2020/3/4 Update

Energy Dispersive X-ray Spectrometer(EDS) Basic Manual

Laboratory of XPS analysis

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Caution

Please keep the following rules.

- Take off your shoes, and change into slippers.
- No food or drink. Clean up the garbage.
- Contact with staff immediately when you encounter apparatus trouble.
- Don’t use the apparatus roughly.
- Don’t take out tools of this laboratory.
- Keep valuables yourself. Lock a door of this laboratory when you leave here.
- Keep the limit of the stage transfer. The stage will hit the detector if you move the stage beyond the limit.
- Return to original setting after using if you change parameters of software and hardware.
- Don’t connect your USB memory to SEM PC. Use the USB of this laboratory, and take out the data via the PC for analysis.
- Don’t touch with your hand anything which put in SEM chamber. Clean the tools if you make it dirty.
- Reserve the apparatus yourself before using, and use the apparatus on time. Changes to reservations must be made the day before. Change of reservation on the day is invalid.
- Respond the all troubles by your laboratory if you use at night, early morning, holiday. Students must let your supervisor know. Emergency contacts are mentioned beside the entrance.
- If the first time using, make contact with the staff before using and reserve the training.
- Don’t put fragile sample, too big sample, gas exhaust sample into the SEM chamber without permission.

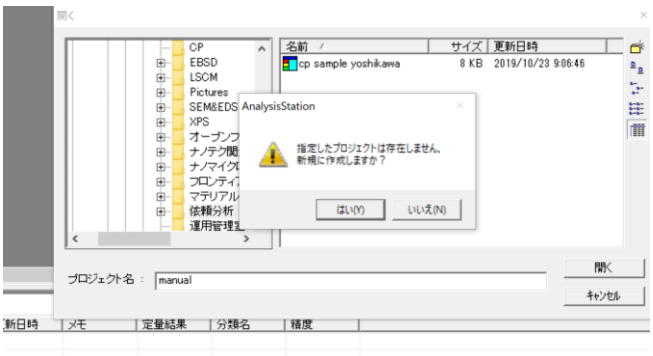
Before analysis

Check the scanning electron microscope (SEM) Basic Manual for the usage of SEM.

EDS power

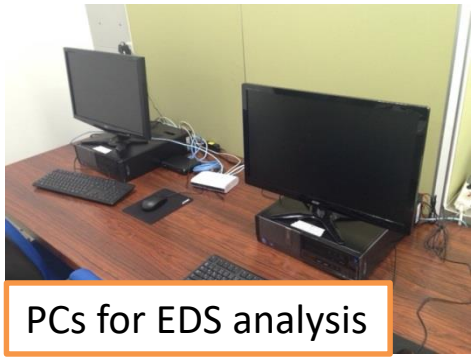
Power on the EDS 10 minutes before starting the EDS analysis software.

After 10 minutes, start EDS software “Analysis Station”.



Create a new project folder or open your existing project folder.

EDS data is saved as project folder unit. SEM images taken into EDS and analytical spectrum data linked to SEM images are saved in the project folder. You can create a new project for each sample, or you can treat some sample groups with one project. Always save the project after measurement. It is better not to change the contents of the project folder.

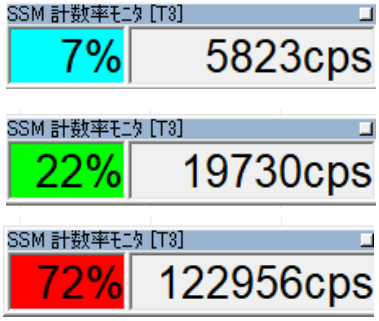


PCs for EDS analysis

EDS software “Analysis Station” has been installed in PCs for analysis. You can open the project and edit the data.

Import analysis area

SSM couting rate monitor



↑Reduce until light blue

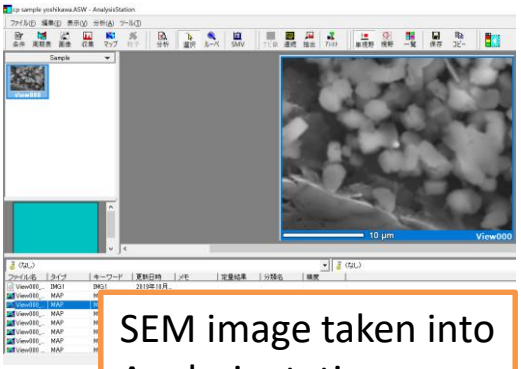
Import the area for EDS analysis into the “Analysis Station”.

Check the “SSM couting rate monitor” that appeared at the same time as the software startup. Adjust the accelerating voltage and spot size of electron beam so that the value of cps (the amount of X-rays) is increased to the required value.

In general, about 5000 cps for spectrum analysis and about 20000 cps for mapping. Acceleration voltage 20 kV and SS 65 is better for analysis. When the main composition is light elements, the acceleration voltage is better to be 10-15kV. If the value of cps is low, signal-noise ratio will be poor. In that case, it can be solved to some extent by increasing the measurement time.

Image

After adjusting the focus and contrast, click the “Image” icon to import the SEM image.

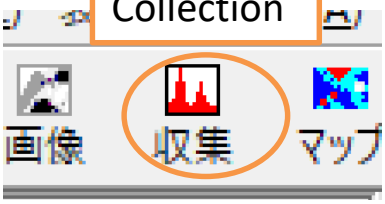


SEM image taken into Analysis station

- The value of acceleration voltage

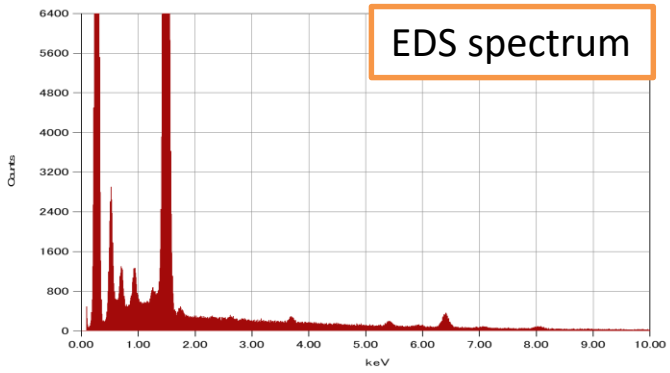
The magnitude of the acceleration voltage necessary for excitation is decided by each characteristic X-ray of each element. Please note that EDS analysis can not be done unless excited! Regarding the acceleration voltage required for excitation, check the periodic table poster behind the SEM apparatus. Usually, it is better to set the acceleration voltage to about 2-3 times the energy of the characteristic X-ray to be measured. Also, the strength of the acceleration voltage effects for the area, equivalent to the characteristic X-ray generation area, that the electron beam diffuse in the specimen. That is equivalent to the characteristic X-ray generation area. Even if you carry out point analysis, actually you measure X-rays from the point deeply and extensively (about 1 µm). For details, refer to the poster’s Castaing formula.

Spectral analysis(Collection/Qualitative)

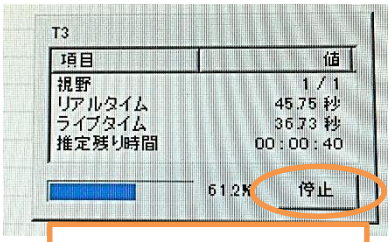


Collection

Analyze the EDS spectrum the whole of the SEM image with “Collection” icon.

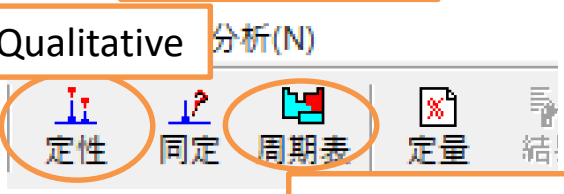


EDS spectrum



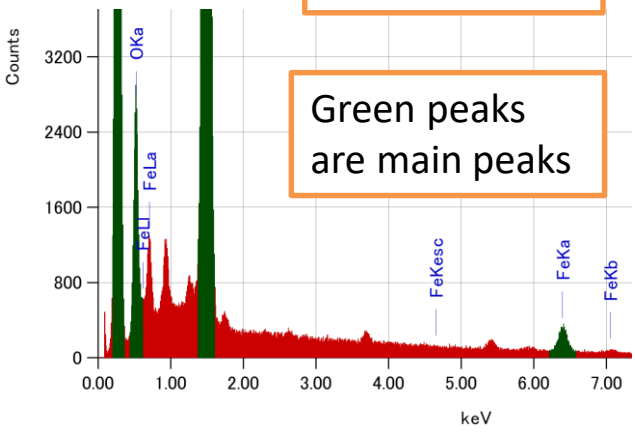
End with “Stop”

Qualitative

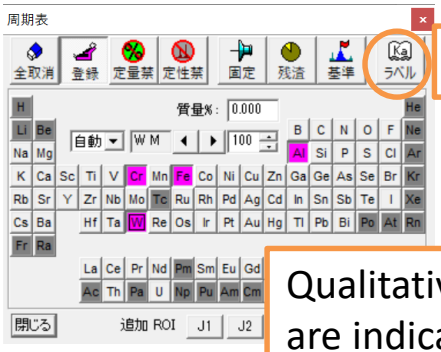


Periodic table

After acquiring the spectrum, identify the peak automatically with “Qualitative” icon in the spectrum window menu. You can check and change the qualitative elements with “Periodic table” icon.

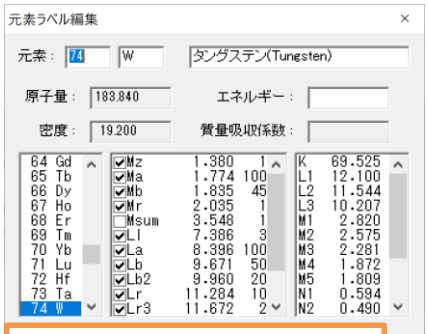


Green peaks are main peaks



Label

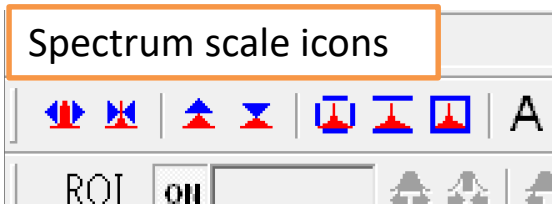
Qualitative elements are indicated in pink



Labeling edit menu

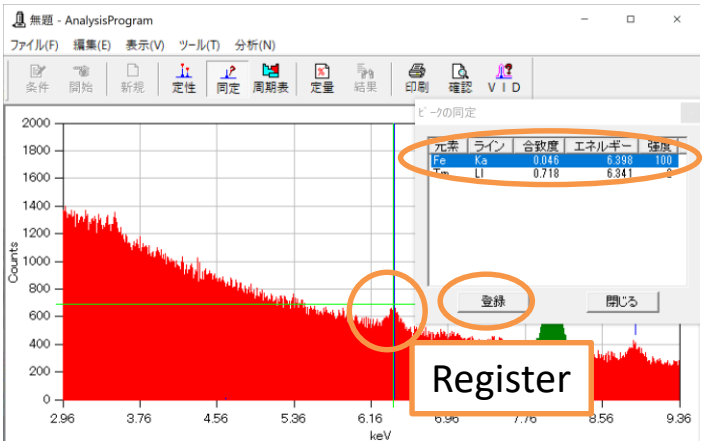
"Label" icon in the periodic table allows you to select the characteristic X-ray labeling of the selected element. All types of characteristic X-rays are not labeled and please change as needed. Especially small peaks are not labeled, so if there are unknown small peaks, you should check here.

Spectral analysis(Identification/Quantification)

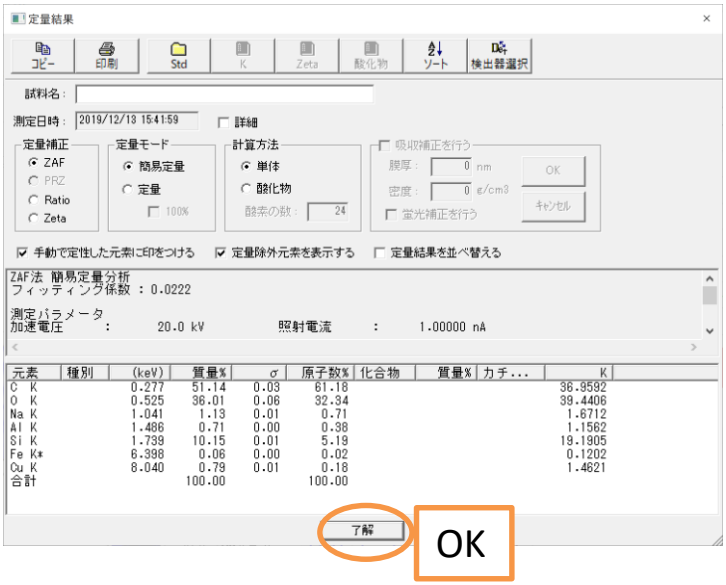


You can change the scale with the icons in the lower left of the spectrum window.

For small peaks that can not be identified by automatic qualitative, it is necessary to do by yourself.

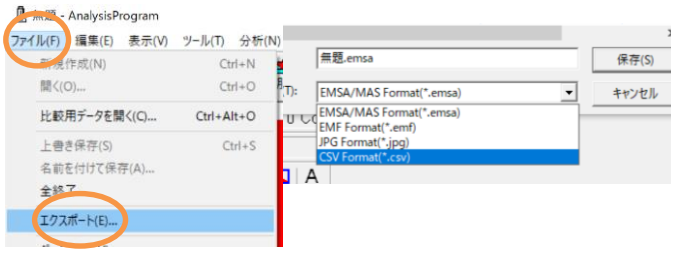


Click on the position an unknown peak in eds spectrum. Click on “Identification” icon, a characteristic X-ray suggestion is displayed for that peak. Selecting and registering the element, it is added to the result of identification.

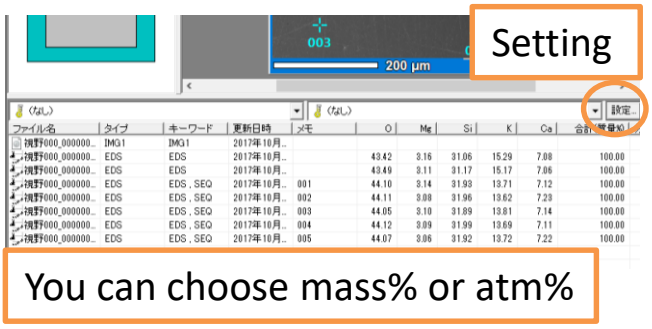


Click the "Quantitative" icon, you can see the results of the relative quantitative values of the qualitative elements. Click “OK” after confirming. You click “Yes” when close the spectrum window, the quantitative results will be registered in the spectrum data.

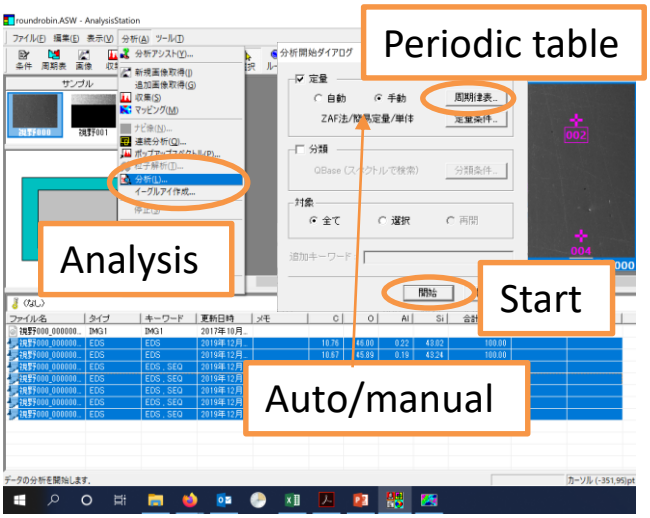
Spectral analysis(Saving/Condition)



Click “ファイル(F)”→”エクスポート(E)” in the menu in the spectrum window. spectrum graph data can be output by selecting the file type “csv”.



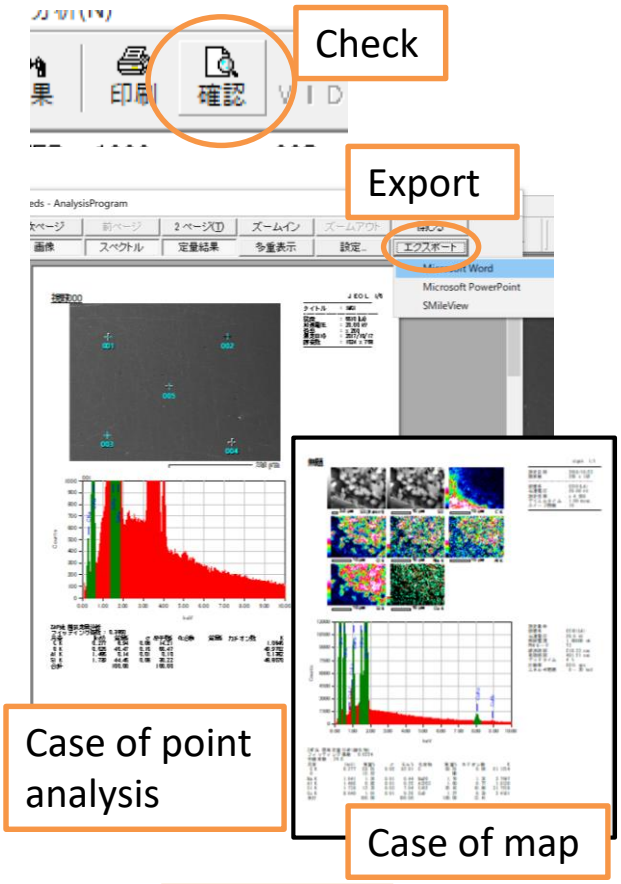
The spectrum data is linked to the captured SEM image and placed in the list below when the image was selected. Quantitative results are also listed. The display format can be changed from the “Setting” button on the list.



If you select multiple spectra data from the list and select “Analysis” from the top menu, you can quantify multiple spectra at once.

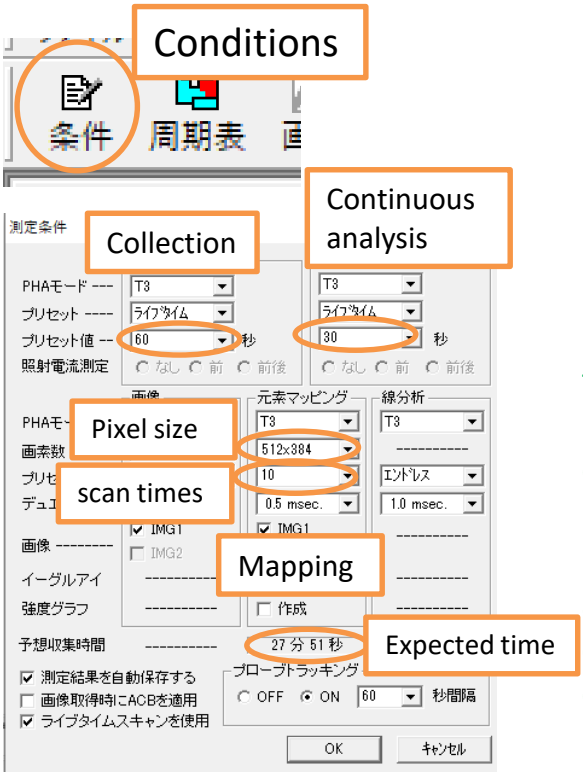
Select multiple spectra data from the list and click “Compare” in the right-click menu to display multiple spectra side by side

Spectral analysis(Saving/Condition)



Click the “Check" icon in the spectrum window menu to display a report window for SEM images, spectrum image, and quantitative results. Click "Export" in the window and selecting Word or PowerPoint, the report will be output. Multiple reports can be output at once by using “Compare” of previous page.

In the case of mapping, click “分析(N)” → “スペクトル表示(D)” from the mapping window menu to open the spectrum window. After performing quantification, click “Check” to display the mapping image, spectrum image, quantification result report.



In “Conditions” window, you can change the integration time of spectrum measurement. It is better to set a long time when the X-ray intensity is low.

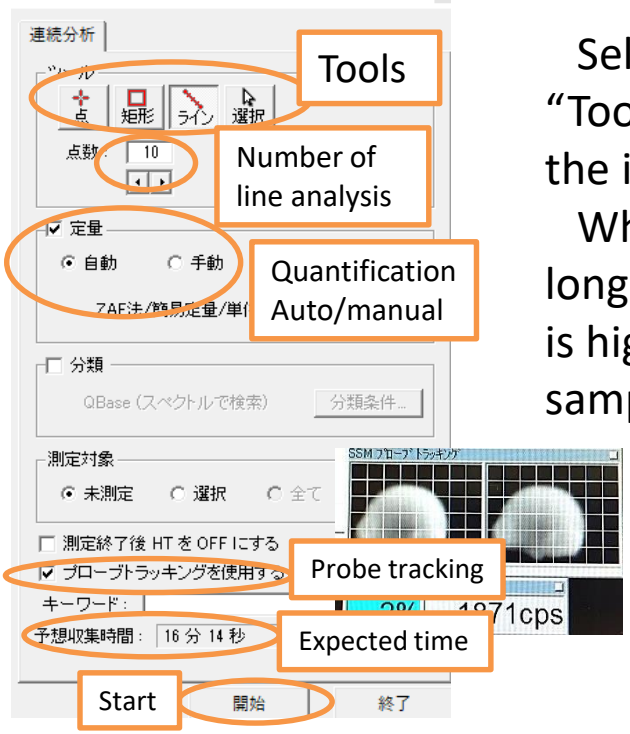
S / N ratio is improved by the route times the integration time. The S / N ratio is doubled at 4 times the integration time. However, no matter how the S / N ratio improves, the detection limit of the peak is about 0.1atm%.

In “Conditions” window, you can change integration time of continuous analysis, pixel size and scan times for elemental mapping, execution interval of probe tracking, etc. If changed, please return to the original parameters at the end.

Continuous analysis

You can carry out the point analysis, area analysis, and line analysis from “Continuous” icon.

You can not select it unless you close the spectrum window. And import SEM image first.

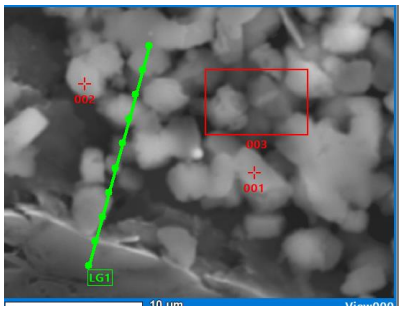


Select the analysis method from the “Tools” and set the analysis position in the imported image.

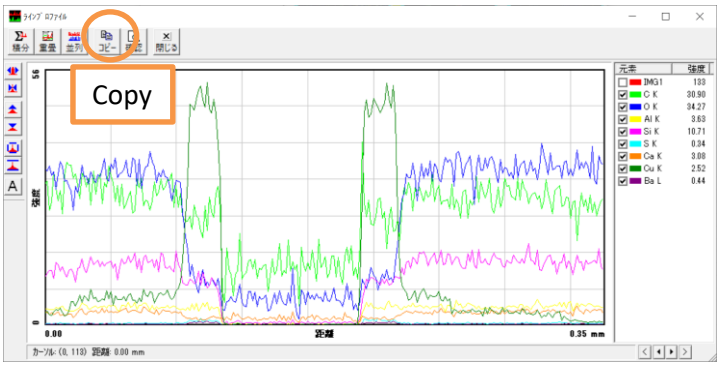
When the measurement time becomes longer or the magnification of the image is high, it is possible to prevent the sample deviation by “Probe tracking”.

“Probe tracking” correct the original analysis position by beam shift when there is a position shift compared to the original captured image. Before using, be sure to reset beam shift before capturing images.

When you check the Quantitation, Quantitation will be carried out automatically.



Click “Start” to take measurements in order.



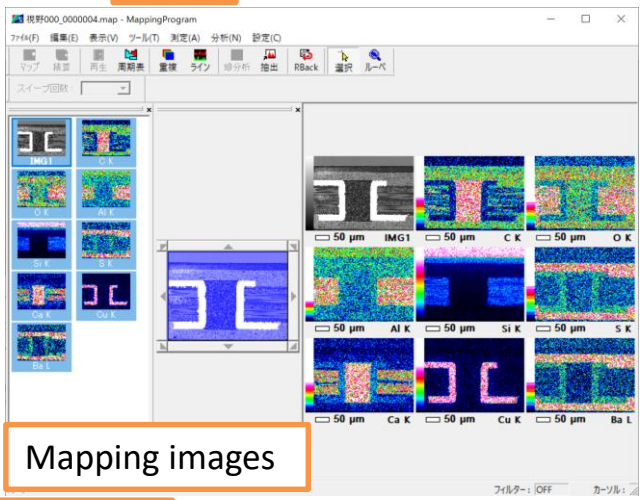
In line analysis, the horizontal axis of the profile can be drawn as distance. Results can be superimposed on SEM images. Click “Copy" to copy the line profile graph data.

Elemental mapping

Map

Click the "Map" icon to draw the intensity distribution of each element in the capture SEM image area.

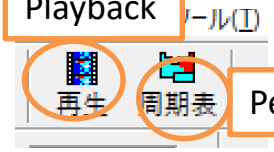
Click the “Condition” icon and set the number of mapping scan times and resolution in advance.



Mapping images

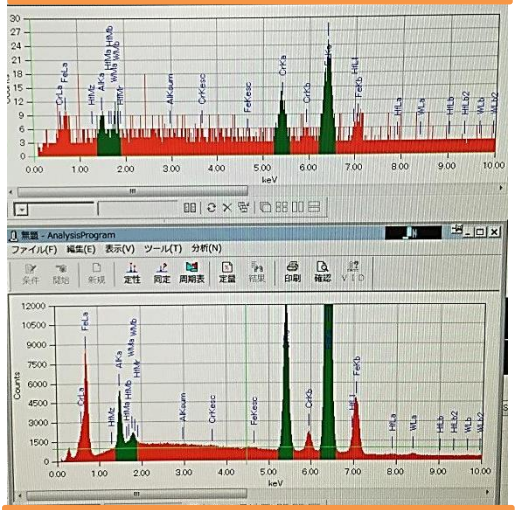
When start mapping, the elements that have been registered in the periodic table are mapped. If you want to add a mapping element, register it in the periodic table (make it pink) and click the "Playback" icon

Playback



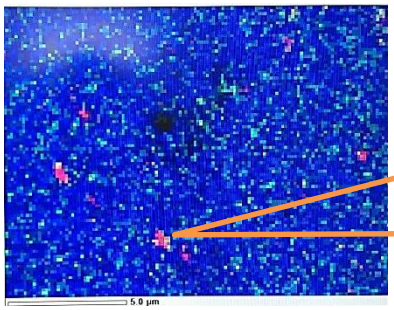
Periodic table

Eagle eye spectrum
(spectrum with smaller count)



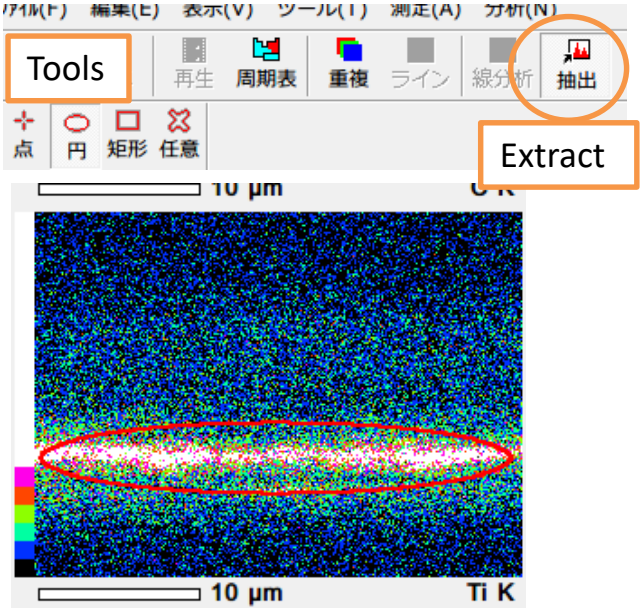
Spectrum of entire SEM image

Two spectrum windows appear at the same time as the mapping. One is the spectrum of the entire SEM image (same as “Collection"). The another is called "Eagle Eye Spectrum", which is a spectrum that shows the superimposition of spectra for each pixel of the image. In the Eagle Eye spectrum, peaks can be found even for element that exist only in a small area. Can be used to find trace element.



Elements that exist only in part of the analysis area may not show peaks in the spectrum of the entire SEM image.

Elemental mapping(extract/line display)



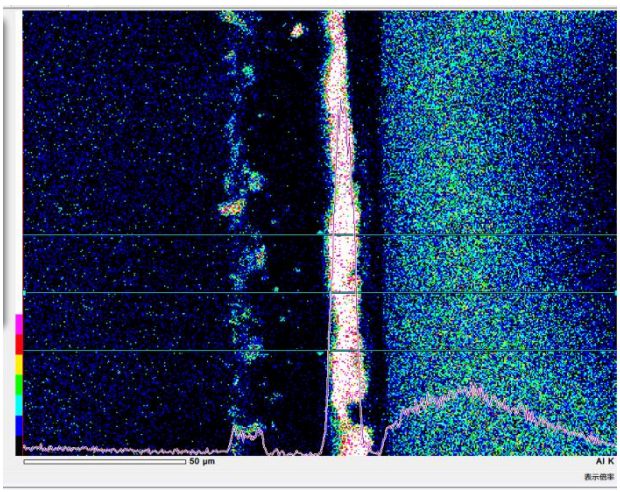
In mapping window, you can partially extract data and generate line profile from mapping measurement data.

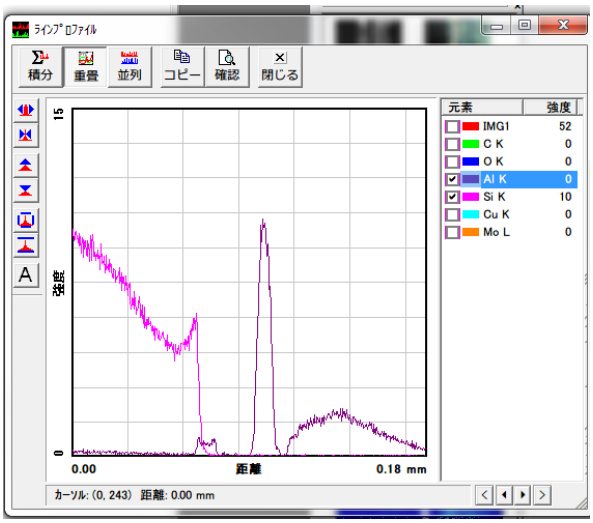
- Extract

Selecting a tool and specifying its position on the SEM or mapping image, create a spectrum in that area.

- Line display

Click the “Line display” icon to display the line profile of each element. The position and width of the line can be adjusted freely.



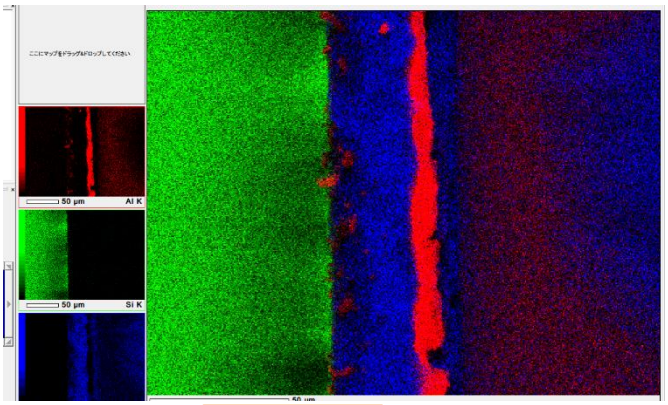


Elemental mapping(overlap/quantitative map)

- Overlap

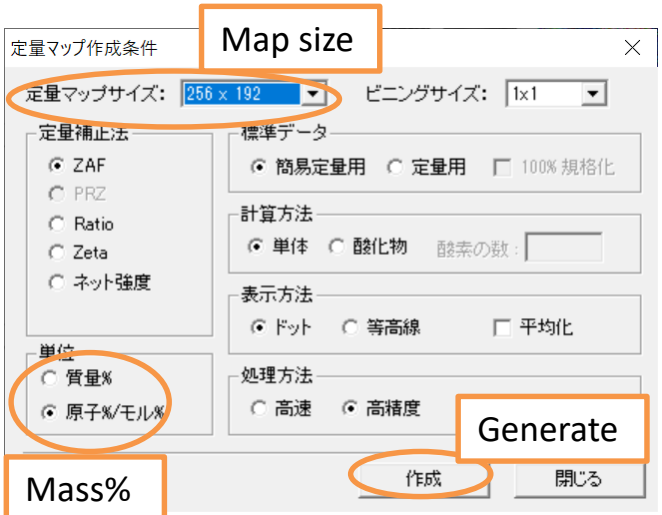
The mapping images of each element can be overlapped in RGB color.

The image can be saved by right-click copy or by “ファイル(F)” → “エクスポート(E)" from the mapping window menu.

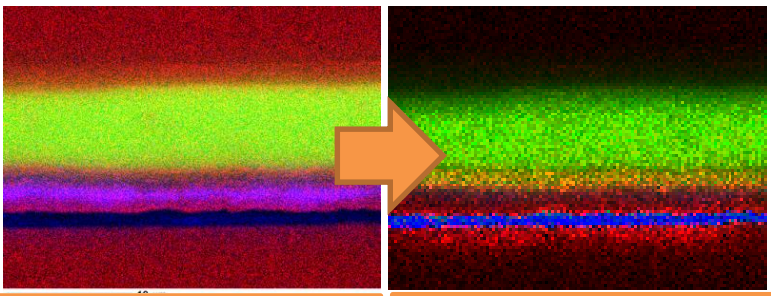


- Quantitative map

Convert intensity mapping data to quantitative maps. Click “分析(N)” → “定量マップ作成(Q)" from the mapping window menu. Select the map size and unit in the generate conditions window, and click ”Generate” to display the quantitative map.



Since the intensity map is an intensity distribution image of the main peak of each element, the distribution of each element can be understood, but the amount cannot be compared between each element. Quantitative map represent quantitative values of each element in color. Intensity map has the influence of shielding of characteristic X-rays (signal attenuation) due to irregularities on the sample surface. Quantitative map has little influence, even if there is some signal attenuation, because the map is calculated quantitative values and describe the distribution.

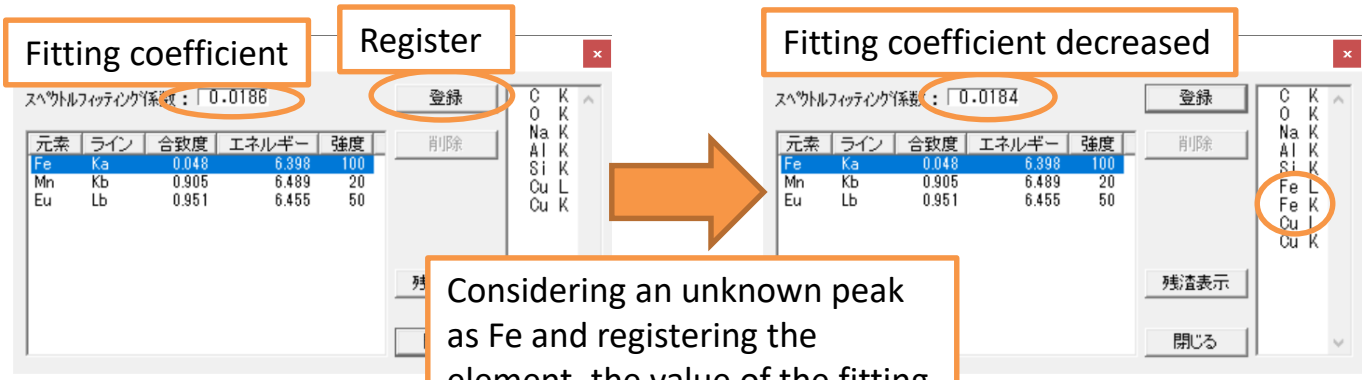


Intensity map Au Al Ti

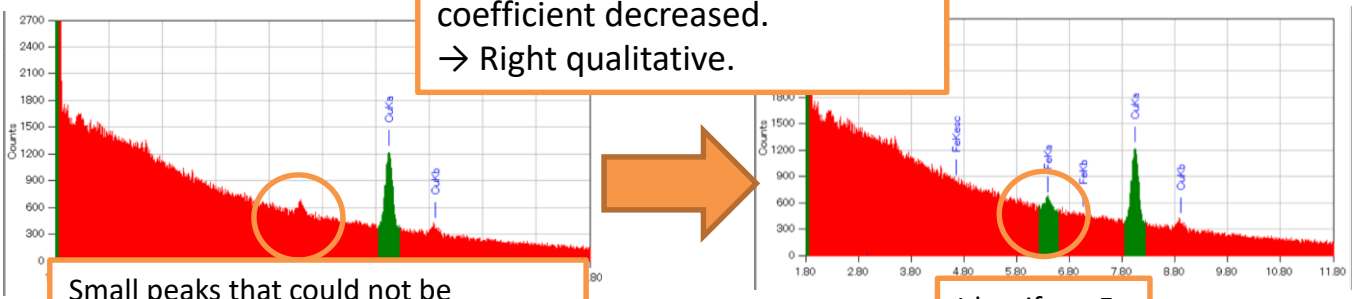
Quantitative map Au Al Ti

Other functions

When identifying a small peak, you can check the spectral fitting coefficient by using "VID" in the spectrum window menu. The correctness of the identified element can be evaluated by fitting calculation.



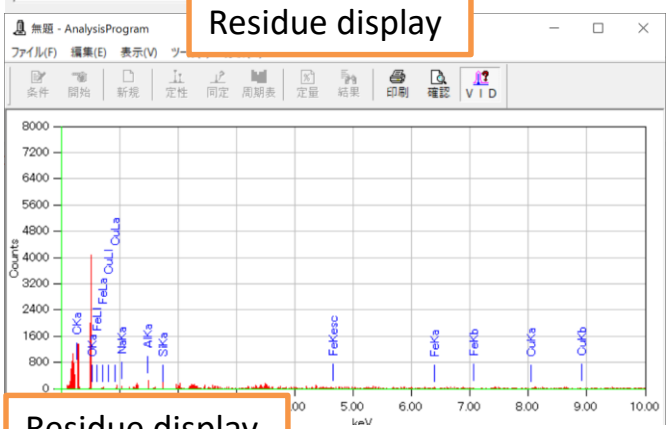
Considering an unknown peak as Fe and registering the element, the value of the fitting coefficient decreased.
→ Right qualitative.



Small peaks that could not be determined by automatic qualification

Identify as Fe

Residue display

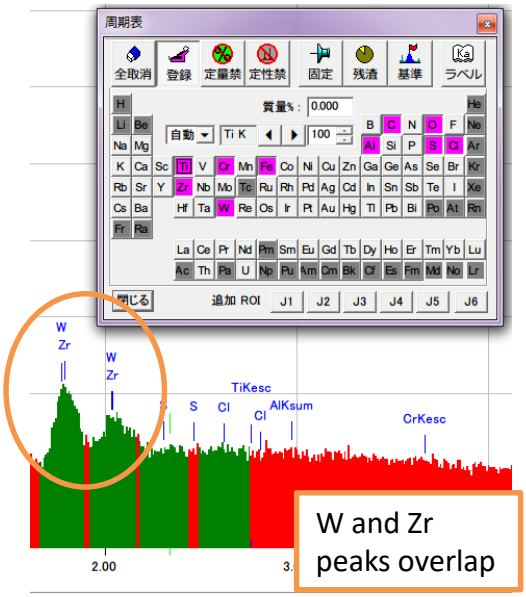


Residue display

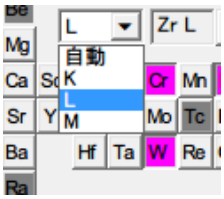
In addition, the residual between the spectrum calculated from the qualitative composition and the measured spectrum can be displayed. The residue display makes it easier to find peaks that have not been qualified.

Other functions

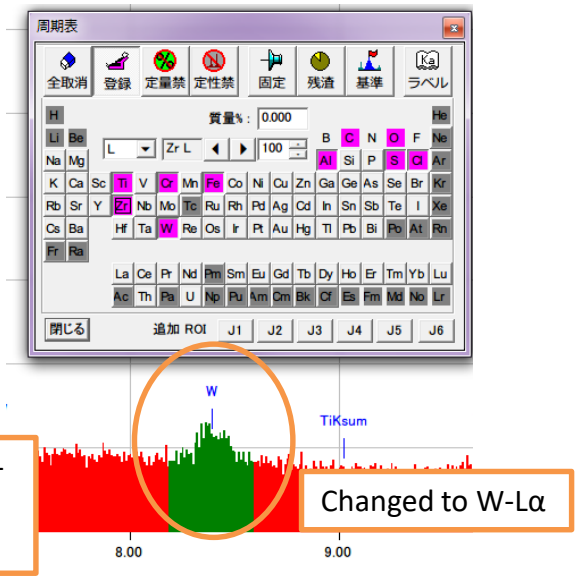
If the peaks overlap, quantification may not work. You can do quantitative calculation or regenerate new intensity map by changing to other X-ray that no overlapping peaks in the "Periodic Table".



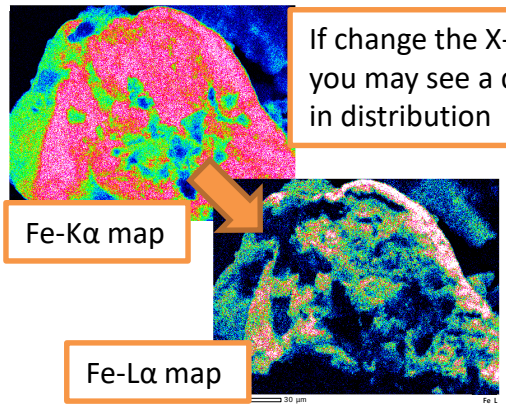
W and Zr peaks overlap



Select the type of X-ray and re-register the element



Changed to W-Lα

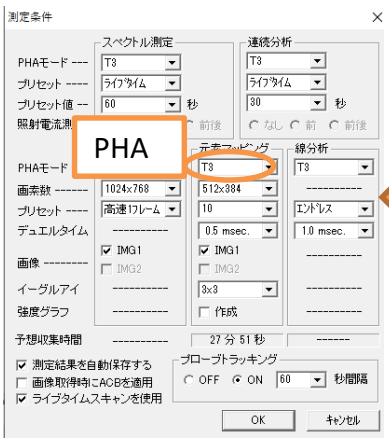


If change the X-ray type, you may see a difference in distribution

Fe-Kα map

Fe-Lα map

If the mass% of a certain element in the sample is known, the mass% can be fixed in the "Periodic Table".



PHA

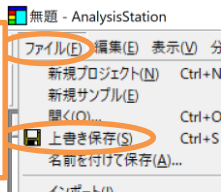
When the X-ray cps is small in the mapping, you can increase the cps and measure by changing “PHA” from T3 to T1 in the “Conditions” window. However, use it for elemental analysis after Na because the energy resolution is reduced. Please be sure to return initial condition after use.

EDS analysis end procedure

Conditions

Initial state

Save project folder



- Please return each parameter to initial state by "Conditions" icon.
- Save project folder.
- Close “Analysis station”.
- EDS power Off.

Don’t forget ！

- Please refer to "SEM Basic Manual" for how to end SEM.

Appendix

This table is an example of peaks overlapping. If this combination exists, use VID to check the peaks well.

Ca−Kα (3.690KeV)	K−Kβ (3.589KeV) Sb−Lα (3.604KeV) Te−Lα (3.769KeV) Sn−Lβ (3.662KeV)
Sc−Kα (4.088KeV)	Ca−Kβ (4.012KeV)
Ti−Kα (4.508KeV)	Ba−Lα (4.465KeV) La−Lα (4.650KeV)
V−Kα (4.949KeV)	Ti−Kβ (4.931KeV)
Cr−Kα (5.411KeV)	V−Kβ (5.426KeV)
Mn−Kα (5.894KeV)	Cr−Kβ (5.946KeV)
Fe−Kα (6.398KeV)	Mn−Kβ (6.489KeV)
Co−Kα (6.924KeV)	Fe−Kβ (7.057KeV)
Ni−Kα (7.471KeV)	Co−Kβ (7.648KeV)
Cu−Kα (8.040KeV)	Ni−Kβ (8.263KeV)
Zn−Kα (8.630KeV)	Cu−Kβ (8.904KeV)
Se−Lα (1.379KeV)	W−Mz (1.380KeV)
Tb−Mα (1.240KeV)	As−Lα (1.282KeV)

Na−Kα (1.041KeV)	Cu−Lα (0.930KeV) Zn−Lα (1.012KeV)
Mg−Kα (1.253KeV)	Ge−Lα (1.188KeV) As−Lα (1.282KeV) Tb−Mα (1.24KeV)
Al−Kα (1.486KeV)	Br−Lα (1.48KeV)
Si−Kα (1.739KeV)	Rb−Lα (1.694KeV) Sr−Lα (1.806KeV) Ta−Mα (1.709KeV) W−Mα (1.774KeV)
P−Kα (2.013KeV)	Zr−Lα (2.042KeV) Ir− Mα (1.977KeV) Pt−Mα (2.048KeV) Au−Mα (2.120KeV) W−Mγ (2.035KeV)
S−Kα (2.307KeV)	Mo−Lα (2.293KeV) Pb−Mα (2.342KeV) Bi−Mα (2.419KeV)
Cl−Kα (2.621KeV)	Ru−Lα (2.558KeV) Rh−Lα (2.696KeV)
K−Kα (3.312KeV)	In−Lα (3.286KeV) Cd−Lβ (3.316KeV)