Bruker EDS on Zeiss SEM
Standard Operating Procedure

These instructions are intended for reference only, and will not replace the thorough training required for proper system operation. Contact the Electron Microscopy staff member with questions or to report a system problem.
1. Enable the Zeiss SEM in BADGER

2. Make sure that the Blue light on the detector is on.

3. Make sure that the receiver behind the monitor is also on.

These two should be always on! Never turn them off.
4. On the main Zeiss PC (left monitor), launch the **RemCon32** application in the task bar.

5. In the **RemCon32** click on the **green tick**.

   This will connect the Zeiss SEM to the Bruker EDS server.

   Yellow texts will show up on the black screen, if the connection goes well.

6. On the third monitor on the right (Bruker PC), load the **ESPIRIT** data acquisition and analysis software.

   Use your login information then press Login.

   **Public** is the general Username, no password.
7. After loading the software, make sure the application window is maximized, and you can see the icons on the left bottom side of the screen.

8. **Workspace** options on the left side of the ESPIRIT application.

   You can only view one of these column at a time.

9. **Workspace** options

   - **Spectra** allows spectrum acquisition and analysis of (saved) X-ray spectra.
   - **Objects** permits point, multipoint, and area EDS analysis.
   - **Line scan** is used to perform qualitative and quantitative line scan EDS analysis.
   - **Mapping** allows the acquisition of maps: intensity maps and HyperMaps (Bruker’s
position- tagged spectrometry tool (spectral imaging)). Mapping also includes the chemical phase analysis tool **AutoPhase**.

**Imaging** allows capturing and processing saved electron microscope images.

<table>
<thead>
<tr>
<th>10. Configuration Bar</th>
</tr>
</thead>
<tbody>
<tr>
<td>The individual configurators display relevant adjustable parameters of the sample, X-ray excitation sources, scan unit, spectrometers, as well as the report and project tools.</td>
</tr>
<tr>
<td>Clicking on the <strong>small arrow</strong> shows the <strong>adjustable parameters</strong>.</td>
</tr>
</tbody>
</table>
### 11. Sample Configuration

The Sample configurator is used to display and manage information about the sample (optional).

- Sample name
- Coating type
- Company
- Batch
- Sample number

### 12. Microscope Configuration

Displays the main parameters of the electron microscope.

**Nothing to change here.**

The WD and Magnification and kV are readings from Zeiss SEM.

### 13. Scan Configuration

**Image resolution:** Image size in pixels

**Dwell time:** Signal collection time of a single pixel during image acquisition

**Frame time**
Do not change these parameter unless you know their functions.

For more info refer to the software Manual page 35

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Image resolution [pixel]</td>
<td>512</td>
</tr>
<tr>
<td>EDS Configuration</td>
<td></td>
</tr>
<tr>
<td>Displays the measurement parameters of the available EDS detector(s).</td>
<td></td>
</tr>
<tr>
<td>1) Make sure the green bar is on</td>
<td></td>
</tr>
<tr>
<td>2) This icon means the detector is inserted</td>
<td></td>
</tr>
<tr>
<td>3) Pulse Throughput (cont.)</td>
<td></td>
</tr>
</tbody>
</table>
### 15. Pulse throughput

This control box lists the signal processor settings available for the spectrometer hardware. When **Automatic** is checked, QUANTAX selects the throughput setting according to the current pulse load at the beginning of a spectrum acquisition or measurement.

**Maximum energy (normally 20 keV).** The available energy ranges are listed. When **Automatic** is checked, QUANTAX sets the energy range according to the microscope high voltage (kV).

**Pulse throughput:**

- Low kcps numbers $\rightarrow$ higher signal quality, but higher dead time ratio (a lot of signal cannot be processed by the detector) $\rightarrow$ The signal throughput (bar 🟢) turns red.
- Higher kcps numbers $\rightarrow$ lower signal quality, but lower dead time ratio $\rightarrow$ all of the received signal will be processed (bar 🟢) is green.

Start the Pulse Throughput with a number 275 kcps and change it to lower numbers and see bar. If the bar turns red select higher kcps number, if the bar is in very low range, you can select smaller kcps number.

### 16. Data Acquisition and saving your data

Your data are shown in some of the examples below:

(For more detailed examples on data quantification please refer to ESPIRIT manual, pages 81-115). Some examples are given at the end of the SOP.

- **Example – Spectrum Acquisition** (page 10)
- **Example – Using Spectrum Chart** (page 12)
- **Example – Map Acquisition** (page 15)
- **Example – Line Scan** (Page 17)
### 17. Finishing Your Session:

1. Make sure you have **saved** your data and projects.

2. **Remove** the **Projects** and **Reports** from the top right of the ESPIRIT software. (they will remain in your folder)

3. Close the ESPIRIT software

### 18. Follow the standard instructions for continuing your SEM imaging session or removing your sample.

### 19. **BADGER LOGOUT:** Don’t forget to disable the tool in badger after you’re done.
Example – Spectrum Acquisition
This section describes the workflow for spectrum acquisition within the Spectra workspace.

<table>
<thead>
<tr>
<th>Step</th>
<th>Examples/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>Choose appropriate measurement conditions on the electron microscope.</td>
<td>The live spectrum appears in the spectrum window. Click the <strong>Preview</strong> button again to stop acquisition.</td>
</tr>
<tr>
<td><strong>1</strong> Select the <strong>Spectra</strong> workspace.</td>
<td></td>
</tr>
<tr>
<td><strong>2</strong> Click the <strong>Preview</strong> button to acquire a live spectrum.</td>
<td></td>
</tr>
<tr>
<td><strong>3</strong> Click <strong>Preview</strong> on the <strong>Preview</strong> button to set timing parameters in the <strong>Preview timing</strong> submenu.</td>
<td></td>
</tr>
<tr>
<td><strong>4</strong> Click <strong>Preview</strong> on the <strong>Acquire</strong> button</td>
<td>Set acquisition time.</td>
</tr>
<tr>
<td>a) Set <strong>Acquisition parameters</strong></td>
<td>Fast (50,000 counts) for major elements, Precise (250,000 counts) for minor elements, Exhaustive (1,000,000 counts) for elements close to the detection limit.</td>
</tr>
<tr>
<td>Manual</td>
<td>If <strong>Manual</strong> is selected, the acquisition has to be stopped manually by clicking the <strong>Stop</strong> button.</td>
</tr>
<tr>
<td>Real Time</td>
<td>The acquisition will stop after the time entered in the dialog box has elapsed.</td>
</tr>
<tr>
<td>Live Time</td>
<td>The acquisition will stop after the dead time corrected acquisition time has elapsed.</td>
</tr>
<tr>
<td>Counts</td>
<td>The acquisition will stop after the predefined number of counts is recorded.</td>
</tr>
<tr>
<td>b) Set <strong>Automatic quantification</strong></td>
<td>If <strong>Continuous/After acquisition</strong> is selected, the spectrum is quantified during/after acquisition. Click <strong>Load</strong> to access the method editor.</td>
</tr>
<tr>
<td>c) Activate <strong>Cyclic acquisition</strong></td>
<td>Load a quantification method using the button.</td>
</tr>
<tr>
<td>d) <strong>Spectrum numbering.</strong></td>
<td>This option acquires several spectra with identical settings from the same area. Set <strong>Cycle count and Pause [s]</strong>. Use <strong>Add to project</strong> to automatically add acquired spectra to project.</td>
</tr>
<tr>
<td></td>
<td><strong>Automatic numbering</strong> can be activated. The numbering will start with the entered <strong>Spectrum number</strong>.</td>
</tr>
</tbody>
</table>
5. Acquire a spectrum.

The acquired spectrum appears in the spectrum chart and the quantification results in the spectrum list. The spectrum can be further processed (element identification, quantification).

6. **Optional**
   - Use the workspace icon to:
     a) Add data to project
     b) Add data to report.
   Alternatively, drag and drop the spectrum from the spectrum list to Project or Report. The project has to be saved manually.

7. **Recommended**
   - Use the spectrum chart icon to:
     a) Save spectrum (or result list, element selection, graphic)
   Highlight spectra in the spectrum list to save several spectra. Alternatively, right click into the spectrum. Various file formats are possible*. To include all meta data use the .spx format.
   Alternatively, drag and drop the spectrum from the spectrum list to Project or Report. The project has to be saved manually.

*optional, license-based
Example – Using Spectrum Chart (similar in all workspaces)

This section describes the features of the Spectrum chart, available in the Spectra, Objects, Line scan, and Mapping workspaces. (See next two pages for the instruction)
Step

The spectrum list shows:

1a Spectrum type: excitation and/or analysis method

1b Spectrum name

1c Spectrum color

1d Options

1e Factor: scaling factor of y-axis

1f Results.

2 Multiple spectra can be selected (checkbox) or highlighted by clicking on the spectrum line (gray outline).

3 To delete a spectrum press the DEL key on the keyboard or on the spectrum chart.

To scale spectrum diagram or zoom, use either:

4a Mouse scroll wheel to change x-scale

4b Click and hold mouse scroll wheel to move spectrum area

4c Use CTRL key + left mouse click into the spectrum diagram + drag the mouse to scale x- and y-axis

4d Right click on x- or y-axis: scale values can be entered manually

Example/hints

The spectrum name has to be unique in the spectrum list. It can be changed after clicking on the name.

Click on the color bar to toggle between filled and unfilled display. Click on to select the color.

Click to select Pulses, cps, Net counts, energy resolution of peak selected by the cursor (FWHM fit).

Available when individual scaling is checked in the menu.

Click to select Spectrum information, identification or Display of quantification results (Mass-%, Mass-% (norm.), Atom-%, Stoich.-%, Stoich.-% (norm.)).

Checkbox selects spectra for display, highlighted spectra can be processed and exported. To highlight several spectra use SHIFT or CTRL keys and left click on the spectra. When exporting multiple spectrum results, the result table contains methodical error values (Sigma).

Alternatively, drag and drop spectrum from the spectrum list onto the icon.
4e Use the [A scale] icon for Automatic scaling (spectrum fills display area)

4f Use the [Zoom] icon to zoom in the spectrum window.

5 Use the [Options] icon or right click on the spectrum chart and select Properties... to change Spectrum display properties.

6 Click next to the x-axis dimension.

7 Click next to the y-axis dimension.

8 Right click on the x or y scale bar.

9 Double click on a spectrum in the spectrum list to access Spectrum properties (Results, Sample info, Detector, Parameter, and Spectrum).

10 Click the [Math] icon to select the desired operator.

11 Click the [Element] icon to select or de-select an element.

12 Select the tab [Finder] in the periodic table.

13 Highlight a region in the spectrum (by dragging the spectrum cursor with the right mouse button pressed) and select a free region (F1-F8).

14 Click the [Search] icon on the right side of the spectrum chart and choose the location where the reference spectra are located. Then click [Start search].

15 Use the spectrum chart [Add] icon to select Add to project in the Element selection section.

Set a rectangle by holding the left mouse key to zoom into the area of interest.

To normalize multiple spectra for comparison, select Individual scaling and Automatic and select an energy region with right mouse button.

Toggle between energy (keV) and channel.

Toggle between cps/eV or channel and pulse/eV or channel.

Adjust the x and y scaling.

Quantification results, sample info, detector (detector parameters), parameter (Acquisition parameters), spectrum (Energy calibration data) can be here retrieved.

The SPECTRA ARITHMETIC dialog opens. Available from the dropdown list are sum, absolute difference, relative difference, quotient, maximum and minimum. The resulting spectrum will be added to the spectra list.

Any element can be selected or de-selected just by clicking the according symbol.

The Finder option supports the identification of unknown peaks in the spectrum. Place the spectrum cursor over the center of a peak or highlight the peak range by dragging the cursor with pressed right mouse button to display a list of all possible elements. The first element of this list is that one with the highest probability of actually being present in the sample.

Up to 8 spectral regions can be assigned to monitor non-analytical peaks or background levels.

SEARCHING FOR SIMILAR SPECTRA dialog opens. Found spectra will be added to the spectrum list in the dialog. Use the sensitivity slider (cross correlation factor) to optimize search results.

Add the Element selection to the project.
Example – Map Acquisition

This section describes the acquisition of maps using the Mapping workspace. Maps can be saved as element distribution images or datacube (HyperMap data, recommended).
Step 1: Select the [Mapping] workspace.

Step 2: Click [ ] on the scan configurator to set Image resolution, Mapping dwell time and Line average.

Step 3: Click [ ] and adjust image.

Step 4: Click [ ] on the [Capture] button to open the Capture parameters menu.

Step 5: [Capture] an image.

Step 6: Click [ ] on the [Acquire] button and set parameters for Map time, After measurement and Map area.

Step 7: [Acquire] a map.

Step 8: Use the workspace [ ] icon to
   a) Save Map data
   b) Add data to project
   c) Add data to report.

Step 9: Use the chart [ ] icon of Map tab to
   a) Save map image (Element selection, Settings, Result table)

Optional:
   b) Add item to project
   c) Add item to report

Step 10: Use the thumbnail bar [ ] icon to
   a) Save Images

Example/hints

- Use high count rate for good count statistics.
- Image resolution defines the pixel resolution.
- Adjust brightness, contrast and magnification on the microscope.
- Adjust Capture parameters for the image: set single
- Mapping is automatically terminated if Measurement time [s] or Cycles is set. When Manual is selected acquisition has to be stopped manually by clicking the [Stop] button. One click terminates scan after finishing the last frame, a second click terminates the measurement immediately.

- Use file format:
  - *bcf*: To save HyperMaps (datacube, hyperspectral data set). Spectra for each pixel are saved. Further processing is possible only, when data is saved as a .bcf file.
  - *rtm*: To save element distribution images without point spectra.
  - *raw*: To save hyperspectral datacube for further processing with third party softwares (NIST Lispix format).

  Note: Only the EM image, composite element images and Map spectrum will be added to a Project and Report, not the whole hyperspectral database (HyperMap).

- Use image file formats (.bmp, .png, .jpg, .tif) to save the composite element image. Alternatively, click with the right mouse button into the map.

  Composite element image will be added to Project or Report.

- Individual element images of selected thumbnails will be saved. To select them mark the individual element images with left mouse-click + SHIFT or CTRL key.
### Example – Line Scan

This section describes the steps of line scan analysis using the Line scan workspace.

<table>
<thead>
<tr>
<th>Step</th>
<th>Example/hints</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Select the Line scan workspace.</td>
<td>Alternatively select Distance [µm] between measurement points.</td>
</tr>
<tr>
<td>2. Capture an image.</td>
<td>Choose Automatic, Manual or Measurement time [s]</td>
</tr>
<tr>
<td>3. Highlight the line and drag and adjust the endpoints to the desired position.</td>
<td>The line scan is automatically terminated if Automatic or Measurement time [s] is selected. When Manual is selected, acquisition can be stopped manually by clicking the Acquire button. One click terminates scan after finishing the last scan (the button changes into Stop), a second click terminates the measurement immediately.</td>
</tr>
<tr>
<td>4. Set Point count of the line scan.</td>
<td>Elements are automatically identified by Auto ID. Add and delete elements by clicking on the Element ID or use the Finder in the Spectrum tab. Identified elements are displayed in the line profile thumbnail bar. The element selection can be changed at any time during or after the line scan acquisition.</td>
</tr>
<tr>
<td>5. Click at the Acquire button to set Scan time.</td>
<td>Optionally use All or None on the right of the thumbnail bar. Use the icon to edit the line profile display settings, e.g. scaling of the x-axis, selection of result types, filter strength, etc.</td>
</tr>
<tr>
<td>6. Acquire a line scan.</td>
<td>Extracted spectrum (named as Range) appears in the Spectrum tab. (optional step). The Scan spectrum in the spectrum tab presents the sum spectrum of the line.</td>
</tr>
<tr>
<td>7. Use the icon to identify elements.</td>
<td>Use .rtf file format to save line scan data including EM and scan images and point spectra.</td>
</tr>
<tr>
<td>8. Select elements in the thumbnail bar by ticking the boxes below the individual element images to display their profiles in the Profiles tab.</td>
<td>Image(s), composite element profiles and scan image will be transferred. Confirm the pop-up window Do you want to save point spectra too? by clicking yes to transfer spectral data.</td>
</tr>
<tr>
<td>9. Use right mouse key in the scan image to extract region of interest spectrum from the line scan.</td>
<td></td>
</tr>
<tr>
<td>10. Use the workspace icon to</td>
<td></td>
</tr>
<tr>
<td>a) Save line scan</td>
<td></td>
</tr>
<tr>
<td>b) Add data to project</td>
<td></td>
</tr>
<tr>
<td>c) Add data to report</td>
<td></td>
</tr>
</tbody>
</table>
11 Use the Profiles tab icon to
   a) Save data
   b) Add data to project
   c) Add data to report.

Save the composite element profile as .bmp, .jpg, .png, .tif file format including Composite element profile, Scan image or both.

Alternatively, right click into the profile and use the local Line scan menu. Confirm the pop-up window Do you want to save point spectra too? by clicking Yes to transfer spectral data.

Highlight elements in profile thumbnails. Select Individual element profiles to add separate profiles for each element. Select Composite element profile to add all selected elements in one diagram and the scan image.

12 Use the thumbnail bar icon to Add Line scan to report.