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"

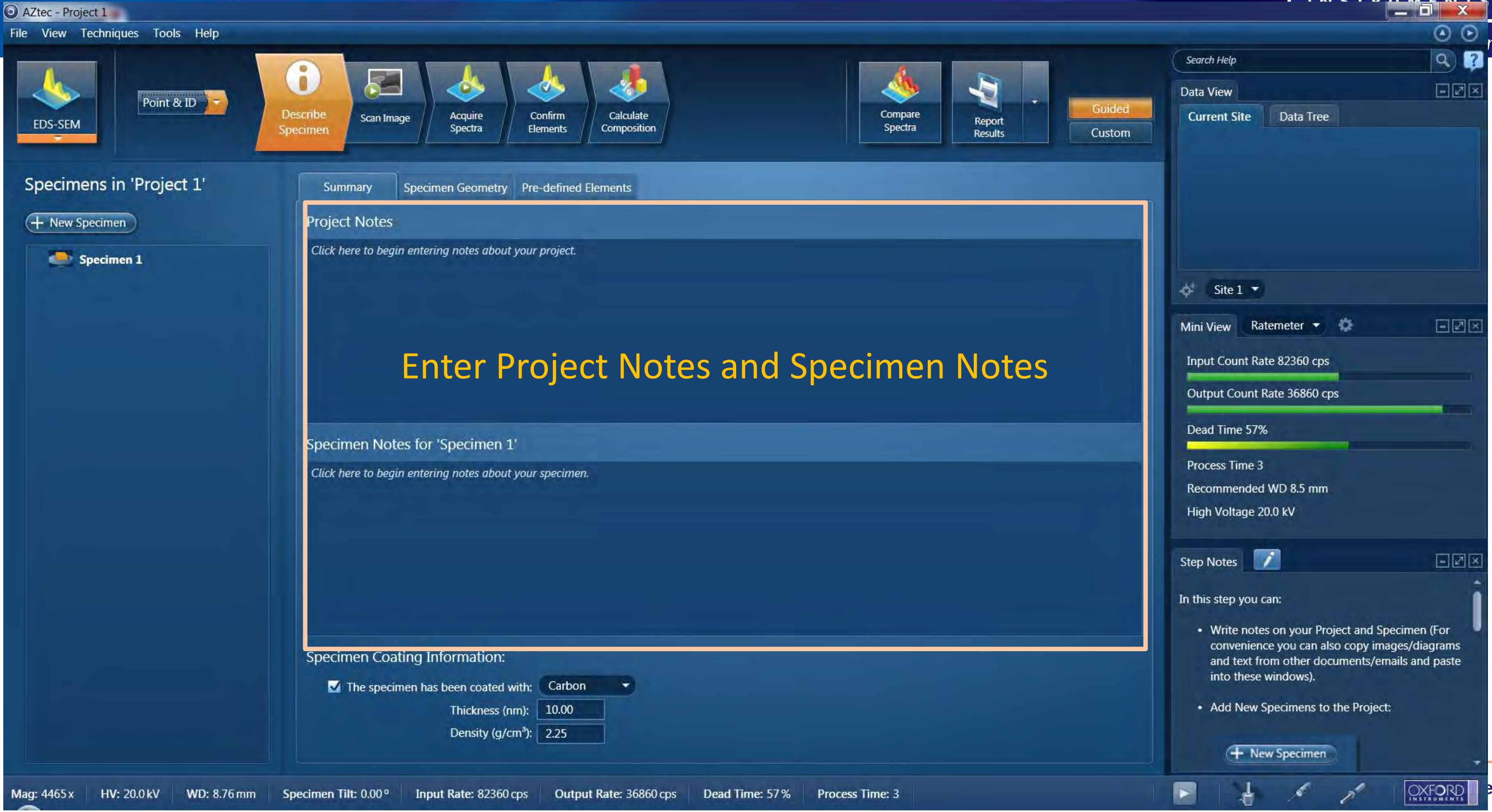
Step 7: Quant in Calculate Composition step.

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



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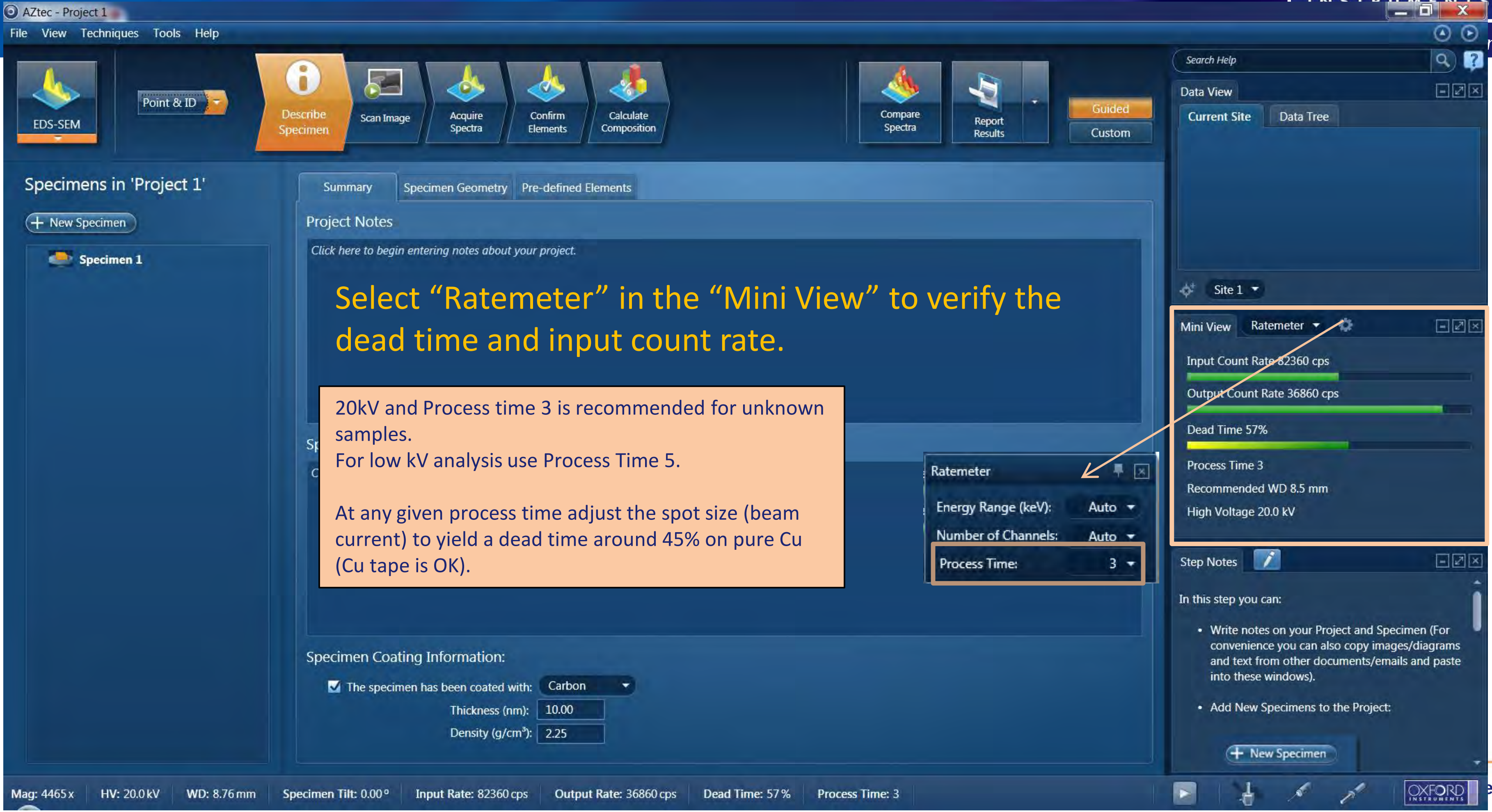
Step 2: Verifying microscope conditions

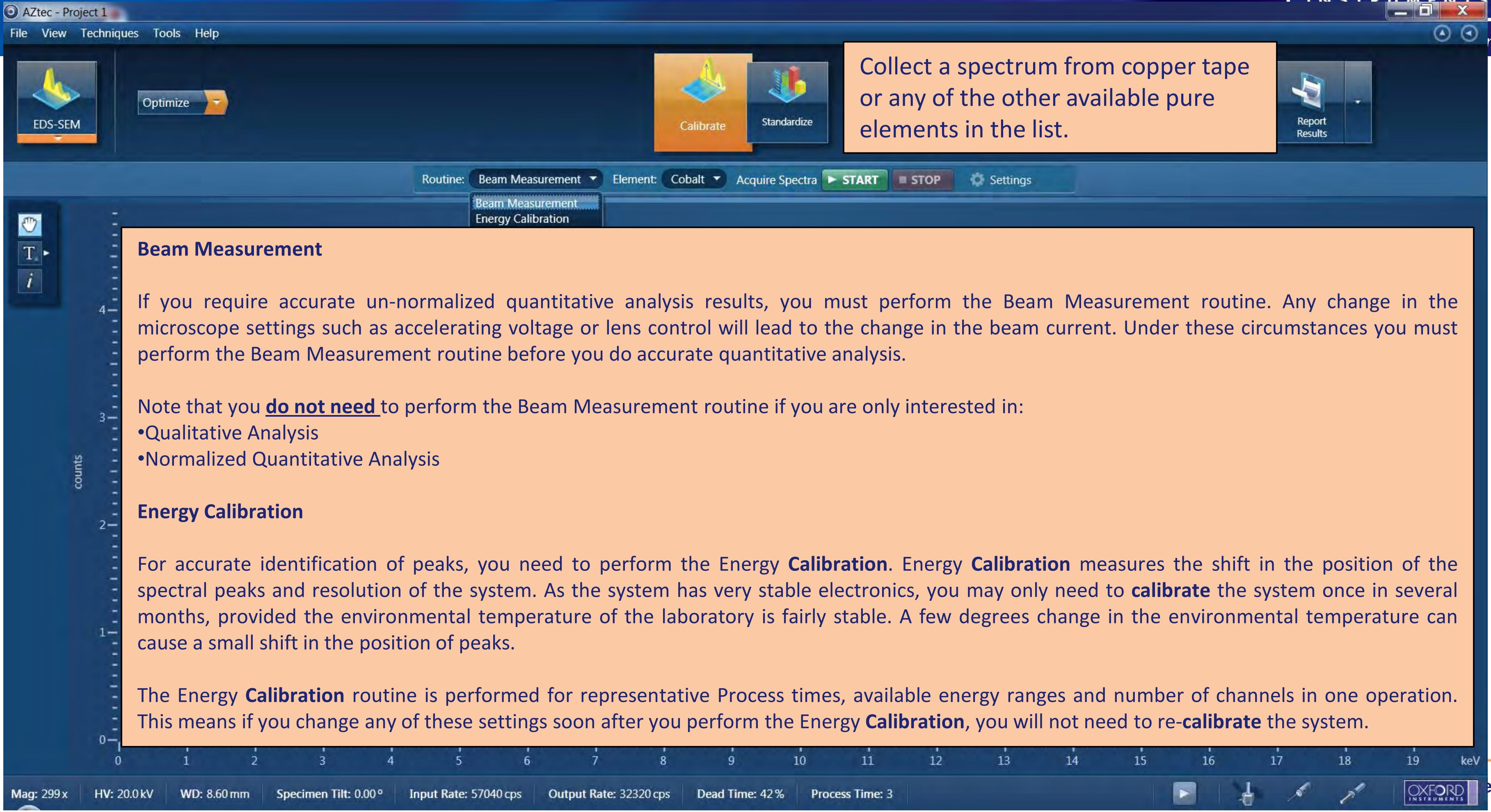


Select “Ratemeter” in the “Mini View” to verify the dead time and input count rate.

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).

Step 3: Beam measurement for un-normalized quant analysis



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

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:

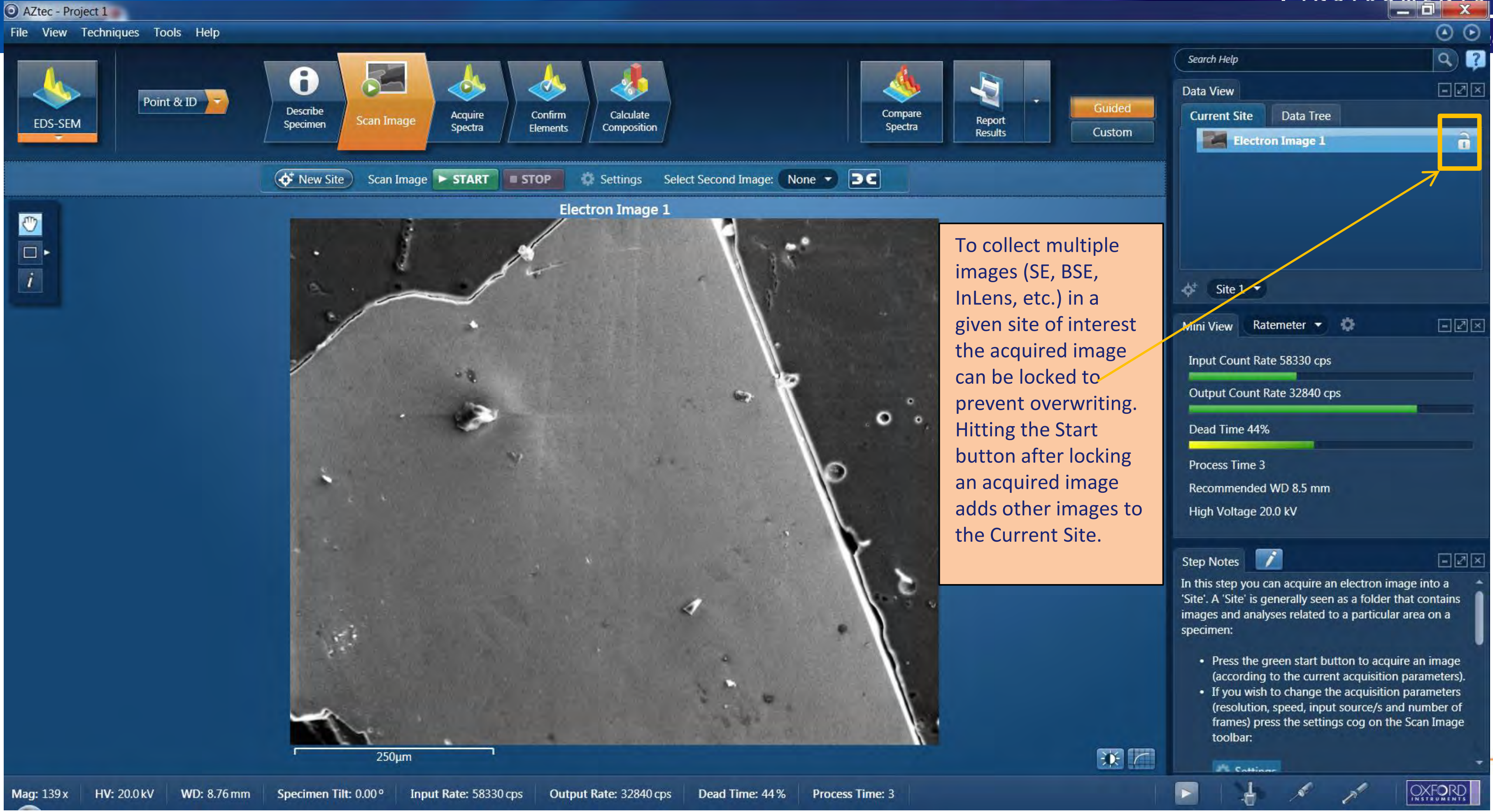
- Qualitative Analysis
- Normalized Quantitative Analysis

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.

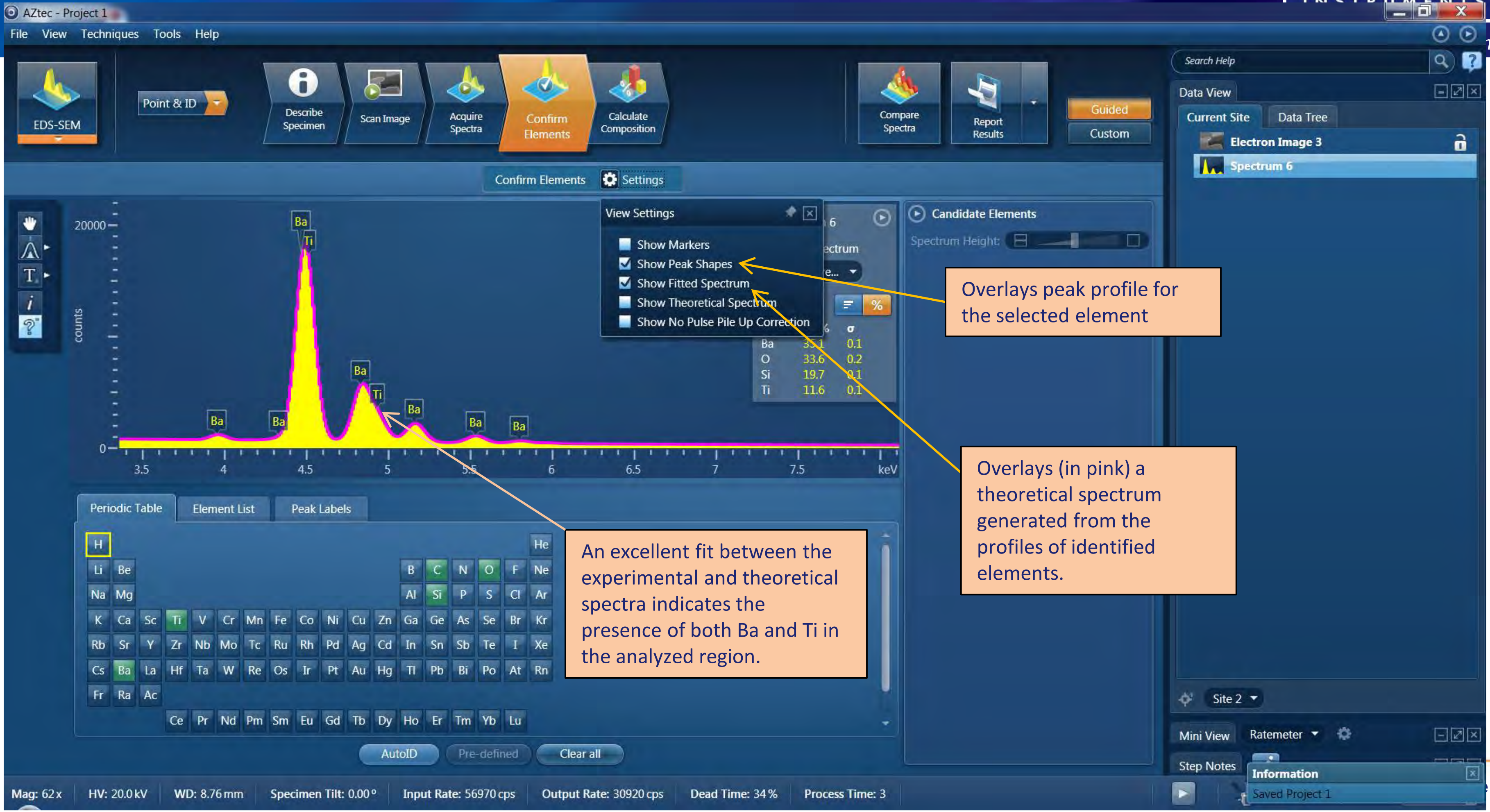
Step 4: Collect a reference image



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

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

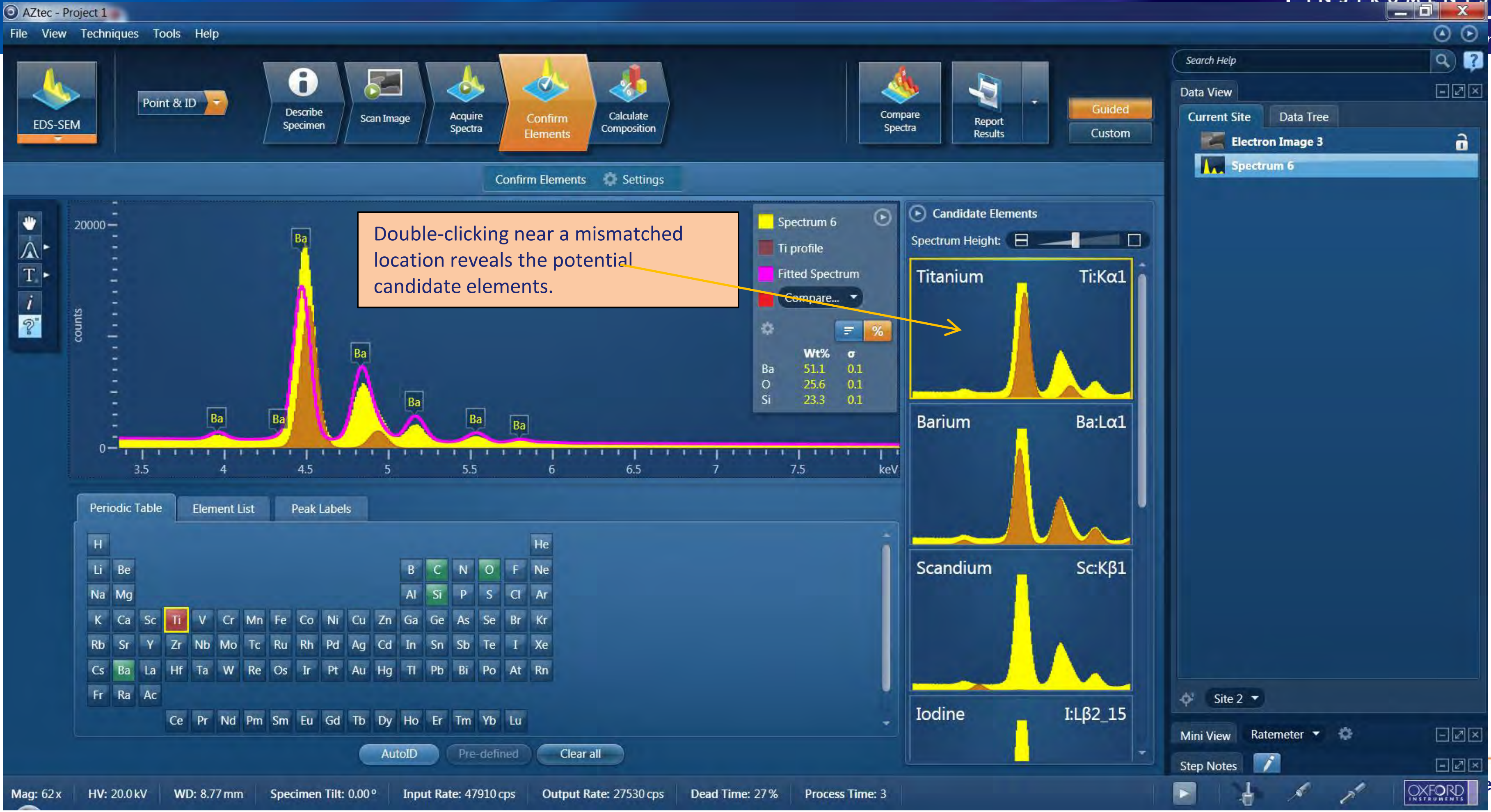


Overlays peak profile for the selected element

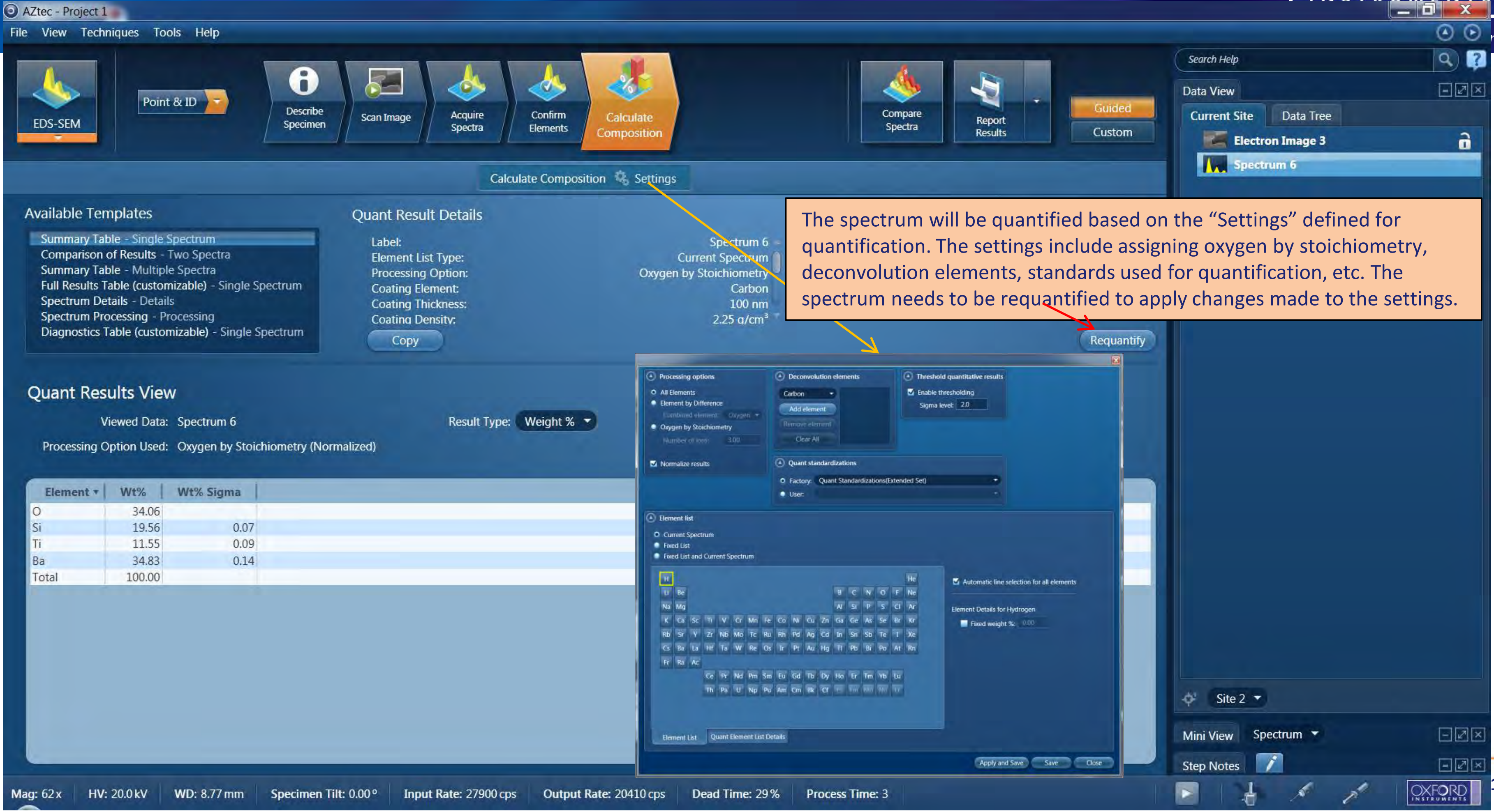
Overlays (in pink) a theoretical spectrum generated from the profiles of identified elements.

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

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

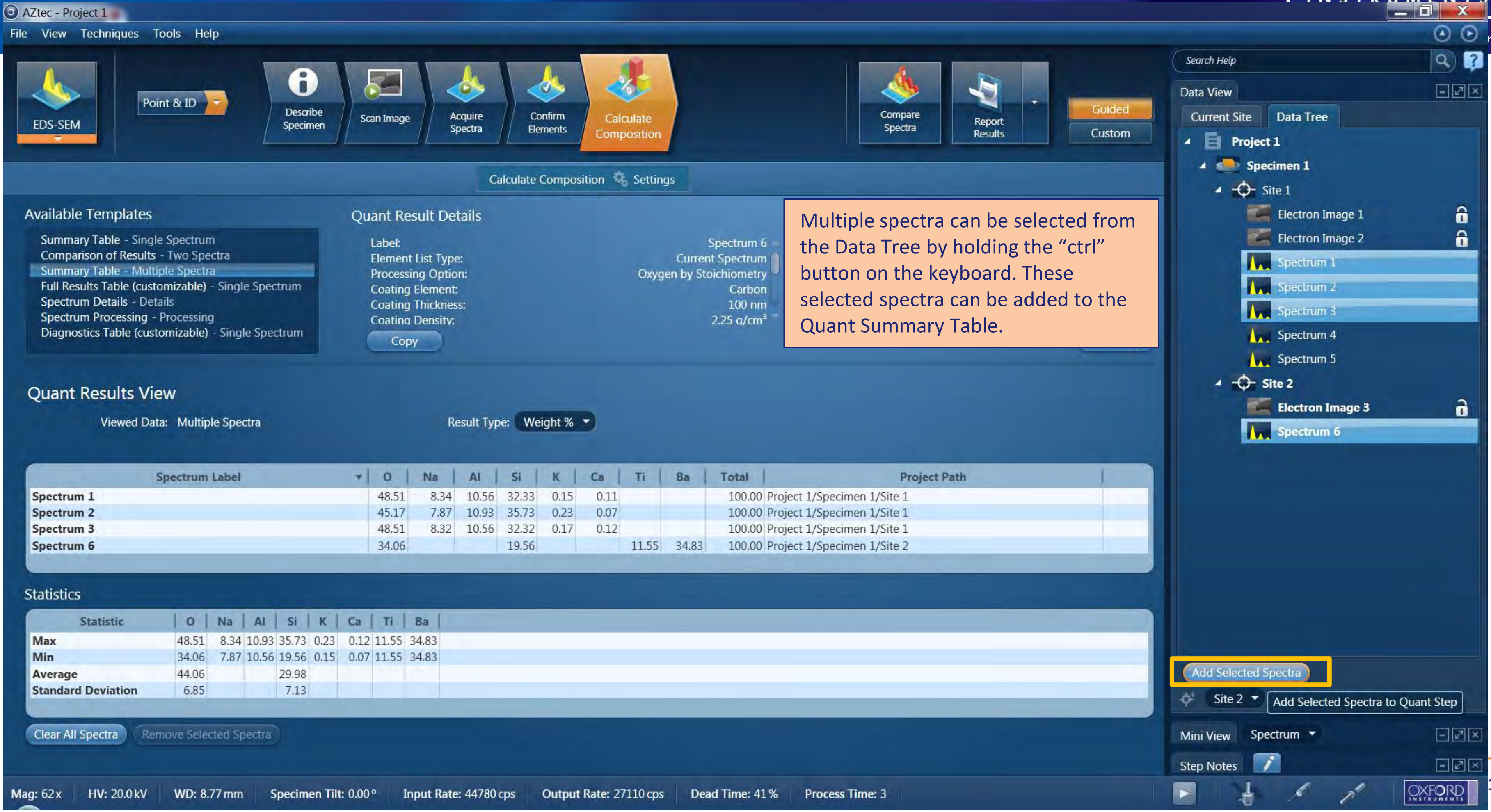


Step 7: Quant



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.

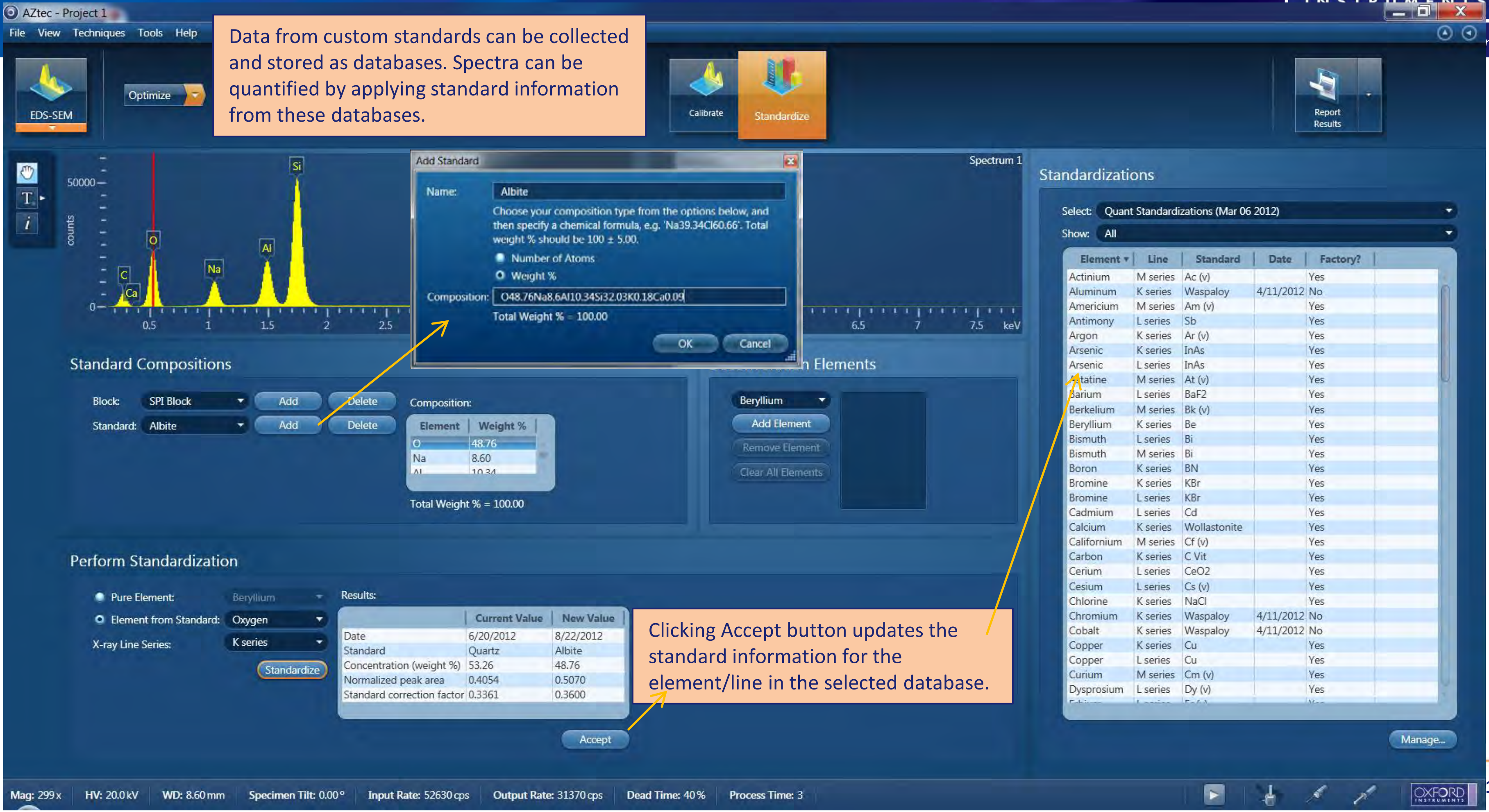
Step 7: Quant - Multiple Spectra Comparison



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.

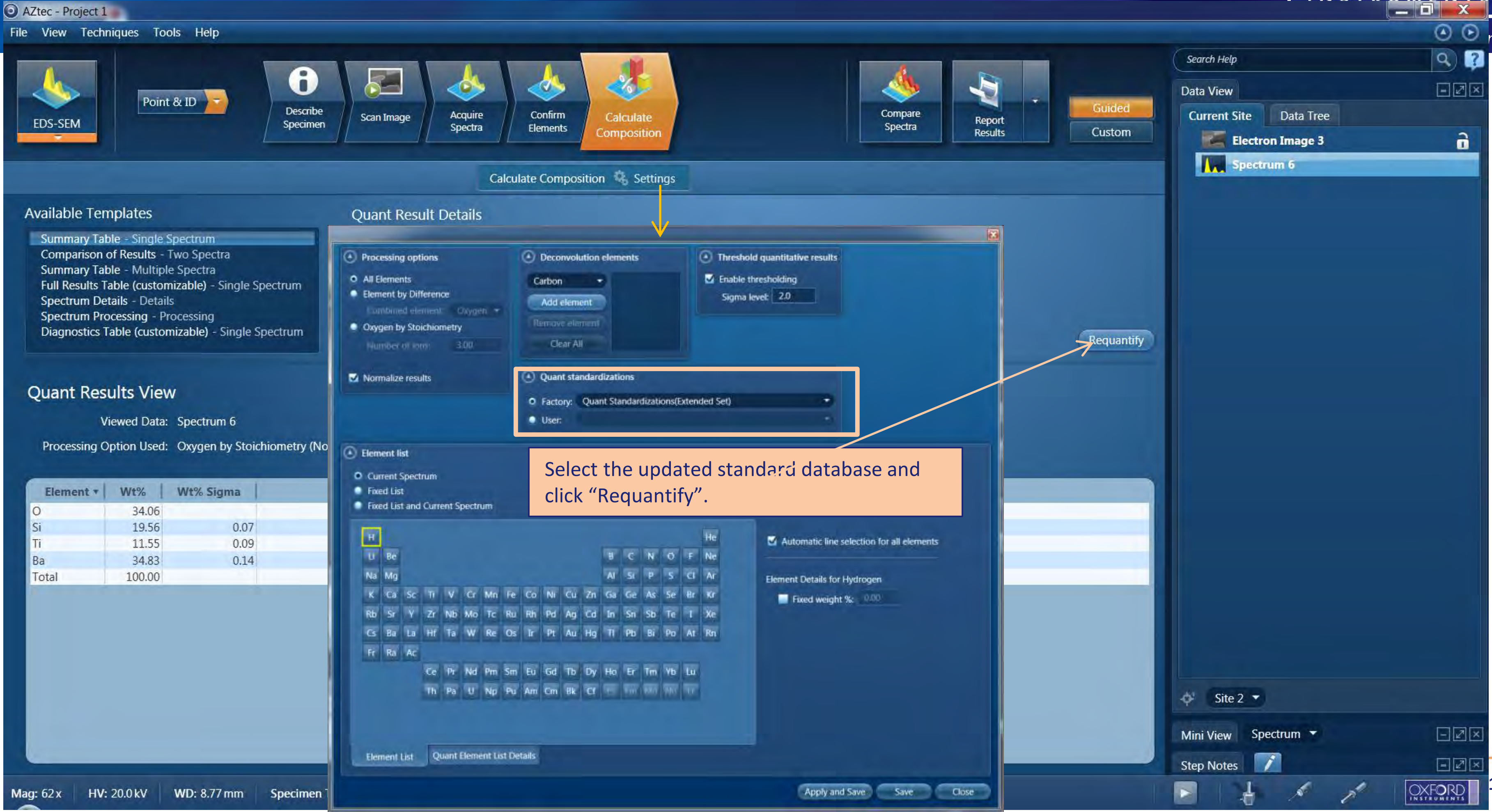
Step 8: Optimize - Standardize

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

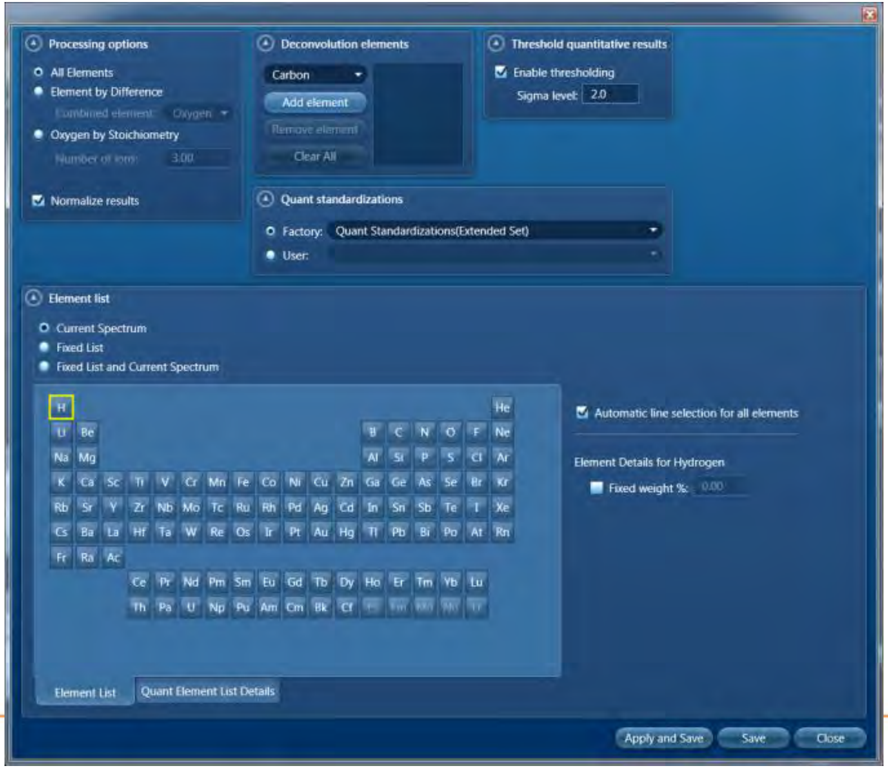


Clicking Accept button updates the standard information for the element/line in the selected database.

Step 9: Re-quantify using new standards database



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



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

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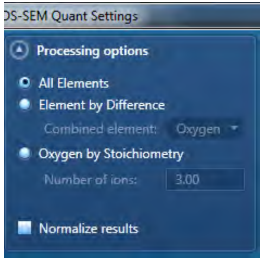
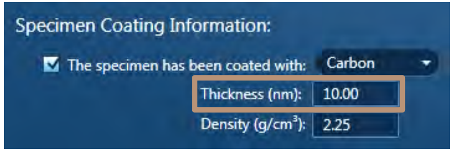
Olivine Certified Values
O: 43.89
Mg: 30.42
Si: 19.44
Mn: 0.08
Fe: 5.87
Ni: 0.3



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

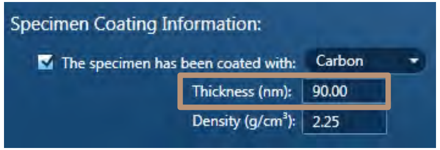
AZtec Quant

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.

Albite: To demonstrate the effect of Pileup peaks

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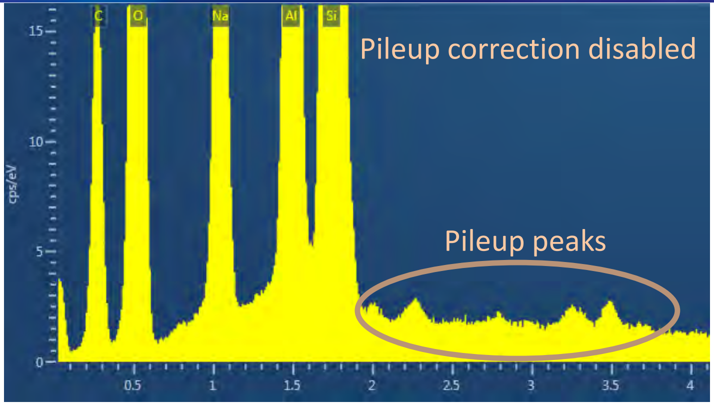
Albite Certified Values

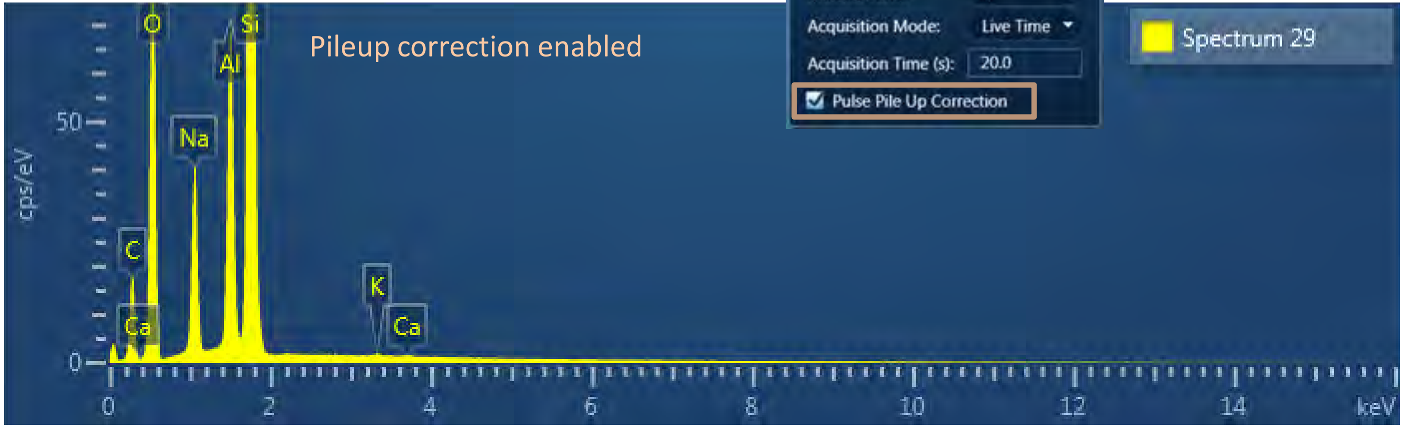
O: 48.76
Na: 8.6
Al: 10.34
Si: 32.03
K: 0.18
Ca: 0.09



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.

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

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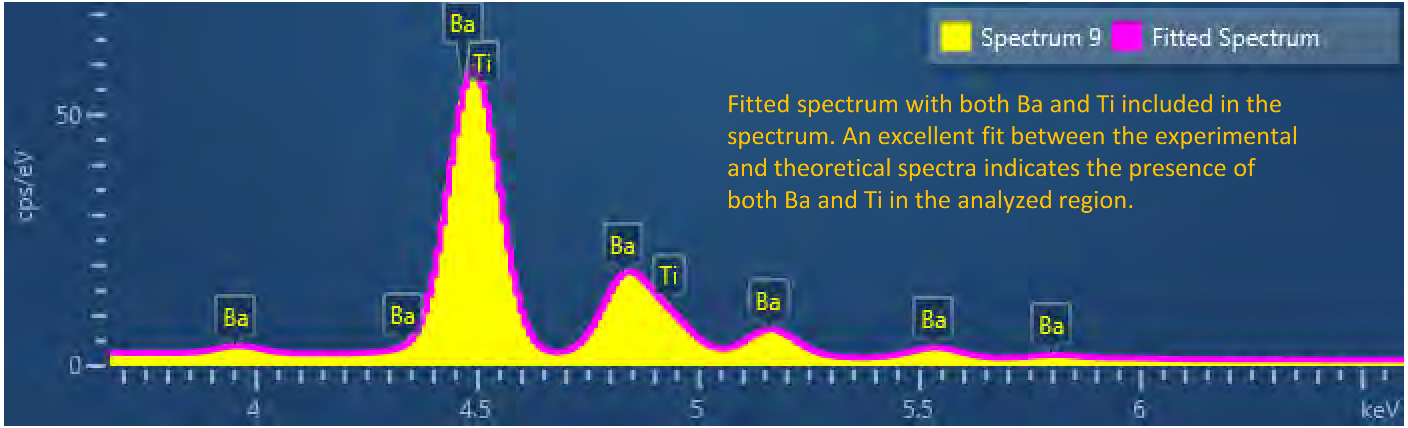
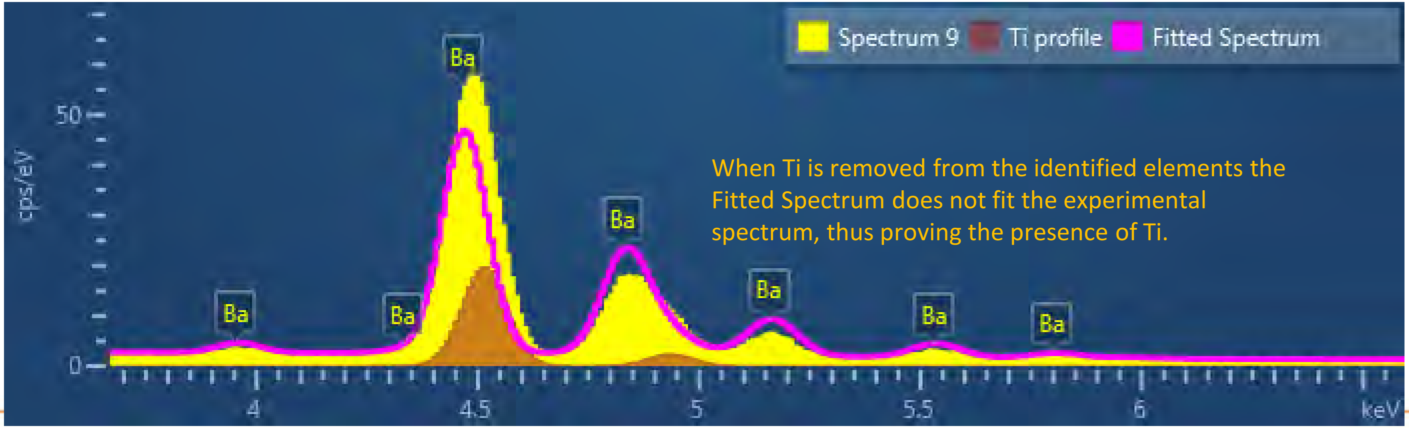
Benitoite: Certified Values

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

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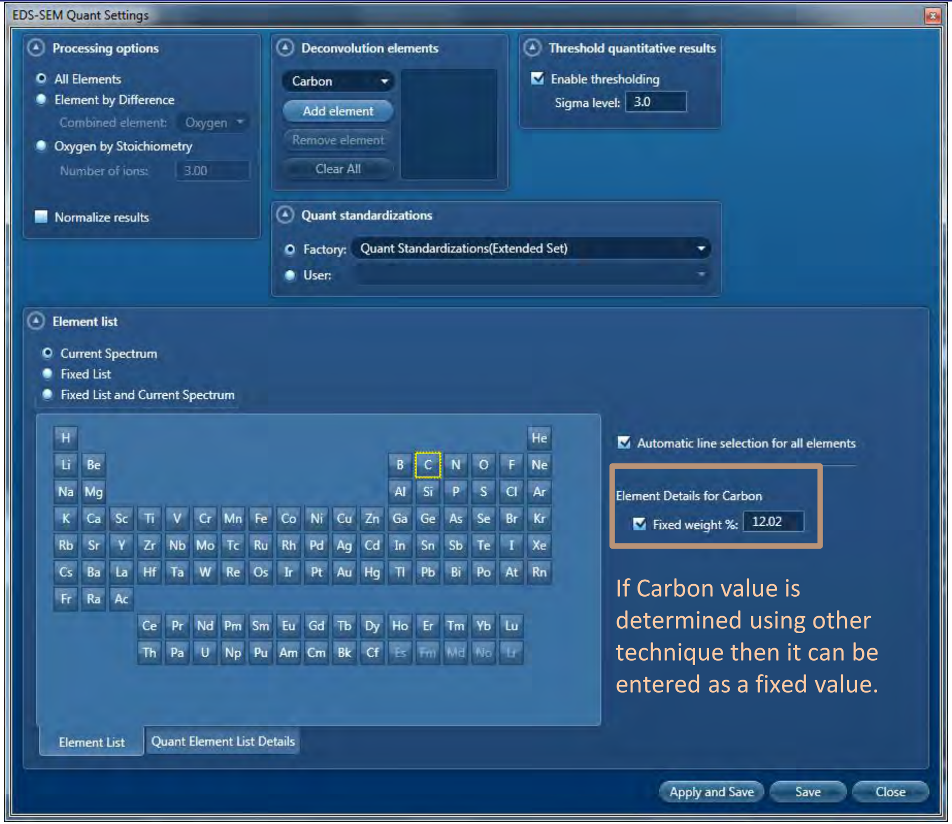
Calcite: Certified Values

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.

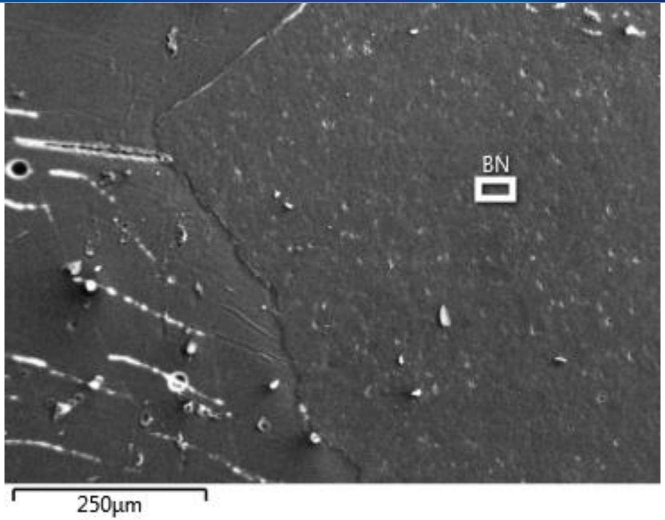
AZtec Quant

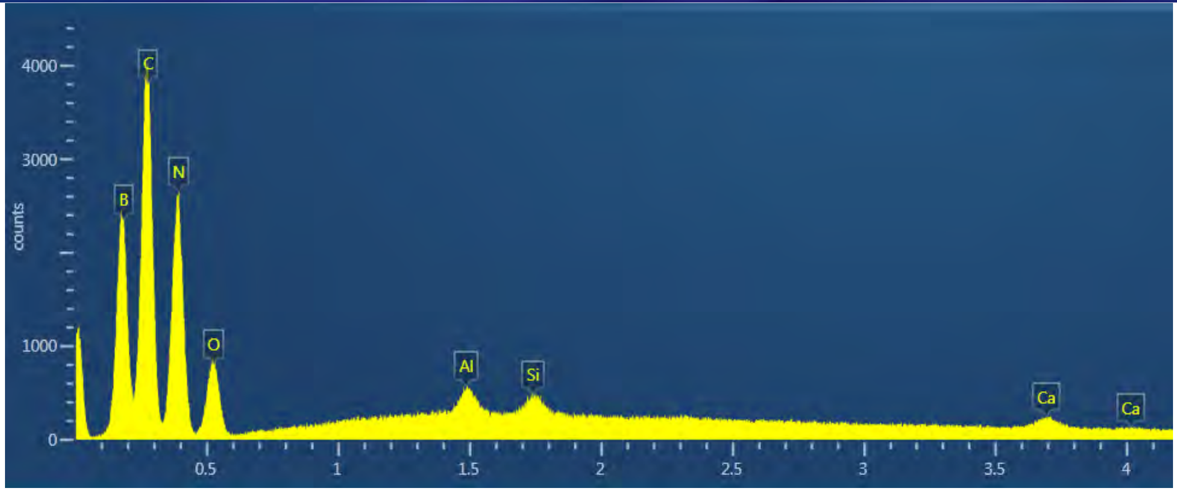
Calcite	Wt%
C (fixed value)	12.02
O	48.02
Ca	39.22
Total	99.26

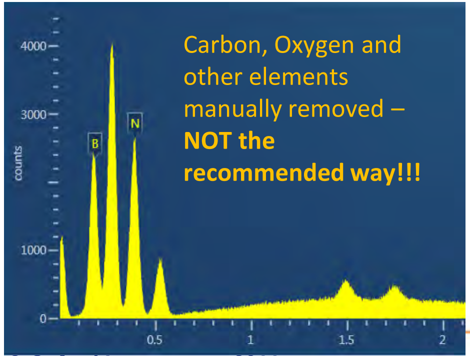


Boron Nitride with Carbon coating and Oxygen contamination

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







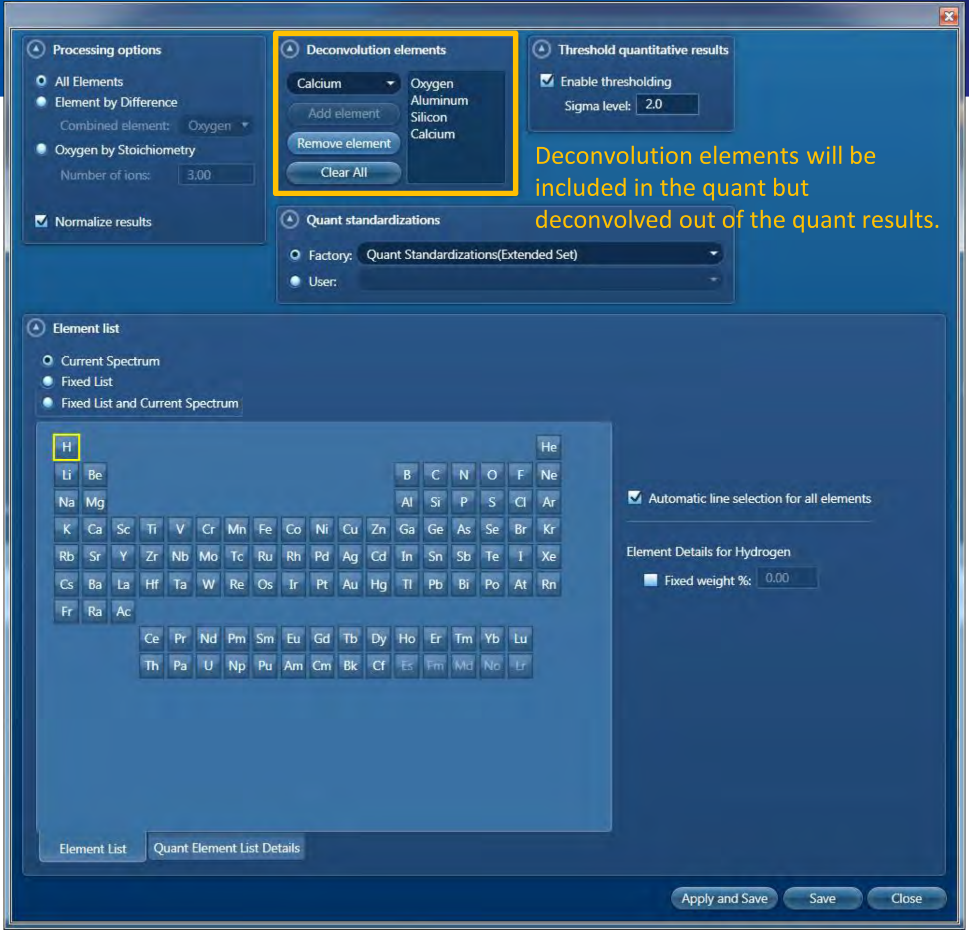
Manually removing surface coating and other contaminant elements from identification will NOT remove their corresponding x-ray contribution. This may lead to inaccurate quantitative analysis.

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.%



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

BN	Atomic %
B	49.93
N	50.07