

Quantitative EDS Analysis using AZtec software platform

Good Practices

Step 1: Enter Project Notes and Specimen Notes for the sample in “Describe Specimen”

Step 2: Select “Ratemeter” in the “Mini View” to verify the dead time and input count rate.

Step 3: Perform a “Beam Measurement” in “Optimize” Calibrate step (If interested in un-normalized quant).

Step 4: Collect a reference image in “Scan Image” step in Point&ID. Skip if not interested in collecting an image.

Step 5: Start spectral acquisition by selecting appropriate tool (spot, rectangular region, etc.) and clicking on the region of interest in Point & ID.

Step 6: Use the Fitted Spectrum tool to verify peak identification in “Confirm elements”

Step 7: Quant in Calculate Composition step.

For Quant using Standards:

Step 8: Acquire a spectrum from a standard as defined above. Standardize the elements of interest in “Standardize” (located in Optimize).

Step 9: Re-quantify spectra from unknown samples using the updated standard database.

Step 1: project/Specimen details

AZtec - Project 1

File View Techniques Tools Help

EDS-SEM Point & ID Describe Specimen Scan Image Acquire Spectra Confirm Elements Calculate Composition Compare Spectra Report Results Guided Custom

Specimens in 'Project 1'

+ New Specimen

Specimen 1

Summary Specimen Geometry Pre-defined Elements

Project Notes

[Click here to begin entering notes about your project.](#)

Enter Project Notes and Specimen Notes

Specimen Notes for 'Specimen 1'

[Click here to begin entering notes about your specimen.](#)

Specimen Coating Information:

The specimen has been coated with: Carbon

Thickness (nm): 10.00

Density (g/cm³): 2.25

Search Help

Data View

Current Site Data Tree

Site 1

Mini View Ratemeter

Input Count Rate 82360 cps

Output Count Rate 36860 cps

Dead Time 57%

Process Time 3

Recommended WD 8.5 mm

High Voltage 20.0 kV

Step Notes

In this step you can:

- Write notes on your Project and Specimen (For convenience you can also copy images/diagrams and text from other documents/emails and paste into these windows).
- Add New Specimens to the Project:

+ New Specimen

Mag: 4465x HV: 20.0kV WD: 8.76 mm Specimen Tilt: 0.00° Input Rate: 82360 cps Output Rate: 36860 cps Dead Time: 57% Process Time: 3

Step 2: Verifying microscope conditions

The screenshot displays the AZtec software interface for 'Project 1'. The top menu includes File, View, Techniques, Tools, and Help. A toolbar contains icons for EDS-SEM, Point & ID, Describe Specimen, Scan Image, Acquire Spectra, Confirm Elements, Calculate Composition, Compare Spectra, Report Results, Guided, and Custom. The main workspace is titled 'Specimens in 'Project 1'' and contains a 'New Specimen' button and a 'Specimen 1' entry. A 'Project Notes' section contains the following text:

Select "Ratemeter" in the "Mini View" to verify the dead time and input count rate.

20kV and Process time 3 is recommended for unknown samples.
For low kV analysis use Process Time 5.

At any given process time adjust the spot size (beam current) to yield a dead time around 45% on pure Cu (Cu tape is OK).

The 'Mini View' panel on the right shows the 'Ratemeter' settings:

- Input Count Rate: 82360 cps
- Output Count Rate: 36860 cps
- Dead Time: 57%
- Process Time: 3
- Recommended WD: 8.5 mm
- High Voltage: 20.0 kV

The 'Specimen Coating Information' section shows:

- The specimen has been coated with: Carbon
- Thickness (nm): 10.00
- Density (g/cm³): 2.25

The bottom status bar displays: Mag: 4465x, HV: 20.0 kV, WD: 8.76 mm, Specimen Tilt: 0.00°, Input Rate: 82360 cps, Output Rate: 36860 cps, Dead Time: 57%, Process Time: 3.

Step 3: Beam measurement for un-normalized quant analysis



Optimize



Collect a spectrum from copper tape or any of the other available pure elements in the list.



Routine: Beam Measurement Element: Cobalt Acquire Spectra **START** **STOP** Settings

Beam Measurement
Energy Calibration

Beam Measurement

If you require accurate un-normalized quantitative analysis results, you must perform the Beam Measurement routine. Any change in the microscope settings such as accelerating voltage or lens control will lead to the change in the beam current. Under these circumstances you must perform the Beam Measurement routine before you do accurate quantitative analysis.

Note that you **do not need** to perform the Beam Measurement routine if you are only interested in:

- Qualitative Analysis
- Normalized Quantitative Analysis

Energy Calibration

For accurate identification of peaks, you need to perform the Energy **Calibration**. Energy **Calibration** measures the shift in the position of the spectral peaks and resolution of the system. As the system has very stable electronics, you may only need to **calibrate** the system once in several months, provided the environmental temperature of the laboratory is fairly stable. A few degrees change in the environmental temperature can cause a small shift in the position of peaks.

The Energy **Calibration** routine is performed for representative Process times, available energy ranges and number of channels in one operation. This means if you change any of these settings soon after you perform the Energy **Calibration**, you will not need to re-**calibrate** the system.

Step 4: Collect a reference image

AZtec - Project 1

File View Techniques Tools Help

EDS-SEM Point & ID Describe Specimen Scan Image Acquire Spectra Confirm Elements Calculate Composition Compare Spectra Report Results Guided Custom

New Site Scan Image **START** STOP Settings Select Second Image: None

Electron Image 1

Mag: 139x HV: 20.0kV WD: 8.76mm Specimen Tilt: 0.00° Input Rate: 58330 cps Output Rate: 32840 cps Dead Time: 44% Process Time: 3

Search Help

Data View

Current Site Data Tree

Electron Image 1

Site 1

Mini View Ratemeter

Input Count Rate 58330 cps
Output Count Rate 32840 cps
Dead Time 44%
Process Time 3
Recommended WD 8.5 mm
High Voltage 20.0 kV

Step Notes

In this step you can acquire an electron image into a 'Site'. A 'Site' is generally seen as a folder that contains images and analyses related to a particular area on a specimen:

- Press the green start button to acquire an image (according to the current acquisition parameters).
- If you wish to change the acquisition parameters (resolution, speed, input source/s and number of frames) press the settings cog on the Scan Image toolbar:

OXFORD INSTRUMENTS

To collect multiple images (SE, BSE, InLens, etc.) in a given site of interest the acquired image can be locked to prevent overwriting. Hitting the Start button after locking an acquired image adds other images to the Current Site.

Step 5: Start Spectral Acquisition

AZtec - Project 1

File View Techniques Tools Help

EDS-SEM Point & ID Describe Specimen Scan Image Acquire Spectra Confirm Elements Calculate Composition Compare Spectra Report Results Guided Custom

Acquire Spectrum **START** **STOP** Settings Select Second Image: Electron Image 2

Live acquisition tools

Spectral reconstruction tools – to reconstruct spectra from x-ray maps

Use any of the live acquisition tools to select an area on the image and start spectral acquisition

Progress bar: Mouse-over reveals the acquisition time & counts.

MiniQuant: Can be minimized

EDS Acquire Spectrum Settings

Energy Range (keV): Auto

Number of Channels: Auto

Process Time: 3

Acquisition Mode: Live Time

Acquisition Time (s): 20.0

Pulse Pile Up Correction

Element	Wt%	σ
O	48.5	0.2
Si	32.3	0.1
Al	10.6	0.1
Na	8.3	0.1
K	0.2	0.0
Ca	0.1	0.0

Search Help

Data View

Current Site Data Tree

- Electron Image 1
- Electron Image 2
- Spectrum 1**

Percent Complete: 46%

Remaining Time: 0:00:19

Run Time: 0:00:16

Live Time: 0:00:09

Area Counts: 568210

Mini View Ratemeter

Input Count Rate 63730 cps

Output Count Rate 34680 cps

Dead Time 46%

Process Time 3

Recommended WD 8.5 mm

High Voltage 20.0 kV

Step Notes

In this step you can acquire spectra from the current electron image (SE/BSE). Compare and Mini Quant options are also available here.

To acquire spectra:

- Press the green Start button to acquire a spectrum from the entire image area.
- Four beam control tools are available on the left side of the window to define areas on the image for spectrum acquisition. These are Point, Rectangle, Ellipse and Freehand:

Mag: 139x HV: 20.0 kV WD: 8.76 mm Specimen Tilt: 0.00° Input Rate: 63730 cps Output Rate: 34680 cps Dead Time: 46% Process Time: 3

Step 6: Fitted Spectrum Tool – to confirm the elements in the spectrum

File View Techniques Tools Help

EDS-SEM Point & ID Describe Specimen Scan Image Acquire Spectra Confirm Elements Calculate Composition Compare Spectra Report Results Guided Custom

Search Help Data View Current Site Data Tree Electron Image 3 Spectrum 6

Confirm Elements Settings

View Settings

- Show Markers
- Show Peak Shapes
- Show Fitted Spectrum
- Show Theoretical Spectrum
- Show No Pulse Pile Up Correction

Candidate Elements

Element	Count	%
Ba	33.1	0.1
O	33.6	0.2
Si	19.7	0.1
Ti	11.6	0.1

counts

keV

Periodic Table Element List Peak Labels

H He

Li Be B C N O F Ne

Na Mg Al Si P S Cl Ar

K Ca Sc Ti V Cr Mn Fe Co Ni Cu Zn Ga Ge As Se Br Kr

Rb Sr Y Zr Nb Mo Tc Ru Rh Pd Ag Cd In Sn Sb Te I Xe

Cs Ba La Hf Ta W Re Os Ir Pt Au Hg Tl Pb Bi Po At Rn

Fr Ra Ac Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu

AutoID Pre-defined Clear all

Mag: 62x HV: 20.0kV WD: 8.76 mm Specimen Tilt: 0.00° Input Rate: 56970 cps Output Rate: 30920 cps Dead Time: 34% Process Time: 3

Site 2 Mini View Ratemeter Step Notes Information Saved Project 1

Overlays peak profile for the selected element

Overlays (in pink) a theoretical spectrum generated from the profiles of identified elements.

An excellent fit between the experimental and theoretical spectra indicates the presence of both Ba and Ti in the analyzed region.

Step 7: Quant

Navigation bar with icons for EDS-SEM, Point & ID, Describe Specimen, Scan Image, Acquire Spectra, Confirm Elements, Calculate Composition, Compare Spectra, Report Results, Guided, and Custom.

Search Help, Data View, Current Site, Data Tree, Electron Image 3, Spectrum 6

- ### Available Templates
- Summary Table - Single Spectrum
 - Comparison of Results - Two Spectra
 - Summary Table - Multiple Spectra
 - Full Results Table (customizable) - Single Spectrum
 - Spectrum Details - Details
 - Spectrum Processing - Processing
 - Diagnostics Table (customizable) - Single Spectrum

Quant Result Details

Label: Spectrum 6
Current Spectrum: Oxygen by Stoichiometry
Coating Element: Carbon
Coating Thickness: 100 nm
Coating Density: 2.25 g/cm³

Buttons: Copy, Requantify

The spectrum will be quantified based on the "Settings" defined for quantification. The settings include assigning oxygen by stoichiometry, deconvolution elements, standards used for quantification, etc. The spectrum needs to be requantified to apply changes made to the settings.

Quant Results View

Viewed Data: Spectrum 6
Result Type: Weight %
Processing Option Used: Oxygen by Stoichiometry (Normalized)

Element	Wt%	Wt% Sigma
O	34.06	
Si	19.56	0.07
Ti	11.55	0.09
Ba	34.83	0.14
Total	100.00	

Settings Dialog

- Processing options: All Elements, Element by Difference, Oxygen by Stoichiometry, Normalize results
- Deconvolution elements: Carbon, Add element, Remove element, Clear All
- Threshold quantitative results: Enable thresholding, Sigma level: 2.0
- Quant standardizations: Factory: Quant Standardizations(Extended Set), User
- Element list: Current Spectrum, Fixed List, Fixed List and Current Spectrum
- Automatic line selection for all elements: checked
- Element Details for Hydrogen: Fixed weight %: 0.00

Buttons: Apply and Save, Save, Close

Step 7: Quant - Multiple Spectra Comparison

Calculate Composition Settings

- Available Templates
- Summary Table - Single Spectrum
 - Comparison of Results - Two Spectra
 - Summary Table - Multiple Spectra
 - Full Results Table (customizable) - Single Spectrum
 - Spectrum Details - Details
 - Spectrum Processing - Processing
 - Diagnostics Table (customizable) - Single Spectrum

Quant Result Details

Label: Spectrum 6
 Element List Type: Current Spectrum
 Processing Option: Oxygen by Stoichiometry
 Coating Element: Carbon
 Coating Thickness: 100 nm
 Coating Density: 2.25 g/cm³

Copy

Multiple spectra can be selected from the Data Tree by holding the "ctrl" button on the keyboard. These selected spectra can be added to the Quant Summary Table.

Quant Results View

Viewed Data: Multiple Spectra Result Type: Weight %

Spectrum Label	O	Na	Al	Si	K	Ca	Ti	Ba	Total	Project Path
Spectrum 1	48.51	8.34	10.56	32.33	0.15	0.11			100.00	Project 1/Specimen 1/Site 1
Spectrum 2	45.17	7.87	10.93	35.73	0.23	0.07			100.00	Project 1/Specimen 1/Site 1
Spectrum 3	48.51	8.32	10.56	32.32	0.17	0.12			100.00	Project 1/Specimen 1/Site 1
Spectrum 6	34.06			19.56			11.55	34.83	100.00	Project 1/Specimen 1/Site 2

Statistics

Statistic	O	Na	Al	Si	K	Ca	Ti	Ba
Max	48.51	8.34	10.93	35.73	0.23	0.12	11.55	34.83
Min	34.06	7.87	10.56	19.56	0.15	0.07	11.55	34.83
Average	44.06			29.98				
Standard Deviation	6.85			7.13				

Clear All Spectra Remove Selected Spectra

Search Help

Data View

Current Site Data Tree

- Project 1
 - Specimen 1
 - Site 1
 - Electron Image 1
 - Electron Image 2
 - Spectrum 1
 - Spectrum 2
 - Spectrum 3
 - Spectrum 4
 - Spectrum 5
 - Site 2
 - Electron Image 3
 - Spectrum 6

Add Selected Spectra

Site 2 Add Selected Spectra to Quant Step

Mini View Spectrum

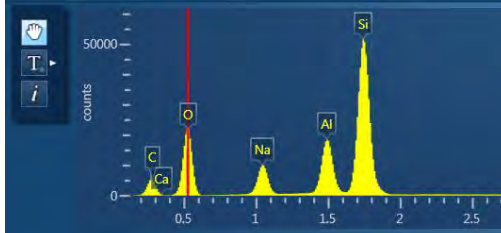
Step Notes

Step 8: Optimize - Standardize

Data from custom standards can be collected and stored as databases. Spectra can be quantified by applying standard information from these databases.



Optimize



Add Standard

Name:

Choose your composition type from the options below, and then specify a chemical formula, e.g. Na39.34Cl60.66. Total weight % should be 100 ± 5.00.

Number of Atoms
 Weight %

Composition:

Total Weight % = 100.00

OK Cancel

Standard Compositions

Block:

Standard:

Element	Weight %
O	48.76
Na	8.60
Al	10.34

Total Weight % = 100.00

Perform Standardization

Pure Element:

Element from Standard:

X-ray Line Series:

	Current Value	New Value
Date	6/20/2012	8/22/2012
Standard	Quartz	Albite
Concentration (weight %)	53.26	48.76
Normalized peak area	0.4054	0.5070
Standard correction factor	0.3361	0.3600

Clicking Accept button updates the standard information for the element/line in the selected database.

Standardizations

Select:

Show:

Element	Line	Standard	Date	Factory?
Actinium	M series	Ac (v)		Yes
Aluminum	K series	Waspaloy	4/11/2012	No
Americium	M series	Am (v)		Yes
Antimony	L series	Sb		Yes
Argon	K series	Ar (v)		Yes
Arsenic	K series	InAs		Yes
Arsenic	L series	InAs		Yes
Astatine	M series	At (v)		Yes
Barium	L series	BaF2		Yes
Berkelium	M series	Bk (v)		Yes
Beryllium	K series	Be		Yes
Bismuth	L series	Bi		Yes
Bismuth	M series	Bi		Yes
Boron	K series	BN		Yes
Bromine	K series	KBr		Yes
Bromine	L series	KBr		Yes
Cadmium	L series	Cd		Yes
Calcium	K series	Wollastonite		Yes
Californium	M series	Cf (v)		Yes
Carbon	K series	C Vit		Yes
Cerium	L series	CeO2		Yes
Cesium	L series	Cs (v)		Yes
Chlorine	K series	NaCl		Yes
Chromium	K series	Waspaloy	4/11/2012	No
Cobalt	K series	Waspaloy	4/11/2012	No
Copper	K series	Cu		Yes
Copper	L series	Cu		Yes
Curium	M series	Cm (v)		Yes
Dysprosium	L series	Dy (v)		Yes

Step 9: Re-quantify using new standards database



Point & ID



Guided
Custom

Calculate Composition Settings

Available Templates

- Summary Table - Single Spectrum
- Comparison of Results - Two Spectra
- Summary Table - Multiple Spectra
- Full Results Table (customizable) - Single Spectrum
- Spectrum Details - Details
- Spectrum Processing - Processing
- Diagnostics Table (customizable) - Single Spectrum

Quant Results View

Viewed Data: Spectrum 6

Processing Option Used: Oxygen by Stoichiometry (No)

Element	Wt%	Wt% Sigma
O	34.06	
Si	19.56	0.07
Ti	11.55	0.09
Ba	34.83	0.14
Total	100.00	

Quant Result Details

Processing options

- All Elements
- Element by Difference
 - Combined element: Oxygen
- Oxygen by Stoichiometry
 - Number of ions: 3.00

Normalize results

Deconvolution elements

Carbon

Add element

Remove element

Clear All

Threshold quantitative results

Enable thresholding

Sigma level: 2.0

Quant standardizations

- Factory: Quant Standardizations(Extended Set)
- User:

Requantify

Select the updated standard database and click "Requantify".

Element list

- Current Spectrum
- Fixed List
- Fixed List and Current Spectrum

Automatic line selection for all elements

Element Details for Hydrogen

Fixed weight %: 0.00

Search Help

Data View

Current Site Data Tree

Electron Image 3

Spectrum 6

Site 2

Mini View Spectrum

Step Notes

A few examples to demonstrate the use of various settings in Calculate Composition Quant Settings

The screenshot displays the 'Calculate Composition Quant Settings' software interface. The main window is titled 'Calculate Composition Quant Settings' and contains several configuration panels:

- Processing options:** Includes radio buttons for 'All Elements', 'Element by Difference', and 'Oxygen by Stoichiometry'. The 'Oxygen by Stoichiometry' option is selected. Below it, 'Fixed element' is set to 'Oxygen' and 'Number of term' is '3.00'. A 'Normalize results' checkbox is checked.
- Deconvolution elements:** A dropdown menu shows 'Carbon'. Below it are buttons for 'Add element', 'Remove element', and 'Clear All'.
- Threshold quantitative results:** An 'Enable thresholding' checkbox is checked, and the 'Sigma level' is set to '2.0'.
- Quant standardizations:** A 'Factory' dropdown is set to 'Quant Standardizations(Extended Set)', and a 'User' dropdown is empty.
- Element list:** Radio buttons for 'Current Spectrum', 'Fixed List', and 'Fixed List and Current Spectrum'. The 'Fixed List' option is selected. Below is a periodic table with 'H' (Hydrogen) highlighted in a yellow box. To the right of the table is a 'Fixed weight %' input field set to '0.00'.

At the bottom of the window, there are buttons for 'Apply and Save', 'Save', and 'Close'. The 'Element List' and 'Quant Element List Details' tabs are visible at the bottom left of the main panel.

Olivine: To demonstrate the effect of Carbon coating on Oxygen measurement

Olivine Certified Values

O: 43.89

Mg: 30.42

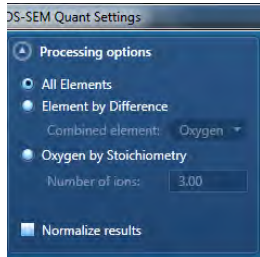
Si: 19.44

Mn: 0.08

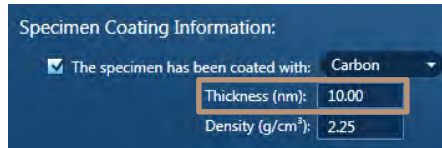
Fe: 5.87

Ni: 0.3

AZtec Quant

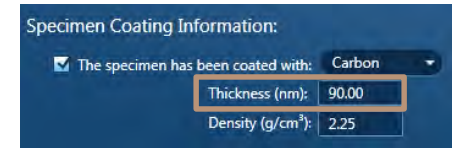


Olivine	Wt%
O	33.14
Mg	28.84
Si	18.85
Mn	0.15
Fe	5.69
Ni	0.23
Total	86.90



Oxygen concentration is severely underestimated due to wrong coating thickness. The actual carbon coating thickness is 90nm – measured using ThinFilmID software.

Olivine	Wt%
O	44.09
Mg	31.00
Si	19.08
Mn	0.16
Fe	5.86
Ni	0.24
Total	100.43



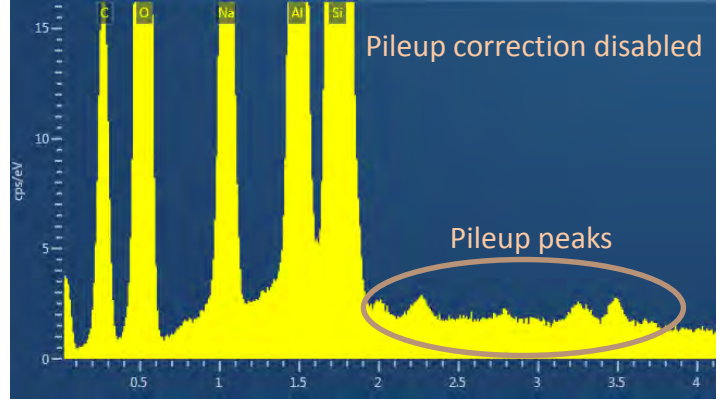
Oxygen concentration is accurately calculated with right Carbon coating thickness. Carbon has an absorption edge near Oxygen, therefore heavily absorbs Oxygen x-rays.

Spectrum quantified by processing all elements, including Oxygen. Normalization was not enabled as beam measurement was performed before the spectral acquisition.

Albite: To demonstrate the effect of Pileup peaks

Albite Certified Values

- O: 48.76
- Na: 8.6
- Al: 10.34
- Si: 32.03
- K: 0.18
- Ca: 0.09



Albite mineral has trace Potassium and Calcium in addition to major concentrations of O, Na, Al and Si. At high count rates the pileup peaks for these major elements fall directly over the trace K and Ca peaks. These can be corrected by enabling Pileup Correction in "Acquire Spectra" settings.

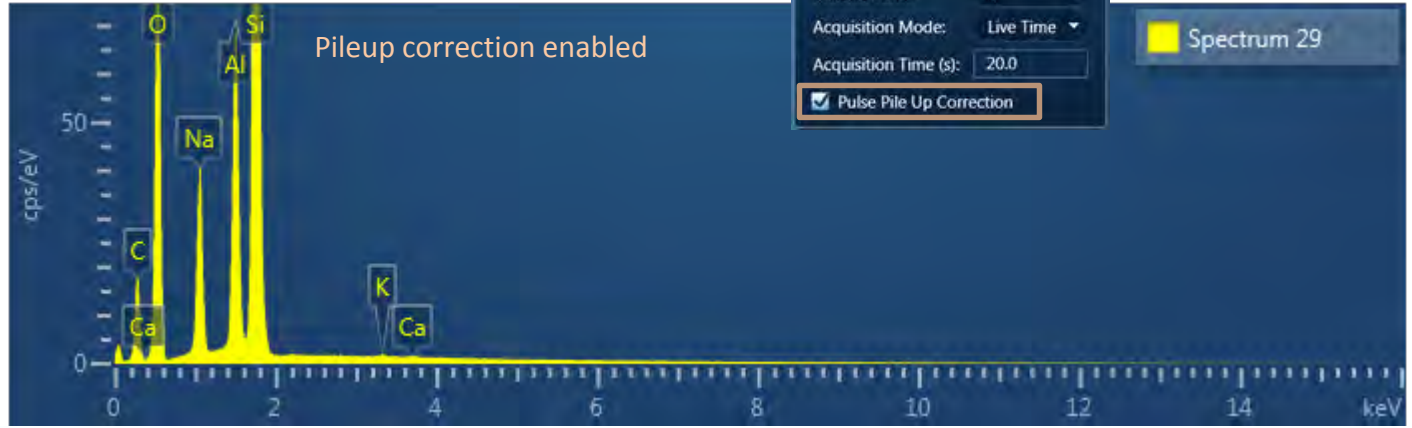
EDS Acquire Spectrum Settings

- Energy Range (keV): Auto
- Number of Channels: Auto
- Process Time: 3
- Acquisition Mode: Live Time
- Acquisition Time (s): 20.0
- Pulse Pile Up Correction

Spectrum 29

AZtec Quant

Albite	Wt%
O	48.90
Na	8.59
Al	10.51
Si	31.81
K	0.10
Ca	0.11
Total	100.02



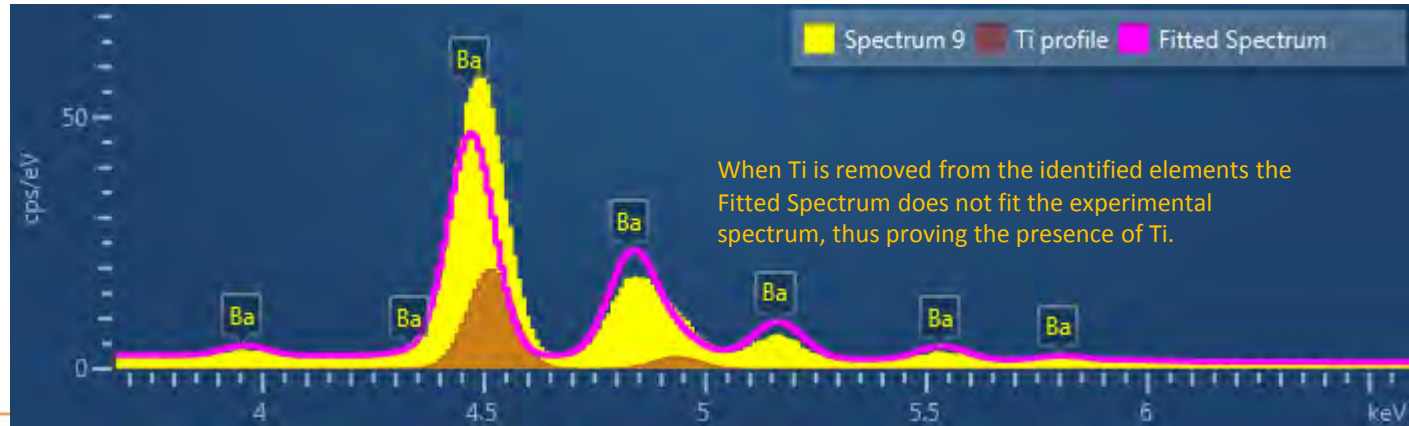
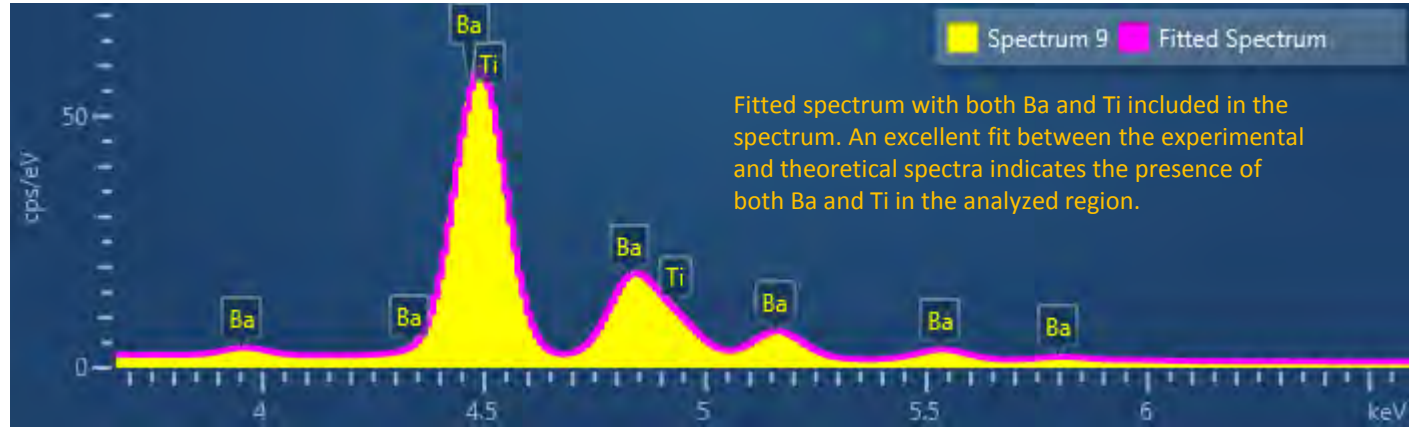
Benitoite: To demonstrate the importance of Fitted Spectrum tool to confirm overlapping peaks

Benitoite: Certified Values

O: 34.82
Si: 20.38
Ti: 11.58
Ba: 33.21

AZtec Quant

Benitoite	Wt%
O	34.81
Si	20.09
Ti	11.37
Ba	34.74
Total	101.01



Calcite: To demonstrate the use of "Fixed Weight %" to incorporate Carbon in the Quant

Calcite: Certified Values

C: 12.02
O: 47.98
Ca: 39.98
Mn: 0.01

Quantifying Carbon using EDS on an SEM is near-impossible as most samples are either coated with carbon or carbon gets sputter deposited by the e-beam during analysis. Therefore, carbon is typically analyzed using alternative techniques. In such cases the Carbon concentration can be entered as "Fixed wt.%" to incorporate it in the matrix corrections.

AZtec Quant

Calcite	Wt%
C (fixed value)	12.02
O	48.02
Ca	39.22
Total	99.26

EDS-SEM Quant Settings

- Processing options
 - All Elements
 - Element by Difference
 - Combined element: Oxygen
 - Oxygen by Stoichiometry
 - Number of ions: 3.00
- Normalize results

Deconvolution elements

Carbon

Add element

Remove element

Clear All

Threshold quantitative results

Enable thresholding

Sigma level: 3.0

Quant standardizations

Factory: Quant Standardizations(Extended Set)

User:

Element list

Current Spectrum

Fixed List

Fixed List and Current Spectrum

Automatic line selection for all elements

Element Details for Carbon

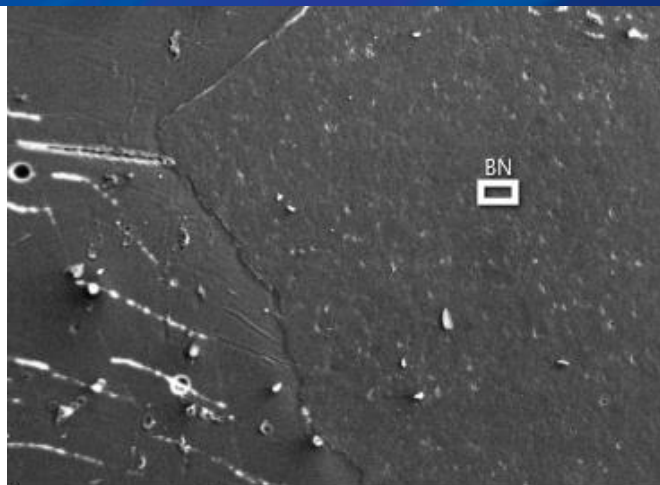
Fixed weight %: 12.02

Apply and Save Save Close

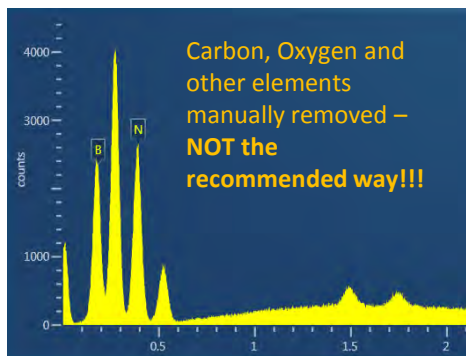
If Carbon value is determined using other technique then it can be entered as a fixed value.

Boron Nitride with Carbon coating and Oxygen contamination

To demonstrate the use of “Deconvolution Elements” in Quant settings



250µm



Manually removing surface coating and other contaminant elements from identification will NOT remove their corresponding x-ray contribution. This may lead to inaccurate quantitative analysis.

The right way to account for these elements is by adding them as “Deconvolution Elements” in the Quant setup (following slide).

Quant by removing coating and contaminant elements

BN	Atomic %
B	55.17
N	44.83

Inaccurate quant – must be 50/50 at.%

Deconvolution elements will be included in the quant but deconvolved out of the quant results.

Quant after applying coating correction and deconvolving contaminant elements out of the analysis.

BN	Atomic %
B	49.93
N	50.07