EDS Training Notebook

Lab Manager: Dr. Perry Cheung
MSE Fee-For-Service Facility
Materials Science and Engineering
University of California, Riverside

September 21, 2023 (rev. 1.3)

Before you begin…

- All EDS users MUST complete SEM training prior to access

SEM Operation

I. Sample Preparation
II. Initiate Software
III. Sample Properties
IV. Microscope
V. Scan Configuration
VI. EDS Configuration
VII. Image Capture
VIII. Spectrum Acquisition
IX. Spectrum Quantification
X. Spectrum Chart
XI. Choice of BI
XII. Choice of Acquire
XIII. Choice of HV
XIV. Object Analysis
XV. Line Scan
XVI. Mapping
XVII. Mapping Processing
XVIII. Cleanup
XIX. QMap

I. Sample Preparation – 1/1

1. Prepare your sample normally for SEM imaging

2. Acquire any high magnification images **BEFORE** configuring for EDS

NOTE: SEM settings for acquiring high magnification images are **NOT** compatible with acquiring high quality EDS spectra!

3. Adjust the ***HV*** to appropriate value
   - Recommend starting ***HV*** value of ***15 kV*** (ADJUST AS NECESSARY)

4. Adjust the ***BI*** value to appropriate value
   - Recommend starting ***BI*** value of ***15*** (ADJUST AS NECESSARY)

5. Set the optimal ***Working Distance*** to ***14 mm*** (must be in ***Resolution Mode***)

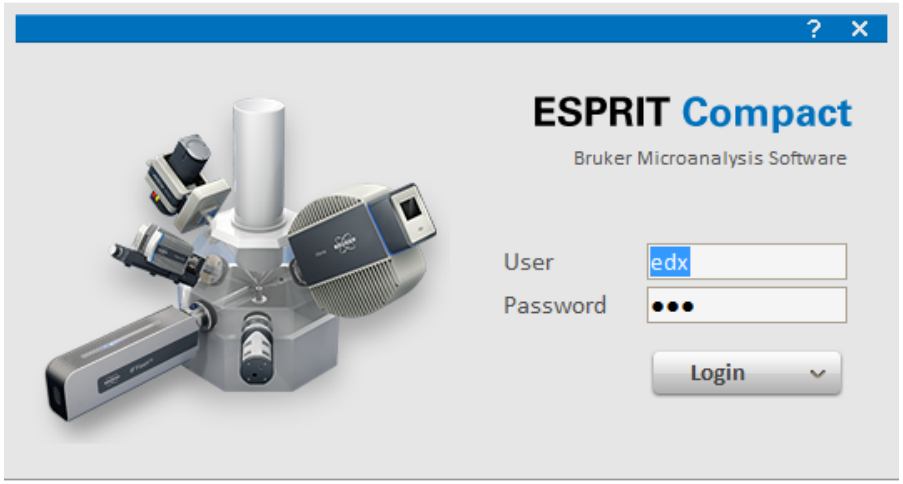
6. **TURN OFF** the ***IR Camera!***

II. Initiate Software – 1/1

1. **Record** your time-in on the **sign-in sheet** located on preparation table (if you haven't already)

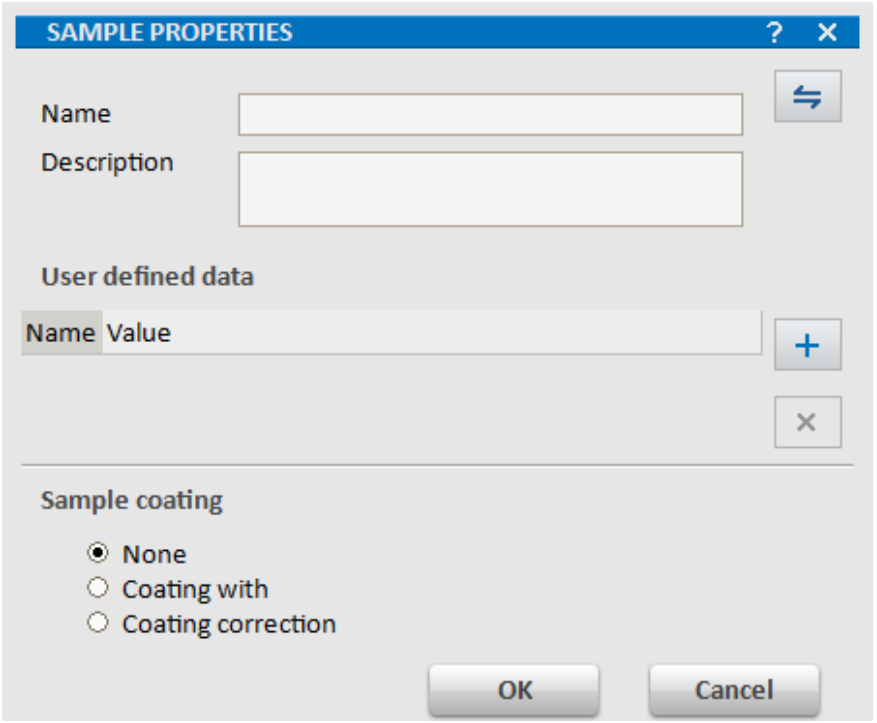
2. Double-click on **ESPRIT Compact** icon to load software on the **RIGHT** (**NOT LEFT**) monitor screen

3. Sign into your user account with your **Username** and **Password** or use the default student user account (edx/edx)



III. Sample Properties – 1/1

1. Click on ***Sample*** tab
2. Input ***Name*** (if desired)
3. Input ***Description*** (if desired)
4. Add new ***User Defined Data*** by clicking on "**+**" (if desired)
5. Select if ***Sample Coating*** was applied



1. None – if sample is not coated

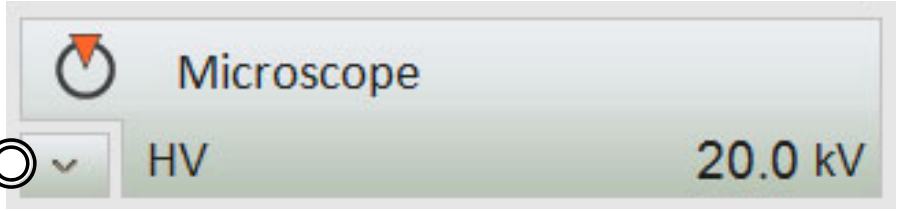
2. Coating with – if sample is coated; select element of coating material in periodic table that pops up (selected element will not be quantified)

Note: Remember to add coating element in ***Elements*** to be deconvoluted for quantification!

3. Coating correction – if sample is coated and a coating correction data file (.ccc) is available

IV. Microscope – 1/1

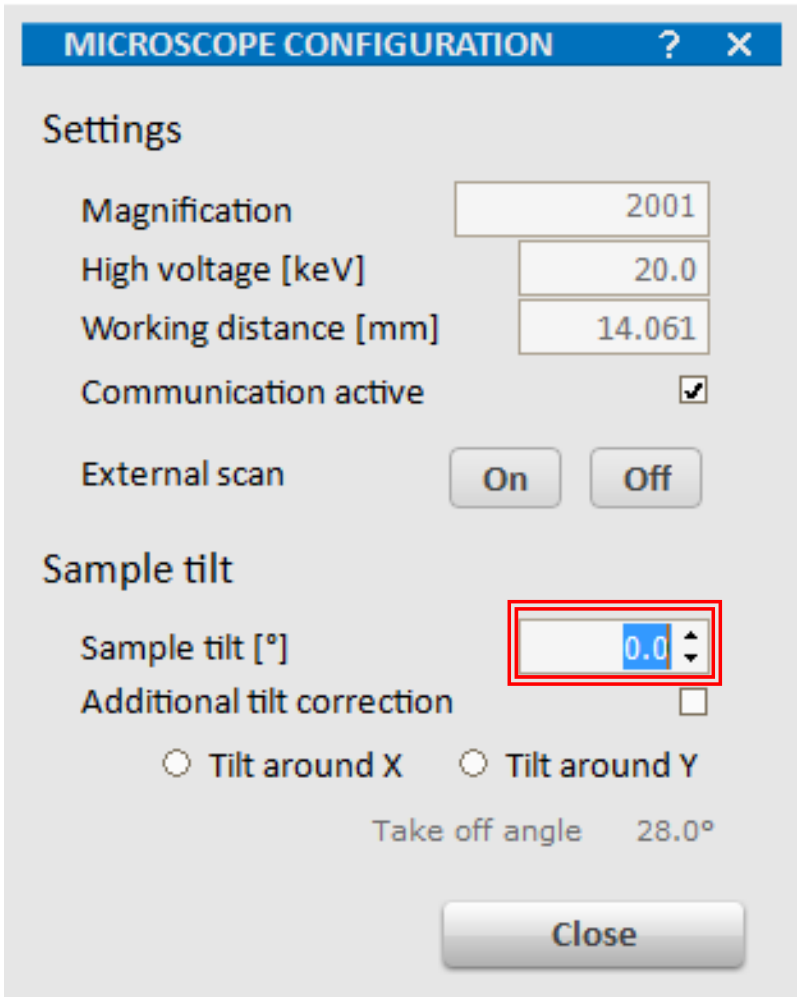
1. Click on ***Microscope*** tab



2. Confirm that Communication active is checked ✓

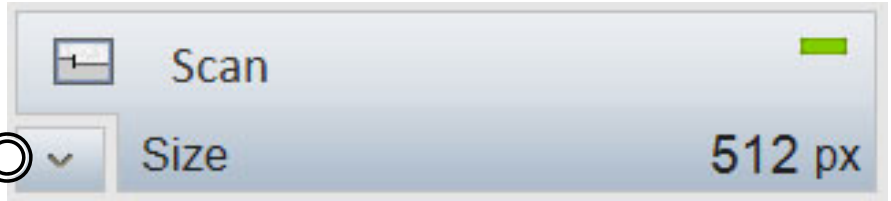
3. If sample is tilted, enter in the value of the ***Sample tilt*** in degrees

4. Additional tilt correction – check this option if sample is tilted and no image tilt correction (used for large tilt angles)

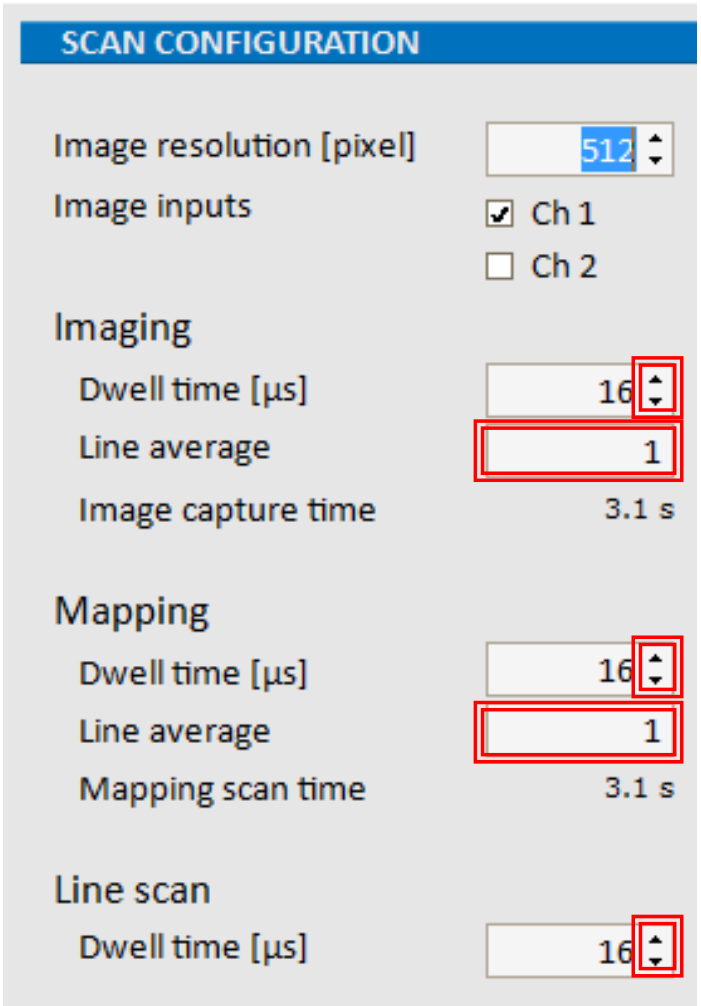


V. Scan Configuration – 1/1

1. Click on ***Scan*** tab
2. Adjust ***Image Resolution*** as necessary for increased resolution (default = ***512 pixels***)
3. Confirm that ***Ch 1*** is checked ✓

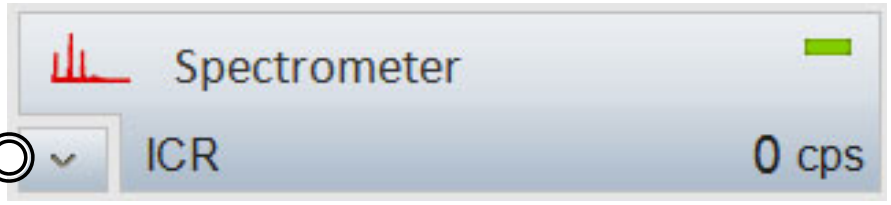


4. Choose appropriate ***Imaging Dwell time***, ***Mapping Dwell time***, and ***Line Scan Dwell time*** with the ⬍ (default = ***16 µs***)
   - ***Dwell time*** = time the electron beam stays on a pixel while capturing an ***image***, acquiring ***EDS map***, or acquiring ***EDS line scan***
5. Choose appropriate ***Line average*** factor (default = ***1***)
   - Live average = controls number of times a horizontal line is scanned before proceeding to next line

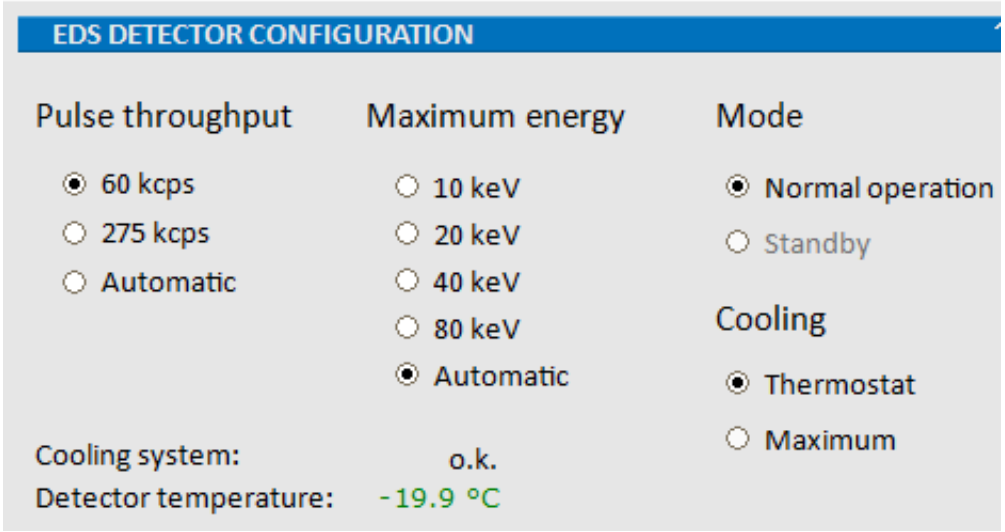


VI. EDS Configuration – 1/1

1. Click on ***Spectrometer*** tab



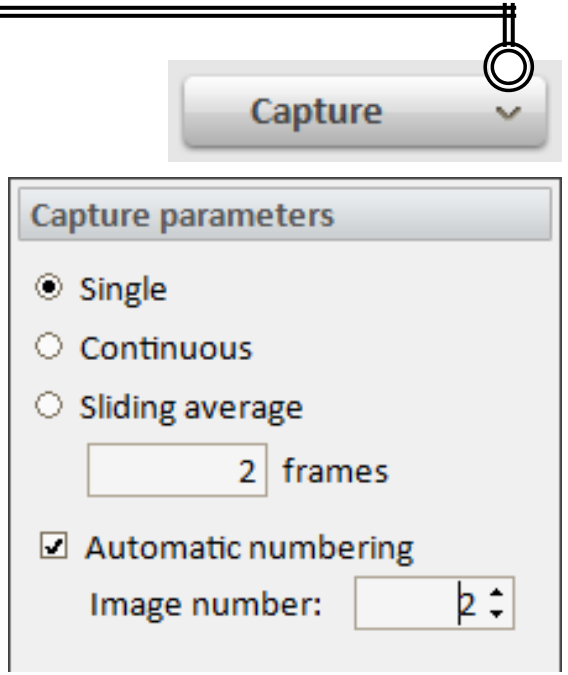
2. Identify desired ***Pulse Throughput***
   - 60 kcps – ideal for peak separation or energy resolution (default)
   - 275 kcps – ideal for max x-ray signal detection + higher BI use



3. Identify appropriate ***Maximum Energy*** for 4096 channels
   - ***Maximum Energy*** value determines the width of an energy channel (i.e. energy resolution)
   - Automatic – sets ***Maximum Energy*** according to ***HV value*** (default)
   - *10 keV = 2.4 eV/channel*
   - *20 keV = 4.8 eV/channel*
   - *40 keV = 9.8 eV/channel*
   - *80 keV = 19.5 eV/channel*

VII. Image Capture – 1/1

1. Click on the ∨ to identify the ***Capture Parameters***



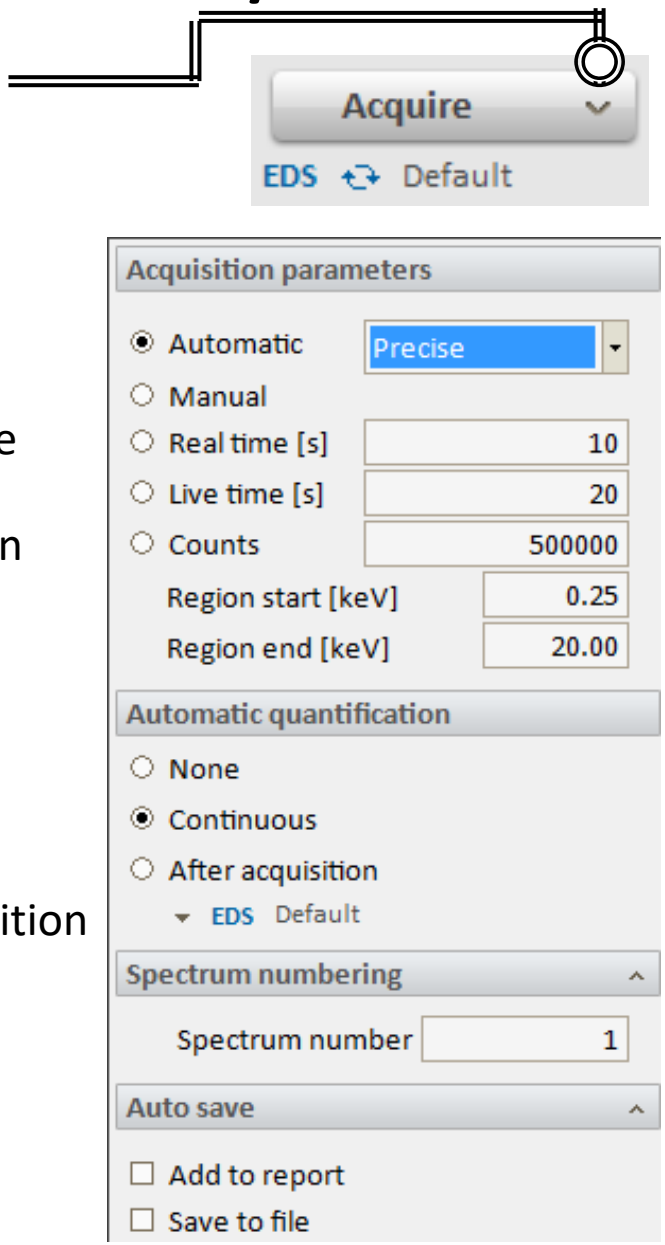
2. Identify the appropriate Image Capture Parameters (based on ***IV. Scan Configuration***)
   - ***Single*** – captures one image frame (recommend)
   - ***Continuous*** – continuously updates image frame
   - ***Sliding average*** – averages # of frames

3. Identify if ***Automatic numbering*** is desired (default)

4. Identify ***Image number***
   - Choose “1” as default for each new sample

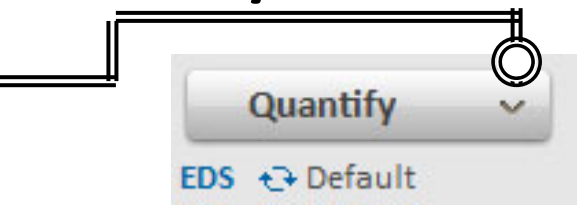
VIII. Spectrum Acquisition – 1/1

1. Click on the ∨ to identify the ***Acquire*** Parameters
2. Identify the appropriate Acquisition parameters
   - ***Automatic*** – acquisition time based on counts
     - ***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 of instrument
   - ***Manual*** – stopped manually by clicking on ***Stop*** button
   - ***Real time*** – actual time on your watch
   - ***Live time*** – estimate of time acquiring counts
   - ***Counts*** – total number of counts accumulated
3. Identify if ***Automatic quantification*** is desired
   - ***None*** – no automatic quantification
   - ***Continuous*** – will continuously quantify during acquisition
   - ***After acquisition*** – only quantify after acquisition
4. Identify ***Spectrum number***
   - Choose "1" as default for each new sample

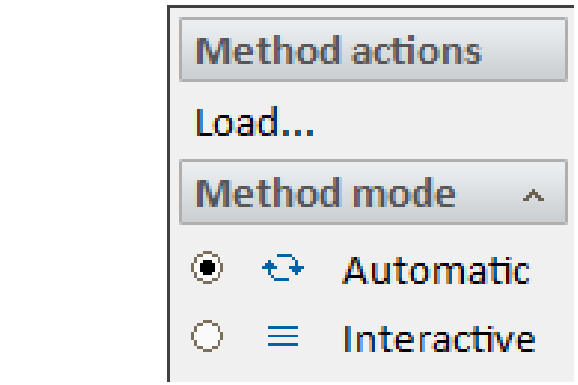


IX. Spectrum Quantification – 1/1

1. Click on the ∨ to identify the ***Quantify*** Parameters



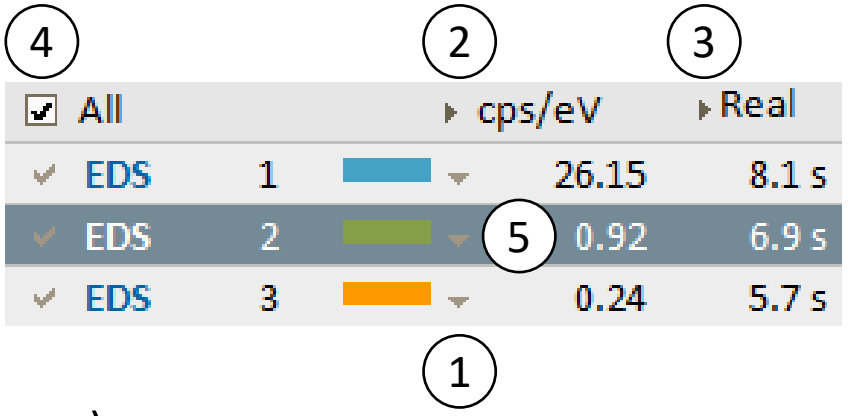
2. Click on ***Load…*** to load pre-configured quantification method (Advanced Users only)



3. Choose the appropriate ***Method Mode***
   - ***Automatic*** – quantification results automatically show up in spectrum list (default)
   - ***Interactive*** – quantification dialog pops up (recommended for complicated spectra)

Note: Remember to add coating element in ***Elements*** to be deconvoluted for quantification!

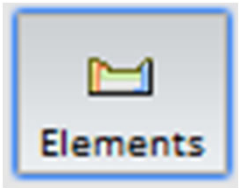
X. Spectrum Chart – 1/2



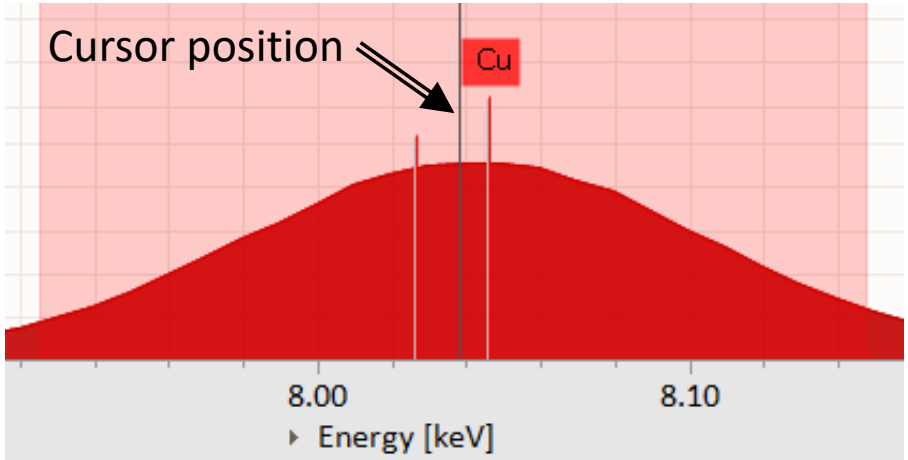
1. ***Spectrum Color***: Click on the ∨ to select the color
2. ***Options***: Click on **>** to select pulses, cps, net counts, or energy resolution
3. ***Results***: Click on **>** to select spectrum information, identification or display of quantification results: mass-%, mass-% (norm.), atom-%, stoich.-%, or stoich.-% (norm)
4. ***Spectra***: Multiple spectra can be selected (checkbox) or select ***All***
5. ***Element ID***: Multiple IDs can be shown by selecting spectrum line (hold CTRL + left-click to select multiple IDs)
6. To scale or zoom the spectrum diagram, use either:
   - Scroll mouse wheel to change x-scale
   - Click and hold mouse wheel up/down to change y-scale
   - Click and hold mouse wheel left/right to move spectrum area
   - Right-click on x- or y-axis to scale values manually
   - Right-click on spectrum and select Auto Scale for automatic scaling

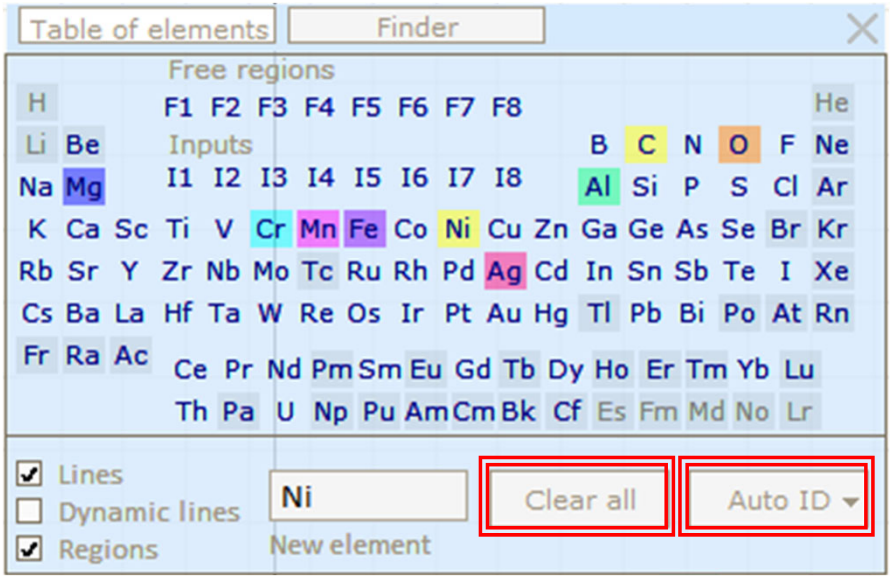
X. Spectrum Chart – 2/2

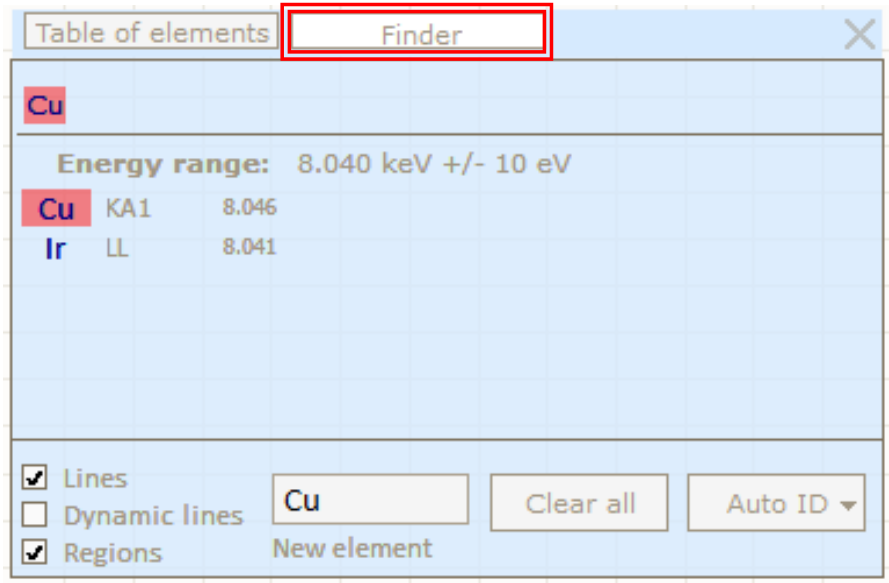
7. Click on ***Elements*** icon to select or de-select elements in spectrum



8. Click ***Clear all*** button to remove all elements from spectrum
9. Click ***Auto ID*** to automatically identify elements from spectrum
10. Click ***Finder*** to see a list of possible elements based on cursor position
11. Add or delete elements by clicking on the element name

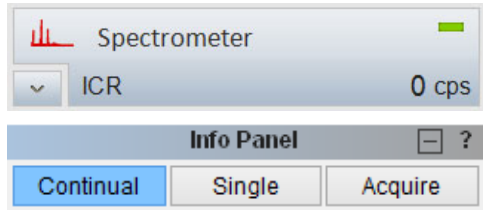


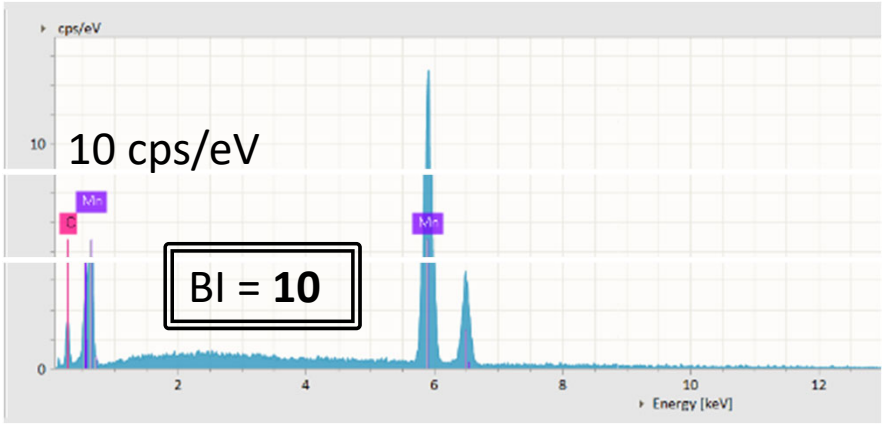




XI. Choice of BI – 1/1

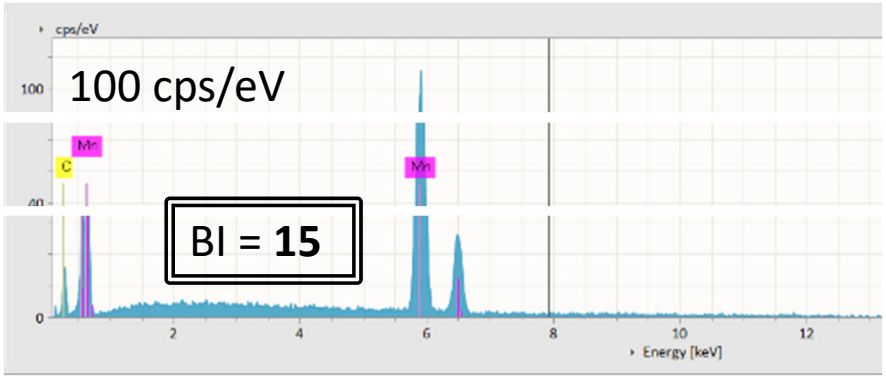
If total counts is 0 cps, check if ***Continuous*** is enabled on SEM!





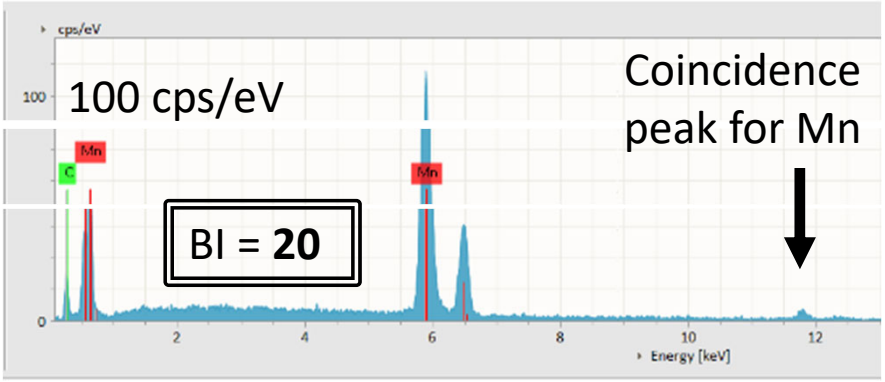
- Low total counts: ≤ 10 kcps (bad)
- Low Dead Time: 0 – 20% (good)
- Coincidence peaks: None (good)

cps/eV	Real	Live	Dead	Pulses	Input	Output
0.07	14 s	14 s	2 %	64177	3543 cps	3477 cps



- High total counts: ≥ 10 kcps (good)
- Moderate Dead Time: 20 – 30% (good)
- Coincidence peaks: None (good)

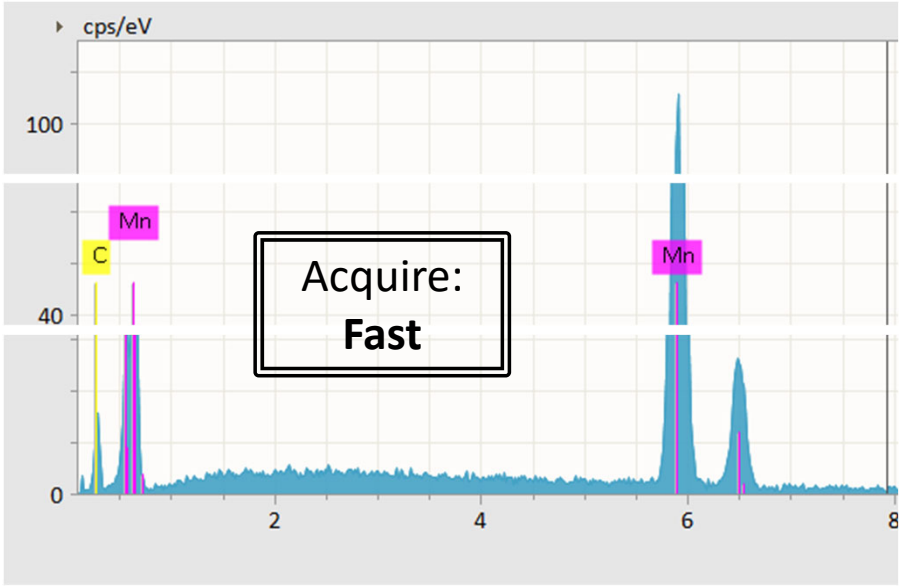
cps/eV	Real	Live	Dead	Pulses	Input	Output
0.51	1.6 s	1.2 s	22 %	59400	41.1 kcps	32.2 kcps



- High total counts: ≥ 10 kcps (good)
- High Dead Time: ≥ 30% (bad)
- Coincidence peaks: Present (bad)

cps/eV	Real	Live	Dead	Pulses	Input	Output
0.99	1.4 s	0.1 s	91 %	56518	388.1 kcps	35.4 kcps

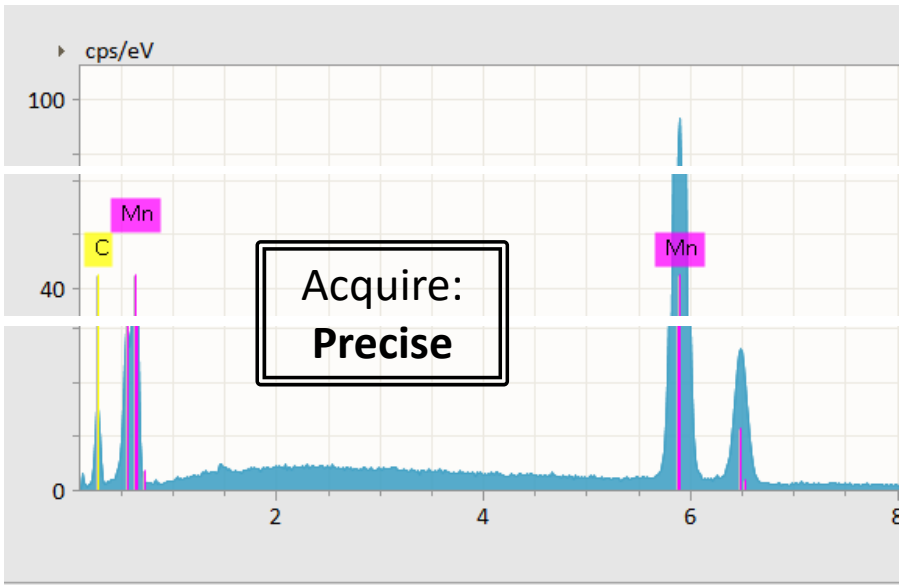
XII. Choice of Acquire – 1/1



Automatic – ***Fast: 50,000 counts***

- Fast spectra collection
  - "Noisy" curves and peaks

cps/eV	Real	Live	Dead	Pulses	Input	Output
0.51	1.6 s	1.2 s	22 %	59400	41.1 kcps	32.2 kcps

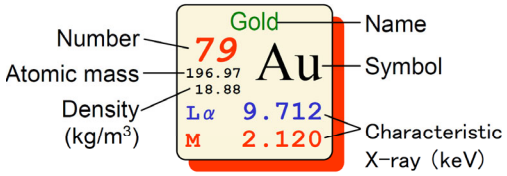


Automatic – ***Precise: 100,000 counts***

- Precise spectra collection
  - "Smoother" curves and peaks

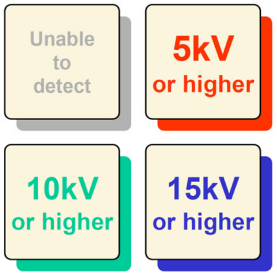
cps/eV	Real	Live	Dead	Pulses	Input	Output
0.45	8.0 s	6.3 s	21 %	298549	39.9 kcps	31.4 kcps

XIII. Choice of HV – 1/3



Note: Density
* 'C' as (graphite), 'P' as (white), 'S' as (alpha), 'Sn' as (white)

Minimum accelerating voltage



The colors mean to detect the characteristic X-ray of the lowest energy for each element.

Minimum accelerating voltage or ***HV*** should be chosen based on:

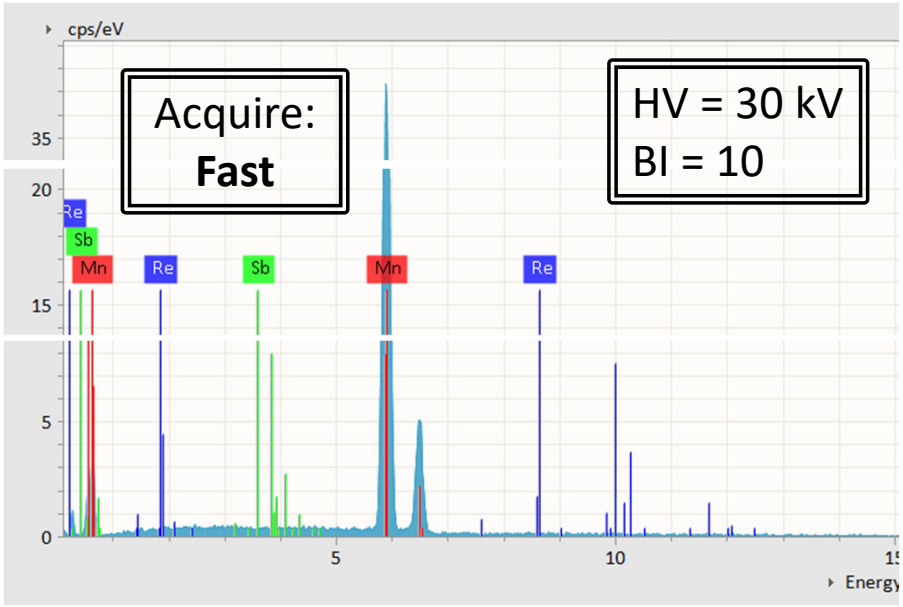
- **Element(s) used for analysis**
  - **Characteristic X-ray(s) of element(s)**

Example: Mn

Kα = 5.894 keV requires 10 kV or higher

Lα = 0.637 keV requires 5 kV or higher

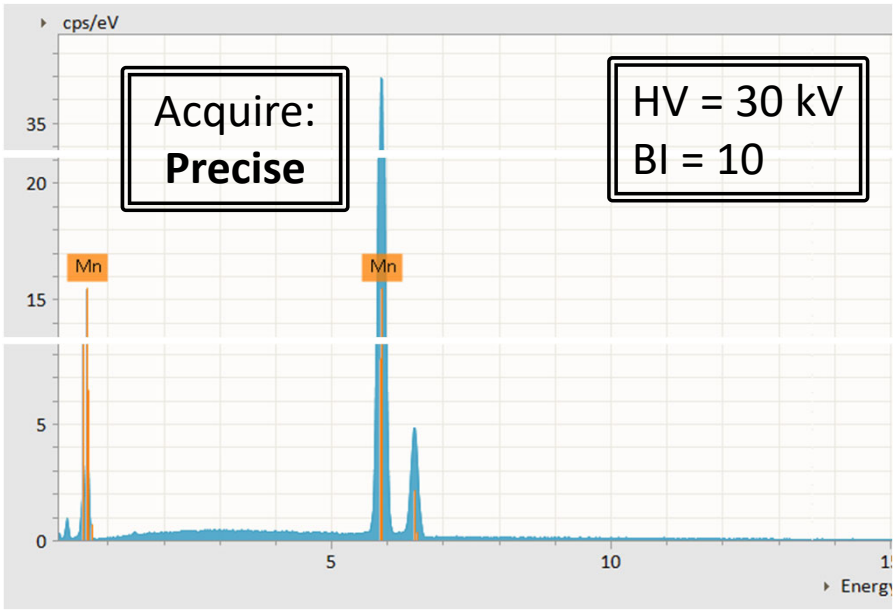
XIII. Choice of HV – 2/3



Increasing HV does not always automatically get you better spectra! Check other settings!

- Automatic – ***Fast: 50,000 counts***
- Fast spectra collection
  - "Noisy" curves and peaks
  - May lead to false ID of peaks

cps/eV	Real	Live	Dead	Pulses	Input	Output
0.00	5.4 s	5.1 s	6 %	59563	9909 cps	9325 cps

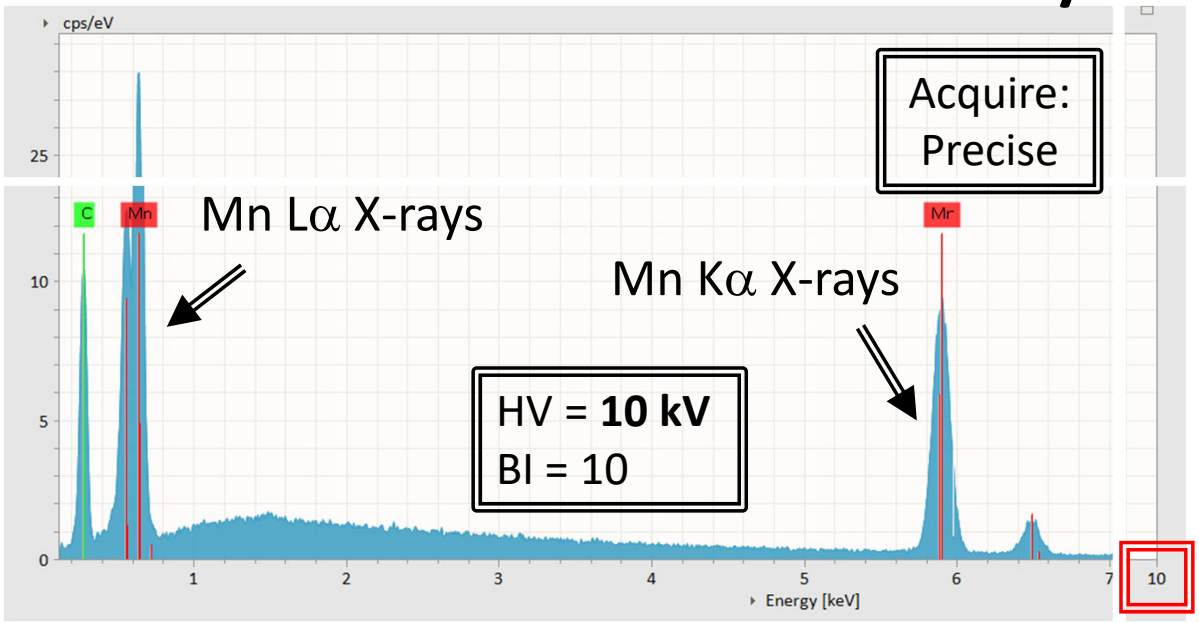


User needs to check if enough counts are present to provide statistical representation of "real" peaks

- Automatic – ***Precise: 100,000 counts***
- Precise spectra collection
  - "Smoother" curves and peaks

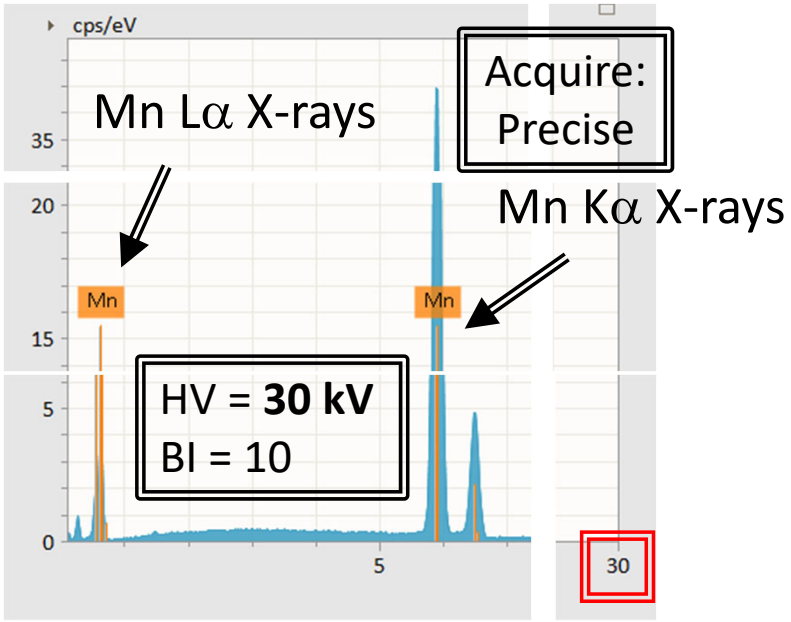
cps/eV	Real	Live	Dead	Pulses	Input	Output
0.09	26 s	25 s	5 %	294614	10.0 kcps	9508 cps

XIII. Choice of HV – 3/3



HV = 10 kV

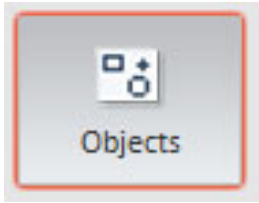
- Energy Range up to 10 keV
- Not all Mn Kα = 5.894 keV X-rays are generated by the 10 kV HV
- Mn Lα = 0.637 keV X-rays are easily generated by the 10 kV HV (higher counts)



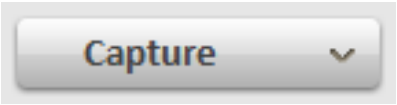
HV = 30 kV

- Energy Range up to 30 keV
  - Unnecessary reduction in resolution
- Mn Kα = 5.894 keV X-rays are now easily produced by the 30 kV HV
- Mn Lα = 0.637 keV X-rays are now absorbed by sample (drop in counts)

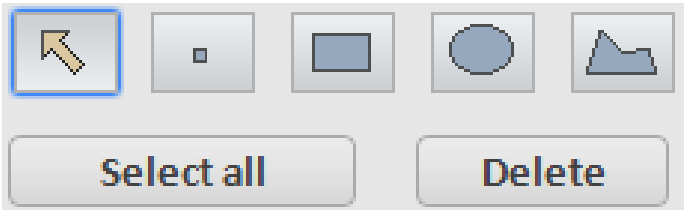
XIV. Object Analysis – 1/2



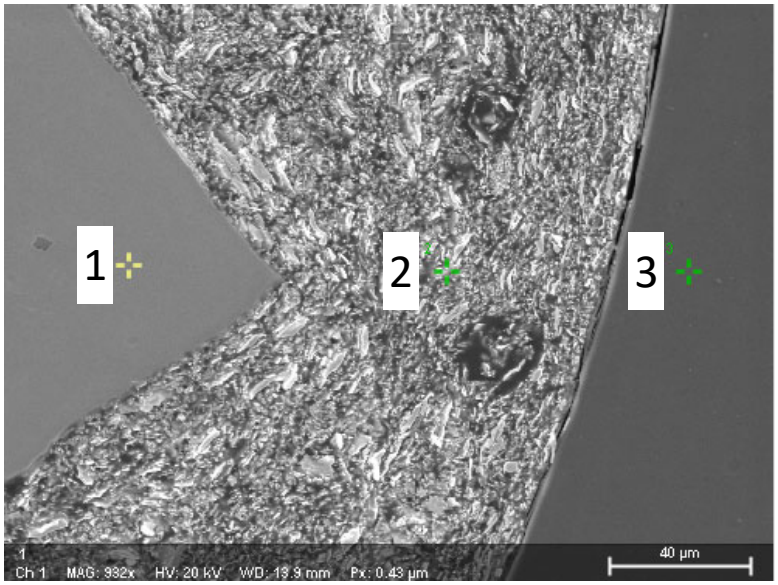
1. Click on ***Objects*** icon for Object Analysis
2. Click ***Capture*** to capture desired image



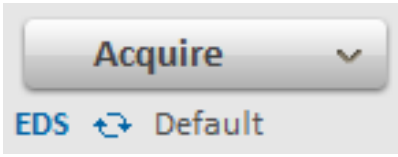
3. Select the desired object type: ***point***, ***rectangle***, ***ellipse***, and ***polygon***



4. Draw an object in the captured image
5. Use ***Select all*** button to highlight all objects



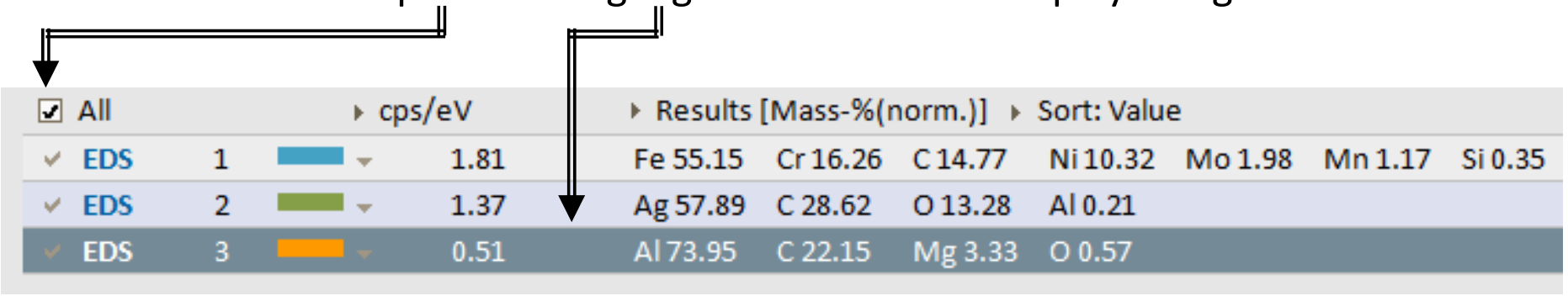
6. Click ***Acquire*** to acquire spectrum for selected objects

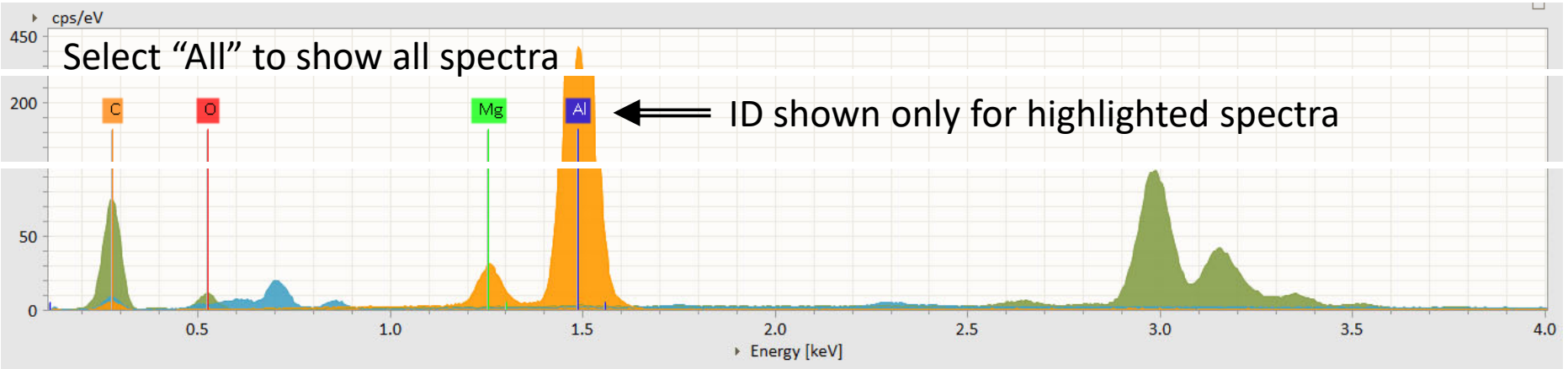


7. Click ***Quantify*** to quantify spectrum if not set to automatic quantification

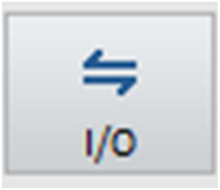
XIV. Object Analysis – 2/2

8. Choose desired spectra or highlight element ID to display using chart below



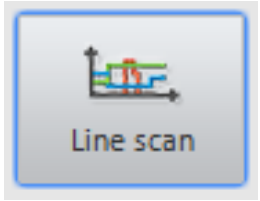


9. Click ***Input/Output*** icon to save or load data
   - Use *.rto file format as it contains all available SEM images including objects and corresponding spectra

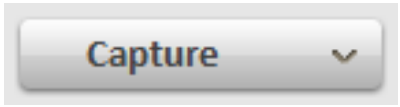


XV. Line Scan – 1/2

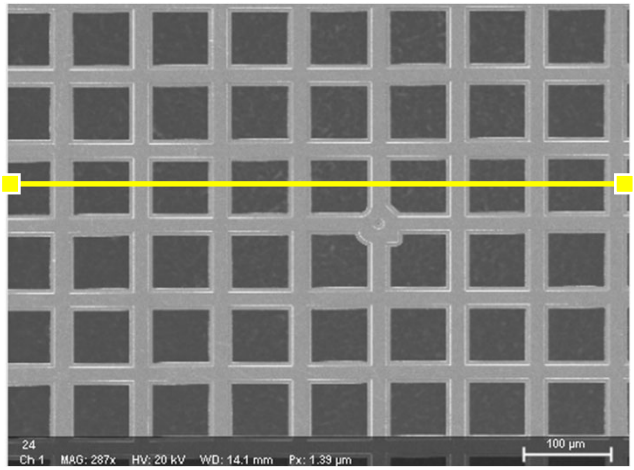
1. Click on ***Line Scan*** icon for Line Scan



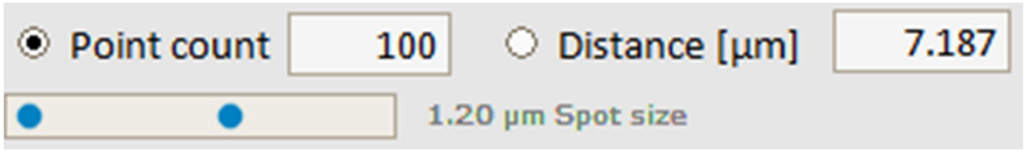
2. Click ***Capture*** to capture desired image



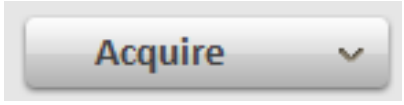
3. Highlight the line and drag and adjust the endpoints to desired position

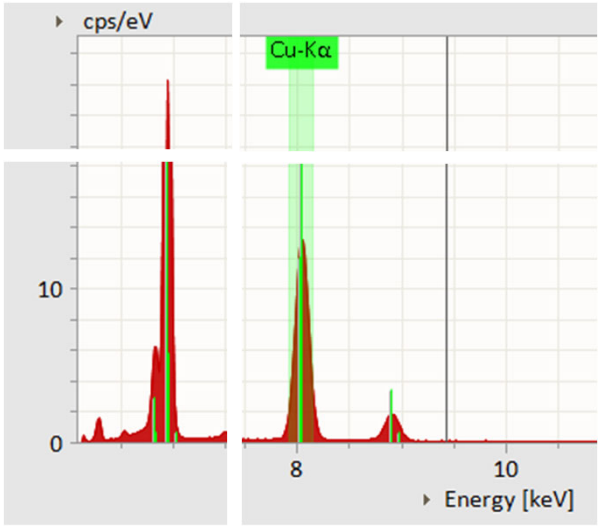


4. Set the ***Point count*** or ***Distance*** between points of the line scan



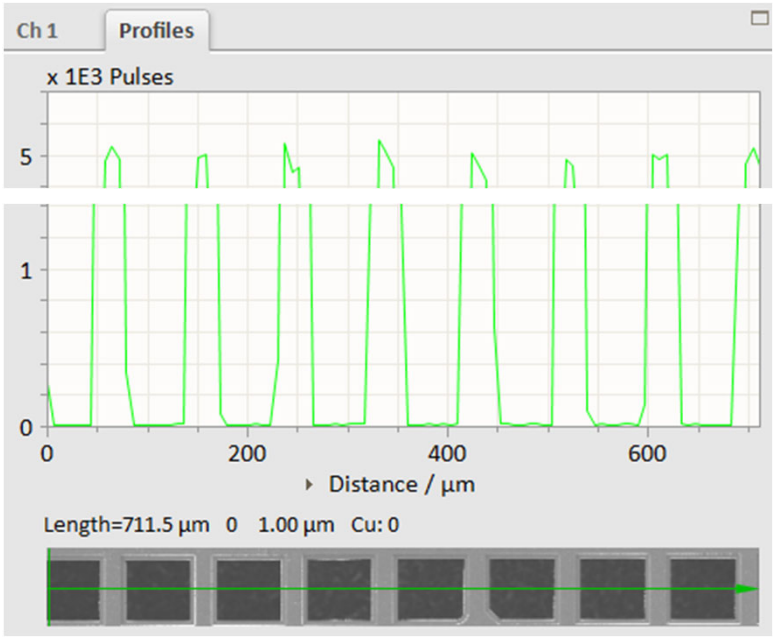
5. Click ***Acquire*** to acquire a spectrum for drawn line



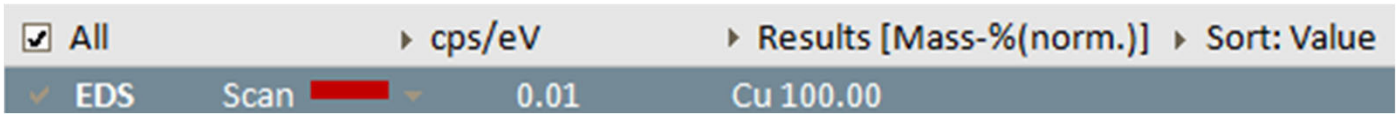


XV. Line Scan – 2/2

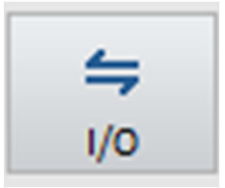
6. Click to view ***Profiles*** or ***Spectrum*** tab on upper right



7. Click ***Quantify*** to quantify spectrum

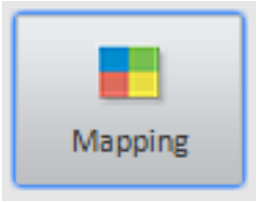


8. Click ***Input/Output*** icon to save or load data

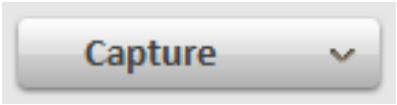


- Use *.rtl file format to save line scan data including SEM and scan images and point spectra
- NOTE: Esprit 2.1 will NOT allow further quantification on *.rtl files, recommend Mapping for future quantification analysis

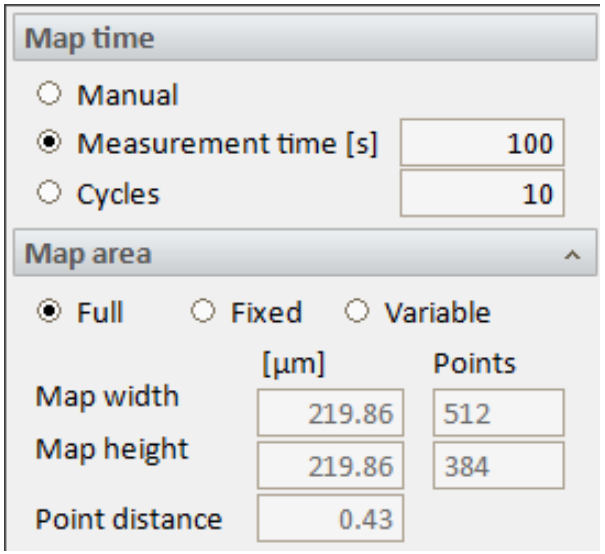
XVI. Mapping – 1/2



1. Click on ***Mapping*** icon for Mapping Analysis
2. Click ***Capture*** to capture desired image

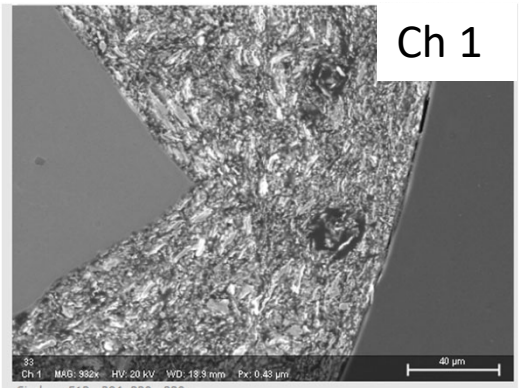


3. Click on the ∨ to identify the ***Acquire*** Parameters for:
   - Map time
     - ***Manual*** – stopped manually by clicking on ***Stop*** button
     - ***Measurement time*** – actual time on your watch
     - ***Cycles*** – total number of cycles accumulated
   - Map area
     - ***Full*** – use full captured image area for mapping
     - ***Fixed*** – use a fixed area for mapping
     - ***Variable*** – use a variable area for mapping each time

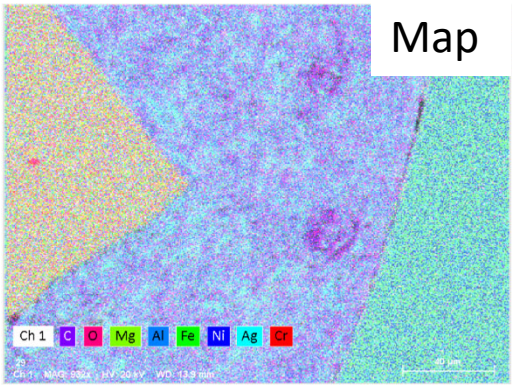


4. Click ***Acquire*** to acquire a map

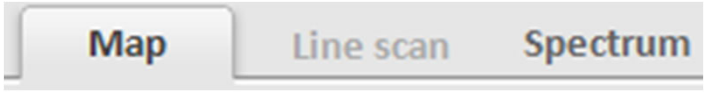
5. Click to view ***Ch1*** or ***Map*** using tab on upper left



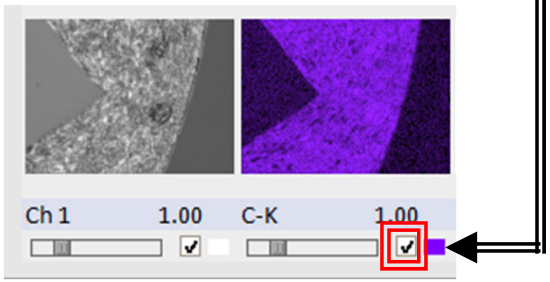
XVI. Mapping – 2/2

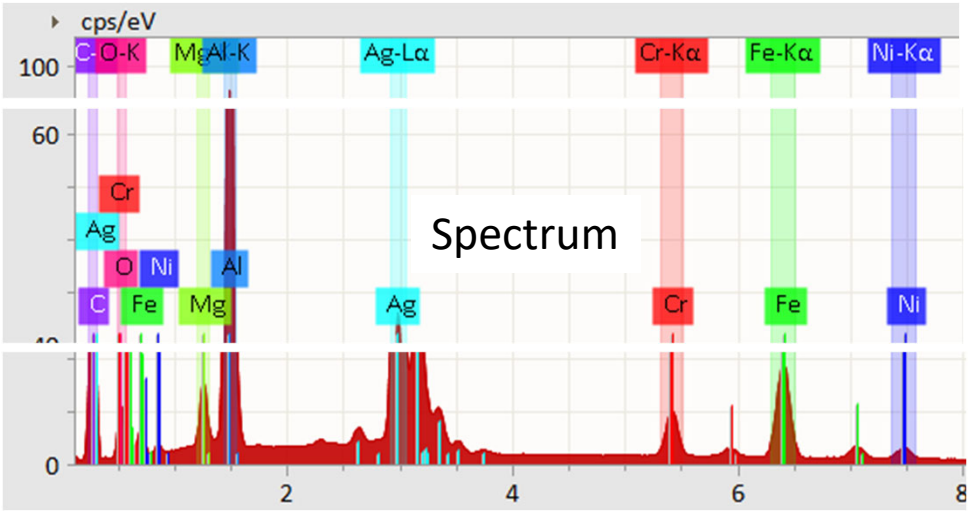


6. Click to view ***Map*** or ***Spectrum*** using tab on upper right



7. Individual element mapping overlays can be turned on/off

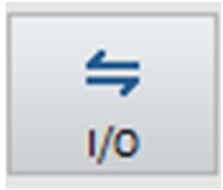




8. Click on ***Quantify*** to quantify the ENTIRE mapped area

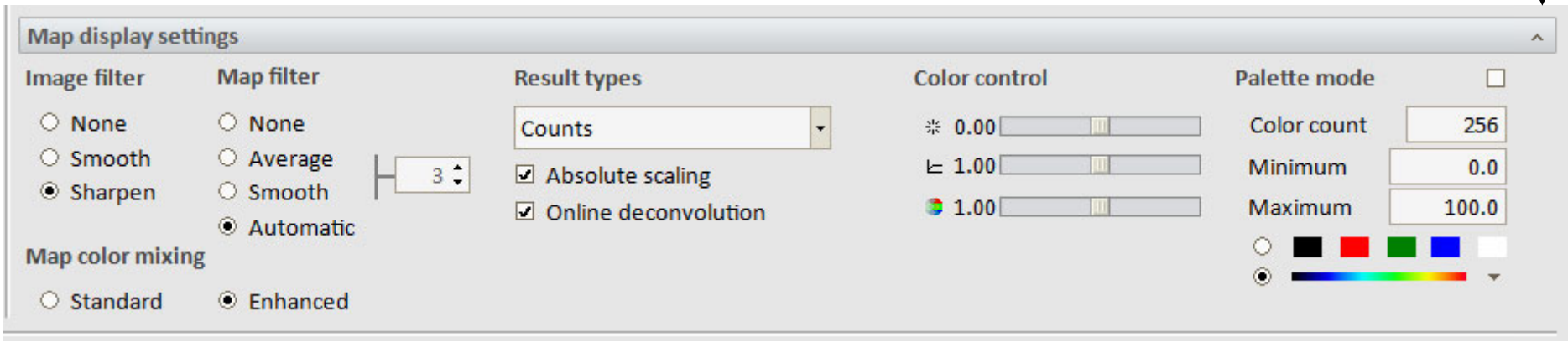
- Only Esprit 2.1 will allow you to quantify each pixel AFTER acquiring map

9. Click ***Input/Output*** icon to save or load data
   - Use *.bcf file format to save HyperMaps (datacube)
   - NOTE: Esprit 2.1 will ALLOW further quantification on *.bcf files

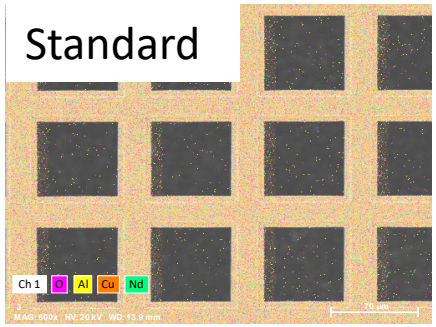


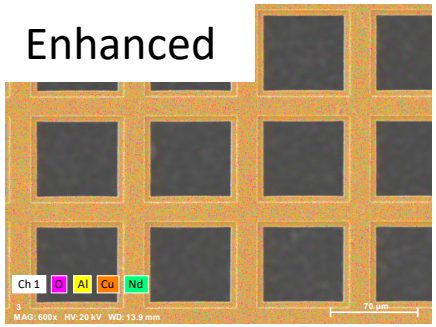
XVII. Mapping Processing – 1/3

1. Click on the ∨ to open up ***Map display settings***

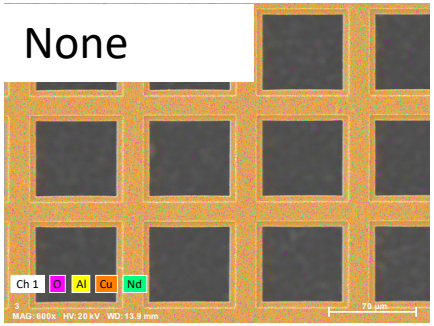


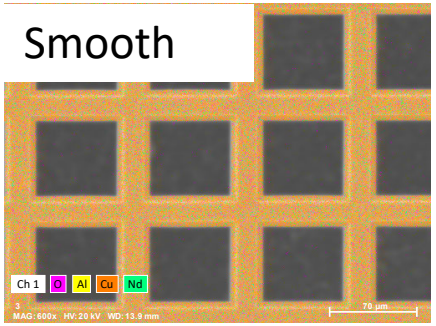
2. Select desired ***Map color mixing***: recommend ***Enhanced***

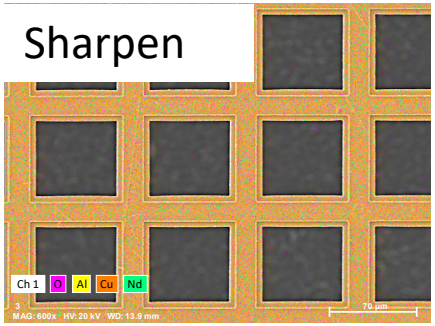




3. Select desired ***Image filter***: recommend ***Sharpen***



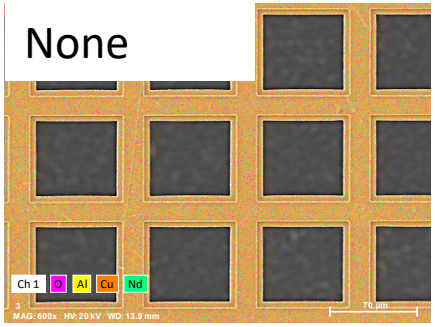




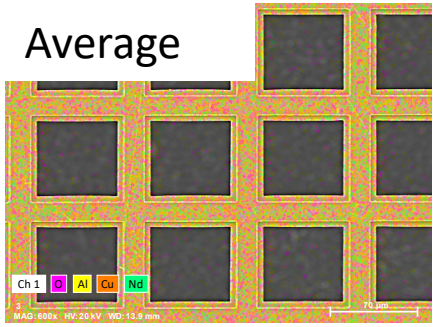
XVII. Mapping Processing – 2/3

4. Select desired ***Map filter*** – recommend ***None*** if sufficient counts

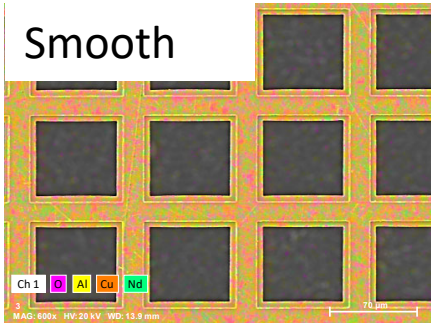
None



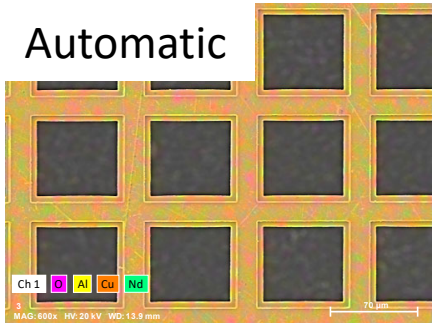
Average



Smooth

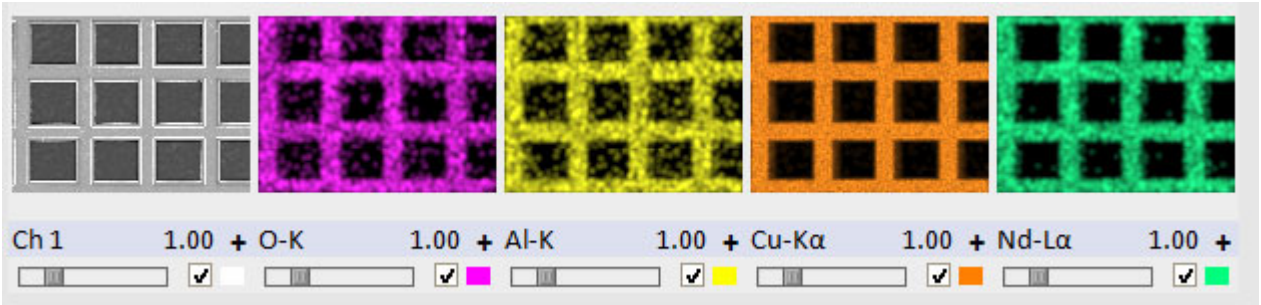


Automatic

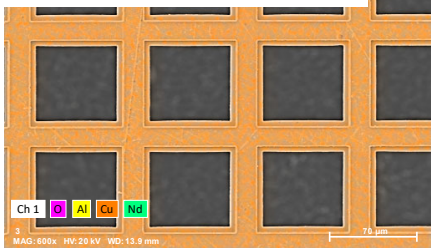


5. Consider adding ***Result type***:

- ***Absolute scaling*** – scales selected elements to a maximum of 100, with color in composite element image being proportional to value of each element

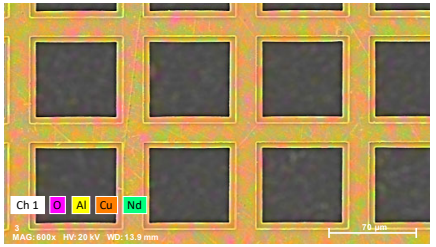


Absolute scaling



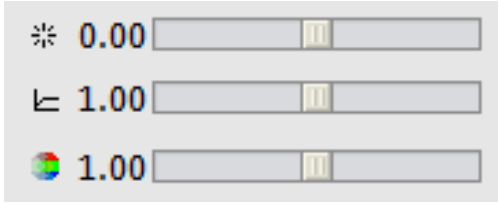
- ***Online deconvolution*** – performs automatic background subtraction and element peak deconvolution

Online deconvolution

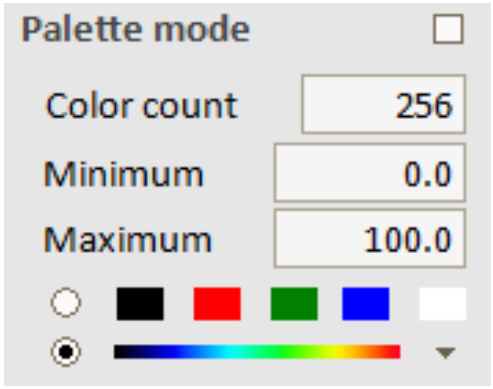


XVII. Mapping Processing – 3/3

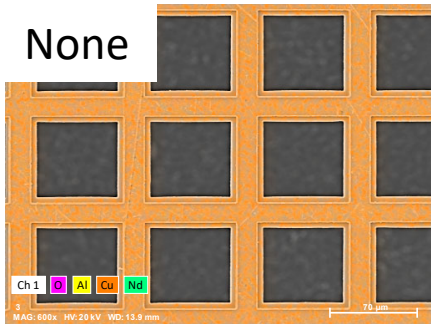
6. Consider changing ***Color control***:
   - Brightness
   - Gamma
   - Color saturation



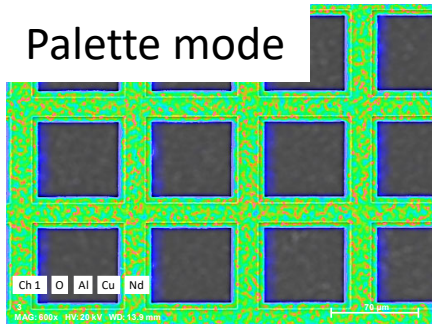
7. Consider changing ***Palette mode***:

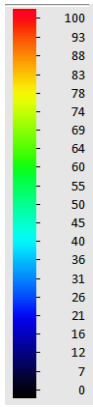


None



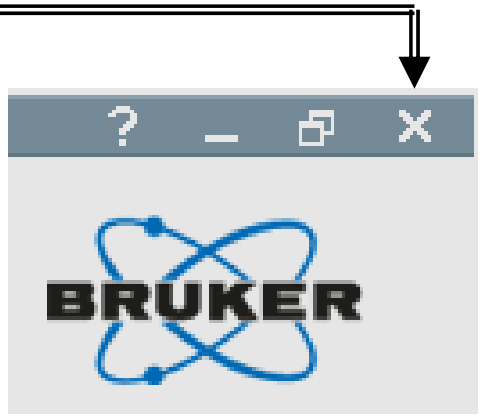
Palette mode





XVIII. Cleanup– 1/1

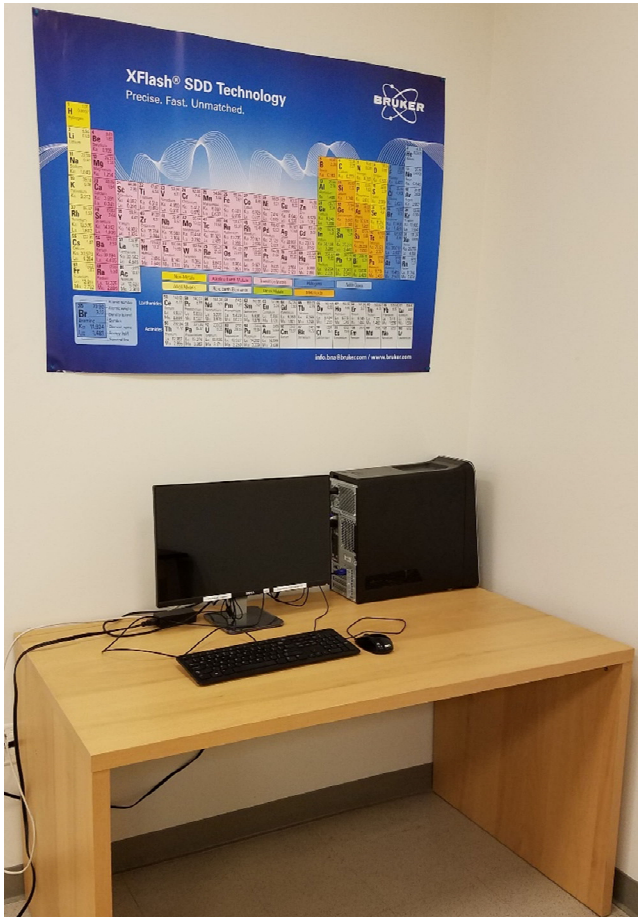
1. Turn off the Esprit Compact software by clicking on the ***Close* X** button



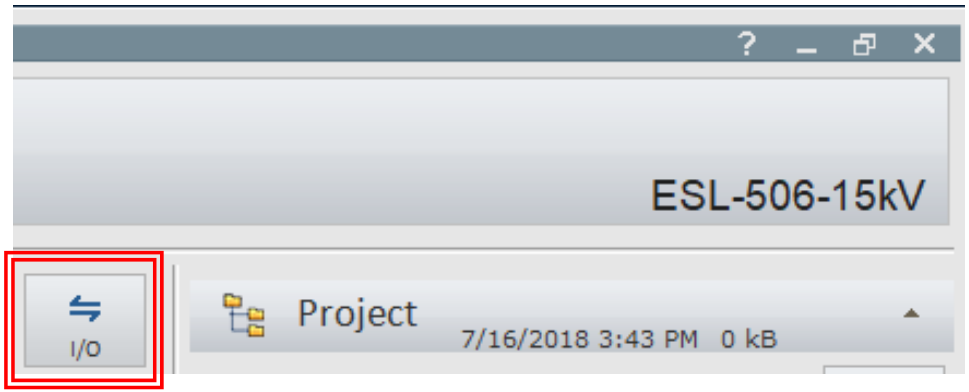
2. Continue to follow the ***XVII. Sample Unloading*** and ***XVIII. Cleanup process*** procedures outlined in the SEM training manual

XIX. QMap – 1/2

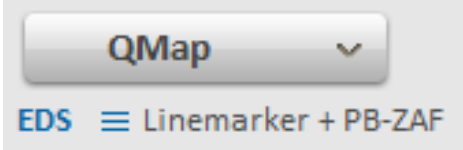
The following description applies to using saved mappings (*.bcf) files to be analyzed in ***Bruker Esprit 2.1*** software accessible on separate EDS computer



1. Double-click on ***ESPRIT 2.1*** icon to load software in separate EDS computer
2. Sign into your user account with your **Username** and **Password** or use the default student user account (student/student)
3. Load desired *.bcf file using the input/output button



XIX. QMap – 2/2



4. Click on ∨ to identify ***QMap*** parameters
5. Load desired Method file if available
6. Choose the appropriate ***Method Mode***
   - ***Automatic*** – quantification results automatically show up in spectrum list (default)
   - ***Interactive*** – quantification dialog pops up
7. Choose desired ***QMap*** options
   - Tile size [pixel] – see estimated time
     - 1x1 – slowest
     - 2x2
     - 4x4
     - 8x8 – fastest
8. Click ***QMap*** to start quantitative mapping
9. Click on ***Result types*** to select desired quantification:
   - Counts
   - Net sum
   - Mass %
   - Mass % (norm.)
   - Atomic % (norm.)