

QUANTAX COMPACT EDX SYSTEM SOP

Revised July 2020

Energy-Dispersive X-Ray Spectroscopy (EDS, EDX, EDXS or XEDS), is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing a unique set of peaks on its electromagnetic emission spectrum.

SOFTWARE OVERVIEW

Workspaces are ESPRIT Compact screen areas in which images and spectra are displayed, the analysis is performed and the results are managed. A workspace can be selected by clicking on the corresponding workspace icon. The active workspace icon is highlighted red.



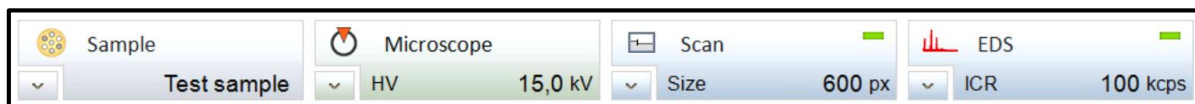
ESPRIT Compact workspace buttons

Objects: permits point, multipoint and area EDS analysis.

Line scan: is used to perform qualitative line scan EDS analysis.

Mapping: allows the acquisition of intensity maps and HyperMaps.

System: is used to adjust global settings and to calibrate the X-ray spectrometer and the image system. Additional functions are the display settings (language, font size), the license management and the communication with the electron microscope.



Configurator bar

The **Sample configurator** is used to display and manage information about the sample. The information, which can be displayed are the following parameters:

- Sample name
- Coating type: If the sample is coated to enhance electrical conductivity, therefore under **Sample coating** the following option could be selected:
 - **None**, if the sample is not coated **OR**
 - **Coating with ...** Select the element of the coating material in the periodic table which pops up after selecting this option.
- Company
- Batch
- Sample

The **Microscope** configurator displays the main parameters of the electron microscope:

- High voltage: Acceleration voltage set on the electron microscope.
- WD (working distance): Sample – pole piece distance in mm.
- Magnification: Image magnification set on the electron microscope.
- Stage position X, Y, Z: Microscope stage position coordinates are displayed.
- Sample tilt angle: Angle of pre-tilted sample surface

The **Scan** configurator displays the scanning parameters:

- Image resolution: Image size in pixels
- Pixel dwell time: Signal collection time of a single pixel during image acquisition
- Frame time

The **EDS** configurator displays the measuring parameters of the available EDS detector(s).

- Pulse rate (input count rate)
- Energy range
- Maximum pulse throughput
- Temperature
- Real time
- Live time
- Dead time

For more details about the operational procedure and other ESPRIT features, please see the **User Manual**. Complimentary videos covering basic operational procedures are also available. Both could be found on the Desktop of Bruker computer.

STARTUP

1. Login in your session on the FOM. Start the SEM imaging as normal.
2. Find the region of interest and adjust magnification. Adjust the beam to **20 kV**, spot size of **10 - 12** and working distance on the microscope **WD=8.5**. Switch ON the beam.

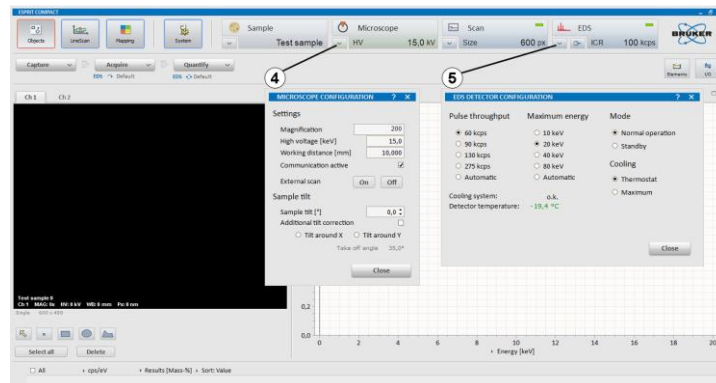
NOTE: For EDX analysis, as a general rule, select an accelerating voltage of at least **2x** the highest peak energy expected. **For example**, if iron is the highest energy peak at 6.39, the minimum kV should be about 14 or 15.

NOTE: Sample tilt **SHOULD** be **0.00 degrees**.

3. Click on Esprit Compact Icon and log into software.

Login: **User**,
Password: **user**


4. Use the icon in the bottom left corner of the Microscope configurator to open the **MICROSCOPE CONFIGURATION** dialog and check the settings.
5. Use the icon in the bottom left corner of the EDX configurator to open the **EDX DETECTOR CONFIGURATION** dialog and
 - a. Set Pulse throughput and Maximum energy
 - b. Check the Detector temperature. Check the detector temperature. The device status indicator should be green.



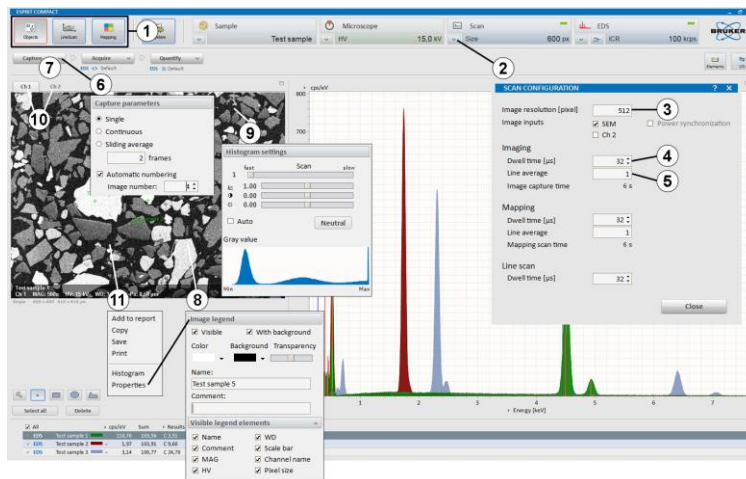
6. Check input count rate for the analysis. The input count rate should be chosen according to the analytical task.
7. Proceed with required analysis.

IMAGE CAPTURE

This section describes how to capture electron microscope images with the ESPRIT Compact software.

1. Select a workspace (,  or ).

2. Use the icon in the bottom left corner of the Scan configurator to open the **SCAN CONFIGURATION** dialog.
3. Set image resolution (pixel). **512 pix** is recommended for Object and Line data acquisition, but can be changed for higher settings, **1000 pix** is max setting in case of Map acquisition.
4. Set dwell time [μ s], **8 μ s** is recommended
5. Set line average, **1** is recommended
6. Click on the **Capture** button to set capture parameters and Image number or Automatic numbering.
7. **Capture** an image.
8. Image legend parameters can be edited by right mouse click in the image and selecting **Properties**.
9. Right mouse click in the image and select **Histogram** to change brightness and contrast settings of the image.
10. Toggle between two image channels if available.
11. Right mouse click in the image to **Save image, Copy image** or **Add image** to report.



ACQUISITION

This section describes the workflow for spectrum acquisition within the Objects workspace.

1. Choose appropriate measurement conditions on the electron microscope.
2. Click on the **Acquire** button to
 - a. Set **Acquisition parameters**.

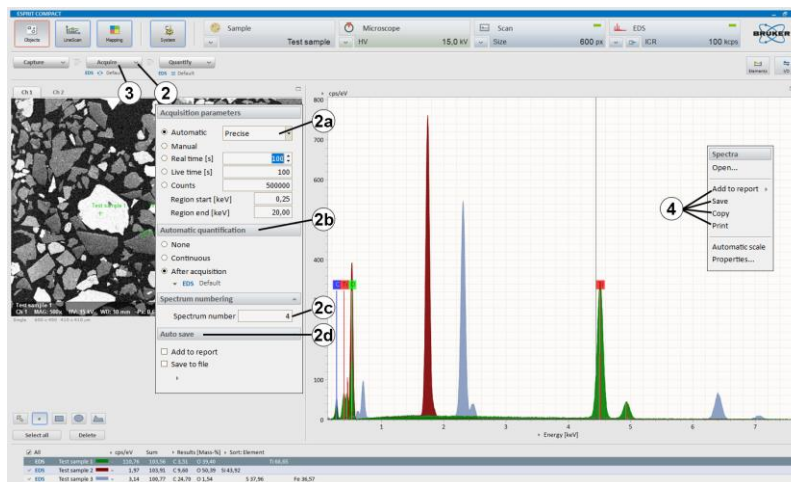
Automatic: **Fast (50,000 counts)** for major elements,
Precise (250,000 counts) for minor elements (**recommended**),
Exhaustive (1,000,000 counts) for elements close to the detection limit.

Manual If Manual is selected, the acquisition has to be stopped manually by clicking the button.

Real Time The acquisition will stop after the time entered in the dialog box has elapsed.

Live Time The acquisition will stop after the dead time corrected acquisition time has elapsed.

Counts The acquisition will stop after the predefined number of counts is recorded.
 - b. Set **Automatic quantification**. If Continuous/After acquisition is selected, the spectrum is quantified during/after acquisition. Load a quantification method using the button.
 - c. **Spectrum numbering**. The numbering will start with the entered Spectrum number.
 - d. **Auto save**. Select Add to report to send the data automatically after acquisition to the report. Select Save to file to save the data automatically after acquisition. In the pop up dialog the data storage location can be chosen.
3. 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).



4. Right mouse click into the spectrum chart to Save the data 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, add item to report or Copy the data.

QUANTITATIVE EDS ANALYSIS OF BULK SAMPLES

This section describes the steps of automatic standardless quantification and quantification using the quantification dialog.

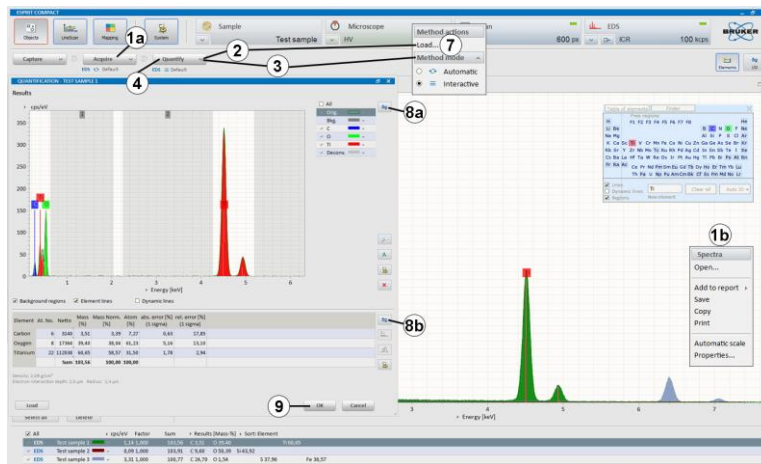
1. Select the **Objects** workspace and
 - a. **Acquire** a spectrum
 - b. Use a right click in the Spectrum chart to **Open...** a spectrum.
2. Click on the **Quantify** button and select **Load...** to load a quantification method. Select a quantification method (e.g. Default or, Oxides).
3. Click on the **Quantify** button and select a **Method mode**.

☰ Interactive method ↻ Automatic method

4. Start quantification by clicking the **Quantify** button.

If quantification is set to automatic (↻), the quantification results appear in the spectrum List. If quantification is set to interactive (☰), the **QUANTIFICATION** dialog pops up.


5. Review spectrum chart. Compare deconvolution results with measured spectrum.
6. Review quantification result table.
7. **Load** another quantification method, if needed. After loading the method, the quantification routine will run immediately and automatically.
8. a. Use the I/O icon in the **QUANTIFICATION** dialog to Save, Print the spectrum diagram or to Add it to report. Use this to export deconvolution results or background-subtracted spectra. Copy and Save the diagram as graphic is also possible.
 - b. Use the Results icon to Copy, Save, Print or Add to report.



9. Click **OK** to close the **QUANTIFICATION** dialog. The results will be displayed in the spectrum list. Click to stop the quantification and come back to the spectrum chart without adopting any changes.

OBJECT ANALYSIS

This section describes the steps of user-defined area analysis with image capture using the Objects workspace.

1. Select the **Object** workspace.
2. **Capture** an image.
3. Select the desired object type. Objects types are point, rectangle, ellipse, and polygon.
4. Draw an object in the captured image. The object can be edited (moved or resized) when the icon is activated. Click **Delete** to delete selected objects.
5. Use **Select all** to highlight all objects.
6. Click at the **Acquire** button to set acquisition parameters. If automatic quantification is desired, check if automatic analysis After acquisition or **Continuous** is selected.
7. **Acquire** a spectrum. Objects are automatically acquired in sequence. Progress is indicated by the acquisition bar. If **Manual** is selected, the acquisition has to be stopped manually.
8. The acquired spectra will appear in the spectrum list.
9. Use the workspace  icon to

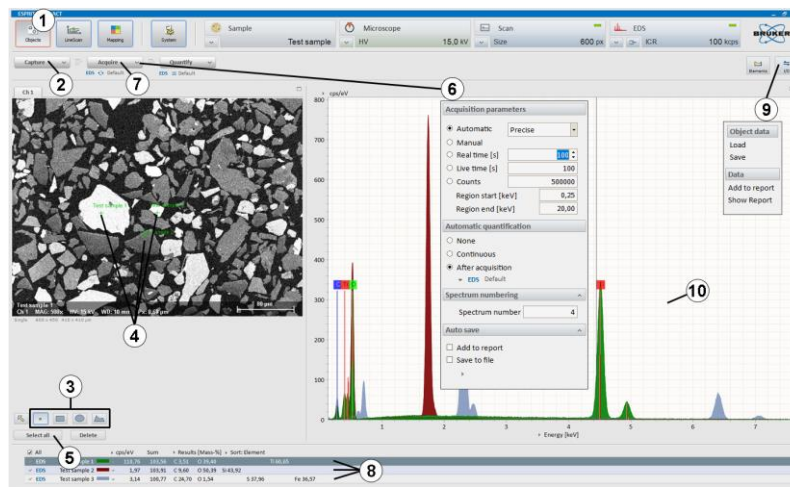
Save object data Use *****.rto** file format (This file type contains all available EM images including objects and corresponding spectra) or Add data to report.

10. Use a right mouse click in the spectrum chart to Save spectrum or Add spectrum to report.

Select Spectra list to add separate diagrams for each spectrum. Select Multiple spectra to add all selected spectra in one diagram.

11. Use a right mouse click in the spectrum list to Save result table or Add result table to report.

Select Single table to add separate tables for each spectrum. Select Multiple tables to add all selected spectra results in one table.




LINE SCAN


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

1. Select the **LineScan** workspace.
2. **Capture** an image.
3. Highlight the line and drag and adjust the endpoints to the desired position.
4. Set **Point count** of the line scan. Alternatively select Distance [μm] between measurement points. The distance between points and electron beam spot size is shown in a display and will change depending on whether Point count or Distance [μm] has been selected. The calculation of the spot size depends on the used high voltage.
5. Click at the **Acquire** button to set
 - a. **Scan type.** Choose Automatic, Manual or Measurement time [s]
 - b. **Auto save.** Select Add to report to send the data automatically after acquisition to the report.

Select Save to file to save the data automatically after acquisition. In the popup dialog the data storage location can be chosen.

6. **Acquire** a line scan. 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.
7. Use the  icon to identify elements.

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.

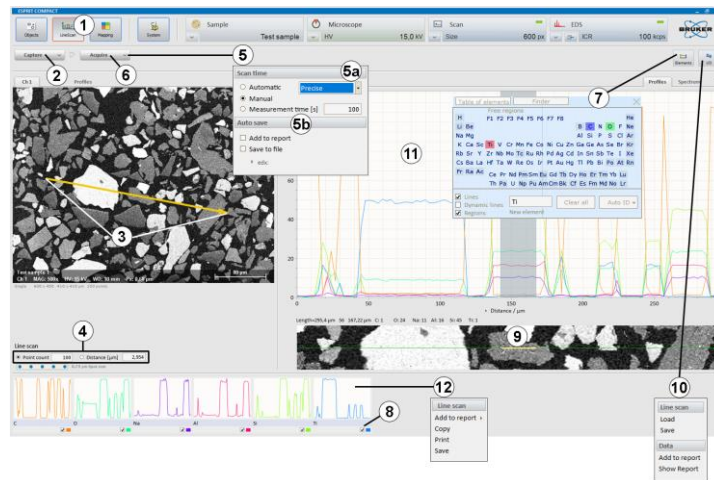
8. Select elements in the thumbnail bar by ticking the boxes below the individual element images to display their profiles in the **Profiles** tab. Use the right mouse click and select **Properties** to edit the line profile display settings, e.g. scaling of the y-axis, selection of normalization, filter strength, etc.
9. Use right mouse key in the scan image to extract region of interest spectrum from the line scan. The 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.
10. Use the workspace  icon to
 - a. Save line scan Use *****.rtl** file format to save line scan data including EM and scan images and point spectra.
 - b. Add data to report Image(s), composite element profiles and scan image will be transferred.

11. Use a right mouse click in the **Profiles** chart to

- a. Save data Use *****.rtf** file format to save line scan data including EM and scan images and point spectra. Save the composite element profile as .bmp, .jpg, .png, .tif file format.
- b. Add data to report. Composite element profile and scan image will be transferred.


12. Use the right mouse click in the thumbnail bar to **Add** a Line scan to report.

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.



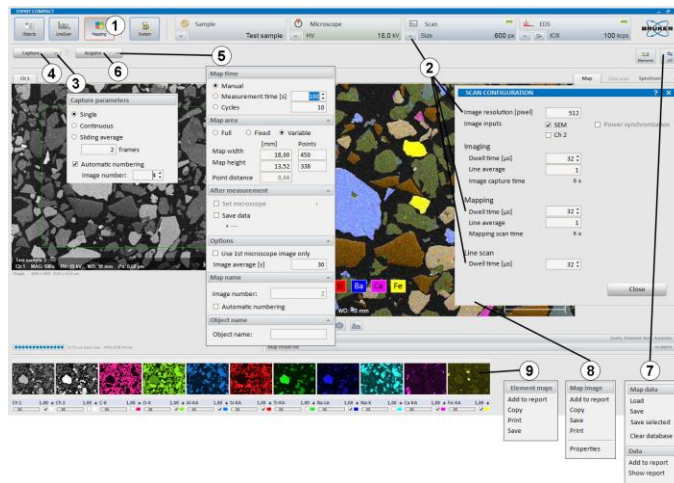
MAP ACQUISITION

This section describes the acquisition of maps using the Mapping workspace. Maps can be saved as element distribution images or datacube (HyperMap data).

1. Select the  workspace. Use high count rate for good count statistics.
2. Click on the Scan configurator to set **Image resolution**, **Mapping dwell time** and **Line average**. Image resolution defines the pixel resolution.
3. Click on the **Capture** button to open the Capture parameters menu. Adjust **Capture parameters** for the image: set single or continuous.
4. **Capture** an image.
5. Click on the **Acquire** button and set parameters for
 - a. **Map time:** 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 **Acquire** button. One click terminates the scan after finishing the last frame, the button will change to **Stop**. Another click on the **Stop** button terminates the measurement immediately.
 - b. **Map area:** Choose as **Map area: Full, Fixed** or **Variable** and the **Map width** or **Map heights** in [μm].
Choose the number of acquisition **Points** or the **Point distance** in [μm]. Points are equal to pixels in the map. The number of points alters the distance between points and the electron beam spot size shown in the display on the left side. The calculation of the spot size depends on the used high voltage.
 - c. **After measurement:** Set microscope to turn off the HV after measurement. Select **Save data** to save the data automatically after acquisition. In the pop up dialog the file name and the data storage location can be chosen.
6. **Acquire** a map.

NOTE: wait time 30 minutes for 10 kcps or less, 5-20 min for 20 kcps or higher.
7. Use the workspace icon to
 - a. **Save Map data.** 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.
 - b. Save **selected** map data, if only a part of the map area needs to be saved Mark the area to be saved by drawing a rectangle in the map chart with the rectangle tool. Only the data of the area within the rectangle will be saved.
 - c. **Add data** to report.
 - d. **Clear database.** HyperMap database will be deleted. Element distribution images without point spectra remain as well as the selected elements. No Elements can be added/selected anymore.

8. Right mouse click into the **Map** tab to
 - a. **Save** map image Use image file formats (.bmp, .png, .jpg, .tif) to save the composite element image.
 - b. Add item to report Composite element image will be added to the report.
9. Right mouse click into the thumbnail bar to
 - a. **Save** images 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.
 - b. Add **images** to report. Individual element element images will be added to report.
10. Process map data as desired.



SHUTTING DOWN PROCEDURE

1. When done, make sure you save all the data (spectrums, reports, etc.) and then exit the Esprit Compact program.
2. Adjust the beam energy to the lower setting ~3-5kV.
3. Finish work with microscope as normal.
4. Retract the EDS detector by turning the knob on the detector clockwise all the way up.