

Bruker M4 Tornado Plus

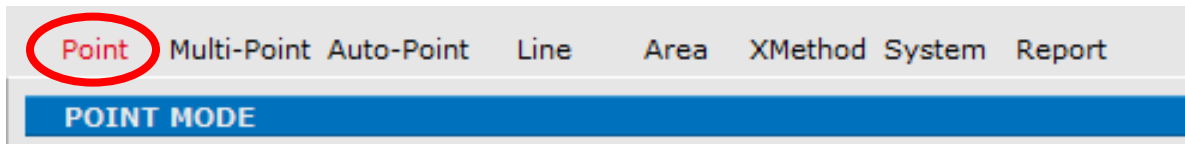
The Bruker M4 Tornado Plus XRF system utilizes polychromatic x-rays from one of two sources to excite fluorescent x-rays from the surface of the sample. Measuring the energy of the fluorescent x-rays using a silicon drift detector will allow the user to identify the elemental composition of the sample up to the information depth.

This XRF system differs from traditional energy dispersive XRF systems because the optics allow for an x-ray spot size of 20 μm . Focusing this x-ray spot on the surface of the sample and rastering the sample underneath the beam, the elemental composition can be mapped.

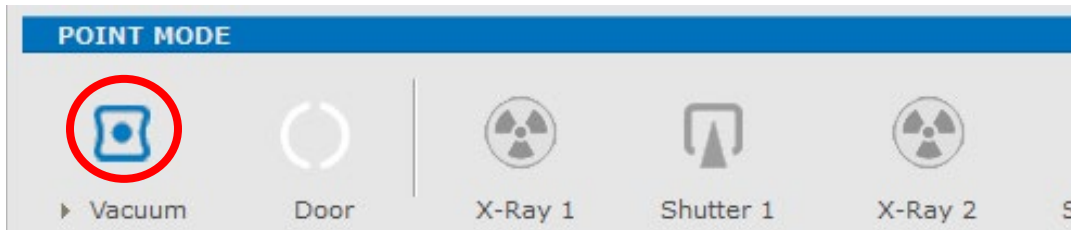
SOP:

Open the M4 Tornado software. You will be prompted to enter a password. The username and password are both xrf.

To load and unload samples, you must be in point mode. If the system is not already, click on Point to enter point mode.



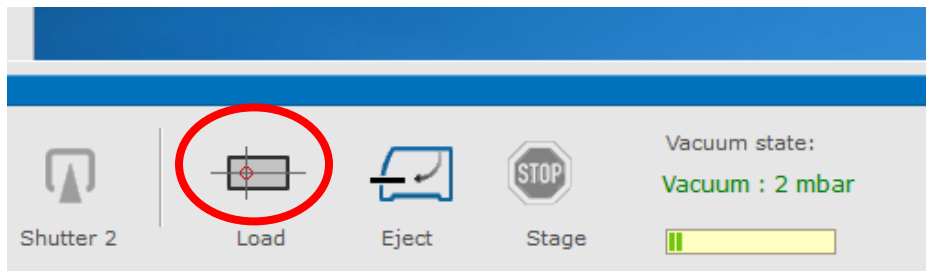
Once the software has opened, you need to vent the system. This is done by clicking the Vacuum button at the top.



When the vacuum state reads 'At Air', you can open the door to load your sample. To load your sample, click on the Eject button. The door will open and the stage will come toward the front.

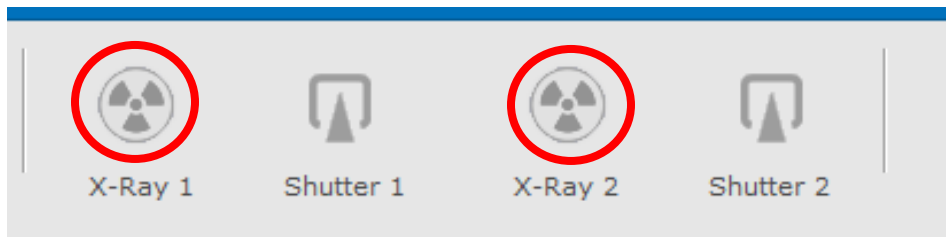



Once you have loaded your samples, click the Load button. This will close the door and center the stage.

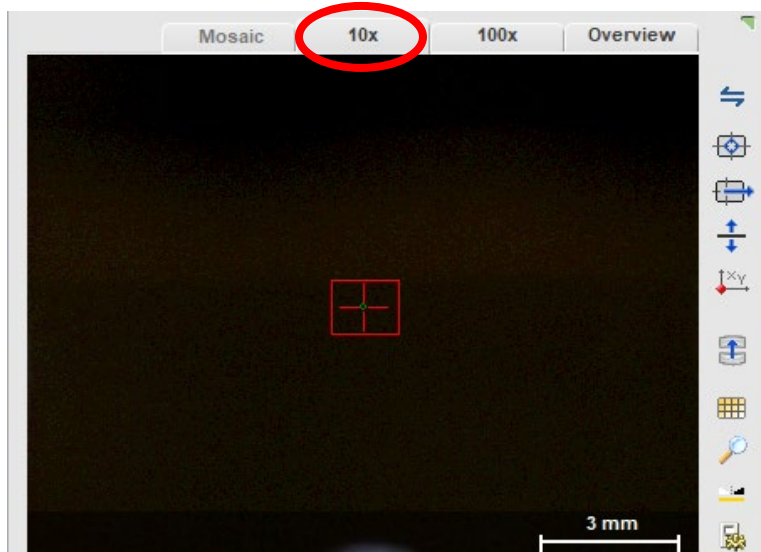


If your sample is not a liquid, click on the Vacuum button to allow the system to pump down. Once the vacuum state reads 2 mbar, the system is ready to use.



Turn on the x-ray source by clicking on either X-ray 1 or X-ray 2 and allowing the tube to fully power on.



Ensure you are looking at the 10x magnification on the sample image. Move the stage up by clicking on the  button and then clicking and holding the cursor in the top half of the image. The larger the arrow, the faster the stage will move. Ensure the sample is not going to crash into the crash guard inside of the system by watching the Overview tab. If you crash the sample into the crash guard, the circle next to stage will turn red and you will need to reset the stage. To reset the stage, go to the Item Descriptions section and look how to do that.



Once you get the sample in focus at 10x, move to the 100x magnification and ensure the sample is in focus.

Move to an area of interest using either  button or  button.

For point mode:

Click on the x-ray tube you want to use for data collection by clicking on the x-ray tube that corresponds to the specific source. Also, select the detector you you want to use for data collection. If the line is dark, the x-ray tube/detector is selected.

| Devices | | | |
|----------------------------------|--------------|---------------------------------------|-----------------------|
| <input checked="" type="radio"/> | Spectrometer | Input | 0.0 kcps |
| <input checked="" type="radio"/> | Spectrometer | Input | 0.0 kcps |
| <input checked="" type="radio"/> | X-Ray tube 1 | Rh / Empty | Actual 0 kV 0 μ A |
| <input type="radio"/> | X-Ray tube 2 | W / Empty / 4500 | Actual 0 kV 0 μ A |
| <input type="radio"/> | Stage | X: 145.00 mm Y: 160.00 mm Z: 10.00 mm | |

If you need to configure the x-ray tube for your experiment (reducing the current, adding a filter, and/or changing the collimator size), this can be done by clicking on the down arrow next to the x-ray tube of interest and selecting the options in the menu.

CONFIGURATION X-RAY TUBE

X-Ray tube

Rh Anode material
30 W Maximum power
Poly capillary lens Optics

50 kV Tube voltage
500 μ A Tube current
25 W Tube power

Standby after 30 min

Filter

- No filter
- Al 12.5 μ m
- Al 25 μ m
- Al 100 μ m
- Al 640 μ m
- Al 100 μ m Ti 100 μ m Cu 25 μ m
- Al 100 μ m Cu 68 μ m Zr 15 μ m Mo 50 μ m
- Aperture_1000
- Aperture_500

Ok Cancel

Click on the down arrow beside the Acquire button and change the settings to your desired preferences. This includes setting the analysis time, the x-ray spot size, the number of cycles, and the name of the spectra. Click OK

Acquire

Measuring time

Automatic Precise

Manual

Real time [s] 600

Live time [s] 60

Counts 4

Region start [keV] 0.20

Region end [keV] 50.00

Cyclic acquisition

Activate

Count 1

Pause [s] 0

SaveTo project

Spot size

20 µm 180 µm

Automatic analysis

None

Continuous

After acquisition

Video image

Automatically save camera images

Low res Camera

High res Camera

Spectrum name

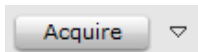
316L_0


Spectrum number: 3

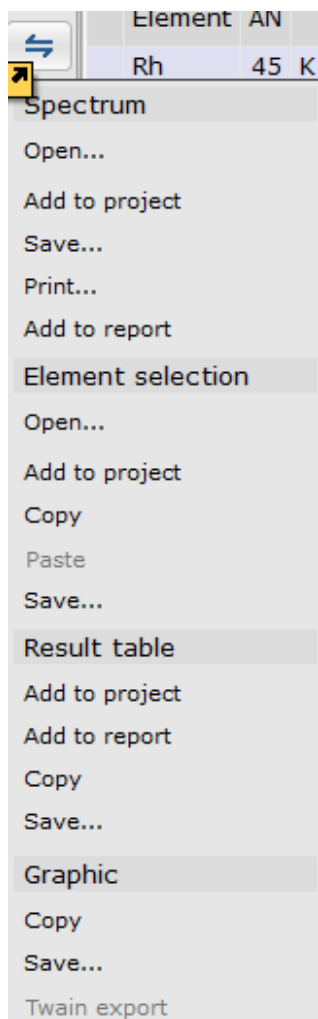
Automatic numbering

OK


Once the options are set, click Acquire.



Save the spectra by clicking on the  button and then saving whatever portion of the data is of interest. By saving the spectra, you can choose to save it in excel format, text format (*.txt), or the Bruker format (*.spx) the last of which will allow you to reopen your data for further analysis. You are also able to save the element selection, the results table from the quantification, and an image of the spectrum.





Item descriptions:


Devices:  This is the list of detectors (called spectrometers) and x-ray tubes that can be used in the system. This section will show the current counts and dead time for the detectors, the current current, voltage, filter, and collimator size for the x-ray tubes, the current stage position, and allow you to change the options for each in the list.


Point mode: This is a mode that allows you to collect fluorescence data from a single point. If using the Rh source, you can select this point diameter to be 20 um or 180 um. If using the W source, you can select a collimator to give a spot size of either 500 um, 1000 um, 2000 um, or 4500 um.

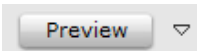
Filter: This allows you to select a filter to be placed between the x-ray source and the sample. Doing this will reduce the overall intensity of the incident x-rays by attenuation. This is useful when attempting to collect/analyze a fluorescence peak whose signal would be blocked/interfered by incident fluorescent peaks. Additionally, this can help to attenuate diffraction peaks which may show up in crystalline samples.

Focus at position:  This button allows you to move the stage in x and y by left clicking once on the area of interest and that becomes the new center position. This button does not move the sample in z.

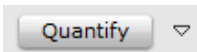
Move stage into direction of cursor:  This button moves the stage in x and y by clicking and holding in the direction you want the sample to move. Placing the cursor further from the center allows the stage to move more quickly.


Move stage into Z-direction:  This button moves the stage in z by clicking and holding the direction you want the sample to move: clicking and holding in the top half of the image causes the stage to move up and clicking and holding in the bottom half of the image causes the stage to move down. The further the cursor is from the center line, the faster the stage moves.


Create/configure mosaic image:  This button allows you to create a mosaic of images that will be stored in the mosaic tab. Assuming your sample is larger than what can be displayed in the window, this is a good way to get an overview of your sample.

Preview:  This button opens the shutter to the x-ray and starts to collect a spectrum. This allows the user to ensure the settings have been correctly set before acquiring a spectrum. This button will not allow the user to save the spectrum but does allow for the assurance of the appropriate dwell time and x-ray current.

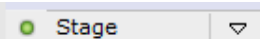
Acquire:  This button acquires a fluorescence spectrum for the sample.

Quantify:  This button will quantify the elements that have been selected from the periodic table using the method selected underneath this button (shown here: automatic).

Autoscale X- and Y-axis once:  This button will autoscale the spectrum to allow the user to see all the data in both the x and y directions.

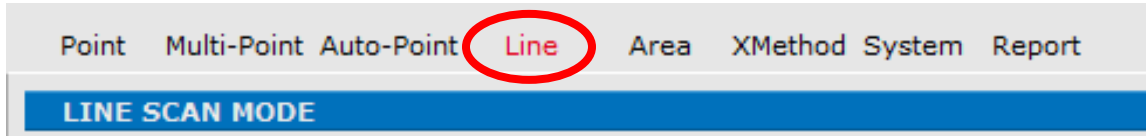
Spectrum display settings:  This allows the user to determine the y-scaling factor for the spectrum.

If you crashed your sample into the crash guard, the stage will stop moving and the circle next to stage will turn red. To reset, click on the down arrow next to stage and click on the option to reference all.



For line scan:

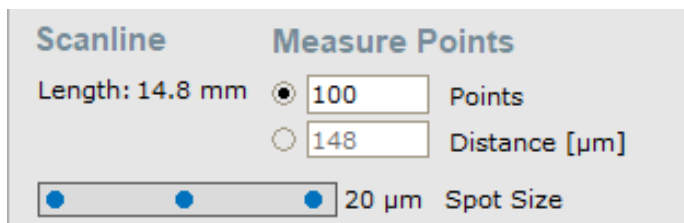
Click on the button labeled Line to bring up the line scan mode. In this mode, the stage will be rastered in a line under the x-ray spot and spectra will be collected at specified intervals. Doing so will allow you to collect spectra at each point and to be able to measure the elemental concentrations along the profile of the line.



Instead of seeing a point in the 10x and 100x magnification, a line will appear with an arrow at one end. This will dictate the direction the stage will move and over what parts of the sample spectra will be collected. To change the size and/or direction of the line, click on the line and a square box will appear at each end. Clicking and dragging these boxes will allow you to change the size and direction of the line. This can be done in the mosaic, the 10x, or the 100x image. Whichever tab is selected when you press acquire will be the line that is drawn on your sample.



To determine the spacing of data collection along the line, go to the dialog box underneath and either select the number of points or the distance between the points. The scanline will tell you how long the line you've drawn is. The image under the dialog boxes shows you the spacing of the spots and the relative x-ray spot size for reference. Once you have changed a number, make sure to hit enter so that it takes.



You will click on the down arrow beside the Acquire button. The settings dialog box will show up and allow you to change the settings of the acquisition. In this settings dialog box, you will see Time/Pixel [ms] which is the amount of time each spot will spend under the x-ray source and how long it will take to collect a spectrum at that spot, Stop Measurement where you will have the option to choose to stop the experiment manually (by clicking the Stop button) or the number of cycles you want this to run, the Measure time which gives an estimate of how long the experiment should run, Fast quantification which allows the system to quantify the data as it goes, and the ability to set the name of the scan.

Measure time

Time/Pixel [ms] 100.00

Stop Measurement 1.5 mm/s

Manually Cycles

10

Measure time: 2:26 min

Quantification

Fast quantification

Scan name

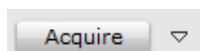
Sperlonga_C2_lineScan_Clast

Image number: 1

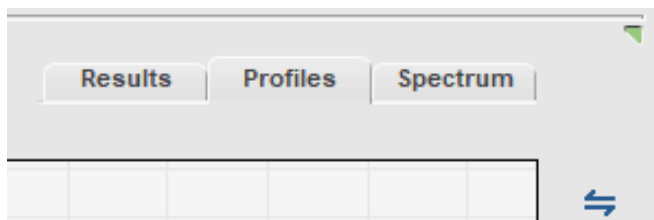
Automatic numbering

OK

Once you are satisfied, click OK and then on Acquire to acquire spectra.



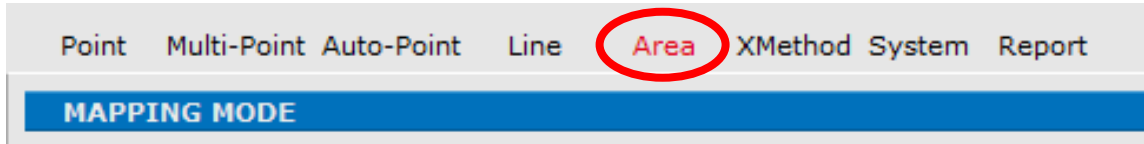
In line scan mode, you have the option of either viewing the spectra or looking at the profiles. These two views are accessible by clicking on the tabs. The Spectrum tab will show you the composite spectrum from the entire line by averaging each point together. The profiles tab will show you relative (or absolute) quantities of elements that are selected in the periodic table as a function of distance.



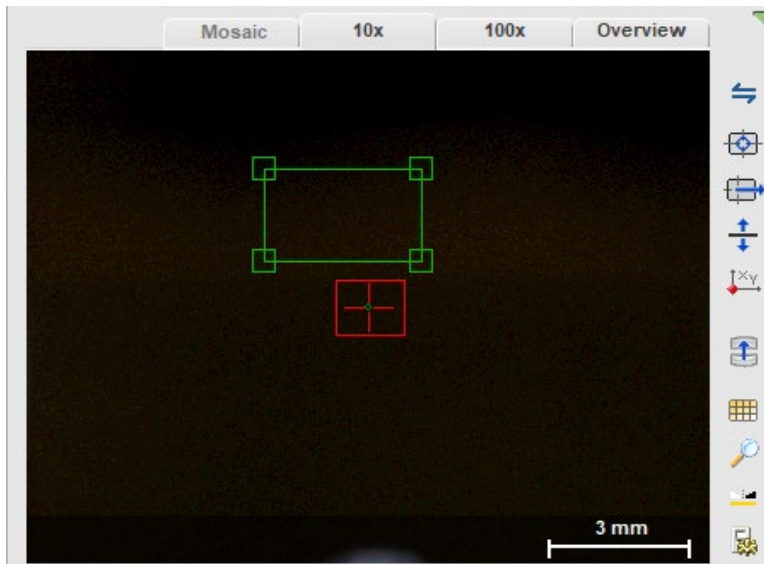
As with point mode, you can autoscale the profile and spectrum by selecting the **A** button. You can save your data by clicking on the **↔** button. You will be able to save the spectra as a Bruker file (*.spx) or a text file (*.txt).

For area scan:

Click on the button labeled Area to bring up the area scan mode. In this mode, the stage will be rastered in a Z pattern under the x-ray spot and the spectra will be collected at specified intervals. Doing so will allow you to collect spectra at each point and to be able to measure the elemental concentrations spatially.



In the 10x and 100x magnification windows, a box will appear. This box dictates the area that will be scanned on the sample and from which the elemental concentrations will be measured. To change the size of the box, click inside of the box and smaller boxes at each corner of the rectangle will appear. By clicking and dragging these smaller boxes, you can change the size of the scan area. This can be done on the mosaic, the 10x, or the 100x window. Whichever tab is selected when you press acquire will be the area over which data will be collected.



To determine the spacing of data collection inside of the area, go to the dialog box underneath and either select the number of points in x and y or the distance between the points. The analytical area will display the x and y coordinates of the top left corner (defined as top and left) and the width and height of the box in mm. The image under the dialog boxes shows you the spacing of the spots and the relative x-ray spot size for reference. Once you've changed a number, make sure to hit enter so that it takes. The image filter is going to be an algorithm that smooths the video image. The element filter gives options for the data processing by improving the element distribution display. To improve the signal to noise, increase the binning number next to Average and select that option. QMap resolution defines the number of pixels to average together for the quantification of the map. The processing time shows the estimated time to do the QMap.

| | | | | |
|--|-----------------------------------|---------------------------------------|---------------------------------|--|
| Analytic Area | | Measure Points | | |
| Left [mm]: | <input type="text" value="5.17"/> | <input type="radio"/> | <input type="text" value="84"/> | x |
| Top [mm]: | <input type="text" value="2.56"/> | <input type="radio"/> | <input type="text" value="48"/> | y |
| Width [mm]: | <input type="text" value="3.35"/> | <input checked="" type="radio"/> | <input type="text" value="40"/> | µm distance |
| Height [mm]: | <input type="text" value="1.93"/> | | | |
| <input checked="" type="radio"/> <input type="radio"/> <input type="radio"/> <input type="radio"/> <input type="radio"/> <input type="radio"/> <input type="radio"/> <input type="radio"/> | | 20 µm | Spot size | |
| Image filter | | Element filter | | |
| <input checked="" type="radio"/> None | <input type="radio"/> Smooth | <input checked="" type="radio"/> None | <input type="radio"/> Average | <input type="text" value="3"/> <input type="button" value="▲"/> <input type="button" value="▼"/> |
| <input type="radio"/> Sharpen | | <input type="radio"/> Smooth | | |
| | | <input type="radio"/> Automatic | | |
| | | | | |
| QMap resolution | | | | |
| <input checked="" type="radio"/> 1x1 | <input type="radio"/> 5x5 | <input type="radio"/> manual | <input type="text" value="3"/> | |
| <input type="radio"/> 3x3 | <input type="radio"/> 9x9 | | | |
| Processing time: | 20 min | | | |

If the Acquire button is grayed out, you will need to select a new project. To do this, you will click the New button shown below.



With the Acquire button available, you will click on the down arrow to the right of it and set the options for the acquisition. The Time/Pixel is the amount of time the system will spend at each spot collecting data. The Stagemode should always remain on Normal. Stop measurement can be set to stop the measurement manually or by collecting data over a number of cycles. Under this, the estimated time to collect this map will be shown. The speed (shown in the image below as 997 µm/s) should remain above 800 µm/s. If you need to dwell longer at a spot, increase the number of cycles. You will also type in the Map name which will name the area scan.

Measure Time

Time/Pixel [ms]

Stagemode 997 $\mu\text{m/s}$

Normal Serpentine

Stop Measurement

Manually Cycles

Measure time: 4:06 min

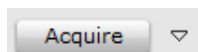
Map name

Map number

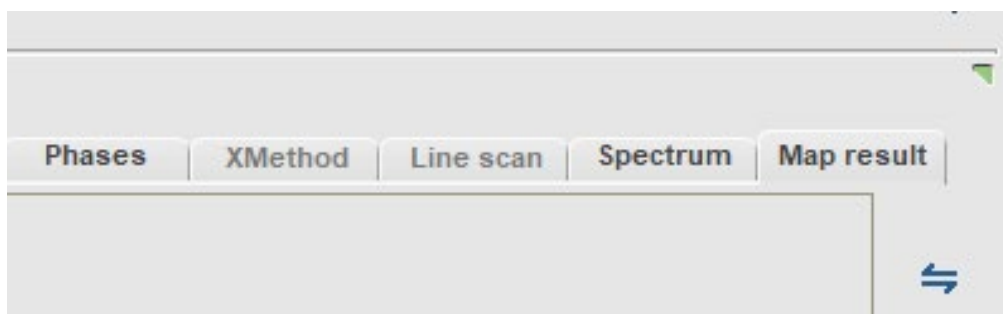
Automatic numbering



Object-Name






Once you are satisfied, click OK and then on Acquire to acquire spectra.

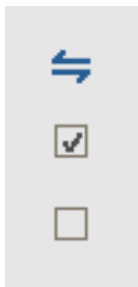



In the area mode, you have the option of either viewing the average spectra or looking at the map result. Clicking on the Spectrum tab will show you the average spectrum of all of the points that were collected in the map image. Clicking on the Map Result tab will show you the optical image of your sample and will show the elemental distribution of elements in your sample based on the elements that you selected.




In the Map Result tab, you will be able to select which elements you want displayed in the map and used to quantify the data by clicking on  button. The  button creates a maximum pixel spectrum

which calculates an artificial spectrum that shows the maximum content of every channel in the map which is helpful to identify hot spots. You can click on the  button which will show you a heat map of the elemental distribution. In this menu, you will be able to change the color display for how the elements appear on the map by showing where the elements are most intense in a given region. You can click on the  button which will show a histogram of intensities of the elements based on a binary or ternary diagram. Clicking on the  button will allow you to define an object for further analysis. Right clicking on this button will allow you to determine whether you want to draw a point, a line, a rectangle, a circle, or a polygon on the map. Doing so will take the spectra inside of that object and average them together to create a spectra of the given object. You can do area determinations, quantifications of the elements inside of those areas, and pull the averaged spectra from that region with this function. The  button allows you to write comments in the map, take measurements, and insert various markings to draw attention to certain features. The  button will change the display options for the map. In this menu, you can change the mix method, the scaling, the way the results are displayed, and legend options. Toward the bottom of the toolbar in the Map Result window are the buttons as shown below. These allow you to select all or deselect all the elemental distribution maps and add them or remove them from the composite map shown. The first button is used to save the elemental distribution images individually.



You can save your data by clicking on the  button. You will be able to save entire dataset by clicking on Save the Database. This will save every spectra at every point and allow you to open this file back up to do further analysis, if desired. Saving the database will save the file as a *.bcf file. You can also save the map data which will save images of the elemental distribution of each element, the elements you selected, and/or the table of results that were obtained through Quantify.

Export image 

- Database**
 - Open...
 - Save...
 - Save selection. . .
 - Save all objects. . .
- Map data**
 - Add to project
 - Insert
 - Copy
 - Save...
 - Print...
 - Add to report
 - Twain export
- Element selection**
 - Open...
 - Add to project
 - Copy
 - Paste
 - Save...
- Result table**
 - Add to project
 - Copy
 - Save...
 - Add to report