

Mick:	255-0650
<b>Cornell Police:</b>	<b>255-5111</b>
Emergency:	<b>911</b>
Phil Carubia:	255-6757

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CCMI

Step 1 - setup:

### Select Objects and click on MultiPoint Spectra Feature Objects Jobs System Report Imaging Object Mode Devices Input O Spectrometer 27.2 kcps Range 10 keV / 60 kcps $\bigtriangledown$ HV 10.0 kV Project Information 4/1/2016 6:35:12 PM 0 Objects Point MultiPoint Line scan Mapping HyperMap

Set the EHT to ~4X the highest energy edge you will be measuring.

Set the aperture to 60um to 230um aperture for high count rates.

Set the working distance to 7mm +/- 1mm.

Click on the triangle next to New



image scan	Х
<ul> <li>Single</li> </ul>	
Continuously	
Average	
Image name: Your image name here	

Select the desired shape

Multiple shapes can be selected for sequential acquisition.

Automatic selection of shape locations can also be chosen.



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# 8.00 Step 1 – setup (continued):

Note the count rate (green circle).

Click on the triangle next to Spectrometer.

Spectra Imaging Feature Objects Jobs System Report Object Mode Devices  $\bigtriangledown$ O Spectrometer (▽ Input 27.2 kcps ange 10 keV / 60 kcps HV 10.0 kV Project Information 4/1/2016 6:35:12 PM 0 Objects Point MultiPoint Line scan Mapping HyperMap

Choose the Max. pulse throughput to be just above the kcps value.

Choose the Maximum energy to include all the edges you are analyzing.

Max. pulse throughput 20 kcps 30 kcps	<ul> <li>Maximum energy —</li> <li>10 keV</li> </ul>	Mode Normal opera	tion	
0 60 kcps	O 20 keV	Standby	Do not o	change
<ul> <li>130 kcps</li> <li>275 kcps</li> </ul>	0 40 keV	Cooling	Mode o	
0 400 kcps	0 80 keV	<ul> <li>Thermostat</li> </ul>		
<ul> <li>600 kcps</li> <li>Automatic</li> </ul>	O Automatic	O Maximum		
Cooling system:	On			
Detector temperature:	-30.1 °C			



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# Step 1 setup (continued):

Click on the triangle next to Acquire.

Spectra Imaging

OK

Object Mode	
Devices 🗢	
	Cps Range 10 keV / 60 kcps Dead time 16 % Real time
Imaging system	HV 10.0 kV Magnification 39088 WD
Project	
Information 4/1/2016 6:35:12 PM 0 Objects	
Point MultiPoint Line scan Mapping HyperM	ap
Image scan Preview New	Spectra Acquire  Quantify  Interactive PB-ZAF line
Measuring time	Set the Real time (or Live time)
	desired. Note Real Time = Live
Automatic     Precise	
O Manual	Time + Dead Time
Real time [s]	
Live time [s] 400	
O Counts 500000	
Region start [keV] 0.00	·
Region end [keV] 10.00	
Automatic analysis	
None	If you enter a Spectrum name a
O Continuous	check Automatic numbering the
<ul> <li>After acquisition</li> </ul>	J J
	that name and incremental
Coating correction	number will be assigned to your
None	spectra.
O Coating correction Carbon only	

EBSD Objects Jobs System Report

Click on OK



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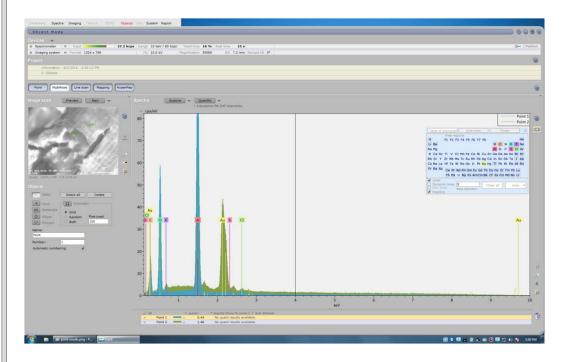
# 8.00 Step 2 - acquisition:

Click on Acquire to collect spectra

The spectra will appear in real time until the acquisition has finished.

Clicking on the 🗀 icon on the right side of the screen will enable the periodic table to appear

A separate spectra will show up for each Point/Rectangle/Ellipse/Polygon



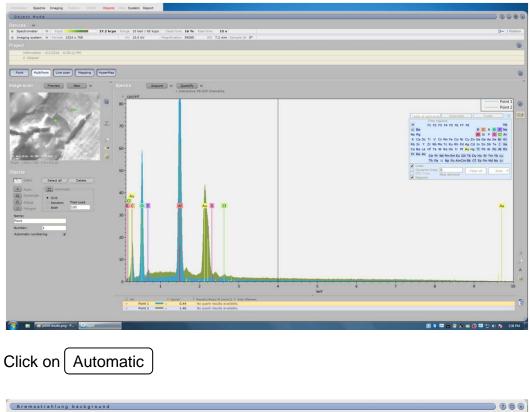
Click on the Auto button Auto 
to automatically identify the peaks or click on the element in the Periodic Table to manually identify the peaks,

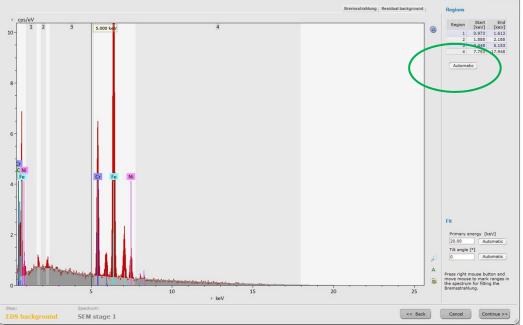


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# 8.00 Step 3 - quantification:

To quantify the elements choose a spectra and click on Quantify



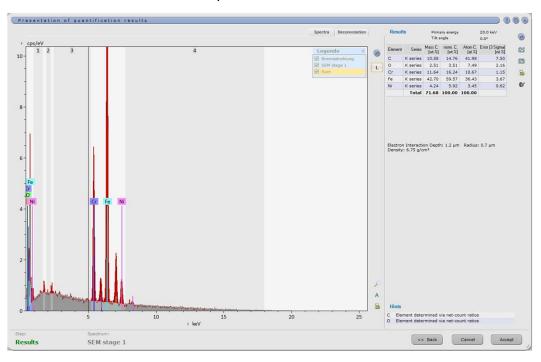




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# 8.00 Step 3 – quantification (continued):

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Note a result table has showed up in the corner:

Click on Accept

Results		Prim Tilt a	ary energy ingle	20.0 ke∨ 0.0°		
Element	Series	Mass C. [wt.%]	norm. C. [wt.%]	Atom C. [at.%]	Error (3 Sigma) [wt.%]	
С	K series	10.58	14.76	41.98	7.50	
0	K series	2.51	3.51	7.49	2.16	
Cr	K series	11.64	16.24	10.67	1.15	
Fe	K series	42.70	59.57	36.43	3.67	
Ni	K series	4.24	5.92	3.45	0.62	
	Total	71.68	100.00	100.00		

Electron Interaction Depth: 1.2  $\mu m$   $\,$  Radius: 0.7  $\mu m$  Density: 6.75 g/cm³  $\,$ 

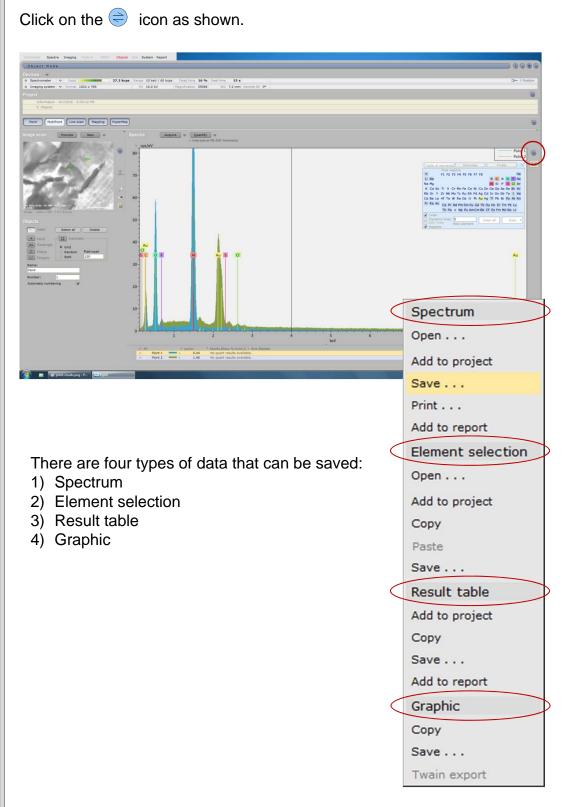
The software displays the results in several ways.

Note also that the electron interaction depth and radius are provided.



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# <u>Step 4 – saving data:</u>





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### 8.00

### Step 4 – saving the data:

Saving the spectra:

Under Spectrum click on Save ... Add to project Save spectrum edx\Data\ Volume 🎟 🔹 🐰 🖿 💼 <u>₩</u> Preview Mod user Add to report 2 Element selection edx Open . . . Add to project Public Copy 💒 C: 2 D: Save . . . - Y: Result table 🖵 Z: File name Information Add to project File type Bruker Nano spectra (\*.spx) Save . . . Text (\*.txt) Add to report Excel 97/2000/XP (\*.xls) EMSA spectra (\*.msa) Graphic 1. Make sure the directory is yours 2. Set the file type to Bruker Nano spectra (\*.spx) Twain export 3. Give the spectra a name 4. Click on Save Υ<u></u> File name Information File type Excel 97/2000/XP (\*.xls) Bruker Nano spectra (\*.spx) Text (\*.txt) EMSA spectra (\*.msa) 5. Under Spectrum click on Save ... again 6. Set the file type to Excel 97/2000/XP (\*.xls) 7. The same file name should populate the File name box



Save

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8. Click on

IF IN DOUBT, ASK

Save

Cancel

Spectrum Open . . .

Save . . .

Print . . .

Paste

Copy

Copy

Save . . .

# Step 4 – saving the data:

Mode ciic 111P

Saving the quantified results:	
	Spectrum
Under Result table click on Save	Open
6	Add to project
Save results	Save
Volumes 💼 edx\Data\ 😽 🖽 🗸 🖻 💼 Preview	Print
Sector User	Add to report
edx	Element selection
	Open
	Add to project
Public	Сору
🚣 C:	Paste
🔐 D:	Save
	Result table
File name Information	Add to project
File type	Сору
Text table (*.txt)	Save
Text table (*.txt) Bitmap images (*.bmp)	Add to report
JPEG images (*.pg) PNG images (*.png)	Graphic
TIF images (*.tif)	Сору
	Save
1. Make sure the directory is yours	Twain export

- 2. Set to Text table (\*.txt)
- 3. The same file name should populate the box with a .txt extension
- 4. Click on Save



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# Step 4 saving the data (continued):

Saving images of spectra:

Important!! Saving an image of the spectra does NOT save the raw data!!! Image files CANNOT be quantitatively analyzed!!

Mode 

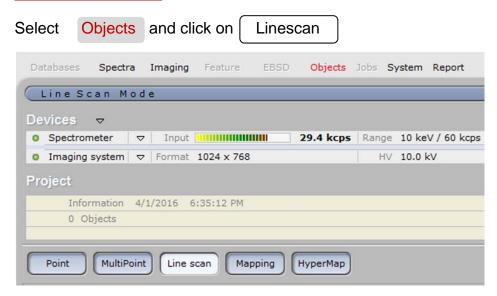
Under	Graphic	click on	Save		Spectrum
					Open
			_		Add to project
					Save
Save					Print
Volume	es 💼 edx\Data\	- 📰 🐐	X 🖻 💼	Preview	Add to report
	user 🗋				Element selection
2					Open
edx	$\downarrow$				Add to project
					Сору
Publi					Paste
🏭 C:					Save
0. D:					Result table
_ ¥:					Add to project
🖵 Z:	File name		In	formation	Сору
	Niobium ong				Save
	PNG images (*.prg		*		Add to report
	Bitmap images (*.br JPEG images (*.jpg PNG images (*.png	)			Graphic
	TIF images (*.tif)				Сору
					Save
			_		Twain export
2. Set to	sure the direct the file type y	ou want (e.			

- 3. Type in the same filename as for your data files
- 4. Click on Save



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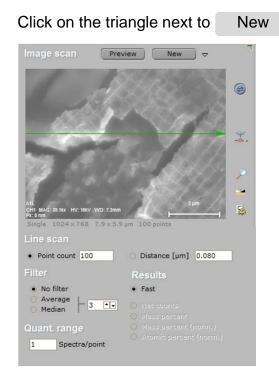
### <u>Step 1 - setup:</u>

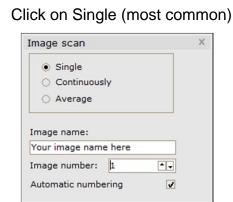


Set the EHT to ~4X the highest energy edge you will be measuring.

Set the aperture to 60um to 230um aperture for high count rates.

Set the working distance to 7mm +/- 1mm.





The point count determines the number of spectra across the image.

Or you can set the desired distance you want to cover



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Click on the image to set the measurement line



Linescan Mod

# 9.00 Step 1 – setup (continued):

Note the count rate (green circle).

Click on the triangle next to Spectrometer.

Spectra EBSD Imaging Feature Objects Jobs System Report Line Scan Mode Devices  $\bigtriangledown$ Input O Spectrometer 29.4 kcps Range 10 keV / 60 kcps HV 10.0 kV Project Information 4/1/2016 6:35:12 PM 0 Objects Point MultiPoint Line scan Mapping HyperMap

Choose the Max. pulse throughput to be just above the kcps value.

Choose the Maximum energy to include all the edges you are analyzing.

Max. pulse throughput 20 kcps 20 kcps	Maximum energy — 10 keV	Mode Normal opera	ition
<ul> <li>30 kcps</li> <li>60 kcps</li> <li>130 kcps</li> <li>275 kcps</li> </ul>	<ul> <li>20 keV</li> <li>40 keV</li> </ul>	O Standby Cooling	Do not change Mode or Cooling
<ul> <li>400 kcps</li> <li>600 kcps</li> <li>Automatic</li> </ul>	<ul><li>80 keV</li><li>Automatic</li></ul>	<ul> <li>Thermostat</li> <li>Maximum</li> </ul>	
Cooling system: Detector temperature:	On -30.1 °C		



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# Step 1 setup (continued):

### Click on the triangle next to Acquire

0	Spectrometer	Input		29.4 kcps	Range	10 keV / 60 kcps	Dead time	16 %	Real time	60
0	Imaging system	Format	1024 x 768		HV	10.0 kV	Magnification	39088	WD	7.2 mn
	0 Objects									

### Set the measuring time

Measuring time	
<ul> <li>Automatic</li> <li>Prec</li> <li>Manual</li> <li>Measuring time [s]</li> </ul>	ise •
Scan name	
Niobium	
Image number: 1	
Automatic numbering	
ОК	

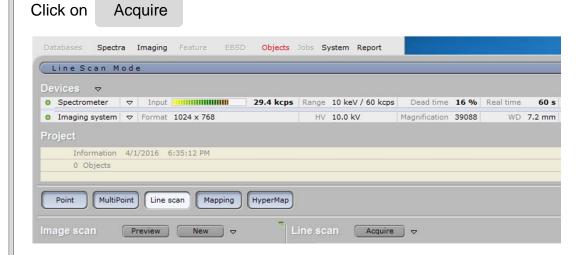


lescan Mod

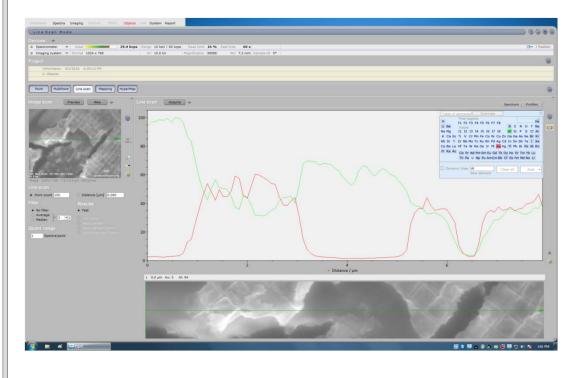
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Step 2 - acquisition:

# **po**M lescan



### Results are displayed as shown below

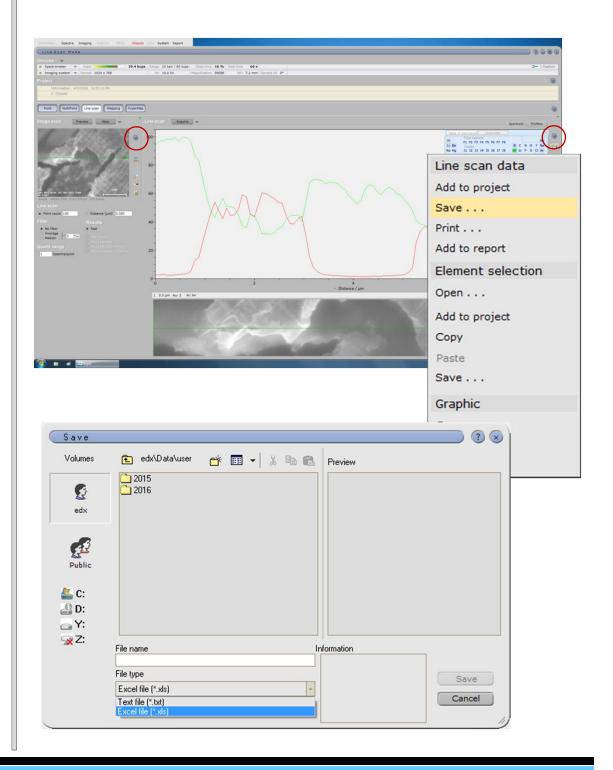




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# Stap 3 – Saving the data:

Click on the  $\Rightarrow$  next to the line scan to save the linescan.

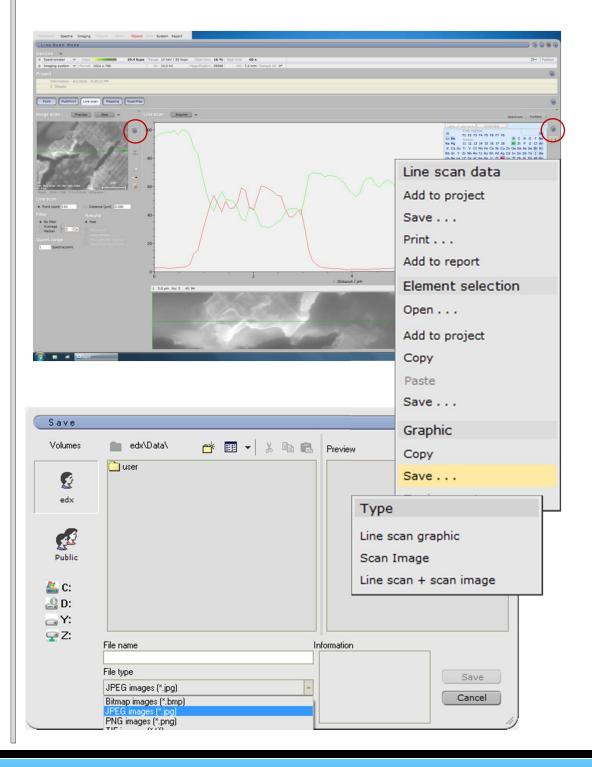




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# Stap 3 – Saving the data:

Click on the inext to the line scan to save the linescan data:



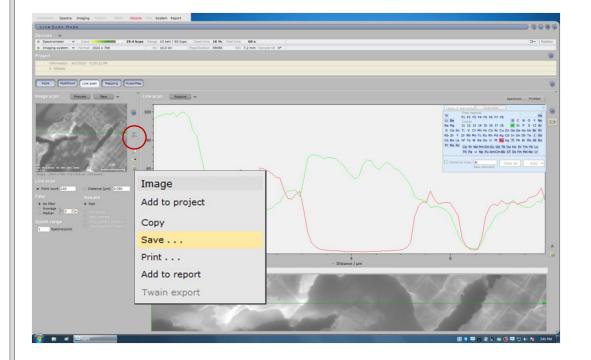


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# Stap 3 – Saving the data:

Click on the e next to the image to save the image.





Save						<b>?</b> ()
Volumes	edx\Data\	<b>r</b> 📰	•   %	þ f	Preview	
edx	C user					
Public						
🏭 C: ⊉ D: Y:						
🖵 Z:	File name			In	formation	
	File type					Save
	JPEG images (*.jpg)			-		
	Bitmap images (*.bmp)					Cancel
	JPEG images (*.jpg) PNG images (*.png)			l		1)



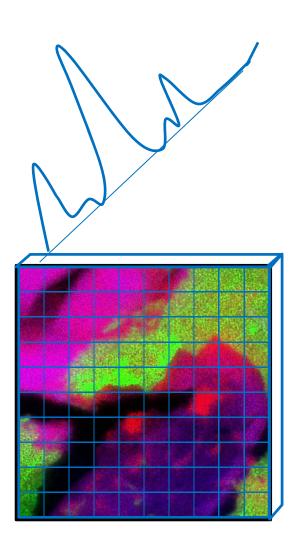
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# <u>10.00</u> **Principles of X-ray mapping:**

In an X-ray map a spectra is recorded at each pixel in the image.

The intensity of the chosen element(s) is then plotted at each pixel .

The map below shows oxygen (in Blue), aluminum (in Red), gold (in Green), When multiple elements are present, combinations of color can be created (e.g. aluminum (blue) and oxygen (red) show up as purple).

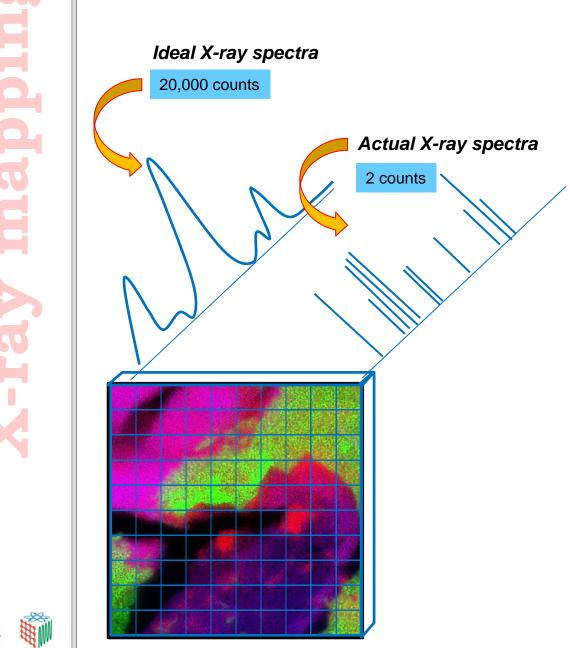




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# <u>10.00</u> *Principles of X-ray mapping:*

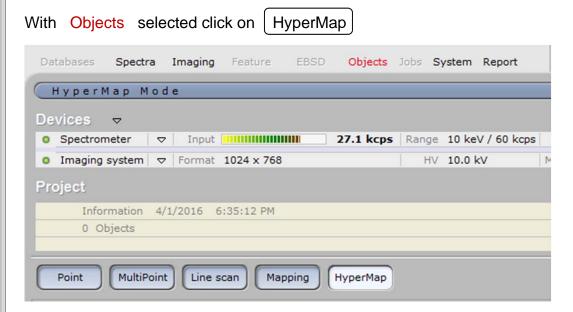
Note that short acquisitions times (or order of milliseconds/pixel) are usually required due to the large number of pixels typically involved. As a result spectra collected in mapping mode are usually very noisy compared to other acquisition modes..





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### <u>Step 1 - setup:</u>

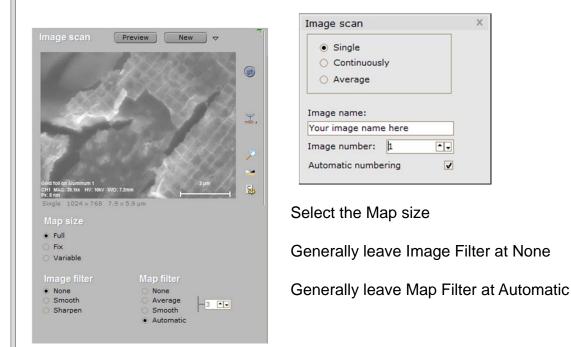


Set the EHT to ~4X the highest energy edge you will be measuring.

Set the aperture to 60um to 230um aperture for high count rates.

Set the working distance to 7mm +/- 1mm.

Click on the triangle next to New





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# <u>10.00</u> <u>Step 1 – setup (continued):</u>

Note the count rate (green circle).

Click on the triangle next to Spectrometer.

Databases	Spectra	Imaging	Feature	EBSD	Objects	Jobs	System	Report	
Hyperi	Мар Моо	de							
Devices									
<ul> <li>Spectror</li> </ul>	meter 🗸 🗢	Input			27.1 kcps	Rang	e 10 ke	V / 60 kcps	
o Imaging	system 🗢	Format	1024 x 768		$\smile$	н	V 10.0 k	٢V	
Project									
Info	rmation 4/	/1/2016 6	:35:12 PM						
0 0	bjects								
						_			
Point	MultiPoin	t Line s	can Mar	oping	HyperMap				

Choose the Max. pulse throughput to be just above the kcps value.

Choose the Maximum energy to include all the edges you are analyzing.

Vax. pulse throughput	Maximum energy	Mode Normal opera	ition	
<ul> <li>30 kcps</li> <li>60 kcps</li> <li>130 kcps</li> <li>275 kcps</li> <li>400 kcps</li> <li>600 kcps</li> <li>Automatic</li> </ul>	<ul> <li>20 keV</li> <li>40 keV</li> <li>80 keV</li> <li>Automatic</li> </ul>	<ul> <li>Standby</li> <li>Cooling</li> <li>Thermostat</li> <li>Maximum</li> </ul>	Do not c Mode or	<u> </u>
Cooling system: Detector temperature: Lighting:	On -30.1 °C		Clos	e



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# <u>Step 1 – setup (continued):</u>



Click on the triangle next to Acquire				
Databases Spectra Imaging Feature EBSD Objects Jo	bs System Report			
(				
(HyperMap Mode				
Devices 🗸				
Spectrometer      □ Input Input 27.1 kcps				
O Imaging system	HV 10.0 kV Magnification 39088 WD 7.2 mm Sample tilt 0°			
Project				
Information 4/1/2016 6:35:12 PM				
0 Objects				
Point MultiPoint Line scan Mapping HyperMap				
Image scan Preview New 🗢 🎙 Ma	p data Acquire ♥ Quantify ♥ Interactive PB-ZAF linemarks			
Measuring time (Fast map)	Click on Cycles and set to 1			
Manual				
	Give the map a name, if desired			
<ul> <li>Measuring time [s]</li> <li>100</li> </ul>				
Cycles	Click on OK			
Switch off microscope				
Options				
Interlaced measurement				
Use 1. microscope image only				
Image average [s] 30				
Map name				
Gold film on Aluminum				
Map number: 172				
Automatic numbering				
Object name				
Map01				
OK				

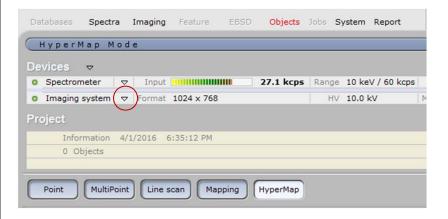


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### <u>Step 1 – setup (continued):</u>

### Click on the triangle next to Imaging System

X-ray mapping



In the window that opens (see below) set the dwell time in microseconds.

A long dwell time will result in better S/N but can take a very long time.

To speed things up, use as large an aperture as you can to get the strongest signal.

Hardware conf	iguration	?
Image resolution [pixel] Image inputs Power synchronisation	512 CH1 CH2	Microscope settings         Magnification       464         High voltage [kV]       20.0         Working distance [mm]       18.2         Communication active
Imaging		Extern scan On Off
Dwell time [µs] Line average Image scan time	2 ••• 1 0.4 s	Tilt     Sample tilt [°]     Additional tilt corr.
Mapping Dwell time [µs] Line average Image scan time Line scan Dwell time [µs]	8192 ••• 1 26 min 32 •••	Tilt around x
		Close

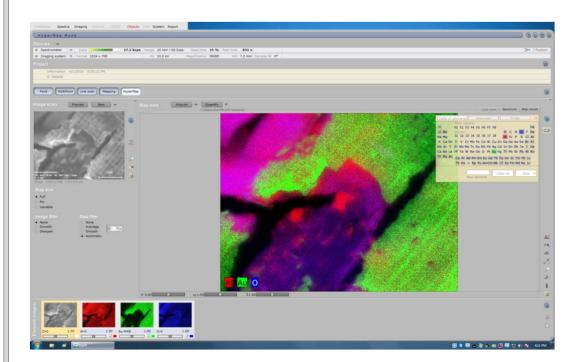


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# <u>Step 2 – acquisition:</u>

### Click on Acquire

X-ray mapping



The individual maps are shown in the lower left corner

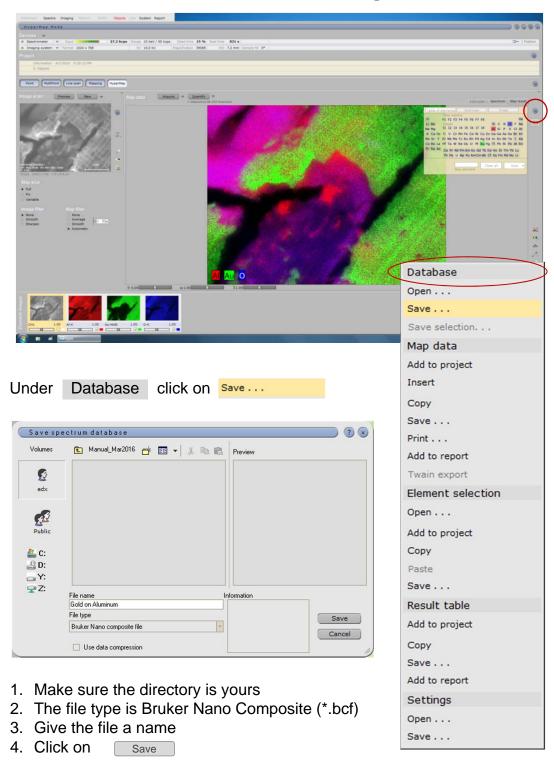
The large composite map is comprised of all the signals checked in the lower images



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# <u>10.00</u> <u>Step 3 – saving data:</u>

Most important – save the raw data: Click on the  $\rightleftharpoons$  icon as shown.

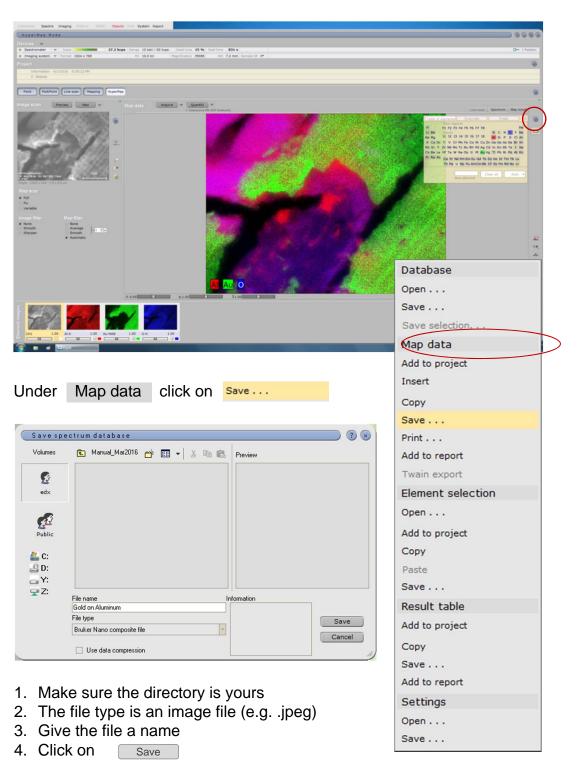




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# <u>Step 3 – saving data (continued):</u>

Saving the composite image: Click on the  $\bigcirc$  icon as shown.

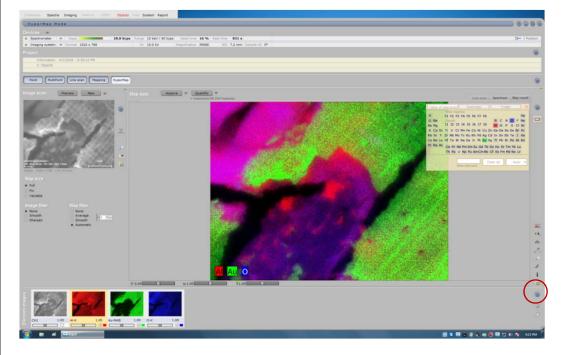






# <u>10.00</u> <u>Step 3 – saving data (continued):</u>

Saving the individual images: Click on the eicon as shown.



Under	Image	click on	Save		
					Image
Savespe	ectrum database			<b>)</b> ? ×	Add to project
Volumes	€ Manual_Mar2016 🗃	X -	Preview		Сору
8					Save
edx					Print
<u>_</u>					Add to report
Public					Twain export
💒 C: 🚑 D:					
👝 Y: 🚅 Z:	File name		Information		
	Gold on Aluminum		Information		
	File type			Save	
	Bruker Nano composite file		-	Cancel	
	Use data compression			//	

1. Make sure the directory is yours

Save

- 2. The file type is an image file (e.g. .jpeg)
- 3. Give the file a name





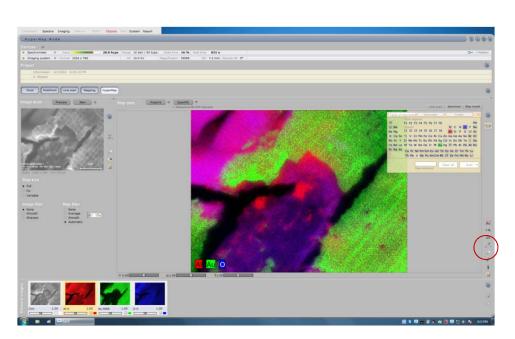


# <u>10.00</u> <u>Step</u>

# Step 4 - quatification:

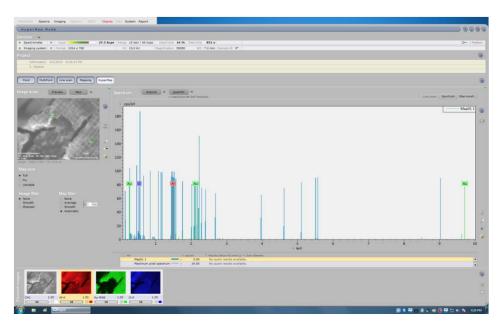
Right click on the eyedropper icon on the lower right hand side





Select the area you want to analyze.

### The spectra will appear as shown

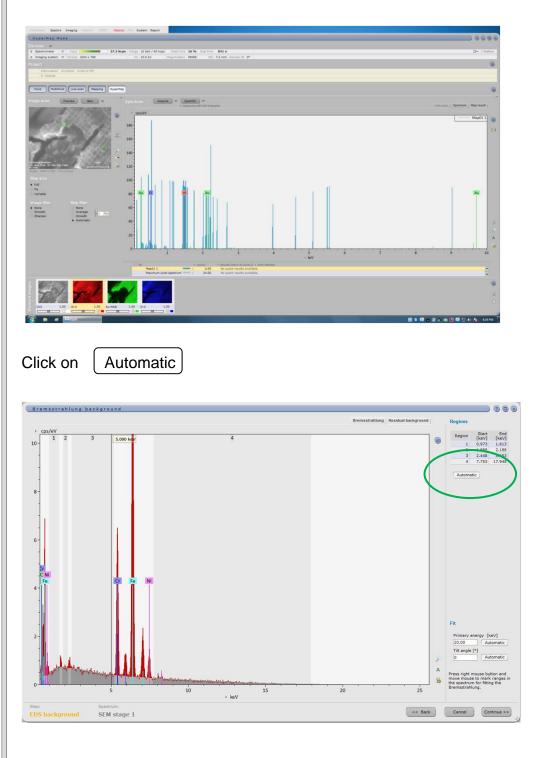




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# 10.00 Step 4 - quantification:

To quantify the elements choose a spectra and click on Quantify

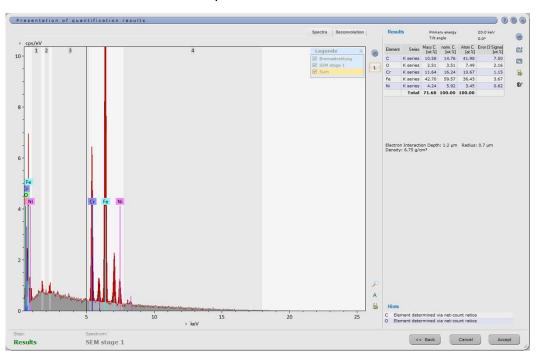




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# <u>10.00</u> <u>Step 4 – quantification (continued):</u>

X-ray mapping



Note a result table has showed up in the corner:

Click on Accept

Result	ts	Primary energy Tilt angle			20.0 keV 0.0°	
Element	Series	Mass C. [wt.%]	norm. C. [wt.%]	Atom C. [at.%]	Error (3 Sigma) [wt.%]	
С	K series	10.58	14.76	41.98	7.50	
0	K series	2.51	3.51	7.49	2.16	
Cr	K series	11.64	16.24	10.67	1.15	
Fe	K series	42.70	59.57	36.43	3.67	
Ni	K series	4.24	5.92	3.45	0.62	
	Total	71.68	100.00	100.00		

Electron Interaction Depth: 1.2  $\mu m$   $\,$  Radius: 0.7  $\mu m$  Density: 6.75 g/cm³  $\,$ 

The software displays the results in several ways.

Note also that the electron interaction depth and radius are provided.

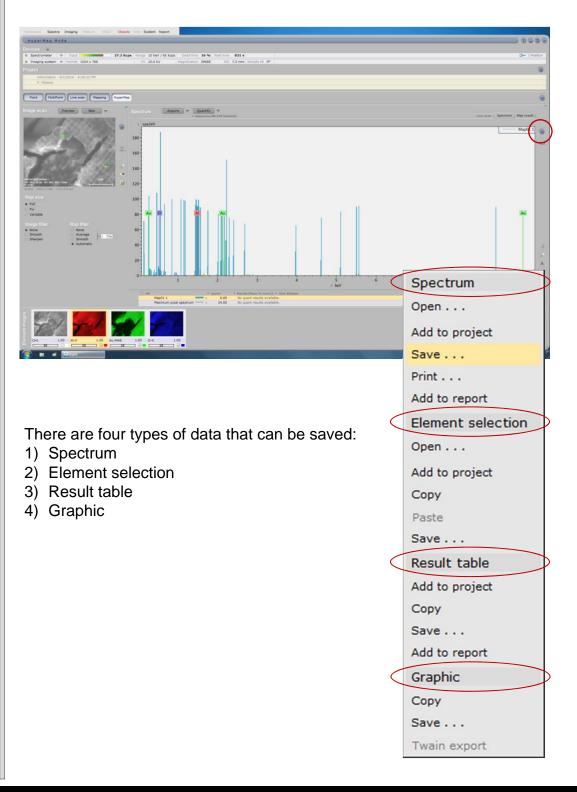


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### <u>Step 5 – saving data:</u>

Click on the 😑 icon as shown.

X-ray mapping





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# Step 5 – saving the data (continued):

X-ray mapping

Saving the spectra:				
Under Spectrum click on Save	Spectrum			
	Open			
- C	Add to project			
Save spectrum	Save			
Volumes 💼 edx\Data\ 😽 📰 👻 🐰 🗈 💼 Preview	Print			
Se user	Add to report			
edx (	Element selection			
	Open			
	Add to project			
Public	Сору			
🚣 C:	Paste			
🔮 D:	Save			
□ Y:	Result table			
File name Information	Add to project			
File type	Сору			
Bruker Nano spectra (*.spx) Bruker Nano spectra (*.spx)	Save			
Text (*.txt) Excel 97/2000/XP (*.xls)	Add to report			
EMSA spectra (*.msa)	Graphic			
	Сору			
1. Make sure the directory is yours	Save			
<ol> <li>Set the file type to Bruker Nano spectra (*.spx)</li> <li>Give the spectra a name</li> </ol>	Twain export			
4. Click on Save				
File name				
File type	Save			
Excel 97/2000/XP (*.xls)	Cancel			
Bruker Nano spectra (*.spx) Text (*.txt) Excel 97/2000/XP (*.xls)	Cancer			
EMSA spectra (*.msa)				
5. Under Spectrum click on Save again				
6. Set the file type to Excel 97/2000/XP (*.xls)				
7. The same file name should populate the File name box				
8. Click on Save				



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# <u>Step 5 – saving the data (continued):</u>

X-ray mapping

Saving the quantified results:	
	Spectrum
Under Result table click on save…	Open
0	Add to project
Save results	Save
Volumes 💼 edx\Data\ 😽 🎫 👻 🖟 Preview	Print
Sector Contraction	Add to report
edx	Element selection
	Open
	Add to project
Public	Сору
🚣 C:	Paste
🔐 D:	Save
	Result table
File name Information	Add to project
File type	Сору
Text table (*.txt)	Save
Text table (".txt) Bitmap images (".bmp)	Add to report
JPEG images (*.jpg) PNG images (*.png)	Graphic
TIF images (".tif)	Сору
	Save
1. Make sure the directory is yours	Twain export

- 2. Set to Text table (\*.txt)
- 3. The same file name should populate the box with a .txt extension
- 4. Click on Save



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# <u>10.00</u> Step 5 - saving the data (continued):

Saving images of spectra:

Important!! Saving an image of the spectra does NOT save the raw data!!! Image files CANNOT be quantitatively analyzed!!

X-ray mapping

Under Graphic click on Save	Spectrum
	Open
	Add to project
	Save
Save	Print
	Add to report
Volumes edx\Data\ 🔆 🖽 🗸 🖻 💼 Preview	Element selection
	Open
edx	Add to project
	Сору
Public	Paste
	Save
▲ C: ▲ D:	Result table
- Y:	Add to project
File name Information	Сору
ng	Save
File type PNG images (*.png)	Add to report
Bitmap images (*.bmp) JPEG images (*.jpg)	Graphic
PNG images (*.ong) TIF images (*.tif)	Сору
	Save
4 Males and the divertements warme	Twain export
<ol> <li>Make sure the directory is yours</li> <li>Set to the file type you want (e.gpng)</li> <li>Type in the same filename as for your data files</li> <li>Click on Save</li> </ol>	



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