [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.002</td>
</tr>
</tbody>
</table>


![Elements icon](image)

g) Use the **Elements** icon to identify elements

h) If **a good signal/noise ratio** has achieved on the spectrum, click **again** to stop acquisition.

i) To save line scan data, click the **button on the top right corner of the workspace window.**

j) To save the profile, click the lower profile chart .

8) **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](image)

<table>
<thead>
<tr>
<th>Size 800 px</th>
</tr>
</thead>
<tbody>
<tr>
<td>16 µs</td>
</tr>
<tr>
<td>8 s</td>
</tr>
<tr>
<td>Drift qual. -- %</td>
</tr>
<tr>
<td>Drift range -- %</td>
</tr>
</tbody>
</table>

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.**

![Acquire button](image)

g) Use the **Elements** icon to identify elements in spectrum window during mapping.
h) **Before stop mapping:**
   > Click **Spectrum** tab above the top right corner of **Map** window, make sure a **good signal/noise ratio** on interested elemental peaks 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 **Acquire** again to stop **Mapping**.

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

k) To save the map image, click the lower image window.

l) To save individual element image in the thumbnail on the bottom, click the **I/O** button.

9 **Closing EDS detection:**
   1) In **PC_SEM** program, click the **OFF** button to **turn off** electron beam
      **Note**: the beam can be left ON if plan to do further SEM imaging.

2) Click **Standby** button in **EDS DETECTOR CONFIGURATION** window to switch the **EDS** detector to **Standby Mode**
3) Check EDS detector window position and **make sure it is switched back to 20 kV**:

**Warning:** It is crucial to set the detector window back to 20 kV to avoid e-beam damage. **Violation** will lead to detector damage and repair charge will be applied to user’s PI account.

4) **Fully retract EDS detector** by clicking in EDS Esprit program and **wait until the detector is fully retracted**.

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

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**.
   - Change the **Cond. Lens1** back to “5.0” in the window below:

   ![Cond. Lens1](image)

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

   ![Exchanging Stages](image)

   - Click **Set** button in the **Stage** tab; uncheck **FG-EDX** box below.
7) **Wait for** the EDS detector temperature rises above 20°C (~ 20 minutes, be patient!) and then take sample out of SEM chamber.

**Warning:** Never start sample transfer while EDS detector is still being cooled. This will induce contamination on the detector if still below 20°C when the Specimen Chamber door is open.

8) **During EDS detector warmup:**
   > Make sure sample holder is at exchange (EXC) position
   > Data transfer is complete
   > Log off FOM on SEM computer in your FOM account.

9) Check to make sure the EDS detector temperature rises above 20°C and then
10) Turn ON small LCD
11) Finish sample unloading from SEM chamber.
12) Turn OFF small LCD
13) Log off FOM on EDS computer
    **Note:** Keep Esprit program ON and maximized on screen
14) Clear the work bench and dispose gloves, Kimwipes…