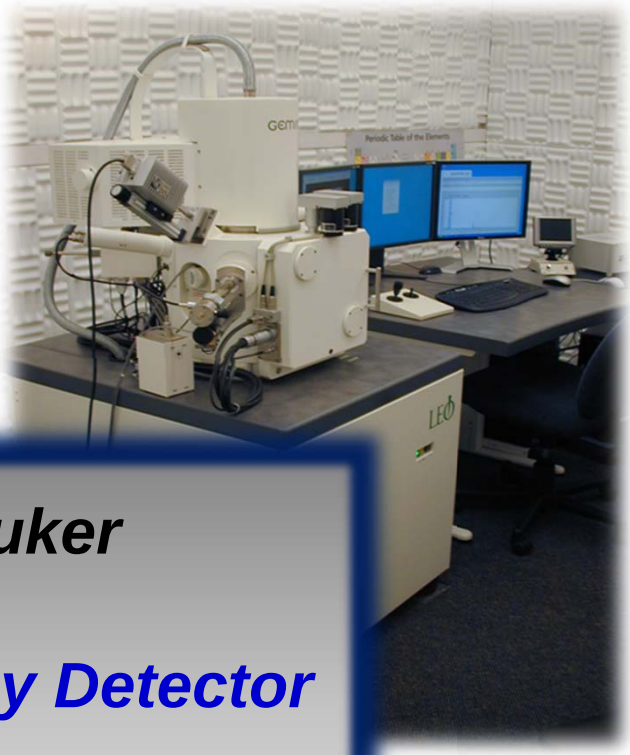
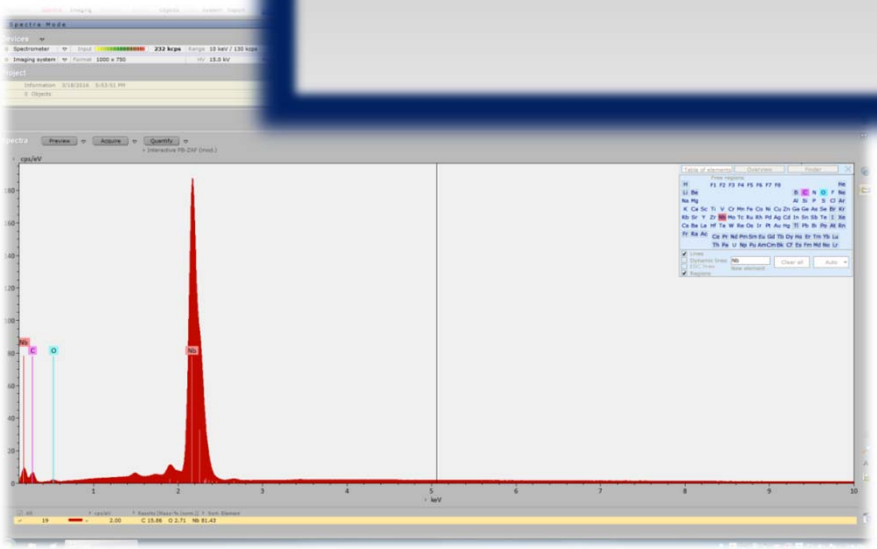
Operating Manual

Part 1



Bruker

SDD X-ray Detector



Contacts

Mick:	255-0650
Cornell Police:	255-5111
Emergency:	911
Phil Carubia:	255-6757

1.00

Laboratory Safety

Users are required to have passed Cornell's on-line laboratory safety training courses before they can use the Bruker X-ray detector on the Keck SEM.

Do not bring any chemicals or hazardous samples or materials into the lab.

Take specimens, stubs, and raw material with you when you leave. Do not store them in the bakeout chamber.

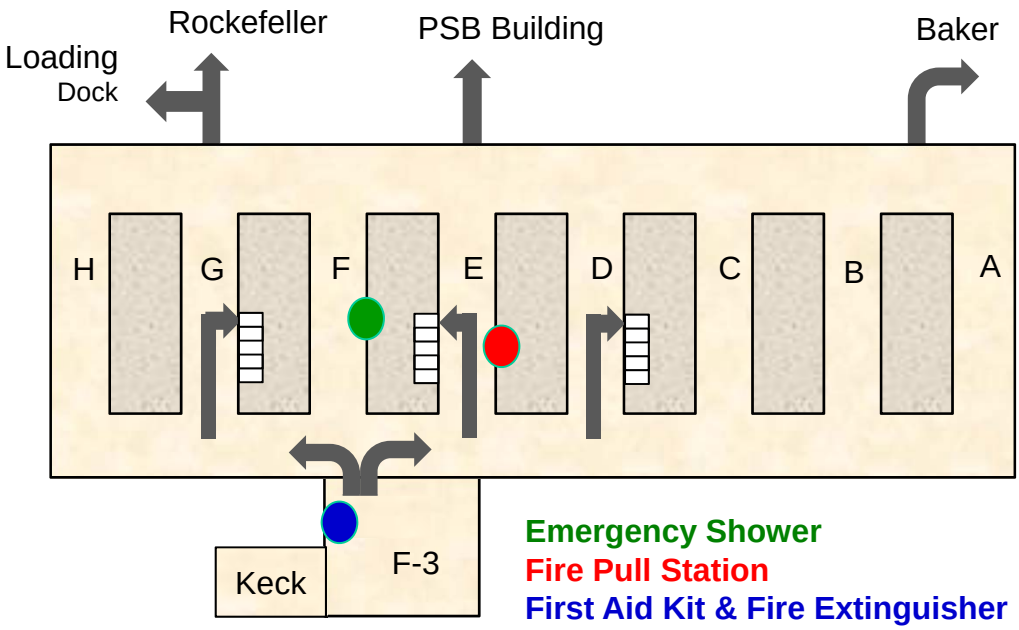
Isopropanol and Aero-Duster are supplied for your use. Wear safety glasses and read the appropriate MSDS before using them.

Nitrogen gas cylinders are changed by CCMR staff only.

Users failing to follow safety rules will be denied access to the lab.

Fire Safety

In case of fire leave immediately and close the door behind you. Do not use the elevators.



1.05

Laboratory Policies

If ANYTHING does not even SEEM right, leave everything as it is and get or call Mick (255-0650)

DO NOT attempt to repair ANY part of the X-ray detector or remove panels, cables, or any part of the X-ray detector..

DO NOT add any software or hardware to the computer

Flash Drives (Memory Sticks) are forbidden
Data MUST be removed by burning a CD or via Cornell DropBox

DO NOT change the room temperature or bring food or drink into F-3 or the SEM room

Wear gloves when venting the SEM, handling samples, or using the pumping station

If you sign up and then cannot use your time, cancel your time via Coral or contact Mick at least one weekday before your scheduled time.

Failure to show up without canceling, unless due to illness or other emergency, can result in use charges

If you are more than 30 minutes late then the microscope becomes available to other users.

If you feel sick, please reschedule your session. You will not be charged if you stay home due to illness.

1.10 Data Storage

Data storage and safety is NOT guaranteed! - users are responsible for their data, and must copy their data in a timely fashion.

Data will only be kept on the hard drive for one year. Periodically the hard drive will be erased, leaving only one years' data there.

1.20 How to Acknowledge CCMR

When research is published using data from this microscope (including the X-ray detector), the facility and the grant number must be acknowledged:

This work made use of the electron microscopy facility of the Cornell Center for Materials Research (CCMR) with support from the National Science Foundation Materials Research Science and Engineering Centers (MRSEC) program (DMR 1120296).

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2.00 Access

User Status	Signup	Room Entry	Access
Novice (< 2 sessions)	Email	Manager	M-F 8am – 5pm
Trained (>2 sessions)	Coral	Card Swipe	M-F 8am – 5pm
Experienced (>4 sessions)	Coral	Card Swipe	24/7

DO NOT use your card to let others into the SEM room

DO NOT lend your card to other users.

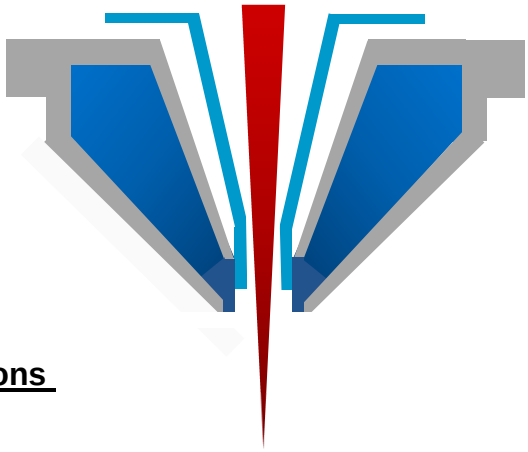
3.00 *About this manual*

This is a ***basic, limited use manual***, intended to help new users get started on the Bruker Quantax X-ray Detector. It provides information on basic data acquisition and X-ray mapping. It is not intended to be exhaustive, and there are many principles about X-ray analysis and features of this detector that are not covered by this manual.

4.00

Understanding the excitation volume:

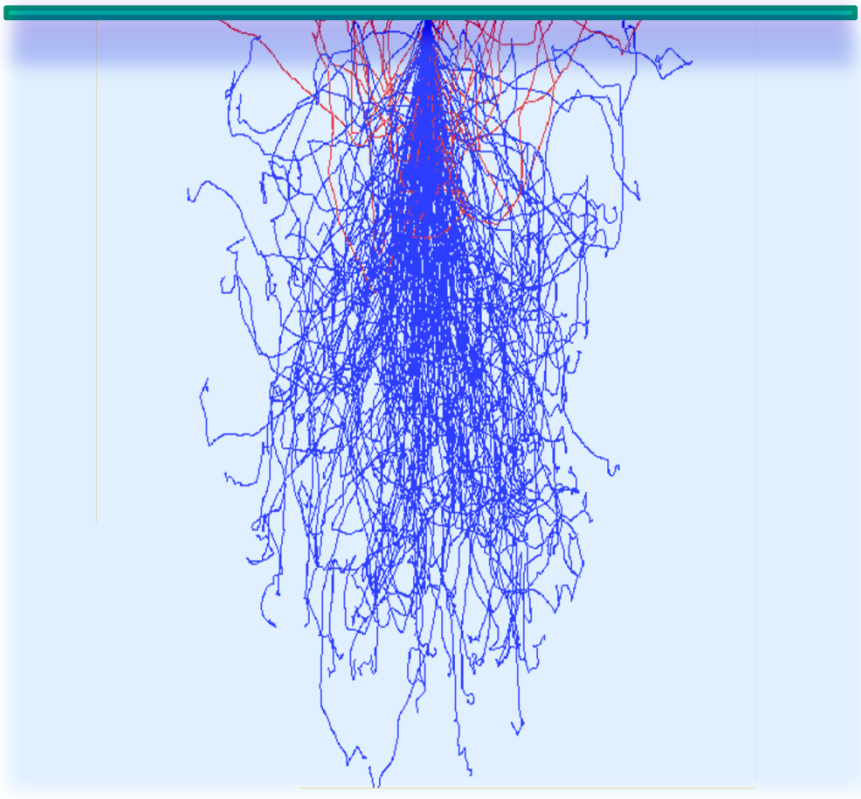
The volume that the x-rays emerge from is much larger than the spot size of the electron beam. This volume is a function of the accelerating voltage and the composition of the sample.



Emission depth:

Secondary Electrons

X-rays

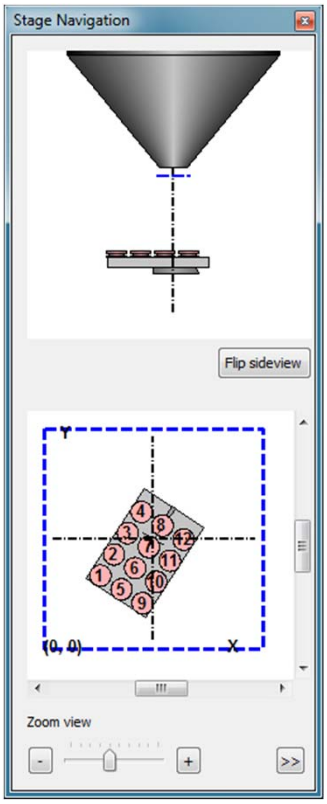


nm scale

micron scale

5.00

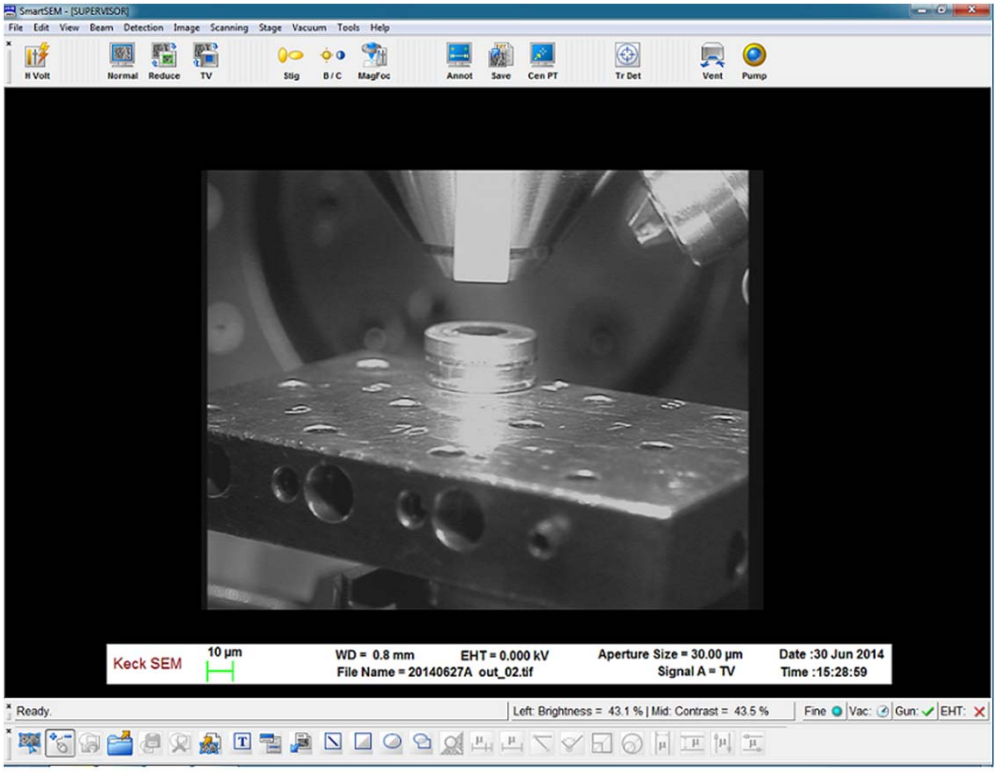
How to position your sample for X-ray analysis:



Move the sample in Z-direction with the joystick to about 7mm from the end of the pole piece as shown below.

Generally a distance between 6mm and 8mm will provide an adequate count rate, but beyond these values in either direction there will be a drop off of X-ray intensity.

Do not trust the cartoon at left showing the gap between the sample and pole piece – your sample height will likely be different!

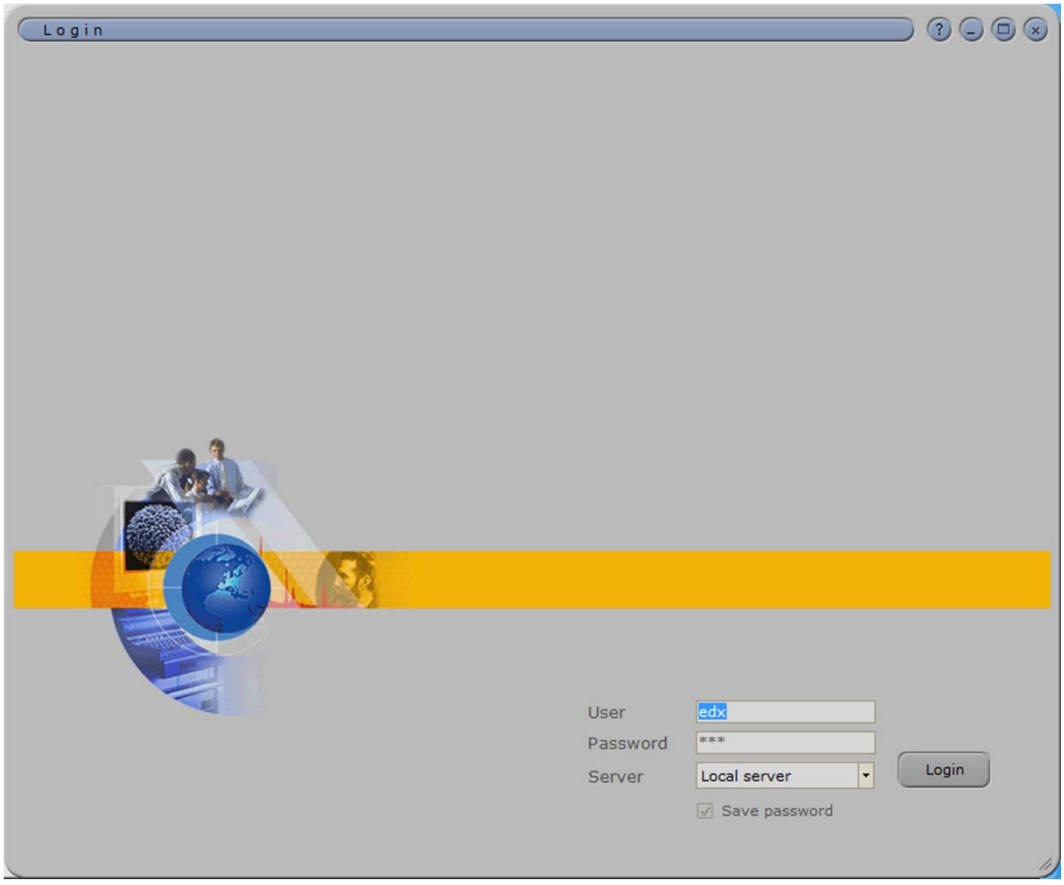


CCMR

5.10 *How to start the software:*

Click on Esprit

Click on Login (password already entered)



6.00

Area Mode

Step 1 - setup:

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

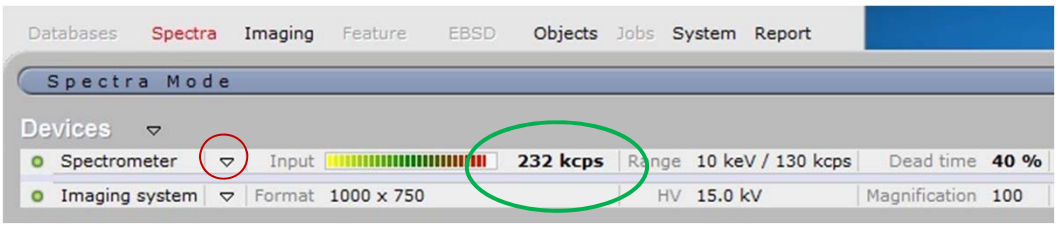
Set the aperture to 60um to 230um aperture for high count rates.

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

Turn on beam in area of interest.

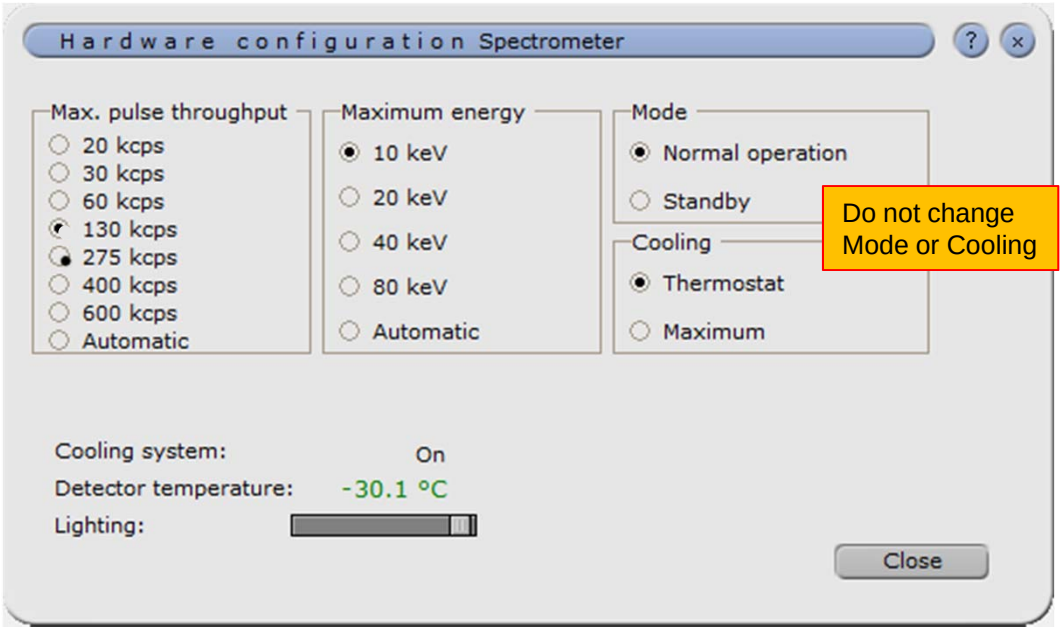
Click on **Spectra** Note the count rate (green circle).

Click on the triangle next to Spectrometer.



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

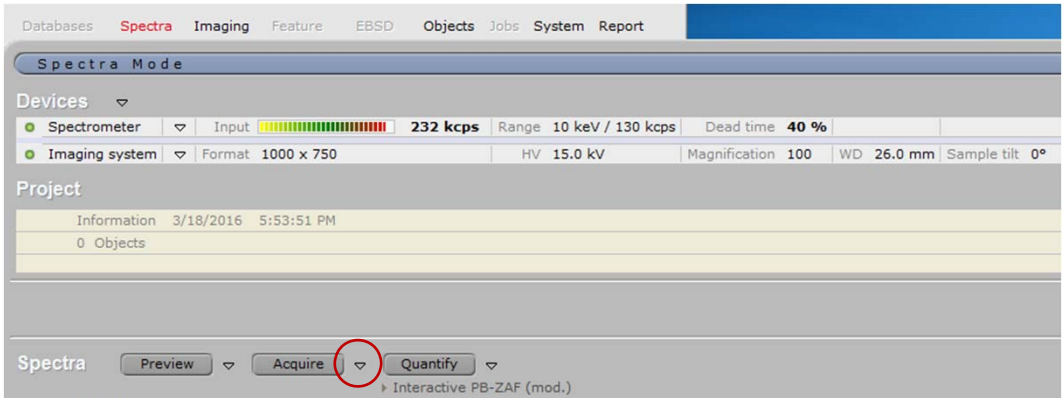
Choose the Maximum energy to include all the edges you are analyzing.

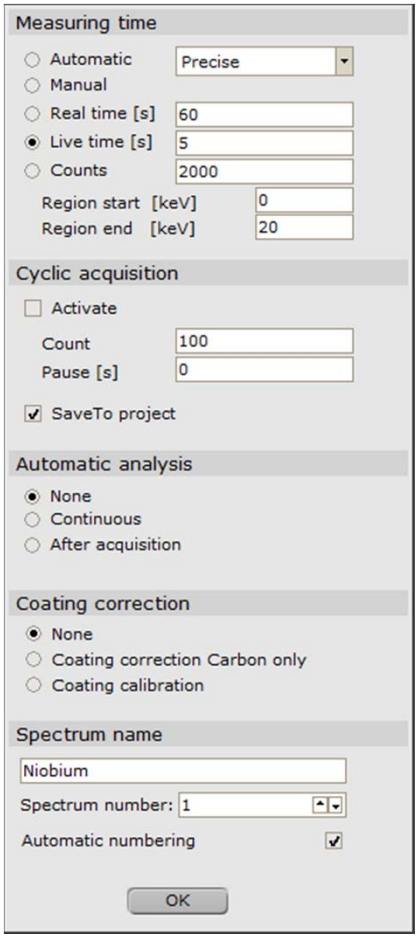


6.00

Step 1 – setup (continued):

Select the triangle next to Acquire





Set the Real time (or Live time) as desired.
Note Real Time = Live Time + Dead Time

.

If you enter a Spectrum name and check Automatic numbering then that name and incremental number will be assigned to your spectra.

Click on OK

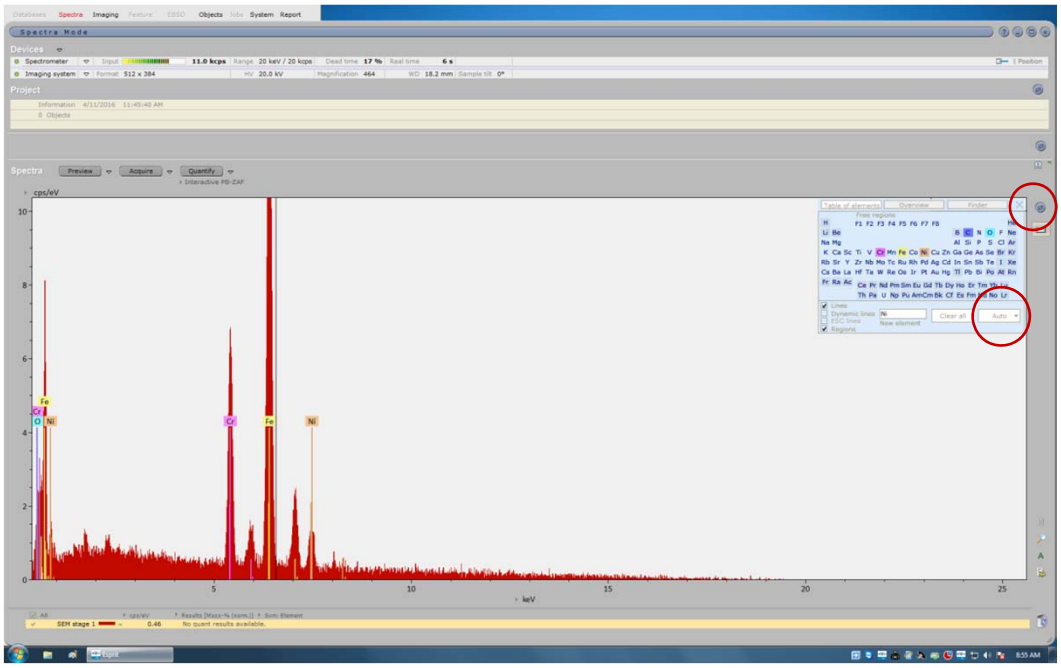
6.00

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

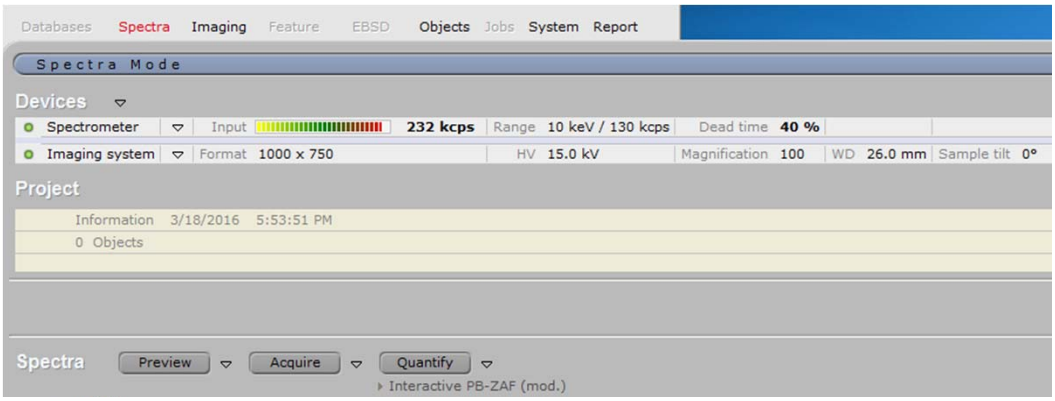


Click on the Auto button Auto ▾ to automatically identify the peaks or click on the element in the Periodic Table to manually identify the peaks,

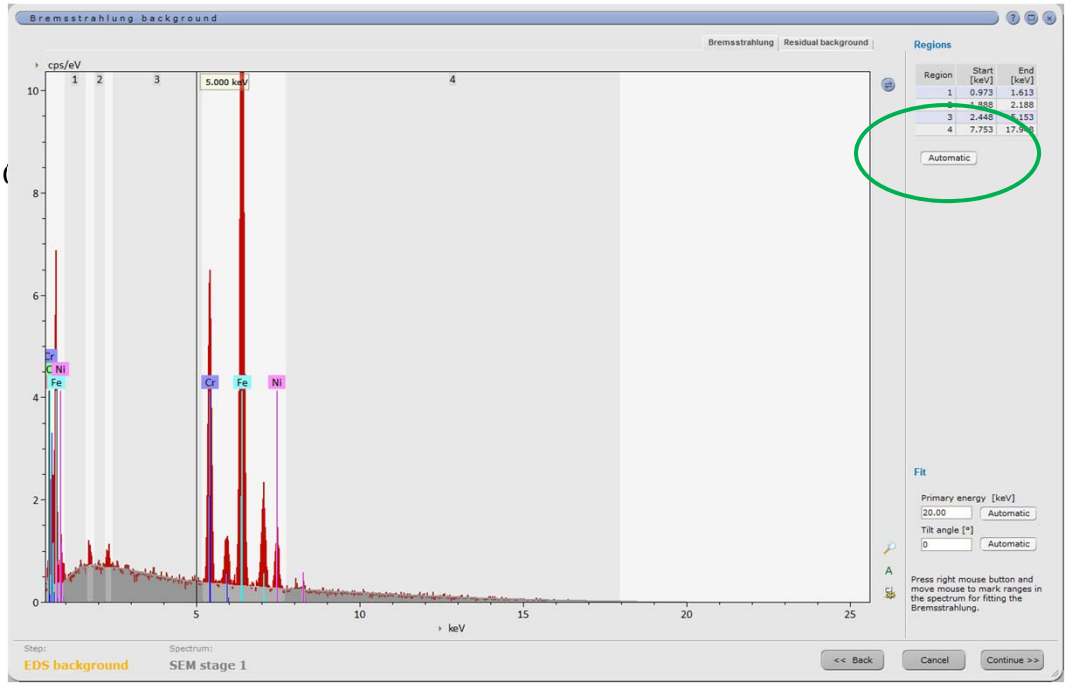
6.00

Step 3 - quantify:

To quantify the elements present in a spectra click on Quantify



Click on Automatic

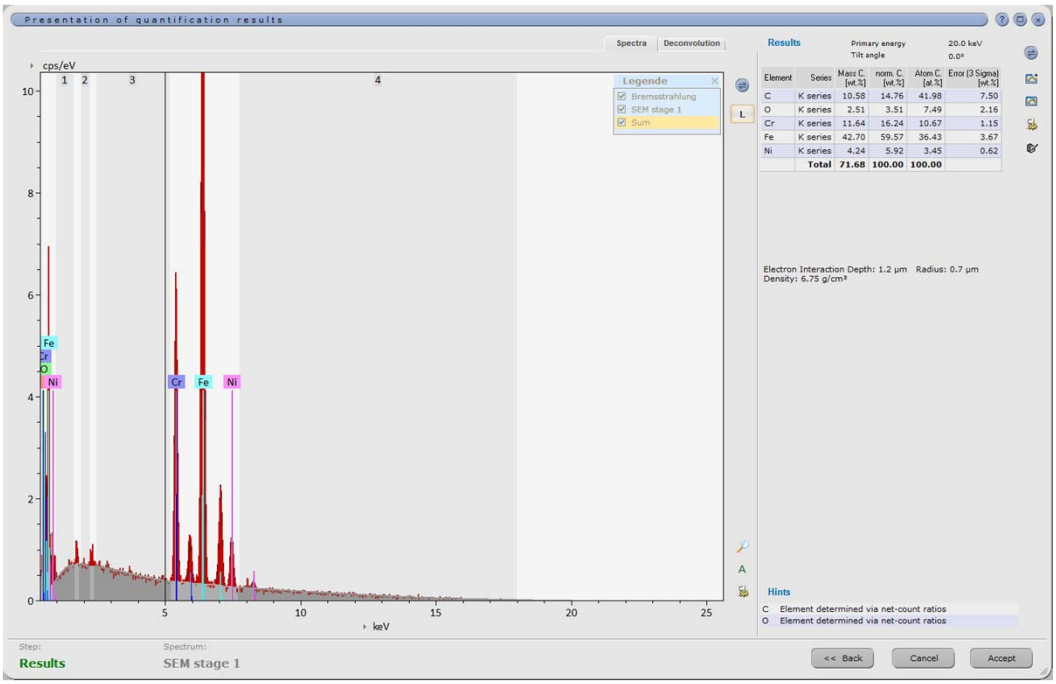


Area Mode

6.00

Step 3 – quantify (continued):

Note a result table has showed up in the corner:



Click on Accept

Results

Primary energy 20.0 keV
Tilt angle 0.0°

Element	Series	Mass C. [wt.%]	norm. C. [wt.%]	Atom C. [at.%]	Error (3 Sigma) [wt.%]
C	K series	10.58	14.76	41.98	7.50
O	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 µm Radius: 0.7 µm
Density: 6.75 g/cm³

The software displays the results in several ways.

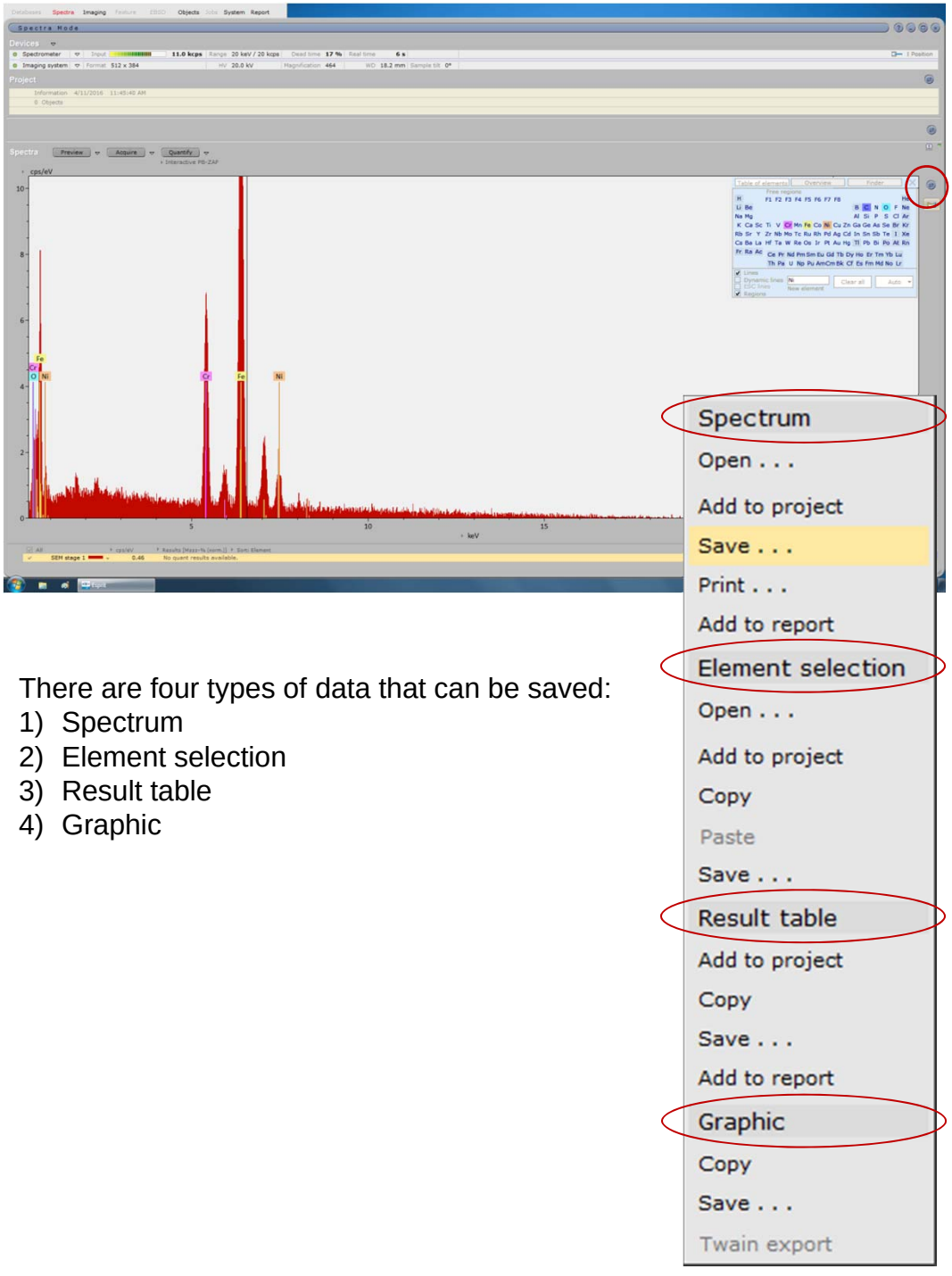
Note also that the electron interaction depth and radius are provided.

Area Mode

6.00

Step 4 – saving the data:

Click on the icon as shown.



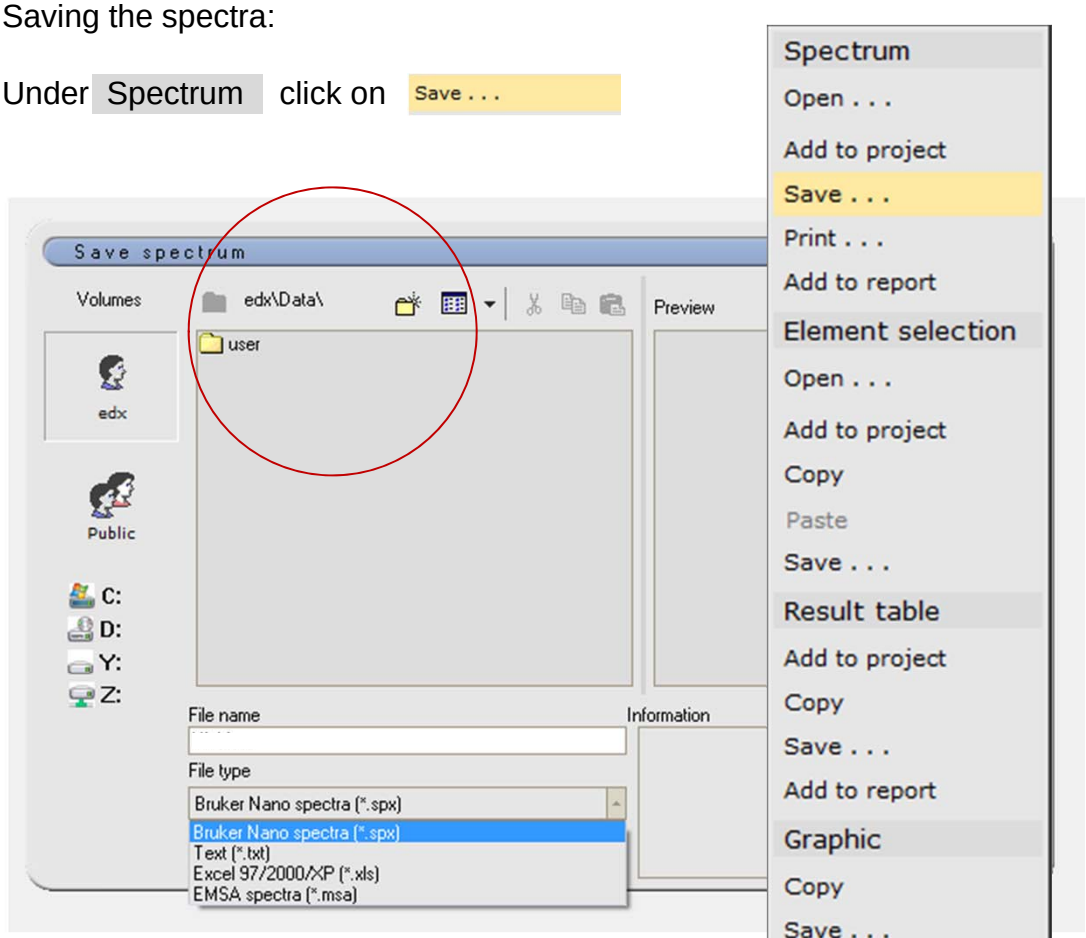
There are four types of data that can be saved:

1) Spectrum
2) Element selection
3) Result table
4) Graphic

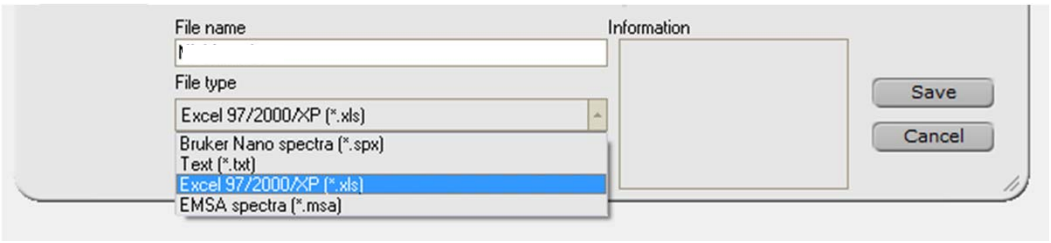
Step 4 – saving the data (continued):

Saving the spectra:

Under Spectrum click on Save . . .



1. Make sure the directory is yours
2. Set the file type to Bruker Nano spectra (*.spx)
3. Give the spectra a name
4. Click on Save



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

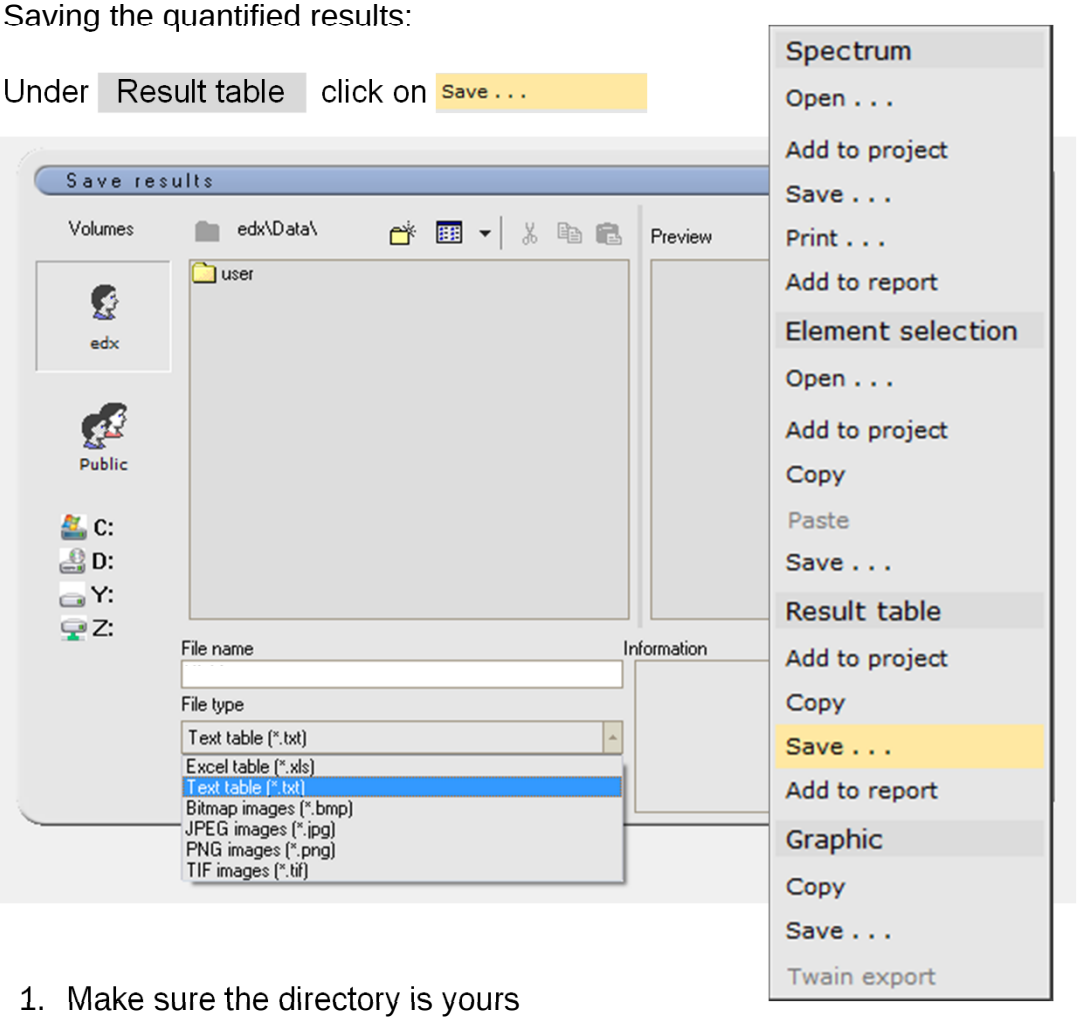
Area Mode

6.00

Step 4 – saving the data (continued):

Saving the quantified results:

Under Result table click on Save . . .



1. Make sure the directory is yours
2. Set to Text table (*.txt)
3. The same file name should populate the box with a .txt extension
4. Click on Save

Area Mode

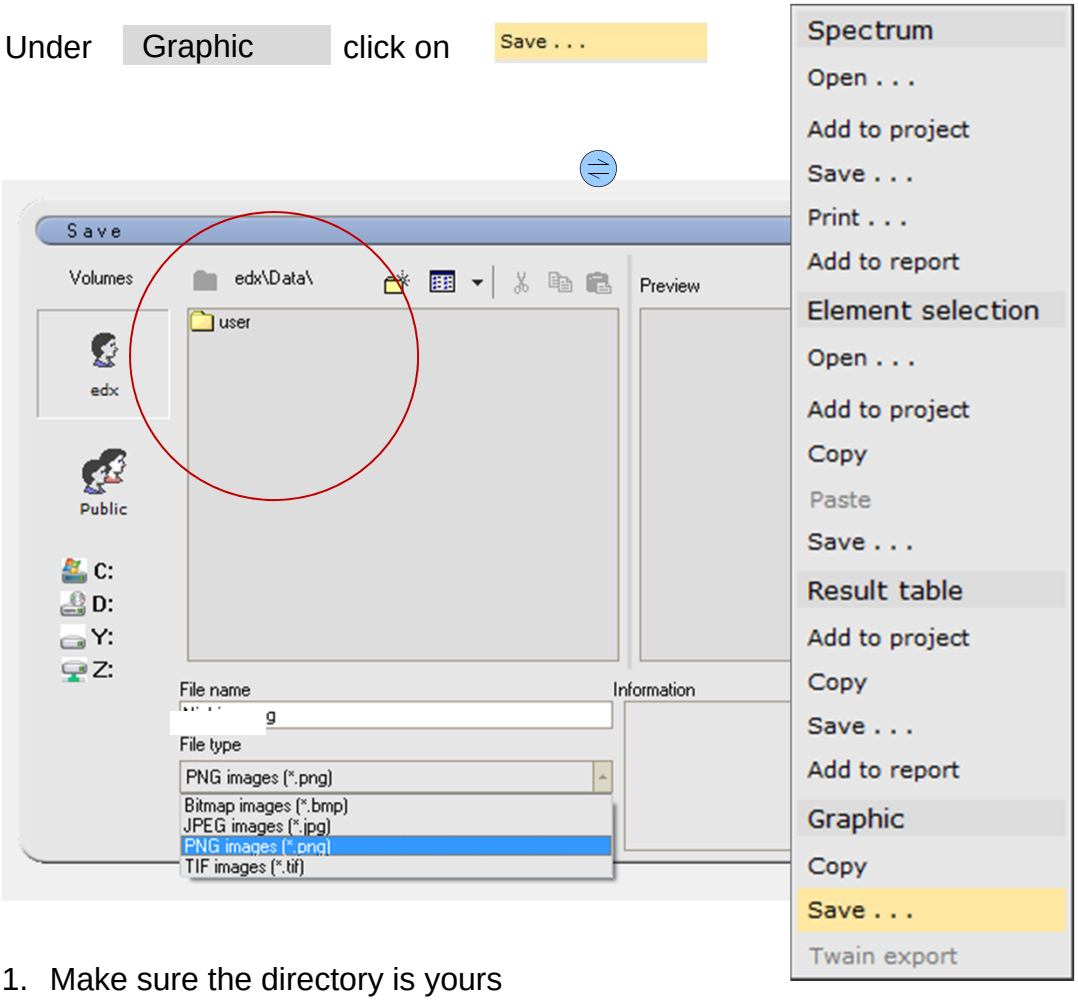
Area Mode

Step 4 – saving the data (continued):

Saving an image of the spectra:

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

Under Graphic click on Save . . .

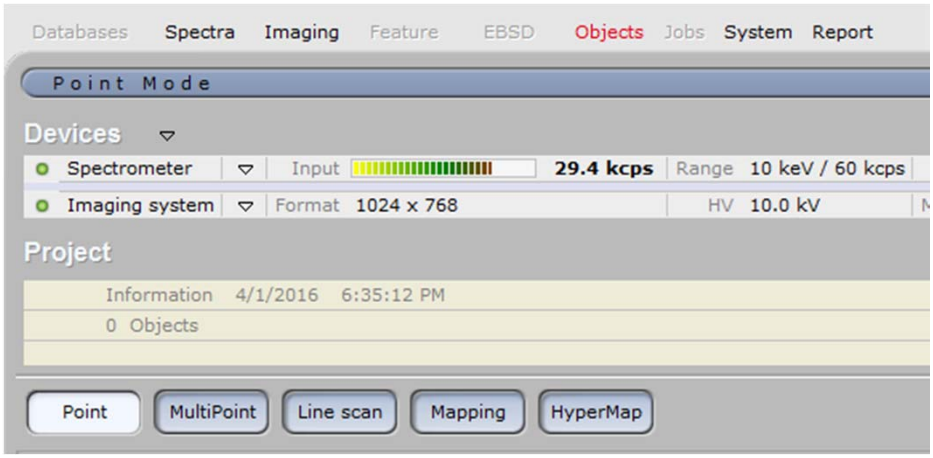


1. Make sure the directory is yours
2. Set to the file type you want (e.g. .png)
3. Type in the same filename as for your data files
4. Click on Save

7.00

Step 1 - setup:

Select Objects and click on Point

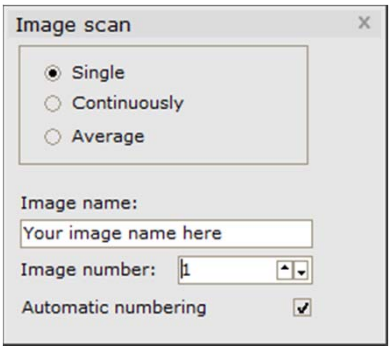


Set the EHT to ~4X the highest energy edge you will be measuring.

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



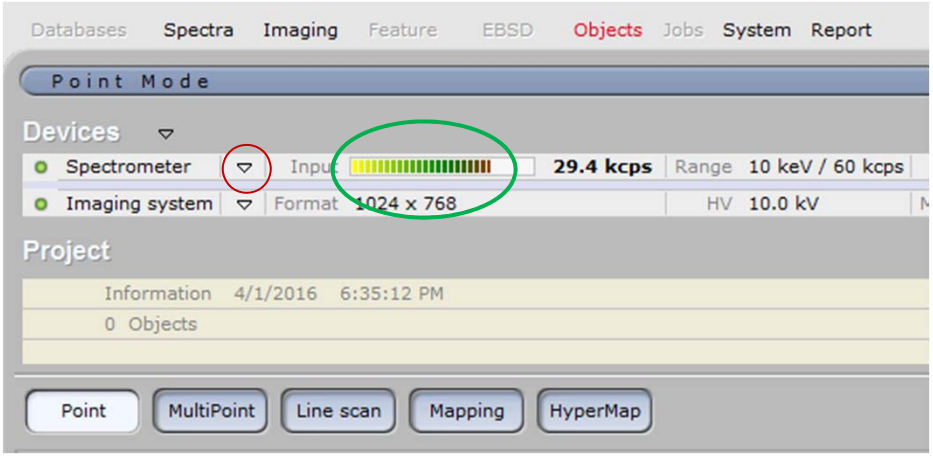
Click on the image to set the measurement point (green crosshair).

Point Mode

Step 1 – setup (continued):

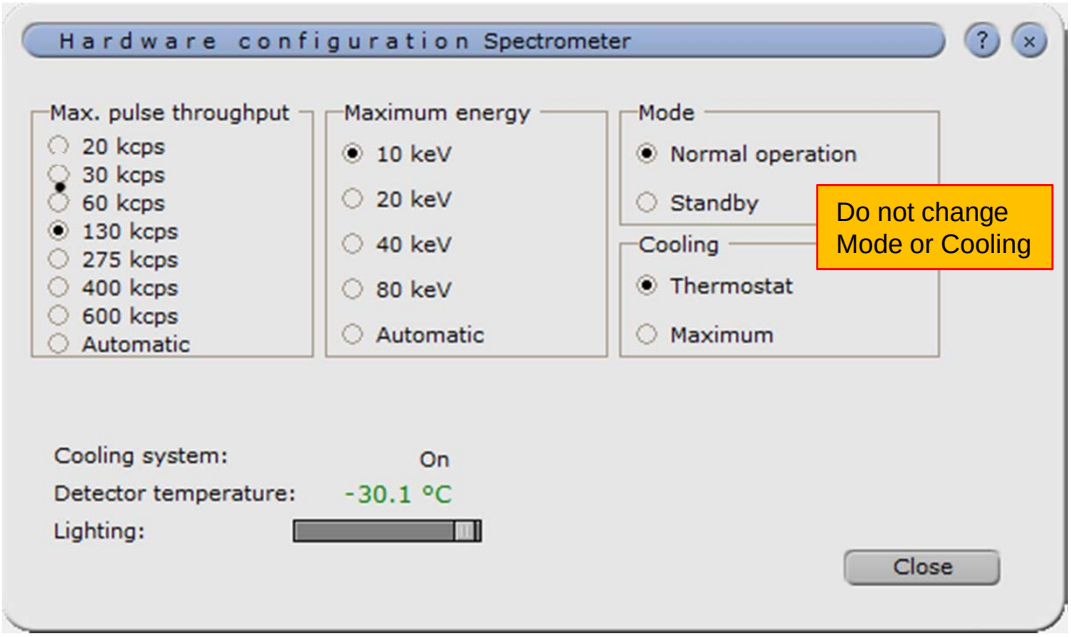
Note the count rate (green circle).

Click on the triangle next to Spectrometer.



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

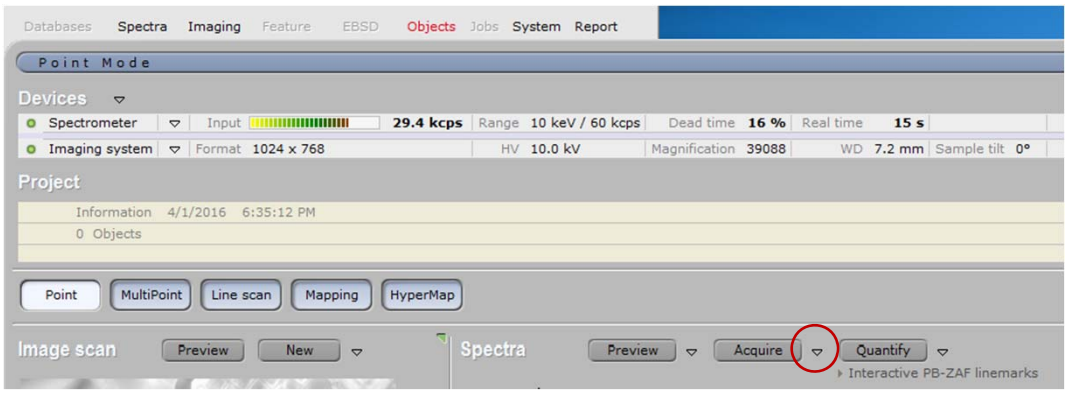
Choose the Maximum energy to include all the edges you are analyzing.

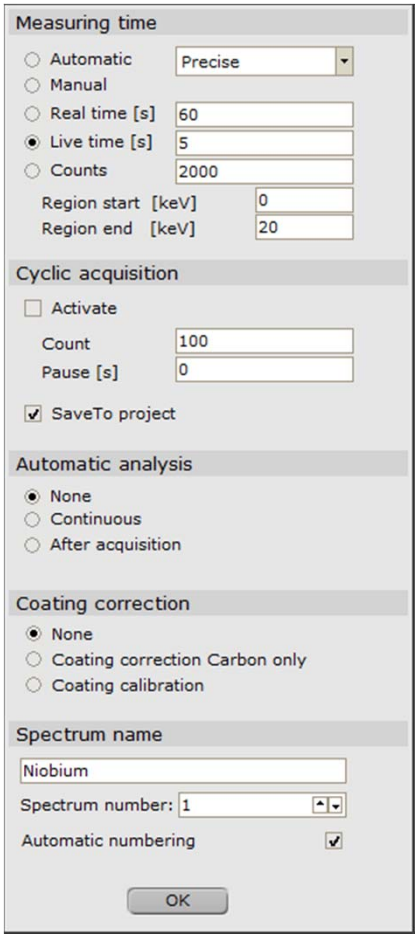


7.00

Step 1 – setup (continued):

Select the triangle next to Acquire





Set the Real time (or Live time) as desired.
Note Real Time = Live Time + Dead Time

.

If you enter a Spectrum name and check Automatic numbering then that name and incremental number will be assigned to your spectra.

Click on OK

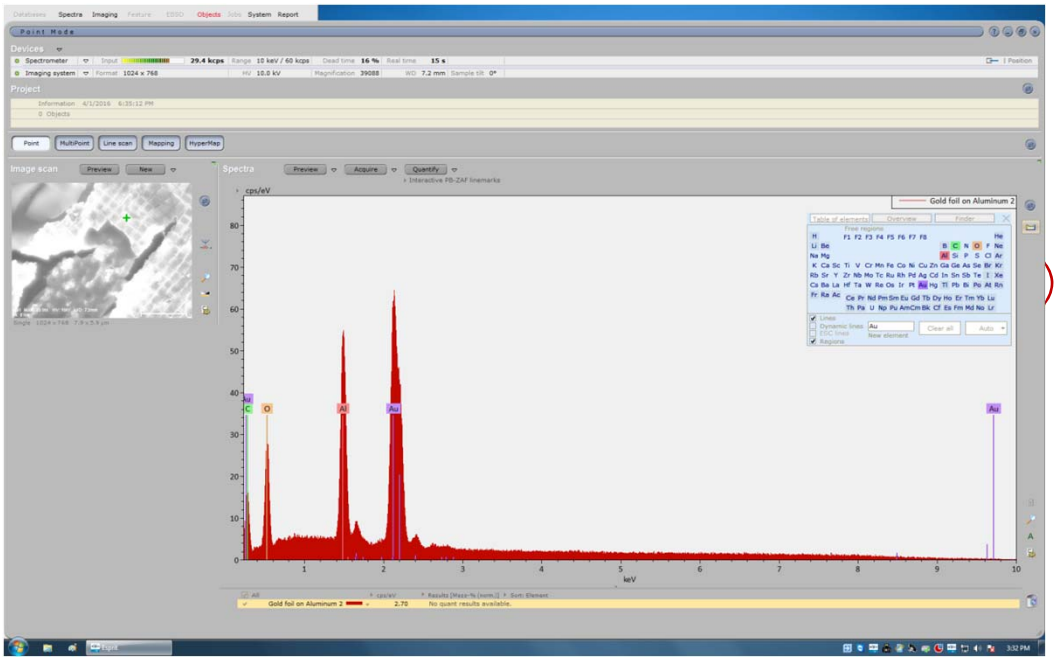
7.00

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

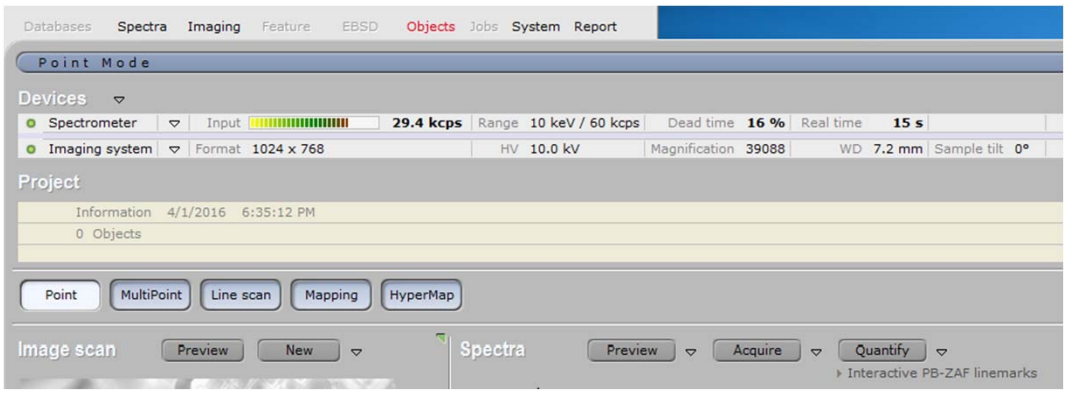


Click on the Auto button Auto ▾ to automatically identify the peaks or click on the element in the Periodic Table to manually identify the peaks,

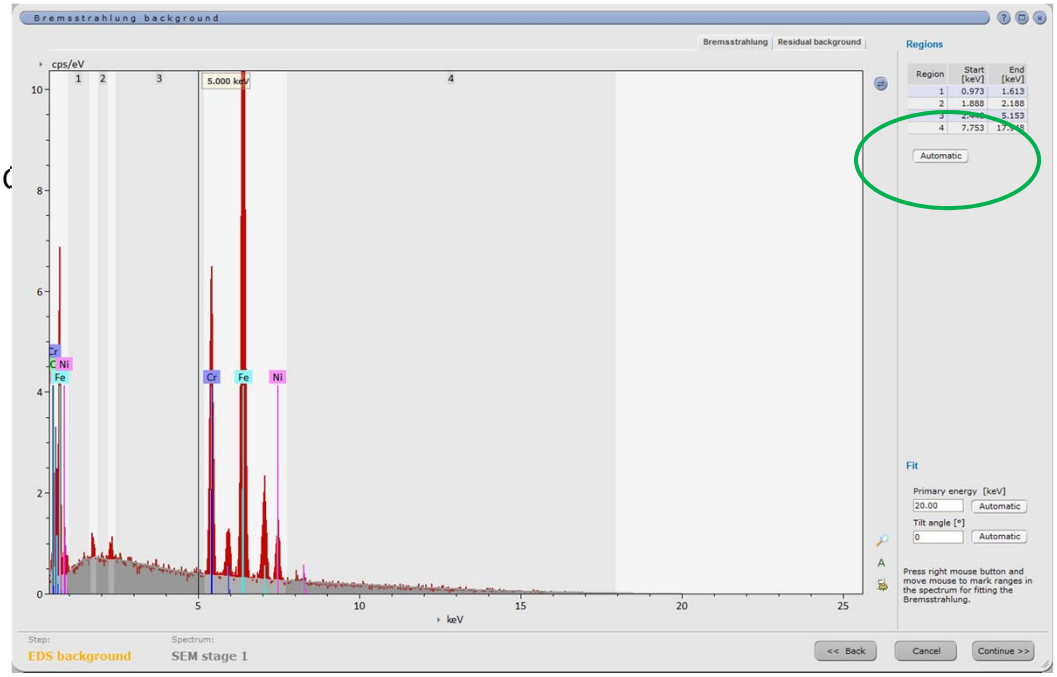
7.00

Step 3 - quantification:

To quantify the elements present in a spectra click on Quantify



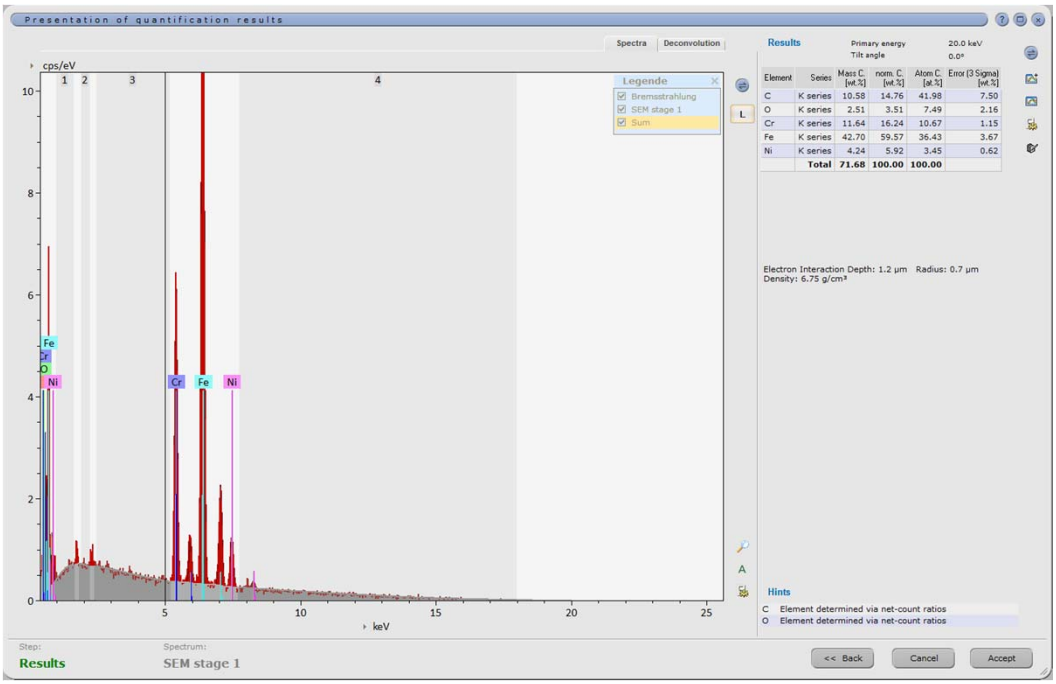
Click on Automatic



7.00

Step 3 – quantification (continued):

Note a result table has showed up in the corner:



Click on 

Results Primary energy 20.0 keV; Tilt angle 0.0°

Element	Series	Mass C. [wt.%]	norm. C. [wt.%]	Atom C. [at.%]	Error (3 Sigma) [wt.%]
C	K series	10.58	14.76	41.98	7.50
O	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 µm Radius: 0.7 µm
Density: 6.75 g/cm³

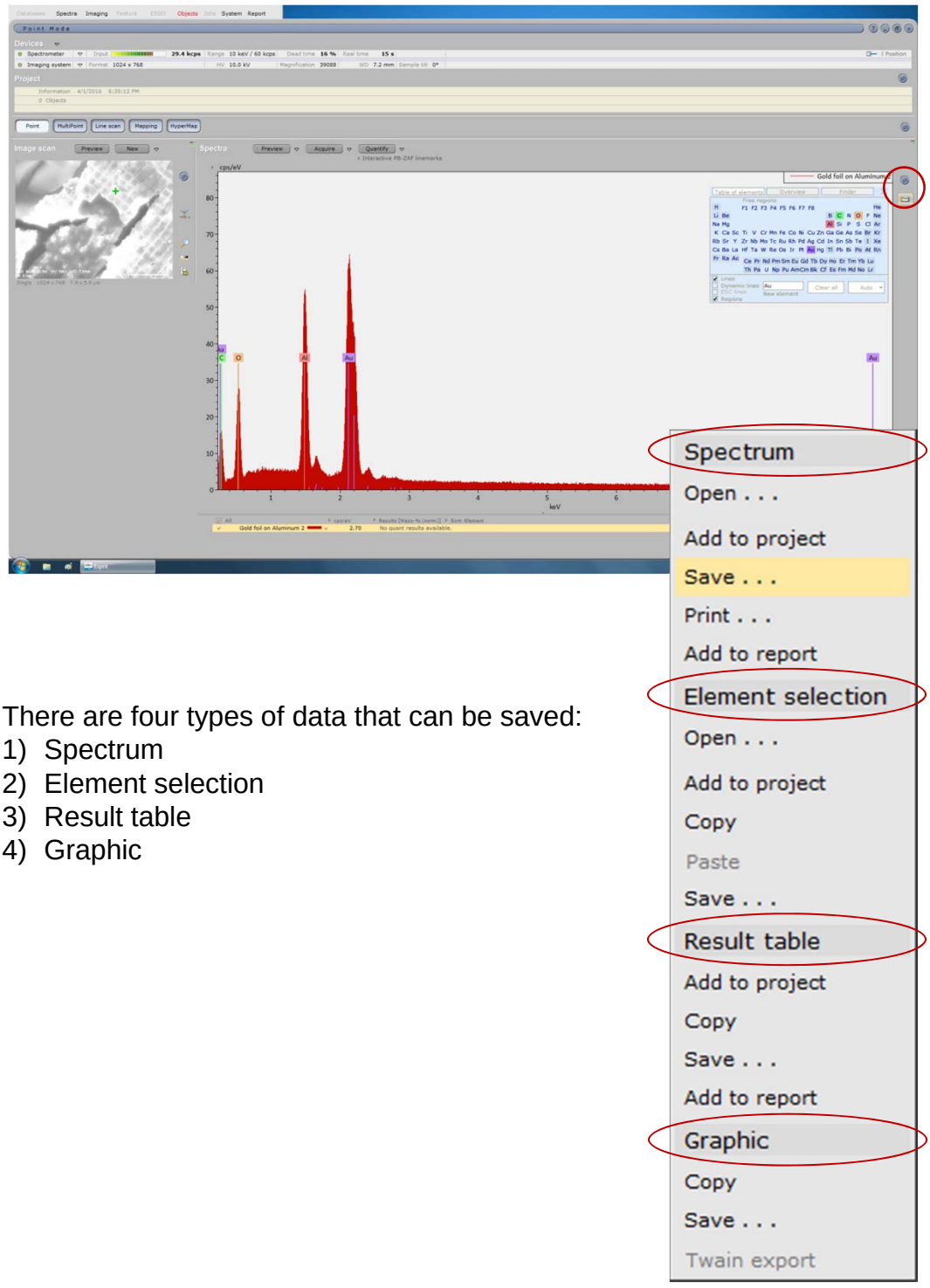
The software displays the results in several ways.

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

7.00

Step 4 – saving data:

Click on the icon as shown.



There are four types of data that can be saved:

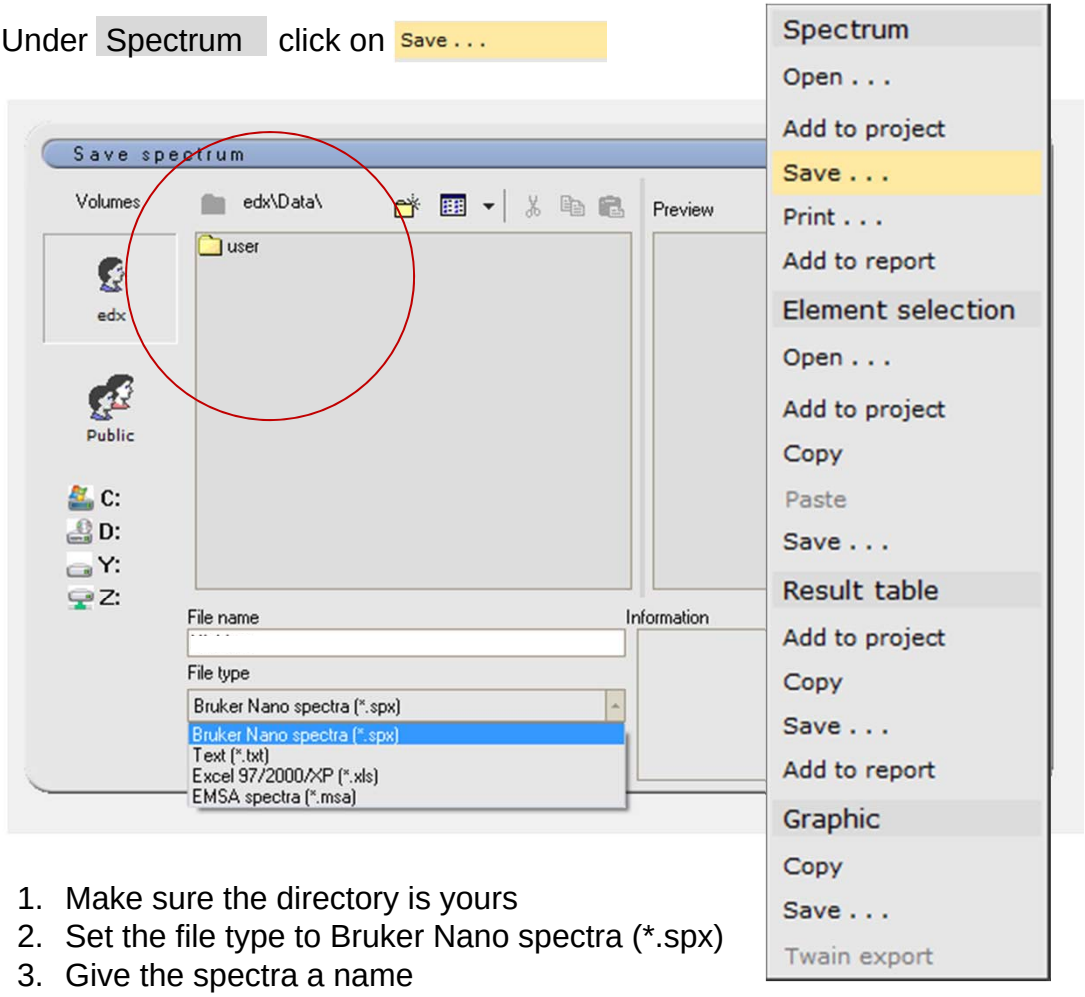
1) Spectrum
2) Element selection
3) Result table
4) Graphic

7.00

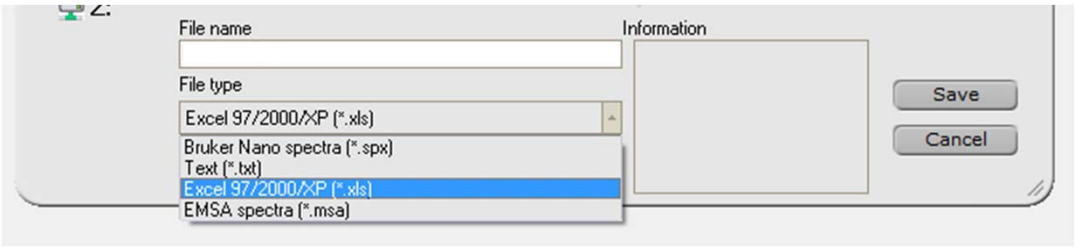
Step 4 – saving the data:

Saving the spectra:

Under Spectrum click on Save . . .



1. Make sure the directory is yours
2. Set the file type to Bruker Nano spectra (*.spx)
3. Give the spectra a name
4. Click on Save



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

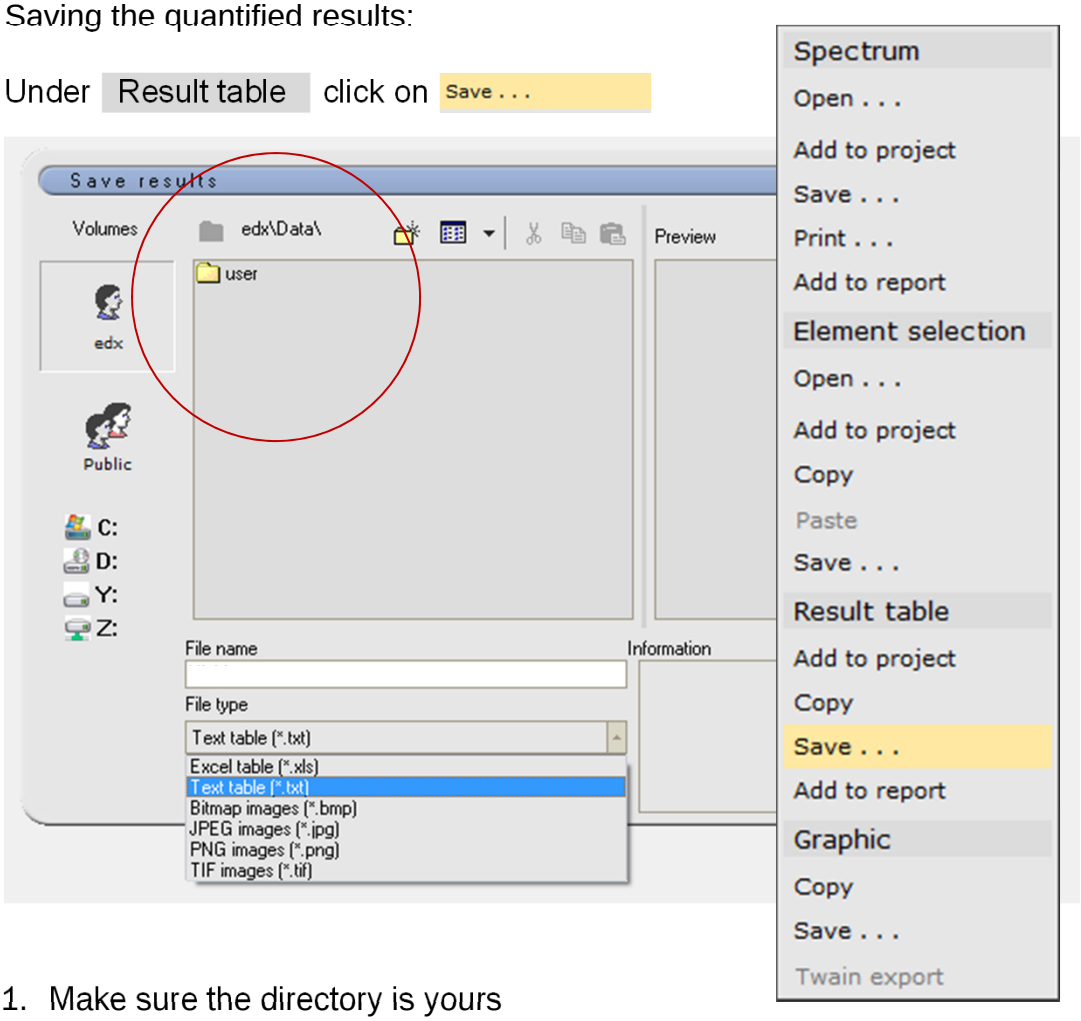
Point Mode

7.00

Step 4 – saving the data:

Saving the quantified results:

Under Result table click on Save . . .



1. Make sure the directory is yours
2. Set to Text table (*.txt)
3. The same file name should populate the box with a .txt extension
4. Click on Save

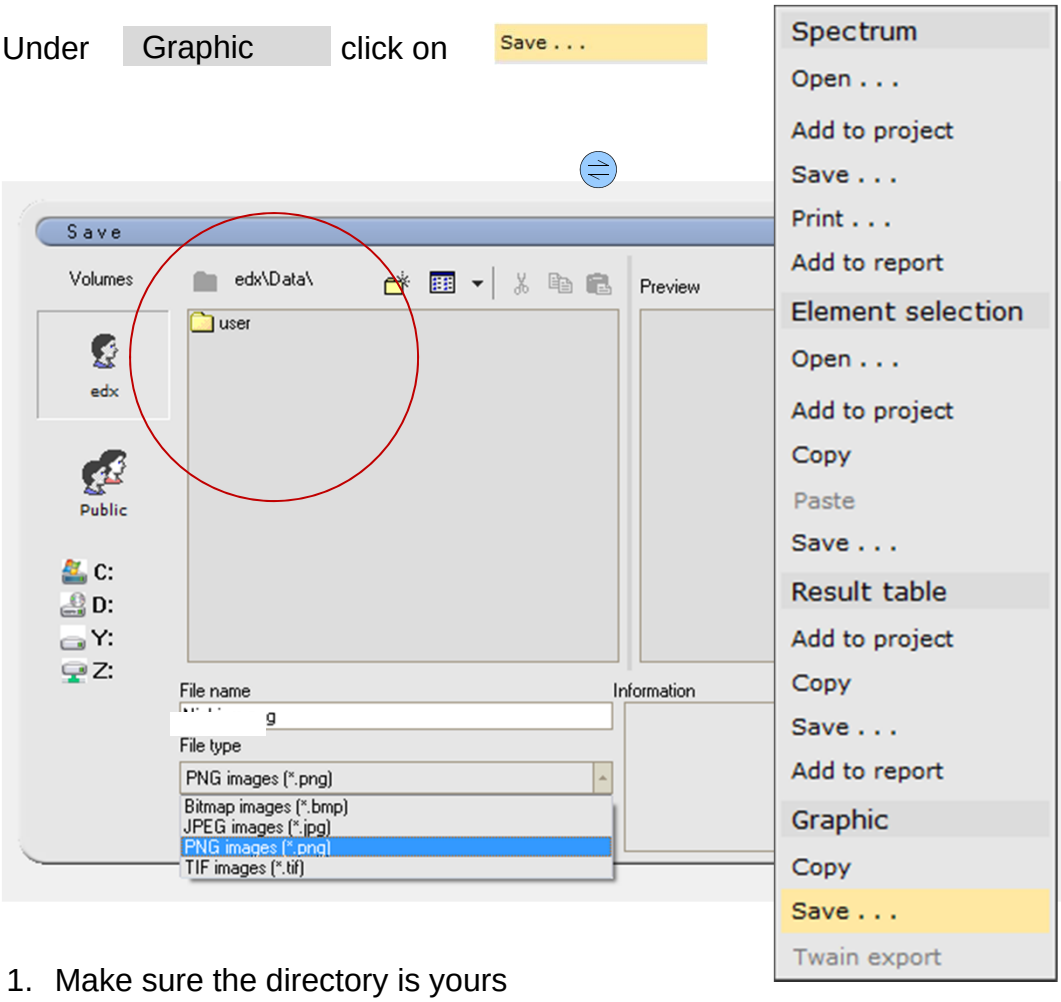
Point Mode

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!!

Under Graphic click on Save . . .



1. Make sure the directory is yours
2. Set to the file type you want (e.g. .png)
3. Type in the same filename as for your data files
4. Click on Save

Point Mode