adolphe merkle institute
excellence in pure and applied nanoscience
UNIVERSITY OF FRIBOURG SWITZERLAND

Focused ion beam

Introduction

Version 1 – February 2022

FIB-SEM EDX

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Quick summary

Start
- Setup the FIB-SEM: Load sample, pump chamber, eucentric heigth, navCam, ...
- Get an image in the SEM
- Stop the chamber view camera
- Assure both PCs have the user logged in ('user')

Counts
- Insert the detector and start the PathFinder software
- Assure the SEM is acquiring
- Spectrum > start spectrum dropdown > high resolution > advanced arrow
- setup the SEM to have stores > 2000 and a dead time < 40-50% (ideal: 20-35%)

Spectrum
- Collect a spectrum
- Identify the elements

Data
- PointID
- Spectral imaging
- Linescan

End
- Export the data from the database
- retract the detector
- Extract your samples, set the FIB-SEM to standby
- Cleanup the desk

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Universal rules

Rule 1: don't touch a control if you are not sure of the outcome of that action

Rule 2: never, ever force anything beyond finger strength

Rule 3: wear gloves when touching anything that goes into the chamber

Rule 4: if in doubt, ask for help

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Demonstration: Ending your session

Prerequisites:
Data recorded and exported

Ready to extract your data from the database

Experiment: end the EDX session

1. Switch on the chamber view
2. Retract the detector entirely (must not be visible anymore in chamber view)
3. Close the pathfinder software
4. Stop the SEM beam and put the FIB-SEM in standby mode

Demonstration: setup the microscope

Prerequisites:
Functional FIB-SEM

Starting your session

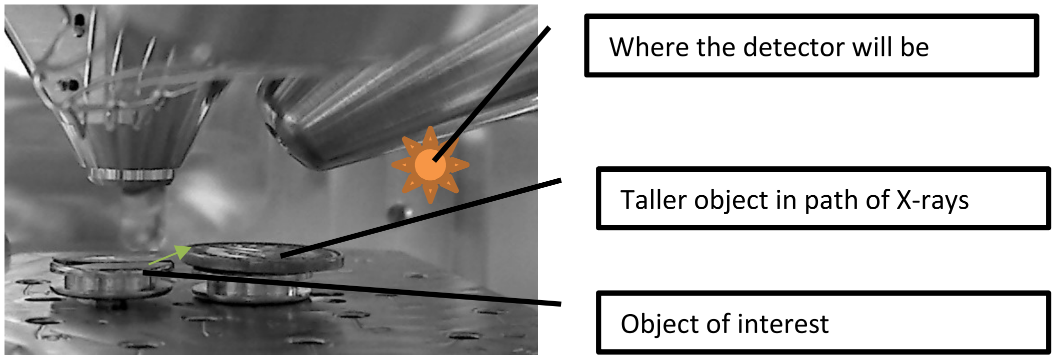
You do not need the FIB beam; the Ga ion beam is destructive and does not produce X-rays.

Experiment

Make sure you are logged in with username ‘**user**’ on the FIB-SEM microscope PC

Setup the basics at the FIB-SEM/SEM imaging
- Load your sample
- Home the stage
- Put the sample in eucentrich height / 7 mm
- Make a navcam picture
- Use the ETD for imaging

Assure that no large sample is blocking the way to right ride of the chamber (in chamber view). Below: suboptimal: the sample on the right might block the path



Demonstration: Saving your data

Prerequisites:
Data recorded

Ready to extract your data from the database

The data is automatically saved in the database. To extract the data from the database:

1. Export full field spectrum

- Look into your folder where you saved your database.
- There will be **an .emsa file** for every full spectrum you recorded[5].

2. Point ID spectra

- Look into your folder where you saved your database.
- There will be **a folder** with the base name of your recording.
- Inside the folder you will find a .psmsa file. This is equivalent to the .emsa files.

3. Extract spectral imaging maps

- Save the maps by File > save map files.
- You will be asked to give the map a type (e.g. counts)
- A new entry will appear in the data list (e.g. filename.counts).
- The maps of all elements can be found in the database root folder as tif files (using the color code as setup in Pathfinder).

3. Extract line scans

- Select your line scan
- Save as CSV or tiff by File > import / export

[5] .emsa files are text files with the spectrum and metadata.

Demonstration: Line scan

Prerequisites:
Detector inserted
Stores / s optimized

Record a line scan at high signal to noise

The functionality of the linescan is similar to the line scan extraction by spectral imaging. However, by only scanning the points along the line (and not the entire map), a longer integration time can be achieved and thus a much better signal to noise ratio.

Experiment: carry out a line scan

- In the microanalysis tab: click Linescan
- Similar to the spectral imaging, record a SEM Image and then record a linescan.
- Adjust the resolution of the scan if needed

Bug alert: You must save your file (File > close project) and reopen the database in order to adjust something to the output.

Demonstration: setup the beam

Prerequisites:
Sample(s) loaded
SEM bean on

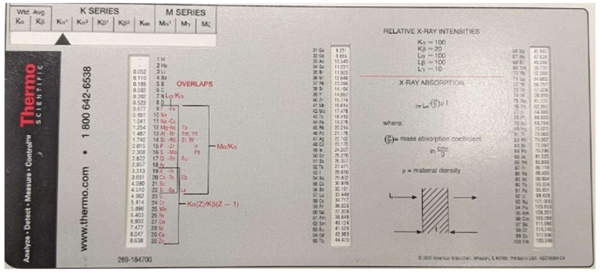
Starting your session

Over-voltage

The choice of the high voltage must be seen in the light of the elements you want to detect. As a rule of thumb:

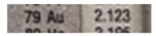
$$HV \geq \frac{E_{x-ray}}{0.5\ to\ 0.7}$$

Or at 30 kV (the maximum on the FIB-SEM), you can detect up to 21 keV edges. Use the Slider tool to extract the information. For example, it Au is the goal:

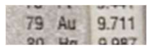


- The K_α edge of Au is at 68.794 keV (= undetectable)

- The M_α edge of Au is at 2.123 keV (= detectable)



- The L_α edge of Au is at 9.711 keV (= detectable if HV > 13.9 kV).



Therefore, 15-20 kV is a good setting to detect Au.

Beam current

The beam current can be set to standard values, e.g 0.4-0.8 nA.

Magnification

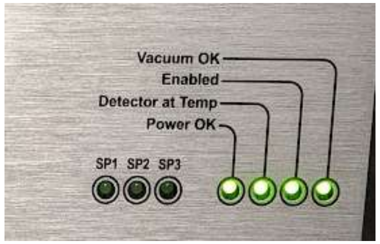
Do not zoom in too much to start. 500-1000X

Demonstration: Start the pathfinder software

Prerequisites:
Proper image under the SEM
Logged in under the username ‘**user**’ on the Suppoer (EDX) PC

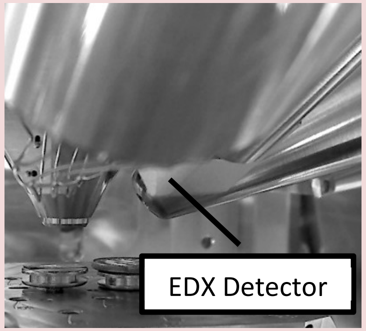
It is absolutely crucial that both PC are logged in with the same username: ‘user’

Before you start: On the right side of the table, check the LED’s on the SSD controller. All four LED’s must be green.



Experiment: Insert the detector

- Insert the Detector using the EDX switch on the table. Press insert for 2-3 seconds until you see the detector extended into the chamber view. The LED on the switch will turn red.



EDX detector retracted

EDX detector inserted

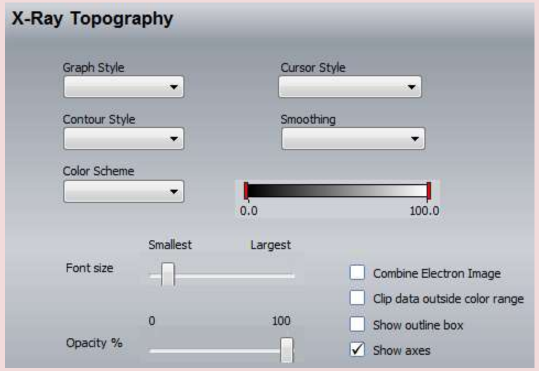
Demonstration: X-ray topography

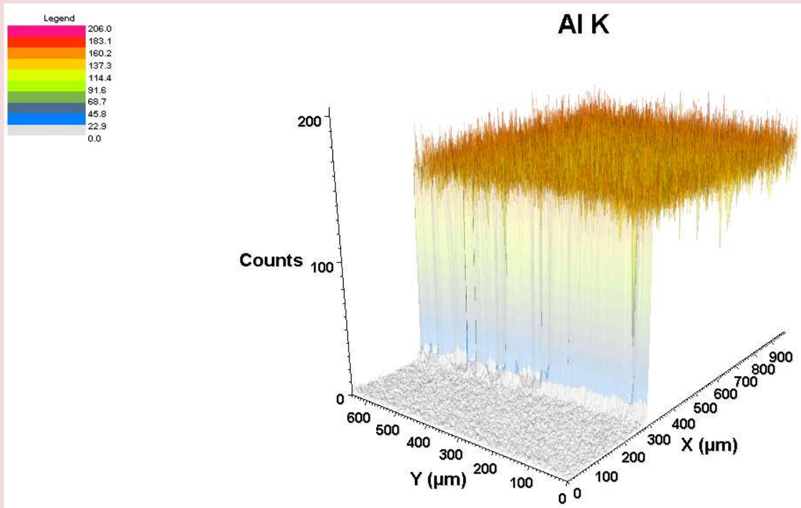
Prerequisites:
Spectral maps recorded

3D graphs of the X-ray distribution

Experiment: X-ray topography plots

- Select surface in the cursor tab
- The workspace changes. A large white field appears on the top left.
- Select an element in the maps
 Choose your settings in the X-ray topography tab





Demonstration: Phase maps

Prerequisites:
Spectral maps recorded

Extract compositionally distinct binary phases

Experiment: use Phases

In Map processing, find phases. You get a list of the elements (uncalculated phases)

Phase map type

Maximum intensity:	Uses MI in each pixel to create the phase maps
X Phase:	proprietary algorithm
Auto	No user input needed
Manual	Allows to set threshold

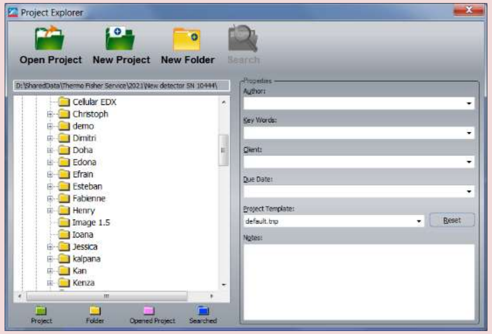
Experiment: start the Pathfinder software and login onto a database

- Start the Pathfinder (icon on the desktop) on the EDX PC

- A splash screen is visible for 10-15 seconds.

- You are asked to use a database. Use an exisiting one or create your own in your user folder.

- You do not have to actively start a cooling



Demonstration: optimizing X-ray counts

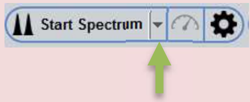
Prerequisites:
Detector inserted

This important setting is very hidden in the software

Before you start recording data, you should optimize the X-ray counts.

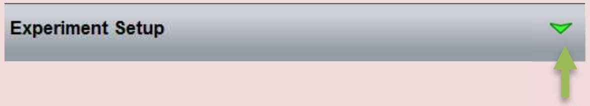
Experiment: finding the X-ray counts

- On the top right, in the microanalysis tab, choose spectrum:

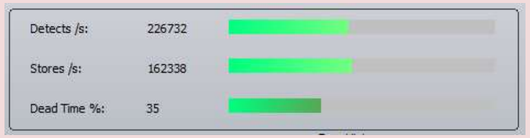


- The click the down arrow next to 'Start spectrum' (Do not start a spectrum!) and choose any setting (e.g. High resolution).

- Then, in the top right of the experiment setup, click the green down arrow



- Now, the counts and stores are at the bottom of the Experiment setup.



Experiment: optimizing the X-ray counts

1. Switch off the chamber view, as this also produces X-rays. Click the lower right quadrant and press F6. Make sure the topleft corner has this symbol: 

Demonstration: Multivariate statistical analysis[4] with COMPASS

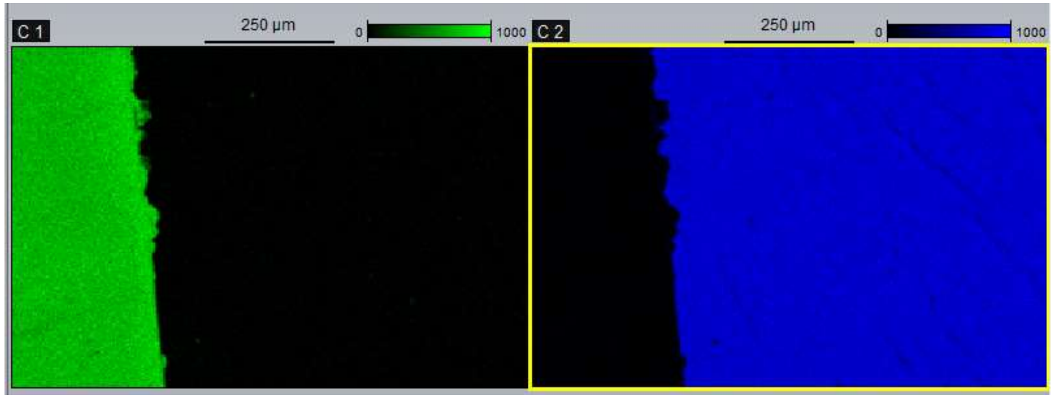
Prerequisites:
Spectral maps recorded

Extract compositionally distinct components without user input

Experiment: use COMPASS

Method

Area:	weigth by nearest neightbor, Recommended for most sample types
Spectral:	No NN. Intended for samples with many small phases
Background	
Using data:	for data with sufficiently high signal to noise
Using model:	for noisy data
Number of components	
0 (auto)	recommended as pilot
1-15	optimization options



[4] Based on principal component analysis

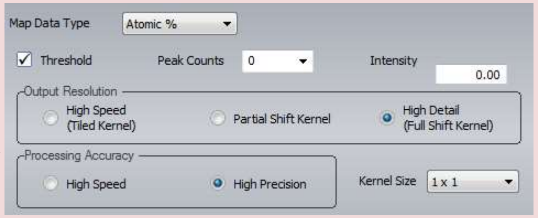
Demonstration: Map quantification

Prerequisites:
Spectral maps recorded

Converting counts into atomic %

Experiment: convert counts to atomic %

- Select QUANT in map processing
- Deselect ‘threshold’ to assure you process all maps
- Set the endpoints you want to achieve:
Atomic %, weight %, …
- time you want to invest (with 256px maps, it is a matter of seconds)
- Kernel will apply a mean filter, increasing The signal to noise at the cost of resolution.
- Click process



The result are maps with a dynamic range of 100 (from 0% to 100% atomic or weight %)

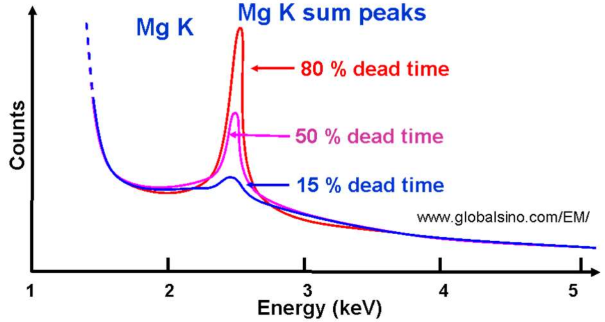
Intensity cursor

Now, use intensity cursor (‘+ cursor’) to output the atomic % for each element at a specific map (xy) position.

2. Start the SEM scanning (i.e. there is NOT the symbol  on the top right quadrant)[1]
   - Image size: e.g. 768 x 512
   - Scan speed for good imaging: 5-10 µs/pixel (depends on the image size)
   - Detector: ETD
3. Adjust the Beam current until you have
   - high stores count (> 2000, better > 10 000)
   - not too high dead times (15 > dead time < 40)

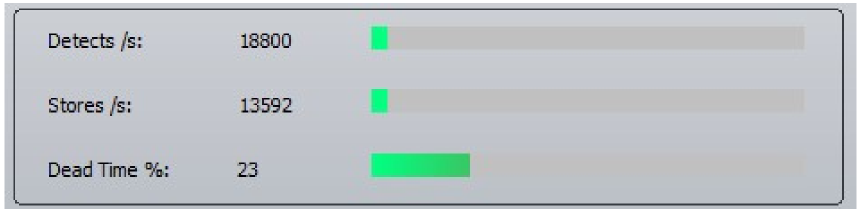
Dead time

- High dead times means inefficient collection
- High dead times cause artefacts, such as higher sum peaks
  A high dead time means the detector is swamped with X-rays while the collection



- However, lower dead times may mean lower stores / s

Example of a good setting



[1] At some point, the software can activate the sacanheads, but not at the first scan.

Demonstration: A word on the time constant

Prerequisites:
Detector inserted
Stores / s optimized

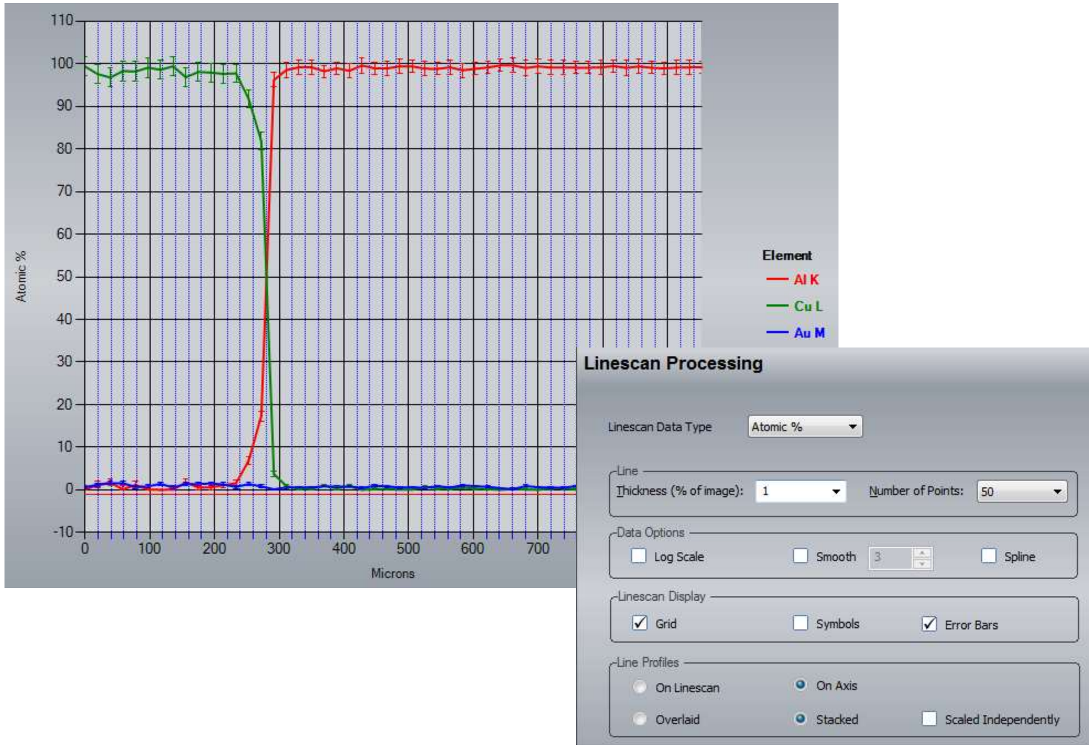
The time constant affects the quality of your detection

X-rays generated from a specimen are subjected to dispersion according to their energy. during acquisition, averaging of this noise is performed to obtain accurate values.

The time constant is a time index to define the noise collection. Changing the time constant, the averaging time for noise collection is changed.

High time constant	good average →	high-energy resolution EDS spectrum higher sum peaks, low throughput
Low time constant	poor average →	lower dead time, efficient imaging, high throughput More noise = lower spectral resolution

Appears on the image.
- Adjust the line (grab the edges)
- On the right: linescan processing
becomes available. Adjust if needed.



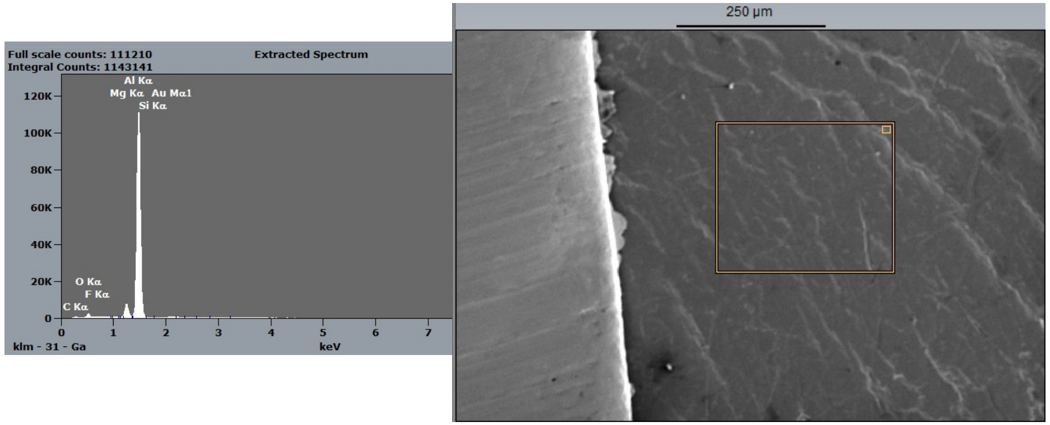
Demonstration: Extracting spectra from maps

Prerequisites:
Spectral maps recorded

Browse through the data in the spectral maps

1. Extract data from points or region of interests

In the bar under the SEM image, select a cursor type (rectangle, spot, …) and select a region in the SEM/Overlay image. The extracted spectrum will show up in the spectrum pane (make sure intensity cursor '+ cursor' is switched off).

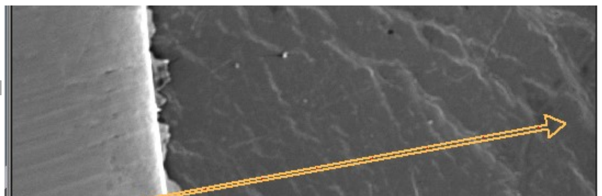


The spectral data is shown either as:

- Area integral — Total X-ray counts from the selected area (default)
- Trace element search — Maximum counts for each eV channel (useful for finding Small concentrations of an element)

2. Extract line plots

- Select 'Line'. An orange line overlay



Demonstration: Record a spectrum

Prerequisites:
Detector inserted
Stores / s optimized

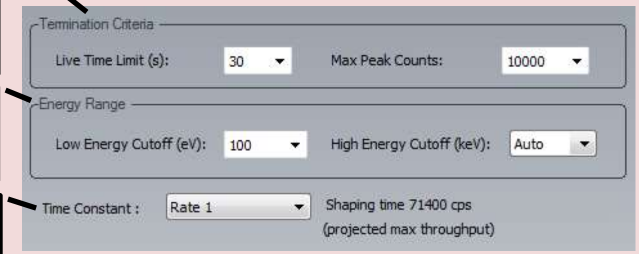
Record a spectrum of the entire field of view

Experiment

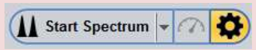
Stop the acquisition after e.g. 30 seconds, or a peak reaches e.g. 10 000 counts

Cut off artefacts/noise below 100 eV. Leave HEC to Auto

High throughput or high spectral resolution

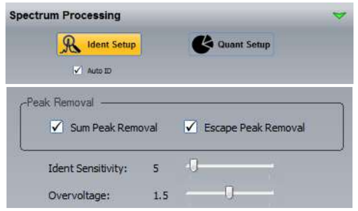


Click start spectrum



Result: Spectrum processing

- Assure that Auto ID is on & click the green arrow
- There are some options for the Auto ID
- Click process to repeat the analysis[2]



[2] Process will only start the identification of the peaks. Clicking Identify button in the bottom left will do the identification and the quantification.

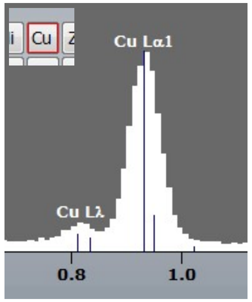
Demonstration: Identify using the periodic system

Prerequisites:
Spectrum recorded

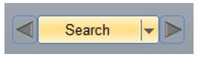
Record a spectrum of the entire field of view

KLM lines

With no auto ID run (all peaks unknown), left click on an element and the peaks will be overlaid on the spectrum.

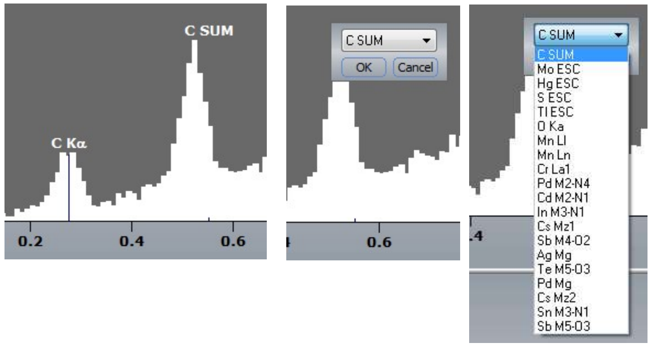


Click left/right on search to go through all elements.



Select the peak element

If the element was detected, double-click to see other available elements.



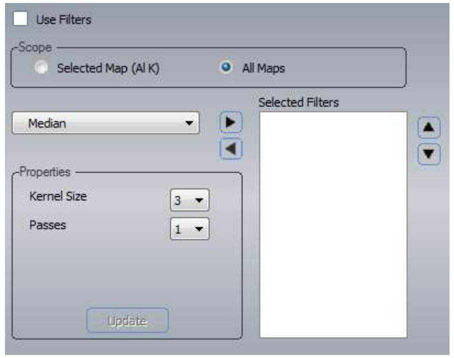
Compare with a synthetic curve

Comparing with a theoretical curve allows you to see if you missed any elements

The software allows to improve noise by filters (especially the smoothing filters).

- Filters will create severe artefacts in data with low dynamic range. Increase dynamic range by recording longer.

- In high dynamic range data, filters are not advisable.



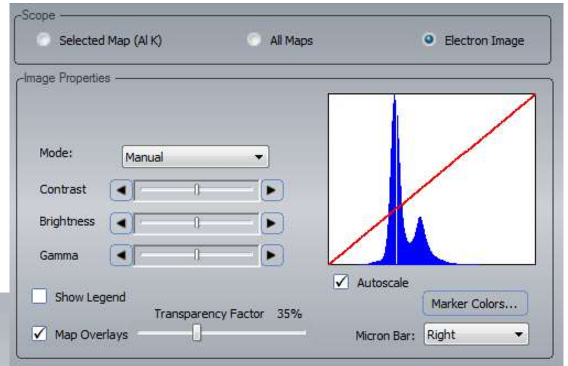
When you use filters, make sure you know what you are doing!

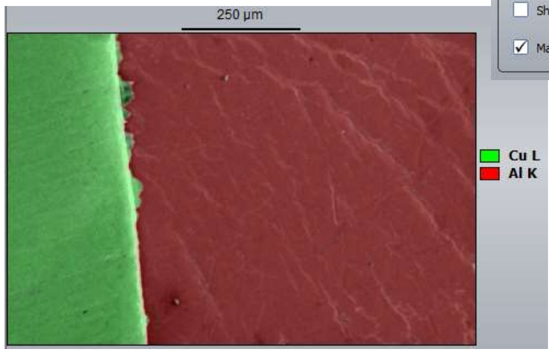
4. Change transparency of the overlays

- In Image settings, choose Electron image.

- BUG ALERT: Click Image filters and then again on Image settings!!

- You can now change the transparency of the overlays





Demonstration: Adjusting the spectral maps

Prerequisites:
Spectral maps recorded

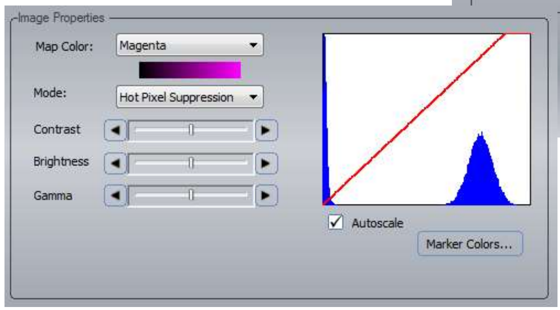
Change the visuals of the maps

1. Change the selected elements

Use the periodic table, right-click on the wrong element and choose 'excluded' to remove elements.

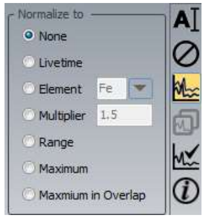
2. Change the false color

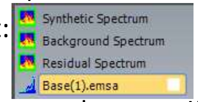
- Left click the map (a yellow border appears).
- Choose image settings
- Choose Selected Map (in this case Al K)
- **BUG ALERT: Click Image filters and then again on Image settings!!**
- Now you can change the colors



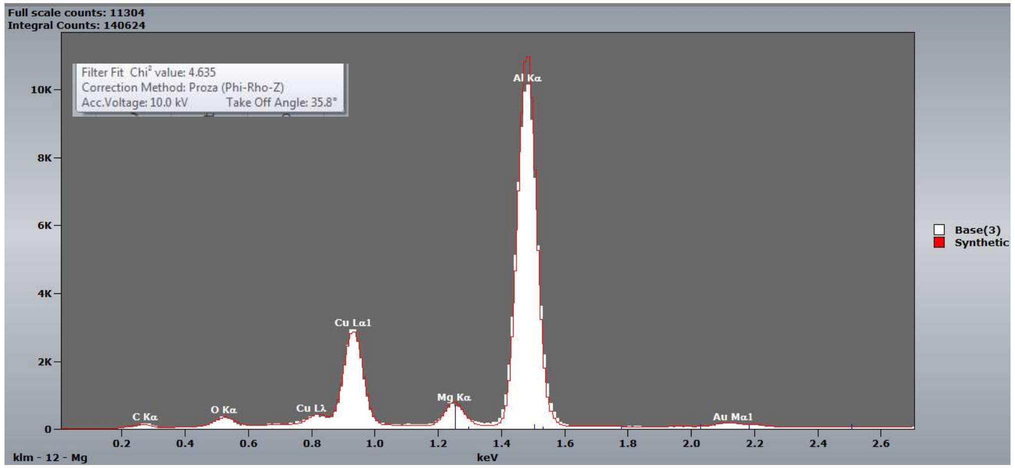
3. Image filters

Select your data from the bottom right and click the compare icon 



- Leave normalization to none, but click the spectra check: 
- 3 new entries become available in the list: 

- click Synthetic spectrum
- the goodness of fit is shown by hoovering over the quantification pane title. The lower the better.

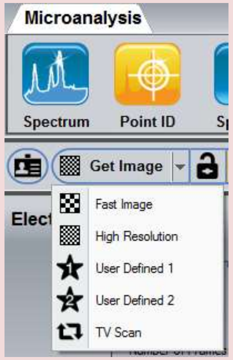


Demonstration: Point ID mode

Prerequisites:
Detector inserted
Stores / s optimized

Record a spectrum of the entire field of view

Experiment: get a point spectrum



1. Record an SEM image: Click Get Image after setting up the imaging details.

2. In the SEM image pane, select the geometry (point, …)
And draw or place your ROI. If instant is selected The scan will start Immediately. Otherwise, Click start spectrum

Demonstration: Spectral imaging

Prerequisites:
Detector inserted
Stores / s optimized

Record an elemental map

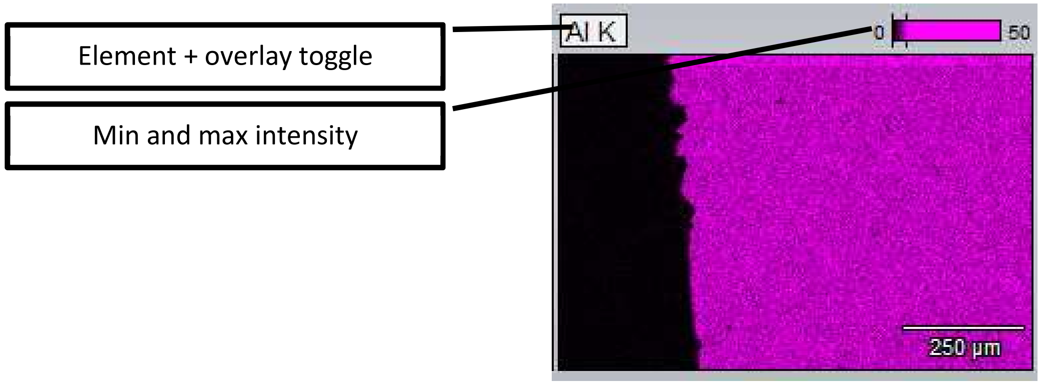
Experiment: acquire a map

1. Acquire an SEM image (see before)

2. Setup the variables. Map scans can take a very long time, and map size is a key setting affecting the time. Do not overdo the map size. Usually, 256px is a good setting[3].

- *Frame time*: Time scan 1 frame
- *Number of frames*: repetitions

3. Start the scan. If the autoID is on (standard), the maps of the detected elements will be shown in the central pane.



[3] It is advisable to have at least 100 values in the scan (this is the dynamic range). More is better.