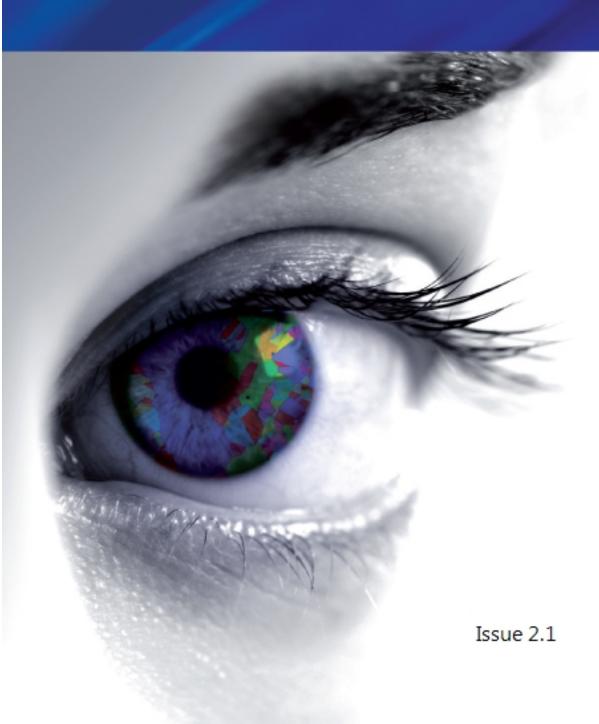
# **AZtec®** User Manual









# Copyright

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# What's New in AZtec 2.1

AZtec 2.1 release contains the following new functionality.

#### ED

- LayerProbe
- QuantLine
- AutoPhaseMap

#### **LayerProbe**

LayerProbe provides a non-destructive tool to measure the thickness and composition of surface and subsurface layers in thin-film structures. It integrates AZtec's robust quantitative-analysis routines with a powerful thin-film analysis engine for reliable results.

- Performs non-destructive analysis with minimal sample preparation
- Predicts solvability and optimum experimental parameters to enable reliable, precise measurements
- Analyses layers down to 1nm thickness\*
- Handles total structure thickness up to several microns\*
- Includes a simulation tool to generate simulated X-ray spectra of thin-film structures

See LayerProbe on page 270

#### **QuantLine (for SEM and TEM)**

Quant LineScan determines elemental concentration variations across a user-defined line on the sample.

- Results can be viewed in either:
  - Schematic linescans for each element, or
  - A table showing the full quantitative data for each point (Wt% or At%). \*
- Points can be binned (by 2,4, 8, 16 and 32)\*
- Spectra can be extracted from any point on the linescan for further investigation\*

See Acquiring linescans on page 391

## **AutoPhaseMap (for SEM and TEM)**

<sup>\*</sup>Precise limits depend on the sample and can be determined using the Solvability Tool supplied with the software.

<sup>\*</sup> This functionality is also available for Line and TruLine.



AutoPhaseMap is a new way to automatically create a map of the distribution of phases in a sample automatically during or after acquisition:

- Turns X-ray map data into Phase Map data in seconds.
- Calculates and displays:
  - Distribution of each phase
  - Spectrum and composition for each phase
  - Area fraction for each phase
- Finds phases and highlights elements that are present at only trace amounts.
- Finds phases at all size ranges including nano-materials.

See Analyze Phases on page 375

#### **EBSD**

- Reanalysis improvements
- Live Monitoring
- Phase fraction
- Improvements in image acquisition for forescatter detectors
- Drift correction using FSD images as references
- Export Raw Unprocessed EBSPs for cross-correlation application
- General performance and other EBSD improvements

#### **Reanalysis improvements**

The reanalysis functionality is now more versatile:

- Multiple reanalysis of any map, so settings can be repeatedly optimised if required.
   This includes changing the solver settings, and adding or removing phases.
- If SmartMap EDS data is collected with an EBSD map, the X-ray data is now included with that map when reanalysed.
- EDS data is viewed and reported together with the EBSD reanalyzed maps.
- Extract point EBSP and X-ray spectrum from any stored map data, including reanalyzed data.

See Reanalysis on page 465

#### **Live Monitoring**

This feature monitors orientation information (EBSP, unit cell, pole figures) in real time to validate data quality.

During acquisition, the orientation information quadrant of 'Construct Maps' provides the following information:

- The EBSP, with optional overlay of the pattern centre and solution simulation,
- The 3D unit cell and list of reflectors,
- Selected pole figure with orientation of current pixel highlighted.

The refresh rate is selectable, up to 1 pattern per second. Acquisition speed is not affected.

If a solution is available, the name of the phase, number of bands, MAD and Euler angles from the collecting point also display at the bottom of the orientation information quadrant.

Switch between monitoring live acquired patterns and stored patterns mode via a Toolbar. The live unprocessed and processed pattern are also available in Mini View.

See Optimize Solver on page 444

#### **Phase fraction**

When collecting an EBSD map, the system now gives the % phase fraction of the phases found:

- Provides a real-time overview of the phases, and % phase fraction in the sample.
- Avoids the need to export data to Channel 5 to see the phase fraction while collecting.

See Construct Maps on page 463.

### Improvements in image acquisition for forescatter detectors

Collect up to 6 individual FSD images simultaneously. The number of images collected depends on the number of independent diodes on the detector. Functions include:

- 6-channel FSD images and a mixed FSD image are acquired and viewed in one interface.
- Automatic optimization of each channel before scanning.
- Offset and gain of each channel can be adjusted manually during image acquisition.
- The weight of signal intensity for each channel in the mixed image is set to get the best image.
- Color may be selected for each channel image.
- Optimization is possible on a reduced area by selecting a reduced area map and the optimization factor applied to the whole map. This is useful for images where very dark regions (such as a hole) or very bright regions can distort the image optimization.

See Scan Image on page 418

#### **Drift correction using FSD images as references**



A new option allows the use of the FSD image as the tracking image to monitor and correct for drift.

On a tilted sample, the FSD image often offers the best image for monitoring drift because it can show more detail than the secondary electron image or backscattered electron image.

See AutoLock Settings on page 138

#### **Export Raw Unprocessed EBSPs for cross-correlation applications**

Raw 12-bit patterns as collected from the camera can be stored as TIFF files. These patterns are not corrected for background, magnetic field, or lens distortion. These patterns are preferred for cross-correlation applications such as CrossCourt.

- Unprocessed EBSPs are created during acquisition and collected in a folder with a selected local destination.
- Image format is uncompressed TIFF.
- If required, the processed EBSPs can also be saved.

See Acquire Map Data - Settings on page 457

### **General performance and other EBSD improvements**

- Monitoring function for extract tool in Phase ID and Optimize Solver.
- AutoID of the peaks in Spectrum Monitor, in Phase ID, and in the Mini View. Easy identification of the elements in the sample.
- The phase key in the phase map lists only those phases found in the map.
- Some improvements to naming consistency, so that the name of EBSD Detail dialog box is constant with the acquired map.

See Identify Phase on page 480, Optimize Solver on page 444, Acquire Map Data on page 450 and Construct Maps on page 463.

#### **General improvements**

- Report Template Generator
- Licensing improvements (AZtec and INCA)
- Network Licensing (AZtec and INCA)
- Interface modifications and GUI usability improvements

#### **Report Template Generator**

In addition to the comprehensive report templates available for reporting, a 'Report Template Generator' now allows the design of personalized report templates.

- User interface is easy to use and intuitive
- Word and Excel templates can be generated at the same time
- Generate multiple-page templates

- A4/Letter format
- Portrait and landscape layouts

See Generating your own report template on page 51.

## Licensing improvements (AZtec and INCA)

Extensive improvements have been made to the AZtec/INCA license activation/deactivation process.

- If a PC where you installed the software products does not have access to the internet, you need a unique unlock code from the internet-based licensing service to enable you to use the software product. This long code needed to be manually copied and taken to a PC with internet access, where it had to be manually pasted.
- There is now no need to manually copy and paste the long licence activation/deactivation codes from one PC to another.\*
- The Licence Manager automatically detects whether a USB stick is inserted in the PC, and stores or loads any codes relevant to the current activation/deactivation process.
- The License Manager utility is also copied to the USB stick for use on internet PCs without AZtec software installed.
- An extensive user guide is included in the AZtec help
- \* Note: except when there is no internet access and no possibility to bring storage media to and from the system PC

#### **Network Licensing (AZtec and INCA)**

An 'AZtec Network licence' is now available, which makes AZtec/INCA available to a large number of offline PCs.

- Network licences are designed for offline processing on multiple PCs.
- Requires installation of the AZtec software on the individual PCs and on the user's local network server.
- All installations on a single server need to be the same package (i.e. Standard, Advanced, Automate)
- Users on a PC connected to the local network server run the AZtec software locally, and it automatically communicates with the server to see if any licences are available. If a license is available, the AZtec software runs as normal. If all licenses have been checked out, the user receives a notification and has to contact the local administrator.
- The local administrator is notified when available licenses are running low.
- The 'Network license' also allows users to 'check-out' an available license for use away from the local server/network. This timed license deactivates when the period has elapsed, and returns to the local server for re-use.

Note: CHANNEL5 still requires a HASP dongle.



## **Interface modifications and GUI usability improvements)**

- AZtec has a new welcome screen that allows:
  - Quick access to recent projects
  - Quick access to demonstration data for training purposes
  - Quick access to Help
  - Creation of a new project with an associated profile
- The Map and TruMap icons have been modified, and moved closer to the Start and Stop buttons.
- Choice of Weight% and Atomic% in the MiniQuant.
- Map consistency: Users can now save not only map/colour selections directly to profiles from mapping navigator, but also the AutoLayer assignment so that the same colours are used when going from area to area.

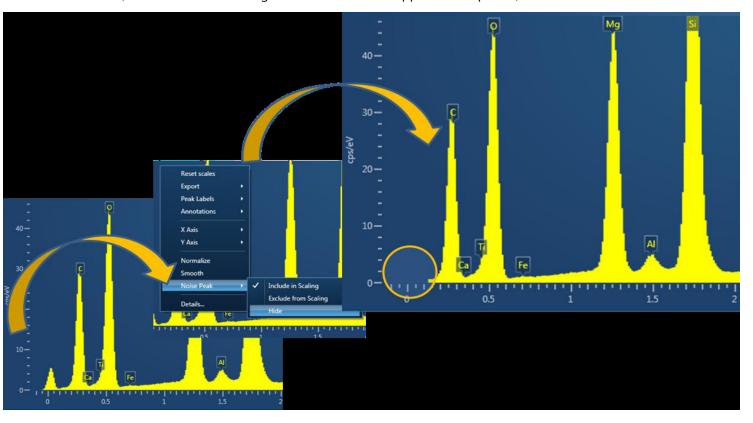
See Acquiring linescans on page 391, Construct Linescans on page 403, Acquire Map Data on page 363, and Construct Maps on page 370.

# What's New in Aztec 2.0 SP1

SP1 contains mostly bug fixes, localization/help updates, some usability modifications and one new functionality implementation.

### **New functionality:**

Users will now have the ability to hide the noise peak in their spectra via the right mouse button (this is a universal setting that once turned on applies to all spectra):



## A selection of bugs that have been fixed in SP1:

- AZtec 2.0 stops working with LN2 detectors systems.
- Irregular spectrum and mapping acquisitions were occasionally failing (when a user draws a free hand acquisition area for spectrum acquisition of mapping, acquisition would occasionally fail to start).
- User defined energy windows in EDS mapping were not saving to profiles.
- Wrong values of pre-tilted specimen holder were used during EBSD mapping (under certain circumstances system uses tilt value from previous map).
- Occasional connection errors to EBSD hardware.



# What's New in Version 2.0

Following are new features and enhancements included in this version of the software:

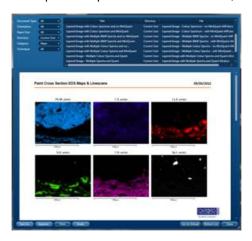
#### **AZtecTEM**

The main focus of AZtec 2.0 has been the introduction of AZtecTEM software.

## Improvements to reporting

The way the report templates are created has been improved:

- Ability to edit/create templates.
- Increased number of report templates to choose from (350+).

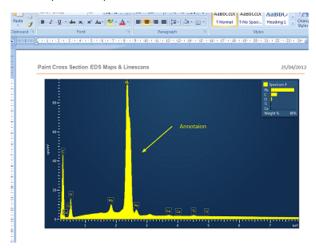


• Site report – Users will have the ability to print a combined report of all data objects in a project with just one click (EDS and EBSD data):



Company logo – The company logo can be changed for all templates, simply by copying the logo image file into the following directory - C:\Program Files\Oxford Instruments NanoAnalysis\AZtec\Reports.

Annotations on spectra – annotations on spectra are now saved in the project and will be visible on report templates:



See the link for details:

Report Results on page 44

#### **General user interface enhancements**

#### Software is now fully 64-bit, which means AZtec can:

- Utilize more of the host PCs RAM.
- Handle larger data sets (Software is now ideally placed to cope with the ever increasing demands for more and more data acquisition and storage).
- Run multiple memory hungry Programs simultaneously without compromising the performance.
- Note: It is important to note that the software will now not run on 32-bit operating system.

## **New navigator selectors**

- More robust user-friendly drop down selectors.
- Future proof (easily copes with any new additions of techniques or navigators):



#### **Batch export of data tree objects**

• Multiple data objects (images, spectra, IPF maps, etc...) can now be batch exported as bmp, gif, jpeg, png, tiff or wmp files:





• Multiple data tree images can be saved at the original resolution, even if each image has a different resolution:



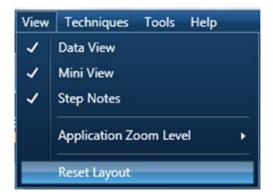
## **Settings panels**

- Settings panels can now behave in two different ways, depending on the users' preference:
  - Stay open until the cog or cross is pressed.
  - Stay open until a mouse click anywhere on the interface:



#### **Interface reset**

• Once activated the interface layout will go back to default:

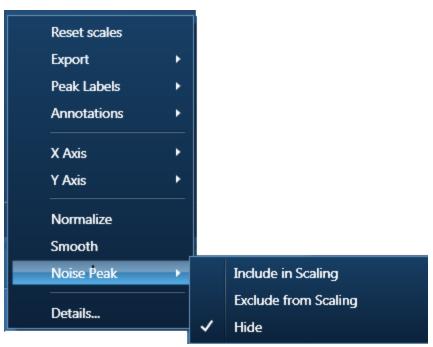


#### kV visible on Mini View



# EDS usability improvements and new functionality

- Noise Peak Option to Include/Exclude the noise peak in the scaling of the spectrum.
- You can hide the noise peak if you do not wish to display it in the spectrum:

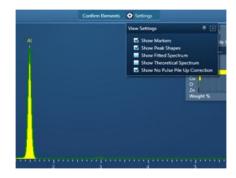


See the link below for details:



#### Context Menus - Spectrum Viewer on page 321

• Pulse Pile Up overlay in 'Confirm Elements' step:



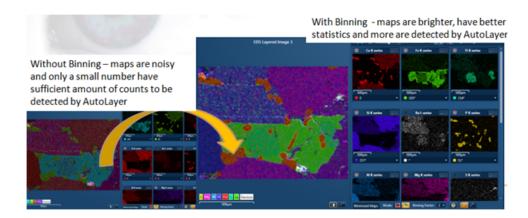
See the link below for details:

Confirm Elements - Settings on page 171

- Pixel Binning available for Maps and Linescans this not only improves the image quality/statistics on an image with low counts, it also allows the large data sets to be processed more easily.
  - No need to decide the pixel resolution when processing data:



• Benefits of binning are illustrated in the screen shot below:



See the link for details below:

How binning affects the quality of your data on page 373

- Compare step is now available in Guided Mode for ease of use.
- Variable spectrum acquisition termination.
  - Now the users have the ability to set their own termination criteria:



• Candidate element list is now collapsible:



## **EBSD** usability improvements and new functionality

#### **Solving Improvements**

- Developments in indexing algorithms. These changes make the indexing more robust so that it is easier to get good quality data.
- The EBSD indexing algorithm:
  - It is less sensitive to selection of number of bands.
  - It is easier to set up for data collection and to achieve a higher hit rate.
  - It is more effective at separating similar phases.
    - 'Band Width' function replaced by 'Grouping Function' accessed from the 'Describe Specimen Step.

See the links below for detail:

Optimize Solver on page 444

Configuring groups of phases on page 417

# Magnetic Field Correction for EBSP's collected using SEMs with Immersion or 'Semi-in Lens' objective lens

We now have the capability to correct EBSPs distorted by the magnetic field in the SEM chamber, this method uses the model described in US Patent 2006 / 0219903.

 This correction is designed to work on Hitachi and JEOL SEMs where the magnetic field is typically required for high resolution imaging. Please check with OI to confirm that your instrument is supported.



■ The solution requires collection of a distorted and undistorted pattern from the same point, and then calculates a correction factor.

See the link below for detail:

Magnetic Field Correction Setup on page 440

#### **Forescatter Detector Control Improvements**

- Additional functionality is included to aid the setting up and collection of FSD images.
- There are 2 default settings:
  - Atomic number contrast.
  - Orientation Contrast, as well as a customized setting.

See the link below for detail:

FSD Control Dialog on page 434

#### **General Performance & Other EBSD Improvements**

- Faster Reanalysis.
- Improved speed of the phase search in Phase ID.
- CTF (Channel Text File) export.
- TIFF export of EBSPs with both CPR and CTF format.
- EBSD Reporting templates, for mapping and Phase ID.
- EDS sum spectra can now be extracted from combined EDS/EBSD maps.

#### **Top Tips Movie**

A 'Top Tips' movie will be installed on to the PC desktop when version 2.0 software is installed. (The directory: C:\Users\Public\Documents\Oxford Instruments Nano-Analysis\Documentation).

The movie reveals the lesser known but useful functionality in the software:



### **Training CD**

A Training CD is now shipped with every system. The CD contains movies on the general operation of the software.



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# **Getting started**

The unique features of the user interface are described in the Application overview followed by the Guided tour of the Application. The details about the software licensing are covered in the frequently asked questions:

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# **Application overview**

There is a great deal of flexibility in the user interface. You can configure the workspace the way you wish to work and save a custom configuration (layout) to come back to every time.

The main application consists of the workspace in the middle area. It is supported by a side panel on the right containing Project Data, Mini View and Step Notes. You can remove each of these components from the view if you wish.

## **Dockable and Floating Window Panes**

The window panes are docked as the default configuration of the user interface. You can undock and free float them. Click and drag them wherever you want them in the interface or to a second monitor.

### **Re-sizable Windows and Dialogs**

Windows and dialogs can be resized by clicking and dragging their edges. The main application window can also be re-sized.

#### Global Menu Bar

There is a Menu bar near the top of the application window. It has the common menu items that you can access wherever you are in the application.

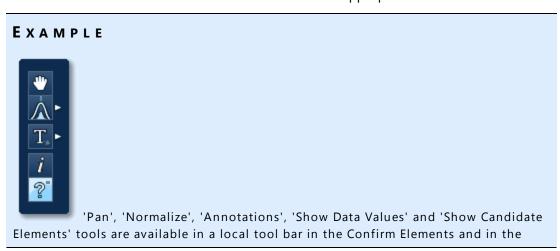


#### **Configurable Status Bar**

The Status Bar is located at the bottom of the application window. You can choose which hardware parameters you wish to display in there. A progress bar also shows up in the Status bar when you are importing or saving a project.

#### **Tool Bars**

Various useful tools are available in local tool bars where appropriate.





Acquire & Confirm windows in Point & ID on the right of the application window.

#### EXAMPLE

This tool bar is available in the Acquire and Confirm window in the Custom Map navigator. You can toggle on/off the user interface components from the display to your preferred layout.

#### **Context Menus**

Many useful menu items are available on the right click of the mouse in the application.

#### EXAMPLE

Image, Spectrum and Map viewers have many useful menu items. For example you can email a spectrum, image or map or append it to your report.

There are two modes of operation, Guided and Custom:

#### **Guided Mode**

The user interface components are laid out in Navigators that take you through your analysis from the Specimen through to the Report. You can navigate backwards and forwards as you wish. Each step has associated F1 (context sensitive) help and Step Notes to assist you at each stage of your analysis.

#### **Custom Mode**

In this mode, the key components are provided in one window. It allows you to perform the analysis in one workspace without having to move away from it. Each component can be undocked to have it free floating or dragged to another monitor to view it in full screen.

To provide you with more workspace, the Navigator area can be collapsed by pressing in the top right of the application window. Press \_\_\_\_ to restore the Navigator.



Below are some key user interface elements which make the software unique:

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# **Navigators**

The software has Navigators to guide the user through the analysis process. There are the following navigators in the software:

- Analyzer Guided on page 75
- Analyzer Custom on page 122
- Point & ID Guided on page 124
- Point & ID Custom on page 199
- Optimize on page 262
- Map Guided on page 362
- Map Custom on page 386
- Linescan Guided on page 389
- Linescan Custom on page 406
- EBSD Map on page 412
- Phase ID on page 473

# **Menu Bar**

There is a menu bar at the top of the application window containing several menu options. Each menu has several items which are described below.



File on the facing page

View Menu on page 9

Technique selector on page 9

Tools Menu on page 10

Help on page 12



# File Menu

Menu	Description
New Project	Removes any existing Projects, prompts to save and then opens a new Project as Project 1.
Open Project	Removes any existing Projects from the data tree, then opens an existing Project, prompts to save any existing Projects if required.
Add	<ul> <li>New Project: Adds a new Project and leaves any open Projects in the data tree.</li> </ul>
	<ul> <li>New Specimen: Adds a new Specimen in the Project that has focus.</li> </ul>
	<ul> <li>New Site: Adds a new</li> <li>Site in the Project that has focus.</li> </ul>
	<ul> <li>Existing Projectadds an existing Project and leaves any open Projects in the data tree.</li> </ul>
Remove Project	Removes the highlighted Project. If there is only one Project in the data tree, the Remove Project menu is dis- abled.
Save Project	Saves the highlighted Project.
Save Project As	Makes a copy of the high- lighted Project, prompts to enter a name and then opens it in the data tree and closes the existing Project.

Menu	Description
Import INCA Project	Imports an INCA Project, adds it to the Project list if existing Projects contain data otherwise replaces an existing "new Project".
Save As INCA Project	Saves the highlighted project as an INCA Project.
Export to CHAN- NEL5 Project	Exports the currently selected EBSD data as a CHANNEL5 project (CPR) file or a Channel Text File (CTF). You may also include EBSD patterns as TIFF files. This option is not available if you have stored EBSPs without solving.
Recent Projects	Allows to load from the recent Projects that you have been working on.
Close	Shuts down the application.



# View Menu

Menu	Description
Data View	It has the Current Site and Data Tree tabs.
Mini View	It has various views such as a live spectrum, image or acquisition progress bar.
Step Notes	Provides a brief description of the main features of each screen.
	Users can write and edit their standard operating procedures (SOPs) for future reference.
Application Zoom Level	Available options are Largest, Large, Medium, Small and Smallest.
Reset Layout	Restores the default layout on restarting the application.

# **Technique selector**

The techniques available are EDS and EBSD. Select the technique you wish to use by pressing the appropriate selector button on the top left of the main screen.

# **Tools Menu**

Menu	Description
Themes	Accessible Theme
	Oxford Instruments Theme
	Light Blue Theme
	Blue Theme
Languages	Default
	English
	French
	German
	Russian
	Chinese Simplified
	Japanese
User Profile	Settings available to create a user profile are:
	EDS Acquire Line Data Settings
	EDS Acquire Map Data Settings
	EDS Element Settings
	EDS Peak Label Setting
	EDS Quant Settings
	Scan Image Settings
	Specimen Tilt Settings



Menu	Description
Preferences	Preferences are saved per user.  Make your selection for the following:
	Auto Save
	Image Viewer
	INCA Image Export
	Reports
	Spectrum Viewer
	Status Bar
	Welcome Screen
Status Mes- sages	

# Help Menu

Menu	Description
Show Help Home Page	Launches the Help Viewer with table of contents in the left pane and useful links to internal and external sites in the right pane.
Show Step Help	Opens the Help Viewer. Pressing F1 on the keyboard loads the help page relevant to the active step of the navigator.
Show NanoAnalysis Advice	Opens links to 'How to' topics.
Launch User Manual	Opens the user manual as a PDF file.
Launch NanoAnalysis Encyclopedia	Opens the Encyclopedia in the Windows Explorer.
Oxford Instruments Website	Launches Oxford Instruments's home page.
About	Provides access to System Info, Assembly Info and Credits.

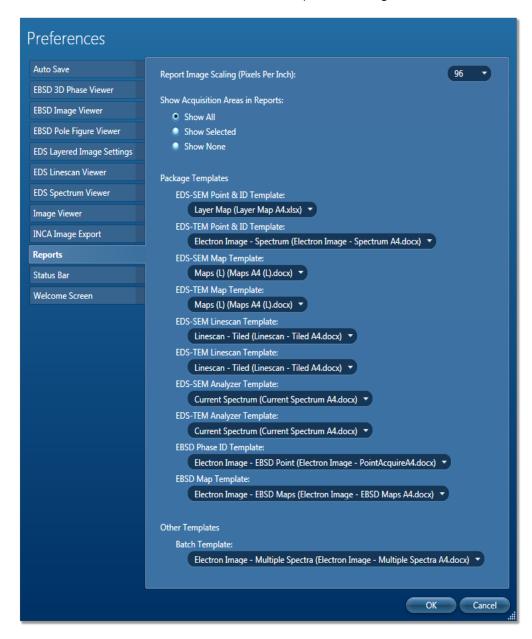


# **Preferences**

You can change the appearance of many parts of the software to suit your own needs by recording your preferences on the Preferences dialog. The software saves your preferences on the computer that you are currently using. When you run the software again, it has the appearance that you prefer. Your preferences will not be available if you run the software on a different computer or with a different logon on the same computer.

### **Setting your preferences**

1. Click the **Tools** menu, then click **Preferences** to open the dialog.



- 2. Click one of the tabs and change the settings as required. Many preferences are available.
- 3. After you change a setting, click **Save**. When you have finished your changes, click **Close**.

#### **Auto Save**

The software can automatically save the data in your current project at regular intervals. If the computer or software fails suddenly, you lose only a few minutes of your most recent work.

Field	Description
Save project after elapsed time	When selected, enable Auto Save.
Save every	Sets the number of minutes that will elapse between each automatic save. A suitable time is 10 minutes.

To save data at any time, click the **File** menu, then click **Save**.

#### **EBSD 3D Phase Viewer**

You can control the default display of the 3D Phase viewer. To temporarily change the current display, right-click it and use the context menu.

Field	Description
Show reflectors	Shows the reflectors as solid lines, edge lines, center lines, or shows no reflectors.
Unit Cell Color	Selects the color of the selected phase.
Max Reflectors	Selects the maximum number of reflectors that you can specify.
Show Unit Cell	Shows an image of the unit cell inside the sphere.
Use Perspective	Makes the image appear three-dimensional.
Use 4 Digit Indices	Selects the 4-digit index notation. Generally, the 3-digit index notation is used.



## **EBSD Image Viewer**

You can control the default display of the Electron Backscatter Diffraction (EBSD) image. To temporarily change the current display, right-click it and use the context menu.

Field	Description
Band Mode	Shows the bands as edges or center lines.
Sum Indices	Shows only those indexes where the sum of the indices does not exceed the number you enter here. For example, if the value entered is three, indices such as 100 and 111 may be labeled, if displayed, on the simulation. However indices whose sum is greater than 3 will not be shown. The value entered must be between 1 and 20.
Min Intensity	Determines how many bands are shown in the simulation. A value of 0 shows all bands. A value of 15 shows only those bands with an intensity greater than 15. The value must be between 0 and 100.
Extend Bands	Shows the bands extended beyond the band detection area.
Use 4 Digit Indices	Selects the 4-digit index notation. Generally, the 3-digit index notation is used.
Display Simulation	Shows a simulation of the solution as an overlay on the EBSP.
Display Band Detection Area	Shows the band detection area as a circle.
Display Bands	Shows the Kikuchi bands.
Display Pattern Center	Shows the pattern center as a green cross.

## **EBSD Pole Figure Viewer**

You can control the default display of pole figures and inverse pole figures. To temporarily change the current display, right-click it and use the context menu.

Field	Description
Projection Type	Offers a choice of the type of projection - equal area or stereographic.
Overlay Inverse Pole Figure on Sphere	Shows the location of the inverse pole figure within the sphere.
Show Great Circles	Shows the latitude and longitude lines on the sphere.
Use 4 Digit Indices	Selects the 4-digit index notation. Generally, the 3-digit index notation is used.

# **EDS Layered Image Settings**

You can control the default display of the layered image to include new X-ray map layers.

#### **EDS Linescan Viewer**

You can select the default line thickness (in pixels) for the linescans.

Field	Description
Default Line Thickness	Offers a thickness from Thin (0.5 pixels) to Thicker (4.0 pixels). The default line thickness is Thick.
	For any other thickness, select <b>User defined</b> , and move the slider bar.

#### **Spectrum Viewer**

You can control the default display of the Spectrum Viewer. To temporarily change the current display, right-click it and use the context menu.



Field	Description
Vertical Scale Type	<ul><li>Linear shows a regular spaced grid.</li><li>Logarithmic is useful for dis-</li></ul>
	playing data that has a wide range.
Show Horizontal Scale Show Vertical Scale	Shows the scales of keV and cps/eV.
Lock Vertical Scale Lock Horizontal Scale	Prevents the Pan tool from moving the graph.
Smooth Spectrum	Shows a smooth representation of the normally spiky spectrum.
Expand Mini Quant	Shows the expanded Mini Quant display in the top right corner of the spectrum.
Noise Peak:  • Include in Scaling	Shows the noise peak, and includes its value if you reset the scales (using the context menu option "Reset Scales").
■ Exclude from Scaling	Shows the noise peak, but excludes its value if you reset the scales (using the context menu option "Reset Scales").
■ Hide	Hides the noise peak. Specify the energy level in the "Cutoff Energy (keV)" box

## **Image Viewer**

You can control the default display of the Image Viewer. To temporarily change the current display, right-click it and use the context menu.

Field	Description
Rescale Image Mode	Changes the scale of the image. For a large image, select Fill Display with Image.

Field	Description
Show Acquisition Areas	Offers a choice of display for the acquisition areas.
Show Short Names	Shortens the labels for the acquisition areas to prevent them from overlapping and masking the text.
Show Header	Shows the label for the image in the header.
Show Color Bar	Shows the Color Bar below the Image Viewer. You can move the slider on the Color Bar to change the contrast of the image.
Show Scale Bar	Shows a micron marker below the Image Viewer.
Show Contrast/Brightness But- tons	Shows the Auto and Manual buttons at the bottom right corner of the image.
Show Annotations	Shows any annotations on the image.
Show Color Key	Shows a color key of the phases in the bottom left part of the Layer Image in the Map application.
Use Image Smoothing	Smoothes the lines in the image. If this check box is not selected, the image might appear more pixilated.

### **INCA Image Export**

If you save your project as an INCA project, you can export a Secondary Electron (SE) or Backscattered electron (BSE) image.

### Reports

You can select the layout, scale and content of your reports. A report is typically a Microsoft® Word document or a Microsoft® Excel spreadsheet. If you do not have those software applications installed, you can still view and print the reports with the Microsoft® viewers that are



supplied with the software. To edit the reports, you need Microsoft® Office 2007 (or later) software installed on your computer.

Field	Description
Report Image Scaling (Pixels Per Inch)	Sets the image scaling.
	The default is 96 pixels per inch, which is suitable for displaying on a computer screen. For high-quality printing, select a higher number.
Show Acquisition Areas in Reports	Offers a choice to show any acquisition areas.
	By default, reports show none.
Package Templates	Offers a choice of templates for each package. A report is a Microsoft® document or spreadsheet in one of several page sizes. Letter is popular in the USA.
	The default paper size, A4 is popular in Europe.
Other Templates	Offers a choice of templates for the batch export of data.

#### **Status Bar**

You can select the parameters (such as microscope voltage and noise levels) for EDS and EBSD that will appear in the Status Bar at the bottom of the software window.

#### **Welcome Screen**

You can choose whether the Welcome Screen appears when you start the software. The Welcome screen shows a list from which you can quickly select any of your recent projects.

#### See also

Status Bar

## **Status Bar**

The Status Bar displays the hardware status. It also allows the access to the Microscope Control, EDS detector and EBSD detector. A progress bar appears in the Status bar when you import, load or save projects.

The user selected parameters are displayed in the Status Bar at the bottom of main application screen. You can choose the parameters you wish to display on the Status Bar tab in the Preferences dialog. To access the Preferences dialog go to the Tools menu on the main tool bar and select Preferences:



Check the relevant check boxes to make your selection and press the Save button. The selected parameters will be displayed in the Status Bar.

You can access the Microscope Control by pressing Status Bar.



located on the right end of the



## See Also:

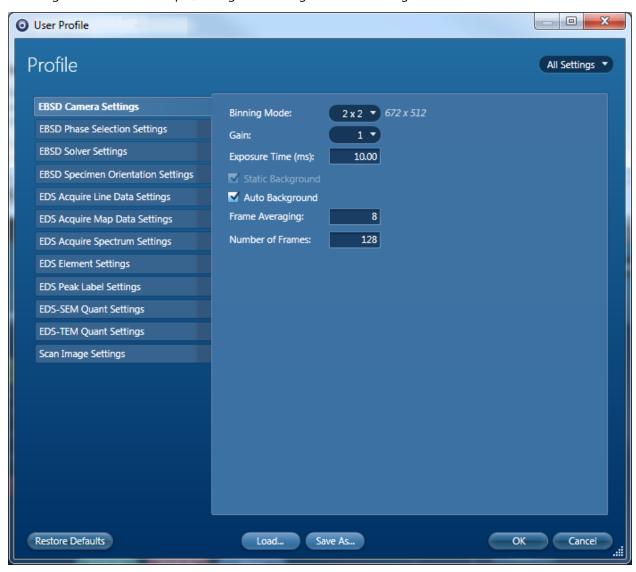
Microscope control on page 493

Microscope Parameters on page 496

# **User Profile**

A user 'Profile' contains all the settings needed to reproduce analytical results obtained on a previous date or by another user. The User Profile dialog is launched from the Tools menu on the main application menu bar.

To show only the settings for your type of analysis, select from the drop-down list in the top right corner. For example, change All Settings to EBSD Settings.



### EXAMPLE

"I am in charge of a service lab and have a number of users reporting to me. We perform many different types of analysis that we carry out, but do not have the luxury of assigning one person to do the same analysis all the time. So it is important that we have a way of reducing the variability in analytical results between different users. At the moment I make sure I check the users' settings before they start "



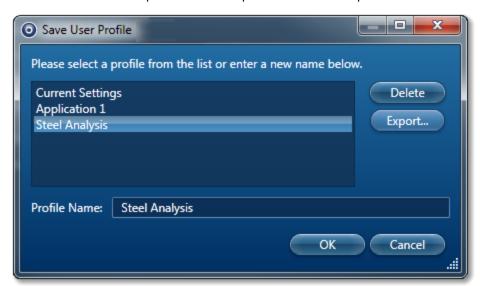
For each analysis type, all the relevant parameters can be saved in a profile, along with personalized step notes to instruct the users on the analysis. Subsequently anytime a user wishes to perform a particular type of analysis, all they have to do is load the relevant profile and all the appropriate settings will be changed and associated step notes will be loaded.



#### EXAMPLE

"My company has several sites all over the world, performing similar types of analysis. We need to ensure that each site carries out the same type of analysis in the same way, so we can compare results" .........."I need some help interpreting recently acquired data....if I send a project to Oxford Instruments Customer Support, how do I ensure that they see what I do?"

For both these cases the user profile can be exported via the user profile window:



The profile will be saved as a .config file and there will be user standards file (with .ois extension) if selected for use. See Managing Standardizations on page 267

Both files must be given to the person who you want to repeat or look at your data. The recipient will have to go to the Load Profile window and import the supplied profile.

#### EXAMPLE

For spectrum acquisition, you can specify the Number of Channels, Energy Range (keV), Process Time, Acquisition Mode and Acquisition Time (s) and save them in the User Profile.

When the User Profile dialog is opened, it stores the backup copy of the current settings.

Load... Press in the User Profile dialog to load a profile. Save As...

in the User Profile dialog to save a profile. OK

in the User Profile dialog to save the settings. This action will close the Press dialog and remove the backup copy.

Cancel Press to close the dialog. This action will restore the current settings from the backup copy.

There are separate tabs for different settings in the User Profile dialog. The details of the settings in each tab are described in the topics which can be accessed from the links below.

#### See Also:

Press

Scan Image - Settings on page 421

Acquire Line Data - Settings on page 252

Acquire Map Data - Settings on page 211

Acquire Spectra - Settings on page 313

**Element Settings below** 

Peak Labels on page 158

Quant Settings on page 187

LayerProbe Settings on page 301

# **EDS Element Settings**

The Element Settings tab in the User Profile dialog is provided with a periodic table. It enables you to define a list of Pre-defined Elements present in the specimen and the elements you wish to exclude from the AutoID routine.

When you press an element symbol in the periodic table, three buttons are enabled which are colored coded:

#### **Include**

#### **Exclude**



#### Clear

Defining an element from the periodic table is a cyclical process. Double-clicking on an element symbol will include this element. It will be colored green. Double-clicking it again will exclude this from the list and it will be colored red. Double-clicking on the symbol third time will clear this element from the list.

#### TIP!

For multiple element selection, hold down the Ctrl key, press on each element in the periodic table that you wish to select and then press the Include, Exclude or Clear button.

#### **AutoID Settings**

You can enable or disable AutoID during acquisition by checking or un-checking the 'Perform AutoID during acquisition' checkbox.

#### **AutoID Confidence Factor**

The default value for the Confidence Factor is set at 3. You can use the slider to set the value. The Confidence Factor is used to determine how AutoID behaves with regard to the sources of error.

#### **Map Element Details**

The 'Map Element Details' dialog allows you to configure the element maps. The default X-ray lines are used for element mapping unless you specify them. You can select the X-ray line for each element that you wish to map from the Map Element Details in the Element Settings tab or in the Construct Maps step.

You can define the energy window width for each element rather than using the default value.

You can select which elements to map and which ones to exclude.

#### See Also:

Auto ID Confidence Factor below

# **Auto ID Confidence Factor**

If the Confidence Factor is set to a high value, AutoID will find the most significant peaks but may miss small peaks that are close to the noise level. If the Confidence Factor is set to a low value, AutoID will detect small peaks but may pick up false positive identifications that are due to statistics or systematic errors.

By default, we set the Confidence Factor to 3 which corresponds to the "3-sigma" confidence level for a normal statistical error distribution.

The Confidence Factor is used to determine how AutoID behaves with regard to the sources of error. AutoID is designed to find a good combination of peak profiles that matches the spectrum and thus identifies the elements present in the specimen. When peaks overlap, the

proportion of constituent profiles is determined by least squares fitting to the sum of peak profiles. Counting statistics introduce fluctuations into the spectrum that are sometimes difficult to distinguish from genuine peaks. The statistical fluctuations introduce "random" errors that are equally likely to be positive or negative. When there are severe peak overlaps, it is even more difficult to distinguish genuine peaks from noise fluctuations.

In addition, chemical bonding effects and inaccuracies in peak profiles may mean that there is no combination of peak profiles that is an exact match to the spectrum, even when there is no statistical noise. If the peak profile is not perfect, this introduces bias or "systematic" error into the results.

If a fitted peak profile is much larger than the random or systematic errors, it is likely that the corresponding element is present in the specimen.

#### Note

To access the AutoID Confidence Factor, select User Profile from the Tools menu and then select the Element Settings tab. AutoID Confidence Factor is available in the AutoID Settings.



# **Support Panel**

The Support Panel is present on the right side of the application window. It has three components, Data View, Mini View and Step Notes. You can add or remove any of these components from the display by selecting the View menu on the Menu bar. You can also minimize, maximize or close each component from the display by pressing the relevant button present at the top right corner of each component.

To increase your work area you may wish to collapse the Support Panel by pressing the arrow button in the top right corner of the application. Pressing this again will restore the Support Panel.

#### **Data View**

Data View has two tabs, one for the Current Site and one for the Data Tree. For details see the Data View topic from the link at the end of this topic.

#### Mini View

In the Mini View you can choose to display a number of different views such as Electron Image, Spectrum Monitor, the Ratemeter or many others depending on the step.

#### **Step Notes**

Step Notes provides the first time user of a navigator with simple instructions on how to complete a typical work flow. It also provides a site administrator or user with the ability to write a standard operating procedure (SOP).

#### See Also:

Mini View on page 93

Step Notes on page 94

# **Data View**

The Data View panel is located on the right of the main application window, By default, it is always displayed. If it has been taken off the view, it can be restored by choosing the Data View from the View menu on the main menu bar.

Data is archived in a logical manner and can be directly viewed via easily recognizable icons.

Acquired data is automatically saved at the end of an acquisition. An auto save option can be enabled from the Auto Save tab of the Preferences on page 13 dialog on the Tools menu.

The Data View panel has two tabs, Current Site and Data Tree.

#### See Also:

Current Site on the facing page

Data Tree on page 85



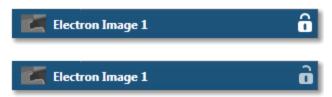
## **Current Site**

The Current Site shows the data for the currently selected Site in the Data Tree, plus the current acquisition and any pending acquisitions. The ordering of items in the Current Site is different to the Data Tree. The new data items are added to the end in the Current Site where as the Data Tree sorts the items under the Site by spectra, electron images and then maps.

The Current Site has some extra features:

#### **Electron Image**

Electron image has a lock/unlock icon. Click once to lock, then again to unlock:



If unlocked, subsequent electron image acquisitions in the same Site will replace the existing electron image.

Locking the Electron Image will prevent the image from being recycled.

#### **Current Acquisition**

Both Spectrum and Map acquisitions show a progress bar and a stop icon:



When acquiring EBSD data, pause/resume and restart icons are also shown. Progress information is also shown in the tool tip.

#### **Pending Acquisitions**

Spectrum shows a cancel icon:



Note spectra and map acquisitions can be queued.

#### See Also:

Data Tree on page 85

Data Tree Menus on page 38

## **Data Tree**

Data is archived in a logical manner and can be directly viewed via easily recognizable icons on the Data Tree. To access the Data Tree, select the Data Tree tab on the Data View panel.

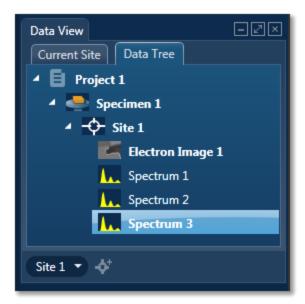
All open Projects and their contents are displayed in the Data Tree. Multiple Projects can be opened and shown in the Data Tree at the same time. If you have multiple Projects, Specimens or Multiple Sites in the Data Tree, you can easily get to your current site by pressing the Current Site tab.

When the application is started a default Project containing a Specimen and a Site is shown. As you acquire data, items are added to the Data Tree. The current items in the Data Tree are shown in bold.

Click on an item on the Data Tree to make it current.

#### Items on the Data Tree

The screen shot below shows an example of the main items in the Data Tree. Each item is described along with their icons below:





#### **Project**

Project is a top level container for data. Each Project is associated with a folder on the file system. The name of the folder is the same as the Project name. The Project folder contains a single file with an .oip extension and optional Data and Reports sub folders.

#### Note

When moving or copying project data ensure that the root project folder is moved/copied, not just the .oip file. The folder can be zipped using the standard



#### Windows compression utilities if required.



#### **Specimen**

Specimen represents the real specimen that you analyze and collect the data from, including images, maps and spectra. There may be many Specimens in a single Project. A Specimen may contain more than one Site.



#### Site

Site represents an area on the Specimen from where you acquire data such as images, spectra and maps. Site can hold multiple images, for example SE and BSE plus any imported images.

The analytical conditions such as kV, Magnification and Calibration are stored with the data.



#### **Electron Image**

Electron Image on each Site can be a secondary electron (SE) image, a backscattered electron (BSE) image, or a forward-scattered electron image. You can acquire two images simultaneously if suitable hardware is available.



#### **FSD Data**

This folder is the container for all FSD data. It contains electron images from each diode, and the FSD mixed image, which is the result of combining some or all of the FSE images.





Folder of images



Image from a single FSD diode



Mixed image



#### **Imported Image**

Any standard Windows Picture files can be imported into the Project for comparison or reporting. The file formats available are JPG, JPEG, BMP, PNG, WDP, GIF, TIF and TIFF. You can import an image using the context menu available from the Site.



#### **Spectrum**

Spectra are acquired from the areas defined on an electron image. Sum Spectra and Reconstructed Spectra are shown under the Map in the Data Tree.

You will see the following items in the Data Tree if you are acquiring element maps in the EDS application:





#### **Map Data**

Map Data is the container for a mapped area(s) in a Site. It can hold EDS Data, EBSD Data or both. One Site can contain more than one Map Data items. In the example above there are two items, Map Data 1 and Map Data 2.



#### **EDS Data**



EDS Data is the container for Map Sum Spectrum, Reconstructed Spectra, X-ray element maps, and Phase Images.



#### **Map Sum Spectrum**

The sum spectrum is calculated from the data acquired from all the pixels in the electron image.



#### **Reconstructed Spectrum**

You can reconstruct spectra from regions of maps or linescans.

#### X-ray Element Maps

The data can be processed as Windows Integral Maps or TruMaps (FLS maps). The Data Tree is populated with the appropriate maps on selection of the map processing option:



#### **Windows Integral Maps**

The standard element maps obtained from the counts in the element energy window including the background.



#### TruMap

The maps are corrected for peak overlaps and any false variations due to X-ray background.





#### **Phase Image**

Phase Image is the container for all the phase maps and their spectra. For example:





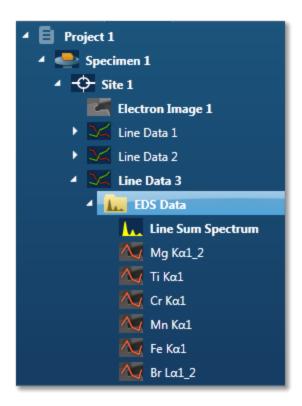
#### **Layered Image**

Layered Image is a composite image created from electron and X-ray map images.



#### **Linescan Data**

The data tree contains a Line item under the Site; this is the container for the line data. By default, this is labeled as 'Line #' where # is an auto-increasing number under the current site(Site 1) as shown below:



The Line item is the container for EDS Data. All linescans and the sum spectrum are contained within the EDS Data.



The Linescans can be processed as Windows Integral Linescans, TruLines or QuantLines. The Data Tree is populated with the appropriate Linescans on selection of the processing option:



Windows Integral Lines- The standard element linescans obtained from the counts in the element energy windows

including the background.

TruLine

The Linescans are corrected for peak overlaps and any false variations due to X-ray background.

QuantLine

The linescans are processed to show relative percentages of each element by weight or number

of atoms.

The label of the element linescan is composed of the element symbol followed by the lines series used for TruLine/Window Integral data analysis. For example Cr K $\alpha$ 1 is the label for a Chromium Linescan obtained from the K $\alpha$ 1 line.



Line Sum Spectrum

The sum spectrum is called Line Sum Spectrum. The region the spectrum comes from is visible on the electron image. This is the same region as where the linescan data is acquired from.



#### **EBSD Data Folder**

The EBSD Data folder is the container for the six Map components as shown in the screen shot below:



These components are described briefly with their respective icons:



#### **Band Contrast**

Band Contrast is an EBSP quality number, higher the number more contrast there is in the EBSP.



#### **Phase Color**

This component colors the pixels in the map based on which phase was identified. The color for each phase is defined in 'Phases for Acquisition'.



#### **Euler Color**

The Map component colors the map based on the Euler color scheme and will help to show different orientations within the map.

Euler 1= R

Euler 2= G

Euler 3= B



#### IPF X Color, IPF Y Color, IPF Z Color

The IPF color components color the pixels based on the orientation of the unit cell and chosen reference direction; x, y or z.

Note that the color key depends on the structure type so it is not always the easiest map to interpret.



#### **EBSD Layered Image**

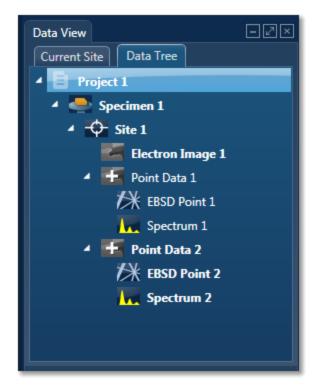
A Layered Image is a composite image created from electron and EBSD map images or element maps if EDS is present as shown in the screenshot above.



#### **Point Data**

In Phase ID, a Point Data node appears in the Data tree when spectra and EBSP are acquired from the points defined on the image:

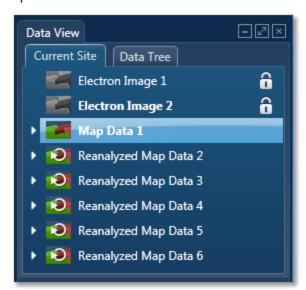






#### Reanalyze Data

If you have acquired an EBSD Map with stored EBSPs it is also possible to reanalyze a map region with new settings such as new solver settings or even solving by including different phases. Re analyzed map data is stored in the data tree as shown in the screen shot below:



#### See Also:

Current Site on page 29

Data Tree Menus below

Moving data to another PC on page 59

#### **Data Tree Menus**

Each item such as Project, Specimen and Site in the Data Tree has its own menu items. Right click with the mouse on a particular item to access the menu entries.

The menu entries for each item on the Data Tree are described below:

#### **Project**

There are two menu items for the Project, Remove and Edit Notes.

- Remove removes the project from the Data Tree. This option is disabled if only one Project is in the Data Tree.
- Edit Notes opens a dialog for editing Project notes.
- Details opens a dialog showing the Project label and Date/time when the Project was created.

#### TIP!

To rename a Project select Save Project As... from the File menu.

#### **Specimen**

There are four menu items for the Specimen, Rename, Delete, Edit Notes and Details.

- Rename allows to rename a Specimen.
- Delete deletes the Specimen from the Project.
- Edit notes opens a dialog for editing Specimen notes.
- Details Opens a dialog showing the Specimen Label, Specimen Orientation and Pretilted Specimen Holder. The Details dialog will also include the specimen coating information if you have selected it in the Describe Specimen step.

#### Site

There are six menu items for the Site, **Rename, Delete**, **Import Image, Batch Report, Print** and , **Email** 

- Rename allows to rename a Site.
- Delete deletes the Site from the Project.
- Import Image imports any standard Windows picture file for comparison or reporting.
- Batch Report this saves the Microsoft® Word or Excel report of all the data in the Site. It uses the report Batch Template selected in the Preferences dialog accessed from the Tools menu.



- Print this prints the Microsoft® Word or Excel report of the data associated with the Site.
- Email this helps to send the report via Email.

#### **Electron Image**

The menu items are:

- Rename renames the Electron Image.
- Delete deletes the Electron Image.
- Add to Layered Image adds an electron image to the current Layered Image.
- Add to Image Viewer- adds an electron image to the current FSD Mixed Image.
- Save As Saves the current electron image in Microsoft® Word or Excel report.
- Print prints the current electron image in Microsoft® Word or Excel report.
- Email sends the image via Email.
- Details opens the dialog showing the image details.

#### Note

You can view reports with the Microsoft® Word/Excel viewers supplied with your system. However, Microsoft® Office need to be installed for editing your reports.

#### Spectrum

There are six menu items for each spectrum on the Data Tree, Rename, Delete, Save As, Print, Email and Details.

- Rename- this renames the spectrum.
- Delete this deletes each spectrum.
- Save As saves the current spectrum in a user selected picture file format.
- Print prints the current spectrum as an image.
- Email sends the spectrum via Email.
- Details opens the dialog showing the spectrum details.

#### Note

Hold **Ctrl** and click on items one by one on the Data Tree for multi -select / de-select. Hold **Shift** and click on children one by one in a branch on the Data Tree for multi-select/de-select.

#### Map

There are six menu items for Map on the Data Tree, **Rename**, **Delete**, **Save As**, **Print**, **Email** and **Details**.

- Rename this renames the current map.
- Delete this deletes the current map.

- Save As saves the current map in a user selected picture file format.
- Print prints the current map.
- Email sends the current map via Email.
- Print prints the current spectrum as an image.
- Details opens the dialog showing the map details.

#### **Layered Image**

There are six menu items for the Layer Image, **Rename**, **Delete**, **Save As**, **Print**, **Email** and **Details:** 

- Rename this renames the Layered Image.
- Delete this deletes the Layered Image.
- Save As saves the current Layered Image in Microsoft® Word or Excel report.
- Print prints the current Layered Image in Microsoft® Word or Excel report.
- Email sends the Layered Image via Email.
- Details opens the dialog showing the Layered Image details.

#### **EDS Data**

There are two menu entries for the EDS Data, Rename and Delete

- Rename this renames the EDS Data.
- Delete this deletes the EDS Data.

#### X-ray Map

There are six menu items for each X-ray Map, **Rename**, **Delete**, **Save As**, **Print**, **Email** and **Details:** 

- Rename this renames the X-ray Map.
- Delete this deletes the X-ray Map.
- Save As saves the current X-ray map in Microsoft® Word or Excel report.
- Print prints the current X-ray map in Microsoft® Word or Excel report.
- Email sends the map via Email.
- Details opens the dialog showing the Layered Image details.

#### **EBSD Data**

There are four menu items for EBSD Data, Rename, Delete, Export... and Details...

- Rename this renames the EBSD Data.
- Delete this deletes the EBSD Data.
- Export exports the currently selected EBSD data as a CHANNEL5 project (CPR) file
  or a Channel Text File (CTF). You may also include EBSPs as TIFF files. This option is
  not available if you have stored EBSPs without solving.
- Details opens the dialog showing EBSD Data Details.



Each map components (Band Contrast, Phase Color, Eulor Color, IPF X, IPF Y and IPF Z) has six menu items:

- Rename this renames the selected component.
- Delete this deletes the selected component.
- Save As this saves the selected component as an image file.
- Print prints the selected component.
- Email sends the selected components via email.
- Details opens the dialog showing details of the selected component.

#### **Point Data**

There are two menu items for the Point Data, Rename and Delete.

#### **EBSD Point n**

There are six menu items for each EBSD Point as in the case of each map component described earlier.

#### Spectrum n

There are six menu items for each Spectrum as in the case of each map component.

## Mini View

The Mini View is an area of the Support Panel dedicated to the display of a number of different views which you can select depending on what data you wish to view. Views containing the current Electron Image , Spectrum Monitor or EDS Ratemeter are examples of such views.

#### **Electron Image**

The full field of view of the currently selected electron image is displayed here. It is often useful to refer to this image in steps where your application area is dedicated to displaying spectra or maps. For example you can view the electron image in the Mini View if you wish to view full size spectrum in the Acquire Spectra step.

The features of the electron image in the Mini View are:

The default state is full image with Scale Bar (micron marker). You can remove the Scale Bar from the display by de-selecting it from the image context menu.

The Context menu items are:

Show Acquisition Areas	Show All
	Show Selected
	Show None

Show Scale Bar	

Features such as Pan, Zoom and User Annotations are not available in the Mini View.

#### **Spectrum Monitor**

It provide a means for the user to see what X-rays are being detected at any given moment. It is useful for a quick survey of the specimen to find an area of interest for analysis. Spectrum Monitor uses the current spectrum acquisition settings with the additional setting of the refresh rate for monitoring the spectrum. This refresh time is referred to as the Buffer Size. The default is 20 but can be changed under the Settings for Spectrum Monitor in the Miniview. Increasing the Buffer Size corresponds to a longer refresh rate.

The settings in the Spectrum Monitor are:

Buffer Size: The default value is 20.

Number of Channels: 1024, 2048 or 4096

Energy Range (keV): 0-10, 0-20 or 0-40

The settings can be selected from the Acquire Spectrum step or Mini View. If you make a change in the setting in one place it is automatically updated in the other.

#### Ratemeter

It is very useful for setting up the microscope beam current while viewing the X-ray acquisition parameters:

Input Count Rate (cps)

Output Count Rate (cps)

Dead Time (%)

Ratemeter also displays the current Process Time and the Recommended WD (mm).

# **Step Notes**

Step Notes provide the first time user of a navigator with simple instructions on how to complete a typical work flow. It also provides a site administrator or user with the ability to write an SOP (Standard operating procedure).

A default editable set of notes are provided for each navigator step. The user can then overwrite these or add notes as required. A reset to default settings is available.

The notes are saved with the current user profile.

#### See Also:

Step Notes Editor on the facing page



# **Step Notes Editor**

The editor allows you to format text as you would using a word processor. You can cut, paste and copy text, left, right and central align the text, change the font size and style, undo and redo, select bulleted list or numbered list and paste in a picture.

# **Report Results**



You can quickly and easily generate reports from the data in your project. Each navigator has a default report template. A report is generated from this template when you click the Report Results button on the main navigation bar.

Once the report is generated, the software automatically displays the new report so you can view, edit or print it.

You can change the default report template by selecting it from the Report Preferences or accessing the Report Templates from the down arrow on the Report Results button.

# Managing your reports

You can click the down arrow on the Report Results button to open a menu of options that enable you to manage your reports. Some options are available only if Microsoft® Word or Excel is installed. In some cases, the appearance of the Report Results button changes to indicate the last action that you performed, so for example, you can send reports by email in quick succession

The following menu options affect individual reports:

Menu option	Appearance of the Report Results icon	Description
(Information at the top of the menu)	AAAA AAAAA - A AAAAA	Shows the name of the report template that is selected by default for the current navigator.  If no name is shown, select Report Templates, choose from a template from the list, and click Set As Default.
Save As		Asks for a file name, then saves the report as a Microsoft® Word or Excel file. The default location for your new report is in the Reports sub-directory of your current project.



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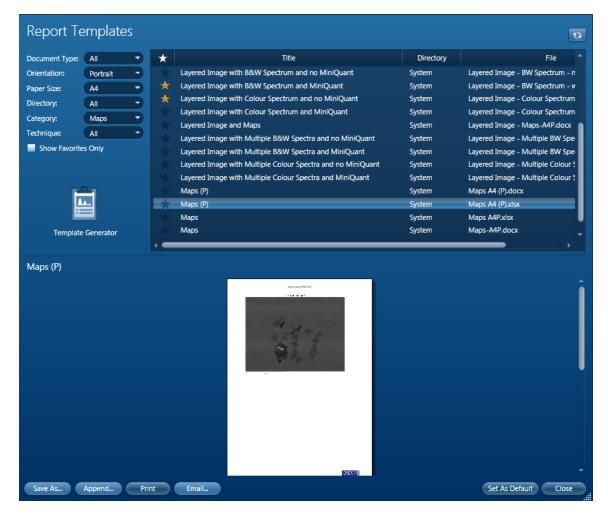
Menu option	Appearance of the Report Results icon	Description
Append (if Micro- soft® Word or Excel is installed)		Appends your report to the open file if a report is already open.  Asks for a file name if no report is open.
Print		Generates a report and prints it immediately on your default printer. As an alternative, you can use Save As, and print the document later.
Email	@	Sends the report using the default email package installed on your computer.

The following menu options affect site reports and report templates:

Menu option	Description
Site Report  (if Microsoft® Word or Excel is installed)	Generates a report about every type of information taken from the site. You can choose the format (Word or Excel), paper size, and file location.
Report Templates	Shows a list of the available templates. See the next section about the Report Templates dialog.

# **Report Templates dialog**

Reports are generated from the templates, which determine the look and feel of the final report. A set of templates are provided with the software. These templates include items which you might like to save, print or email, for example Quant Results and Spectra. You can view the complete set of templates by clicking the down arrow on the Report Results button, then selecting the 'Report Templates' option:



If Microsoft® Word or Excel is installed on your computer, the lower part of the dialog shows a preview of your report. You can click on the title of any report in the list above it to see different layouts. To change the magnification, right click the preview and use the context menu.

Note that the preview works only if your version of Microsoft® Word can save a document as XPS format. Office 2010 is supported, but Office 2003 is not. For Office 2007, see the File menu option, 'Save as'. If your Office 2007 does not have the XPS option, you can copy a 'plug-in' from the product DVD (in folder, Customer Support\Office2007). Alternatively, you can download from: http://www.microsoft.com/download/en/details.aspx?id=7

#### Filtering the list of templates

Initially, the list on the top right of the dialog shows all the available templates. To find a suitable template quickly, use the drop-down lists in the top left of the dialog to filter the long list and show fewer templates.

Menu	Description
Document Type	Shows only the templates that are in Microsoft® Word or Excel format.
Orientation	Shows only the templates that have portrait or landscape layout.



Menu	Description
Paper Size	Shows only the templates that are suitable for printing on a paper size of Letter or A4. Letter is popular in the USA. A4 is popular in Europe.
Directory	<ul> <li>Shows templates from all directories or only one directory.</li> <li>"System" shows the templates as supplied with the software. Unless more templates are created, you see templates only this directory.</li> <li>"Current User" shows your own templates.</li> <li>"All Users" shows templates you share with other users on the same computer, and possibly on the same network.</li> </ul>
Category	Shows only the templates that are suitable for images, maps or spectra.
Technique	Shows only the templates that are suitable for EDS or EBSD analysis.
Show Favor- ites Only	Shows only your favorite report templates. To mark a favorite, click the star in the column to the left of the title.

If extra templates have been created for you and other users, you might see more templates if you do not select System from the Directory drop-down list:

- "Current User" shows your own templates. They are available only when you logon to your user account.
- "All Users" shows templates you share with other users on the same computer, and possibly on the same network (depending on the network configuration).

### Using the dialog buttons

The buttons around the dialog allow you to print or email the report, for example.

Button	Description
O	Updates the list of templates here if you have added a new template, or changed a template design, or changed some configuration information.
	Opens a dialog where you can generate your own report template.

Button	Description
Save As	Asks for a file name, then saves the selected report as a Microsoft® Word or Excel file. Alternatively, you can double-click a template in the list. The default location for your new report is in the Reports sub-directory of your current project.
Append	Appends your selected report to the open file if a report file is already open.
Print	Generates a report and prints it immediately on your default printer. As an alternative, you can use Save As, and print the document later.
Email	Sends the report using the default email package installed on your computer.
Set As Default	Sets the selected template as your preferred default report template for the current navigator. For example, you can change the current template to Excel format instead of Microsoft® Word format.
	You can also change the default report template from the Tools menu by changing it in the Report Preferences.

# **Report Preferences**

The preferences used for generating reports are accessed via the Tools menu on the main application screen. Select Preferences and then select the Reports tab. Preferences allow you to specify how your images will look in your final reports and which Report Template is associated with each navigator.

The following dialog shows a typical choice of report templates. Your list of reports might be different because of the products that are installed.



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The resolution of images in your reports can be specified by setting the Dots per Inch (DPI) option. You can specify whether the area selections are shown in the Electron Images.

The Report Preferences also allow you to specify which Report Template is associated with Batch Reporting mode. This is useful for generating similar reports for many sites.

## Generating similar reports for many sites

The software has a Batch reporting feature which allows you to generate similar reports from any number of sites. For example you can generate a report containing only electron images and spectra from every site.

Decide which report template to use for generating your batch report. You need to do this only once. For example, you want reports of spectra always to be Word documents for printing later in Letter size:

- 1. On the Tools menu, select Preferences, and then select the Reports tab.
- 2. On the Reports tab under Batch Report, select a suitable template, for example: Image & Spectra Letter.docx.

To generate a Batch report at any time:

- 1. On the Data Tree, select one or more sites, then right click, and select Batch Report.
- 2. Select a folder for your reports, and click Select Folder.

One document is generated for each site. The documents are named according to the project name, the site name, and the current date and time.

## Compile a report on the fly

You can prepare your report as you acquire data. The image, spectrum and map viewers have a range of context menus which allow you to export live or stored data. You can copy images, spectra or maps to the clipboard and paste them into Microsoft® Word or Excel. You can manipulate images, spectra and maps using various settings such as width, height, aspect ratio and zoom before exporting to a third party reporting application. You can email or print an image, spectrum or map from the respective viewer.

### **See Also**

Context Menu - Report Templates below

Preferences on page 13

Changing the logo in your reports on the facing page

Generating your own report template on the facing page

## **Context Menu - Report Templates**

After you select a template for a report, you can change the magnification of the preview in the lower part of the dialog. Right click on the preview to see the following menu:

Menu option	Description
Increase Zoom	Increases or decreases the mag-
Decrease Zoom	nification of the preview.
	Alternatively, you can hold down the
	Control key and move the mouse wheel
	to zoom in and out.
Fit to Height	Adjusts the magnification of the preview so that a single page fits within the full area of the window.



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Menu option	Description
Fit to Width	Adjusts the magnification of the preview so that the page fits the full width of the window.

# Changing the logo in your reports

A standard report typically contains a logo, usually at the foot of the first page. If you prefer to use your own logo, you can replace the supplied logo. To make this change, you must have an administrator password.

- 1. In the folder where you installed the software (usually C:\Program Files), find the file logo.png in a subfolder called Reports.
- 2. Open the file in a suitable graphic editor, such as Microsoft® Paint.
- 3. Record the size of the original logo, for example, width x height =  $150 \times 75$ . (In Microsoft® Paint, this information is in Properties.)
- 4. In another folder, create your own logo.
  - If your logo is smaller than the original logo, surround your logo with white space to increase the image size.
  - If your logo is larger, surround it with white space to increase the image size, and then crop the white space to create an image that has the same aspect ratio as the original image, for example 400 x 200.
- 5. Name your logo file as logo.png, and copy it into the installation folder. You might be prompted for an administrator password.

All subsequent reports will display your new logo. If the Report Template dialog is currently open, and your new logo does not appear immediately, click Refresh List and wait a few seconds.

# Generating your own report template

Although the software includes a comprehensive list of report templates, you might sometimes need to create a new report template. The Report Template Generator allows you to design a new report template and publish it for personal use or to be shared with other users. You can use the new template straightaway for producing Microsoft Word and Excel reports. You can also save the template in an intermediate form, which allows you to continue to update the template design over time.

To create a report template:

1. In the Report Templates dialog, click the Template Generator button:



- 2. In the Report Template Generator dialog, enter a title for your template.
- 3. To ensure that you can easily find your template later, specify its properties by selecting the technique, category, paper size, and orientation.
- 4. Under Components, select the information that will appear in your report. For example, to add the date, go to General and click Date, which has this icon to denote it as a piece of text:



An area appears on the white page.

- 5. Move your cursor onto the page, then move or stretch the area, as required. You can also delete any unwanted component. For a context menu, right-click the area.
- 6. Repeat the previous steps to select further components and complete your template.
- 7. To add an extra page, click the "Add New Page" button, which is in the bottom right corner:



- 8. To generate the templates, click Generate (in the menu bar). In the "Template Generation Options" dialog, enter the details. If you will be the only user of this template, select "Current User". You can create Word and Excel templates.
- 9. Click OK to create the report templates. You cannot change the design of these report templates.
- 10. If you intend to change the design of your template later, click File > Save or File > Save As to create an intermediate (XML) file at a convenient location.
- 11. Click OK to close the dialog.
- 12. To close the Report Template Generator dialog, click "X" in the top right corner.
- 13. To quickly find your new report template, look in your list of favorites.

### Changing an existing report template

If you saved your previous design as an intermediate (XML) file earlier, you can open the file again, and continue working on your template design.

1. In the Report Templates dialog, click the Template Generator button:



- 2. In the Report Template Generator dialog, click File > Load.
- 3. Using the file browser, locate the intermediate (XML) file, and click Open.
- 4. Continue working on your design.



# **Themes**

There are different themes i.e., color schemes available to choose to display the User Interface for example:

Accessible Theme

Light Blue Theme

Blue Theme

## **Search Tool**

Enter one or more keywords in the Search Help field near the top right corner of the appli-

cation window or Help Home Viewer and press. The Help Viewer is displayed with links to a list of topics containing the keywords. When you click a topic link, the topic is displayed in the Help Viewer. You can go back to the list of topics by pressing the back arrow near the top left corner of the Help Viewer.

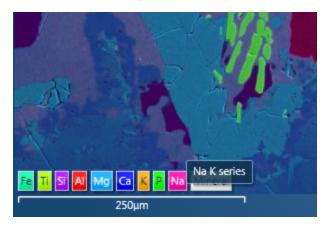
The keywords are highlighted in each topic . To remove all search highlighting, click this icon in the toolbar above:





# **Color key**

The color key is an optional feature on many images. This key (or legend) explains the colors. For more information, hover the cursor over each colored square. For example:



To show or hide the color key:

- 1. On the image, right-click to open the context menu.
- 2. Select View, then Color Key.

# FAQs about Software Licensing

### How can I activate my License Code?

You will need Internet access to Activate your License Codes. If the software is installed on a computer with Internet access you can simply enter the License Code into the License Manager and press the Activate button. The Licence Manager will then automatically send your License Code number to the remote licence server and, provided your License Code is valid, an Unlock Code number will be returned to the Licence Manager that will unlock the software you have purchased. Once Activated you will not need to do this again unless you want to Deactivate a License Code. Each software platform has its own separate License Code so you will need to do this for each platform.

# My system computer does not have Internet access so how can I activate my License Code?

If your system does not have direct access to the Internet you can still Activate your License Code using any other computer with Internet access.

See NLS Getting Started Card and User Guide for details.

### Can I install the software on more than one computer (PC or Laptop) at a time?

Yes you can. The terms of the license allow you to install the software on any number of PCs within your organization, but you will only be able to use the software on computers which have a valid License Code installed and Activated.

### What is a single license?

Unless you specifically ordered multiple licenses when you purchased your system, you will have a single licence which means you can Activate your software only on one computer at a time. (If you need to run the software on more computers you can Deactivate one computer and Activate another or you can buy additional licenses.)

### What is a multiple license?

If you purchased multiple licenses you will be able to run the software on the corresponding number of computers at the same time. For example, if you purchased five licenses you can Activate the software on a maximum of five computers, then if you try Activating a sixth computer you will be advised that all of your licenses are in use. Unless you specifically ordered multiple licenses when you purchased your system, you will have a single licence which means your software can only be Activated on one computer at a time. (If you need to run the software on more computers you can of course Deactivate one computer and Activate another or buy additional licenses.)

### I ordered multiple licenses so why have I only got one License Code?

The License Code contains details of the software you have purchased and are entitled to use and the number of licenses (computers) you are entitled to Activate at the same time. So if you purchased five licenses you will only receive one License Code but it will allow you to Activate five computers at the same time using the same License Code.



### What is a License Code?

A License Code is a unique 18-digit number which you will have received with your system. It contains encrypted details of the software you have purchased including the modules (functionality) and the number of licenses. When this number is entered into the software Licence Manager and the code is Activated the software functionality you purchased and are entitled to use is unlocked.

### Can I use my single software on a different PC or Laptop?

Yes, you can. If you have your software installed on another computer you will need to Deactivate the license on the existing computer, and then Activate the license on the other computer. Transferring the Active license to the second PC does of course mean that your software will no longer run on the first (Deactivated) computer (because you only have a single license). Each software platform has its own separate License Code so you will need to do this for each platform.

### Can I transfer my (single) license between computers more than once?

Yes, you can transfer (Deactivate and Activate) a license any number of times, but remember you can only Activate a single license on one computer at a time.

### Can I use my software on more than one PC or Laptop at the same time?

Although you can install your software on more than one computer at the same time, if you have a single license you can only activate it on one of them at a time. If you have a multiple licenses you can activate the license on as many computers as your license allows. If you want to use your software on more computer than you currently have licenses for you can easily buy additional licenses as required from your representative.

### I have forgotten the password. What can I do??

If you cannot remember a password when re-activating a license, you can request a new password to be sent to the email address that was given during registration.

This feature is available only if the mail server has been configured correctly.

**Tip**: Alternatively, you can register the license code with a new user name and password.

To set up the mail server:

- 1. On the licensing server, open the License Manager. For details, see section 1.4.
- 2. Select the "Settings" tab.
- 3. Enter the fully qualified domain name of the mail server, for example: mail.e-xample.com.
- 4. Click Save.

To request the new password:

- 1. On the registration page, click the "Forgotten password" link.
- 2. Follow the instructions on-screen.

### How do I upgrade my license?

If you are upgrading your license, you need to deactivate and reactivate the license to pick up the new modules. For details, see the sections in the Software Licensing User Guide. You will need to know the user name and password that was set at the time of registration.

If you forget the password, you can recover it by an email method. You need to know the email address that was registered with the user name. If you do not know the user name and password, you can activate the software product by registering with a new user name and password. You will need the license code.

### Note

You can access the License Manager from Tools in Oxford Instruments NanoAnalysis group of programs.



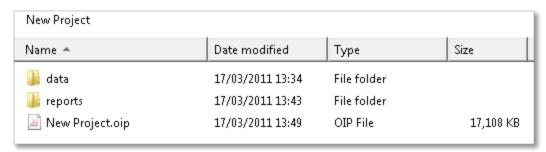
# Moving data to another PC

When you start a new Project, a Project folder is created. The Project folder contains a Project file with an '.oip' (Oxford Instruments Project) extension. By default the Project folder and Project file use the same project name, however it is possible to rename either of these provided the project is not open in the software.

A Project folder may contain two sub folders, 'data' and 'reports'. Acquired data is either stored in the '.oip' file or in the 'data' folder if additional data files are required. It is possible for a Project to not have a 'data' sub folder.

The 'reports' folder is the default location for saving any reports generated from the Project by the user. Reports can be saved in other locations where necessary. Before moving a project, please close any reports saved in the 'reports' subfolder.

An example of a project folder is shown in the screen shot below:



To move the project, either copy or move the Project folder to it's new location. This will maintain the folder structure, and allow the project to be opened from it's new location.

The Project can be opened on a second PC provided the software and an appropriate license are installed.

### To open the Project:

- Launch the software either from the shortcut on the desktop or from the Oxford Instruments NanoAnalysis folder in All Programs on the Windows Start menu.
- Select Open Project from the File menu on the menu bar.
- Browse to the Project folder.
- Select the Project file with .oip extension and press Open.
- The Project is loaded and the data items are populated in the Data Tree.



# **Getting Help**

Various elements of Help available in the software are described below:

### • Context Sensitive Help (F1)

The active workspace in the application has an associated help topic. Press F1 to access the help topic. Each help topic has useful links for further information.

### Step Notes

A default editable set of notes are provided for each navigator step. The user can then overwrite these or add notes as required. A reset to default settings is available.

### On-Line Help

There are six options available from the Help menu on the application menu bar:

### A. Show Help (F1)

This opens the Help Viewer with TOC in the left pane and the help topic on the

right. You can also launch the Help Home Page by pressing in the top right corner of the application window. The Home Page has the facility for searching the Help by entering the key words in the search field. It has links to the following four help items:

### Getting Started



This opens a page with links to topics to give you information about the main features of the user interface and Frequently asked questions about the software licensing.

### Oxford Instruments Website



This opens the Oxford Instruments Website.

### Support



This links to Oxford Instruments Support Website.

### NanoAnalysis Encyclopedia



This opens the Encyclopedia that contains help topics with interactive diagrams and movies. It provides the background information, theory and instrumentation of Microanalysis techniques.

### • Show Nano Analysis Advice

It has a number of topics which provide step by step advice on most frequently performed tasks.

### • Launch User Manual

This opens the User Manual in PDF format. The manual is supplied with a table of content and a comprehensive Index.

### • Launch Nano Analysis Encyclopedia

This is another way of launching the Encyclopedia.

### E. Visit Oxford Instruments Website

This launches Oxford Instruments website.

### About

The About dialog opens. It has Software version number and Copyright statement. You can access License information, System information, Assembly information and Credits.

### Search Help

The Search Help field is near the top right corner of the application window.

To find a single word or any combination of words, type each keyword here. To find a specific phrase, include double quote marks around the keywords. Then press:



The Help Viewer is displayed with links to a list of topics containing the keywords or the phrase. When you click a topic link, the topic is displayed in the Help Viewer. You can go back through the list of topics by pressing the back arrow near the top left corner of the Help Viewer:





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# **Setup for EDS**

There are two calibration routines available in the Calibrate step of Optimize navigator, Energy Calibration and Beam Measurement. To ensure that you understand when to calibrate your system and which calibration routine to use, read the comprehensive details below:

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### **Calibrate**



Two calibration routines available are Energy Calibration and Beam Measurement. For qualitative and normalized quantitative analyses, you will need to perform only the Energy Calibration part. However, if you are interested in accurate quantitative analysis with unnormalized results, you will need to perform the Beam Measurement routine too.

#### Note

You can access the Calibration routines from the Calibrate step in the Optimize navigator.

### **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.

See details on how to perform the routine below.

### **Beam Measurement**

If you are an expert user, and you need more than relative concentrations and 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

See details on how to perform the routine below.

### **Details on Energy Calibration**



# Why do we need to perform Energy Calibration?

- Ambient temperature changes will alter the gain of the system and this will affect where peaks appear in the spectrum. The exact peak positions and the resolution of the system are needed to precisely identify individual peak components in the spectrum.
- If peaks overlap, the relative sizes of individual peaks can only be calculated accurately if the width and position of each peak is accurately known. By measuring the position of one known peak, the system can be optimized to determine the position of all other peaks.

# How often should I perform Energy Calibration?

The electronics used is carefully designed to provide good temperature stability. Since a change of 10°C produces only a 1 eV shift in peak position, most routine analysis can be performed without re-optimizing peak position. However, if you need the software to resolve very closely overlapped peaks, you should perform Energy Calibration and re-optimize if the ambient temperature changes by a few degrees. With a good laboratory temperature control you may not need to optimize for many months.

## **How to perform Energy Calibration**

Energy Calibration requires the acquisition of a high quality spectrum from a suitable element from which details of the spectrometer gain are calculated and stored. One element can be used for both the Energy Calibration and Beam Measurement or you can use two different elements if you wish.

You can use an element as an energy calibration standard as long as the calibration peak is not overlapped by other peaks. There should not be any peaks within 100 eV of the calibration peak.

To perform Energy Calibration follow the steps:

- Select Energy Calibration from the Calibration Routine drop-down list.
- Select an element from the Calibration Element drop-down list.
- Get the element standard in the field of view of the microscope. Adjust the working distance to the recommended value and the beam current to achieve an optimum count rate.

- Press to start acquisition of the calibration spectrum. The current settings will be used to acquire the spectrum. A window will be painted across the peaks of the element X-ray line series.
- A progress bar near the top of the Calibrate window displays the estimated time for the completion of calibration spectrum acquisition.
- On completion of spectrum acquisition, a message is displayed asking you if you
  wish to perform the Energy Calibration. Press Yes if you wish to perform the Energy
  Calibration.

Note that the details of the Energy Calibration can be found in the Spectrum Details dialog in the Calculate Composition step when a spectrum has been quantified.

### **Details on Beam Measurement**

For Beam Measurement, you must use a pure element standard and it must be stable under the beam. See Calibration Element.

Note that the acquisition settings chosen in the Acquire Spectra step are carried across to the Optimize Navigator and vice versa. Changing the settings in one will automatically change the settings in the other.

# Why do we need to perform Beam Measurement?

The microscope beam current may vary with time. If we want to measure absolute concentrations, we need to make a comparison of intensity of a peak with that from a known material. If we measure a known material, we can then make accurate intensity measurements on unknowns, provided the beam current doesn't alter after the optimization.

# How often should I perform Beam Measurement?

■ If you wish to calculate un-normalized totals, the frequency with which you perform the optimization will depend on the stability of the beam current. Repeated measurement of a known standard will indicate whether the beam current is varying. The variation in the analysis total will be in direct proportion to the change in current since the last optimization.

## **How to perform Beam Measurement**

The Beam Measurement routine requires the acquisition of a high quality spectrum from a suitable element from which details of the beam current is calculated and stored. One element can be used for both the Beam Measurement and Energy Calibration or you can use two different elements if you wish.

To perform Beam Measurement follow the steps:



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- Select a calibration routine from the drop-down list of Energy Calibration and Beam Measurement.
- Select an element from the Calibration Element drop-down list.
- Choose the spectrum acquisition parameters from the Settings cog. For details see Beam Measurement- Settings.
- Get the element standard in the field of view of the microscope. Adjust the working distance to the recommended value and the beam current to achieve an optimum count rate.
- Press to start acquisition of the calibration spectrum. The current settings will be used to acquire the spectrum. A window will be painted across the peaks of the element X-ray line series.
- A progress bar near the top of the Calibrate window displays the estimated time for the completion of Beam Measurement routine. The progress is also displayed in the Current Site tab of Data View in the Support Panel.
- On completion of the Beam Measurement routine, a message is displayed to save it if you wish. If you decide to save it, the calibration spectrum is saved in your Project.

Note that the details of Beam Measurement can be found in the Spectrum Details dialog in the Calculate Composition step when a spectrum has been quantified.

### See also:

Calibration Element on next page

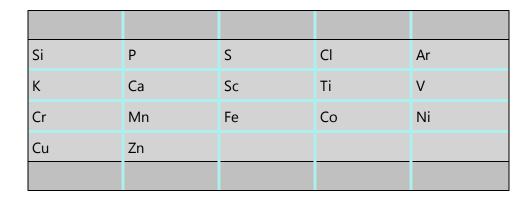
Calibrate for Beam Measurement- Settings on page 71

### **Calibration Element**

Select the element that you wish to use for calibration from the drop down list. The choice of element depends on whether you are doing Energy Calibration or Beam Measurement.

### For Energy Calibration

The elements available for Energy Calibration are tabulated below:



X-ray peaks often involve multiple lines and in order to achieve accurate calibration, a large peak with well-known line energies and intensities is required.

Therefore, for this reason we recommend that for energy calibration a pure element is used. If a pure element is not available compounds that have K lines that are not overlapped may be used instead. However, there may be some loss of accuracy because the line energy in a compound can sometimes be up to 2eV different from that in the pure element.

### For Beam Measurement

The system standards were calibrated using Co as the beam measurement element. Pure Co resists oxidation and polishes well and is therefore the most suitable choice to monitor beam current when you want to obtain accurate un-normalized or "absolute" estimates of composition. However, below 15kV, Co K is weakly excited and it is preferable to choose another pure element for monitoring. The system will then make suitable corrections to allow the following pure elements to be used:

Si	Ti	V	Cr	Mn
Fe	Co	Ni	Cu	Zn

If the beam measurement standard is oxidized, contaminated or has a rough surface, then this will have a direct effect on analysis totals.



# Calibrate for Beam Measurement- Settings

When you select Beam Measurement, the Settings icon is enabled. These are the same X-ray acquisition parameters which you have selected in the Acquire Spectra step. You can change the settings here if you wish. The parameters selected in the Optimize navigator will be used when you start acquiring spectra from your specimen.

### **Energy Range (keV)**

The appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited.

In the Auto mode, the system checks for the accelerating voltage selected on the microscope and sets a suitable energy range in the software.

### **Number of Channels**

Select the number of channels from the drop down list of 1024, 2048 and 4096 with which you display the spectrum. The number of eV/channel will depend on both the energy range and the number of channels you select.

In the Auto mode, the system checks for the energy range selected and sets the appropriate number of channels.

### **Process Time**

Select the Process Time from the drop-down list of 1 to 6. The Process time is the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process Time, the lower the noise. If noise is minimized, the resolution of the peak displayed in the spectrum is improved, in other words, the peak is narrower and it becomes easier to separate or resolve, from another peak that may be close by in energy.

If Default is selected, the Process Time is automatically set to a suitable value. There is a trade off between the Process time that is used, and the speed at which data can be acquired into the X-ray spectrum.

### **Total counts in spectrum**

The default value for 'Total number of counts in spectrum' is displayed here. This value is used to acquire a spectrum before the Beam Calibration is performed. You can enter the value that you wish to use.

The default value for the total number of counts is 600,000. It is the total number of counts in a Co spectrum acquired at 20 kV. It gives an approximate precision of 1% in the value of beam current over a spread of  $\pm 0.5\%$ .

### See Also:

Acquire Spectra - Settings on page 313

How to	
In this section users are provided step by step advice on the most tasks. The following tasks are described in details:	frequently performed
EDS Qualitative Analysis	72



# **EDS Qualitative Analysis**

In EDS, the qualitative analysis is the process of identifying elements present in a specimen. It involves acquiring a spectrum from the specimen and then identifying the peaks in the spectrum. Peaks can be manually identified to confirm elements using sophisticated tools available in the software. Once you have identified all the elements you can produce a Microsoft® Word or Excel report. You can email the spectrum to your customer directly from the spectrum viewer provided your system is connected to the network and it has appropriate software installed.

You can use the confirmed elements list for elemental maps and quantitative analysis.

Below is step by step guide for qualitative analysis to get the most accurate results out of your system with minimal effort:

- If you know what elements are present in your specimen and you only want to see peak labels or X-ray maps or X-ray linescans for those elements, then you can select them in the Pre-defined Elements tab in the Describe Specimen step of Point & ID, Map or Linescan navigator.
- If you are interested in seeing what other elements might be present, then select the AutoID option by checking the 'Perform AutoID During Acquisition' check box.

#### Note

Check the EDS Element Settings in the User Profile dialog (available from the Tools menu) to ensure that the elements that you have pre-defined in the Describe Specimen step are not in the Exclusion Elements List. Users can have different exclusion lists.

- Acquire an electron image from an area on your specimen in the Scan Image step available in the Point & ID, Map and Linescan navigators.
- Navigate to the Acquire Spectra step. Press
   from the entire image. If you wish to acquire a spectrum from a point or an area on
   the image, select the appropriate tool from the toolbar on the left side of the
   screen. For details see How to acquire spectra on page 148
- For details of spectrum manipulation and annotation see Acquire Spectra Toolbar on page 151
- You can see your MiniQuant results in a table or a bar chart during analysis. Real time Compare and instant MiniQuant options are available in the Acquire Spectra, Confirm Elements and Calculate Composition (Comparison of Results Two Spectra template) steps. You can compare your current spectrum to a control spectrum during acquisition. For further details see Compare Spectra & MiniQuant Results on page 195

- Peaks in the spectrum are identified and labeled automatically using the AutoID and Pre-defined elements lists. If too many peaks are close together, you can move the peak labels for clarity. Select the Annotation tool available in the toolbar on the left of user interface in Acquire Spectra and Confirm Elements steps. Click on the label to select it and then drag it to a new position. For details of configuring peak labels see Peak Labels on page 158
- Navigate to the Confirm Elements step to manually confirm the elements identified by AutoID (if selected). Extensive tools including Show Markers, Show Peak Shapes, Show Fitted Spectrum, Show Theoretical Spectrum (from the Settings) and Show Candidate Elements (from the toolbar on the left) are available to assist you in confirming elements manually. For details see Confirm Elements - Tools on page 174
- Once all elements in the spectrum have been identified and confirmed, you can email the spectrum to your customer or generate a Microsoft® Word or Microsoft® Excel report using the Export option available on the context menu on the Spectrum viewer. For further details of context menus available on the Spectrum viewer see Context Menus - Spectrum Viewer on page 321
- You can use the confirmed element lists to acquire element maps and linescans and perform quantitative analysis.

### Note

You can navigate to the Confirm Elements step from a quick link, within the Construct Maps and Construct Linescans steps to confirm an element for map and linescan acquisitions.



# **Analyzer - Guided**

Analyzer is a microscope centric application. X-ray spectra are acquired from the regions on the specimen scanned by the microscope beam. There are two modes of operation in the Analyzer application, Guided and Custom.

In the Guided mode, the Analyzer navigator has five following steps:

Describe Specimen	76
Acquire Spectra	96
Confirm Elements	115
Calculate Composition	117
Compare Spectra	120

# **Describe Specimen**



In this step there are two tabs, Summary and Pre-defined Elements.

### **Summary**

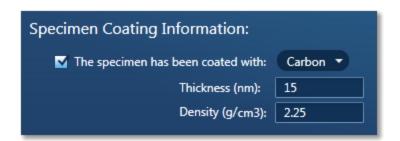
In the Summary view you can write notes on the Project and the Specimen present in the Project. (For convenience you can also copy images/diagrams and text from other documents/emails and paste into these windows). Notes are saved with the Project and you are allowed to edit notes in any step of the Navigator. It helps to capture the important information during the analysis. Click with the right mouse button on the Project or Specimen in the Data Tree and then select Edit Notes to write/modify the relevant notes.

You can add new Specimens to the current Project by pressing the New Specimen button:



Click on the Specimen in the Project Overview dialog. This action displays the 'Specimen Notes for Specimen 1' text box. Here you are provided with the text formatting tools. You are allowed to write notes about each Specimen and save them.

You can add coating information for each specimen (This information is used for the calculation of the quantitative results):



You have access to the periodic table for choosing the coating element for each Specimen. Two parameters, coating density and thickness exist which are required for a full coating correction to be applied for the calculation of the quantitative results. The default element is carbon, the defaults for thickness and density are 10 nm and 2.25 g/cm<sup>3</sup> respectively. Note that where appropriate the default density is that of the element at room temperature and pressure. The thickness and density may then be modified if required.

### **Pre-defined Elements**



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If you know what elements are present in your Specimen and you only want to see peak labels or X-ray maps for those elements, then you can select them in the Pre-defined Elements tab.

Press the Pre-defined Elements tab to access the periodic table:

If you wish to enable the AutoID option check the 'Perform AutoID During Acquisition' option.



Double -click on the element symbols that you wish to include in the analysis. All the included elements will be marked with the green color key in the periodic table. To save the Pre-

defined Elements in the current User Profile press . It means when you load the User Profile next time these elements will be included in the analysis.

If you have already created a User Profile with the Pre-defined Elements in the User Profile dialog press Load from Profile .

Pressing will deselect the Pre-defined Elements from the periodic table and they will not be included in the current analysis.

The peaks for the Pre-defined Elements if included in the analysis are labeled in the Acquire Spectra step. MiniQuant will display the quant results for these elements as Wt% or a bar chart.

The Pre-defined Elements will be marked as Pre-defined in the Confirm Elements list box in the Confirm Elements Step. There may be other elements in the Specimen which are identified by AutoID routine if the 'Perform AutoID during acquisition' option has been checked in the User Profile dialog.

### Тiр

Right click on the Project or Specimen in the Data Tree and select Edit Notes to write or edit notes in any step of the Navigator.

### See also:

Why are specimen coated? on the facing page

Coating Techniques on page 80

Element Lists on page 191

Data Tree on page 85

Mini View on page 93

Step Notes on page 94



# Why Are Specimen Coated?

- Samples are sometimes coated with a thin conductive layer prior to observation in the SEM to ensure that there is a good electrical path to ground. This prevents non-conducting specimens, as well as the oxides which are present on the surface of many samples, from charging under the electron beam.
- Carbon is generally the preferred coating material for microanalysis applications because of its minimal effect on the X-ray intensities.
- Gold is also commonly used for coating specimens since it increases the secondary electron yield providing improved SEM image quality. However, the number of peaks produced by the gold coating can cause overlap problems with the peaks of interest.
- A guide for the desirable thickness range of coatings is of the order of 5-30 nm.
- To minimize the effect of your coatings aim to make them as thin as is practically possible. Thick coatings will, for some samples, result in poor analysis.

### Effect of the Coating on the X-ray spectrum

The coating will have three main effects:

- 1. Energy loss of the primary electron beam as it passes through the coating.
- 2. Attenuation of the emerging X-rays.
- 3. The contribution of characteristic peaks to the X-ray spectrum. Thus, carbon coated samples will always display a carbon peak in the spectrum.

In the software, you can specify a coating element for a particular sample. During spectrum processing any X-ray peaks arising from this element are automatically **deconvolved** in addition to two other corrections (loss of X-ray intensity due to absorption of the emergent X-rays, reduction of primary keV). Application of these corrections is particularly important for low kV spectra in particular (  $\sim 5~{\rm keV}$ ). The final quantitative results could have significant errors if no such corrections were applied.

- Sample step input of additional parameters : coating density and thickness
- Standardization here the normalized area of the standard (I(std) / I(optimization)) is corrected for the coating.
- Full calculations the quantitative results are corrected for the coating.

Described below are the expected modes of operation for the locations described above.

### **Describe Specimen step**

In the Describe Specimen step, two parameters exist which are required for a full coating correction to be applied: coating density and thickness. This part of the program behaves in the following way:

- New project/specimen For example, select coating correction, the default element is carbon, the defaults for thickness and density are 10 nm and 2.25 g/cm3 respectively. Note that where appropriate the default density is that of the element at room temperature and pressure. The thickness and density may then be modified if required.
- Existing specimen If a coating element was selected this will remain as before with the density set to the default value and the thickness set to zero. With these settings the coating element will be deconvolved as before but no coating correction will take place. The full coating correction will be enabled by setting the thickness to a non zero value.

#### Standardization

The value of the Standard Correction Factor is adjusted to take account of the selected coating. The adjusted value will be used in the quant calculations.

### Quantification

During quantification for a coated specimen corrections will be made for the reduction in the effective kV for the primary beam when entering the specimen and the reduction in the emergent X-ray intensity due to the additional absorption of the coating layer. Thus for a particular specimen the values of the concentrations will increase when the coating correction is enabled and in general the effect will be most pronounced in the case of spectra acquired at low kV.

## **Coating Techniques**

Unless you are working with a variable pressure or low vacuum microscope, it is important that the sample you are analyzing conducts sufficiently so that it does not charge under the electron beam. It also may not be a problem if you are using very low voltages. There are a number of ways to reduce charging including coating your sample with a conductive material prior to observation in the SEM. Coatings are usually applied by using a vacuum evaporator, or a sputtering device. For X-ray microanalysis, we recommend that you use carbon since it does not generally interfere with elements of interest in the sample. Gold, usually used when a good secondary electron image is required, is not recommended for X-ray microanalysis, because of the large number of lines in the gold X-ray spectrum which may overlap with the lines of interest from your sample.

### **Evaporation**

The most common form of coating with carbon is using evaporation. A high current is passed through carbon rods, under vacuum. The heating effect causes the carbon to evaporate, and in turn, this is deposited as a thin film on the surface of the sample. The actual process of formation of a thin film is a complex process, and not fully discussed here.

- Ensure that one of the carbon rods is sharp. The end of one rod is usually flattened, while the other will be pointed.
- Place your sample in the evaporator.
- Evacuate the chamber.
- Once the desired vacuum has been achieved, usually about 1x10<sup>-5</sup> Pa, it is important to out gas the rods. This is done by heating them until they glow a dull red.
- The pressure in the chamber will rise, and the pressure should then be restored before the evaporation process.
- Pass a high current through the carbon rods.



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- The heating effect causes the carbon to evaporate and deposit a thin film on the surface of the sample.
- It is often useful to place a piece of white paper or filter paper beneath the sample to help you gauge the amount of carbon you deposit. Although the degree of coating you need will depend on the conductivity of the sample, as rule of thumb, when the paper appearance of the paper becomes light brown, this should be sufficient carbon.

### **Element Lists**

Any list of elements in the software can be split into the following three categories:

- Pre-defined Elements elements expected in specimen
- Identified Elements typically based on automatic peak identification (Auto ID)
- Fixed List used for Quantitative analysis

### **Pre-defined Elements**

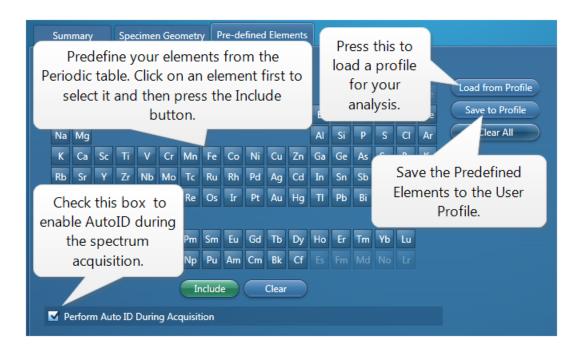
You may have prior knowledge of your Specimen and know what elements to look for.

### Examples

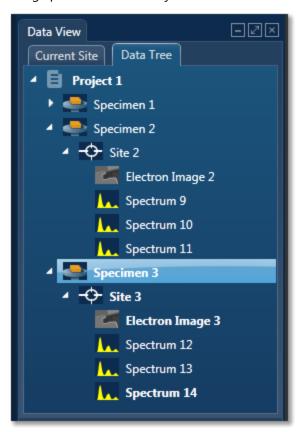
'I want to look for a particular list of elements. (I am not interested in any other elements)...I may want to see their labels on spectra, their X-ray maps or both....I want to see these even if the element is not present'.

' I know what's in my sample.... I want to look for a specific set of elements (I want to see these even if the element is not present.) .....but I would like to know if there is anything else in my sample too'.

You can define these elements in the 'Pre-defined Elements' tab in the Describe Specimen step. If you want to save the Pre-defined Elements to a profile you must first press 'Save to Profile' button, then save the profile via the drop down menu. When you want to analyze your Specimen, you can load this profile or another profile by pressing the 'Load Profile' button in the Describe Specimen step as shown in the screen shot below:



Note that the 'Pre-defined Elements' are saved with the current Specimen. Changing the 'Predefined Elements' will only update the Pre-defined Elements in the current Specimen. It will not update any existing Specimens in the Project.





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The current specimen is the one that you are presently analyzing/processing the data from. For example, in the screen shot above, Specimen 3 is the current specimen, Specimens 1 and 2 are the other specimens in the Project.

### **Identified Elements**

The 'Identified Elements will include:

- Any Pre-defined Elements
- Elements identified by Auto ID
- Any additional Elements identified manually

If the 'Pre-defined Elements have been specified, these will be included for identifying and labeling peaks in the current spectrum automatically.

Note that the 'Identified Elements' are saved in the Spectrum.

'Perform Auto ID During Acquisition' option is enabled by default and can be deactivate by un-checking it in the Describe Specimen step as shown in the screen shot above. You can then AutoID at any time by pressing the button.

Additional peaks in the spectrum can be identified manually by using the 'Show Candidate Elements' tool in the Confirm Element step. Click on the question mark icon to select the Show Candidate Element tool. Position the cursor at the center of a peak by double-clicking with the mouse. The list of elements spectra corresponding to the energy at the cursor is displayed in the panel on the right. By highlighting an element in this list, you will see the markers showing all the lines for this element.

Note that the 'Identified Elements' will be quantified if you have selected the Current Spectrum, or the Fixed List and Current Spectrum Element List in the Quant Settings in the Calculate Composition step or EDS Quant Settings in the User Profile dialog.

### Note

EDS Quant Settings are available in the User Profile Dialog accessed from the Tools menu. These settings are also available from the Calculate Composition step.

### **Fixed List**

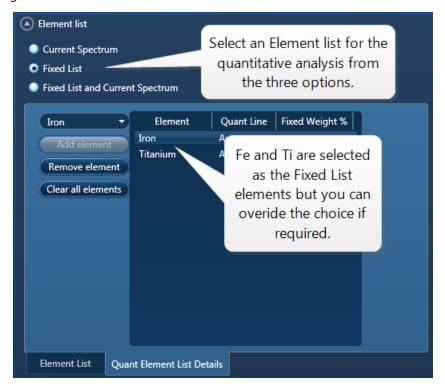
The elements in the 'Fixed List' are defined in the Quant Settings dialog which is available in the User Profile and the Calculate Composition window.

Note that the Fixed List is only used for calculating composition in quantitative analysis.

### Example

'I want to do quantitative analysis on my glass samples and want to compare results from one batch to another batch. I am always looking for the same specified list of elements'.

You can specify the Element List for Quant from the three available options in the Quant Settings dialog as shown in the screen shot below:



### **Current Spectrum**

This list includes the Pre-defined Elements and elements identified by Auto ID and manually using the Candidate Element tool.

### **Fixed List**

You select the Fixed List option if you know what elements you wish to quantify. Choose the elements from the drop-down list as shown in the screen shot above.

### **Fixed List and Current Spectrum**

To quantify the elements in the above two lists, select the Fixed List and Current Spectrum option.

MiniQuant results table will clearly display which list is being used. A lock icon will be displayed against the 'Fixed List' elements as shown in the screen shot below:





In this example, Fe and Ti are selected in the Fixed List. The rest of the elements in the chart results are from the Current Spectrum because the Element List selected for quantification was 'Fixed List and Current Spectrum'.

Note that the 'Fixed List' is saved in a User Profile.

#### See also:

Describe Specimen on page 76

Acquire Spectra on page 350

Confirm Elements on page 353

Calculate Composition on page 185

User Profile on page 22

# **Data Tree**

Data is archived in a logical manner and can be directly viewed via easily recognizable icons on the Data Tree. To access the Data Tree, select the Data Tree tab on the Data View panel.

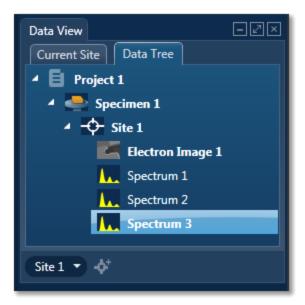
All open Projects and their contents are displayed in the Data Tree. Multiple Projects can be opened and shown in the Data Tree at the same time. If you have multiple Projects, Specimens or Multiple Sites in the Data Tree, you can easily get to your current site by pressing the Current Site tab.

When the application is started a default Project containing a Specimen and a Site is shown. As you acquire data, items are added to the Data Tree. The current items in the Data Tree are shown in bold.

Click on an item on the Data Tree to make it current.

### Items on the Data Tree

The screen shot below shows an example of the main items in the Data Tree. Each item is described along with their icons below:





### **Project**

Project is a top level container for data. Each Project is associated with a folder on the file system. The name of the folder is the same as the Project name. The Project folder contains a single file with an .oip extension and optional Data and Reports sub folders.

### Note

When moving or copying project data ensure that the root project folder is moved/copied, not just the .oip file. The folder can be zipped using the standard Windows compression utilities if required.



# Specimen

Specimen represents the real specimen that you analyze and collect the data from, including images, maps and spectra. There may be many Specimens in a single Project. A Specimen may contain more than one Site.



### Site

Site represents an area on the Specimen from where you acquire data such as images, spectra and maps. Site can hold multiple images, for example SE and BSE plus any imported images.



The analytical conditions such as kV, Magnification and Calibration are stored with the data.



### **Electron Image**

Electron Image on each Site can be a secondary electron (SE) image, a backscattered electron (BSE) image, or a forward-scattered electron image. You can acquire two images simultaneously if suitable hardware is available.



### **FSD Data**

This folder is the container for all FSD data. It contains electron images from each diode, and the FSD mixed image, which is the result of combining some or all of the FSE images.





Folder of images



Image from a single FSD diode



Mixed image



### **Imported Image**

Any standard Windows Picture files can be imported into the Project for comparison or reporting. The file formats available are JPG, JPEG, BMP, PNG, WDP, GIF, TIF and TIFF. You can import an image using the context menu available from the Site.



### **Spectrum**

Spectra are acquired from the areas defined on an electron image. Sum Spectra and Reconstructed Spectra are shown under the Map in the Data Tree.

You will see the following items in the Data Tree if you are acquiring element maps in the EDS application:





# **Map Data**

Map Data is the container for a mapped area(s) in a Site. It can hold EDS Data, EBSD Data or both. One Site can contain more than one Map Data items. In the example above there are two items, Map Data 1 and Map Data 2.



### **EDS Data**

EDS Data is the container for Map Sum Spectrum, Reconstructed Spectra, X-ray element maps, and Phase Images.



### **Map Sum Spectrum**

The sum spectrum is calculated from the data acquired from all the pixels in the electron image.



# **Reconstructed Spectrum**

You can reconstruct spectra from regions of maps or linescans.

# X-ray Element Maps



The data can be processed as Windows Integral Maps or TruMaps (FLS maps). The Data Tree is populated with the appropriate maps on selection of the map processing option:



### **Windows Integral Maps**

The standard element maps obtained from the counts in the element energy window including the background.



# TruMap

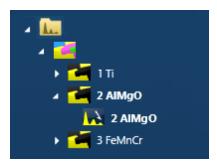
The maps are corrected for peak overlaps and any false variations due to X-ray background.

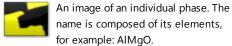




# **Phase Image**

Phase Image is the container for all the phase maps and their spectra. For example:







A spectrum extracted from all the points in a phase.



# **Layered Image**

Layered Image is a composite image created from electron and X-ray map images.



### **Linescan Data**

The data tree contains a Line item under the Site; this is the container for the line data. By default, this is labeled as 'Line #' where # is an auto-increasing number under the current site(Site 1) as shown below:



The Line item is the container for EDS Data. All linescans and the sum spectrum are contained within the EDS Data.

The Linescans can be processed as Windows Integral Linescans, TruLines or QuantLines. The Data Tree is populated with the appropriate Linescans on selection of the processing option:



Windows Integral Lines- The standard element linescans obtained from the counts in the element energy windows including the background.



TruLine

The Linescans are corrected for peak overlaps and any false variations due to X-ray background.



QuantLine

The linescans are processed to show relative percentages of each element by weight or number of atoms.

The label of the element linescan is composed of the element symbol followed by the lines series used for TruLine/Window Integral data analysis. For example Cr K $\alpha$ 1 is the label for a Chromium Linescan obtained from the K $\alpha$ 1 line.



Line Sum Spectrum

The sum spectrum is called Line Sum Spectrum. The region the spectrum comes from is visible on the electron image. This is the same region as where the linescan data is acquired from.





# **EBSD Data Folder**

The EBSD Data folder is the container for the six Map components as shown in the screen shot below:



These components are described briefly with their respective icons:



### **Band Contrast**

Band Contrast is an EBSP quality number, higher the number more contrast there is in the EBSP.



### **Phase Color**

This component colors the pixels in the map based on which phase was identified. The color for each phase is defined in 'Phases for Acquisition'.



### **Euler Color**

The Map component colors the map based on the Euler color scheme and will help to show different orientations within the map.

Euler 1= R

Euler 2= G

Euler 3= B



# IPF X Color, IPF Y Color, IPF Z Color

The IPF color components color the pixels based on the orientation of the unit cell and chosen reference direction; x, y or z.

Note that the color key depends on the structure type so it is not always the easiest map to interpret.



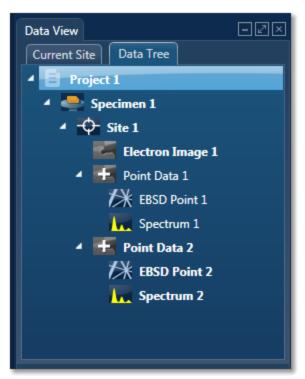
# **EBSD Layered Image**

A Layered Image is a composite image created from electron and EBSD map images or element maps if EDS is present as shown in the screenshot above.



### **Point Data**

In Phase ID, a Point Data node appears in the Data tree when spectra and EBSP are acquired from the points defined on the image:

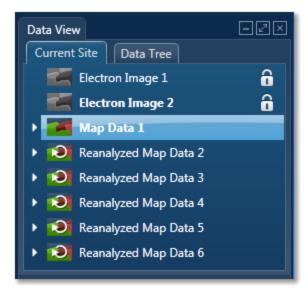




# **Reanalyze Data**



If you have acquired an EBSD Map with stored EBSPs it is also possible to reanalyze a map region with new settings such as new solver settings or even solving by including different phases. Re analyzed map data is stored in the data tree as shown in the screen shot below:



#### See Also:

Current Site on page 29

Data Tree Menus on page 38

Moving data to another PC on page 59

# Mini View

The Mini View is an area of the Support Panel dedicated to the display of a number of different views which you can select depending on what data you wish to view. Views containing the current Electron Image, Spectrum Monitor or EDS Ratemeter are examples of such views.

### **Electron Image**

The full field of view of the currently selected electron image is displayed here. It is often useful to refer to this image in steps where your application area is dedicated to displaying spectra or maps. For example you can view the electron image in the Mini View if you wish to view full size spectrum in the Acquire Spectra step.

The features of the electron image in the Mini View are:

The default state is full image with Scale Bar (micron marker). You can remove the Scale Bar from the display by de-selecting it from the image context menu.

The Context menu items are:

Show Acquisition Areas	Show All
	Show Selected
	Show None
Show Scale Bar	

Features such as Pan, Zoom and User Annotations are not available in the Mini View.

### **Spectrum Monitor**

It provide a means for the user to see what X-rays are being detected at any given moment. It is useful for a quick survey of the specimen to find an area of interest for analysis. Spectrum Monitor uses the current spectrum acquisition settings with the additional setting of the refresh rate for monitoring the spectrum. This refresh time is referred to as the Buffer Size. The default is 20 but can be changed under the Settings for Spectrum Monitor in the Miniview. Increasing the Buffer Size corresponds to a longer refresh rate.

The settings in the Spectrum Monitor are:

Buffer Size: The default value is 20.

Number of Channels: 1024, 2048 or 4096

Energy Range (keV): 0-10, 0-20 or 0-40

The settings can be selected from the Acquire Spectrum step or Mini View. If you make a change in the setting in one place it is automatically updated in the other.

### Ratemeter

It is very useful for setting up the microscope beam current while viewing the X-ray acquisition parameters:

Input Count Rate (cps)

Output Count Rate (cps)

Dead Time (%)

Ratemeter also displays the current Process Time and the Recommended WD (mm).

# **Step Notes**

Step Notes provide the first time user of a navigator with simple instructions on how to complete a typical work flow. It also provides a site administrator or user with the ability to write an SOP (Standard operating procedure).



A default editable set of notes are provided for each navigator step. The user can then overwrite these or add notes as required. A reset to default settings is available.

The notes are saved with the current user profile.

# See Also:

Step Notes Editor on page 43

# **Acquire Spectra**

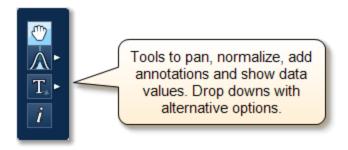


Analyzer is microscope centric application. You can acquire a spectrum from a region on the specimen scanned by the electron beam.

There is an acquisition toolbar near the top of the workspace. It has controls for starting and stopping the spectrum acquisition.

There is a Settings cog for selecting the acquisition parameters. For details see Acquire Spectra - Settings on page 313.

The toolbar located on the left side of the workspace has various tools for spectrum manipulation and annotation as shown in the screen shot below:



For description of each tool, see Acquire Spectra - Toolbar on page 316

The Compare Spectra & MiniQuant Results on page 195 option is available in the top right corner of the Spectrum viewer. You can compare the current spectrum with any other spectrum from an opened Project on the Data Tree. Instant MiniQuant results can be viewed in a table or a bar chart.

A number of useful shortcut menus are available as right mouse click in the spectrum viewer. For details see Context Menus - Spectrum Viewer on page 321.

# **Acquire Spectra - Settings**

The settings are described in detail below:

# **Energy Range (keV)**

Select a spectrum energy range from the available options of Auto, 0-10, 0-20 or 0-40 keV from the Energy Range drop down list.



An appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited.

In the Auto mode, the system checks for the accelerating voltage set on the microscope and selects a suitable energy range in the software.

# **Number of Channels**

Select number of channels from the drop down list of Auto, 1024, 2048 or 4096 (4K) with which you wish to display the spectrum. The number of eV/channel will depend on both the energy range and the number of channels you select:

Energy Range (keV)	Number of Channels	eV/channel
0-10	4096	2.5
0-10	2048	5
0-10	1024	10
0-20	4096	5
0-20	2048	10
0-20	1024	20
0-40	4096	10
0-40	2048	20
0-40	1024	40

In the Auto mode, the system checks for the energy range selected and sets the appropriate number of channels.

# Note

The Energy Calibration routine is performed for all process times and for all available energy ranges and number of channels. It means if you change any of these settings soon after you had performed the Energy Calibration you do not need to re-optimize the system.

### **Process Time**

Select the Process Time from the drop-down list of Process Times, Default and 1 to 6. The Process time is the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process time, the lower the noise. If noise is

minimized, the resolution of the peak displayed in the spectrum is improved, in other words, the peak is narrower and it becomes easier to separate or resolve, from another peak that may be close by in energy.

If Default is selected, the Process Time is automatically set to a suitable value.

There is a trade off between the Process time that is used, and the speed at which data can be acquired into the X-ray spectrum. Process time 1 is the shortest, and as such, gives the highest X-ray acquisition rates, but at some cost to resolution. Process time 6 is the longest, and gives the highest resolution, but at some cost to maximum acquisition rate. The longer the Process time, the slower data can be acquired, i.e. the higher the system Deadtime will be for a given input count rate. (The input rate is not affected by the pulse processor).

# Which Process Time should I use?

When you start your application first time, the Process Time is set to Default. This is a suitable choice for many routine applications where you are looking for good resolution of peaks and fast acquisition.

For the first look at a specimen you should use a long process time (5 or 6) to start with in order that you do not miss any detail in your spectrum. For example, when identifying peaks particularly those closely spaced and overlapping, it is important to get good peak separation. Good resolution is also important for looking at a series of lines that are very closely spaced, like an L series and process times 4 to 6 should be chosen. Common overlaps include the Mo L and the S K lines.

If there are no closely spaced peaks then you can afford to use a shorter Process Time such as 1-3 which will enable you to increase the acquisition rate by increasing the beam current. A compromise between acquisition speed and resolution should be found if there are peak overlaps.

When acquiring SmartMap data you should choose your Process Time carefully.

- You may have been working on a Specimen in either Analyzer or Point & ID where
  you have setup your acquisition parameters to optimize your quantitative analysis.
  If you now wish to acquire SmartMap data and you think you may wish to reconstruct spectra from your SmartMap data and then quantify these spectra, you
  should maintain these acquisition parameters. This means that you may have to
  acquire data with a long Process Time to maximize resolution but limit the maximum
  acquisition rate.
- 2. You may have been working in either Analyzer or Point & ID and you want to view the distribution of elements whose main peaks do not overlap as a map or a linescan. In this case you should use a shorter Process Time which will mean that you can work with higher acquisition rates and shorter acquisition times. The choice of Process Time will very much depend on your sample and what you wish to do with your SmartMap data once it has been acquired.
- 3. If you have started your Project in Map and you are analyzing an unknown sample, we recommend that you use a long Process Time in order that you do not miss any detail in your spectrum. However if you only wish to map certain elements whose



main lines do not overlap, you can afford to shorten the Process Time and increase the acquisition rate by increasing the beam current.

# **Acquisition Mode**

There are three options to terminate the acquisition, Auto, Live Time and Counts.

If Auto mode is selected, acquisition continues until enough counts are collected in the spectrum for quantification.

You can choose to terminate acquisition at the end of a preset Live Time. Enter the required time in seconds into the text box. This is the time for which the system is processing counts into the spectrum. The live time clock runs slower than the real time clock so that the acquisition for '100' live seconds takes longer than 100 real seconds. This time is extended to compensate for the output rate being less than the input rate by the degree of Deadtime.

You can choose to terminate acquisition at the end of a preset number of counts. Enter the value in the Count Limit text box. The default value is 500.000.

# **Pulse Pile Up Correction**

Check Pulse Pileup Correction check box if you wish to automatically correct the spectrum for pulse pileup peaks. Uncheck the box if you wish to disable this correction.

Pileup peaks can occur when a second pulse arrives and triggers the measuring system during the time required to process a previous pulse. When this happens, neither pulse will appear in its correct position. The result being a peak at a higher energy equivalent to the sum of the energy of the two photons.

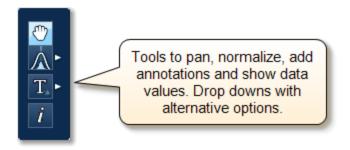
The largest pileup peaks will be seen at twice the energy of the main peaks - e.g. Fe Ka pile up peaks will be seen around 12.8 keV.

#### Notes

The pileup correction algorithm assumes that the count rate at every energy is constant throughout the analysis period. Therefore, the correction works best when analysis is performed on single pixels, points or areas of same composition. Bad results may be obtained if the beam is rastered over an area where composition is changing or if a spectrum is reconstructed from a SmartMap over a region where the composition is changing.

# **Acquire Spectra - Toolbar**

The Acquire Spectra screen has a toolbar on the left side of the workspace shown in the screen shot below:



#### Pan

The Pan tool allows to expand the spectrum along the vertical axis and move the spectrum along the horizontal axis. To expand the spectrum along the horizontal axis with Pan tool selected, hold down the Ctrl key while dragging the spectrum with the left mouse.

### **Normalize Spectra**

You can normalize two spectra over a selected point or a region.

# **Normalize Spectra (Point)**

- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Point) option from the toolbar. The cursor turns into an up down arrow ( ).
- Double-click in the spectrum to set a normalization point along the X-axis. A window is drawn on either side of this point. Both the spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

# **Normalize Spectra (Region)**

- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Region) option from the toolbar. The cursor turns into a crosshair (+).
- Click in the spectrum viewer to select the start point of the energy window. A default window is displayed about this point. Drag the mouse to define your window and then release it. A window will be drawn between the first point and the end point where you release the mouse. Both spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

#### **Annotations**

Five tools available to add annotations on the current spectrum and the image are Caliper, Angle, Text, Rectangle and Ellipse. Select the tool by clicking on it and then click on the spectrum/image to add annotation. For example to add text select the Text annotation tool, click on the spectrum where you wish to enter the text and then start typing the text. To delete annotation double click on it to select it and then press the Delete key on the keyboard.



#### **Show Data Values**

With this tool you can view the Energy (keV) and counts in any channel of the spectrum. Simply select the Show Data Values tool from the toolbar and then hover on spectrum. The values will be displayed as you move from channel to channel.

# **Acquisition and Settings Toolbar**

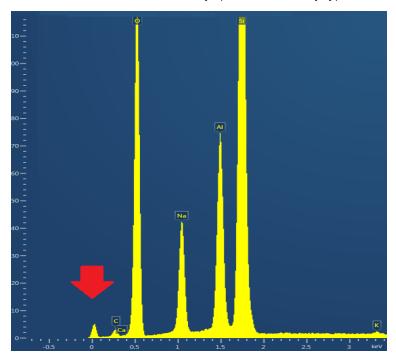
Near the top of the Acquire Spectra window, there are buttons for starting and stopping acquisition as shown below:



Press the Start button to acquire a spectrum. There is a Settings cog in the toolbar. For details of settings see Acquire Spectra - Settings on page 313.

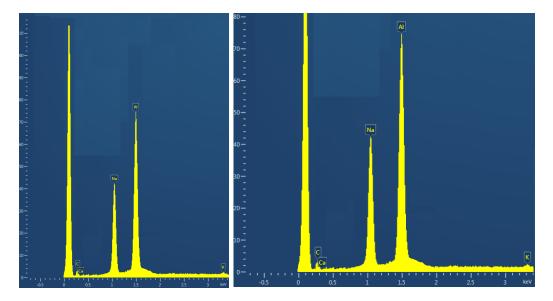
# About the noise peak

In an X-ray spectrum, a noise peak sometimes appears near the zero voltage level. This small amount of electrical noise is always present from any type of detector.



Peaks at low energies below 1 keV might indicate various elements (such as the  $K\alpha$  line for carbon,  $L\alpha$  line for calcium, or  $M\alpha$  line for lanthanum). A noise peak can be confusing because it appears to represent an element but has no label. You can choose to hide this peak by specifying a cut-off voltage below 1 keV, which removes lower energy values from the spectrum.

If the noise peak is very high, it determines the maximum value for the counts when you reset the spectrum's vertical scale. This is shown in the left picture. If you exclude this effect, you can compare the proportions of elements more easily. This is shown in the right picture.



### See Also:

Excluding the noise peak when resetting scales below

Setting the cut-off voltage for the noise peak below

# **Excluding the noise peak when resetting scales**

Note that if the value for the cut-off voltage is near 1 keV, the low-energy peaks for some elements might be excluded from scaling.

To exclude the noise peak when resetting the scales in any spectrum:

- 1. On the Tools menu, select Preferences.
- 2. On the Preferences dialog, select the EDS Spectrum Viewer tab.
- 3. On the tab, under Noise Peak, select Exclude from Scaling.

To include the noise peak, follow the same steps, and instead select Include in Scaling. This automatic scaling occurs whenever a new spectrum is acquired, unless the vertical scale is locked.

To exclude the noise peak when resetting the scales in the current spectrum:

- 1. Right click in the Spectrum Viewer to open the context menu.
- 2. Select Noise Peak, then Exclude From Scaling.
- 3. Right click in the Spectrum Viewer to open the context menu. Select Reset Scales.

# Setting the cut-off voltage for the noise peak

To set the cut-off voltage for the noise peak in any spectrum:

- 1. On the Tools menu, select Preferences.
- 2. On the Preferences dialog, select the EDS Spectrum Viewer tab.
- 3. On the tab, under Noise Peak, select Hide or Exclude from Scaling.



4. In the Cutoff Energy (keV) box, type a value between 0 and 1. The recommended value is 0.12 keV.

If the value for cut-off is near 1 keV, the low-energy peaks for some elements might not be visible or they might be excluded from scaling.

# **Context Menus - Spectrum Viewer**

A number of useful shortcut menus available as right mouse click in the spectrum viewer are shown in the table below:

Context Menu Item		
Reset Scales		
Export	Save As	
	EMSA	
	Сору	
	Print	
	Email	
	Settings	
Peak Labels	Show	
	Reset Posi- tions	
Annotations	Show	
	Select All	
	Style	
	Delete	
X Axis	Show	

Context Menu Item		
THE TECH		
	Locked This	
	is a useful	
	option if	
	you are	
	looking for	
	a particular	
	energy	
	range. You	
	do not want	
	the energy	
	scale to	
	change	
	when you	
	load a new	
	spectrum in	
	the viewer	
	for exam-	
	ination or	
	for report-	
	ing. Locking	
	the X Axis	
	will main-	
	tain the hor-	
	izontal	
	energy	
	scale. If you	
	do not lock	
	it, it will	
	change to	
	the full	
	scale when	
	you load a	
	different	
	spectrum.	
	The default	
	energy	
	range is full scale.	
	Adjust	



Context Menu Item		
Y Axis	Show	
	Locked	

Ctt		
Context Menu Item		
Menu Item	Units You have the choice between cps/eV(per channel) and Counts for the units along the Y-Axis of the spectrum. For easy comparison of spectra, cps/eV is an ideal choice because there is very small variation in the range. You do not need to normalize prior to comparison of spectra using different energy ranges, number of channels and live times. This is vital when comparing a stored spectrum with one that is still	cps/eV



Context Menu Item		
	acquiring.	
		Counts
	Scale	Linear
		Logarithmic

Context Menu Item	
Normalize	
Normalize is	
a useful func-	
tion for com-	
paring two	
spectra	
acquired	
using dif-	
ferent input	
X-ray count	
rates for	
example	
spectra	
acquired	
with two dif-	
ferent beam	
currents. Nor-	
malize is	
available	
from the	
toolbar on	
the left of	
the screen in	
Acquire	
Spectra, Con-	
firm Ele-	
ments,	
Acquire &	
Confirm and	
Compare	
Spectra	
steps of	
Point & ID	
and	
Analyzer. For	
details of	
how to nor-	
malize using	
a point or a	

Context	
Menu Item	
region on a	
spectrum	
see the Com-	
pare Spectra	
help topic.	
Note that	
Normalize	
can be	
switched on	
and off from	
the shortcut	
menu avail-	
able as right	
mouse click	
in the spec-	
trum viewer.	
If a point or	
region has	
already been	
defined on	
the spec-	
trum, switch-	
ing	
Normalize	
on or off will	
maintain the	
point or	
region. If a	
point or	
region has	
not been	
defined	
already,	
switching it	
on will nor-	
malize the	
spectra to	
the peak at	
zero.	

Context Menu Item	
Smooth This	
is useful	
when com-	
paring spec-	
tra where	
small dif-	
ferences	
may be	
obscured by	
statistical	
scatter. The	
smooth func-	
tion applies	
an energy-	
dependent	
filter to the	
spectrum.	
This has the	
effect of	
slightly	
broadening	
the peaks	
and also fil-	
tering out	
the rapid	
fluctuations	
due to sta-	
tistics. Sta-	
tistical	
fluctuations	
can some-	
times appear	
like a real	
peak. When	
it is difficult	
to decide	
whether a	
peak is	
present or	
not, the	



Context Menu Item		
smooth function substantially reduces the statistical fluctuation so that any real peak becomes more visible.		
Noise Peak	Include in Scaling	Shows the noise peak, and includes its value when the context menu option "Reset Scales" is used.
	Exclude from Scal- ing	Shows the noise peak, but excludes its value when the context menu option "Reset Scales" is used.
	Hide	Hides the noise peak.
Details		

# **Compare Spectra & MiniQuant Results**

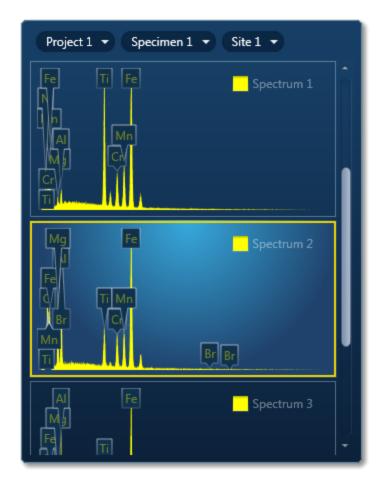
Real time Compare and instant MiniQuant options are available in the Acquire Spectra, Confirm Elements and Calculate Composition (Comparison of Results - Two Spectra template) steps. User can see results without having to move away from the acquisition mode. Using these options you can:

- See your results during analysis.
- Compare your current spectrum to a control spectrum during acquisition.
- View MiniQuant results in a table or a bar chart.

Click in the top right corner of the Spectrum Viewer in Acquire Spectra, Confirm Elements or the Calculate Composition window to access the Compare & MiniQuant options:



In the above example, Spectrum 1 is the current spectrum and Spectrum 2 is the comparison spectrum. You can select the comparison spectrum from a Project currently available in the Data Tree. It can be from any Project, any Specimen and any Site of Interest currently available in the Data tree. To choose the comparison spectrum click on the down arrow (Spectrum 2 in the above example). Spectra available in the current Project, Specimen and Site of Interest are displayed as below:





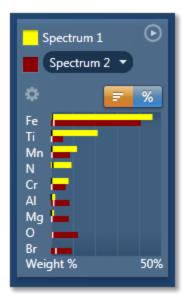
Click on the spectrum in the display to select it for comparison. The selected spectrum will be overlaid as a line spectrum over the current spectrum. The MiniQuant results are displayed in a table as shown in the example below:



The results are displayed as wt% (weight%).

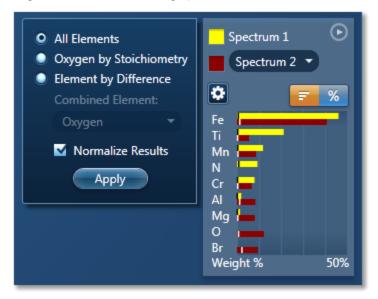
The statistical error is displayed as  $\sigma$  (weight% sigma) for the calculated wt%. It is the overall confidence figure for the analysis. You can use sigma to assess the results especially when an element is present at low concentration. For example, if an element concentration is 0.2 wt% and the  $\sigma$  is 0.12 wt%, the element might be detected at a statistically significant level if the acquisition time for the spectrum is extended. If the  $\sigma$  is 0.4 wt%, it is pointless to extend the acquisition time and it is safe to assume that the element if present, is at a level above the limit of detection for this technique.

Press to display the results in a chart:



The sigma values are displayed as black or white vertical bands across the bars in the chart results as shown in the example above. In this case the full scale of the bar chart is 50%.

If you wish to change the MiniQuant Settings press



Make your selection by clicking on the radio button and then press the Apply button. The results will be updated immediately .

### Note

The Quant Settings in the MiniQuant and Calculate Composition are the same. Updating one updates the other and vice versa.



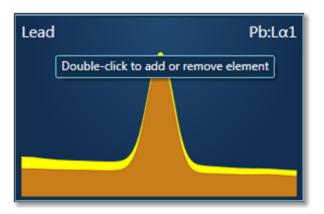
# **Confirm Elements**



This step has been designed to help you confirm the elements that have been identified by AutoID in your spectrum. These elements are then used to create a confirmed elements list for qualitative and quantitative analyses. Extensive tools including Element Series Markers, Overlays, Element Profiles and Show Candidate Elements are available here to assist you in confirming elements manually.

### How to confirm elements:

Start with the largest peaks. Press the question mark icon to select the Show Candidate Elements tool from the tool bar on the left hand side of the interface, then double click on a peak in the spectrum viewer. The candidate elements are displayed in a stacked spectra view on the right hand side of the window (you can double click on any of these elements to add or remove it from the confirm elements list).



You can control what overlays you see in the Spectrum viewer via the 'Confirm Elements Settings'. These overlays can be very useful in helping you to interrogate complex spectra.



 Press Include/Exclude once you are satisfied with the identification of each element to build your list of the confirmed elements.

# See Also:

Confirm Elements - Settings on page 171

Confirm Elements - Tools on page 174

Element Lists on page 191

Peak Labels on page 158

Compare Spectra & MiniQuant Results on page 195

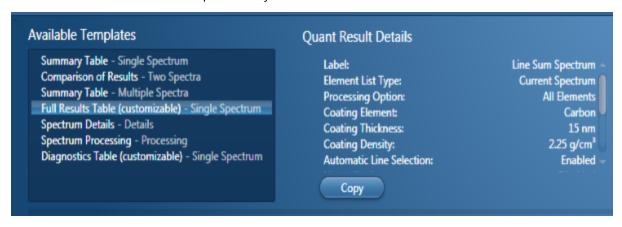


# **Calculate Composition**

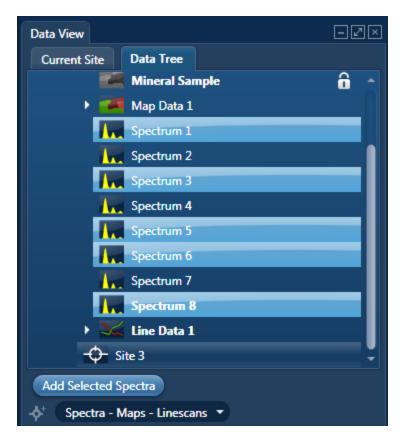


In this step you can view quant results in more detail using any of the 'Available Templates'.

To view result select the template that you wish to use:



- If you want to see a comprehensive set of results from a single spectrum, then choose the 'Full Results Table (customizable) - Single Spectrum' template and whichever spectrum is highlighted in the Data Tree will have its results shown in this template.
- To populate a multiple spectra template, hold the Ctrl key down while choosing spectra on the Data Tree and then press the 'Add Selected Spectra' button at the bottom of the Data Tree window.



- To compare quant results from two spectra, select 'Comparison of Results Two Spectra' template. Then select the comparison spectrum from the Compare option in the 'Mini Quant and Compare' option. The compare spectrum will be overlaid on the current spectrum in the Spectrum Viewer. The quant results will be displayed in the table below.
- If you wish to change the Quant Settings press the Settings button to display the Quant Settings dialog. Apply the changes and close the dialog.
- Press the Requantify button to display the recalculated results.

# **Quant Results Details**

You can see the settings used for calculating the composition in the Quant Results Details list box:

Parameter	Description
Label (Spectrum Label)	E.g., Spectrum 1
Element List Type	Current Spectrum, Fixed List or Combined List
Processing Options	All Elements, Element by Difference, Combined Element or Oxygen by Stoichiometry
Apply Coating Correction	Enabled or Disabled



Parameter	Description
Coating Element	E.g., Carbon
Coating Thickness	E.g., 15 nm
Coating Density	E.g., 2.25 g/cm3
Automatic Line Selection	Enabled or Disabled
Normalization	Enabled or Disabled
Thresholding	Enabled or Disabled
Deconvolution Elements	None/Selected
Factory Standards	Standardizations file supplied with the system
User Standards	Standardizations file defined by the user
Pulse Pile Up Correction	Enabled/Disabled
Detector File	Indicates file that has been used to characterize detector
Efficiency	Calculated/File based

# **Quant Results View**

The information displayed in the Quant Results View depends on which template has been selected. You can view Spectrum Details, Spectrum Processing and Diagnostics table in addition to quant results.

# See Also:

Quant Settings on page 187

Element Lists on page 191

Compare Spectra & MiniQuant Results on page 195

# **Compare Spectra**



This step in both the Point & ID and Analyzer Navigators allows you to compare spectra acquired from different sites of interest and specimens from the currently opened projects.

You can compare spectra acquired using different settings for example energy ranges (0-10, 0-20 or 0-40 keV) and number of channels (1024, 2048 or 4096).

Spectra associated with the current Site can be added to the Compare table by holding down the control key and pressing Add Selected Spectra. Note that if you have acquired the spectra in Point & ID, the positions of all the spectra associated with the current Site of Interest are displayed on the image. You can add spectra from any Project, Specimen and Site of Interest from the Data tree into this table.

Select which spectra you wish to compare by selecting them individually from the Data Tree, Press 'Add Selected Spectra'. This will add all the spectra you wish to compare into the 'Compare' table:



If you want to remove any spectra from the table, highlight the spectrum and press

You can choose which of the available spectra you wish all the others to be compared to by selecting your reference spectrum from the compare table and pressing Select Reference Spectrum.

### Settings

You can change the color of individual spectra by selecting the color from the drop down list in the compare table.

To change the line thickness of individual spectra, select it from the Line Thickness drop down list in the Compare table.

To apply the chosen line thickness globally, select it from the Compare Spectra Settings from near the top of the Compare viewer.

### Normalize



Normalize is a useful function for comparing spectra acquired using different input X-ray count rates such as spectra acquired with two different beam currents. Note that you can normalize spectra using a point or a region.

#### **Normalize Spectra (Point)**

- Select this option from the toolbar near the top left of the Compare Spectra screen.
- Double-click in the spectrum to set a normalization point along the X-axis.
- A window is drawn on either side of this point. The spectra in the Compare viewer are scaled to the average value of cps/eV (Y-axis) in the window.

#### **Normalize Spectra (Region)**

- Select this option from the toolbar near the top left of the Compare Spectra screen.
- Click and drag to set a normalization region along the X-axis.
- The spectra in the Compare viewer are scaled to the average value of cps/eV (Y-axis) in the normalization region selected in the previous step.

#### **Smooth**

The Smooth function is available from the context menus of the spectrum viewer. This is useful when comparing spectra where small differences may be obscured by statistical scatter.

The smooth function applies an energy-dependent filter to the spectrum. This has the effect of slightly broadening the peaks and also filtering out the rapid fluctuations due to statistics. Statistical fluctuations can sometimes appear like a real peak. When it is difficult to decide whether a peak is present or not, the smooth function substantially reduces the statistical fluctuation so that any real peak becomes more visible.

Analyzer -	Custom
------------	--------

Analyzer is microscope centric application. X-ray spectra are acquired from the regions on the specimen scanned by the microscope beam. There are two modes of operation in the Analyzer application, Guided and Custom.

In the Custom mode, the Analyzer navigator has three steps.

The Describe Specimen and Compare Spectra steps are explained in the previous section. The new step is described below:

Acquire and Confirm. 123



# **Acquire and Confirm**



Acquire and Confirm is the main step of the Analyzer Navigator in the Custom mode. Three components are available in the single workspace. The Acquire Spectra component is located in the top half of the workspace, Quant Results in the bottom left and Confirm Elements in the bottom right.

The three components are combined in the Custom mode to give you a single workspace called Acquire and Confirm. It offers the convenience of working in one window without having to move away from it for acquiring a spectrum and obtaining the quant results.

You can choose which component you wish to display in the workspace by pressing the rel-

evant button in the toolbar, from the view.

Press to un-dock a component as a free floating window located in the top right corner of the view. Press to switch it into a full screen view.

To re-dock the free floating window back into the main workspace press .

Each component has identical functionality as its counterpart in the Guided Navigator. To get help on each application follow the links below:

Acquire Spectra on page 96

Confirm Elements on page 353

Calculate Composition on page 185

# Point & ID - Guided

Point & ID is an image centric application that requires the acquisition of an electron image prior to X-ray spectra acquisition. There are two modes of operation, Guided and Custom.

In the Guided mode, the Point & ID navigator has six steps.

Describe Specimen and Compare Spectra are explained in the earlier section. The steps which are unique to Point & ID - Guided are explained here.

Scan Image	125
Acquire Spectra	146
Confirm Elements.	170
Calculate Composition.	185



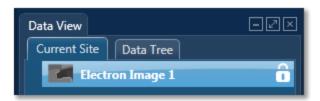
# **Scan Image**



In the Scan Image step, you can acquire an electron image into a 'Site'. A 'Site' is like a folder, which contains images and analyses for a particular area on a specimen.

For EDS, if you do not want to collect an image and just want to acquire spectra, you can skip this step and go straight to the Acquire Spectra step.

You can have any number of images in a site. Just ensure that the images you want to keep are padlocked in the data tree to stop them being overwritten, as shown in the screen shot below:



You can toggle between saving or replacing the current image with successive image acquisition.

If your specimen is drifting, click the Settings cog and activate AutoLock.

The Scan Image step has several tools for manipulating and enhancing electron images:

- The acquisition toolbar above the electron image and other nearby controls.
- Scan Image toolbar (a vertical toolbar on the left) for manipulating and annotating the image.
- If a forward-scatter electron detector (FSD) is fitted, extra controls are available for combining the signals from each diode into a mixed image.

#### Acquisition toolbar and other nearby controls

The acquisition toolbar, above the electron image and below the Navigator, has buttons for starting and stopping the image acquisition, the Settings cog for selecting the image acquisition parameters and a button to link/unlink images for manipulation.



Control	Description
(FSD only)	Displays up to three combinations of mixed image and individual images. Your selections are retained in your user profile.
► START	Click to start the image acquisition according to the current acquisition parameters.
■ STOP	Click to stop image acquisition. Acquisition stops at the end of the current frame. Click again to stop immediately. If you navigate away from the step, acquisition stops at the end of the current frame.
Settings	To change the acquisition parameters, click the Settings cog on the Acquisition Toolbar to display a dialog.
	You can select Image Scan Size, Dwell Time (µs), Input Signal the labels here reflect whatever was set during the installation for example SE, BSE or FSD), either Continuous Scan or Number of Frames and Frame Time (secs).
	If your specimen is drifting, you can activate AutoLock to ensure that any analysis corresponds to the true location on your image.
Mixing Mode (drop-down list)	Combines signals from the diodes to form a mixed image. Some options are available only if the required diodes are installed and configured.
(FSD only)	<ul> <li>FSD Z Contrast uses upper and side FSD detector channel images. Select this mode if you are inter- ested in seeing density/atomic Z contrast signal.</li> </ul>
	<ul> <li>FSD Topo/Orientation uses lower FSD detector chan- nel images. Select this mode if you are interested in seeing orientation contrast signal.</li> </ul>
	<ul> <li>Custom - include and exclude FSD detector channel images of your choice.</li> </ul>
	When you select either of the first two modes, the software automatically uses the FSD diode channels associated with that mode. For example, FSD Z mixing mode uses the upper and side FSD diode channels. The FSD Topo/Orientation mixing mode always uses the lower FSD diode channels. Custom mode allows you to mix any FSD diode channels.



Control	Description
Select Second Image	Selects further images to compare with the electron images, for example, a forward-scattered electron image. This control is available only when the map display is for an image only:
(FSD only)	Sets the number of images per row in the Standard and Interactive displays.
Standard ▼	Offers a choice of image display :
(FSD only)	<ul> <li>Standard - you can add individual images or remove them from the mixed image.</li> </ul>
	<ul> <li>Interactive - similar to Standard. You can also change the weighting and color contributed by each image.</li> </ul>
	<ul> <li>Summary - similar to Interactive and in a more compact display.</li> </ul>
	Links images. You can simultaneously manipulate all the layers using the pan or zoom controls.
	Unlinks images. You can manipulate individual layers using pan or zoom controls.
	Use the mouse wheel to zoom in and out of the image.
	Use these tools (near the bottom right of the screen) to adjust manual and automatic brightness, contrast and color.
Context Menus	Right-click the electron image to display context menus for copying, exporting and printing images.

#### See Also:

Scan Image Toolbar on page 424

Scan Image - Settings on page 421

FSD diode controls on page 432

Context Menus - Image Viewer on page 157

## Scan Image - Settings

The selectable parameters that control image acquisition (Image Scan Size, Dwell Time and Number of Frames) should be chosen according to your specific requirements. Both the time taken and the data storage size of the image are dependent on these parameters.

For a quick look at the specimen select the lowest image scan size and the fastest speed. This will enable you to decide whether you require either a higher pixel density, in order to observe finer detail such as small features, or a longer dwell time in order to improve the image quality by reducing the noise.

The available acquisition parameters are:

- Image Scan Size
- Dwell Time (µs)
- Mains Synchronize
- Input Signal
- Software Tilt Correction
- Continuous Scan
- Number of Frames
- Frame Time (secs)
- FSD Control
- AutoLock

## **Image Scan Size**

In general, the resolution of an image or Image Scan Size is defined as the number of picture points or pixels along the x and y axes e.g., 256 x 256, 512 x 512 or 1024 X 1024. The quality of the image improves as the resolution at which an image is acquired is increased. However, a microscope monitor/CRT is usually a rectangular display (rather than square), so the resolution is displayed as a rectangle i.e., 256 x 200 in order to take into account the aspect ratio. The y dimension is set at installation, when imaging is calibrated. It will vary with each system.

Select the Image Scan Size for image acquisition from the following drop down options available: 64, 128, 256, 512, 1024, 2048, 4096, 8192

## Dwell Time (µs)

Images can be acquired using different speeds. The beam dwells on each pixel for a specified length of time while the signal is collected and then it moves to the next pixel. So the speed at which an image is acquired depends on the dwell time.



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Speed	Dwell time
Fastest	1 μs
Fast	5 µs
Normal	10 μs
Medium	35 µs
Slow	65 µs
Slowest	400 μs

## **Mains Synchronize**

Selecting Mains Synchronize on the Image Setup window, synchronizes the start of each scanned line to the mains supply. This will help to reduce mains borne interference in the image. Note that the acquisition time will be marginally longer than when mains synchronize is not selected.

Note that Mains Synchronize will only be visible if the appropriate hardware is installed.

## **Input Signal**

Select the signals from the detectors on the microscope.

For EDS, secondary electron imaging is generally most appropriate if you are imaging a sample which has topography whereas backscattered imaging is a very useful means of identifying areas of different composition on flat samples. Secondary electron imaging is the most common form of imaging and for a first look at your sample, choose this mode.

If you are analyzing a flat, polished sample and you can see weak contrast, switch to back-scattered imaging which will tend to enhance this contrast by showing up areas of different phases.

For EBSD, Forward Scattered Imaging is often used. Because of the high angle of tilt dictated by the collection geometry required for EBSD, many electrons are scattered forward and down towards the bottom of the phosphor screen. Using Forward Scattered Electron (FSE) imaging diffraction contrast is enhanced and the resultant signal makes the presence of individual grains easy to identify. The forward scattered electron signal produced is therefore ideal for EBSD investigations. However, the user may use any electron signal as required for the reference image.

If you select the Auto checkbox before you start a new FSE acquisition, the software automatically adjusts the signal from each diode for optimal brightness and contrast. The optimized electron image then appears after a delay of a few seconds. When you first start the software, the Auto check box is already selected for you.

#### **Software Tilt Correction**

Enables the use of imaging tilt correction. This is an important function when working with tilted samples. If no tilt correction is done, images and areas will be distorted. If the software tilt correction is enabled, it will be possible to correct scanned images and areas based on information about the tilt angle and the scanning tilt axis.

#### **Continuous Scan**

If the Continuous Scan option is checked, you will see the image start to scan down the window and it will continue to refresh after each frame. If there are any instabilities in your specimen (e.g., charging or drifting problems) then these will be apparent as the image may shift slightly after each scan.

In order to stop the continuous scan, press the Stop button.

- Click Stop once and the scan will stop when the current frame is complete.
- Click Stop twice and it will stop immediately.

If you navigate to a different step, the scan will stop at the end of a frame.

#### **Number of Frames**

Enter the number of times you wish the beam to scan the site of interest for image acquisition.

### Frame Time (secs)

The frame time is displayed in seconds. The value of frame time depends on the resolution, speed and mains synchronize if available.

#### **FSD Control**

This section appears only if a forward-scatter detector is correctly configured. To control settings for the signals from the forward-scatter detection diodes, click the Settings button to open another dialog.

If you select the Auto checkbox before you start a new FSD acquisition, the software automatically adjusts the signal from each diode for optimal brightness and contrast. The optimized electron image then appears after a delay of a few seconds. When you first start the software, the Auto check box is already selected for you.

#### See Also:

AutoLock on page 133

Scan Image on page 418

## Scan Image Toolbar

The Toolbar is located near the top left side of the Scan Image window. Tools are provided to manipulate and annotate the image:





#### **Pan and Zoom**

You can move the image using the Pan tool. Use the wheel mouse to zoom in and out.



#### **Annotation**

There are five different tools to add annotations on the current image as shown in the screen shot below:



To edit an annotation double-click on it to select it, the editing handles will be displayed. Use the handles to edit the annotation.

To delete an annotation, select it by double-clicking on it and then press the Delete key on the keyboard.

To delete all annotations on an image, choose Select All from the Annotations context menu on the image viewer and then press the Delete key on the keyboard.

#### Information

Select the Show Data Values tool from the toolbar and click anywhere on the image to display the Intensity value at that point.



#### **FSE** area optimization

If an FSD system is currently fitted, this tool is available. It helps you to improve the brightness and contrast of any area of the FSD mixed image.



Normally, the software calculates optimized brightness and contrast values for the mixed image over the *whole* area. Some areas of the image might be very dark or very light, so that features within an area of interest might not be well defined.

- 1. Select the rectangle tool from the toolbar.
- 2. On the mixed image, find the area of interest.
- 3. Click and drag to form a rectangle around the area.

When you release the mouse button, the software calculates new values of brightness and contrast, then applies them to your area of interest. If necessary, you can repeat this step or reacquire the image and try again.

# **Export - Settings**

In this window, you can change the appearance of the image, and then print it or send it by email for example. This window appears when you right-click an image or spectra to open the context menu, and select Export, then Settings.

Control	Description
Preview Original	These two tabs show the image you are now creating and the original image (which you saw when you selected Export, then Settings).
Units	Offers a choice of units, such as inches or pixels, so that you can correctly size the image before you print it or add it to a web page.
Width, Height, Keep Aspect Ratio	Here you can type the actual size of your image. When Keep Aspect Ratio is selected, you need only change Width or Height to keep your image correctly proportioned.
Vertical Scale Type	This drop-down list is available for spectra only.  To display data that has a wide range, select Logarithmic.



Control	Description
(Check boxes)	Depending on the type of image, several check boxes might appear. Select the options to add extra information to your image.
Zoom	Changes the size of the image for easier viewing. This does not affect the size of the image when printed.
Сору	Copies the image. You can then, for example, paste the image into a Microsoft Word document.
Print Setup	Opens the standard Microsoft Windows printer dialog, where you can print the image immediately or apply the settings to use later.
Print	Immediately prints the image using existing settings for the printer.
Email	Opens your email service (if available), and attaches the image to a new message.
Save As	Saves the image as a bitmap file, which has a bmp file name extension.

# **AutoLock**

AutoLock is designed to increase the stability of data acquisition on SEMs and TEMs where the image may shift. This image shift can occur for a number of reasons, such as sample movement due to temperature changes at high magnifications or charging of the sample.

AutoLock works by acquiring an image, comparing it with the reference image acquired at that Site, determining the image shift and adjusting the scan position to compensate for the

shift. AutoLock will correct for an image shift at the interval specified while acquisition is in progress.

#### Note

AutoLock is accessed from the Settings in the Scan Image step in the Guided mode and Acquire and Confirm step in the Custom mode of Point and ID navigator. In Map and Linescan, AutoLock is accessed from the Settings in the Scan Image step in the Guided mode and Acquire and Construct step in the Custom mode.

To display the status of AutoLock in the Status Bar (On, Off, Due, Acquiring) check the AutoLock Status check box in the Status Bar tab in the Preferences dialog. You can access Preferences from the Tools menu.

#### **Setting up the AutoLock:**

1. Enable the AutoLock from the Settings in the Scan Image screen. Set it to Auto or Custom mode using the appropriate button.

There are three modes,

#### Off

Press the 'Off' button to disable AutoLock .

#### Auto

In the Auto mode, the system chooses a set of default settings. This would typically use a 50% reduced scan at low resolution, providing AutoLock for a distance of half the scan area. If you require extra correction, use the Custom setting. The tracking image settings are displayed but the Scan Settings and Predictive Settings are hidden in the Auto mode.

#### Custom

In the Custom mode, you can define all AutoLock settings including Tracking Image, Scan Settings and Predictive Correction as shown in the screen shot below:





2. Start image acquisition by pressing the Start button. It will initialize the AutoLock and then acquire an electron image. The AutoLock Settings can only be changed if no data has been captured for a given electron image; in this case, an existing electron image will be deleted and replaced unless you have locked the image in the Data Tree.

#### Note

If data has already been collected, changing the AutoLock Settings will require starting a new Site.

During data acquisition you can choose to display the status, history and diagnostic information of AutoLock in the Mini View:

#### **AutoLock Progression**

The drift profile is shown as snail trail (yellow). It represents the amount of drift that has occurred.

The full, dark area around the image represents the total imaging area, if a full 100% scan was captured. The sample image, as appears in the main user interface, is shown overlaid on this.

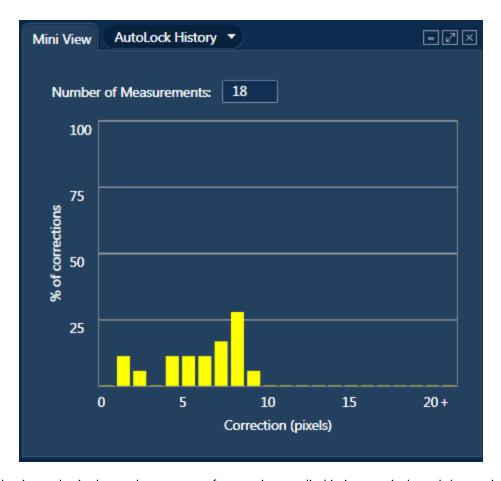
If the sample image reaches the edge of the dark surround, that indicates the limit of Auto-Lock has been reached. If drift continues in that manner, some parts of the sample will be outside the viewable area of the microscope. Note that the software will terminate the current experiment if the tracking area (or the scan region in In-field mode) reaches the edge. Any queued experiments will be immediately canceled if they are also outside the edge of the image.

The trail may extend outside the sample image but not outside the total imaging area. The trail will reset for each acquisition, i.e. it will only show the drift from the current acquisition.

#### **AutoLock History**

The AutoLock performance histogram, like the drift profile trail, also shows the amount of drift that has been applied during a single acquisition e.g., map acquisition. However, it provides a histogram view that allows rapid assessment of drift.

Consider the diagram below showing the histogram display:



The horizontal axis shows the amount of correction applied in image pixels and the vertical axis shows the percentage of the correction that had to be applied. For a stable sample, the amount of correction should be low – this will appear as very high values to the left of the histogram – maybe 100% across 1-2 pixels. For less stable samples, the histogram will spread to the right.



In the worst case, the histogram will show large peaks to the right hand side, indicating that large amounts of drift have been detected. In this case, you may want to stop the sample and take corrective action.

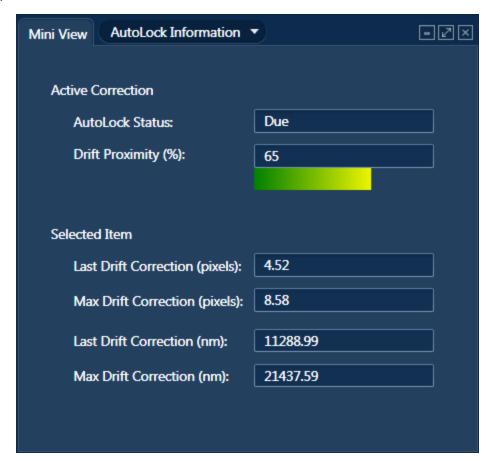
Very large drift values will be clamped to the right side of the histogram once they get beyond a certain value. The cut-off point will be based on the AutoLock Settings.

The histogram will reset for each acquisition, i.e., it will only show the drift from the current acquisition.

You can export the raw drift values (times and distances) as a CSV file, via the context menu on the Mini View.

#### **AutoLock Information**

The following diagnostic data is displayed if you select AutoLock Information in the Mini View:



#### **Active Correction**

AutoLock Status and Drift Proximity are displayed here:

#### **AutoLock Status**

The current status is displayed here.

#### **Drift Proximity (%)**

The remaining drift range (as a percentage of the field width) is displayed as Drift Proximity (%). The proximity display shows a colored bar that extends from green to yellow to red as the limits of AutoLock are reached. The bar will be full and in the red if AutoLock is exceeded (i.e., the scan region has drifted to the edge of the image).

#### **Selected Item**

Details of the drift correction for an item selected on the Data Tree in the current Project are displayed here.

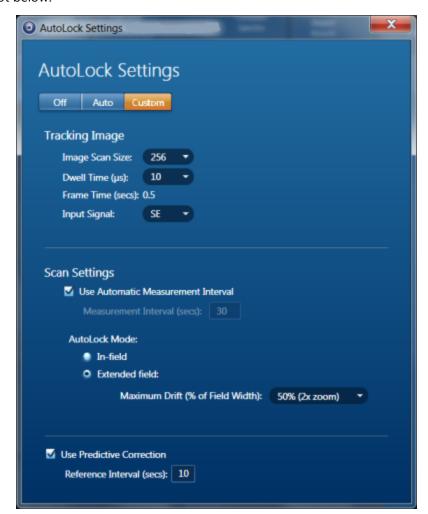
#### See Also:

AutoLock Settings below

FAQs about AutoLock on page 140

## **AutoLock Settings**

AutoLock is set to Off, Auto or Custom mode. In the Auto mode, the system chooses a set of default settings. In the Custom mode, you can specify the AutoLock settings shown in the screen shot below:



#### **Tracking Image**



You need to specify the tracking image settings to use, including the resolution (Image Scan Size), speed (Dwell time in  $\mu$ s) and Input Signal.

#### **Image Scan Size**

You can choose the size of the reference/tracking image from the Image Scan Size drop down list. The available options are 64 to 2048.

#### **Scan Settings**

You need to specify the drift measurement interval, the AutoLock mode and enable/disable 'Use Predictive Correction'.

#### **Measurement Interval**

Here you can specify the interval (in seconds) between tracking images. Drift correction is applied after each tracking image is collected. For slow drift, long intervals are acceptable. For fast drift or for drift that changes direction frequently, shorter intervals are more accurate (but will slow down acquisition). The default value is 30 seconds.

#### **Use Automatic Measurement Interval**

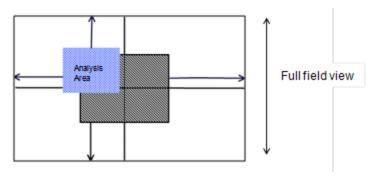
If you select this option, the program will determine the measurement interval for you. The interval is adaptive; as the speed of drift changes over time, the measurement interval will be increased or decreased accordingly. If Predictive Correction is also enabled, the automatic measurement interval will adjust to improve the quality of the predictive correction.

#### **AutoLock Mode**

There are two modes of operation, In-field and Extended field:

#### In-field

In this mode, the scanned area (field of view) is viewed in full all the time. It means that mapping or analysis is done on an area within this field. It also means that the drift correction is applied within this field. Digital zoom is not available in this mode. The In-field mode is illustrated in the figure below:



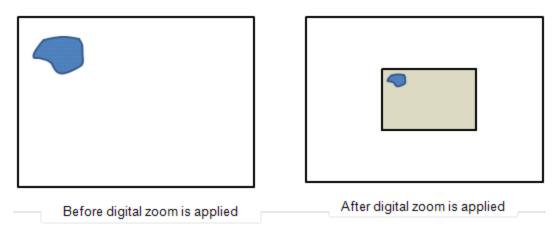
The amount of drift correction that can be applied is shown by the arrows from the analysis area/tracking area to the boundaries of the field of view.

#### **Extended field**

In this mode, the maximum drift available is 50% (2x zoom) and 150% (4x zoom). Setting the zoom level reduces the size of the captured area. If 2x zoom is selected only the middle 50% of the field of view is captured. The full field of view can be preserved by using the 'Maintain subject size' option described below.

#### Maintain subject size

You select an area you wish to map/analyze on the microscope, AutoLock preserves this area after the digital zoom is applied if you have checked the option, 'Maintain subject size' as illustrated in the screen shot below:



This is done automatically by setting a new magnification on the microscope before acquisition starts. This value is determined by dividing the current magnification by 2 (2x digital zoom) or by 4 (4x digital zoom) if 25% is selected.

The Maintain subject size' option is not available in the In-field mode.

#### Note

If the microscope does not have column control then 'Maintain subject size' will not be available.

#### **Use Predictive Correction**

If 'Use Predictive Correction' is selected, a correction is applied periodically at selected intervals between tracking images. It is a useful option if your sample has a fairly consistent amount of drift. At the start two reference images are acquired. You can define the interval between the reference images. The Predictive correction will be updated when a tracking image is acquired.

## **FAQs about AutoLock**

AutoLock is designed to aid in the stability of data acquisition on SEMs and TEMs where the image may shift. This image shift can occur for a number of reasons, such as sample movement due to temperature changes at high magnifications or charging of the sample.

AutoLock works by acquiring an image, comparing it with a reference image acquired from the same area and determining the image shift. The scan position is then adjusted to compensate for the shift.



#### What is the difference between Reactive and Predictive Drift Correction?

There are 2 modes of AutoLock operation:

#### **Reactive Drift Correction**

It collects an image after a certain time lag. This time can be manually set or automatically calculated. This image is compared to the original reference image and the drift between the two images is measured. If there is a drift between the two images then beam shift is used to correct it.

Reactive drift correction can be used independently, or can be combined with Predictive Drift Correction.

#### **Predictive Drift Correction**

It applies a linear extrapolation taken from the last two drift measurements over time. It continues to correct until the next full drift measurement. Predictive correction requires two initial reference frames which are acquired before user data acquisition begins.

This method of drift correction is typically used when acquiring EBSD data as it typically suits the type of drift seen when analyzing tilted samples.

# Why does AZtec only require 2 image scans for Predictive Drift Correction when Fast Acquisition required at least 3?

The predictive algorithm in AZtec is different from Fast Acquisition and as such we only need two images. However, there might be gains to be made in increasing the initial predictive reference interval times.

#### What Is the Measurement Interval?

The measurement interval is the time in seconds between the tracking images. Drift correction is applied after each tracking image is collected. For small amounts of drift a longer measurement interval is sufficient. If the sample drifts a lot then shorter measurements are required for accurate data collection. The default value is 30 seconds.

#### What is the Automatic Measurement Interval (AMI)?

If the Automatic Measurement Interval is selected then the system will determine a suitable measurement interval for you. This interval is adaptive, so if the rate of drift changes so will the drift interval.

This option will work with both predictive and reactive drift. This measurement interval is determined based on a maximum target drift between corrections, and this maximum is 1.5 pixels.

#### How is the Automatic Measurement Interval (AMI) Set?

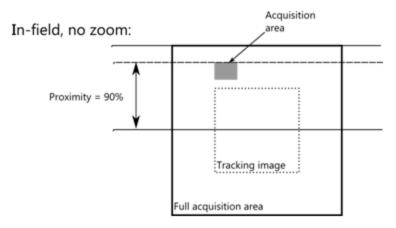
The automatic drift is calculated in the following way:

a. When the run starts if there are not enough measurements to determine a suitable interval, use either the minimum allowed user-specifiable interval (currently 5

- seconds) or use the frame time for the tracking image, whichever is larger. If Predictive Drift Correction is enabled, this limit will be either the predictive reference interval or the frame time, whichever is larger. Start accumulating drift in pixels.
- b. Once two reference images have been collected, the accumulated drift over time is determined. AZtec then determines if drift exceeds the target max drift value in pixels (the target max drift is 1.5 pixels).
- c. Assuming the target max drift has not been exceeded, the system will estimate when that target will be reached and the next measurement interval is scheduled for this point.
- d. If the target max drift has been exceeded, the system will estimate when that target was reached, and use this to determine the measurement interval. We also reset our accumulated drift to zero and restart the associated timer.
- e. The same limits apply to the automatic measurement, as are in place for the values which can be manually entered (currently 5 seconds to 30 minutes). (For Predictive Drift Correction, the minimum time is at least as big as the predictive reference interval).

#### What is 'In-Field' Mode?

In this mode the entire scanned area (i.e. field of view) is always viewed. The tracking image used is equivalent to a 2x zoom image from the center of the field of view. The acquisition area (from which the data is collected) is then a smaller area within the field, but not necessarily within the tracking image. See the figure below. In this mode the analysis will stop if either the acquisition area or the tracking image reaches the limit of the original field of view.

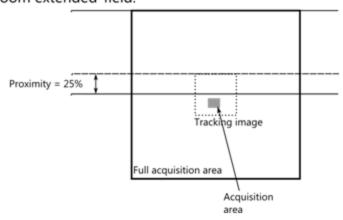


What is 'Extended-Field' Mode?



This mode requires that the image is zoomed at 2x, 4x or 8x. This reduces the size of the field being analyzed; the acquisition area has to be within the tracking image area. If the image is zoomed 2x then the center 50% of the image is taken for the tracking image. If 4x zoom is used the inner 25% of the original image is used for data collection. The amount of potential 'correction' available is therefore larger when higher zooms are used.

#### 4x zoom extended-field:



#### **But What If I want to Maintain Subject Size?**

When using extended field mode you can maintain the size of a feature on the image by enabling "Maintain Subject Size". The magnification on the SEM is changed to maintain the same field of view, based on the zoom that is selected.

Note: This option will only be available if your microscope supports it.

#### How can I solve large jumps in the Image?

If the tracking interval is set too long and the image has drifted significantly then the drift correction will be applied in a single jump. The result could be seen as a large 'shift' within the image. If you see this then set a shorter measurement interval, use the Automatic Measurement Interval, or enable Predictive Drift Correction.

#### How do the predictive and reactive drift intervals interact?

The predictive interval ("Predictive Reference Interval") determines how long AutoLock waits between the reference frames when starting predictive drift correction. The reactive drift interval ("Measurement Interval") determines how often AutoLock re-measures the drift once acquisition starts. Generally, these settings are independent. However, if the Automatic Measurement Interval is enabled, the measurement interval won't go lower than the predictive reference interval once acquisition has started.

In most cases, where the sample is either fairly stable or settling into stability, a good balance of measurement interval vs accuracy should be achievable by using the Automatic Measurement Interval with a predictive reference interval of up to a minute (where applicable).

#### What is Drift Proximity?

Drift Proximity (%) is the potential drift range as a percentage of the field width. The proximity display also shows a colored bar that extends from green to yellow to red as the limits of AutoLock are reached; the bar will be full and in the red if AutoLock limits are exceeded. The proximity value gives the worst-case drift range for the acquisition area and the tracking image. In extended-field mode, the tracking image area always determines the proximity, as it contains the acquisition area. For in-field mode, either the tracking image area or the acquisition area determines the proximity, depending on which one is closer to the edge of the field. See the illustrations under "What is 'In-Field' Mode?" and "What is 'Extended-Field' Mode?", above.

### What does the AutoLock Histogram show?

The AutoLock performance histogram shows the number of drift corrections at various distances (in tracking image pixels) that have been applied during a single acquisition; the histogram allows a rapid assessment of drift. For a stable sample most histogram values should be at or near zero. Large numbers of adjustments towards the right of the histogram (long distances) might indicate that you would benefit from using a shorter measurement interval.

# **Context Menus - Image Viewer**

A number of useful shortcut menus available as right mouse click in the image viewer are shown in the table below:

Context Menu Item	
Rescale Image	Fit Image to Display
	Fill Display with Image
View	Color Bar
	Header
	Scale Bar
	Show Con- trast/Brightness Buttons
	Color Key
Set Image Colors	



Context Menu Item	
Export	Save As (Original Resolution)
	Save As
	Сору
	Print
	Email
	Settings
Annotations	Show
	Select All
	Style
	Delete
Show Acquisition Areas	Show All
	Show Selected
	Show None
	Show Short Names
Details	

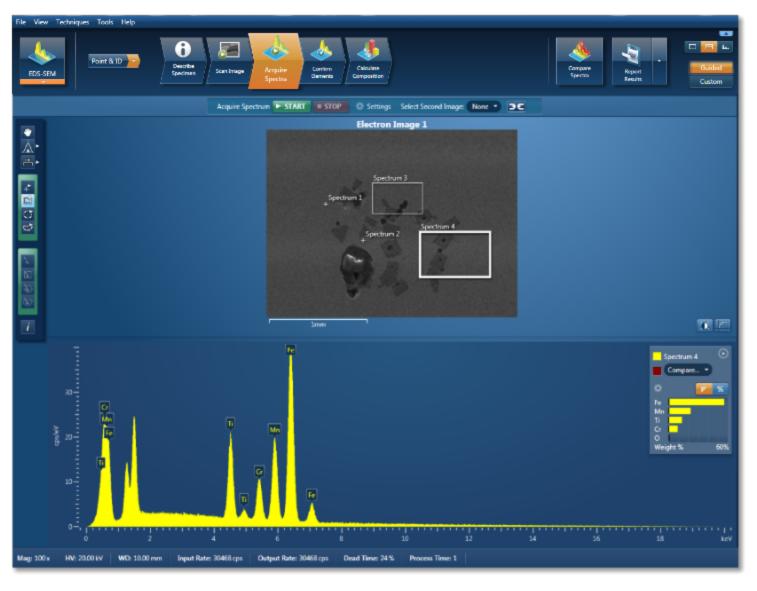
# **Acquire Spectra**



In this step you acquire spectra from the current electron image (SE/BSE). You can also reconstruct spectra from a Layered Image or X-ray map if you have already acquired SmartMaps. Real time Compare and instant MiniQuant options are also available here.

You can display the components that you are working on such as image and spectrum using

the controls on this toolbar located in the top right side of the screen. You have choice of displaying image and spectrum as shown in the screen shot below or just an image or a spectrum full screen:





There is an acquisition toolbar near the top of the workspace:



It has controls for starting and stopping the spectrum acquisition. There is a Settings cog for selecting the acquisition parameters. For details see Acquire Spectra - Settings link below.

The toolbar located on the left side of the workspace has various tools for image and spectrum manipulation, enhancement, annotation and area selection. For details see Acquire Spectra - Toolbar topic from the link below.

There are manual and auto brightness, contrast and color controls available for the image view. You can use these controls to enhance and high light certain features in the image.

The Compare Spectra & MiniQuant Results option is available in the top right corner of the Spectrum viewer. You can compare the current spectrum with any other spectrum from an opened Project on the Data Tree. Instant MiniQuant results can be viewed in a table or a bar chart.

#### See also:

How to acquire spectra on next page

Modes of X-ray spectrum acquisition on page 150

Acquire Spectra - Toolbar on page 151

Acquire Spectra - Settings on page 313

Context Menus - Spectrum Viewer on page 321

Export - Settings on page 132

Peak Labels on page 158

Element Lists on page 191

# How to acquire spectra

You can acquire a spectrum from the entire image, a single point or area on an image. You can also acquire spectra from multiple points and/or areas on an image. Tools are provided to specify points and areas on the image, for detailed description read the topic, Modes of X-ray spectrum acquisition on page 150.

The screen shot below shows the defined points and areas on the image and the spectra being populated on the Data Tree:



Spectra have been acquired from the areas and points (white) labeled as Spectrum 1, 2 and 3. Spectrum 4 is currently being acquired from the area (yellow) labeled as Spectrum 4. Spectrum 5 (blue) is in the queue.

For step by step guidance on how to acquire spectra see below:

#### Spectrum acquisition from a single point

- Having acquired an image in the Scan Image step, navigate to the Acquire Spectra step.
- Select the Point Tool from the Toolbar on the left of the window.
- Click on the image with the mouse to acquire spectrum from a point.



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  - Spectrum acquisition starts from that point. The position of the point is marked as a cross on the image and it is labeled as Spectrum 1. It is added to the Data Tree just below the image it is acquired from.
  - The progress of spectrum 1 acquisition is displayed on the Current Site tab.

#### Spectrum acquisition from multiple points

- Having acquired an image in the Scan Image step, navigate to the Acquire Spectra step.
- Select the Point Tool from the toolbar on the left of the window.
- Click on different locations on the image to define multiple points.
- Spectrum acquisition starts from the first point as soon as you click on the image.
   The rest of the points are queued up for acquisition.
- The position of each point on the image is marked as a cross and it is labeled as Spectrum x (1, 2, 3...).
- As spectra are being acquired, they are added to the Data Tree just below the image they are acquired from.
- Status of each point is color coded i.e., current point from which a spectrum is being acquired is yellow, all queued up points are blue and post acquisition points are white.

#### Spectrum acquisition from an area

- Having acquired an image in the Scan Image step, navigate to the Acquire Spectra step.
- Select an area selection tool from the Rectangle, Ellipse and Freehand in the toolbar on the left of the window.
- Click and drag the mouse to outline a rectangular, ellipsoid or irregular shaped area on the image with the Rectangle, Ellipse or Freehand Tool respectively.
- Spectrum acquisition starts from the defined area as soon as you release the mouse. The position of the defined area is marked with the relevant shape and it is labeled as Spectrum 1.
- Spectrum 1 is added to the Data Tree just below the image it is acquired from.

#### Spectrum acquisition from multiple points and areas

- Having acquired an image in the Scan Image step, navigate to the Acquire Spectra step.
- Select the Point Tool from the toolbar on the left of the window.
- Click on different locations on the image to define multiple points.
- Select an area selection tool from the Rectangle, Ellipse and Freehand in the toolbar on the left of the window.
- Click and drag the mouse to outline a rectangular, ellipsoid or irregular shaped area on the image with the Rectangle, Ellipse or Freehand Tool respectively.

- Spectrum acquisition starts in the order you have defined the points and areas. Status of each point/area is color coded i.e., current point/area from which a spectrum is being acquired is yellow, all queued up points/areas are blue and post acquisition points/areas are white.
- All points and areas defined on the image are labeled as Spectrum x (1, 2, 3...).
- As spectra are being acquired, they are added to the Data Tree just below the image they are acquired from.

# **Spectrum Acquisition Tools**

There is a toolbar on the left side of the Acquire Spectra and Acquire & Confirm windows that has four point and area selection tools. You can use these tools to define points and regions on an image to acquire spectra. See the table below for detailed description of each tool:

### **Spectrum Acquisition Tools**



#### **Point**

Click on this icon to select the Point Tool from the toolbar and then click on the image to start the spectrum acquisition from that point. This is a useful tool for quick survey of a homogenous specimen.



#### Rectangular

Click on this icon to select the Rectangular Tool. Click and drag the mouse on the image to outline a rectangular area. When you release the mouse button, an area will be outlined and a spectrum will be acquired from it. This is a useful tool for examining regular shaped features and areas on an image.



#### **Spectrum Acquisition Tools**



#### **Ellipse**

Click on this icon if you wish to define an ellipsoid area on the image. Click and drag the mouse on the image to outline an area. When you release the mouse button, an area will be outlined and a spectrum will be acquired from it. This tool allows you the flexibility of outlining an oval or ellipsoid feature on an image.

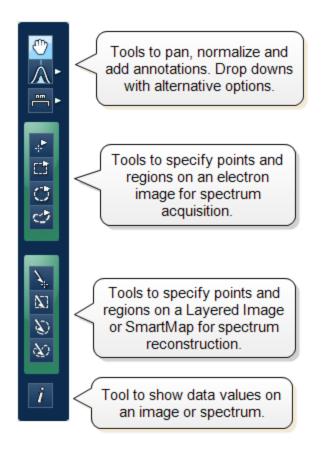


#### **Freehand**

Click on this icon if you wish to acquire a spectrum from an irregular shaped feature on the image. Click and drag the mouse around the feature on the image. Once you have defined the feature release the mouse button. A spectrum will be acquired from it.

# **Acquire Spectra - Toolbar**

The Acquire Spectra screen has a toolbar on the left side of the workspace shown in the screen shot below:



#### Pan

The Pan tool allows to expand the spectrum along the vertical axis and move the spectrum along the horizontal axis. To expand the spectrum along the horizontal axis with Pan tool selected, hold down the Ctrl key while dragging the spectrum with the left mouse.

#### **Normalize Spectra**

You can normalize two spectra over a selected point or a region.

#### **Normalize Spectra (Point)**

- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Point) option from the toolbar. The cursor turns into an up down arrow ( ).
- Double-click in the spectrum to set a normalization point along the X-axis. A window is drawn on either side of this point. Both the spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

#### **Normalize Spectra (Region)**



- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Region) option from the toolbar. The cursor turns into a crosshair (+).
- Click in the spectrum viewer to select the start point of the energy window. A default window is displayed about this point. Drag the mouse to define your window and then release it. A window will be drawn between the first point and the end point where you release the mouse. Both spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

#### **Annotations**

Five tools available to add annotations on the current spectrum and the image are Caliper, Angle, Text, Rectangle and Ellipse. Select the tool by clicking on it and then click on the spectrum/image to add annotation. For example to add text select the Text annotation tool, click on the spectrum where you wish to enter the text and then start typing the text. To delete annotation double click on it to select it and then press the Delete key on the keyboard.

#### **Spectrum Acquisition Tools**

There are four spectrum acquisition tools, Point, Rectangle, Ellipse and Freehand. Select a tool to acquire spectra from points and/or regions on the image. For details see Modes of X-ray spectrum acquisition on page 150

For step by step advice on spectrum acquisition see How to acquire spectra on page 148.

#### **Spectrum Reconstruction Tools**

You can reconstruct spectra from SmartMaps using Point, Rectangle, Ellipse and Freehand tools. These tools are enabled on map acquisition.

#### **Show Data Values**

With this tool you can view the Energy (keV) and counts in any channel of the spectrum. Simply select the Show Data Values tool from the toolbar and then hover on spectrum. The values will be displayed as you move from channel to channel.

#### Acquisition and Settings Toolbar

Near the top of the Acquire Spectra window, there are buttons for starting and stopping acquisition as shown below:



Press the Start button to acquire a spectrum from a point or region specified on the electron image.

There is also a Settings cog in the toolbar. For details of settings see Acquire Spectra - Settings on page 313.

You can choose to display two electron images (SE/BSE) side by side by selecting the second image from the drop down list in the toolbar. Images can be linked for manipulation using the Link button.

## **Acquire Spectra - Settings**

The settings are described in detail below:

## **Energy Range (keV)**

Select a spectrum energy range from the available options of Auto, 0-10, 0-20 or 0-40 keV from the Energy Range drop down list.

An appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited.

In the Auto mode, the system checks for the accelerating voltage set on the microscope and selects a suitable energy range in the software.

### **Number of Channels**

Select number of channels from the drop down list of Auto, 1024, 2048 or 4096 (4K) with which you wish to display the spectrum. The number of eV/channel will depend on both the energy range and the number of channels you select:

Energy Range (keV)	Number of Channels	eV/channel
0-10	4096	2.5
0-10	2048	5
0-10	1024	10
0-20	4096	5
0-20	2048	10
0-20	1024	20
0-40	4096	10
0-40	2048	20
0-40	1024	40

In the Auto mode, the system checks for the energy range selected and sets the appropriate number of channels.



#### Note

The Energy Calibration routine is performed for all process times and for all available energy ranges and number of channels. It means if you change any of these settings soon after you had performed the Energy Calibration you do not need to re-optimize the system.

#### **Process Time**

Select the Process Time from the drop-down list of Process Times, Default and 1 to 6. The Process time is the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process time, the lower the noise. If noise is minimized, the resolution of the peak displayed in the spectrum is improved, in other words, the peak is narrower and it becomes easier to separate or resolve, from another peak that may be close by in energy.

If Default is selected, the Process Time is automatically set to a suitable value.

There is a trade off between the Process time that is used, and the speed at which data can be acquired into the X-ray spectrum. Process time 1 is the shortest, and as such, gives the highest X-ray acquisition rates, but at some cost to resolution. Process time 6 is the longest, and gives the highest resolution, but at some cost to maximum acquisition rate. The longer the Process time, the slower data can be acquired, i.e. the higher the system Deadtime will be for a given input count rate. (The input rate is not affected by the pulse processor).

#### Which Process Time should I use?

When you start your application first time, the Process Time is set to Default. This is a suitable choice for many routine applications where you are looking for good resolution of peaks and fast acquisition.

For the first look at a specimen you should use a long process time (5 or 6) to start with in order that you do not miss any detail in your spectrum. For example, when identifying peaks particularly those closely spaced and overlapping, it is important to get good peak separation. Good resolution is also important for looking at a series of lines that are very closely spaced, like an L series and process times 4 to 6 should be chosen. Common overlaps include the Mo L and the S K lines.

If there are no closely spaced peaks then you can afford to use a shorter Process Time such as 1-3 which will enable you to increase the acquisition rate by increasing the beam current. A compromise between acquisition speed and resolution should be found if there are peak overlaps.

When acquiring SmartMap data you should choose your Process Time carefully.

1. You may have been working on a Specimen in either Analyzer or Point & ID where you have setup your acquisition parameters to optimize your quantitative analysis. If you now wish to acquire SmartMap data and you think you may wish to reconstruct spectra from your SmartMap data and then quantify these spectra, you

- should maintain these acquisition parameters. This means that you may have to acquire data with a long Process Time to maximize resolution but limit the maximum acquisition rate.
- 2. You may have been working in either Analyzer or Point & ID and you want to view the distribution of elements whose main peaks do not overlap as a map or a linescan. In this case you should use a shorter Process Time which will mean that you can work with higher acquisition rates and shorter acquisition times. The choice of Process Time will very much depend on your sample and what you wish to do with your SmartMap data once it has been acquired.
- 3. If you have started your Project in Map and you are analyzing an unknown sample, we recommend that you use a long Process Time in order that you do not miss any detail in your spectrum. However if you only wish to map certain elements whose main lines do not overlap, you can afford to shorten the Process Time and increase the acquisition rate by increasing the beam current.

## **Acquisition Mode**

There are three options to terminate the acquisition, Auto, Live Time and Counts.

If Auto mode is selected, acquisition continues until enough counts are collected in the spectrum for quantification.

You can choose to terminate acquisition at the end of a preset Live Time. Enter the required time in seconds into the text box. This is the time for which the system is processing counts into the spectrum. The live time clock runs slower than the real time clock so that the acquisition for '100' live seconds takes longer than 100 real seconds. This time is extended to compensate for the output rate being less than the input rate by the degree of Deadtime.

You can choose to terminate acquisition at the end of a preset number of counts. Enter the value in the Count Limit text box. The default value is 500,000.

## **Pulse Pile Up Correction**

Check Pulse Pileup Correction check box if you wish to automatically correct the spectrum for pulse pileup peaks. Uncheck the box if you wish to disable this correction.

Pileup peaks can occur when a second pulse arrives and triggers the measuring system during the time required to process a previous pulse. When this happens, neither pulse will appear in its correct position. The result being a peak at a higher energy equivalent to the sum of the energy of the two photons.

The largest pileup peaks will be seen at twice the energy of the main peaks - e.g. Fe Ka pile up peaks will be seen around 12.8 keV.

#### Notes

The pileup correction algorithm assumes that the count rate at every energy is constant throughout the analysis period. Therefore, the correction works best when analysis is performed on single pixels, points or areas of same composition. Bad results may be obtained if the beam is rastered over an area where composition is changing or if a spectrum is reconstructed from a SmartMap over a region where the composition is changing.



# **Context Menus - Image Viewer**

A number of useful shortcut menus available as right mouse click in the image viewer are shown in the table below:

Context Menu Item	
Rescale Image	Fit Image to Display
	Fill Display with Image
View	Color Bar
	Header
	Scale Bar
	Show Contrast/Brightness Buttons
	Color Key
Set Image Colors	
Export	Save As (Original Resolution)
	Save As
	Сору
	Print
	Email
	Settings
Annotations	Show
	Select All
	Style
	Delete
Show Acquisition Areas	Show All

Context Menu Item	
	Show Selected
	Show None
	Show Short Names
Details	

## **Peak Labels**

Peaks in the spectrum are automatically labeled during acquisition if AutoID is enabled in Predefined Elements tab in the Describe Specimen step.

You can configure peak labels from the Peak Labels tab available in two places in the software:

- User Profile
- Confirm Elements step

## **Default Peak Label Behavior**

The default peak labeling behavior is controlled by the User Profile. You can access the User Profile screen from the Tools menu on the main application window. Select the Peak Labels tab from the User Profile screen:



You can select how you wish to display labels on the peaks from the following three options:



- 1. Element Symbols (e.g., Mn)
- 2. Element Symbol & Line (e.g., Mn Kα1)
- 3. Element name (e.g., Manganese)

Make your selection by checking the relevant radio button. This configuration is saved with the User Profile. Note that peaks will be labeled according to your new selection on the next spectrum acquisition. The labels on the current spectrum will not be automatically updated. To update the labels on an existing spectrum, use the 'Apply Profile' button on the Confirm Elements step shown in the screen shots later on in this topic.

When you click on an element symbol on the Periodic table, a list of X-ray lines together with their energies is displayed. This list corresponds to those in the X-ray database. The lines which are checked are the ones used that will be used for labeling peaks. You can check or uncheck a line to select or deselect it for labeling. To restore the settings to the factory set-

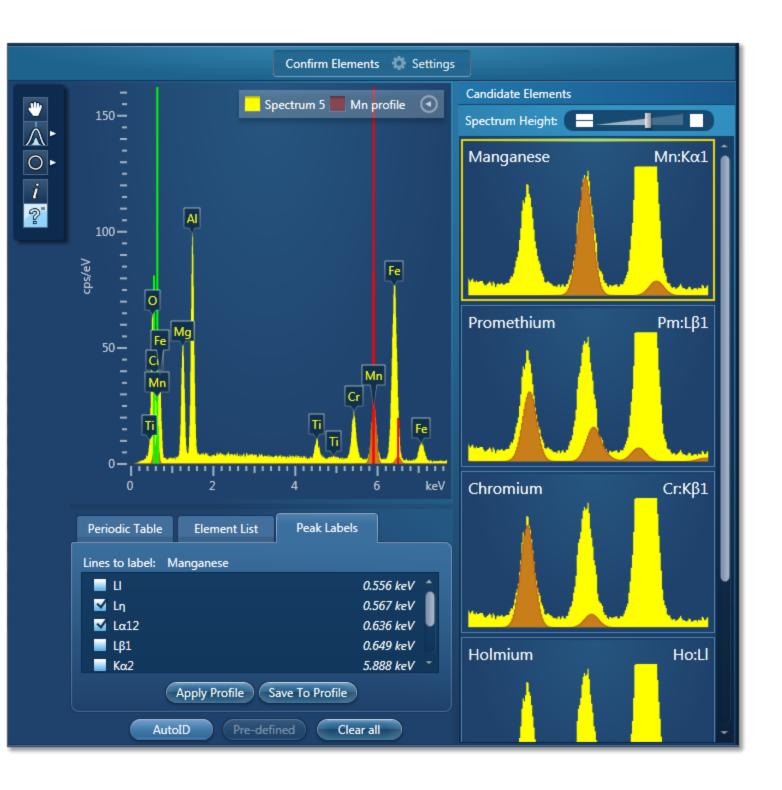
tings for this element press the Reset Element buti

You may have configured lines for other elements. To restore them to the factory settings press the button. This action will restore all the elements to the default settings.

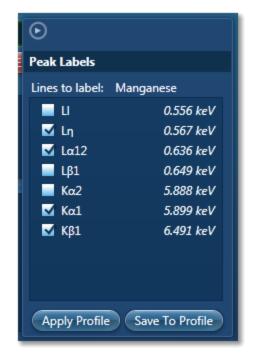
## **Refining Peak Labels Manually**

You can manually refine the peak labels for an existing spectrum in the Confirm Elements step.

You can configure which lines you wish to be labeled for each element from the Peak Labels tab or Peak Labels slide-out menu as shown the screen shots below:







The lines which are checked are the ones used for labeling the peaks in the current spectrum. You can check or uncheck a line to add or remove the label. To save your labeling con-

figuration for an element in the User Profile, press the button after checking the lines.

To use the labeling configuration saved in the User profile press the button. This action will remove all labels from the current spectrum and apply labels according to the scheme saved in the User Profile.

## **Moving Peak Labels Manually**

Click on the peak label that you wish to move and drag it to a new position by holding the left mouse button.

After moving the peak labels on the current spectrum you can restore them to their original position by selecting 'Reset Positions' from the Peak Labels section of the context menu on the Spectrum viewer.

#### **Notes**

- If there are too many labels too close together only main peak labels are displayed. The hidden labels will reappear if you stretch the spectrum along the x-axis.
- You may find that previously hidden labels show up when labels are moved or deleted.
- All existing peak labels are removed and the spectrum is relabeled according to the User Profile whenever the Element List changes e.g., when an element is manually added or removed or when AutoID is used.

## **Element Lists**

Any list of elements in the software can be split into the following three categories:

- Pre-defined Elements elements expected in specimen
- Identified Elements typically based on automatic peak identification (Auto ID)
- Fixed List used for Quantitative analysis

#### **Pre-defined Elements**

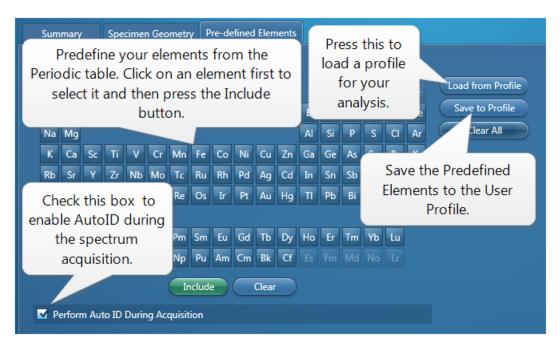
You may have prior knowledge of your Specimen and know what elements to look for.

## Examples

'I want to look for a particular list of elements. (I am not interested in any other elements)...I may want to see their labels on spectra, their X-ray maps or both....I want to see these even if the element is not present'.

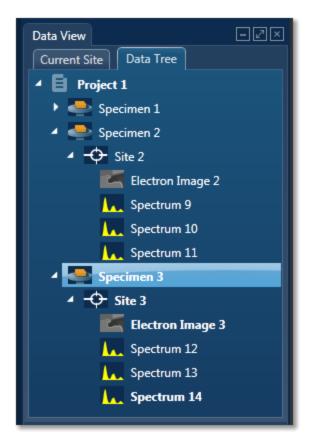
' I know what's in my sample.... I want to look for a specific set of elements (I want to see these even if the element is not present.) .....but I would like to know if there is anything else in my sample too'.

You can define these elements in the 'Pre-defined Elements' tab in the Describe Specimen step. If you want to save the Pre-defined Elements to a profile you must first press 'Save to Profile' button, then save the profile via the drop down menu. When you want to analyze your Specimen, you can load this profile or another profile by pressing the 'Load Profile' button in the Describe Specimen step as shown in the screen shot below:



Note that the 'Pre-defined Elements' are saved with the current Specimen. Changing the 'Predefined Elements' will only update the Pre-defined Elements in the current Specimen. It will not update any existing Specimens in the Project.





The current specimen is the one that you are presently analyzing/processing the data from. For example, in the screen shot above, Specimen 3 is the current specimen, Specimens 1 and 2 are the other specimens in the Project.

## **Identified Elements**

The 'Identified Elements will include:

- Any Pre-defined Elements
- Elements identified by Auto ID
- Any additional Elements identified manually

If the 'Pre-defined Elements have been specified, these will be included for identifying and labeling peaks in the current spectrum automatically.

Note that the 'Identified Elements' are saved in the Spectrum.

'Perform Auto ID During Acquisition' option is enabled by default and can be deactivate by un-checking it in the Describe Specimen step as shown in the screen shot above. You can then AutoID at any time by pressing the button.

Additional peaks in the spectrum can be identified manually by using the 'Show Candidate Elements' tool in the Confirm Element step. Click on the question mark icon to select the Show Candidate Element tool. Position the cursor at the center of a peak by double-clicking

with the mouse. The list of elements spectra corresponding to the energy at the cursor is displayed in the panel on the right. By highlighting an element in this list, you will see the markers showing all the lines for this element.

Note that the 'Identified Elements' will be quantified if you have selected the Current Spectrum, or the Fixed List and Current Spectrum Element List in the Quant Settings in the Calculate Composition step or EDS Quant Settings in the User Profile dialog.

## Note

EDS Quant Settings are available in the User Profile Dialog accessed from the Tools menu. These settings are also available from the Calculate Composition step.

#### **Fixed List**

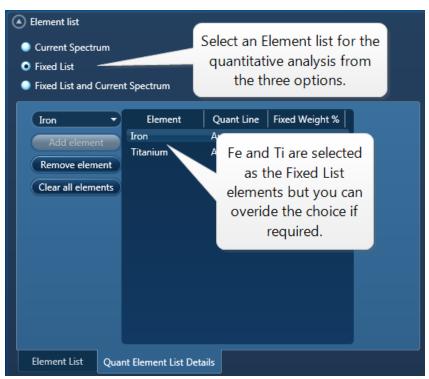
The elements in the 'Fixed List' are defined in the Quant Settings dialog which is available in the User Profile and the Calculate Composition window.

Note that the Fixed List is only used for calculating composition in quantitative analysis.

## Example

'I want to do quantitative analysis on my glass samples and want to compare results from one batch to another batch. I am always looking for the same specified list of elements'.

You can specify the Element List for Quant from the three available options in the Quant Settings dialog as shown in the screen shot below:



## **Current Spectrum**



This list includes the Pre-defined Elements and elements identified by Auto ID and manually using the Candidate Element tool.

#### **Fixed List**

You select the Fixed List option if you know what elements you wish to quantify. Choose the elements from the drop-down list as shown in the screen shot above.

## **Fixed List and Current Spectrum**

To quantify the elements in the above two lists, select the Fixed List and Current Spectrum option.

MiniQuant results table will clearly display which list is being used. A lock icon will be displayed against the 'Fixed List' elements as shown in the screen shot below:



In this example, Fe and Ti are selected in the Fixed List. The rest of the elements in the chart results are from the Current Spectrum because the Element List selected for quantification was 'Fixed List and Current Spectrum'.

Note that the 'Fixed List' is saved in a User Profile.

## See also:

Describe Specimen on page 76

Acquire Spectra on page 350

Confirm Elements on page 353

Calculate Composition on page 185

User Profile on page 22

## **Compare Spectra & MiniQuant Results**

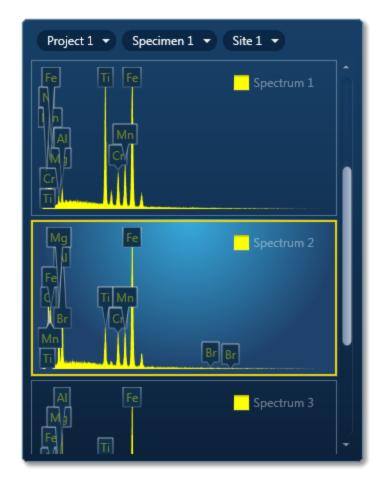
Real time Compare and instant MiniQuant options are available in the Acquire Spectra, Confirm Elements and Calculate Composition (Comparison of Results - Two Spectra template) steps. User can see results without having to move away from the acquisition mode. Using these options you can:

- See your results during analysis.
- Compare your current spectrum to a control spectrum during acquisition.
- View MiniQuant results in a table or a bar chart.

Click in the top right corner of the Spectrum Viewer in Acquire Spectra, Confirm Elements or the Calculate Composition window to access the Compare & MiniQuant options:



In the above example, Spectrum 1 is the current spectrum and Spectrum 2 is the comparison spectrum. You can select the comparison spectrum from a Project currently available in the Data Tree. It can be from any Project, any Specimen and any Site of Interest currently available in the Data tree. To choose the comparison spectrum click on the down arrow (Spectrum 2 in the above example). Spectra available in the current Project, Specimen and Site of Interest are displayed as below:



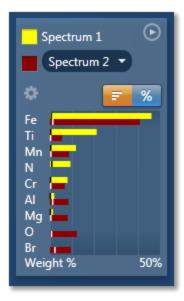
Click on the spectrum in the display to select it for comparison. The selected spectrum will be overlaid as a line spectrum over the current spectrum. The MiniQuant results are displayed in a table as shown in the example below:



The results are displayed as wt% (weight%).

The statistical error is displayed as  $\sigma$  (weight% sigma) for the calculated wt%. It is the overall confidence figure for the analysis. You can use sigma to assess the results especially when an element is present at low concentration. For example, if an element concentration is 0.2 wt% and the  $\sigma$  is 0.12 wt%, the element might be detected at a statistically significant level if the acquisition time for the spectrum is extended. If the  $\sigma$  is 0.4 wt%, it is pointless to extend the acquisition time and it is safe to assume that the element if present, is at a level above the limit of detection for this technique.

Press to display the results in a chart:



The sigma values are displayed as black or white vertical bands across the bars in the chart results as shown in the example above. In this case the full scale of the bar chart is 50%.

If you wish to change the MiniQuant Settings press





Make your selection by clicking on the radio button and then press the Apply button. The results will be updated immediately .

## Note

The Quant Settings in the MiniQuant and Calculate Composition are the same. Updating one updates the other and vice versa.

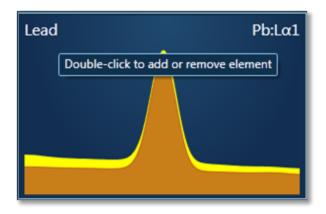
## **Confirm Elements**



This step has been designed to help you confirm the elements that have been identified by AutoID in your spectrum. These elements are then used to create a confirmed elements list for qualitative and quantitative analyses. Extensive tools including Element Series Markers, Overlays, Element Profiles and Show Candidate Elements are available here to assist you in confirming elements manually.

#### How to confirm elements:

Start with the largest peaks. Press the question mark icon to select the Show Candidate Elements tool from the tool bar on the left hand side of the interface, then double click on a peak in the spectrum viewer. The candidate elements are displayed in a stacked spectra view on the right hand side of the window (you can double click on any of these elements to add or remove it from the confirm elements list).



You can control what overlays you see in the Spectrum viewer via the 'Confirm Elements Settings'. These overlays can be very useful in helping you to interrogate complex spectra.





 Press Include/Exclude once you are satisfied with the identification of each element to build your list of the confirmed elements.

#### See Also:

Confirm Elements - Settings below

Confirm Elements - Tools on page 174

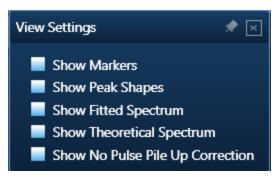
Element Lists on page 191

Peak Labels on page 158

Compare Spectra & MiniQuant Results on page 195

## **Confirm Elements - Settings**

To display the Confirm Elements Settings, click on the cog icon near the top of the screen. The various option available are shown in the screen shot here:



To select any of these options simply check the associated check box.

## **Show Markers**

Clicking on one of the elements in the confirmed elements list puts up the markers for that element. Lines series are color coded. K series lines are marked in red, L series lines are marked in green and M series lines are marked in purple.

#### **Show Peak Shapes**

Peak Shapes (one for each line series for each element) are fitted to the spectrum using the Filtered Least Squares, FLS approach. This effectively removes the effect of background and corrects for peak overlap. The Fitted Spectrum shows the fitted peak shapes superimposed on a close approximation to the background and this makes it easy to see if a particular element has been missed or a peak shape is a bad match to the observed peak shape.

## **Show Fitted Spectrum**

This tool is very useful for checking for the presence of small amounts of elements whose peaks are heavily overlapped in the spectrum.

It overlays a fitted spectrum onto the current spectrum. The shape of the fitted spectrum is based on the elements labeled in the current spectrum (AutoID). If any peaks are incorrectly labeled, or any elements missed, then the fitted spectrum will not overlay correctly on your current spectrum. Use the Show Candidate Elements tool to identify elements on the part of the spectrum where the fitted spectrum and the current spectrum have their greatest discrepancy. Then add the possible elements identified manually by double clicking on them individually in the stacked spectra view and test if they improve the overlay fit.

#### **Show Theoretical Spectrum**

This tool calculates a full x-ray spectrum from the analyzed composition. This calculation includes the efficiency of excitation of all lines, the effects of absorption and backscatter within the sample and calculates the relative intensity of both lines and bremsstrahlung background. Although the theory is not perfect, it normally predicts peaks and background within about 10% accuracy. If elements have been misidentified or element composition ratios are incorrect, then the Theoretical Spectrum will appear significantly different from the observed spectrum, either in terms of peak intensities or background. When the theoretical spectrum is a good match to the observed spectrum, this provides useful confirmation that the analysis results are sensible.

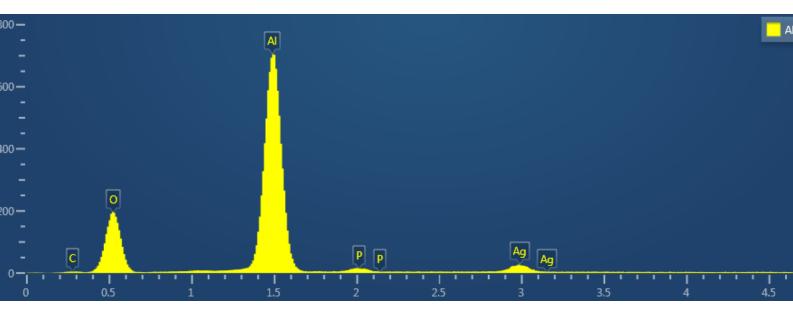
Note that the Theoretical Spectrum is calculated assuming that the specimen is flat and homogeneous. If the specimen shows a lot of topography or has composition that varies throughout the information volume, then the calculation will not be relevant. For example, if a spectrum is obtained from a point that is not directly visible by the detector, then the emitted x-rays will be absorbed on their way to the detector by an unknown amount or unknown material so the overall effect cannot be predicted.

#### **Show No Pulse Pile Up Correction**

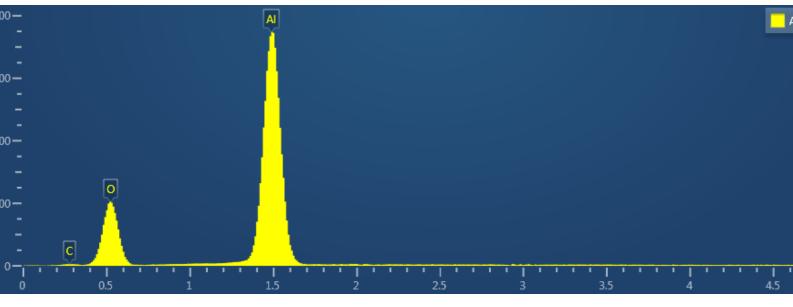
This is a useful tool for showing the pile up peaks (sum peaks) that have been removed from the spectrum by the pulse pile up procedure.

The pile up peaks need to be removed from the spectrum, because if left they can be misassigned as characteristic X-ray lines of elements which are not present in the specimen.

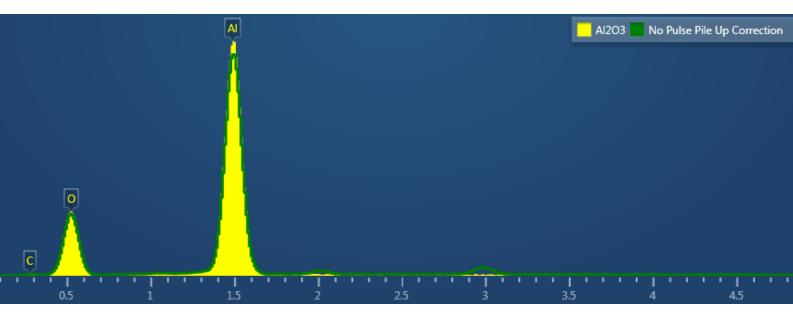
The  ${\rm Al}_2{\rm O}_3$  spectrum below contains pile up peaks which are identified as P and Ag:



When pile up correction is applied to a spectrum, the pile up artifacts are removed, but there is no indication of where in the spectrum the correction has been applied. The pile up corrected spectrum is shown below:



When the 'Show No Pile Up Correction' option is checked, the current spectrum is overlaid with a version that has no pile up correction applied to it. The differences seen between the pile up corrected spectrum and the 'Show No Pile Up Correction' overlay indicate where the pile up artifacts have been removed from the spectrum as shown in the spectrum below. The overlay can help to identify which peaks in a spectrum are pile up peaks.



## See Also:

Pulse Pile Up Correction

## **Confirm Elements - Tools**

A toolbar is located near the top left side of the Confirm Elements step. There are five different tools to manipulate the spectrum. These are Pan, Normalize, Annotations, Show Data Values and Show Candidate Elements:



## Pan

The Pan tool allows to expand the spectrum along the vertical axis and move the spectrum along the horizontal axis. To expand the spectrum along the horizontal axis with Pan tool selected, hold down the Ctrl key while dragging the spectrum with the left mouse.

## **Normalize Spectra**

You can normalize two spectra over a selected point or a region.

## **Normalize Spectra (Point)**



- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Point) option from the toolbar. The cursor turns into an up down arrow ( ).
- Double-click in the spectrum to set a normalization point along the X-axis. A window is drawn on either side of this point. Both spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

## Normalize Spectra (Region)

- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Region) option from the toolbar. The cursor turns into a crosshair (+).
- Click in the spectrum viewer to select the start point of the energy window. A default window is displayed about this point. Drag the mouse to define your window and then release it. A window will be drawn between the first point and the end point where you release the mouse. Both spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

#### **Annotations**

Three tools available to add annotations on the current spectrum are Text, Rectangle and Ellipse. Select the tool by clicking on it and then click on the spectrum to add annotation. For example to add text select the Text annotation tool, click on the spectrum where you wish to enter the text and then start typing the text. To delete annotation double click on it to select it and then press the Delete key on the keyboard.

## TIP!

To delete all annotations at once go into the context menu on the spectrum viewer. Choose Select All from the Annotations menu and then select Delete from the Annotations menu. This operation will remove all the annotations from the current spectrum.

## **Show Data Values**

With this tool you can view the Energy (keV) and counts in any channel of the spectrum. Simply select the Show Data Values tool from the toolbar and then hover on spectrum. The values will be displayed as you move from channel to channel.

#### **Show Candidate Elements**

Click on the question mark icon to select the Show Candidate Element tool. Position the cursor at the center of a peak by double-clicking with the mouse. Note, you may wish to expand the spectrum horizontally by holding down the control key and dragging the spectrum with

the mouse. The list of elements spectra corresponding to the energy at the cursor is displayed in the panel on the right. By highlighting an element in this list, you will see the markers showing all the lines for this element.

The profile of each candidate element is overlaid on the current spectrum. The color of the profile is different from the current spectrum to enhance the display and assist you in identifying and confirming elements.

## **Spectrum Height**

This tool aids in the manipulation of the height of each candidate element spectrum using the slider bar:



## **Display MiniQuant & Compare**

Real time compare and instant MiniQuant options are available in Acquire Spectra, Confirm Elements and Calculate Composition steps. You can see results without having to move away from the acquisition mode. You can also compare the current spectrum to a control spectrum during acquisition. You can view MiniQuant results compared in a table or as a bar chart.

The element list is taken from the Quant Settings in the Calculate Composition step. The default Element List is Current Spectrum.

## **Element Lists**

Any list of elements in the software can be split into the following three categories:

- Pre-defined Elements elements expected in specimen
- Identified Elements typically based on automatic peak identification (Auto ID)
- Fixed List used for Quantitative analysis

#### **Pre-defined Elements**

You may have prior knowledge of your Specimen and know what elements to look for.

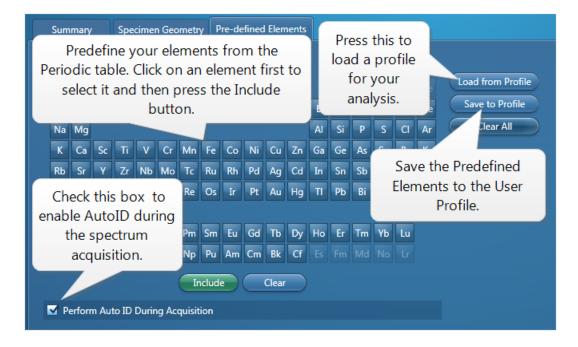
#### Examples

'I want to look for a particular list of elements. (I am not interested in any other elements)...I may want to see their labels on spectra, their X-ray maps or both....I want to see these even if the element is not present'.

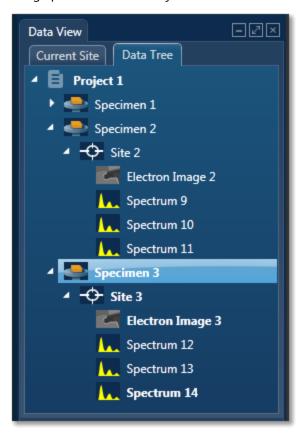
' I know what's in my sample.... I want to look for a specific set of elements (I want to see these even if the element is not present.) .....but I would like to know if there is anything else in my sample too'.

You can define these elements in the 'Pre-defined Elements' tab in the Describe Specimen step. If you want to save the Pre-defined Elements to a profile you must first press 'Save to Profile' button, then save the profile via the drop down menu. When you want to analyze your Specimen, you can load this profile or another profile by pressing the 'Load Profile' button in the Describe Specimen step as shown in the screen shot below:





Note that the 'Pre-defined Elements' are saved with the current Specimen. Changing the 'Predefined Elements' will only update the Pre-defined Elements in the current Specimen. It will not update any existing Specimens in the Project.



The current specimen is the one that you are presently analyzing/processing the data from. For example, in the screen shot above, Specimen 3 is the current specimen, Specimens 1 and 2 are the other specimens in the Project.

#### **Identified Elements**

The 'Identified Elements will include:

- Any Pre-defined Elements
- Elements identified by Auto ID
- Any additional Elements identified manually

If the 'Pre-defined Elements have been specified, these will be included for identifying and labeling peaks in the current spectrum automatically.

Note that the 'Identified Elements' are saved in the Spectrum.

'Perform Auto ID During Acquisition' option is enabled by default and can be deactivate by un-checking it in the Describe Specimen step as shown in the screen shot above. You can then AutoID at any time by pressing the button.

Additional peaks in the spectrum can be identified manually by using the 'Show Candidate Elements' tool in the Confirm Element step. Click on the question mark icon to select the Show Candidate Element tool. Position the cursor at the center of a peak by double-clicking with the mouse. The list of elements spectra corresponding to the energy at the cursor is displayed in the panel on the right. By highlighting an element in this list, you will see the markers showing all the lines for this element.

Note that the 'Identified Elements' will be quantified if you have selected the Current Spectrum, or the Fixed List and Current Spectrum Element List in the Quant Settings in the Calculate Composition step or EDS Quant Settings in the User Profile dialog.

## Note

EDS Quant Settings are available in the User Profile Dialog accessed from the Tools menu. These settings are also available from the Calculate Composition step.

#### **Fixed List**

The elements in the 'Fixed List' are defined in the Quant Settings dialog which is available in the User Profile and the Calculate Composition window.

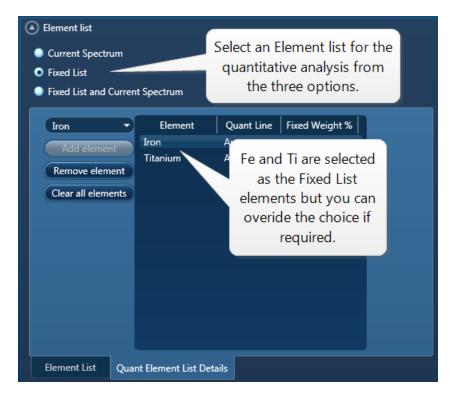
Note that the Fixed List is only used for calculating composition in quantitative analysis.

## Example

'I want to do quantitative analysis on my glass samples and want to compare results from one batch to another batch. I am always looking for the same specified list of elements'.

You can specify the Element List for Quant from the three available options in the Quant Settings dialog as shown in the screen shot below:





## **Current Spectrum**

This list includes the Pre-defined Elements and elements identified by Auto ID and manually using the Candidate Element tool.

#### **Fixed List**

You select the Fixed List option if you know what elements you wish to quantify. Choose the elements from the drop-down list as shown in the screen shot above.

## **Fixed List and Current Spectrum**

To quantify the elements in the above two lists, select the Fixed List and Current Spectrum option.

MiniQuant results table will clearly display which list is being used. A lock icon will be displayed against the 'Fixed List' elements as shown in the screen shot below:



In this example, Fe and Ti are selected in the Fixed List. The rest of the elements in the chart results are from the Current Spectrum because the Element List selected for quantification was 'Fixed List and Current Spectrum'.

Note that the 'Fixed List' is saved in a User Profile.

## See also:

Describe Specimen on page 76

Acquire Spectra on page 350

Confirm Elements on page 353

Calculate Composition on page 185

User Profile on page 22

## **Compare Spectra & MiniQuant Results**

Real time Compare and instant MiniQuant options are available in the Acquire Spectra, Confirm Elements and Calculate Composition (Comparison of Results - Two Spectra template) steps. User can see results without having to move away from the acquisition mode. Using these options you can:

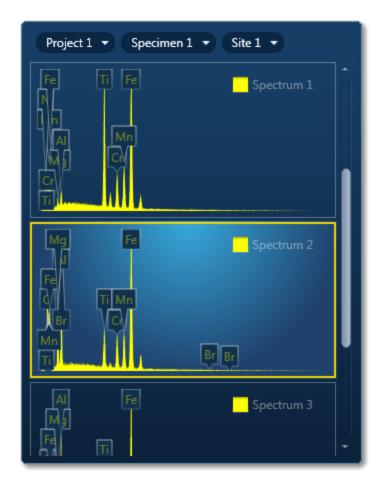
- See your results during analysis.
- Compare your current spectrum to a control spectrum during acquisition.
- View MiniQuant results in a table or a bar chart.

Click in the top right corner of the Spectrum Viewer in Acquire Spectra, Confirm Elements or the Calculate Composition window to access the Compare & MiniQuant options:





In the above example, Spectrum 1 is the current spectrum and Spectrum 2 is the comparison spectrum. You can select the comparison spectrum from a Project currently available in the Data Tree. It can be from any Project, any Specimen and any Site of Interest currently available in the Data tree. To choose the comparison spectrum click on the down arrow (Spectrum 2 in the above example). Spectra available in the current Project, Specimen and Site of Interest are displayed as below:



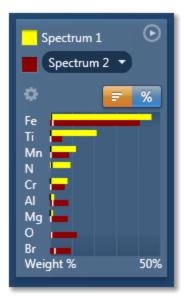
Click on the spectrum in the display to select it for comparison. The selected spectrum will be overlaid as a line spectrum over the current spectrum. The MiniQuant results are displayed in a table as shown in the example below:



The results are displayed as wt% (weight%).

The statistical error is displayed as  $\sigma$  (weight% sigma) for the calculated wt%. It is the overall confidence figure for the analysis. You can use sigma to assess the results especially when an element is present at low concentration. For example, if an element concentration is 0.2 wt% and the  $\sigma$  is 0.12 wt%, the element might be detected at a statistically significant level if the acquisition time for the spectrum is extended. If the  $\sigma$  is 0.4 wt%, it is pointless to extend the acquisition time and it is safe to assume that the element if present, is at a level above the limit of detection for this technique.

to display the results in a chart:

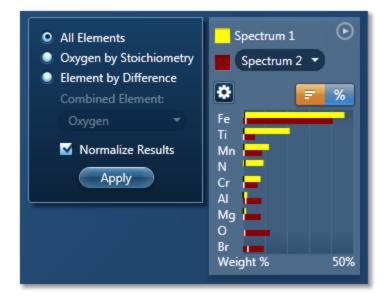


The sigma values are displayed as black or white vertical bands across the bars in the chart results as shown in the example above. In this case the full scale of the bar chart is 50%.

If you wish to change the MiniQuant Settings press







Make your selection by clicking on the radio button and then press the Apply button. The results will be updated immediately .

## Note

The Quant Settings in the MiniQuant and Calculate Composition are the same. Updating one updates the other and vice versa.

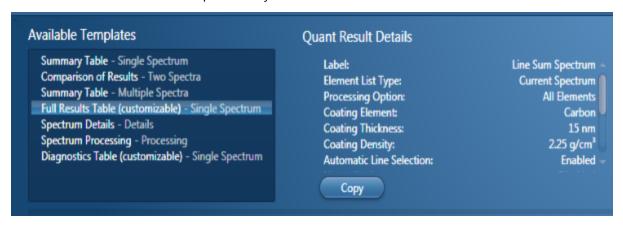


# **Calculate Composition**

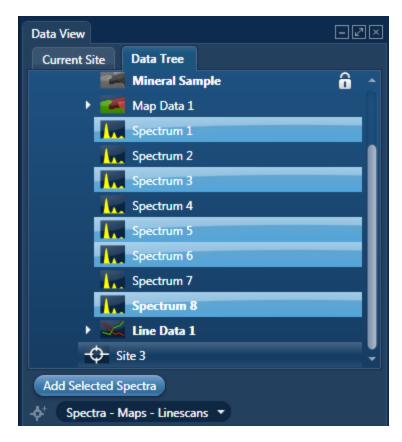


In this step you can view quant results in more detail using any of the 'Available Templates'.

To view result select the template that you wish to use:



- If you want to see a comprehensive set of results from a single spectrum, then choose the 'Full Results Table (customizable) - Single Spectrum' template and whichever spectrum is highlighted in the Data Tree will have its results shown in this template.
- To populate a multiple spectra template, hold the Ctrl key down while choosing spectra on the Data Tree and then press the 'Add Selected Spectra' button at the bottom of the Data Tree window.



- To compare quant results from two spectra, select 'Comparison of Results Two Spectra' template. Then select the comparison spectrum from the Compare option in the 'Mini Quant and Compare' option. The compare spectrum will be overlaid on the current spectrum in the Spectrum Viewer. The quant results will be displayed in the table below.
- If you wish to change the Quant Settings press the Settings button to display the Quant Settings dialog. Apply the changes and close the dialog.
- Press the Requantify button to display the recalculated results.

## **Quant Results Details**

You can see the settings used for calculating the composition in the Quant Results Details list box:

Parameter	Description
Label (Spectrum Label)	E.g., Spectrum 1
Element List Type	Current Spectrum, Fixed List or Combined List
Processing Options	All Elements, Element by Difference, Combined Element or Oxygen by Stoichiometry
Apply Coating Correction	Enabled or Disabled



Parameter	Description
Coating Element	E.g., Carbon
Coating Thickness	E.g., 15 nm
Coating Density	E.g., 2.25 g/cm3
Automatic Line Selection	Enabled or Disabled
Normalization	Enabled or Disabled
Thresholding	Enabled or Disabled
Deconvolution Elements	None/Selected
Factory Standards	Standardizations file supplied with the system
User Standards	Standardizations file defined by the user
Pulse Pile Up Correction	Enabled/Disabled
Detector File	Indicates file that has been used to characterize detector
Efficiency	Calculated/File based

## **Quant Results View**

The information displayed in the Quant Results View depends on which template has been selected. You can view Spectrum Details, Spectrum Processing and Diagnostics table in addition to quant results.

#### See Also:

**Quant Settings below** 

Element Lists on page 191

Compare Spectra & MiniQuant Results on page 195

# **Quant Settings**

The Quant Settings are described below:

## **Processing Options**

To make the correct selection, a little knowledge of the specimen is required. For example, can all elements in the specimen be detected and analyzed, or are you analyzing a mineral where it is more usual to calculate the oxygen present?

## All Elements

This option is used when processing spectra from specimens in which all elements yield X-rays which can be readily detected e.g., steels, alloys and other materials with insignificant amounts of elements lighter than sodium.

## • Element by Difference

This option can be used if you can readily measure X-ray signal from all elements except one in the specimen. The omitted element is called the combined element. The concentration of the combined element is not measured, but it is calculated assuming that the difference between the analyzed total and 100% is due only to the presence of this element. Intensity corrections are calculated assuming the presence of this element. The total from this type of analysis is always 100%.

This option can be used when analyzing a specimen in which a significant quantity of a light element which cannot be detected, is known to be present. This method can also be used in cases where an element for which no standard is available is present.

## • Oxygen by Stoichiometry

Use this option if you want the concentration of oxygen to be calculated assuming that it is bound by predefined stoichiometry to all the other analyzable elements. The stoichiometry is defined by the valency of the oxygen ions and the valencies of other measured elements:

#### Number of Ions

Enter the number of oxygen ions that are combined stoichiometrically to the other elements. The calculations are based on the number of oxygen ions and how many atoms there are in each unit cell.

#### Valency

When 'Oxygen by Stoichiometry' is selected, the option for choosing the valency for each analyzable element becomes available. The most common value for the valency of the element is displayed when you click on an element symbol on the periodic table. To use a different value, enter the new value in the Valency text box.

#### **Normalize Results**

When this option is selected the analytical total of an analysis carried out using All elements or Oxygen by Stoichiometry is forced to 100%.

Use of this needs care in interpreting the final result since if, for example, the element list is incomplete, there will be serious errors in the result, even though the total is 100%.

This is often used as an expedient where the beam current is unstable or the specimen is unpolished. With normalization, you need not worry about beam current fluctuations but you must take care not to omit any major elements from the element list because this will not be obvious in an analysis total which is forced to be 100%.

#### **Element List**

You can select a different type of element list depending on how you want your spectra/spectrum to be quantified:

## Current Spectrum



If this option is selected, each spectrum will be quantified using the elements confirmed in the Confirm Elements step for the current spectrum.

#### Fixed List

Select this option if you wish to define a list of elements with which to quantify your spectra. For example you may only wish to quantify your spectra using certain elements if you are constantly quantifying similar spectra. Define your Fixed List using the Periodic Table. To include an element in the list, click on the element symbol on the Periodic Table and press



or double-click on the element symbol.

## Fixed List and Current Spectrum

Select this option when you know what elements there are in your specimen and you also wish to include any other element that may be present. You define your Fixed List using the Periodic Table as described in bullet number 2 above. The Confirmed Elements List is from the Current Spectrum. This list includes all the elements identified by AutoID and any other elements that may have been added to the Confirmed Element list manually. What elements are quantified when you select 'Fixed List and Current Spectrum' are shown in the examples below in a table:

	Fixed List	<b>Current Spectrum</b>	Combined List
Spectrum 1	Si, O	Si, O, Al, Ca	Si, O, Al, Ca
Spectrum 2	Si, O	B, N	Si, O, B, N

#### **Quant Element List Details**

From this tab you can view the details of each element in the list. The default setting is that the X-ray lines to be used for Quant are automatically selected. You can manually select the X-ray line for each element if you un-check the 'Automatic selection of line for all elements' option. You can check the 'Fixed weight %' option and enter the value.

#### **Deconvolution Elements**

Deconvolution elements may be used to select elements present in the spectrum that should not be quantified, but whose influence needs to be accounted for when processing the spectral data. For example elements present in an oxidation layer, or in a substrate layer.

If you wish to deconvolve elements from a spectrum, select the required element from the drop down list and press 'Add element'. Further elements can be added or removed using Add element or Remove element respectively.

Selecting an element for deconvolution means the peaks will automatically be deconvolved from the spectrum but the element will normally not be quantified. The deconvolution element will only be quantified if its' composition is entered as fixed wt% or is calculated by stoichiometry or difference.

## **Threshold Quantitative Results**

Quantitative results are displayed with +/- value which is one sigma (standard deviation) based on counting statistics. Typically, results which are less than 3 sigma have reduced significance and so it may be desirable to set them to zero. Thresholding may be applied so that results below the selected sigmas are set to zero. Thresholding will also ensure that negative insignificant values, which sometimes result from trace element analysis, are set to zero.

To enable 'Threshold Quantitative Results', check this option in the Quant Settings.

The default value for Sigma is 3 which represents 99.7% confidence level.

## **Quant Standardizations**

The system is supplied with factory standardizations. To use your own standards for quantitative analysis, you first need to acquire spectra from standards and perform at least one standardization using the Standardize step on the Optimize navigator. When you have done this your user file will be available to select for use by the "User" radio button under Quant standardizations in EDS Quant Settings. If the "Factory" button is selected, factory standardizations will be used for the calculation of quantitative results.

In the default "Quant Standardizations" file some low energy lines have been deliberately omitted because there are many potential sources of error for quantification (For example close overlaps, chemical shifts, anomalous excitation and absorption effects on individual lines, inaccurate absorption coefficients and presence of carbon and oxide layers that give large peaks at low kV) If you understand the issues you can still use your own standards to perform analysis for these lines. However, they have been omitted from the default file.

At low kV, fewer x-ray lines are excited and if the recommended line is not available, quantification is not possible. In this case the spectrum overlays cannot be calculated and will not appear in the Confirm Elements step. If you want to work at low kV the factory standardizations file "Quant Standardizations(Extended Set)" has additional low energy L and M lines that will allow you to obtain a concentration result and enable the spectrum overlays. You may find it convenient to use the same file at both high and low kV but it is important to realise that concentration estimates obtained using these extra low energy lines may sometimes be inaccurate.

## **Apply and Save**

If you make a change to the Quant Settings and press , the settings are saved and the currently selected spectra are quantified. The quant results are updated immediately.

#### Save

If you make a change to the Quant Settings and press, the new settings are saved to be used when you do quantification next time.

## **Element Lists**

Any list of elements in the software can be split into the following three categories:

- Pre-defined Elements elements expected in specimen
- Identified Elements typically based on automatic peak identification (Auto ID)
- Fixed List used for Quantitative analysis

## **Pre-defined Elements**

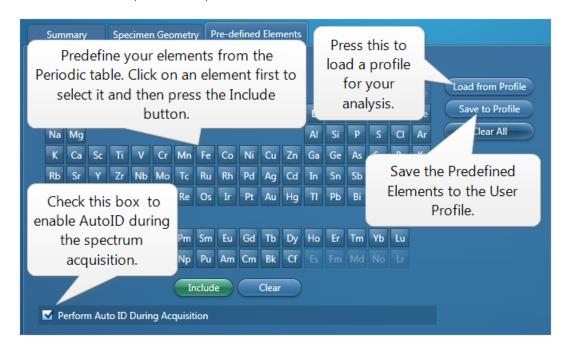
You may have prior knowledge of your Specimen and know what elements to look for.

## Examples

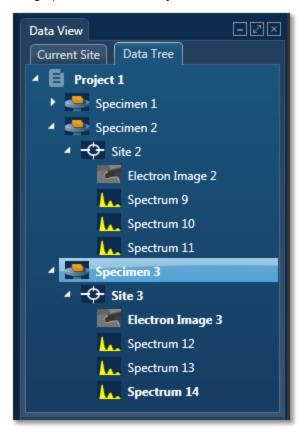
'I want to look for a particular list of elements. (I am not interested in any other elements)...I may want to see their labels on spectra, their X-ray maps or both....I want to see these even if the element is not present'.

' I know what's in my sample.... I want to look for a specific set of elements (I want to see these even if the element is not present.) .....but I would like to know if there is anything else in my sample too'.

You can define these elements in the 'Pre-defined Elements' tab in the Describe Specimen step. If you want to save the Pre-defined Elements to a profile you must first press 'Save to Profile' button, then save the profile via the drop down menu. When you want to analyze your Specimen, you can load this profile or another profile by pressing the 'Load Profile' button in the Describe Specimen step as shown in the screen shot below:



Note that the 'Pre-defined Elements' are saved with the current Specimen. Changing the 'Predefined Elements' will only update the Pre-defined Elements in the current Specimen. It will not update any existing Specimens in the Project.



The current specimen is the one that you are presently analyzing/processing the data from. For example, in the screen shot above, Specimen 3 is the current specimen, Specimens 1 and 2 are the other specimens in the Project.

## **Identified Elements**

The 'Identified Elements will include:

- Any Pre-defined Elements
- Elements identified by Auto ID
- Any additional Elements identified manually

If the 'Pre-defined Elements have been specified, these will be included for identifying and labeling peaks in the current spectrum automatically.

Note that the 'Identified Elements' are saved in the Spectrum.

'Perform Auto ID During Acquisition' option is enabled by default and can be deactivate by un-checking it in the Describe Specimen step as shown in the screen shot above. You can then AutoID at any time by pressing the button.



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Additional peaks in the spectrum can be identified manually by using the 'Show Candidate Elements' tool in the Confirm Element step. Click on the question mark icon to select the Show Candidate Element tool. Position the cursor at the center of a peak by double-clicking with the mouse. The list of elements spectra corresponding to the energy at the cursor is displayed in the panel on the right. By highlighting an element in this list, you will see the markers showing all the lines for this element.

Note that the 'Identified Elements' will be quantified if you have selected the Current Spectrum, or the Fixed List and Current Spectrum Element List in the Quant Settings in the Calculate Composition step or EDS Quant Settings in the User Profile dialog.

#### Note

EDS Quant Settings are available in the User Profile Dialog accessed from the Tools menu. These settings are also available from the Calculate Composition step.

#### **Fixed List**

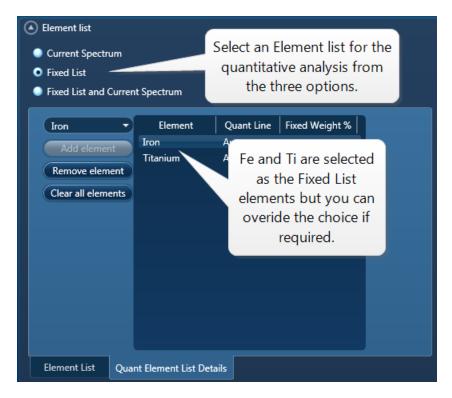
The elements in the 'Fixed List' are defined in the Quant Settings dialog which is available in the User Profile and the Calculate Composition window.

Note that the Fixed List is only used for calculating composition in quantitative analysis.

#### Example

'I want to do quantitative analysis on my glass samples and want to compare results from one batch to another batch. I am always looking for the same specified list of elements'.

You can specify the Element List for Quant from the three available options in the Quant Settings dialog as shown in the screen shot below:



#### **Current Spectrum**

This list includes the Pre-defined Elements and elements identified by Auto ID and manually using the Candidate Element tool.

#### **Fixed List**

You select the Fixed List option if you know what elements you wish to quantify. Choose the elements from the drop-down list as shown in the screen shot above.

#### **Fixed List and Current Spectrum**

To quantify the elements in the above two lists, select the Fixed List and Current Spectrum option.

MiniQuant results table will clearly display which list is being used. A lock icon will be displayed against the 'Fixed List' elements as shown in the screen shot below:



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In this example, Fe and Ti are selected in the Fixed List. The rest of the elements in the chart results are from the Current Spectrum because the Element List selected for quantification was 'Fixed List and Current Spectrum'.

Note that the 'Fixed List' is saved in a User Profile.

#### See also:

Describe Specimen on page 76

Acquire Spectra on page 350

Confirm Elements on page 353

Calculate Composition on page 185

User Profile on page 22

## **Compare Spectra & MiniQuant Results**

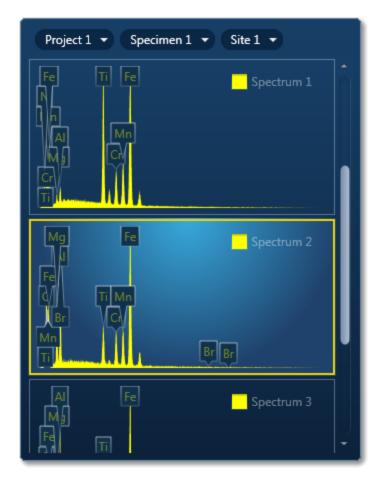
Real time Compare and instant MiniQuant options are available in the Acquire Spectra, Confirm Elements and Calculate Composition (Comparison of Results - Two Spectra template) steps. User can see results without having to move away from the acquisition mode. Using these options you can:

- See your results during analysis.
- Compare your current spectrum to a control spectrum during acquisition.
- View MiniQuant results in a table or a bar chart.

Click in the top right corner of the Spectrum Viewer in Acquire Spectra, Confirm Elements or the Calculate Composition window to access the Compare & MiniQuant options:



In the above example, Spectrum 1 is the current spectrum and Spectrum 2 is the comparison spectrum. You can select the comparison spectrum from a Project currently available in the Data Tree. It can be from any Project, any Specimen and any Site of Interest currently available in the Data tree. To choose the comparison spectrum click on the down arrow (Spectrum 2 in the above example). Spectra available in the current Project, Specimen and Site of Interest are displayed as below:



Click on the spectrum in the display to select it for comparison. The selected spectrum will be overlaid as a line spectrum over the current spectrum. The MiniQuant results are displayed in a table as shown in the example below:

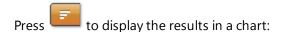


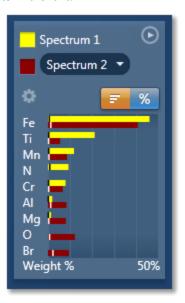
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The results are displayed as wt% (weight%).

The statistical error is displayed as  $\sigma$  (weight% sigma) for the calculated wt%. It is the overall confidence figure for the analysis. You can use sigma to assess the results especially when an element is present at low concentration. For example, if an element concentration is 0.2 wt% and the  $\sigma$  is 0.12 wt%, the element might be detected at a statistically significant level if the acquisition time for the spectrum is extended. If the  $\sigma$  is 0.4 wt%, it is pointless to extend the acquisition time and it is safe to assume that the element if present, is at a level above the limit of detection for this technique.

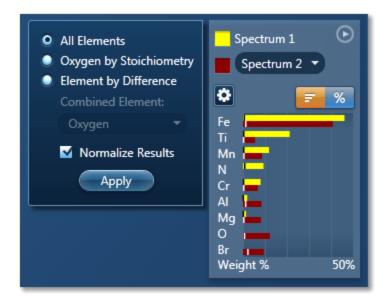




The sigma values are displayed as black or white vertical bands across the bars in the chart results as shown in the example above. In this case the full scale of the bar chart is 50%.

If you wish to change the MiniQuant Settings press





Make your selection by clicking on the radio button and then press the Apply button. The results will be updated immediately .

#### Note

The Quant Settings in the MiniQuant and Calculate Composition are the same. Updating one updates the other and vice versa.



## Point & ID - Custom

Point & ID is an image centric application that requires the acquisition of an electron image prior to X-ray spectra acquisition. There are two modes of operation, Guided and Custom.

In the Custom mode, the Point & ID navigator has three steps.

Describe Specimen and Compare Spectra are explained in the earlier section. The step which is unique to Point & ID - Custom is explained here.

Acquire and Confirm.	201
Recommended way of working in Point & ID - Custom Mode	202



## **Acquire and Confirm**



In Acquire and Confirm step, four operations are combined into one window. Acquire and Confirm is the main step of the Point & ID Navigator in the Custom mode. It is aimed for users who do not require any guidance during their analyses. The workspace is divided into four quadrants. Each quadrant represents an application. For example, Scan Image is located in the top left quadrant, Acquire Spectra in the top right quadrant, Quant Results in the bottom left quadrant and Confirm Elements in the bottom right quadrant.

Press the relevant button in the toolbar, from the view in any quadrant.

Press to un-dock a quadrant view into a free floating window located in the top right corner of the view. Press to switch it into a full screen view.

To re-dock the free floating window back into the main application window press  $\square$ 

Each application has identical functionality as its counterpart in the Guided Navigator. To get help on each application follow the links below:

Scan Image on page 418

Acquire Spectra on page 350

Confirm Elements on page 353

Calculate Composition on page 185

# Recommended way of working in Point & ID - Custom Mode

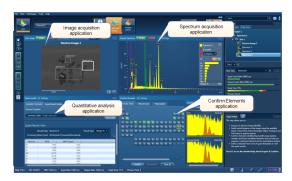
In the Custom mode, four smart components are available in one window. You can acquire, review, confirm and process data in one window. You do not need to move away from it during the analysis.

The four smart components are docked in the workspace as the default layout. Each component can be un-docked and have it as a free floating window. It can be resized and dragged to a second monitor to be viewed in full screen. There is a great deal of flexibility in the user interface to customize the layout to suit your requirements.

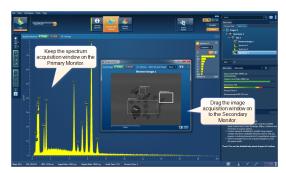
#### EXAMPLE

One way of working in the Custom mode is described with screen shots here.

The four components in the Acquire and Confirm window are docked in the four quadrants of the work space as shown in the screen shot below:



- 1. Press in the top left quadrant to undock the Acquire Image window. The upper two quadrants are now filled with the Acquire Spectrum window.
- 2. Click with the left mouse button in the title bar of the floating Acquire Image window and drag it to a second monitor.





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3. Toggle off the % button in the toolbar near the top right of the main application to remove the Quant Results window from the display. The Confirm Elements window will slide in under the Acquire Spectrum window as shown in the screen shot below:



4. The Acquire Spectrum window can be removed from the display if you need to maximize the Confirm Elements display as shown below:



- 5. Once you have confirmed the elements, you may not require the Confirm Elements window. You can remove it from the display if you wish.
- 6. To be able to view the Quant Results, switch the Quant Results display on from the toolbar:



## Map - Guided

In the Guided mode, the Map navigator has the four steps.

The Describe Specimen and Scan Image steps are covered in the earlier section. The steps which are unique to this navigator are described here.

Acquire Map Data	205
Construct Maps.	219
Analyze Phases	226



## **Acquire Map Data**



In this step, you can acquire X-ray maps from the full frame or selected regions of the specimen. The maps show the spatial distribution of all elements in the specimen. The results can be displayed as a Layered Image, where colors for each element are mixed together and overlaid on the electron image, or as individual maps. Spectra from selected regions can be reconstructed during or after data acquisition. Generating a Layered Image or X-ray maps can be a very useful way to find out what is going on in your specimen.

#### EXAMPLE

'I want to know where certain key elements are distributed over a defect. Once I have this information, I can determine what caused the defect and advise my production department.'

#### **How to Acquire and Manipulate Maps**

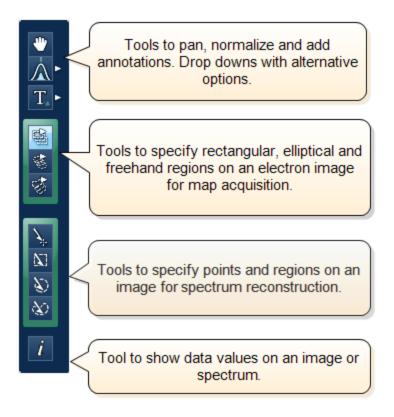
There are two different types of maps that you can acquire, Window Integral Maps or Tru-Maps.

Historically, Window Integral Maps have been the standard mode for X-ray maps. These are ideal when there are no overlapping peaks and you are not looking for trace elements in your specimen.

The second mode of mapping is with TruMaps which are ideal for specimen containing elements with overlapping peaks, and removes false variations due to X-ray background.

You can easily switch between the two modes of mapping during acquisition by pressing the Map or TruMap button above the map display.

- Select the acquisition parameters from the Settings cog on the acquisition toolbar,
   and press
   to acquire map data from the full frame.
- To acquire maps from a region, select a map acquisition tool from Rectangle, Ellipse and Freehand tools available from the toolbar:



- Click on the image and drag with the left mouse to outline a region on the image.
   Maps will be acquired from the scanned region. During TruMap acquisition, a progressing green line is the map acquisition line followed by a yellow map processing line.
- A layered image, element maps and an electron image/s (SE/BSE) are displayed. You can choose how you wish to view your data from Standard, Interactive or Summary view available from the drop-down list.
- Adjust the slider bar to choose the number of maps per row.
- The layered image allows you to see the X-ray maps overlaid on the electron image.
- You can add or remove an X-ray map from the layered Image (combined Electron and X-ray map image) by toggling the Layered Image icon in the top left hand corner of each map.
- If you have lots of maps, it may be useful to minimize some of them pressing the minimize icon in the top right hand corner of each map.
- You may want to delete a map from the analysis completely. In which case press the delete icon in the top right hand corner of each map. This means this element will not be identified automatically (AutoID) and will be excluded from the current analysis. Note: If an element is present in a specimen, deleting or excluding it will affect the TruMap results.



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- In the map display settings you can sort maps alphabetically, by atomic number or by maximum map intensity. You can also smooth maps by choosing the smoothing level from the Settings.
- Using the Auto Brightness and Gamma buttons on the bottom right hand corner of the Map display window allows you to change the Brightness/Contrast and Gamma for all the maps. The Auto Brightness button optimizes the maps to give the best Layered Image and the Auto Gamma enables you to see all the map data including background noise.
- You can choose the color for your maps, adjust intensities and decide which maps to add to the Layered image. Alternatively, you can let the software do this auto
  - matically. Pressing the AutoLayer button (which is located in the bottom right hand corner of the Map Display window) will automatically scale and color all the maps and select the best ones to provide an effective color image that delineates regions of different composition. Maps will be auto-brightness corrected and those that show similar structure will be assigned the same color. Maps that are very noisy will be shown in grey. The most significant map for each assigned color will be added to the Layered Image. See AutoLayer on page 222 for detailed information.
- If your data contains a lot of noise, binning might help you to achieve a better Auto-Layer result. Select the binning factor from the drop-down list below the maps.

#### **Data from Map Acquisition**

The Data Tree gets populated with the new items as data is being acquired as shown in the screen shot below:



#### **Electron image**

It can be a secondary electron image (SE), backscatter electron image (BSE), or a forward-scattered electron image. Appropriate detector hardware needs to be installed. You can also import an image into the Project.

#### **Map Data**

The EDS and EBSD Map data are contained in the Map Data folder. The EDS Data folder contains Map Sum Spectrum and X-ray Element Maps.

#### X-ray Element Maps

Two modes of mapping are available, Window Integral Maps and TruMaps. To select the mapping mode, press the Map or TruMap button above the map display.

Standard Window Integral maps (counts in the energy window) are acquired for the element list chosen for analysis. These are raw X-ray maps which are not corrected for background or peak overlaps.

Second mode of mapping is TruMap. You can process the map data as Tru-Maps which are corrected for background and peak overlaps.

#### **EDS Layered Image**

It is a composite image generated by overlaying selected X-ray maps on top of the electron image.

#### **Viewing and Manipulating Maps**

You can choose how you wish to view your data. Various tools are available to manipulate and view the X-ray maps.

#### **Map Size**

You can choose the number of maps per row using the slider bar for displaying maps you wish to view in the Standard and Interactive display mode.



#### **Display Modes**



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Maps can be viewed in three different display modes available from the drop-down list on the Display toolbar:

- Standard
- Interactive

In the Interactive mode, you can change the color of individual maps from the Hue dropdown list. The Layer Mode can also be chosen from Mix and Overlay modes available from the electron image.

Summary

In the Summary view, you see details of the energy window and X-ray line used for each map in addition to other details such as Layer Name, Source (AutoID or User), Map Color and if it is selected for the Layered Image. See the screen shot below:

Image	Layer Name	Source	Map Color		Energy Window (keV)	Line	Visible lay	er
	Fe Kα1	AutoID	300	D	6.32 - 6.49	Κα1	8	<u></u>
	Ti Kα1	AutoID	<b>0</b>	Ð	4.44 - 4.58	Κα1	\$	<b>T</b>
	Cr Kα1	AutoID	<b>120</b>	Đ	5.34 - 5.49	Κα1	\$	<b>T</b>
	Mn Kα1	AutoID	<b>180</b>	Đ	5.82 - 5.98	Κα1	\$	<b>T</b>
	Br Lα1_2	AutoID	<b>3</b> 0	Đ	1.43 - 1.53	Lα1_2	\$	<b>T</b>
	Al Kα1	AutoID	<b>210</b>	Đ	1.44 - 1.54	Κα1		<b>T</b>
	Mg Kα1_2	AutoID	<b>60</b>	Đ	1.21 - 1.30	Κα1_2	\$	<b>T</b>
	Ν Κα1_2	AutoID	90	Đ	0.35 - 0.43	Κα1_2		<b>T</b>
	Ο Κα1	AutoID	<b>150</b>	Đ	0.48 - 0.57	Κα1		<b>T</b>
\$17	Electron Image 1		••	Đ			\$	<u>¥</u>

#### Link/Unlink



to link images for manipulation of all layers using the Pan or Zoom control.

Press to unlink images. You can manipulate individual layers using Pan or Zoom control.

#### **Brightness and Contrast**

You can adjust the brightness and contrast of the currently selected image or map. Press



on the Display toolbar to open the Brightness and Contrast dialog.

#### **Auto Brightness and Auto Gamma**

Using the Auto brightness and Gamma buttons on the bottom right hand corner of the Map display window allows you to change the Brightness/Contrast and Gamma for all the maps. The Auto Brightness button optimizes the maps to give the best Layered Image and the Auto Gamma enables you to see all the map data including background noise.

#### **Element Maps View - Settings**

You can manipulate and view the data by using various parameters available in the Settings:



### **Sort Order**

There are three different ways of sorting maps:

Alphabetically

By atomic number

By maximum intensity in map - sorts on the value of the brightest pixel in cps.

## **Layer Visibility Selection**

You can choose how the visibility of layers selected in the layered image. There are two options: Manual and Automatic. In the Manual mode, you must select which X-ray maps to be included in the layered image.

In the Automatic mode, first N maps (Number of Map that you entered) are selected by the maximum intensity.

## Smoothing Level

The maps may contain a lot of statistical noise if there is not sufficient data. The noise can mask the distribution of elements in the maps. You can filter out some of this noise by



applying Smoothing Level. This operation applies a lowpass filter to an image to smooth the data. If you are using TruMap, it might be more effective to use binning.

## Smoothing Level, 3X3

The lowpass filter uses the following 3x3 kernel:

1/9 1/9 1/9

1/9 1/9 1/9

1/9 1/9 1/9

## Smoothing Level, 5x5

The lowpass filter uses the following 5x5 kernel:

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

## **ACB** while acquiring

Check this option if you wish to apply automatic brightness or automatic gamma to maps during acquisition depending on your pre-selection of Auto Brightness or Auto Gamma.

#### See also:

Acquire Map Data - Settings below

Context Menu - Map Viewer on page 223

How binning affects the quality of your data on page 373

Layer modes in the interactive map display on page 216

## **Acquire Map Data - Settings**

The settings cog is located near the top of the Acquire Map Data screen. Clicking on it with the mouse displays the available settings:



#### Resolution

You can set the resolution of your maps by choosing the number of pixels in the X dimension over which the beam scans. The number of pixels in the Y dimension will depend on the aspect ratio of your microscope image.

If you are collecting X-ray data from the entire image using the number of pixels used will be the number set in the map resolution option.

If you define an area using the rectangle, Ellipse or Freehand tool X-ray data will be collected from only this defined area with a proportional number of pixels.

#### **Acquisition Time**

There are two options available for maps acquisition time:

#### **Until Stopped**

If you choose this option the system will carry on acquiring data until you stop it.

#### **Fixed Duration**

You can choose number of frames you wish to acquire by entering the number in the Frame Count dialog.

#### **Number of Channels**

Select the number of channels from the drop down list of 1024 and 2048 with which you display the spectrum. The number of eV/channel will depend on both the energy range and the number of channels you select.



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Energy Range (keV)	Number of Channels	eV/channel
0-10	2048	5
0-10	1024	10
0-20	2048	10
0-20	1024	20
0-40	2048	20
0-40	1024	40

In the Auto mode, the system checks for the energy range selected and sets the appropriate number of channels.

#### **Energy Range (keV)**

Select a spectrum energy range from the available options of Auto, 0-10, 0-20 or 0-40 keV from the Energy Range drop down list.

The appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited.

In the Auto mode, the system checks for the accelerating voltage set on the microscope and selects a suitable energy range in the software.

#### **Process Time**

Select the Process Time from the drop-down list of Process Times, Default and 1 to 6. The Process time is the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process time, the lower the noise. If noise is minimized, the resolution of the peak displayed in the spectrum is improved, in other words, the peak is narrower and it becomes easier to separate or resolve, from another peak that may be close by in energy.

If Default is selected, the Process Time is automatically set to a suitable value.

There is a trade off between the Process time that is used, and the speed at which data can be acquired into the X-ray spectrum. Process time 1 is the shortest, and as such, gives the highest X-ray acquisition rates, but at some cost to resolution. Process time 6 is the longest, and gives the highest resolution, but at some cost to maximum acquisition rate. The longer the Process time, the slower data can be acquired, i.e. the higher the system Deadtime will be for a given input count rate. (The input rate is not affected by the pulse processor).

#### Pixel Dwell Time (µs)

The default value for dwell time per pixel is 100 µs.

#### Frame Live Time (s)

The value of frame live time depends on the map resolution and pixel dwell time.

## **Brightness, Contrast and Gamma Controls**

You can adjust the brightness and contrast of the currently selected image or map. Press



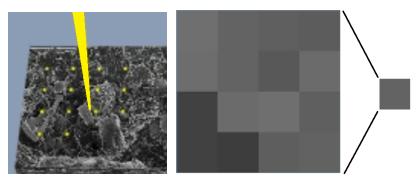
on the Display toolbar to open the Brightness and Contrast dialog.

#### **Auto Brightness and Auto Gamma**

Using the Auto brightness and Gamma buttons on the bottom right hand corner of the Map display window allows you to change the Brightness/Contrast and Gamma for all the maps. The Auto Brightness button optimizes the maps to give the best Layered Image and the Auto Gamma enables you to see all the map data including background noise.

# How binning affects the quality of your data

As the electron beam scans a line or area of the specimen, data is acquired from numerous points. Inevitably, the signal from each point includes some noise. If data is not acquired for long enough, the noise level will be high. By combining signals from several neighboring points, the binning technique produces an averaged signal, which has less noise overall. The effect is similar to collecting the original data at a lower resolution and a longer dwell time.



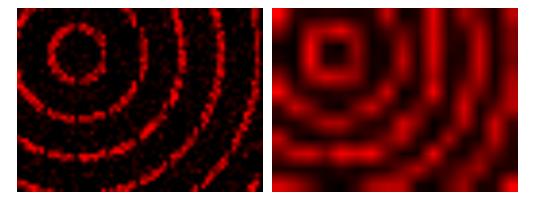
You can select the binning factor from the drop-down list below the map or linescan display.

#### Effect of binning on element maps

A binning factor of four combines 16 adjacent pixels from each 4x4 square into one new pixel. For example:



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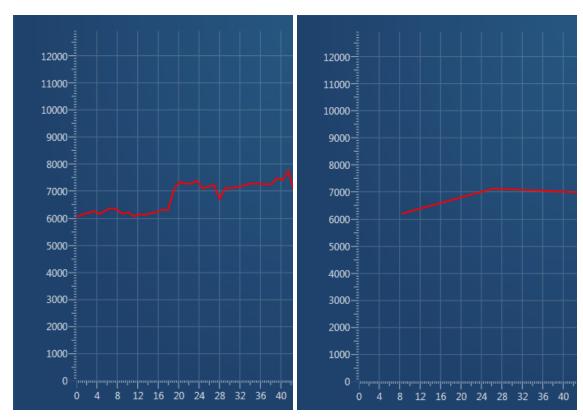
Binning is useful when you are using the TruMap mode for mapping because of the improved statistics. Binning also enables AutoLayer to combine similar maps more successfully because of the lower noise.

#### See also:

AutoLayer on page 222

#### Effect of binning on linescans

Binning combines a group of pixels into one new pixel. The binning appears to shorten the linescan at each end. In the following example with a binning factor of 16, the first value appears at approximately  $8~\mu m$  because the first binned point is midway between the first 16 values.



Original data at 1 µm spacing

Binned data

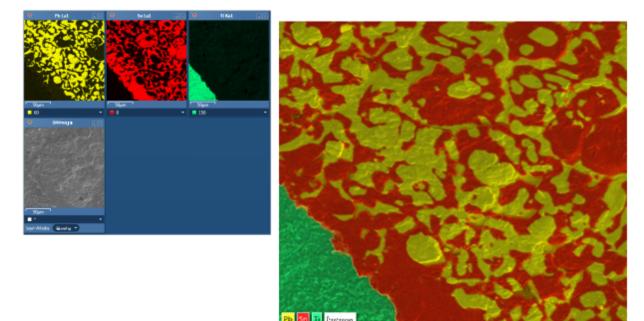
## Layer modes in the interactive map display

Layer modes on electron images can be useful for showing topographical and compositional detail in a layered image. Both layer modes add the color components for each map together at each pixel.

- In Overlay mode, the brightness of each pixel is determined by the electron image. The color is determined by the composite (red, green and blue) of all the other images. This mode shows more clearly the relative changes in intensity between the maps.
- In Mix mode, the color is determined by the composite (red, green and blue) of all the images (including the electron image). This mode tends to make variations in the absolute intensities of the maps more visible.

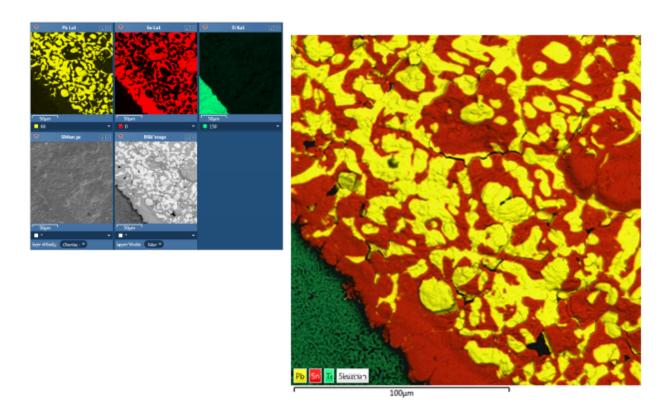
At the Acquire Map Data step, select Interactive from the drop-down list above the maps and images. You can then choose a layer mode from a drop-down list on the electron image.

By default, the layered image for the electron image is in Overlay mode. This is ideal for viewing the overall chemical distribution of elements on a specimen image. See the next example, which shows the selected maps and the layered image.



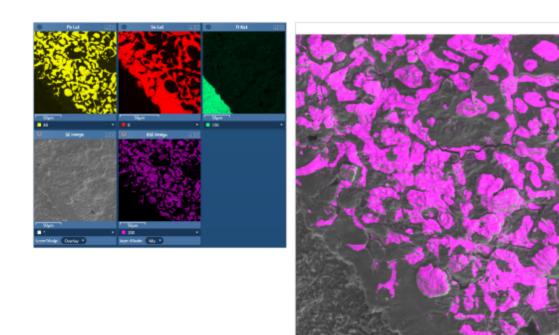
The next example uses the same area and adds the backscatter electron (BSE) image in Mix mode. The addition of the compositional information in the BSE image to the topographical information in the SE image gives better definition to the overall layered image.

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To overlay the density information in a BSE image on the secondary electron (SE) image:

- 1. Remove all other maps from the layered image.
- 2. Ensure that the map display is set to Interactive.
- 3. Below the BSE image, use the drop-down list to change the color of the BSE image.
- 4. Below the BSE image, select the Mix layer mode.
- 5. Adjust the brightness and contrast on the BSE image to emphasize the layered image as required.





## **Construct Maps**



In this step, you can select which elements to map and which ones to exclude.

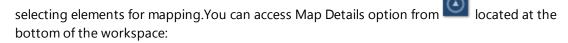
You can change the default X-ray line used for Window Integral Maps for any given element. It is also possible to define energy windows whose widths you can specify yourself rather than using the auto width calculation.

Tools are provided to interrogate the map data to confirm the elemental composition of user specified areas of interest. You can navigate to the Confirm Elements step from within the Construct Maps step.

To reduce the effects of noise in the maps, you can apply a binning factor.

#### **Map Details**

Map Details dialog allows you to choose elements you wish to include or exclude for mapping. You may have pre-defined the known elements in your specimen in the Describe Specimen step. You can map these elements by pressing the Pre-defined button in the Map Details dialog. There may be unexpected peaks in the spectrum. You can use AutoID for





To include an element for mapping either double-click on its symbol on the periodic table or click the Include button. All the elements selected for mapping are green color coded on the periodic table. To exclude an element from being identified by AutoID and excluded from being mapped, click on its symbol on the periodic table to select it and then press the Exclude button. This element will be removed from the analysis. All excluded elements will be red color coded on the periodic table. Note: An excluded element if present in the specimen may affect the TruMap results.

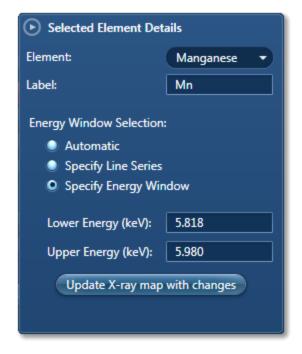
To remove an element map from the display, select it by clicking on it and then press the Clear button. It will be removed from the display. Press the Clear All button to remove all maps from the display. Remember the maps will be displayed again when you press AutoID or include elements manually.

#### Manual selection of energy windows and X-ray lines:

Press on the Map Details dialog to open the Selected Elements Details:



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The default setting is Automatic X-ray lines and energy window width selection.

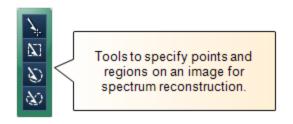
- To manually define the width of the energy window, select the Specify Energy Window option. Enter the values for Lower Energy (keV) and Upper Energy (keV) and press
  Update X-ray map with changes
  . The map will be acquired using the defined window width.
- To manually select the X-ray line for mapping an element, select the Specify Line Series option. Select the line from the Line Series drop-down list and press
   Update X-ray map with changes

   The map will be acquired using the specified X-ray line.

## Reconstruct spectra from Layered Image or Maps using Point, Rectangle, Ellipse and Freehand tools

The spectrum reconstruction tools are available in the toolbar on the left of the workspace:

• Select the spectrum reconstruction tool from the four available options:



- Click on any image and drag with the left mouse to select a region. A reconstructed spectrum is displayed in the spectrum viewer and it is also added to the data tree.
- MiniQuant results of the reconstructed spectrum are displayed. You can compare the sum spectrum and the reconstructed spectra.
- To confirm the elemental composition of a phase you can navigate to the Confirm Elements step in the Point & ID package from the link below the Layered Image Viewer. For details refer to Online Help.

#### See Also:

AutoLayer below

Context Menu - Map Viewer on the facing page

How binning affects the quality of your data on page 373

## **AutoLayer**

By identifying and combining the elements that vary in a sample, AutoLayer helps you visualize both phase and element distribution using a single image.

You can choose a color hue for each of the X-ray maps and adjust brightness and contrast on each map individually, or use the "AutoBrightness" or "AutoGamma" buttons to apply an automatic setting to all maps. Colored maps can be mixed together or "layered" on top of an electron image to generate the "Layered Image" view and there is a control on each map window to select whether to include the map in the Layered Image combination. The idea of mixing is to make regions of different material content appear in different colors. An overlay can be used to relate this material content to the topographic detail that is usually visible in the secondary electron image. If the electron image is a backscattered electron (BSE) image, the intensity may be controlled more by material content than by topography. You may therefore prefer to assign a color to a BSE image and an option is provided on the electron image to either assign a color and "mix" it (as if it was an X-ray map) or use a grey scale and "overlay" the mixture of colored X-ray maps. If you choose different colors for similar maps and mix them together, the resulting mixture color will not bear any relationship to the original colors so it will be difficult to make sense of the layered image. If you are analyzing specimens where you know what elements are likely to appear, then manual color mixing is straightforward. However, for more complex situations or when you are dealing with an unknown specimen you should find the AutoLayer function helpful.

If you have a series of X-ray maps displayed, the "AutoLayer" button will run an algorithm to analyze the spatial content in the maps to decide and select the best maps to use for the layered image. It will also adjust contrast and brightness and assign a suitable color hue to the maps to give a useful layered image. If maps have a lot of statistical noise, they are unlikely to be selected, otherwise the layered image would show a lot of random colored dots. When a set of maps show similar spatial content, they will be assigned the same hue, but only the best map will be chosen for the mix. Sometimes a noisy map will be assigned a color but it will not influence the mix. If a map has not been assigned a color, it either has too much noise, or enough different maps have already been found to provide a good mix. In the case where



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there are many different materials in the field of view, it is worth checking those maps that have not been allocated a color to see if they show any features that are not obvious in the layered image.

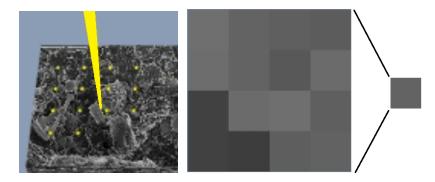
## **Context Menu - Map Viewer**

A number of useful shortcut menus available as right mouse click in the map viewer are shown in the table below:

Context Menu Item	
Reset Image Scale	
View	Color Bar
	Scale Bar
Export	Save As (Original Resolution)
	Save As
	Сору
	Print
	Email
	Settings
Details	

# How binning affects the quality of your data

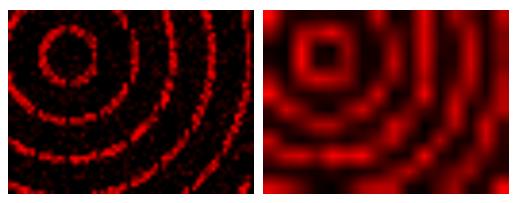
As the electron beam scans a line or area of the specimen, data is acquired from numerous points. Inevitably, the signal from each point includes some noise. If data is not acquired for long enough, the noise level will be high. By combining signals from several neighboring points, the binning technique produces an averaged signal, which has less noise overall. The effect is similar to collecting the original data at a lower resolution and a longer dwell time.



You can select the binning factor from the drop-down list below the map or linescan display.

#### **Effect of binning on element maps**

A binning factor of four combines 16 adjacent pixels from each 4x4 square into one new pixel. For example:



Binning is useful when you are using the TruMap mode for mapping because of the improved statistics. Binning also enables AutoLayer to combine similar maps more successfully because of the lower noise.

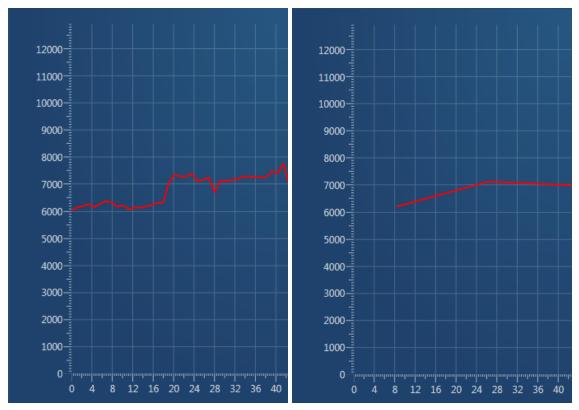
#### See also:

AutoLayer on page 222

#### **Effect of binning on linescans**

Binning combines a group of pixels into one new pixel. The binning appears to shorten the linescan at each end. In the following example with a binning factor of 16, the first value appears at approximately 8  $\mu$ m because the first binned point is midway between the first 16 values.

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Original data at 1  $\mu m$  spacing

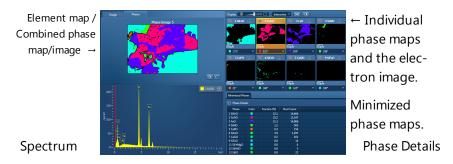
Binned data

## **Analyze Phases**



In this step, the software automatically converts X-ray maps into phase maps. The phase maps help you to see the constituent elements of the phase, and how the phases are distributed over the specimen.

The window has several sections:



- The individual phase maps (top right) are presented according to the distribution and size of each phase. The first maps show large areas of closely grouped elements. Later maps show smaller areas that are more finely distributed. You can change the colors in the phase maps to better represent interesting groups of elements (or "phases").
- On the Image tab (top left), you can view any image in your project, such as the EDS layered image. The Phases tab shows the combination of images that you select from the many phase maps and the electron image.
- The spectrum (bottom left) shows a spectrum extracted from the pixels in the currently selected phase. You can also closely examine the spectrum at any point or area of interest using tools on the left toolbar.
- Phase Details (bottom right) shows the area of each phase in pixels and as a percentage of the total area of the map. You can copy these results into a spreadsheet.

Toolbars around the window enable you to change the processing of the data, and display and analyze the data.

#### See Also:

About phase maps on page 377

Analyze Phases toolbars on page 383



## Finding phases

In the Analyze Phases step, the software automatically converts X-ray maps into phase maps. The phase maps help you to see the constituent elements of the phase, and how the phases are distributed over the specimen. You can run this step while map data is acquired, or afterwards.

If map data is being acquired, the phase analysis repeats periodically until acquisition finishes or you click the "Cancel Processing" button. The phase analysis produces the best results if run after all the map data has been acquired and elements have been identified.

1. In the acquisition toolbar, click the green "Find Phases" button to start processing. When processing is finished, phase map data appears on the Data Tree, for example:



- 2. To re-run the processing with different settings, select the Settings cog icon from the top toolbar. Any changed settings are retained in your User Profile, and become your default settings when you next run this analysis.
- 3. After the processing, you can manipulate the phases as required:
- Examine the spectrum at any point or area on the phase, using the tools in the left toolbar, to confirm the composition.
- Select individual phase maps to include in the combined phase map. You can also include the electron image.
- Merge phases that you want to analyze as a single phase.
- Remove phases that are not relevant to your analysis.
- Rename the phase maps in the Data Tree to identify possibly significant phases. For example, rename CaSiO to Calcium Metasilicate.
- Change the color of phases for convenient identification.

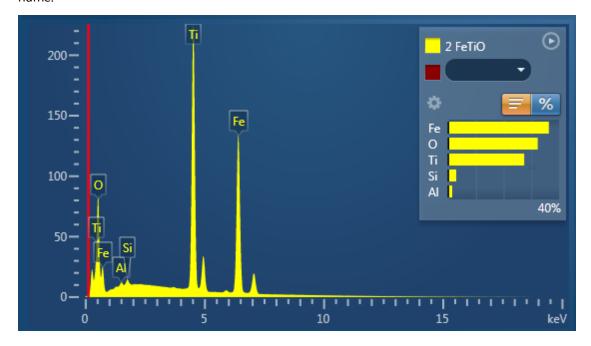
#### See Also:

Analyze Phases toolbars on page 383

## **About phase maps**

The name of each phase is derived from its main elements. For example, a phase with the following spectrum might be named "2 FeTiO". Although the spectrum contains Si and Al, the

quantities are much smaller, and therefore those elements are not included in the phase name.



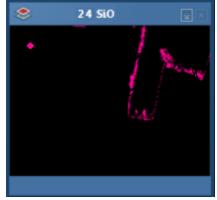
The names of the cations and anions are arranged in an order that closely resembles the name of a chemical compound. The name of the phase is a *possible indication* of a chemical compound.

### The order of the phases

The order of the phases depends on the distribution and quantity of each group of elements. The first phases indicate large areas of closely grouped elements. Later phases show smaller areas that are more thinly distributed, for example:

- 2 SiO shows one large area or a few large areas that contain Si and O.
- 24 SiO shows smaller and probably more scattered areas that contain Si and O.





### The number of phases

You can display more or fewer phases by changing the analysis settings, or by merging phases manually.

#### See Also:



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Analyze Phases settings on page 381

Merging phases on page 379

### Merging phases

To simplify the results of the phase analysis, you can merge some phases. The result is a new phase with a combined spectrum, and fewer phases overall.

For example, these phases have similar spectra and might be considered to be the same phase:



You can merge similar phases in several ways:

- Set a higher Grouping Level, to automatically combine any number of similar phases.
- Manually merge any two phases.

To manually merge two phases:

- 1. In the phase maps, select a phase.
- 2. Right-click, and in the context menu, select "Merge into ...".
- 3. Select the other phase from the list.

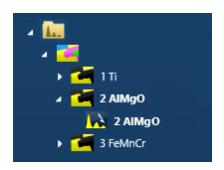
The first phase disappears. The spectrum, phase name and color of the second phase are updated to include information from the first phase.

#### See Also:

Analyze Phases settings on page 381 (Grouping Level)

### Phase maps in the Data Tree

The EDS Data folder contains phase map data in a folder, which by default is labeled as 'Phase Image #', where # is an automatically increasing number. For example:



Icon	Description
	The EDS Data container contains the Phase Image folder, which contains all the phase maps.
	The name of a phase map is composed of a number and its elements, for example: 2 AlMgO. To rename the phase , right-click the icon and select Rename.
	A spectrum extracted from one of the phases. For details, right click the icon.

#### See Also:

Data Tree on page 85

Analyze Phases on page 375

# **Analyze Phases settings**

The software processes data acquired from the maps according to the settings for Boundary Threshold and Grouping Level. To see the settings, click on the Settings button. The default settings are stored in the User Profile.

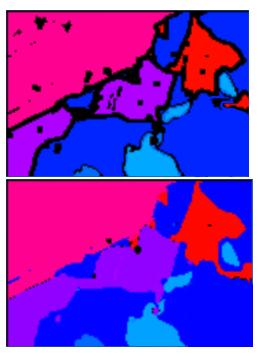


#### **Boundary Tolerance**

Boundary tolerance controls the behavior at the boundaries of each phase.

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- If the boundary tolerance is low, each pixel in the phase represents a pure spectrum. If a pixel has contributions from several phases, it cannot be identified, and appears black.
- If the boundary tolerance is high, all pixels are placed in the phase that fits them most closely. The spectra for each phase shows small contributions from adjacent phases.



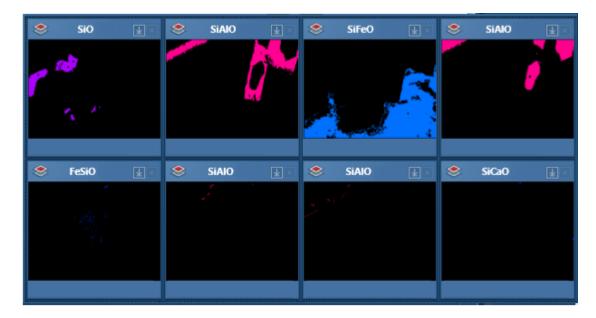
Low setting of boundary tolerance

High setting of boundary tolerance

### **Grouping Level**

Phase analysis identifies all the phases in the sample. Grouping combines phases that are similar, and can create a smaller, more manageable number of phases overall.

- If the grouping level is low, a larger number of phases are displayed. The later maps show phases that occupy small areas, and might indicate trace compounds.
- If the grouping level is high, a smaller number of phases are displayed. The maps typically show a few large areas.



If the Grouping Level is too high, areas that you consider as separate phases will be merged together. To resolve this problem, you can reduce the Grouping Level until all the expected phases are shown, and then manually merge any phases that you prefer to analyze as a single phase.

#### See Also:

Analyze Phases on page 375

User Profile on page 22

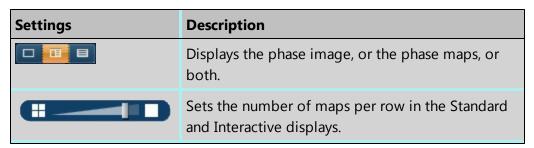
Merging phases on page 379

# **Analyze Phases toolbars**

These toolbars are available in the Analyze Phases step.

### Map display tools

These controls are above and below the phase maps.





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Settings	Description
Standard •	Offers a choice of map display:
	<ul> <li>Standard - you can add individual images or remove them from the combined image.</li> </ul>
	<ul> <li>Interactive - similar to Standard. Links the zoom and pan of all the maps.</li> </ul>
	<ul> <li>Summary - a compact display, where you can change the color of each phase.</li> </ul>
	Links images for manipulation of all layers using the Pan or Zoom controls.
	Changes the brightness and contrast of the currently selected image or map.
	Adds the selected phase map or electron image to the combined phase image.
	Minimizes a map. The Minimize icon is in the top right hand corner of each map. This is useful if you have too many maps in view. The map moves to the Minimized Phases tab, below the displayed maps.
	Removes a map or electron image from the phase analysis. This icon is in the top right hand corner of the image.
<b>T</b>	Restores a minimized map to its normal size. The minimized maps are on the tab below the displayed maps. This icon is in the top right hand corner of each map.
■ 180* ▼	Sets the color of the phase. The list is available only in the Interactive and Summary map display.

### See Also:

Analyze Phases on page 375

### **Processing toolbar**

These controls are at the top of the window, above the phase maps.

Icon	Description
Find Phases	Starts processing the element maps to create each phase map. A Cancel button and a progress bar appear during the processing.
	Shows the progress of the processing. For more details, hover the cursor over the progress bar for a few seconds.
Cancel Processing	Stops the processing.
	Adjusts the settings used during the processing:  Boundary Tolerance controls the behavior at the boundaries of each phase.  The Grouping Level determines the numbers of phases that you see.

### See Also:

Analyze Phases on page 375

Analyze Phases settings on page 381

Finding phases on page 376

### **Toolbar for Phase Map**

These controls are at the top left side of the window.

Icon	Description
	Moves the image. Click the Pan tool, then click and drag the image. Use the mouse wheel to zoom in and out.
	Normalizes the spectra.



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Icon	Description
	Adds annotations to the current image. The tools include Caliper, Angle, Text, Rectangle and Ellipse.
7 2 N N	Defines points and regions on a map image to extract spectra.
i	Shows the energy and counts at any point in the spectrum viewer (in the bottom left corner).
	Shows the phase at any point in the phase maps (in the top right corner).

### See Also:

Analyze Phases on page 375

Map -	Custom
-------	--------

In the Custom mode, the Map navigator has two steps.

The Describe Specimen step is covered in the earlier section. The step which is unique to this navigator is described here.

Acquire and Construct 237



# **Acquire and Construct**



Scan Image, Acquire Map Data and Construct Maps are laid out as separate steps in the Guided mode of the Map application. There are four components that make up these three steps: an Image window, an X-ray map window, a Spectrum reconstruction window and a Confirm elements window. These four components are combined in the Custom mode to give you a single workspace called Acquire and Construct. It offers the convenience of working in one window without having to move away from it.

The user interface components are docked in the workspace in the four quadrants. Each component can be undocked in a free floating window. It can be dragged on to another monitor, resized or displayed in the full screen view.

There is a toolbar located near the top right corner of the workspace with icons which allow you to toggle on/off each component.

The user interface elements are described below:

#### **Electron Image/Layered Image**

The Scan Image component is docked in the top left quadrant. It allows you to acquire an electron image and a Layered image. You can choose to display either the electron image or the Layered Image.

Press to acquire or display the electron image. Press to acquire map data or display the Layered Image.

#### **Element Maps**

The Element Maps component is docked in the top right quadrant of the Acquire and Construct workspace. You can acquire element maps here and view them in three different ways, Standard, Interactive or Summary view. To get details of all the functions follow the Acquire Map Data link below.

#### Spectrum Viewer

The bottom left quadrant displays the current spectrum. It can be a Sum Spectrum or a Reconstructed Spectrum. At the top right corner of the Spectrum Viewer, there is a link to the Confirm Elements step of the Point & ID Navigator. It is a useful option for identifying and confirming small peaks in the spectrum. Select Map to get back into the Acquire and Confirm workspace from the Confirm Element screen.

#### **Selected Element Details**

The Map Details is located in the bottom right quadrant of the workspace. From the Selected Element Details, you can select which elements you wish to map. You can define the energy windows for window integral maps and select the X-ray lines you wish to use for mapping instead of using the automatically selected energy window and lines. When you select a map from the element map display, the energy window and X-ray lines markers for this element are displayed in the spectrum viewer. To read details of defining energy windows and choosing X-ray lines, follow the link to Construct Maps topic below.

#### See also:

Acquire Map Data on page 363

Construct Maps on page 370

Acquire Map Data - Settings on page 211

Context Menu - Map Viewer on page 223



# **Linescan - Guided**

In the Guided mode the Linescan navigator has four steps:

Describe Specimen, Scan Image, Acquire Line Data and Construct Linescans. The two new steps are described here:

Acquiring linescans	241
Displaying and manipulating linescans	243
Measuring the distance between two points	245
Viewing element counts and percentages	246
Comparing element quantities	247
Smoothing the linescans.	248
Linescan Data	249
Exporting the linescan data	250
Extracting a single spectrum from the linescan	251
Extracting multiple spectra from the linescan	252
Construct Linescans	255

### **Acquire Line Data**



In this step you can acquire element linescans along a line defined on the electron image or map. The data can be processed in several ways:

- Line, also known as Window Integral, obtains the counts in the element energy windows including the background. Line gives a fast and simple representation of the X-ray energies.
- TruLine, also known as Filtered Least Squares (FLS), applies further processing. Sometimes the standard X-ray mapping (Line) gives misleading results because some elements have overlapping energy windows. For example, a Titanium linescan

might include Barium information. The TruLine option eliminates the problem by comparing the X-ray line series with the expected peak shape for each element. The linescans are corrected for peak overlaps and any false variations due to X-ray background.

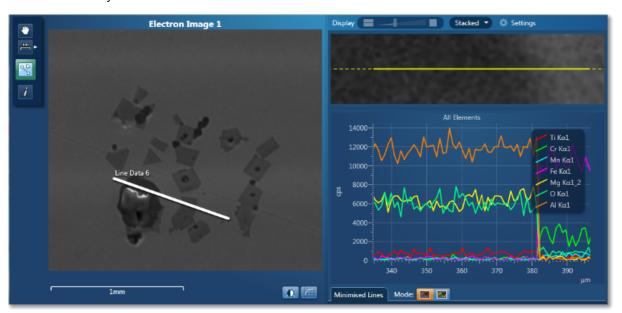
• QuantLine further processes the data, showing the atomic or weight percentages of elements at every point on the line.



# **Acquiring linescans**

The elements for which linescans are being acquired are chosen in the Describe Specimen step by selecting the Auto ID option, Pre-defined Elements, or both. To see the acquisition settings, click the Settings cog in the toolbar in the title bar.

- 1. Select the Acquire Line tool from the toolbar on the left.
- 2. Click on the image to set the start point and then drag the mouse to define the line. Release the mouse to set the end point. A line with start and end points is defined on the image.
- 3. Press to start acquisition. A relevant section of the image is zoomed and rotated above the Linescan viewer. This action aligns the defined line horizontally to match the x-axis of the Linescan viewer.



The progress of line data acquisition is displayed in Current Site tab in the Data View:



- 4. Element linescans start to populate the Linescan viewer as the data is being acquired.
- 5. You can stop the acquisition by pressing or the red STOP Button. To cancel the line processing, click the "Cancel Processing" button in the acquisition tool bar.
- 6. From the controls above the Linescan viewer, choose how to process the data, for example: TruLine.

The TruLine data processing will use the TruLine settings from the EDS Element settings tab in the User Profile screen. You can access the User Profile from the Tools menu. You can specifically define whether the threshold is on by selecting "Apply threshold for TruLine" and entering a Sigma Threshold between 0.0 and 3.0.

#### See Also:

User Profile on page 22

Acquire Line Data - Settings on page 252



# Displaying and manipulating linescans

Three different views are available from the controls in the top right corner of the Acquire Line Data screen:

■ Di	splay	<sup>,</sup> Electron	Image	Ful	l Screen
------	-------	-----------------------	-------	-----	----------



 Display Linescans and Electron Image



■ Display Linescans Full Screen



Several options are available to view the Linescans:

- Stacked multiple linescans overlaid are displayed in a single view.
- Vertical Tiles individual element linescans are displayed in a separate view. You can change the height of each view using the Display slider bar.
- Table values for each point on the line.

You can pan and zoom linescans using the mouse controls. Both the viewers, (image and line view) respond synchronously to the mouse interactions.

- Left mouse button down:
  - Move left / right pan left/ right (if view is expanded). Tip: If the list of elements and lines obscures the right end of the linescan, pan fully to the left.
  - Move up / down change the scale height
- Mouse wheel
  - Zooms x range in/out around current x value (defined by mouse location). The image will expand/shrink to match the data displayed. If the data is not visible in the viewer because of pan/zoom state, the line on the rotated image will change to a dotted yellow line to indicate there is more data.
- Linescan viewer specifics:
  - Dragging directly on the axis will pan the range.
  - Mouse wheel on the axis will expand / reduce the range.

The thickness of the line in the plots can be set globally from the Linescans Viewer tab in the Preferences screen. The thickness values in pixels are as tabulated below:

	Thickness in Pixels
Thin	0.5
Normal	1.0

	Thickness in Pixels
Thick	2.0
Thicker	4.0

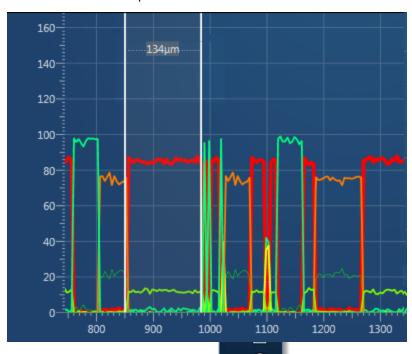
In addition you can change the color and thickness of individual lines from the Settings in the Linescan viewer toolbar.



# Measuring the distance between two points

The distance between two points in the Linescan viewer can be measured using the Caliper tool.

- 1. Select the tool from the toolbar on the left, and move the mouse over a Linescan viewer (stacked or tiled). This cursor will track the mouse movement.
- 2. Double-click to set the first measurement point.
- 3. Move the mouse to paint a region on the viewer, which shows the distance between the two points. This will update as the second cursor is moved.
- 4. Double-click to fix the position of the second cursor.



#### NOTE

Unlike calipers on an image, the linescan calipers are not saved with the data. When you switch to a different tool the caliper information will disappear (and will not reappear when you switch back).

A new region can be drawn by double clicking in the viewer at the new (start) point. You can then define the extent of the measurement by placing the second marker, as described above.

To get rid of the region on the viewer, you should switch to another tool on the toolbar.

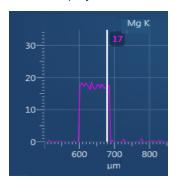
In the Vertical Tiles view, you can use the Caliper tool on individual linescans to display the distance between two points.

# Viewing element counts and percentages

1. Select the "Show data values" tool from the toolbar on the left:



2. Click on the line in the Linescan viewer. A vertical cursor appears at that location, and displays the value for each element in that location. For example:



If no sigma threshold is applied to TruLine processing, some values might be negative. To see the sigma threshold setting, go to the Tools menu, select User Profile, and then the EDS Element Settings tab.



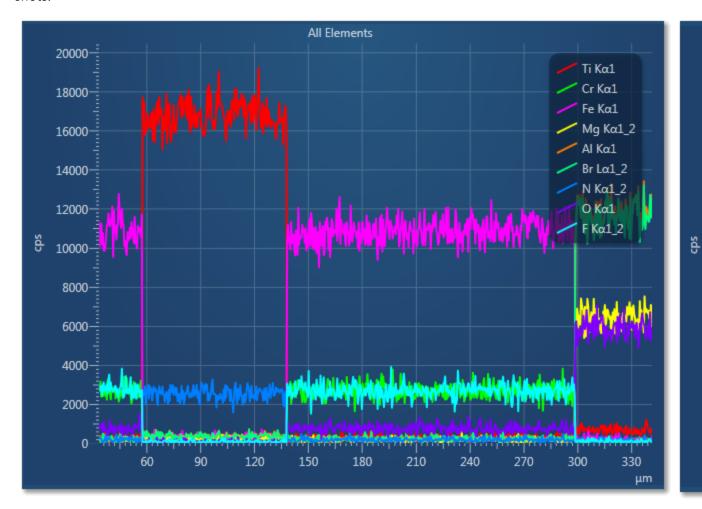
# **Comparing element quantities**

The Normalize Y-Axis option is available on the context menu of the Linescan viewer, when in Stacked view. It allows you to compare linescans with very different maximum count rates, and is useful in QuantLine displays. The maxima of linescans are scaled to the full height of the viewer. It allows you to view the details of the minima of linescans with low count rate.

Note that there is no Y-axis on the normalized linescans because the absolute scale is meaningless.

# **Smoothing the linescans**

The Smoothing Factor option is available from the Linescan viewer settings. It allows you to smooth the linescans after normalization for visual clarity as demonstrated in the screen shots:



Typical linescan

Smoo

The Smoothing Factor uses moving averages to remove fluctuations of data.

The options available are:

- 1: No smoothing applied
- 3: Data averaged over three points
- 5: Data averaged over five points

As an alternative way to reduce the effects of noise in the linescans, you can apply a binning factor. Binning is particularly effective when using TruLine.

### See Also:

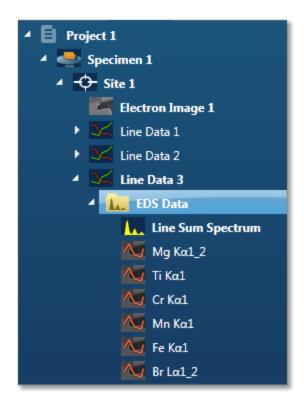
How binning affects the quality of your data on page 373.



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# **Linescan Data**

The data tree contains a Line item under the Site; this is the container for the line data. By default, this is labeled as 'Line Data #' where # is an auto-increasing number under the current site (Site 1) as shown below:



Icon	Description
	The Line item is the container for EDS Data. All linescans and the sum spectrum are contained within the EDS Data container. For details, right click the icon.
	The sum spectrum is called Line Sum Spectrum.
<b>△ △ △ ★</b>	The name of the element linescan (Line, TruLine or QuantLine) is composed of the element symbol and the line.
	A spectrum extracted from a single point on the linescan.

### See Also:

Data Tree on page 85

# **Exporting the linescan data**

Right click on the linescan viewer to access the Export menu. It has several different ways of exporting the linescans: Save As, Copy, Print and Email. The exported image includes the relevant information from the Caliper or Show data values tool.

To export some or all of the data to a spreadsheet program such as Microsoft® Excel®:

- 1. On the toolbar above the linescan viewer, select Table from the drop-down list.
- 2. To select all the data, click one row in the table, then press Control and A. To select only some of the data, click a row then drag, or use click and Shift-click.
- 3. Right-click and select Copy.
- 4. Paste the data into your spreadsheet.

#### See Also:

Toolbars on page 253



# Extracting a single spectrum from the linescan

You can examine the spectrum and element quantities at any point along the line.

1. In the toolbar on the left of the image viewer, click the Reconstruct Line Point Spectrum tool:



- 2. Move the cursor to any point on a line in the linescan viewer.
- 3. Click to extract the spectrum for the point into the project.

You can view the spectrum in the Mini View. The data is also saved in the Data Tree with this icon:



# Extracting multiple spectra from the linescan

You can examine the spectrum and element quantities at many points along the line. To prevent an excessive amount of data, you can apply a binning factor to limit the number of points.

1. Click the Extract Spectra button below the linescans:



- 2. In the Reconstruct Spectra dialog, select a binning factor.
- 3. Click START to extract the spectra for the points into the project.

The data is saved in the Data Tree with this icon:

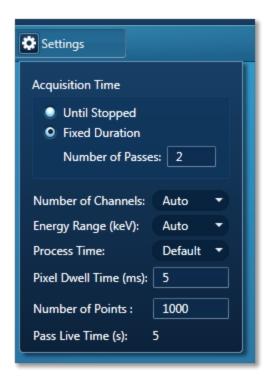


#### See Also:

How binning affects the quality of your data on page 373

# **Acquire Line Data - Settings**

A settings cog in the Acquire Line Data window provides access to the settings for acquiring linescans:



You can specify the Acquisition Time, Energy Range (keV), Number of Channels, Process Time, Pixel Dwell Time (ms), Number of Points in the line or the separation between points, and Live Time (s) per Pass.



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In the Auto mode, the system checks for the accelerating voltage selected on the microscope and sets the appropriate spectrum Energy Range. Based on the Energy Range selected, an appropriate value for the Number of Channels is set automatically.

Use the Default Process Time if you wish to acquire good quality data at the optimum speed.

### **Toolbars**

### **Acquire Line Data - Acquisition toolbar**

These controls are at the top of the window, above the electron image and the line scan.

Control	Description
► START	Starts the acquisition of linescan data.
■ STOP	Stops the acquisition of linescan data.
Settings	Opens a window where you can specify acquisition parameters such as the number of points along the line.
	Offers a choice of processing: Line, TruLine or Quant- Line.

### LineScan viewer - toolbar

These controls are above and below the line scan.

Control	Description		
	Changes the height of each element linescan in the Stacked view.		
	Offers a choice of display:		
	<ul> <li>Stacked - overlays multiple linescans in a sin- gle view.</li> </ul>		
	<ul> <li>Vertical Tiles - displays individual element linescans in a separate view. You can change the height of each view using the Display slider bar.</li> </ul>		
	<ul> <li>Table - displays data for each point and ele- ment in a table layout.</li> </ul>		

Control	Description	
Settings	Allows you to change the color and thickness of each line, and the smoothing factor for all the linescans.	
K.	Extracts all spectra from the line.	
Result Type	Shows the percentages by weight or the number of atoms . Available for QuantLine processing only.	
Binning Factor	Sets the binning factor. Binning produces an averaged signal, which has less noise overall.	

### **Acquire Line Data - left toolbar**

These controls are at the top left side of the electron image.

Control	Description
•	Pans and zooms the image or linescan.
nm	Adds annotation to the image.
	The caliper tool measures distances between points on the image and the linescan.
<b>Y</b>	Marks the line on the sample, then acquires the data.
N. Control	Extracts a spectrum from a point on the linescan.
i	Shows intensity at any point on the electron image.
_	Shows the counts per second or element percentage on each linescan.

### See Also:

Acquiring linescans on page 391

Acquire Line Data - Settings on page 252

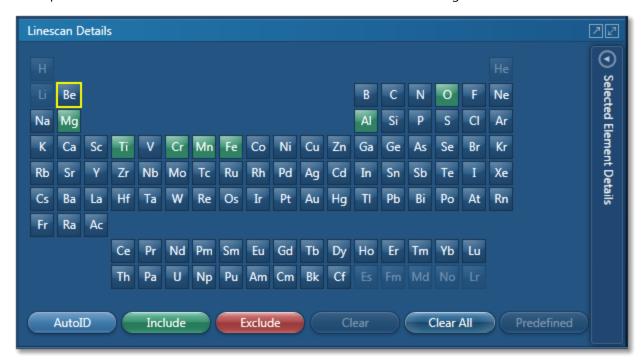


### **Construct Linescans**

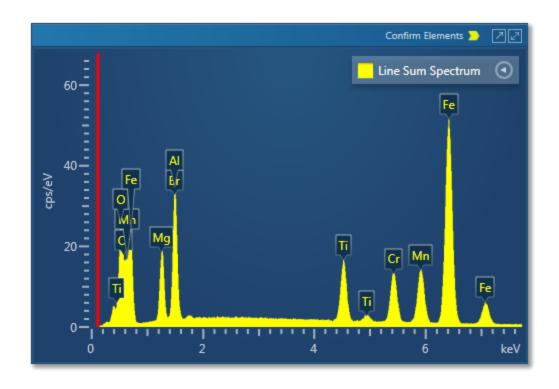


In this step you can define energy windows and configure X-ray line series to update the display of element linescans in the viewer.

You can use AutoID for initial display and then add or remove elements as you wish using the periodic table and the controls available in the Linescan Details dialog:

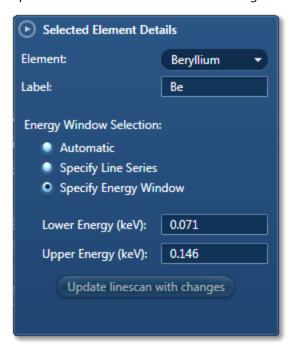


In addition, you can view the Line Sum Spectrum and navigate to the Confirm Elements step from within the Construct Linescan step to manually confirm elements:



# Manual selection of energy windows and X-ray lines

1. Press to open the Selected Elements Details dialog:



The default settings are automatic X-ray line series and energy window width selection.



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press

2. To manually define the width of the energy window, check the 'Specify Energy Window' option. Enter the values for Lower Energy (keV) and Upper Energy (keV) and Update linescan with changes

3. To manually select the X-ray line for an element linescan, check the 'Specify Line Series' option. Select the line from the Line Series drop-down list and press

Update linescan with changes

### **Linescans Display**

There are three different display options available from the controls, near the top right corner of the Construct Linescans screen:



- Display image full screen
- Display linescans and image
- Display linescans full screen

#### See Also:

How binning affects the quality of your data on page 373

Line	escan -	Custom		
Line	escan -	Custom	 	

In the Custom Mode, the Linescan navigator has two steps:

The Describe Specimen step is explained in the earlier section. The new step is described below:

Acquire and Construct - Linescans 259



# **Acquire and Construct - Linescans**



The three components, Scan Image, Acquire Line Data and Construct Linescans are laid out as separate steps in the Guided mode of the Linescan application. These three components are combined in the Custom mode to give you a single workspace called Acquire and Construct. It provides the convenience of working in one screen without having to move away from it.

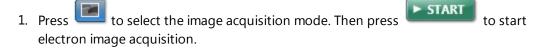
The user interface components are docked in the four quadrants in the workspace. Each component can be undocked as a free floating window. It can be dragged on to another monitor, resized or displayed in the full screen view.

There is a toolbar located near the top right corner of the workspace with icons which allows you to toggle on/off each component.

The user interface elements are described below.

### Acquiring an electron image and line data

In the top left quadrant, you can acquire an electron image first and then define a line to acquire the line data.



- 2. On completion of image acquisition, press to switch to the line data acquisition mode.
- 3. Press to select the line tool in the toolbar on the left.
- 4. Click on the image to set the start point and then drag the mouse to define the line. Release the mouse to set the end point. A line with start and end points is defined on the image.
- 5. Press to start the line data acquisition. A relevant section of the image is zoomed and rotated above the Linescan viewer. This action aligns the defined line horizontally to match the x-axis of the Linescan viewer.

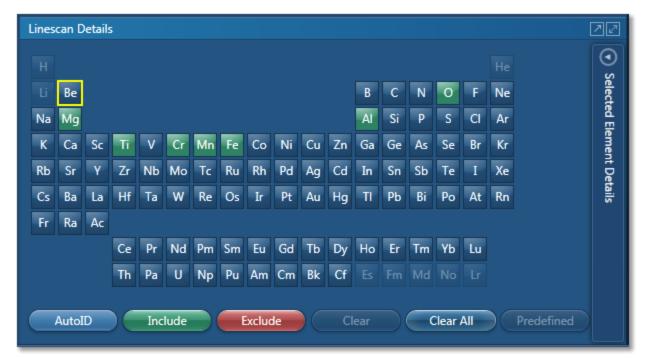
Element linescans start to populate in the Linescan viewer from in the top right quadrant as the data is being acquired as shown in the next screen shot:



#### **Selected Element Details**

In the bottom right quadrant, you can define energy windows and configure X-ray line series from Selected Element Details.

AutoID can be used for initial display. You can add or remove elements as you wish using the periodic table:

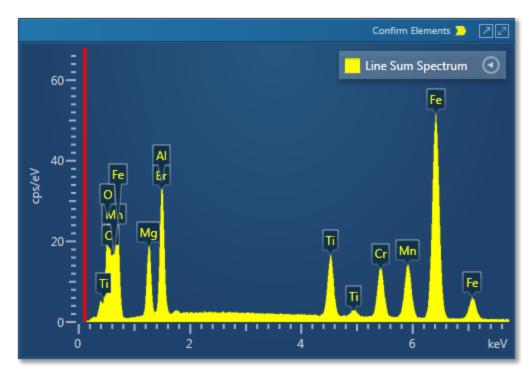


**Spectrum Viewer** 



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The spectrum is available in the bottom left quadrant. You can view the Line Sum Spectrum and navigate to the Confirm Elements step from within this component to manually confirm elements:



Optimize	
There are two components in the Optimize navigator, Calibrate ar	nd Standardize.
The Calibrate step is described in an earlier section. The Standard	ize step is described here.
Standardize	263



### **Standardize**

The Standardize application allows you to setup your own standards for quantitative analysis. It is accessed from the Optimize navigator:



Use the links below for further details about:

Why Standardize? below

Getting Started with Standardization on page 265

Managing Standardizations on page 267

### Why Standardize?

Quantitative analysis can be performed without the need to measure standard materials since your system is supplied with a complete set of default standardizations. However, in specific cases, using your own standards will lead to an improvement in your quantitative results. The purpose of Standardize is therefore to enable you to set up your own standards for quantitative analysis. When you use your system for the first time and click on the Standardize step, your own standards database will be created. At this stage your database will contain only the Factory standardizations. Once you have standardized entries, your standards database will be modified accordingly.

Quantitative analysis of elements in any Specimen, requires an accurate measure of the intensity of peaks, before the concentration of elements in a Specimen can be calculated. In determining peak areas in spectra, two problems arise:

- A typical spectrum contains characteristic peaks, which are superimposed on a slowly varying background, which is 'noisy' because of statistical variations.
   This background contribution needs to be carefully subtracted from the spectrum.
- The energy resolution imposes a limit on the separation of peaks. Identification of peaks is generally not a problem, but overlapping peaks require deconvolution, before being able to extract the true peak intensities relevant to the elements present in the Specimen.

Once these intensities have been determined, a comparison is then made with standards of known composition, followed by application of matrix corrections, before the concentration of each element can be determined.

#### What does standardization do?

In order to make a direct comparison between intensity and concentration, a standard Specimen is referred to in which the relationship between Istd and Cstd is accurately known where Istd is the intensity from the standard and Cstd is the concentration of the standard. Once this is known, this ratio can be used to determine the concentration of that element in an unknown Specimen (Cspe) since the intensity of the element in the Specimen (Ispe) can be measured. Let Cxspe be the concentration of element X in the unknown Specimen and Ixspe be the intensity of the relevant peak from element X in the unknown Specimen. All intensities are assumed to have been corrected for background.

The concentration of element X in the unknown Specimen can be approximated as:

Cxspe={Cxstd(Ixspe/Ixstd)}

and is often referred to as the Apparent concentration or the uncorrected concentration. Once these 'Apparent concentrations' have been determined, the element weight percents are then calculated by applying a matrix correction to the measured intensity ratios. These corrections attempt to account and correct for the effects of X-rays traversing the Specimen matrix such as absorption of X-rays in the material.

The ratio in value between the Apparent concentration and the true concentration is a measure of the matrix corrections which need to be included in the calculation. However, the need for the correction is minimized if the composition of the standard Specimen is as similar as possible to the composition of the unknown Specimen. This simply means that the effect on the X-ray intensity of X-rays traversing the Specimen and standard is similar. Since these matrix corrections can be calculated with only a certain degree of accuracy, the choice of standard material is very important if the quantification is to be as accurate as possible.

#### Why Standardize?

The need to standardize depends very much on the level of accuracy you require from your analysis on any one material. As a rule of thumb:

- If you require accuracy such that the relative errors are less than 2%, you should standardize.
- If you are quantifying elements whose X-ray lines are in the low energy region of the spectrum (which may be the case if you are using an accelerating voltage of less than 15kV, or the element you are quantifying has an atomic number less than 11), standardization will improve your quantitative results.
- If the matrix corrections are high such as would be in the case of quantifying Al in a Pd matrix, (e.g. light in heavy, heavy in light), where your intensity correction Ic is either >1.25 or < 0.8, you should standardize. The intensity correction is given in the quantitative results.

Not necessary to standardize if



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- You only want to know which phase it is.
- Your standards are of a poor quality (rough, dirty).

### **Getting Started with Standardization**

Quantitative analysis can be performed without the need to measure standard materials since your system is supplied with a complete set of Factory standardizations. However, in specific cases, using your own standards will lead to an improvement in your quantitative results. The purpose of Standardize is therefore to enable you to set up your own standards for quantitative analysis.

When you use your system for the first time and click on the Standardize step, your own standards database will be created. At this stage your database will contain only the Factory standardizations. Once you have standardized entries, your standards database will be modified accordingly.

Standardize allows you to add your own standards blocks and enter compositions of all standards on each block. You can standardize on any of the elements available in the standards.

#### Adding composition of standards

You can build your standards database by adding blocks of standards and the compositions of individual standards on each block. Note that if you wish to use pure element standards you do not need to add standards.

- Press Standardize on the Optimize navigator. The Standardize screen is displayed.
- Press Add to add a block in the Standards Compositions area.
- Enter the name for the block and press OK. Your block will appear in the Block list box.
- Press Add to add a standard. The following dialog is displayed:



- Enter name for the standard.
- Enter composition of the standard by choosing either Number of Atoms or Weight%. When you know the chemical formula select the option, Number of Atoms. The formula is validated as you enter it. If you enter it in a wrong format for example feS2 or FES2, it will not be accepted. For Weight% option, you need to enter the element symbol followed by weight e.g., for FeS2, it will be Fe 46.55 S 53.45. It will be symbol weight symbol weight until you have added all elements. The total weight% has to be between 95% and 105%. If it falls outside this range you will be warned about it.

#### Standards

Standards are materials which are used to relate the intensity of a peak in a spectrum to the concentration of that element in the specimen. They are materials in which the concentrations of all the elements are accurately known. Standards may be pure elements or compounds. High quality reference standards are essential to perform accurate quantitative microanalysis in the SEM. Not only must their chemical composition be well characterized, but they must also be microscopically homogeneous, stable under the electron beam and prepared with a flat, polished surface.

#### How to standardize

- Ensure that the spectrum to be used for standardization is displayed in the spectrum viewer. This may be a spectrum you have just acquired in either the Analyzer or in Point & ID or you may wish to standardize using a stored spectrum.
- Select the standards block you wish to use from the Block drop-down list in the Standards Composition section.
- Select the standard that you wish to use from the Standard drop-down list.
- If you wish to set up a Deconvolution element, select it and add it to the Deconvolution text box.
- Select either Pure Element or Element from Standard (Compound standard) as appropriate from the Perform Standardization section. X-ray line markers will be displayed for the selected element (K lines are in red, L lines in green, M lines in purple and N lines in cyan.)
- Select the line you wish to standardize on from the X-ray Line Series drop-down list.
- Having selected the element and the line for standardization, press to perform the standardization. The current and new values of standardization will be displayed.

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#### Note

The Accept button will be grayed out if the Standardizations file is selected for quant. You need to deselect it in the Quant Settings dialog (available from the Calculate Composition screen) if you wish to standardize on any entry in this file.

- In Calculate Composition step, when you press Requantify, the new standardization will be used and displayed in Quant Results Details if you select the Full Results template.
- If you have not accepted the new values for standardization, no changes will be made to the existing standardization.

### **Managing Standardizations**

Your system is supplied with a Factory Standardizations database. When you use your system for the first time and click on the Standardize step, your own standards database will be created. At this stage your database will contain only the Factory Standardizations. Once you have standardized entries, your standards database will be modified accordingly. To manage your Standardizations the following functions are provided:

#### Creating a new standardization file

To create a new standardization file:

- Press the Manage button in the Standardize screen and then select the Create File
- Select an existing file from the Factory Files or User Files. This file will be used to populate the entries in the new file.
- Enter the name for the new file and press the Create button. A new file will be created. You can overwrite the existing entries in the new file with your own standardizations.
- To use this new standardization file, select it in the Quant Settings in the Calculate Compositions step for quantitative analysis.

#### Note

The Factory Standardization file is available to all users of the system. However, files created by individual users are user specific.

#### Deleting a user standardization file

Remember that you can not delete the Factory Standardization file. User created files can be deleted.

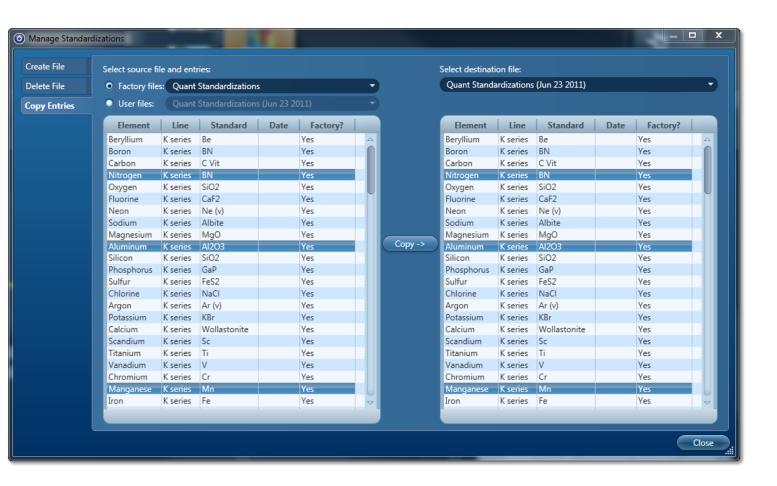
To delete a User Standardization file:

- Select the Delete File option from the Manage Standardization dialog.
- Select the standardization file to delete from the drop-down list.
- Press the Delete button. The selected file will be deleted. Note that this file will not be deleted if it has been selected for quant. You will be warned about it when you press the Delete button.

#### **Restoring standardization entries**

The Copy Entries option allows you to restore standardizations from the Factory File to a User File or from one User File to another User File:

- Press the Manage button in the Standardize screen and then select the Copy Entries tab.
- Select User or Factory Standardization File as the Source File.
- Select your standardization file which contains the modified entries as the Destination file.
- Select the entry in the Factory Standardization file that you wish to restore by clicking on it.
- Then Press the Copy button to restore this entry across to your file. The modified entry in your file will be overwritten with the Factory entry. For copying multiple entries hold down the Ctrl key while selecting individual entries:





• Press the Copy button to copy the selected entries.

#### **Sharing standardization files**

You can share standardization files with other users of the system by using the Export and Import options available in the User Profile dialog from the EDS Quant Setting tab:

- Select User Profile from the Tools menu.
- Select EDS Quant Settings in the User Profile dialog.
- Select the Standardization file from the Quant Standardizations.
- Press the Save As button. The Save User Profile dialog is displayed.
- Enter the name for the User Profile or select an existing User Profile from the list in the Save User Profile dialog.
- Press the Export button. The Standardization file with .ois extension and User Profile file with .config extension are exported.
- You can share these files with other users using the Import option in the User Profile dialog.

# **LayerProbe**

The LayerProbe navigator has the following steps:

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.....



### **LayerProbe**

#### Introduction

LayerProbe is designed to calculate the thicknesses and compositions of multilayer structures on a substrate. The program contains tools which enable you to determine the optimum conditions (kV, beam current, livetime and analysis lines) to be used for subsequent acquisition of spectra from a particular type of sample.

To use LayerProbe you need to be able to define which elements exist in each layer, their approximate concentrations, and the order and approximate thicknesses of the layers. You can then select which thicknesses and concentrations you want to determine (unknowns), and define those you already know (fixed variables). It is also necessary to define the X-ray lines to be measured and which acceleration voltage (kV) to use.

The choice of X-ray lines and kV will depend on the problem to be solved and that requires expert judgment. LayerProbe includes an automatic tool, "Calculate Solubility" which helps to find the best lines and kV to use for a particular problem. The tool will look at what you wish to determine ("unknowns") and what you do know (fixed variables) and will scan a range of kVs trying to find a kV where the problem can be solved.

LayerProbe has to determine all the unknowns you have asked for by measuring the X-ray line intensities for the elements present. If more unknowns are to be determined than there are lines available, the problem will be unsolvable. It can be more complicated than that because at different kV, different lines (K, L, M) are excited, the electron beam may not penetrate all the layers and some X-ray lines may be absorbed on their way out to the detector. Furthermore, sometimes there are various combinations of unknowns that produce an identical set of line intensities in which case the problem is unsolvable under these conditions. The "Calculate Solubility" tool takes all this into consideration and will work out those kV ranges where the problem is solvable and those where it is unsolvable. It also takes into account how the intensity of lines varies with kV and will calculate the relative statistical variation in all the determined unknowns so that a figure of merit can be plotted as a function of kV. The kV at which the problem is solvable is marked in green and where it is unsolvable is marked in red. The "Calculate Solubility" tool will try to find a kV where the problem is both solvable and where best precision is achieved for the unknowns to be determined. At this kV, the tool will show the best X-ray lines to use for the analysis. If you are an expert, this choice can be over-ridden.

Furthermore, an expert can over-ride the default choice of allowed lines that the Calculate Solubility algorithm is allowed to use for each element. However, in most cases, this intervention will not be necessary.

If you want to find out exactly what sort of precision can be expected from the analysis, there is a simulation tool that synthesizes the spectrum that would be achieved in real life on the microscope. If the simulated spectrum is then analyzed, you can see what sorts of results are generated and what precision is achieved in the unknown variables. If the precision is not good enough, you can increase the acquisition time or count rate and try again. All this can

be done very quickly without having to waste any time on the microscope before it is known that the method will work.

Sometimes the best conditions will fail and this will usually be because there are too many unknowns, or because the kV range is not extensive enough. The way around this is not to ask for so many unknowns. In many problems there are certain concentrations or thicknesses that are critical and others that are not important. For example, there might be a layer of contamination present but is not what the experiment is setting out to measure. By making these unimportant variables fixed rather than unknown, a previously unsolvable problem may now be solvable. When a problem is unsolvable, the best conditions tool will sometimes be able to advise which unknown variables are making the problem unsolvable and fixing one or more of these will often work.

Of course, making a thickness or composition fixed means having to estimate the value rather than determine it. LayerProbe allows you to make a guess at the value. The obvious question is 'does it matter how good my guesses are?' and this depends on the exact details of the problem. However, there is another tool available called "Sensitivity analysis" and that will show just how the uncertainty in the guesses will affect the values of the critical thicknesses and concentrations that are being determined. For example, you may find out that provided a contamination layer is less than a certain thickness it will have no serious effect on the accuracy of the results. Again, this can be investigated using the simulation tool to synthesize realistic spectra that can be analyzed without wasting time on the microscope.

The general principle is that the more precisely the problem can be defined, the better chance that LayerProbe will be able to solve it and the more precise the results will be for a certain measurement time. When you tell LayerProbe the approximate values of layer thickness that is to be determined, the Calculate Solubility tool will try to anticipate all possible values when working out if the problem is solvable.

When you are ready to do a real analysis on the microscope, the system will allow acquisition of spectra and will also optimize the accuracy of the quantitative measurements. Data is then saved in a project and spectra then analyzed using the LayerProbe software. The results will show the sample description, the determined values for the unknown thicknesses and concentrations and the calculated precision in these results. As previously described, the sensitivity analysis will show how any guesses in the fixed values would affect the results and also shows how a small variation in beam current during the experiment would affect the results.



# Describe Specimen



In this step there are two tabs, Summary and Pre-defined Elements.

#### Summary

In the Summary view you can write notes on the Project and the Specimen present in the Project. (For convenience you can also copy images/diagrams and text from other documents/emails and paste into these windows). Notes are saved with the Project and you are allowed to edit notes in any step of the Navigator. It helps to capture the important information during the analysis. Click with the right mouse button on the Project or Specimen in the Data Tree and then select Edit Notes to write/modify the relevant notes.

You can add new Specimens to the current Project by pressing the New Specimen button:



Click on the Specimen in the Project Overview dialog. This action displays the 'Specimen Notes for Specimen 1' text box. Here you are provided with the text formatting tools. You are allowed to write notes about each Specimen and save them.

#### **Pre-defined Elements**

If you know what elements are present in your Specimen and you only want to see peak labels or X-ray maps for those elements, then you can select them in the 'Pre-defined Elements' tab.

Press the Pre-defined Elements tab to access the periodic table:

If you wish to enable the AutoID option check the 'Perform AutoID During Acquisition' option.



Double -click on the element symbols that you wish to include in the analysis. All the included elements will be marked with the green color key in the periodic table. To save the Pre-

defined Elements in the current User Profile press Save to Profile. It means when you load the User Profile next time these elements will be included in the analysis.

If you have already created a User Profile with the Pre-defined Elements in the User Profile dialog press Load from Profile .

Pressing will deselect the Pre-defined Elements from the periodic table and they will not be included in the current analysis.

The peaks for the Pre-defined Elements if included in the analysis are labeled in the Acquire Spectra step. MiniQuant will display the quant results for these elements as Wt% or a bar chart.

The Pre-defined Elements will be marked as Pre-defined in the Confirm Elements list box in the Confirm Elements Step. There may be other elements in the Specimen which are identified by AutoID routine if the 'Perform AutoID during acquisition' option has been checked in the User Profile dialog.



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#### Тiр

Right click on the Project or Specimen in the Data Tree and select Edit Notes to write or edit notes in any step of the Navigator.

#### See also:

Element Lists on page 191

Data Tree on page 85

Mini View on page 93

Step Notes on page 94

### **Describe Model**



In this step, the specimen is described by a model, defined by a (multi) layered structure on a substrate. In order to setup the model, you will need to know the sequence of layers and their constituent elements.

There are four simple steps to completing the model:

- 1. Defining the layers
- 2. Defining the composition of each layer
- 3. Defining the thickness of each layer
- 4. Defining the density of each layer

#### 1. Defining the layers

The Model area of the screen is used to define the number of layers and their order. You are able to add new layers, re-order and delete existing layers or clear the model. The 'Clear All' button will remove all the layers, leaving an empty 'Bulk' entry.

Initially when you start to describe a new Model or when the 'Clear All' button has been pressed, the program will display a single 'Bulk' layer. When you add additional layers to the model, the 'Bulk' layer becomes a 'Substrate'.

#### To add a layer to the structure:

- 1. Press "Add Above". The Bulk layer changes to the Substrate layer. The first layer will always be the Substrate layer.
- 2. To define a layer on the substrate press "Add Above". This layer in the structure is layer 1.
- 3. Using the "Add Above" button adds further layers to the layer structure.

#### 2. Defining the composition of each layer

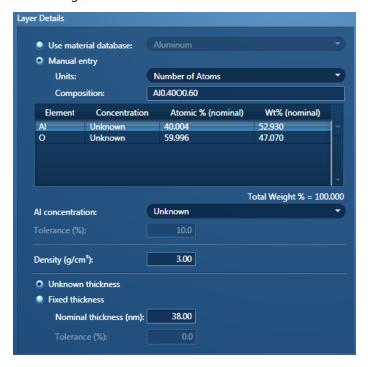
Enter the layer composition of the substrate first followed by the individual layers. The layer details shown on the right hand side of the work space are of the currently selected layer:

- 1. For each layer, the composition is specified by one of the two methods, either by selecting an entry from the materials database or by manually entering the composition as a chemical formula or in weight%.
- 2. When you select an entry from the materials database, all the relevant fields for the layer composition will be automatically populated.
- 3. Select the Manual entry radio button to manually enter the layer details.



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- 4. To enter the composition, choose one of the two modes of entry, Number of Atoms or Weight%.
  - Selecting Number of Atoms allows the entry of the chemical formula, e.g.,
     Al2O3 for aluminium oxide.
  - Selecting Weight% allows entry by specifying the elements and weight% values e.g., Al52.93O47.07:



- 5. Once the layer composition has been entered, the concentration type for each element in the layer defaults to Fixed.
- 6. Select the element in the table and then select the desired concentration type from the drop-down list of Fixed, Difference and Unknown.

The selection of concentration type depends on whether you know the composition or want to determine it. It is important to consider carefully what to define as unknowns because choosing too many unknowns will prevent the software from obtaining a solution.

#### **Fixed**

If Fixed concentration is specified for an element, this means that the concentration is known. However when performing the sensitivity analysis, it is important to enter a percentage tolerance which will be applied to the concentration of that element during analysis. Note that the tolerance % is a relative not an absolute parameter. For example if the concentration of an element is 60%, the tolerance of 10% allows the concentration to be  $60\pm6\%$ .

#### **Difference**

This option can be used if you are able to analyze all elements except one. The concentration of the element selected as "Difference" is not measured, but is calculated assuming that the difference between the analyzed total and 100% is due only to the presence of this element.

Intensity corrections are then calculated assuming the presence of this element. It should be noted that the total from this type of analysis is always 100%.

Setting an element to difference can be used when analyzing a specimen in which a significant quantity of a light element which cannot be detected but is known to be present. It can also be used in cases where an element is present for which no standard is available.

#### Unknown

If the element is defined as "Unknown" the concentration entered will be used as a starting value for the model refinement in the calculation. The closer this estimate is to

the real composition the quicker the LayerProbe calculation engine will be to achieving a result.

A layer must have two or more unknown elements to use the "Unknown" concentration option. (Note that if there is only one element in a layer then this must be defined as "Fixed" and the concentration must be entered as 100 Wt%).

#### 3. Defining the density of each layer

 If you know the density of a layer, enter it in the Density text box. It is expressed in g/cm3.

Note that when a composition containing a single element is entered, the density is automatically set to the element density.

Internally, LayerProbe calculates the mass thickness of each layer. The mass thickness is defined as the product of the density of the material and its thickness. Hence to determine the thickness, the density of a layer must be known and be entered into the model. Therefore it is important to realise that the accuracy of the value in density directly affects the measured thickness.

#### 4. Defining the thickness of each layer

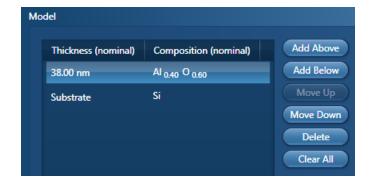
- 1. Select either Unknown or Fixed thickness.
- 2. If Unknown thickness is selected, enter the thickness in nm.
- 3. Specify a percentage tolerance to be applied to the nominal thickness during analysis.

Note that the tolerance % of the nominal thickness is a relative not an absolute parameter. For example if the layer thickness is 40 nm, a tolerance of 15% will allow the thickness to be  $40\pm6$  nm.

Once the composition and thickness of a layer in a structure is defined, it is displayed in a table on the left side of the workspace under the Model. The thickness in nm is displayed in the first column. Then the composition is shown with each element symbol followed by its atomic fraction:



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#### Import/Export/Restore/Save Model

If a model already exists (.tfe or .tff file), you can import it by pressing the 'Import' button:

- .tfe files are ThinFilmID specific.
- .tiff files are Stratagem type files that can be used in ThinFilmID and in LayerProbe.



You can also export your model to a file allowing the use of existing models and the sharing of settings between systems.

Once the model is defined, save it by pressing the Save button. The model is stored in the current User Profile settings and can be restored from the User Profile by pressing the Restore button.

### Scan Image



In the Scan Image step, you can acquire an electron image into a 'Site'. A 'Site' is like a folder, which contains images and analyses for a particular area on a specimen.

For EDS, if you do not want to collect an image and just want to acquire spectra, you can skip this step and go straight to the Acquire Spectra step.

You can have any number of images in a site. Just ensure that the images you want to keep are padlocked in the data tree to stop them being overwritten, as shown in the screen shot below:



You can toggle between saving or replacing the current image with successive image acquisition.

If your specimen is drifting, click the Settings cog and activate AutoLock.

The Scan Image step has several tools for manipulating and enhancing electron images:

- The acquisition toolbar above the electron image and other nearby controls.
- Scan Image toolbar (a vertical toolbar on the left) for manipulating and annotating the image.
- If a forward-scatter electron detector (FSD) is fitted, extra controls are available for combining the signals from each diode into a mixed image.

#### Acquisition toolbar and other nearby controls

The acquisition toolbar, above the electron image and below the Navigator, has buttons for starting and stopping the image acquisition, the Settings cog for selecting the image acquisition parameters and a button to link/unlink images for manipulation.





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Control	Description
(FSD only)	Displays up to three combinations of mixed image and individual images. Your selections are retained in your user profile.
► START	Click to start the image acquisition according to the current acquisition parameters.
■ STOP	Click to stop image acquisition. Acquisition stops at the end of the current frame. Click again to stop immediately. If you navigate away from the step, acquisition stops at the end of the current frame.
Settings	To change the acquisition parameters, click the Settings cog on the Acquisition Toolbar to display a dialog.
	You can select Image Scan Size, Dwell Time (µs), Input Signal the labels here reflect whatever was set during the installation for example SE, BSE or FSD), either Continuous Scan or Number of Frames and Frame Time (secs).
	If your specimen is drifting, you can activate AutoLock to ensure that any analysis corresponds to the true location on your image.

Control	Description
Mixing Mode (drop-down list)	Combines signals from the diodes to form a mixed image. Some options are available only if the required diodes are installed and configured.
(FSD only)	<ul> <li>FSD Z Contrast uses upper and side FSD detector channel images. Select this mode if you are inter- ested in seeing density/atomic Z contrast signal.</li> </ul>
	<ul> <li>FSD Topo/Orientation uses lower FSD detector chan- nel images. Select this mode if you are interested in seeing orientation contrast signal.</li> </ul>
	<ul> <li>Custom - include and exclude FSD detector channel images of your choice.</li> </ul>
	When you select either of the first two modes, the software automatically uses the FSD diode channels associated with that mode. For example, FSD Z mixing mode uses the upper and side FSD diode channels. The FSD Topo/Orientation mixing mode always uses the lower FSD diode channels. Custom mode allows you to mix any FSD diode channels.
Select Second Image	Selects further images to compare with the electron images, for example, a forward-scattered electron image. This control is available only when the map display is for an image only:
(FSD only)	Sets the number of images per row in the Standard and Interactive displays.
Standard •	Offers a choice of image display :
(FSD only)	<ul> <li>Standard - you can add individual images or remove them from the mixed image.</li> </ul>
	<ul> <li>Interactive - similar to Standard. You can also change the weighting and color contributed by each image.</li> </ul>
	<ul> <li>Summary - similar to Interactive and in a more compact display.</li> </ul>
30	Links images. You can simultaneously manipulate all the layers using the pan or zoom controls.



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Control	Description
	Unlinks images. You can manipulate individual layers using pan or zoom controls.
	Use the mouse wheel to zoom in and out of the image.
	Use these tools (near the bottom right of the screen) to adjust manual and automatic brightness, contrast and color.
Context Menus	Right-click the electron image to display context menus for copying, exporting and printing images.

#### See Also:

Scan Image Toolbar on page 424

Scan Image - Settings on page 421

FSD diode controls on page 432

Context Menus - Image Viewer on page 157

Export - Settings on page 132

AutoLock on page 133

# **Acquire Spectra**



#### Introduction

Having defined the model for your specimen and determined the best conditions (kV and X-ray lines) for analysis aided by using the Simulate Spectra and Set up Solver steps, you are ready to acquire spectra from your specimen on the microscope. After spectrum acquisition from the current electron image (SE/BSE) of your specimen, each spectrum is then quantified in real time. These instant quantitative results can be viewed using MiniQuant. The real time spectrum compare options are also available to view your results.

#### **Acquiring Spectra**

- Set the spectrum acquisition conditions such as kV and X-ray lines aided by using the Simulate Spectra and Set up Solver steps or from prior specimen knowledge.
- Perform a Beam Measurement using a pure element standard such as Co or Mn in the Calibrate step of the Optimize navigator. Repeat the Beam Measurement as often as is required in order to check beam current drift. For details of the Beam Measurement routine refer to the Calibrate help topic link below.
- Acquire a spectrum from the specimen. The spectrum will then be added to the data tree in the current project.
- View the MiniQuant results on completion of spectrum acquisition.

#### **Notes**

The Beam current must be the same for both the standard and specimen. It is vital that the beam current is stable to achieve good quantitative analysis.

For accurate identification of peaks, you will need to perform an Energy Calibration. However, the AZtec system has very stable electronics and therefore you may only need to calibrate the system once in several months, provided the environmental temperature of the laboratory is fairly stable.

The Sensitivity analysis display in the Confirm Analysis step shows the effects of beam current variation. In some analyses, a 1% variation in beam current could give significant errors in results and therefore stability in beam current is important.

#### **Display options**

You can display the components that you are working on such as image and spectrum using

the controls on this toolbar



located in the top right side of the screen. You



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have choice of displaying image and spectrum as shown in the screen shot below or just an image or a spectrum full screen:

#### **Acquisition Toolbar**

There is an acquisition toolbar near the top of the workspace:



It has controls for starting and stopping the spectrum acquisition. There is also a Settings cog for selecting the acquisition parameters. For details see Acquire Spectra - Settings link below.

The toolbar located on the left side of the workspace has various tools for image and spectrum manipulation, enhancement, annotation and area selection. For details see Acquire Spectra - Toolbar topic from the link below.

#### Manual and Auto Color/Contrast/Brightness/Gamma controls

There are manual and automatic color, contrast, brightness and gamma controls available for the image view. You can use these controls to enhance and high light certain features in the image. For details of these controls see the link below.

#### Compare & MiniQuant Results option

The Compare Spectra & MiniQuant Results options are available in the top right corner of the Spectrum viewer. Instant MiniQuant results are displayed on completion of spectrum acquisition. Results are separated into layers. The layer thickness heads each line in the display. Elements are listed next to the layer heading along with either the Atomic or Weight Fraction value as selected from the MiniQuant Settings cog. The substrate layer is displayed at the bottom of the list.

The composition will be displayed in different ways according to the result type. For example if the result type is Atomic fraction, then Sodium Chloride (NaCl) will be shown as Na0.50Cl0.50. If the result type is weight fraction, then the composition will be shown as Na[0.61]Cl[0.39]. Note the use of square brackets when describing weight fraction.



#### **Comparing spectra**

The current spectrum can be compared with another spectrum in the Data Tree from the same project or from another project. The compare spectrum can be chosen via the 'Compare drop down'. The selected spectrum is overlaid on the current spectrum as a line spectrum. It is a very useful feature for visually identifying any differences in the spectra.

#### See Also

Acquire Spectra - Settings on page 313

Acquire Spectra - Toolbar on page 151

Calibrate on page 66

Brightness, Contrast and Gamma Controls on page 214

Context Menus - Spectrum Viewer on page 321



# Confirm Analysis



In this step, the thickness and composition values for the model structure defined in the 'Describe Model' step are calculated from the experimental data selected in the data management using the nominal values of the unknowns as starting points. The quality of the solution can be assessed visually by overlaying the theoretical spectrum and the fitted spectrum over the experimental spectrum or numerically by comparing predicted and measured kratios and reviewing the sensitivity of the solutions to assumptions in the model or beam current fluctuations.

#### **Spectrum Overlays**

The options of theoretical and fitted spectrum overlays are available from the Settings cog.

#### ■ Theoretical Spectrum

Under 'Settings', when you check the 'Show Theoretical Spectrum' option, a theoretical spectrum is overlaid on the experimental spectrum. This option calculates a full X-ray spectrum from the composition defined for the current model. The calculation includes the efficiency of excitation of all X-ray lines, the effects of absorption and back-scatter within the specimen and calculates the relative intensities of all lines in addition to the Bremsstrahlung background. Although the theory is not perfect, it normally predicts peaks and background within about 10% accuracy. If any elements have been misidentified or the element composition ratios are incorrect, the theoretical spectrum will appear significantly different from the experimental spectrum, either in terms of peak intensities and/or background.

When the theoretical spectrum is a good match to the experimental spectrum, this provides useful confirmation that the analysis results are sensible.

#### Fitted Spectrum

Under 'Settings', when you check the 'Show Fitted Spectrum' option, a fitted spectrum is overlaid onto the experimental spectrum. The choice of peaks in the fitted spectrum is based on the elements defined for the composition of the model in the 'Describe Model' step and their heights are obtained by individual fitting. If any elements are missed, the fitted spectrum will not overlay correctly on the experimental spectrum. This is a useful way to confirm that the elements in the experimental spectrum are the same as defined in the model.

#### **Analysis Lines**

The X-ray lines used for analysis ('Analysis Lines') are displayed in the 'Analysis Lines' table below the Spectrum Viewer. Lines can be selected or deselected by checking or unchecking the relevant box.

Alternatively, you may have used the 'Calculate Solubility' procedure in the 'Set Up Solver step' to optimize the analysis conditions and determine the appropriate analysis lines to use for a particular model. To use these lines, press the 'Use Selected Conditions' button.

#### Quantify

When 'Quantify' is pressed, the current spectrum is processed. The element list is fixed and determined by the set up of the model and the deconvolution element list. For each element line, the peak area from FLS processing is used to calculate a k-ratio which is the ratio of intensity of the line to the intensity that would be observed from a pure element under the same conditions. The intensity for the pure element is calculated from the 'Beam current measurement' and the standards file.

The k-ratios are then used in the Stratagem engine. The nominal values specified in the sample model are used as the starting point of the true sample description. The Stratagem engine then calculates what k-ratios would be expected for this guess and compares these with the measured k-ratios from spectrum processing. It then refines the guess to form a new estimate of the true sample model. This process proceeds by iteration until a sample model is achieved that is consistent with the measured k-ratios. The systematic errors between the predicted and measured values are shown as '% rel.diff' and these can be compared with the statistical variation expected for the measured k-ratios shown as '% rel.sigma'.

It is important that you review '% rel.diff' to see how good the fit is to the data. If '% rel. diff' is high then the results should not be relied upon and you should consider ways to improve your results. When the difference between the measured and predicted k ratio is greater than six times the '% rel.sigma', the % relative difference will be displayed in red.

#### How large is the error of the result?

If the theory for X-ray generation and the measurement of k-ratios were perfect, the reported values for layer thickness and element concentrations would give predicted k ratios that agreed with measured k-ratios to within the statistical precision of the measurement. The relative statistical precision is shown as '% rel.sigma' and this should be interpreted as follows: if repeat analyses are performed, the observed '% rel.diff' value will fluctuate but will typically stay within  $\pm$  3 × '% rel.sigma'.

Statistical variation can always be reduced by increasing the Livetime for acquisition or the count rate. If you simulate a typical spectrum and quantify it, you can see what sort of sigma values you are likely to get in a real measurement and then adjust the acquisition conditions to bring the sigma values within your desired level of accuracy for the measured thicknesses and/or concentrations.

It is impossible to be certain there are no systematic errors but the k ratios provides a useful consistency check. If the magnitude of '% rel.diff' exceeds  $3 \times$  '% rel.sigma, this suggests there is some error in either the theory or the measured k-ratios. Errors in measured k-ratios can be reduced by using standardization in AZtec.



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When a k-ratio has a large '% rel.diff' with magnitude exceeding  $3 \times$  '% rel.sigma' this suggests that there is a systematic error. In that case, any of the measured thicknesses and/or concentrations could be subject to a similar '% rel.diff'. For example, if there is a k-ratio with a '% rel.diff' of 25%, it could be expected that one or more of your determined thicknesses or concentrations could also be subject to a large error. In some cases, it may be possible to exclude this X-ray line from the analysis. In such a case, it is recommended that you manually remove this X-ray line from the sample model in the Setup Solver step and determine if the problem is soluble without this line. When the analysis is repeated, you should then find a better fit between the measured and predicted k-ratios which will provide more confidence in your results.

#### **Analysis**

Sample Summary

This shows a summary of the model of your sample. Note that the results are displayed in atomic fractions.

Sensitivity

The 'Sensitivity' choice in the drop down box shows how variations in the beam current can affect the analysis. Also shown is how the uncertainty in the fixed quantities will affect the calculation of unknown quantities.

In terms of the sensitivity of the analysis with respect to small changes in beam current, since the SEM beam current may change between the measurement of the Beam optimization standard and the measurement of the spectrum from the sample, the k-ratios will change in proportion. The 'Sensitivity' output shows how a change of -1% and +1% will affect each determined unknown.

#### **Options**

Press the 'Options' cog to open the 'Options' window. In this window, deconvolution elements and standards files can be selected as well as thresholding of results and the window artefact correction.

#### Deconvolution Elements

You may select Deconvolution elements here in order to remove peaks arising from spurious peaks. To exclude any element from the quantitative calculation, select the required element/s from the drop down list and press 'Add element'. More than one element can be selected for deconvolution. To remove any elements from the list, highlight the required element and press 'Remove element'. The peaks associated with all the elements which have been selected for deconvolution will automatically be deconvolved from the spectrum prior to quantification. This procedure involves fitting to the peak and removing the peak's contribution from the spectrum. This becomes important when there is an overlap with neighbouring peaks.

Thresholding

If thresholding is enabled, any X-ray line whose k-ratio value is below the minimum value allowed by the Stratagem engine is assigned the minimum allowed k ratio value in order for the calculation to proceed without an error message.

#### Window artefacts

If you check the 'Correct for Window artefacts' box, a correction is applied to compensate for the presence of a thin window in front of the X-ray detector.

In some situations, you may observe a carbon K energy peak in the spectrum even though there is no carbon in your specimen. This is an artefact caused by the detector vacuum window. The real background (bremsstrahlung) spectrum should be smooth with no bump at Carbon. The thin detector window is made from polymer with a high carbon content and the absorption of X-rays changes dramatically at the carbon absorption edge energy. Any other materials in the detector window will cause a similar absorption artifact. The artifact at carbon will give a significant carbon peak area. For example, if the thickness of a carbon film on silicon were being determined, the artefact could make the film thickness appear larger than it really is.

The 'Correct for window artefacts' feature takes account of all absorption steps due to whatever window material has been used in the detector and removes the systematic errors caused by such artefacts. The effect of the correction can be seen by performing analysis with and without the box checked.

#### **Quant Standardizations**

Select whether you wish to use the Quant Standardizations as supplied by the factory (note that the extended set is also available) or the User defined set of standardizations.

If you need accurate results, although good results can be obtained by using factory standards, you can improve the accuracy of your quantitative results by using your own standards.

#### **Standardize**

In AZtec, quantitative analysis can be carried out without the need to measure standard materials since your AZtec system is supplied with sets of factory standardizations.

If the factory standards are not good enough, then you must make standards. As a rule of thumb, we recommend that you analyze a well characterized material which is typical of the sample you will be working on. Note that standards must be bulk materials.

Typically you may need to standardize if:

- Accelerating voltages are less than 10kV.
- You are analyzing light elements.
- You are using X-rays lines less than 2 keV.

The need to standardize depends very much on the level of accuracy you require from your analysis on any one material. As a rule of thumb:

• If you require accuracy such that the relative errors are less than 2%, you should standardize.



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- If you are quantifying elements whose X-ray lines are in the low energy region of the spectrum (which may be the case if you are using an accelerating voltage of less than 15kV, or the element you are quantifying has an atomic number less than 11), standardization will improve your quantitative results.
- If the matrix corrections are high such as would be in the case of quantifying Al in a Pd matrix, (e.g. light element in heavy matrix or vice-versa), you should standardize.

### Calculate Layers.



This Step allows you to view your quantitative results in more detail using a number of available templates. It also provides the opportunity to produce tables containing results from multiple spectra. Such tables may be exported to third-party software for further processing using the Copy and Paste function.

For multiple spectra, statistical parameters such as average composition and layer thickness are also calculated. Note that if a particular spectrum has been previously quantified, the existing results are displayed unless the Requantify button has been pressed.

#### **Available templates**

- Select the template that you wish to use.
- For customizable templates, you can select which data type you wish to show in the columns when you view the results. Press Edit Columns to show the window where you can hide/show such columns such as Density, Thickness, Weight %.

#### **Templates for Single Spectra**

- If you want to see a comprehensive set of results from a single spectrum, choose the 'Full Results Table (customizable) - Single Spectrum' template. The results are shown for the spectrum currently highlighted in the Data View.
- Templates that show results for a single spectrum present each layer as a separate row. You may decide what information to display in the columns by pressing the Edit Columns button. Select the type of information to display in the Edit Columns window.

#### **Templates for Multiple Spectra**

- To populate a multiple spectra template, hold the Ctrl key down while choosing spectra on the Data View and then press the 'Add Selected Spectra' button at the bottom of the Data View window.
- Templates that show results for multiple spectra present each spectrum in a separate row. In a given row there are there are repeated sets of columns, one set per layer. This is followed by another set of columns which refer to the next layer etc.
- Templates that show results for multiple spectra include a statistics table that shows Max, Min, Average and Standard Deviation for each visible results column in the main table.

#### **Additional notes**



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- All templates that display Quantitative results include an 'Unknowns Only' check box which is un-checked by default. You can check this box to exclude all Fixed thicknesses and concentrations from the results and statistics tables.
- If you wish to change which standardization file or deconvolution elements to use, press the Options button to display the LayerProbe Options window.
- Press the Requantify button to display the recalculated results.

### **Edit Materials**



In this step you can build a 'database' of materials that can be used to populate layers within a model. The composition and density of commonly used materials can be stored here so that they do not have to be re-entered every time.

#### How to add an entry to the Database?

- 1. Enter the name of the material in the Name text box.
- Choose a composition type from the two available options, Number of Atoms or Weight%. Select Number of Atoms if you know the chemical formula e.g., for aluminum oxide, enter Al2O3 in the Composition text box. The elements and their Atomic % and Weight % are populated automatically in the table.
- 3. Enter the Density (g/cm³) if you know it. For a pure element, the density is set automatically and is set to 1.0 for a compound.
- 4. Press the Add button to save this material entry to the Database.

#### Name

Materials are stored under unique names specified by you. Each material contains one or more elements along with their atomic % and weight % stored with each element.

#### Composition

The composition is specified as a chemical formula (for example Aluminum Oxide is entered as Al2O3), or elements with weight % (Al52.926, O47.074).





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When entering compositions as weight %, it is important to note that the total weight % for a material must add up to (100 +/-0.1)%.

In addition, you are only allowed to add valid materials (valid element symbols/chemical formulae) to the database. A flashing red rectangle means that the material entry is not valid.

#### Density

If you know the density, you can enter the value of the material in g/cm<sup>3</sup>. When a composition containing a single element is entered, the density is automatically set to the element density. However if a compound is entered, the density is always set to 1.0 by default. It should be noted that if a pure element is specified which is normally present in the gaseous state, then the density is given a default value of 1.0 g/cm<sup>3</sup>.

#### **Additional notes**

When adding a material, if a material with the same name is already stored in the database you will be warned and given the option to overwrite the existing entry.

If you wish to delete a material from the database, select it and press delete. Note that a warning will be shown before deletion.

# Simulate Spectra



In this step you can simulate a spectrum based on the model of a layered structure. This may be helpful when setting up the experiments or when refining the model of a partially unknown specimen. A spectrum is selected from the data tree, which acts as the template spectrum for synthesizing a spectrum. Parameters such as geometry settings and beam calibration will be read from this spectrum.

It is important to note that the template spectrum must be beam calibrated. Spectrum acquisition parameters are then selected in the various fields and a spectrum is simulated.

#### To simulate a spectrum:

- Set up a model including all of the layers and the substrate of the specimen you
  want to simulate, either using the Describe Model step or the interface provided in
  the Simulate Spectra step. Save the model by pressing the Save button. Your model
  will be saved in the User Profile.
- 2. Select a spectrum from the Data Tree in order to use it as a template. Note that the values for the tilt, elevation and azimuth angles displayed are of the template spectrum and are not editable.
- 3. The livetime and the beam current factor are user selectable. You can change these settings to find the optimum conditions. Note that the calculated count rate is a result of the spectrum simulation and is not directly editable.
- 4. The accelerating voltage and acquisition range are taken from the template spectrum in the data tree but can be changed in order to explore how the spectrum depends on the accelerating voltage used.
- 5. You may add the simulated spectrum to the Data Tree by pressing the Add Spectrum button.

#### Setting up a model

Set up a model of your specimen by including all of the layers and the substrate of the specimen you want to simulate. This may be done either by using the Describe Model step or the interface provided in the Simulate Spectra step. Once you are satisfied with the model, you may save it by pressing the Save button. This will ensure that your model is saved in the User Profile.

It should be noted that if you are generating a new model, all values for the thickness and the concentration are set to 'fixed' by default.

You may import a previously defined model (.tfe or .tff files) by pressing the import button or export the current model by pressing the Export button to share the model between sys-



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tems. Note that if you import a model, edit some of the values, previous settings in the user profile can be restored by pressing Restore.

#### **Layer Structure**

Defining the composition of each Layer

Enter the composition of the substrate first followed by the individual layers. The layer details shown are of the currently selected layer:

- For each layer, the composition is specified by one of the two methods, either by selecting an entry from the materials database or by manually entering the composition as a chemical formula or in weight%.
- When you select an entry from the materials database, all the relevant fields for the layer composition will be automatically populated.
- Select the Manual entry radio button to manually enter the layer details.
- To enter the composition, choose one of the two modes of entry, Number of Atoms or Weight%.

Selecting Number of Atoms allows the entry of the chemical formula, e.g., Al2O3 for aluminium oxide.

Selecting Weight% allows entry by specifying the elements and weight% values e.g., Al52.93O47.07:

#### Defining the density of each layer

• If you know the density of a layer, enter it in the Density text box. It is expressed in g/cm3.

Note that when a composition containing a single element is entered, the density is automatically set to the element density.

Internally, LayerProbe calculates the mass thickness of each layer. The mass thickness is defined as the product of density of the material and its thickness. Hence, to determine the thickness, the density of a layer must be known and be entered into the model. Therefore it is important to realise that the value of the density directly affects the measured thickness.

#### Defining the thickness of each layer

• Enter the thickness of each layer in nm.

#### **Simulated Spectrum conditions**

■ **Specimen tilt, Elevation and Azimuth**: Select a spectrum from the data tree which acts as the template spectrum. Values for the tilt, elevation and azimuth angles are read directly from this spectrum. In addition, the beam calibration is also read. The values for the tilt, elevation and azimuth angles displayed are of the template spectrum and are not editable.

- **Livetime (s)**: The Livetime is user selectable. When you select a spectrum in the data tree, the Livetime for the simulated spectrum is set to match to that of the selected spectrum.
- **Beam current factor**: The beam current used for simulation is equal to the beam current factor times the beam current used for beam measurement when calibrating. This is user selectable. When you select a spectrum from the data tree, the beam current factor is reset to one. One is the value if the beam current is the same as that used for the beam calibration for the template spectrum.
- Calculated Count Rate (kcps): This is the number of counts in the simulated spectrum divided by the Livetime. The calculated count rate for the simulated spectrum is not editable. However it is useful to see how the calculated count rate changes when the Beam current factor or Accelerating Voltage is changed.
- **Number of Channels**: The numbers of channels for the simulated spectrum are user selectable: 1024, 2048 and 4096.
- **Energy Range (keV)**: The energy range of the simulated spectrum used or selected is user selectable from 0-10, 0-20 and 0-40 keV. The energy range to use will depend on the elements present in the specimen and the X-ray lines to use for analysis.
- Accelerating Voltage (kV): The accelerating voltage of a simulated spectrum is user selectable in the range from 2.0 to 30.0 kV. On selection of a template spectrum in the data tree, the accelerating voltage for simulation is initially set to match that of the template spectrum.



# **Set Up Solver**



The purpose of this step is to investigate if a particular problem is soluble and to suggest experimental and analysis conditions to use when calculating unknown layer thicknesses and/or concentrations. There are various parts to this step, all of which are necessary to complete before you can calculate the Solubility:

.....

- 1. Make sure that the model of the structure you wish to characterize is set up correctly (see the 'Describe Model' step for details).
- 2. Select a spectrum from the data tree to use as a template spectrum. The template spectrum provides both microscope-detector geometry information and detector characterization information.
- 3. Define a set of accelerating voltages to use for the solubility calculation as well as the target count rate and spectrum Livetime. Press Update.
- 4. Decide whether to disallow any particular X-ray lines for the solubility calculation by deselecting them under the relevant accelerating voltage.
- 5. Press 'Calculate Solubility' to perform the calculation.

When you press the 'Calculate Solubility' button, the inputs from the model are taken into consideration and are used to work out under which conditions the problem is soluble and those where it is insoluble for a set of predefined accelerating voltages. It then suggests a set of analysis conditions to use:

- Accelerating voltage (kV)
- The X-ray lines to use for each element in the specimen
- The acquisition range (0 10 keV, 0 20 keV or 0 40 keV)

In more detail, the calculation takes into account how the intensity of the X-ray lines vary with kV and it will calculate the relative statistical variation in all the determined unknowns. At different kVs, different lines (K, L, M) are excited, the electron beam may not penetrate all the layers and some X-ray lines may be absorbed on their way out to the detector. The 'Calculate Solubility' procedure will try to find a kV where the problem is both soluble and invertible and where best precision is achieved for the unknowns to be determined. At this kV, the best X-ray lines to use for the analysis will be shown. Note that you may override the default choice of allowed lines that the 'Calculate Solubility' algorithm uses for each element in the 'Confirm Analysis' step under the 'Analysis Lines' section. However, in most cases, this intervention will not be necessary. The acquisition conditions that the 'Calculate Solubility' procedure suggests are indicated by the radio button at the base of the 'Solubility calculation' results section.

In summary, for each predefined kV, the calculation works out whether the problem you have setup to calculate is solvable and invertible. If these criteria are met, Layer Probe then calculates statistics which then enable the software to suggest the best kV to use based on the statistics.



## LayerProbe Settings

There are three tabs in the LayerProbe Settings window:

Quant Setup, Model and Analysis Lines.

#### **Quant Setup**

The Quant Setup tab allows you to select the deconvolution elements, the threshold for quantitative results, the correction for detector window artefacts and select quantitative standardizations.

#### • Deconvolution elements

Deconvolution elements may be used to select elements present in the spectrum that should not be quantified, but whose influence needs to be accounted for when processing the spectral data.

If you wish to deconvolve elements from a spectrum, select the required element from the drop down list and press 'Add element'. Further elements can be added or removed using Add element or Remove element respectively.

#### • Threshold Quantitative Results

To enable 'Threshold Quantitative Results', check this option in the Quant Setup tab.

If thresholding is enabled, any X-ray line whose k-ratio value is below the minimum value allowed by the Stratagem engine is assigned the minimum allowed k ratio value in order for the calculation to proceed without an error message.

#### Correct for window artefacts

To enable Correct for window artefacts, check this option in the Quant Setup tab. Checking this option ensures that the contribution to the spectral peaks from the detector window material is removed prior to quantification of the spectrum.

#### Quantitative Standardizations

The system is supplied with factory standardizations. To use your own standards for quantitative analysis, you first need to acquire spectra from standards and perform at least one standardization using the Standardize step on the Optimize navigator. When you have done this your user file will be available to select under Quant standardizations in the EDS LayerProbe settings. If the Factory button is selected, only Factory standardizations can be used for the calculation of quantitative results.

#### Model

In the Model tab, you can view the model. Note that it is a read-only screen.

#### **Analysis Lines**

In the Analysis Lines tab, you can view the analysis lines selected for quantification in the Confirm Analysis step. It also gives you the details of the lines selected as part of the suggested analysis conditions in the Setup Solver step. Note that this is a read only screen.



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Optimize	
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In Optimize there is only one step, Calibrate.

There is one calibration routine, Energy Calibration.

Calibrate on next page

## **Calibrate**



There is Energy Calibration routine available from the Calibrate screen.

#### **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.

See details on how to perform the routine below:

# Why do we need to perform Energy Calibration?

- Ambient temperature changes will alter the gain of the system and this will affect where peaks appear in the spectrum. The exact peak positions and the resolution of the system are needed to precisely identify individual peak components in the spectrum.
- If peaks overlap, the relative sizes of individual peaks can only be calculated accurately if the width and position of each peak is accurately known. By measuring the position of one known peak, the system can be optimized to determine the position of all other peaks.

# How often should I perform Energy Calibration?

The electronics used is carefully designed to provide good temperature stability. Since a change of 10°C produces only a 1 eV shift in peak position, most routine analysis can be performed without re-optimizing peak position. However, if you need the software to resolve very closely overlapped peaks, you should perform Energy Calibration and re-optimize if the



ambient temperature changes by a few degrees. With a good laboratory temperature control you may not need to optimize for many months.

## **How to perform Energy Calibration**

Energy Calibration requires the acquisition of a high quality spectrum from a suitable element from which details of the spectrometer gain are calculated and stored.

You can use an element as an energy calibration standard as long as the calibration peak is not overlapped by other peaks. There should not be any peaks within 100 eV of the calibration peak.

To perform Energy Calibration follow the steps:

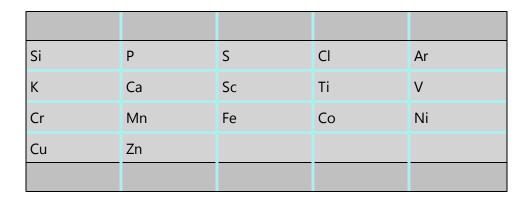
- Select Energy Calibration from the Calibration Routine drop-down list.
- Select an element from the Calibration Element drop-down list.
- Get the element standard in the field of view of the microscope. Adjust the working distance to the recommended value and the beam current to achieve an optimum count rate.
- Press to start acquisition of the calibration spectrum. The current settings will be used to acquire the spectrum. A window will be painted across the peaks of the element X-ray line series.
- A progress bar near the top of the Calibrate window displays the estimated time for the completion of calibration spectrum acquisition.
- On completion of spectrum acquisition, a message is displayed asking you if you
  wish to perform the Energy Calibration. Press Yes if you wish to perform the Energy
  Calibration.

Note that the details of the Energy Calibration can be found in the Spectrum Details dialog in the Calculate Composition step when a spectrum has been quantified.

## **Calibration Element**

Select the element that you wish to use for Energy calibration from the drop down list.

The elements available for Energy Calibration are tabulated below:



X-ray peaks often involve multiple lines and in order to achieve accurate calibration, a large peak with well-known line energies and intensities is required.

Therefore, for this reason we recommend that for energy calibration a pure element is used. If a pure element is not available compounds that have K lines that are not overlapped may be used instead. However, there may be some loss of accuracy because the line energy in a compound can sometimes be up to 2eV different from that in the pure element.



## Analyzer - Guided

There are two modes of operation in the Analyzer application, Guided and Custom.

In the Guided mode, the Analyzer navigator has five following steps:

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## **Describe Specimen**



In this step there are two tabs, Summary and Pre-defined Elements.

#### **Summary**

In the Summary view you can write notes on the Project and the Specimen present in the Project. (For convenience you can also copy images/diagrams and text from other documents/emails and paste into these windows). Notes are saved with the Project and you are allowed to edit notes in any step of the Navigator. It helps to capture the important information during the analysis. Click with the right mouse button on the Project or Specimen in the Data Tree and then select Edit Notes to write/modify the relevant notes.

You can add new Specimens to the current Project by pressing the New Specimen button:



Click on the Specimen in the Project Overview dialog. This action displays the 'Specimen Notes for Specimen 1' text box. Here you are provided with the text formatting tools. You are allowed to write notes about each Specimen and save them.

#### **Pre-defined Elements**

If you know what elements are present in your Specimen and you only want to see peak labels or X-ray maps for those elements, then you can select them in the 'Pre-defined Elements' tab.

Press the Pre-defined Elements tab to access the periodic table:

If you wish to enable the AutoID option check the 'Perform AutoID During Acquisition' option.





Double -click on the element symbols that you wish to include in the analysis. All the included elements will be marked with the green color key in the periodic table. To save the Pre-

defined Elements in the current User Profile press . It means when you load the User Profile next time these elements will be included in the analysis.

If you have already created a User Profile with the Pre-defined Elements in the User Profile dialog press Load from Profile.

Pressing will deselect the Pre-defined Elements from the periodic table and they will not be included in the current analysis.

The peaks for the Pre-defined Elements if included in the analysis are labeled in the Acquire Spectra step. MiniQuant will display the quant results for these elements as Wt% or a bar chart.

The Pre-defined Elements will be marked as Pre-defined in the Confirm Elements list box in the Confirm Elements Step. There may be other elements in the Specimen which are identified by AutoID routine if the 'Perform AutoID during acquisition' option has been checked in the User Profile dialog.

### Тiр

Right click on the Project or Specimen in the Data Tree and select Edit Notes to write or edit notes in any step of the Navigator.

#### See also:

Element Lists on page 191

Data Tree on page 85

Mini View on page 93

Step Notes on page 94



## **Acquire Spectra**

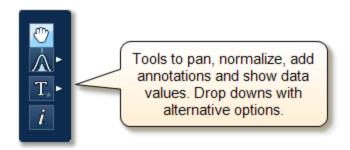


Analyzer is microscope centric application. You can acquire a spectrum from a point or an area on the specimen either generated by a static probe or in STEM mode.

There is an acquisition toolbar near the top of the workspace. It has controls for starting and stopping the spectrum acquisition.

There is a Settings cog for selecting the acquisition parameters. For details see Acquire Spectra - Settings below.

The toolbar located on the left side of the workspace has various tools for spectrum manipulation and annotation as shown in the screen shot below:



For description of each tool, see Acquire Spectra - Toolbar on page 316

The Compare Spectra & MiniQuant Results on page 318 option is available in the top right corner of the Spectrum viewer. You can compare the current spectrum with any other spectrum from an opened Project on the Data Tree. Instant MiniQuant results can be viewed in a table or a bar chart.

A number of useful shortcut menus are available as right mouse click in the spectrum viewer. For details see Context Menus - Spectrum Viewer on page 321.

## **Acquire Spectra - Settings**

The settings are described in detail below:

### **Energy Range (keV)**

Select a spectrum energy range from the available options of Auto, 0-10, 0-20 or 0-40 keV from the Energy Range drop down list.

An appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited.

In the Auto mode, the system checks for the accelerating voltage set on the microscope and selects a suitable energy range in the software.

#### **Number of Channels**

Select number of channels from the drop down list of Auto, 1024, 2048 or 4096 (4K) with which you wish to display the spectrum. The number of eV/channel will depend on both the energy range and the number of channels you select:

Energy Range (keV)	Number of Channels	eV/channel
0-10	4096	2.5
0-10	2048	5
0-10	1024	10
0-20	4096	5
0-20	2048	10
0-20	1024	20
0-40	4096	10
0-40	2048	20
0-40	1024	40

In the Auto mode, the system checks for the energy range selected and sets the appropriate number of channels.

#### Note

The Energy Calibration routine is performed for all process times and for all available energy ranges and number of channels. It means if you change any of these settings soon after you had performed the Energy Calibration you do not need to re-optimize the system.

#### **Process Time**

Select the Process Time from the drop-down list of Process Times, Default and 1 to 6. The Process time is the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process time, the lower the noise. If noise is minimized, the resolution of the peak displayed in the spectrum is improved, in other words, the



peak is narrower and it becomes easier to separate or resolve, from another peak that may be close by in energy.

If Default is selected, the Process Time is automatically set to a suitable value.

There is a trade off between the Process time that is used, and the speed at which data can be acquired into the X-ray spectrum. Process time 1 is the shortest, and as such, gives the highest X-ray acquisition rates, but at some cost to resolution. Process time 6 is the longest, and gives the highest resolution, but at some cost to maximum acquisition rate. The longer the Process time, the slower data can be acquired, i.e. the higher the system Deadtime will be for a given input count rate. (The input rate is not affected by the pulse processor).

#### Which Process Time should I use?

When you start your application first time, the Process Time is set to Default. This is a suitable choice for many routine applications where you are looking for good resolution of peaks and fast acquisition.

For the first look at a specimen you should use a long process time (5 or 6) to start with in order that you do not miss any detail in your spectrum. For example, when identifying peaks particularly those closely spaced and overlapping, it is important to get good peak separation. Good resolution is also important for looking at a series of lines that are very closely spaced, like an L series and process times 4 to 6 should be chosen. Common overlaps include the Mo L and the S K lines.

If there are no closely spaced peaks then you can afford to use a shorter Process Time such as 1-3 which will enable you to increase the acquisition rate by increasing the beam current. A compromise between acquisition speed and resolution should be found if there are peak overlaps.

When acquiring SmartMap data you should choose your Process Time carefully.

- 1. You may have been working on a Specimen in either Analyzer or Point & ID where you have setup your acquisition parameters to optimize your quantitative analysis. If you now wish to acquire SmartMap data and you think you may wish to reconstruct spectra from your SmartMap data and then quantify these spectra, you should maintain these acquisition parameters. This means that you may have to acquire data with a long Process Time to maximize resolution but limit the maximum acquisition rate.
- 2. You may have been working in either Analyzer or Point & ID and you want to view the distribution of elements whose main peaks do not overlap as a map or a linescan. In this case you should use a shorter Process Time which will mean that you can work with higher acquisition rates and shorter acquisition times. The choice of Process Time will very much depend on your sample and what you wish to do with your SmartMap data once it has been acquired.
- 3. If you have started your Project in Map and you are analyzing an unknown sample, we recommend that you use a long Process Time in order that you do not miss any detail in your spectrum. However if you only wish to map certain elements whose

main lines do not overlap, you can afford to shorten the Process Time and increase the acquisition rate by increasing the beam current.

#### **Acquisition Mode**

There are three options to terminate the acquisition, Auto, Live Time and Counts.

If Auto mode is selected, acquisition continues until enough counts are collected in the spectrum for quantification.

You can choose to terminate acquisition at the end of a preset Live Time. Enter the required time in seconds into the text box. This is the time for which the system is processing counts into the spectrum. The live time clock runs slower than the real time clock so that the acquisition for '100' live seconds takes longer than 100 real seconds. This time is extended to compensate for the output rate being less than the input rate by the degree of Deadtime.

You can choose to terminate acquisition at the end of a preset number of counts. Enter the value in the Count Limit text box. The default value is 500,000.

#### **Pulse Pile Up Correction**

Check Pulse Pileup Correction check box if you wish to automatically correct the spectrum for pulse pileup peaks. Uncheck the box if you wish to disable this correction.

Pileup peaks can occur when a second pulse arrives and triggers the measuring system during the time required to process a previous pulse. When this happens, neither pulse will appear in its correct position. The result being a peak at a higher energy equivalent to the sum of the energy of the two photons.

The largest pileup peaks will be seen at twice the energy of the main peaks - e.g. Fe Ka pile up peaks will be seen around 12.8 keV.

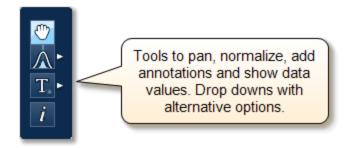
#### Notes

The pileup correction algorithm assumes that the count rate at every energy is constant throughout the analysis period. Therefore, the correction works best when analysis is performed on single pixels, points or areas of same composition. Bad results may be obtained if the beam is rastered over an area where composition is changing or if a spectrum is reconstructed from a SmartMap over a region where the composition is changing.

## **Acquire Spectra - Toolbar**

The Acquire Spectra screen has a toolbar on the left side of the workspace shown in the screen shot below:





#### Pan

The Pan tool allows to expand the spectrum along the vertical axis and move the spectrum along the horizontal axis. To expand the spectrum along the horizontal axis with Pan tool selected, hold down the Ctrl key while dragging the spectrum with the left mouse.

#### **Normalize Spectra**

You can normalize two spectra over a selected point or a region.

#### **Normalize Spectra (Point)**

- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Point) option from the toolbar. The cursor turns into an up down arrow ( ).
- Double-click in the spectrum to set a normalization point along the X-axis. A window is drawn on either side of this point. Both the spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

#### Normalize Spectra (Region)

- You have the current spectrum in the spectrum viewer.
- Select the second spectrum using the Compare drop down. The second spectrum is overlaid on the current spectrum.
- Select the Normalize Spectra (Region) option from the toolbar. The cursor turns into a crosshair (+).
- Click in the spectrum viewer to select the start point of the energy window. A default window is displayed about this point. Drag the mouse to define your window and then release it. A window will be drawn between the first point and the end point where you release the mouse. Both spectra are scaled along the Y-axis to the average value (usually cps/eV) in the window.

#### **Annotations**

Five tools available to add annotations on the current spectrum and the image are Caliper, Angle, Text, Rectangle and Ellipse. Select the tool by clicking on it and then click on the spectrum/image to add annotation. For example to add text select the Text annotation tool, click

on the spectrum where you wish to enter the text and then start typing the text. To delete annotation double click on it to select it and then press the Delete key on the keyboard.

#### **Show Data Values**

With this tool you can view the Energy (keV) and counts in any channel of the spectrum. Simply select the Show Data Values tool from the toolbar and then hover on spectrum. The values will be displayed as you move from channel to channel.

#### **Acquisition and Settings Toolbar**

Near the top of the Acquire Spectra window, there are buttons for starting and stopping acquisition as shown below:



Press the Start button to acquire a spectrum. There is a Settings cog in the toolbar. For details of settings see Acquire Spectra - Settings on page 313.

## **Compare Spectra & MiniQuant Results**

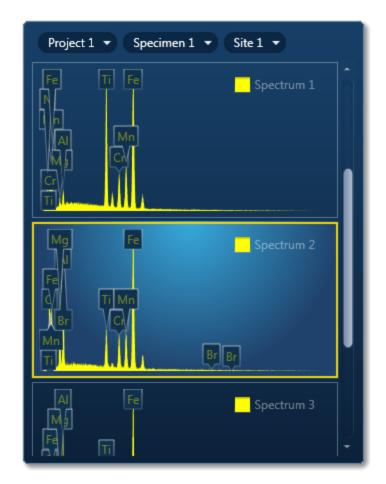
Real time Compare and instant MiniQuant options are available in the Acquire Spectra, Confirm Elements and Calculate Composition (Comparison of Results - Two Spectra template) steps. User can see results without having to move away from the acquisition mode. Using these options you can:

- See your results during analysis.
- Compare your current spectrum to a control spectrum during acquisition.
- View MiniQuant results in a table or a bar chart.

Click in the top right corner of the Spectrum Viewer in Acquire Spectra, Confirm Elements or the Calculate Composition window to access the Compare & MiniQuant options:



In the above example, Spectrum 1 is the current spectrum and Spectrum 2 is the comparison spectrum. You can select the comparison spectrum from a Project currently available in the Data Tree. It can be from any Project, any Specimen and any Site of Interest currently available in the Data tree. To choose the comparison spectrum click on the down arrow (Spectrum 2 in the above example). Spectra available in the current Project, Specimen and Site of Interest are displayed as below:



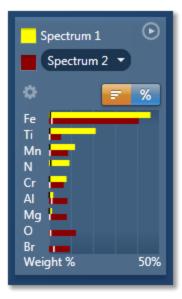
Click on the spectrum in the display to select it for comparison. The selected spectrum will be overlaid as a line spectrum over the current spectrum. The MiniQuant results are displayed in a table as shown in the example below:



The results are displayed as wt% (weight%).

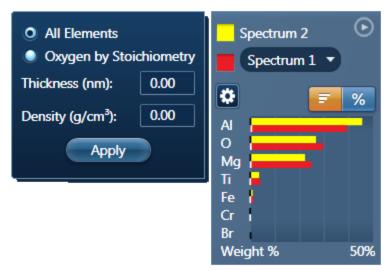
The statistical error is displayed as  $\sigma$  (weight% sigma) for the calculated wt%. It is the overall confidence figure for the analysis. You can use sigma to assess the results especially when an element is present at low concentration. For example, if an element concentration is 0.2 wt% and the  $\sigma$  is 0.12 wt%, the element might be detected at a statistically significant level if the acquisition time for the spectrum is extended. If the  $\sigma$  is 0.4 wt%, it is pointless to extend the acquisition time and it is safe to assume that the element if present, is at a level above the limit of detection for this technique.

Press to display the results in a chart:



The sigma values are displayed as black or white vertical bands across the bars in the chart results as shown in the example above. In this case the full scale of the bar chart is 50%.

If you wish to change the MiniQuant Settings press



Make your selection by clicking on the radio button and then press the Apply button. The results will be updated immediately .



#### Note

The Quant Settings in the MiniQuant and Calculate Composition are the same. Updating one updates the other and vice versa.

## **Context Menus - Spectrum Viewer**

A number of useful shortcut menus available as right mouse click in the spectrum viewer are shown in the table below:

Context Menu Item		
Reset Scales		
Export	Save As	
	EMSA	
	Сору	
	Print	
	Email	
	Settings	
Peak Labels	Show	
	Reset Posi- tions	
Annotations	Show	
	Select All	
	Style	
	Delete	
X Axis	Show	

Context		
Menu Item		
	Locked This is a useful option if you are looking for a particular energy range. You do not want the energy scale to change when you load a new spectrum in the viewer	
	the viewer for examination or for reporting. Locking the X Axis will maintain the horizontal energy scale. If you do not lock it, it will	
	change to the full scale when you load a different spectrum. The default energy range is full scale. Adjust	



Context Menu Item		
Y Axis	Show	
	Locked	

Context		
Menu Item		
	Units You have the choice between cps/eV(per channel) and Counts for the units along the Y-Axis of the spectrum. For easy comparison of spectra, cps/eV is an ideal choice because there is very small variation in the range. You do not need to normalize prior to comparison of spectra using different energy ranges, number of channels and live times. This is vital when comparing a stored spectrum with one that is still	cps/eV



Context Menu Item		
	acquiring.	
		Counts
	Scale	Linear
		Logarithmic

Context Menu Item	
Normalize	
Normalize is	
a useful func-	
tion for com-	
paring two	
spectra	
acquired	
using dif-	
ferent input	
X-ray count	
rates for	
example	
spectra	
acquired	
with two dif-	
ferent beam	
currents. Nor-	
malize is	
available	
from the	
toolbar on	
the left of	
the screen in	
Acquire	
Spectra, Con-	
firm Ele-	
ments,	
Acquire &	
Confirm and	
Compare	
Spectra	
steps of	
Point & ID	
and	
Analyzer. For	
details of	
how to nor-	
malize using	
a point or a	



Context	
Menu Item	
region on a	
spectrum	
see the Com-	
pare Spectra	
help topic.	
Note that	
Normalize	
can be	
switched on	
and off from	
the shortcut	
menu avail-	
able as right	
mouse click	
in the spec-	
trum viewer.	
If a point or	
region has	
already been	
defined on	
the spec-	
trum, switch-	
ing	
Normalize	
on or off will	
maintain the	
point or	
region. If a	
point or	
region has	
not been	
defined	
already,	
switching it	
on will nor-	
malize the	
spectra to	
the peak at	
zero.	

Context	
Menu Item	
Smooth This	
is useful	
when com-	
paring spec-	
tra where	
small dif-	
ferences	
may be	
obscured by	
statistical	
scatter. The	
smooth func-	
tion applies	
an energy-	
dependent	
filter to the	
spectrum.	
This has the	
effect of	
slightly broadening	
the peaks	
and also fil-	
tering out	
the rapid	
fluctuations	
due to sta-	
tistics. Sta-	
tistical	
fluctuations	
can some-	
times appear	
like a real	
peak. When	
it is difficult	
to decide	
whether a	
peak is	
present or	
not, the	



Context Menu Item		
smooth function substantially reduces the statistical fluctuation so that any real peak becomes more visible.		
Noise Peak	Include in Scaling	Shows the noise peak, and includes its value when the context menu option "Reset Scales" is used.
	Exclude from Scal- ing	Shows the noise peak, but excludes its value when the context menu option "Reset Scales" is used.
	Hide	Hides the noise peak.
Details		

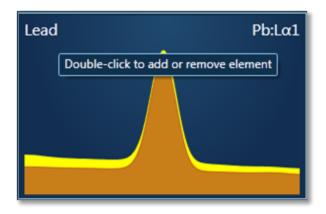
## **Confirm Elements**



This step has been designed to help you confirm the elements that have been identified by AutoID in your spectrum. These elements are then used to create a confirmed elements list for qualitative and quantitative analyses. Extensive tools including Element Series Markers, Overlays, Element Profiles and Show Candidate Elements are available here to assist you in confirming elements manually.

#### How to confirm elements:

Start with the largest peaks. Press the question mark icon to select the Show Candidate Elements tool from the tool bar on the left hand side of the interface, then double click on a peak in the spectrum viewer. The candidate elements are displayed in a stacked spectra view on the right hand side of the window (you can double click on any of these elements to add or remove it from the confirm elements list).



You can control what overlays you see in the Spectrum viewer via the 'Confirm Elements Settings'. These overlays can be very useful in helping you to interrogate complex spectra.





 Press Include/Exclude once you are satisfied with the identification of each element to build your list of the confirmed elements.

#### See Also:

Confirm Elements - Settings

Confirm Elements - Tools on page 174

Element Lists on page 191

Peak Labels on page 158

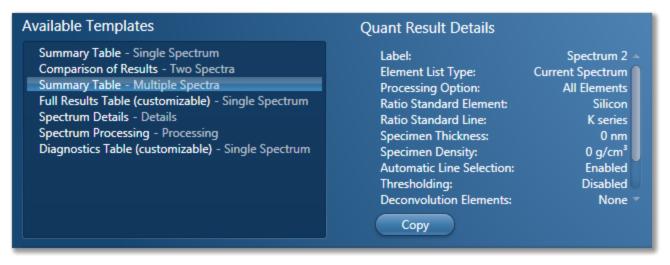
Compare Spectra & MiniQuant Results on page 318

## **Calculate Composition**



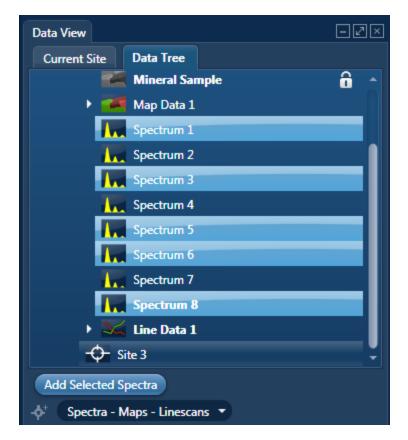
In this step you can view quant results in more detail using any of the 'Available Templates'.

To view result select the template that you wish to use:



- If you want to see a comprehensive set of results from a single spectrum, then choose the 'Full Results Table (customizable) Single Spectrum' template and whichever spectrum is highlighted in the Data Tree will have its results shown in this template.
- To populate a multiple spectra template, hold the Ctrl key down while choosing spectra on the Data Tree and then press the 'Add Selected Spectra' button at the bottom of the Data Tree window.





- To compare quant results from two spectra, select 'Comparison of Results Two Spectra' template. Then select the comparison spectrum from the Compare option in the 'Mini Quant and Compare' option. The compare spectrum will be overlaid on the current spectrum in the Spectrum Viewer. The quant results will be displayed in the table below.
- If you wish to change the Quant Settings press the Settings button to display the Quant Settings dialog. Make the changes and press . The recalculated results will be displayed. Alternatively make the changes to the quant settings and press . Press the Close button to close the Quant Settings dialog.
- Then press the Requantify button to display the recalculated results.

#### **Quant Results Details**

You can see the settings used for calculating the composition in the Quant Results Details list box:

Parameter	Description
Label (Spectrum Label)	E.g., Spectrum 1
Element List Type	Current Spectrum, Fixed List or Combined List

Parameter	Description
Processing Options	All Elements or Oxygen by Stoichiometry
Ratio Standard Element	Silicon
Ratio Standard Line	K Series
Specimen Thickness	x nm
Specimen Density	x g/cm <sup>3</sup>
Automatic Line Selection	Enabled or Disabled
Thresholding	Enabled or Disabled
Deconvolution Elements	None/Selected
Pulse Pile Up Correction	Enabled/Disabled
Detector File	Indicates file that has been used to characterize detector
Efficiency	Calculated/File based

#### **Quant Results View**

The information displayed in the Quant Results View depends on which template has been selected. You can view Spectrum Details, Spectrum Processing and Diagnostics table in addition to quant results.

#### See Also:

**Quant Settings below** 

Element Lists on page 191

Compare Spectra & MiniQuant Results on page 318

## **Quant Settings**

The Quant Settings are described below:

#### **Processing Options**

To make the correct selection, a little knowledge of the specimen is required. For example, can all elements in the specimen be detected and analyzed, or are you analyzing a mineral where it is more usual to calculate the oxygen present?

#### All Elements



This option is used when processing spectra from specimens in which all elements yield X-rays which can be readily detected e.g., steels, alloys and other materials with insignificant amounts of elements lighter than sodium.

#### Oxygen by Stoichiometry

Use this option if you want the concentration of oxygen to be calculated assuming that it is bound by predefined stoichiometry to all the other analyzable elements. The stoichiometry is defined by the valency of the oxygen ions and the valencies of other measured elements:

#### Number of Ions

Enter the number of oxygen ions that are combined stoichiometrically to the other elements. The calculations are based on the number of oxygen ions and how many atoms there are in each unit cell.

#### Valency

When 'Oxygen by Stoichiometry' is selected, the option for choosing the valency for each analyzable element becomes available. The most common value for the valency of the element is displayed when you click on an element symbol on the periodic table. To use a different value, enter the new value in the Valency text box.

#### **Element List**

You can select a different type of element list depending on how you want your spectra/spectrum to be quantified:

#### Current Spectrum

If this option is selected, each spectrum will be quantified using the elements confirmed in the Confirm Elements step for the current spectrum.

#### Fixed List

Select this option if you wish to define a list of elements with which to quantify your spectra. For example you may only wish to quantify your spectra using certain elements if you are constantly quantifying similar spectra. Define your Fixed List using the Periodic Table. To include an element in the list, click on the element symbol on the Periodic Table and press



or double-click on the element symbol.

#### • Fixed List and Current Spectrum

Select this option when you know what elements there are in your specimen and you also wish to include any other element that may be present. You define your Fixed List using the Periodic Table as described in bullet number 2 above. The Confirmed Elements List is from the Current Spectrum. This list includes all the elements identified by AutoID and any other elements that may have been added to the Confirmed Element list manually. What elements are quantified when you select 'Fixed List and Current Spectrum' are shown in the examples below in a table:

	Fixed List	<b>Current Spectrum</b>	Combined List
Spectrum 1	Si, O	Si, O, Al, Ca	Si, O, Al, Ca
Spectrum 2	Si, O	B, N	Si, O, B, N

#### **Quant Element List Details**

From this tab you can view the details of each element in the list. The default setting is that the X-ray lines to be used for Quant are automatically selected. You can manually select the X-ray line for each element if you un-check the 'Automatic selection of line for all elements' option. You can check the 'Fixed weight %' option and enter the value.

#### **Deconvolution Elements**

Deconvolution elements may be used to select elements present in the spectrum that should not be quantified, but whose influence needs to be accounted for when processing the spectral data. For example elements present in an oxidation layer, or in a supporting grid.

If you wish to deconvolve elements from a spectrum, select the required element from the drop down list and press 'Add element'. Further elements can be added or removed using Add element or Remove element respectively.

Selecting an element for deconvolution means the peaks will automatically be deconvolved from the spectrum but the element will normally not be quantified. The deconvolution element will only be quantified if it is oxygen and its composition is calculated by stoichiometry.

#### **Ratio Standard**

A proportionality factor, known as the k factor is used to relate the characteristic X-ray intensities  $I_A/I_B$  in a thin sample to the actual concentrations in wt% in the form:

$$C_A/C_B = K_{AB}I_A/I_B$$

where I = Intensity, C = concentration and K = K factor

#### K factors

The k-factor relates the intensity of two elements. Where more than two elements are to be analyzed, if all ratios are taken with respect to a single element (this is called the ratio standard element), these values can be regarded as elemental sensitivity factors, more usually known as k-factors.

The theoretical k factor value is determined using the X-ray line type (K series, L series, etc) for the ratio standard you select. For a given X-ray line, A, and ratio standard line, R, the k factor  $k_{\Delta R}$  is calculated as follows:

$$k_{AR} = A_A \omega_R Q_B \varepsilon_R / A_R \omega_A Q_A \varepsilon_A$$

where A = atomic weight;  $\omega$  = fluorescent yield; Q = ionisation cross section; and  $\epsilon$  = the efficiency of detector window at that line energy.



Once k factors are known relative to the ratio standard, any other k factors can be calculated using the formula

$$k_{AB} = k_{AR} / k_{BR}$$

You can use any element as you are using theoretically derived k factors. Conventionally, Si is used.

### **Specimen Thickness and Density**

If you want to apply an absorption correction to the calculated concentration, you will need to know the density and thickness of the material. The absorption correction is recorded in the Full results section of the quant results.

Enter the thickness (nm) and density (g/cm<sup>3</sup>) into the spaces provided. These values are then used next time spectra are quantified. To recalculate the current analysis with different values, edit the required value and go to the Calculate Composition step.

### **Threshold Quantitative Results**

Quantitative results are displayed with +/- value which is one sigma (standard deviation) based on counting statistics. Typically, results which are less than 3 sigma have reduced significance and so it may be desirable to set them to zero. Thresholding may be applied so that results below the selected sigmas are set to zero. Thresholding will also ensure that negative insignificant values, which sometimes result from trace element analysis, are set to zero.

To enable 'Threshold Quantitative Results', check this option in the Quant Settings.

The default value for Sigma is 3 which represents 99.7% confidence level.

### **Apply and Save**

If you make a change to the Quant Settings and press aved and the currently selected spectra are quantified. The quant results are updated immediately.

#### Save

If you make a change to the Quant Settings and press saved to be used when you do quantification next time.

### **Compare Spectra**



This step in both the Point & ID and Analyzer Navigators allows you to compare spectra acquired from different sites of interest and specimens from the currently opened projects.

You can compare spectra acquired using different settings for example energy ranges (0-10, 0-20 or 0-40 keV) and number of channels (1024, 2048 or 4096).

Spectra associated with the current Site can be added to the Compare table by holding down the control key and pressing Add Selected Spectra. Note that if you have acquired the spectra in Point & ID, the positions of all the spectra associated with the current Site of Interest are displayed on the image. You can add spectra from any Project, Specimen and Site of Interest from the Data tree into this table.

Select which spectra you wish to compare by selecting them individually from the Data Tree, Press 'Add Selected Spectra'. This will add all the spectra you wish to compare into the 'Compare' table:



If you want to remove any spectra from the table, highlight the spectrum and press

Remove Spectrum

You can choose which of the available spectra you wish all the others to be compared to by selecting your reference spectrum from the compare table and pressing Select Reference Spectrum.

#### Settings

You can change the color of individual spectra by selecting the color from the drop down list in the compare table.

To change the line thickness of individual spectra, select it from the Line Thickness drop down list in the Compare table.

To apply the chosen line thickness globally, select it from the Compare Spectra Settings from near the top of the Compare viewer.

### **Normalize**



Normalize is a useful function for comparing spectra acquired using different input X-ray count rates such as spectra acquired with two different beam currents. Note that you can normalize spectra using a point or a region.

### **Normalize Spectra (Point)**

- Select this option from the toolbar near the top left of the Compare Spectra screen.
- Double-click in the spectrum to set a normalization point along the X-axis.
- A window is drawn on either side of this point. The spectra in the Compare viewer are scaled to the average value of cps/eV (Y-axis) in the window.

### **Normalize Spectra (Region)**

- Select this option from the toolbar near the top left of the Compare Spectra screen.
- Click and drag to set a normalization region along the X-axis.
- The spectra in the Compare viewer are scaled to the average value of cps/eV (Y-axis) in the normalization region selected in the previous step.

#### **Smooth**

The Smooth function is available from the context menus of the spectrum viewer. This is useful when comparing spectra where small differences may be obscured by statistical scatter.

The smooth function applies an energy-dependent filter to the spectrum. This has the effect of slightly broadening the peaks and also filtering out the rapid fluctuations due to statistics. Statistical fluctuations can sometimes appear like a real peak. When it is difficult to decide whether a peak is present or not, the smooth function substantially reduces the statistical fluctuation so that any real peak becomes more visible.

Analyzer -	Custom
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There are two modes of operation in the Analyzer application, Guided and Custom.

In the Custom mode, the Analyzer navigator has three steps.

The Describe Specimen and Compare Spectra are explained in the previous section. The one new step is described below:



## **Acquire and Confirm**



Acquire and Confirm is the main step of the Analyzer Navigator in the Custom mode. Three components are available in the single workspace. The Acquire Spectra component is located in the top half of the workspace, Quant Results in the bottom left and Confirm Elements in the bottom right.

The three components are combined in the Custom mode to give you a single workspace called Acquire and Confirm. It offers the convenience of working in one window without having to move away from it for acquiring a spectrum and obtaining the quant results.

You can choose which component you wish to display in the workspace by pressing the rel-

evant button in the toolbar, Vou can toggle to switch on/off a component from the view.

Press to un-dock a component as a free floating window located in the top right corner of the view. Press to switch it into a full screen view.

To re-dock the free floating window back into the main workspace press .

Each component has identical functionality as its counterpart in the Guided Navigator. To get help on each application follow the links below:

Acquire Spectra on page 313

Confirm Elements on page 330

Calculate Composition on page 355

## Point & ID - Guided

Point & ID is an image centric application that requires the acquisition of an electron image prior to X-ray spectra acquisition. There are two modes of operation, Guided and Custom.

In the Guided mode, the Point & ID navigator has six steps:

Describe Specimen	343
Scan Image	346
Acquire Spectra	350
Confirm Elements.	353
Calculate Composition.	355
Compare Spectra	358



## **Describe Specimen**



In this step there are two tabs, Summary and Pre-defined Elements.

### **Summary**

In the Summary view you can write notes on the Project and the Specimen present in the Project. (For convenience you can also copy images/diagrams and text from other documents/emails and paste into these windows). Notes are saved with the Project and you are allowed to edit notes in any step of the Navigator. It helps to capture the important information during the analysis. Click with the right mouse button on the Project or Specimen in the Data Tree and then select Edit Notes to write/modify the relevant notes.

You can add new Specimens to the current Project by pressing the New Specimen button:



Click on the Specimen in the Project Overview dialog. This action displays the 'Specimen Notes for Specimen 1' text box. Here you are provided with the text formatting tools. You are allowed to write notes about each Specimen and save them.

#### **Pre-defined Elements**

If you know what elements are present in your Specimen and you only want to see peak labels or X-ray maps for those elements, then you can select them in the 'Pre-defined Elements' tab.

Press the Pre-defined Elements tab to access the periodic table:

If you wish to enable the AutoID option check the 'Perform AutoID During Acquisition' option.



Double -click on the element symbols that you wish to include in the analysis. All the included elements will be marked with the green color key in the periodic table. To save the Pre-

defined Elements in the current User Profile press Save to Profile. It means when you load the User Profile next time these elements will be included in the analysis.

If you have already created a User Profile with the Pre-defined Elements in the User Profile dialog press Load from Profile .

Pressing will deselect the Pre-defined Elements from the periodic table and they will not be included in the current analysis.

The peaks for the Pre-defined Elements if included in the analysis are labeled in the Acquire Spectra step. MiniQuant will display the quant results for these elements as Wt% or a bar chart.

The Pre-defined Elements will be marked as Pre-defined in the Confirm Elements list box in the Confirm Elements Step. There may be other elements in the Specimen which are identified by AutoID routine if the 'Perform AutoID during acquisition' option has been checked in the User Profile dialog.



### Тiр

Right click on the Project or Specimen in the Data Tree and select Edit Notes to write or edit notes in any step of the Navigator.

### See also:

Element Lists on page 191

Data Tree on page 85

Mini View on page 93

Step Notes on page 94

### **Scan Image**



In the Scan Image step, you can acquire an electron image into a 'Site'. A 'Site' is like a folder, which contains images and analyses for a particular area on a specimen.

For EDS, if you do not want to collect an image and just want to acquire spectra, you can skip this step and go straight to the Acquire Spectra step.

You can have any number of images in a site. Just ensure that the images you want to keep are padlocked in the data tree to stop them being overwritten, as shown in the screen shot below:



You can toggle between saving or replacing the current image with successive image acquisition.

If your specimen is drifting, click the Settings cog and activate AutoLock.

The Scan Image step has several tools for manipulating and enhancing electron images:

- The acquisition toolbar above the electron image and other nearby controls.
- Scan Image toolbar (a vertical toolbar on the left) for manipulating and annotating the image.
- If a forward-scatter electron detector (FSD) is fitted, extra controls are available for combining the signals from each diode into a mixed image.

### Acquisition toolbar and other nearby controls

The acquisition toolbar, above the electron image and below the Navigator, has buttons for starting and stopping the image acquisition, the Settings cog for selecting the image acquisition parameters and a button to link/unlink images for manipulation.





Control	Description
(FSD only)	Displays up to three combinations of mixed image and individual images. Your selections are retained in your user profile.
► START	Click to start the image acquisition according to the current acquisition parameters.
■ STOP	Click to stop image acquisition. Acquisition stops at the end of the current frame. Click again to stop immediately. If you navigate away from the step, acquisition stops at the end of the current frame.
Settings	To change the acquisition parameters, click the Settings cog on the Acquisition Toolbar to display a dialog.
	You can select Image Scan Size, Dwell Time (µs), Input Signal the labels here reflect whatever was set during the installation for example SE, BSE or FSD), either Continuous Scan or Number of Frames and Frame Time (secs).
	If your specimen is drifting, you can activate AutoLock to ensure that any analysis corresponds to the true location on your image.

Control	Description				
Mixing Mode (drop-down list)	Combines signals from the diodes to form a mixed image. Some options are available only if the required diodes are installed and configured.				
(FSD only)	<ul> <li>FSD Z Contrast uses upper and side FSD detector channel images. Select this mode if you are inter- ested in seeing density/atomic Z contrast signal.</li> </ul>				
	<ul> <li>FSD Topo/Orientation uses lower FSD detector chan- nel images. Select this mode if you are interested in seeing orientation contrast signal.</li> </ul>				
	<ul> <li>Custom - include and exclude FSD detector channel images of your choice.</li> </ul>				
	When you select either of the first two modes, the software automatically uses the FSD diode channels associated with that mode. For example, FSD Z mixing mode uses the upper and side FSD diode channels. The FSD Topo/Orientation mixing mode always uses the lower FSD diode channels. Custom mode allows you to mix any FSD diode channels.				
Select Second Image	Selects further images to compare with the electron images, for example, a forward-scattered electron image. This control is available only when the map display is for an image only:				
(FSD only)	Sets the number of images per row in the Standard and Interactive displays.				
Standard •	Offers a choice of image display :				
(FSD only)	<ul> <li>Standard - you can add individual images or remove them from the mixed image.</li> </ul>				
	<ul> <li>Interactive - similar to Standard. You can also change the weighting and color contributed by each image.</li> </ul>				
	<ul> <li>Summary - similar to Interactive and in a more compact display.</li> </ul>				
30	Links images. You can simultaneously manipulate all the layers using the pan or zoom controls.				



Control	Description
	Unlinks images. You can manipulate individual layers using pan or zoom controls.
	Use the mouse wheel to zoom in and out of the image.
	Use these tools (near the bottom right of the screen) to adjust manual and automatic brightness, contrast and color.
Context Menus	Right-click the electron image to display context menus for copying, exporting and printing images.

### See Also:

Scan Image Toolbar on page 424

Scan Image - Settings on page 421

FSD diode controls on page 432

Context Menus - Image Viewer on page 157

Export - Settings on page 132

AutoLock on page 133

## **Acquire Spectra**



In this step you acquire spectra from the current electron image (SE/BSE). You can also reconstruct spectra from a Layered Image or X-ray map if you have already acquired SmartMaps. Real time Compare and instant MiniQuant options are also available here.

You can display the components that you are working on such as image and spectrum using

the controls on this toolbar located in the top right side of the screen. You have choice of displaying image and spectrum as shown in the screen shot below or just an image or a spectrum full screen:





There is an acquisition toolbar near the top of the workspace:



It has controls for starting and stopping the spectrum acquisition. There is a Settings cog for selecting the acquisition parameters. For details see Acquire Spectra - Settings link below.

The toolbar located on the left side of the workspace has various tools for image and spectrum manipulation, enhancement, annotation and area selection. For details see Acquire Spectra - Toolbar topic from the link below.

There are manual and auto brightness, contrast and color controls available for the image view. You can use these controls to enhance and high light certain features in the image.

The Compare Spectra & MiniQuant Results option is available in the top right corner of the Spectrum viewer. You can compare the current spectrum with any other spectrum from an opened Project on the Data Tree. Instant MiniQuant results can be viewed in a table or a bar chart.

#### See also:

How to acquire spectra on page 148

Modes of X-ray spectrum acquisition on page 150

Acquire Spectra - Toolbar on page 151

Acquire Spectra - Settings on page 313

Context Menus - Spectrum Viewer on page 321

Export - Settings on page 132

Peak Labels on page 158

Element Lists on page 191



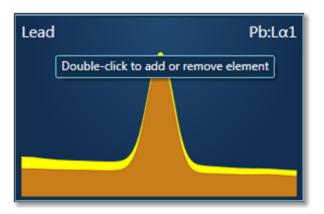
### **Confirm Elements**



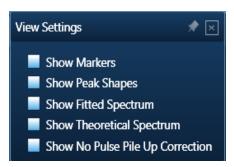
This step has been designed to help you confirm the elements that have been identified by AutoID in your spectrum. These elements are then used to create a confirmed elements list for qualitative and quantitative analyses. Extensive tools including Element Series Markers, Overlays, Element Profiles and Show Candidate Elements are available here to assist you in confirming elements manually.

### How to confirm elements:

Start with the largest peaks. Press the question mark icon to select the Show Candidate Elements tool from the tool bar on the left hand side of the interface, then double click on a peak in the spectrum viewer. The candidate elements are displayed in a stacked spectra view on the right hand side of the window (you can double click on any of these elements to add or remove it from the confirm elements list).



You can control what overlays you see in the Spectrum viewer via the 'Confirm Elements Settings'. These overlays can be very useful in helping you to interrogate complex spectra.



 Press Include/Exclude once you are satisfied with the identification of each element to build your list of the confirmed elements.

### See Also:

Confirm Elements - Settings on page 171

Confirm Elements - Tools on page 174

Element Lists on page 191

Peak Labels on page 158

Compare Spectra & MiniQuant Results on page 195

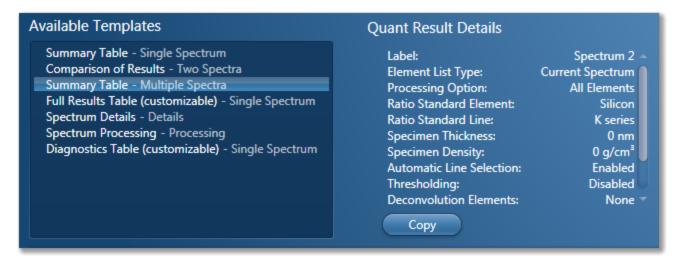


## **Calculate Composition**



In this step you can view quant results in more detail using any of the 'Available Templates'.

To view result select the template that you wish to use:



- If you want to see a comprehensive set of results from a single spectrum, then choose the 'Full Results Table (customizable) - Single Spectrum' template and whichever spectrum is highlighted in the Data Tree will have its results shown in this template.
- To populate a multiple spectra template, hold the Ctrl key down while choosing spectra on the Data Tree and then press the 'Add Selected Spectra' button at the bottom of the Data Tree window.



- To compare quant results from two spectra, select 'Comparison of Results Two Spectra' template. Then select the comparison spectrum from the Compare option in the 'Mini Quant and Compare' option. The compare spectrum will be overlaid on the current spectrum in the Spectrum Viewer. The quant results will be displayed in the table below.
- If you wish to change the Quant Settings press the Settings button to display the Quant Settings dialog. Make the changes and press culated results will be displayed. Alternatively make the changes to the quant settings and press Save.
   Press the Close button to close the Quant Settings dialog.
- Then press the Requantify button to display the recalculated results.

### **Quant Results Details**

You can see the settings used for calculating the composition in the Quant Results Details list box:

Parameter	Description
Label (Spectrum Label)	E.g., Spectrum 1
Element List Type	Current Spectrum, Fixed List or Combined List



Parameter	Description
Processing Options	All Elements or Oxygen by Stoichiometry
Ratio Standard Element	Silicon
Ratio Standard Line	K Series
Specimen Thickness	x nm
Specimen Density	x g/cm <sup>3</sup>
Automatic Line Selection	Enabled or Disabled
Thresholding	Enabled or Disabled
Deconvolution Elements	None/Selected
Pulse Pile Up Correction	Enabled/Disabled
Detector File	Indicates file that has been used to characterize detector
Efficiency	Calculated/File based

### **Quant Results View**

The information displayed in the Quant Results View depends on which template has been selected. You can view Spectrum Details, Spectrum Processing and Diagnostics table in addition to quant results.

### See Also:

Quant Settings on page 334

Element Lists on page 191

Compare Spectra & MiniQuant Results on page 318

### **Compare Spectra**



This step in both the Point & ID and Analyzer Navigators allows you to compare spectra acquired from different sites of interest and specimens from the currently opened projects.

You can compare spectra acquired using different settings for example energy ranges (0-10, 0-20 or 0-40 keV) and number of channels (1024, 2048 or 4096).

Spectra associated with the current Site can be added to the Compare table by holding down the control key and pressing Add Selected Spectra. Note that if you have acquired the spectra in Point & ID, the positions of all the spectra associated with the current Site of Interest are displayed on the image. You can add spectra from any Project, Specimen and Site of Interest from the Data tree into this table.

Select which spectra you wish to compare by selecting them individually from the Data Tree, Press 'Add Selected Spectra'. This will add all the spectra you wish to compare into the 'Compare' table:



If you want to remove any spectra from the table, highlight the spectrum and press

You can choose which of the available spectra you wish all the others to be compared to by selecting your reference spectrum from the compare table and pressing Select Reference Spectrum.

#### Settings

You can change the color of individual spectra by selecting the color from the drop down list in the compare table.

To change the line thickness of individual spectra, select it from the Line Thickness drop down list in the Compare table.

To apply the chosen line thickness globally, select it from the Compare Spectra Settings from near the top of the Compare viewer.

### Normalize



Normalize is a useful function for comparing spectra acquired using different input X-ray count rates such as spectra acquired with two different beam currents. Note that you can normalize spectra using a point or a region.

### **Normalize Spectra (Point)**

- Select this option from the toolbar near the top left of the Compare Spectra screen.
- Double-click in the spectrum to set a normalization point along the X-axis.
- A window is drawn on either side of this point. The spectra in the Compare viewer are scaled to the average value of cps/eV (Y-axis) in the window.

### **Normalize Spectra (Region)**

- Select this option from the toolbar near the top left of the Compare Spectra screen.
- Click and drag to set a normalization region along the X-axis.
- The spectra in the Compare viewer are scaled to the average value of cps/eV (Y-axis) in the normalization region selected in the previous step.

#### **Smooth**

The Smooth function is available from the context menus of the spectrum viewer. This is useful when comparing spectra where small differences may be obscured by statistical scatter.

The smooth function applies an energy-dependent filter to the spectrum. This has the effect of slightly broadening the peaks and also filtering out the rapid fluctuations due to statistics. Statistical fluctuations can sometimes appear like a real peak. When it is difficult to decide whether a peak is present or not, the smooth function substantially reduces the statistical fluctuation so that any real peak becomes more visible.

Point & ID - Cu	stom	1
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Point & ID is an image centric application that requires the acquisition of an electron image prior to X-ray spectra acquisition. There are two modes of operation, Guided and Custom.

In the Custom mode, the Point & ID navigator has three steps.

Describe Specimen and Compare Spectra are described in the earlier sections. The new step is described here.



### **Acquire and Confirm**



In Acquire and Confirm step, four operations are combined into one window. Acquire and Confirm is the main step of the Point & ID Navigator in the Custom mode. It is aimed for users who do not require any guidance during their analyses. The workspace is divided into four quadrants. Each quadrant represents an application. For example, Scan Image is located in the top left quadrant, Acquire Spectra in the top right quadrant, Quant Results in the bottom left quadrant and Confirm Elements in the bottom right quadrant.

Press the relevant button in the toolbar, from the view in any quadrant.

Press to un-dock a quadrant view into a free floating window located in the top right corner of the view. Press to switch it into a full screen view.

To re-dock the free floating window back into the main application window press

Each application has identical functionality as its counterpart in the Guided Navigator. To get help on each application follow the links below:

Scan Image on page 418

Acquire Spectra on page 313

Confirm Elements on page 330

# Map - Guided

In the Guided mode, the Map navigator has the four steps.

The Describe Specimen and Scan Image steps are covered in the earlier section. The steps which are unique to this navigator are described here.

Acquire Map Data	363
Construct Maps.	370
How binning affects the quality of your data	373



### **Acquire Map Data**



In this step, you can acquire X-ray maps from the full frame or selected regions of the specimen. The maps show the spatial distribution of all elements in the specimen. The results can be displayed as a Layered Image, where colors for each element are mixed together and overlaid on the electron image, or as individual maps. Spectra from selected regions can be reconstructed during or after data acquisition. Generating a Layered Image or X-ray maps can be a very useful way to find out what is going on in your specimen.

#### EXAMPLE

'I want to know where certain key elements are distributed over a defect. Once I have this information, I can determine what caused the defect and advise my production department.'

### **How to Acquire and Manipulate Maps**

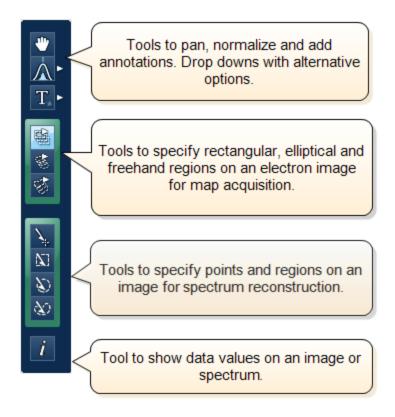
There are two different types of maps that you can acquire, Window Integral Maps or Tru-Maps.

Historically, Window Integral Maps have been the standard mode for X-ray maps. These are ideal when there are no overlapping peaks and you are not looking for trace elements in your specimen.

The second mode of mapping is with TruMaps which are ideal for specimen containing elements with overlapping peaks, and removes false variations due to X-ray background.

You can easily switch between the two modes of mapping during acquisition by pressing the Map or TruMap button above the map display.

- Select the acquisition parameters from the Settings cog on the acquisition toolbar,
   and press
   to acquire map data from the full frame.
- To acquire maps from a region, select a map acquisition tool from Rectangle, Ellipse and Freehand tools available from the toolbar:



- Click on the image and drag with the left mouse to outline a region on the image.
   Maps will be acquired from the scanned region. During TruMap acquisition, a progressing green line is the map acquisition line followed by a yellow map processing line.
- A layered image, element maps and an electron image/s (SE/BSE) are displayed. You can choose how you wish to view your data from Standard, Interactive or Summary view available from the drop-down list.
- Adjust the slider bar to choose the number of maps per row.
- The layered image allows you to see the X-ray maps overlaid on the electron image.
- You can add or remove an X-ray map from the layered Image (combined Electron and X-ray map image) by toggling the Layered Image icon in the top left hand corner of each map.
- If you have lots of maps, it may be useful to minimize some of them pressing the minimize icon in the top right hand corner of each map.
- You may want to delete a map from the analysis completely. In which case press the delete icon in the top right hand corner of each map. This means this element will not be identified automatically (AutoID) and will be excluded from the current analysis. Note: If an element is present in a specimen, deleting or excluding it will affect the TruMap results.



- In the map display settings you can sort maps alphabetically, by atomic number or by maximum map intensity. You can also smooth maps by choosing the smoothing level from the Settings.
- Using the Auto Brightness and Gamma buttons on the bottom right hand corner of the Map display window allows you to change the Brightness/Contrast and Gamma for all the maps. The Auto Brightness button optimizes the maps to give the best Layered Image and the Auto Gamma enables you to see all the map data including background noise.
- You can choose the color for your maps, adjust intensities and decide which maps to add to the Layered image. Alternatively, you can let the software do this auto
  - matically. Pressing the AutoLayer button (which is located in the bottom right hand corner of the Map Display window) will automatically scale and color all the maps and select the best ones to provide an effective color image that delineates regions of different composition. Maps will be auto-brightness corrected and those that show similar structure will be assigned the same color. Maps that are very noisy will be shown in grey. The most significant map for each assigned color will be added to the Layered Image. See AutoLayer on page 222 for detailed information.
- If your data contains a lot of noise, binning might help you to achieve a better Auto-Layer result. Select the binning factor from the drop-down list below the maps.

### **Data from Map Acquisition**

The Data Tree gets populated with the new items as data is being acquired as shown in the screen shot below:



### **Electron image**

It can be a secondary electron image (SE), backscatter electron image (BSE), or a forward-scattered electron image. Appropriate detector hardware needs to be installed. You can also import an image into the Project.

#### **Map Data**

The EDS and EBSD Map data are contained in the Map Data folder. The EDS Data folder contains Map Sum Spectrum and X-ray Element Maps.

### X-ray Element Maps

Two modes of mapping are available, Window Integral Maps and TruMaps. To select the mapping mode, press the Map or TruMap button above the map display.

Standard Window Integral maps (counts in the energy window) are acquired for the element list chosen for analysis. These are raw X-ray maps which are not corrected for background or peak overlaps.

Second mode of mapping is TruMap. You can process the map data as Tru-Maps which are corrected for background and peak overlaps.

### **EDS Layered Image**

It is a composite image generated by overlaying selected X-ray maps on top of the electron image.

### **Viewing and Manipulating Maps**

You can choose how you wish to view your data. Various tools are available to manipulate and view the X-ray maps.

### **Map Size**

You can choose the number of maps per row using the slider bar for displaying maps you wish to view in the Standard and Interactive display mode.



### **Display Modes**



Maps can be viewed in three different display modes available from the drop-down list on the Display toolbar:

- Standard
- Interactive

In the Interactive mode, you can change the color of individual maps from the Hue dropdown list. The Layer Mode can also be chosen from Mix and Overlay modes available from the electron image.

Summary

In the Summary view, you see details of the energy window and X-ray line used for each map in addition to other details such as Layer Name, Source (AutoID or User), Map Color and if it is selected for the Layered Image. See the screen shot below:

Image	Layer Name	Source	Map Color		Energy Window (keV)	Line	Visible lay	er
	Fe Kα1	AutoID	300	D	6.32 - 6.49	Κα1	8	<b>T</b>
	Ti Kα1	AutoID	<b>0</b> 0	Ð	4.44 - 4.58	Κα1	\$	<b>T</b>
	Cr Kα1	AutoID	<b>120</b>	Ð	5.34 - 5.49	Κα1	\$	<b>T</b>
	Mn Kα1	AutoID	<b>180</b>	·	5.82 - 5.98	Kα1	\$	<b>T</b>
	Br Lα1_2	AutoID	<b>3</b> 0	·	1.43 - 1.53	Lα1_2	\$	<b>T</b>
	Al Kα1	AutoID	210	·	1.44 - 1.54	Kα1		<b>T</b>
	Mg Kα1_2	AutoID	<b>60</b>	·	1.21 - 1.30	Κα1_2	\$	<b>T</b>
	Ν Κα1_2	AutoID	90	Ð	0.35 - 0.43	Κα1_2		<b>T</b>
	Ο Κα1	AutoID	<b>150</b>	Đ	0.48 - 0.57	Κα1		<b>T</b>
\$17	Electron Image 1		••	Ð			\$	$\overline{\bot}$

### Link/Unlink



to link images for manipulation of all layers using the Pan or Zoom control.

Press to unlink images. You can manipulate individual layers using Pan or Zoom control.

### **Brightness and Contrast**

You can adjust the brightness and contrast of the currently selected image or map. Press



on the Display toolbar to open the Brightness and Contrast dialog.

### **Auto Brightness and Auto Gamma**

Using the Auto brightness and Gamma buttons on the bottom right hand corner of the Map display window allows you to change the Brightness/Contrast and Gamma for all the maps. The Auto Brightness button optimizes the maps to give the best Layered Image and the Auto Gamma enables you to see all the map data including background noise.

### **Element Maps View - Settings**

You can manipulate and view the data by using various parameters available in the Settings:



### **Sort Order**

There are three different ways of sorting maps:

Alphabetically

By atomic number

By maximum intensity in map - sorts on the value of the brightest pixel in cps.

### **Layer Visibility Selection**

You can choose how the visibility of layers selected in the layered image. There are two options: Manual and Automatic. In the Manual mode, you must select which X-ray maps to be included in the layered image.

In the Automatic mode, first N maps (Number of Map that you entered) are selected by the maximum intensity.

### Smoothing Level

The maps may contain a lot of statistical noise if there is not sufficient data. The noise can mask the distribution of elements in the maps. You can filter out some of this noise by



applying Smoothing Level. This operation applies a lowpass filter to an image to smooth the data. If you are using TruMap, it might be more effective to use binning.

### Smoothing Level, 3X3

The lowpass filter uses the following 3x3 kernel:

1/9 1/9 1/9

1/9 1/9 1/9

1/9 1/9 1/9

### Smoothing Level, 5x5

The lowpass filter uses the following 5x5 kernel:

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

1/25 1/25 1/25 1/25 1/25

### **ACB** while acquiring

Check this option if you wish to apply automatic brightness or automatic gamma to maps during acquisition depending on your pre-selection of Auto Brightness or Auto Gamma.

### See also:

Acquire Map Data - Settings on page 211

Context Menu - Map Viewer on page 223

How binning affects the quality of your data on page 373

Layer modes in the interactive map display on page 216

### **Construct Maps**



In this step, you can select which elements to map and which ones to exclude.

You can change the default X-ray line used for Window Integral Maps for any given element. It is also possible to define energy windows whose widths you can specify yourself rather than using the auto width calculation.

Tools are provided to interrogate the map data to confirm the elemental composition of user specified areas of interest. You can navigate to the Confirm Elements step from within the Construct Maps step.

To reduce the effects of noise in the maps, you can apply a binning factor.

#### **Map Details**

Map Details dialog allows you to choose elements you wish to include or exclude for mapping. You may have pre-defined the known elements in your specimen in the Describe Specimen step. You can map these elements by pressing the Pre-defined button in the Map Details dialog. There may be unexpected peaks in the spectrum. You can use AutoID for

selecting elements for mapping. You can access Map Details option from located at the bottom of the workspace:



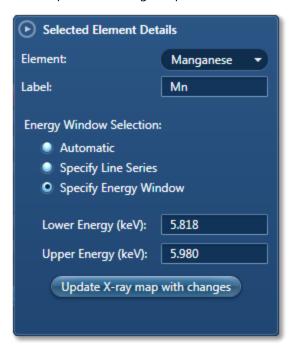


To include an element for mapping either double-click on its symbol on the periodic table or click the Include button. All the elements selected for mapping are green color coded on the periodic table. To exclude an element from being identified by AutoID and excluded from being mapped, click on its symbol on the periodic table to select it and then press the Exclude button. This element will be removed from the analysis. All excluded elements will be red color coded on the periodic table. Note: An excluded element if present in the specimen may affect the TruMap results.

To remove an element map from the display, select it by clicking on it and then press the Clear button. It will be removed from the display. Press the Clear All button to remove all maps from the display. Remember the maps will be displayed again when you press AutoID or include elements manually.

### Manual selection of energy windows and X-ray lines:

Press on the Map Details dialog to open the Selected Elements Details:



The default setting is Automatic X-ray lines and energy window width selection.

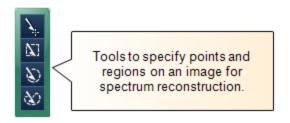
window width.

- To manually define the width of the energy window, select the Specify Energy Window option. Enter the values for Lower Energy (keV) and Upper Energy (keV) and
   Update X-ray map with changes
   The map will be acquired using the defined
- To manually select the X-ray line for mapping an element, select the Specify Line Series option. Select the line from the Line Series drop-down list and press

# Reconstruct spectra from Layered Image or Maps using Point, Rectangle, Ellipse and Freehand tools

The spectrum reconstruction tools are available in the toolbar on the left of the workspace:

• Select the spectrum reconstruction tool from the four available options:



- Click on any image and drag with the left mouse to select a region. A reconstructed spectrum is displayed in the spectrum viewer and it is also added to the data tree.
- MiniQuant results of the reconstructed spectrum are displayed. You can compare the sum spectrum and the reconstructed spectra.
- To confirm the elemental composition of a phase you can navigate to the Confirm Elements step in the Point & ID package from the link below the Layered Image Viewer. For details refer to Online Help.

### See Also:

AutoLayer on page 222

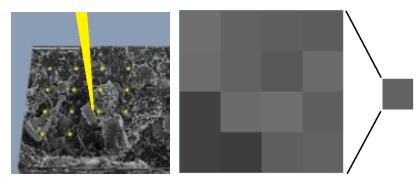
Context Menu - Map Viewer on page 223

How binning affects the quality of your data on the facing page



### How binning affects the quality of your data

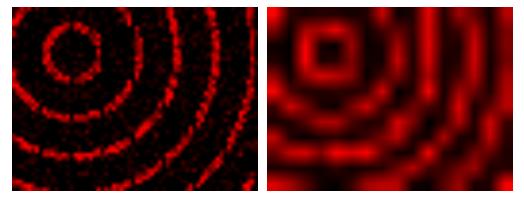
As the electron beam scans a line or area of the specimen, data is acquired from numerous points. Inevitably, the signal from each point includes some noise. If data is not acquired for long enough, the noise level will be high. By combining signals from several neighboring points, the binning technique produces an averaged signal, which has less noise overall. The effect is similar to collecting the original data at a lower resolution and a longer dwell time.



You can select the binning factor from the drop-down list below the map or linescan display.

#### Effect of binning on element maps

A binning factor of four combines 16 adjacent pixels from each 4x4 square into one new pixel. For example:



Binning is useful when you are using the TruMap mode for mapping because of the improved statistics. Binning also enables AutoLayer to combine similar maps more successfully because of the lower noise.

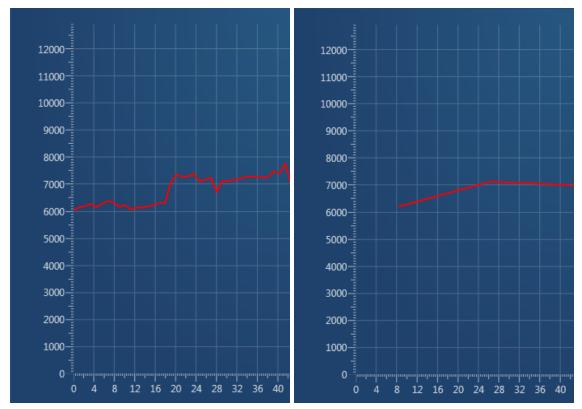
#### See also:

AutoLayer on page 222

#### Effect of binning on linescans

Binning combines a group of pixels into one new pixel. The binning appears to shorten the linescan at each end. In the following example with a binning factor of 16, the first value

appears at approximately 8  $\mu m$  because the first binned point is midway between the first 16 values.



Original data at 1 µm spacing

Binned data

.....

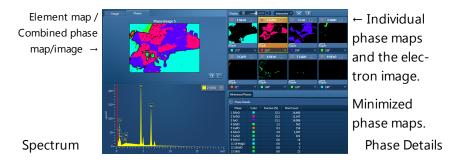


# **Analyze Phases**



In this step, the software automatically converts X-ray maps into phase maps. The phase maps help you to see the constituent elements of the phase, and how the phases are distributed over the specimen.

The window has several sections:



- The individual phase maps (top right) are presented according to the distribution and size of each phase. The first maps show large areas of closely grouped elements. Later maps show smaller areas that are more finely distributed. You can change the colors in the phase maps to better represent interesting groups of elements (or "phases").
- On the Image tab (top left), you can view any image in your project, such as the EDS layered image. The Phases tab shows the combination of images that you select from the many phase maps and the electron image.
- The spectrum (bottom left) shows a spectrum extracted from the pixels in the currently selected phase. You can also closely examine the spectrum at any point or area of interest using tools on the left toolbar.
- Phase Details (bottom right) shows the area of each phase in pixels and as a percentage of the total area of the map. You can copy these results into a spreadsheet.

Toolbars around the window enable you to change the processing of the data, and display and analyze the data.

#### See Also:

About phase maps on page 377

Analyze Phases toolbars on page 383

### **Finding phases**

In the Analyze Phases step, the software automatically converts X-ray maps into phase maps. The phase maps help you to see the constituent elements of the phase, and how the phases are distributed over the specimen. You can run this step while map data is acquired, or afterwards.

If map data is being acquired, the phase analysis repeats periodically until acquisition finishes or you click the "Cancel Processing" button. The phase analysis produces the best results if run after all the map data has been acquired and elements have been identified.

 In the acquisition toolbar, click the green "Find Phases" button to start processing. When processing is finished, phase map data appears on the Data Tree, for example:



- 2. To re-run the processing with different settings, select the Settings cog icon from the top toolbar. Any changed settings are retained in your User Profile, and become your default settings when you next run this analysis.
- 3. After the processing, you can manipulate the phases as required:
- Examine the spectrum at any point or area on the phase, using the tools in the left toolbar, to confirm the composition.
- Select individual phase maps to include in the combined phase map. You can also include the electron image.
- Merge phases that you want to analyze as a single phase.
- Remove phases that are not relevant to your analysis.
- Rename the phase maps in the Data Tree to identify possibly significant phases. For example, rename CaSiO to Calcium Metasilicate.
- Change the color of phases for convenient identification.

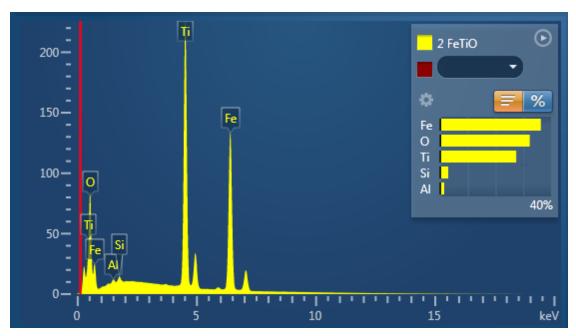
#### See Also:

Analyze Phases toolbars on page 383



# **About phase maps**

The name of each phase is derived from its main elements. For example, a phase with the following spectrum might be named "2 FeTiO". Although the spectrum contains Si and Al, the quantities are much smaller, and therefore those elements are not included in the phase name.



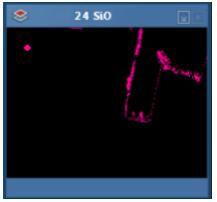
The names of the cations and anions are arranged in an order that closely resembles the name of a chemical compound. The name of the phase is a *possible indication* of a chemical compound.

### The order of the phases

The order of the phases depends on the distribution and quantity of each group of elements. The first phases indicate large areas of closely grouped elements. Later phases show smaller areas that are more thinly distributed, for example:

- 2 SiO shows one large area or a few large areas that contain Si and O.
- 24 SiO shows smaller and probably more scattered areas that contain Si and O.





# The number of phases

You can display more or fewer phases by changing the analysis settings, or by merging phases manually.

#### See Also:

Analyze Phases settings on page 381

Merging phases on the facing page



### **Merging phases**

To simplify the results of the phase analysis, you can merge some phases. The result is a new phase with a combined spectrum, and fewer phases overall.

For example, these phases have similar spectra and might be considered to be the same phase:



You can merge similar phases in several ways:

- Set a higher Grouping Level, to automatically combine any number of similar phases.
- Manually merge any two phases.

To manually merge two phases:

- 1. In the phase maps, select a phase.
- 2. Right-click, and in the context menu, select "Merge into ...".
- 3. Select the other phase from the list.

The first phase disappears. The spectrum, phase name and color of the second phase are updated to include information from the first phase.

#### See Also:

Analyze Phases settings on page 381 (Grouping Level)

# Phase maps in the Data Tree

The EDS Data folder contains phase map data in a folder, which by default is labeled as 'Phase Image #', where # is an automatically increasing number. For example:



Icon	Description
	The EDS Data container contains the Phase Image folder, which contains all the phase maps.
<b>4</b>	The name of a phase map is composed of a number and its elements, for example: 2 AlMgO. To rename the phase , right-click the icon and select Rename.
	A spectrum extracted from one of the phases. For details, right click the icon.

#### See Also:

Data Tree on page 85

Analyze Phases on page 375



## **Analyze Phases settings**

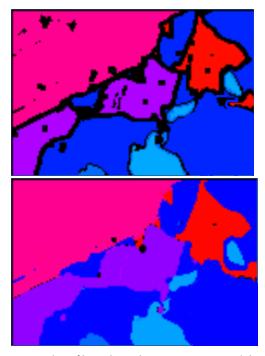
The software processes data acquired from the maps according to the settings for Boundary Threshold and Grouping Level. To see the settings, click on the Settings button. The default settings are stored in the User Profile.



#### **Boundary Tolerance**

Boundary tolerance controls the behavior at the boundaries of each phase.

- If the boundary tolerance is low, each pixel in the phase represents a pure spectrum. If a pixel has contributions from several phases, it cannot be identified, and appears black.
- If the boundary tolerance is high, all pixels are placed in the phase that fits them most closely. The spectra for each phase shows small contributions from adjacent phases.



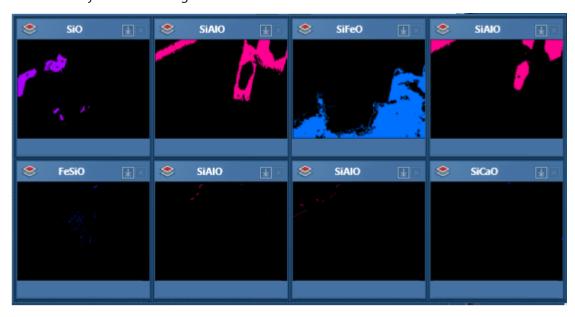
Low setting of boundary tolerance

High setting of boundary tolerance

#### **Grouping Level**

Phase analysis identifies all the phases in the sample. Grouping combines phases that are similar, and can create a smaller, more manageable number of phases overall.

- If the grouping level is low, a larger number of phases are displayed. The later maps show phases that occupy small areas, and might indicate trace compounds.
- If the grouping level is high, a smaller number of phases are displayed. The maps typically show a few large areas.



If the Grouping Level is too high, areas that you consider as separate phases will be merged together. To resolve this problem, you can reduce the Grouping Level until all the expected phases are shown, and then manually merge any phases that you prefer to analyze as a single phase.

#### See Also:

Analyze Phases on page 375

User Profile on page 22

Merging phases on page 379



# **Analyze Phases toolbars**

These toolbars are available in the Analyze Phases step.

# Map display tools

These controls are above and below the phase maps.

Settings	Description
	Displays the phase image, or the phase maps, or both.
	Sets the number of maps per row in the Standard and Interactive displays.
Standard •	Offers a choice of map display:
	<ul> <li>Standard - you can add individual images or remove them from the combined image.</li> </ul>
	<ul> <li>Interactive - similar to Standard. Links the zoom and pan of all the maps.</li> </ul>
	<ul> <li>Summary - a compact display, where you can change the color of each phase.</li> </ul>
	Links images for manipulation of all layers using the Pan or Zoom controls.
	Changes the brightness and contrast of the currently selected image or map.
	Adds the selected phase map or electron image to the combined phase image.
	Minimizes a map. The Minimize icon is in the top right hand corner of each map. This is useful if you have too many maps in view. The map moves to the Minimized Phases tab, below the displayed maps.
	Removes a map or electron image from the phase analysis. This icon is in the top right hand corner of the image.

Settings	Description
	Restores a minimized map to its normal size. The minimized maps are on the tab below the displayed maps. This icon is in the top right hand corner of each map.
■ 180* -	Sets the color of the phase. The list is available only in the Interactive and Summary map display.

#### See Also:

Analyze Phases on page 375

# **Processing toolbar**

These controls are at the top of the window, above the phase maps.

Icon	Description
Find Phases	Starts processing the element maps to create each phase map. A Cancel button and a progress bar appear during the processing.
	Shows the progress of the processing. For more details, hover the cursor over the progress bar for a few seconds.
Cancel Processing	Stops the processing.
	Adjusts the settings used during the processing:  Boundary Tolerance controls the behavior at the boundaries of each phase.  The Grouping Level determines the numbers of phases that you see.

See Also:



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Analyze Phases on page 375

Analyze Phases settings on page 381

Finding phases on page 376

# **Toolbar for Phase Map**

These controls are at the top left side of the window.

Icon	Description
	Moves the image. Click the Pan tool, then click and drag the image. Use the mouse wheel to zoom in and out.
	Normalizes the spectra.
	Adds annotations to the current image. The tools include Caliper, Angle, Text, Rectangle and Ellipse.
7 2 N 8	Defines points and regions on a map image to extract spectra.
i	Shows the energy and counts at any point in the spectrum viewer (in the bottom left corner).
	Shows the phase at any point in the phase maps (in the top right corner).

#### See Also:

Analyze Phases on page 375

Map -	Custom
-------	--------

In the Custom mode, the Map navigator has two steps.

The Describe Specimen step is covered in the earlier section. The step which is unique to this navigator is described here.

Acquire and Construct 387



### **Acquire and Construct**



Scan Image, Acquire Map Data and Construct Maps are laid out as separate steps in the Guided mode of the Map application. There are four components that make up these three steps: an Image window, an X-ray map window, a Spectrum reconstruction window and a Confirm elements window. These four components are combined in the Custom mode to give you a single workspace called Acquire and Construct. It offers the convenience of working in one window without having to move away from it.

The user interface components are docked in the workspace in the four quadrants. Each component can be undocked in a free floating window. It can be dragged on to another monitor, resized or displayed in the full screen view.

There is a toolbar located near the top right corner of the workspace with icons which allow you to toggle on/off each component.

The user interface elements are described below:

#### **Electron Image/Layered Image**

The Scan Image component is docked in the top left quadrant. It allows you to acquire an electron image and a Layered image. You can choose to display either the electron image or the Layered Image.

Press to acquire or display the electron image. Press to acquire map data or display the Layered Image.

#### **Element Maps**

The Element Maps component is docked in the top right quadrant of the Acquire and Construct workspace. You can acquire element maps here and view them in three different ways, Standard, Interactive or Summary view. To get details of all the functions follow the Acquire Map Data link below.

#### Spectrum Viewer

The bottom left quadrant displays the current spectrum. It can be a Sum Spectrum or a Reconstructed Spectrum. At the top right corner of the Spectrum Viewer, there is a link to the Confirm Elements step of the Point & ID Navigator. It is a useful option for identifying and confirming small peaks in the spectrum. Select Map to get back into the Acquire and Confirm workspace from the Confirm Element screen.

#### **Selected Element Details**

The Map Details is located in the bottom right quadrant of the workspace. From the Selected Element Details, you can select which elements you wish to map. You can define the energy windows for window integral maps and select the X-ray lines you wish to use for mapping instead of using the automatically selected energy window and lines. When you select a map from the element map display, the energy window and X-ray lines markers for this element are displayed in the spectrum viewer. To read details of defining energy windows and choosing X-ray lines, follow the link to Construct Maps topic below.

#### See also:

Acquire Map Data on page 363

Construct Maps on page 370

Acquire Map Data - Settings on page 211

Context Menu - Map Viewer on page 223



## **Linescan - Guided**

In the Guided mode the Linescan navigator has four steps:

Describe Specimen, Scan Image, Acquire Line Data and Construct Linescans. The two new steps are described here:

Acquiring linescans	391
Displaying and manipulating linescans	393
Measuring the distance between two points	395
Viewing element counts and percentages	396
Comparing element quantities.	397
Smoothing the linescans.	398
Linescan Data	399
Exporting the linescan data	400
Extracting a single spectrum from the linescan	401
Extracting multiple spectra from the linescan	402
Construct Linescans	403

### **Acquire Line Data**



In this step you can acquire element linescans along a line defined on the electron image or map. The data can be processed in several ways:

- Line, also known as Window Integral, obtains the counts in the element energy windows including the background. Line gives a fast and simple representation of the X-ray energies.
- TruLine, also known as Filtered Least Squares (FLS), applies further processing. Sometimes the standard X-ray mapping (Line) gives misleading results because some elements have overlapping energy windows. For example, a Titanium linescan

might include Barium information. The TruLine option eliminates the problem by comparing the X-ray line series with the expected peak shape for each element. The linescans are corrected for peak overlaps and any false variations due to X-ray background.

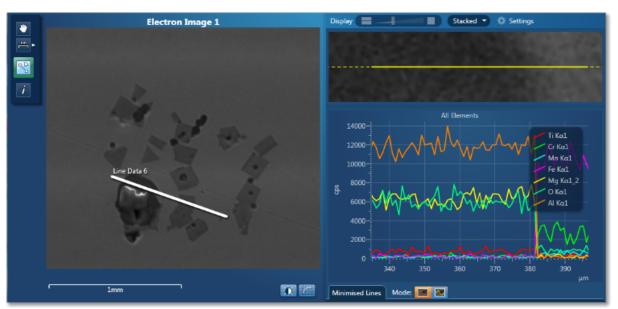
• QuantLine further processes the data, showing the atomic or weight percentages of elements at every point on the line.



### **Acquiring linescans**

The elements for which linescans are being acquired are chosen in the Describe Specimen step by selecting the Auto ID option, Pre-defined Elements, or both. To see the acquisition settings, click the Settings cog in the toolbar in the title bar.

- 1. Select the Acquire Line tool from the toolbar on the left.
- 2. Click on the image to set the start point and then drag the mouse to define the line. Release the mouse to set the end point. A line with start and end points is defined on the image.
- 3. Press to start acquisition. A relevant section of the image is zoomed and rotated above the Linescan viewer. This action aligns the defined line horizontally to match the x-axis of the Linescan viewer.



The progress of line data acquisition is displayed in Current Site tab in the Data View:



- 4. Element linescans start to populate the Linescan viewer as the data is being acquired.
- 5. You can stop the acquisition by pressing or the red STOP Button. To cancel the line processing, click the "Cancel Processing" button in the acquisition tool bar.
- 6. From the controls above the Linescan viewer, choose how to process the data, for example: TruLine.

The TruLine data processing will use the TruLine settings from the EDS Element settings tab in the User Profile screen. You can access the User Profile from the Tools menu. You can specifically define whether the threshold is on by selecting "Apply threshold for TruLine" and entering a Sigma Threshold between 0.0 and 3.0.

#### See Also:

User Profile on page 22

Acquire Line Data - Settings on page 252



### Displaying and manipulating linescans

Three different views are available from the controls in the top right corner of the Acquire Line Data screen:

■ Display Electron Image Full Screen



 Display Linescans and Electron Image



■ Display Linescans Full Screen



Several options are available to view the Linescans:

- Stacked multiple linescans overlaid are displayed in a single view.
- Vertical Tiles individual element linescans are displayed in a separate view. You can change the height of each view using the Display slider bar.
- Table values for each point on the line.

You can pan and zoom linescans using the mouse controls. Both the viewers, (image and line view) respond synchronously to the mouse interactions.

- Left mouse button down:
  - Move left / right pan left/ right (if view is expanded). Tip: If the list of elements and lines obscures the right end of the linescan, pan fully to the left.
  - Move up / down change the scale height
- Mouse wheel
  - Zooms x range in/out around current x value (defined by mouse location). The image will expand/shrink to match the data displayed. If the data is not visible in the viewer because of pan/zoom state, the line on the rotated image will change to a dotted yellow line to indicate there is more data.
- Linescan viewer specifics:
  - Dragging directly on the axis will pan the range.
  - Mouse wheel on the axis will expand / reduce the range.

The thickness of the line in the plots can be set globally from the Linescans Viewer tab in the Preferences screen. The thickness values in pixels are as tabulated below:

	Thickness in Pixels
Thin	0.5
Normal	1.0

	Thickness in Pixels
Thick	2.0
Thicker	4.0

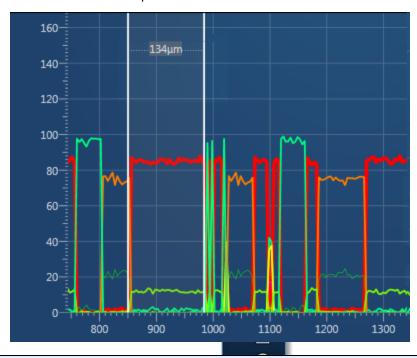
In addition you can change the color and thickness of individual lines from the Settings in the Linescan viewer toolbar.



### Measuring the distance between two points

The distance between two points in the Linescan viewer can be measured using the Caliper tool.

- 1. Select the tool from the toolbar on the left, and move the mouse over a Linescan viewer (stacked or tiled). This cursor will track the mouse movement.
- 2. Double-click to set the first measurement point.
- 3. Move the mouse to paint a region on the viewer, which shows the distance between the two points. This will update as the second cursor is moved.
- 4. Double-click to fix the position of the second cursor.



#### NOTE

Unlike calipers on an image, the linescan calipers are not saved with the data. When you switch to a different tool the caliper information will disappear (and will not reappear when you switch back).

A new region can be drawn by double clicking in the viewer at the new (start) point. You can then define the extent of the measurement by placing the second marker, as described above.

To get rid of the region on the viewer, you should switch to another tool on the toolbar.

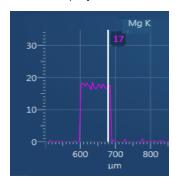
In the Vertical Tiles view, you can use the Caliper tool on individual linescans to display the distance between two points.

## Viewing element counts and percentages

1. Select the "Show data values" tool from the toolbar on the left:



2. Click on the line in the Linescan viewer. A vertical cursor appears at that location, and displays the value for each element in that location. For example:



If no sigma threshold is applied to TruLine processing, some values might be negative. To see the sigma threshold setting, go to the Tools menu, select User Profile, and then the EDS Element Settings tab.



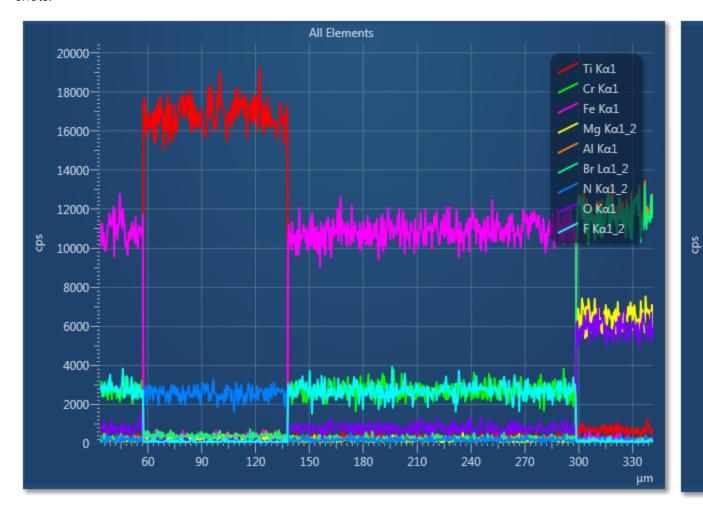
# **Comparing element quantities**

The Normalize Y-Axis option is available on the context menu of the Linescan viewer, when in Stacked view. It allows you to compare linescans with very different maximum count rates, and is useful in QuantLine displays. The maxima of linescans are scaled to the full height of the viewer. It allows you to view the details of the minima of linescans with low count rate.

Note that there is no Y-axis on the normalized linescans because the absolute scale is meaningless.

# **Smoothing the linescans**

The Smoothing Factor option is available from the Linescan viewer settings. It allows you to smooth the linescans after normalization for visual clarity as demonstrated in the screen shots:



Typical linescan

Smoo

The Smoothing Factor uses moving averages to remove fluctuations of data.

The options available are:

- 1: No smoothing applied
- 3: Data averaged over three points
- 5: Data averaged over five points

As an alternative way to reduce the effects of noise in the linescans, you can apply a binning factor. Binning is particularly effective when using TruLine.

#### See Also:

How binning affects the quality of your data on page 373.

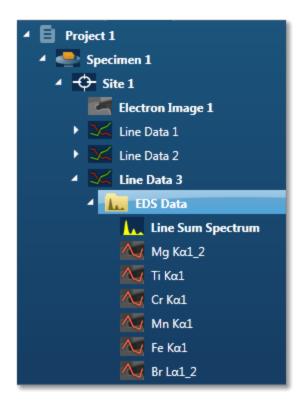
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### **Linescan Data**

The data tree contains a Line item under the Site; this is the container for the line data. By default, this is labeled as 'Line Data #' where # is an auto-increasing number under the current site (Site 1) as shown below:



Icon	Description
	The Line item is the container for EDS Data. All linescans and the sum spectrum are contained within the EDS Data container. For details, right click the icon.
	The sum spectrum is called Line Sum Spectrum.
<b>₹</b>	The name of the element linescan (Line, TruLine or QuantLine) is composed of the element symbol and the line.
	A spectrum extracted from a single point on the linescan.

#### See Also:

Data Tree on page 85

## **Exporting the linescan data**

Right click on the linescan viewer to access the Export menu. It has several different ways of exporting the linescans: Save As, Copy, Print and Email. The exported image includes the relevant information from the Caliper or Show data values tool.

To export some or all of the data to a spreadsheet program such as Microsoft® Excel®:

- 1. On the toolbar above the linescan viewer, select Table from the drop-down list.
- 2. To select all the data, click one row in the table, then press Control and A. To select only some of the data, click a row then drag, or use click and Shift-click.
- 3. Right-click and select Copy.
- 4. Paste the data into your spreadsheet.

#### See Also:

Toolbars on page 253



# Extracting a single spectrum from the linescan

You can examine the spectrum and element quantities at any point along the line.

1. In the toolbar on the left of the image viewer, click the Reconstruct Line Point Spectrum tool:



- 2. Move the cursor to any point on a line in the linescan viewer.
- 3. Click to extract the spectrum for the point into the project.

You can view the spectrum in the Mini View. The data is also saved in the Data Tree with this icon:



# **Extracting multiple spectra from the linescan**

You can examine the spectrum and element quantities at many points along the line. To prevent an excessive amount of data, you can apply a binning factor to limit the number of points.

1. Click the Extract Spectra button below the linescans:



- 2. In the Reconstruct Spectra dialog, select a binning factor.
- 3. Click START to extract the spectra for the points into the project.

The data is saved in the Data Tree with this icon:



#### See Also:

How binning affects the quality of your data on page 373

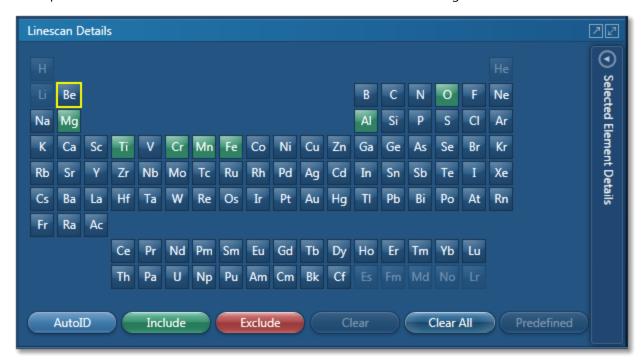


### **Construct Linescans**

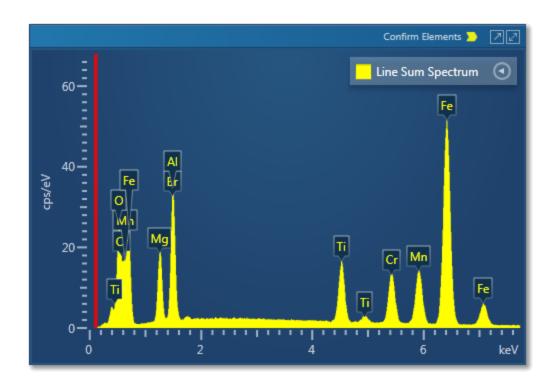


In this step you can define energy windows and configure X-ray line series to update the display of element linescans in the viewer.

You can use AutoID for initial display and then add or remove elements as you wish using the periodic table and the controls available in the Linescan Details dialog:



In addition, you can view the Line Sum Spectrum and navigate to the Confirm Elements step from within the Construct Linescan step to manually confirm elements:



# Manual selection of energy windows and X-ray lines

1. Press to open the Selected Elements Details dialog:



The default settings are automatic X-ray line series and energy window width selection.



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2. To manually define the width of the energy window, check the 'Specify Energy Window' option. Enter the values for Lower Energy (keV) and Upper Energy (keV) and

Update linescan with changes

3. To manually select the X-ray line for an element linescan, check the 'Specify Line Series' option. Select the line from the Line Series drop-down list and press

Update linescan with changes

#### **Linescans Display**

There are three different display options available from the controls, near the top right corner of the Construct Linescans screen:



- Display image full screen
- Display linescans and image
- Display linescans full screen

#### See Also:

How binning affects the quality of your data on page 373

Linescan -	Custom
LIIICSCAII	

In the Custom Mode, the Linescan navigator has two steps:

The Describe Specimen step is explained in the earlier section. The new step is described below:



### **Acquire and Construct - Linescans**



The three components, Scan Image, Acquire Line Data and Construct Linescans are laid out as separate steps in the Guided mode of the Linescan application. These three components are combined in the Custom mode to give you a single workspace called Acquire and Construct. It provides the convenience of working in one screen without having to move away from it.

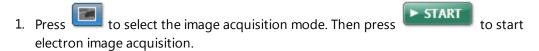
The user interface components are docked in the four quadrants in the workspace. Each component can be undocked as a free floating window. It can be dragged on to another monitor, resized or displayed in the full screen view.

There is a toolbar located near the top right corner of the workspace with icons which allows you to toggle on/off each component.

The user interface elements are described below.

### Acquiring an electron image and line data

In the top left quadrant, you can acquire an electron image first and then define a line to acquire the line data.



- 2. On completion of image acquisition, press to switch to the line data acquisition mode.
- 3. Press to select the line tool in the toolbar on the left.
- 4. Click on the image to set the start point and then drag the mouse to define the line. Release the mouse to set the end point. A line with start and end points is defined on the image.
- 5. Press to start the line data acquisition. A relevant section of the image is zoomed and rotated above the Linescan viewer. This action aligns the defined line horizontally to match the x-axis of the Linescan viewer.

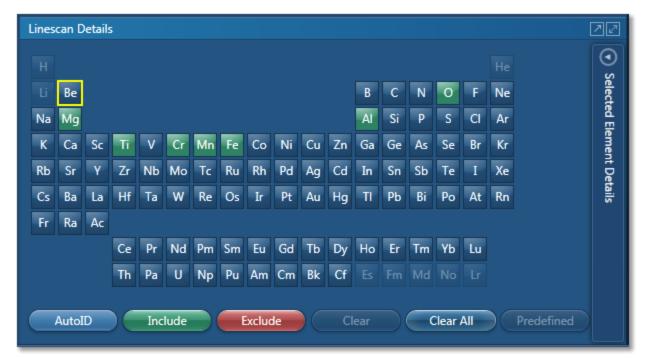
Element linescans start to populate in the Linescan viewer from in the top right quadrant as the data is being acquired as shown in the next screen shot:



#### **Selected Element Details**

In the bottom right quadrant, you can define energy windows and configure X-ray line series from Selected Element Details.

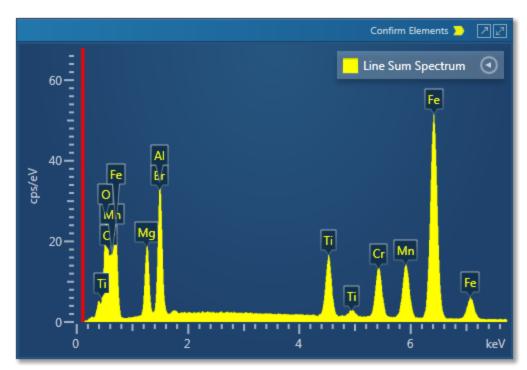
AutoID can be used for initial display. You can add or remove elements as you wish using the periodic table:



**Spectrum Viewer** 



The spectrum is available in the bottom left quadrant. You can view the Line Sum Spectrum and navigate to the Confirm Elements step from within this component to manually confirm elements:





# **EBSD**

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# EBSD - Map

The Map navigator has six steps which are described below:

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### **Describe Specimen**



In this step, you can record information about your specimen, such as its coating, and view phase information.

### Summary

In the Summary view you can write notes on the project and the specimen within the project. (For convenience you can also copy images or diagrams and text from other documents or email and paste into these windows). Notes are saved with the Project and you can edit notes in any step of the Navigator.

The notes help you to capture the important information during your experiments. Right click with the mouse on the Project or Specimen entries in the Data Tree and then select Edit Notes to write/modify the relevant notes.

You can add new Specimens to the current Project by pressing the New Specimen button:



Note that you may write notes about each Specimen and save them.

#### **EBSD**

The Phases for Acquisition are listed in this view together with the number of reflectors, color and the option to include/exclude them during analysis.

### **EDS**

You can add coating information for each specimen. This information is used later during the calculation of the quantitative results.



To specify a coating element, check the coating option and select an element from the Periodic table.

Type the thickness and coating density so that the software applies a full coating correction when calculating the quantitative results. The default element is Carbon with the default values for thickness of 10 nm and a density of 2.25 g/cm3. The default density of the element is at room temperature and pressure where appropriate.

You can change the thickness and density if required. The peaks for the coating element are automatically deconvolved from the spectrum before quantification but are excluded from the calculation of composition.

Please see the topics on Specimen Geometry and Phases via the links below:

#### See Also

Specimen Geometry below

Phases on page 416

Data Tree on page 85

Mini View on page 93

Step Notes on page 94

### **Specimen Geometry**

### **Specimen Tilt**

This area of the application allows you to define the way that the Specimen is tilted in the chamber with respect to the horizontal plane.

Diffracted electrons only escape from a depth in the order of a few tens of nanometers deep from the sample surface. At low tilt angles the total interaction volume close to the surface is very small compared to the interaction volume deep within the material. Consequently, at zero or low tilt, the proportion of diffracted electrons in the overall electron yield may be so low as to be undetectable. Tilting the sample improves the diffracted component to background yield ratio by increasing the volume of near surface material excited. Thus EBSD is generally carried out at approximately 70 degrees tilt.

### **Pretilted Specimen Holder**

If you are using a Pretilted Specimen holder for your experiment, check the corresponding box and enter the corresponding Tilt Angle. Note that this option is only enabled in the application if it has been checked in the Configuration Tool under 'Enable Software Features in Application'. If you are not using a pretilted holder this angle will be zero. Remember to switch off the Pretilt option when you are not using a pretilted holder since it will affect EDS and EBSD data.

#### Stage Tilt



The Stage tilt value is automatically read from the microscope via the Microscope Control. However, if this value is not automatically read, you may enter the value in the Microscope Control window.

### **Total Specimen Tilt**

This value is the sum of the Stage tilt and the angle of the Pretilted holder. This is the tilt angle which is being used by the software for analysis, so check that the specimen tilt is correct before doing any experiments. This can also be shown in the Status Bar and is shown by default.

### **Specimen Orientation**

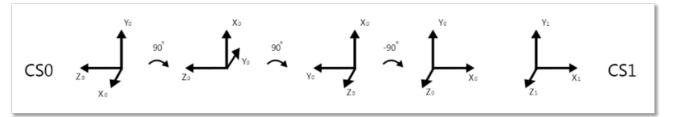
In the Specimen Orientation tab it is possible to define a data rotation which is saved with the Project.

The data shown in the application is always the original, unrotated data so the selection in the Specimen Orientation tab does not affect the way the data is displayed however it works as a placeholder for saving information about the Specimen Orientation. If a rotation transformation is saved with the data then the .cpr file after export to CHANNEL5 format will also contain information about this rotation, so that data viewed in the CS0 coordinate system (Sample Primary Coordinate system) will be shown with the saved rotation. Once the data is exported it can also be rotated from CHANNEL5's Virtual Chamber tool and this tool can also be used to alter any rotation saved from the software.

The six buttons shown are associated with the six standard orientations and are a quick way of specifying the Specimen Orientation. The lower seventh button allows the user to manually type in a rotation in the numeric fields to the right.

When EBSD data is rotated it is between two coordinate systems; CS1 (Data Acquisition Coordinate system) and CS0 (Sample Primary Coordinate system). If the sample coordinate system and acquisition coordinate system are coincident, then the default rotation will be 0, 0, 0.

The data rotation is defined as the rotation from CS0 to CS1. For example the rotation 90, 90, -90 means that the CS0 coordinate system is related to CS1 in following way:



The first angle is the rotation around the coordinate systems z axis, the second angle is the rotation around the rotated x axis and the third angle is the rotation around the rotated z axis required in order to make the two coordinate systems coincide.

Note that the scanning system always has the x axis horizontal, the y axis vertical and the z axis pointing out of the image so the orientation of the CS1 is always the same.

### **Phases**

In order to index an EBSP, the software needs to know what phase(s) is present in the specimen. This step provides the functions necessary to locate a phase in a database and add phases to the list of phases to include during acquisition.

#### **Phases in Database**

You can select a database by using the drop down menu in the Database menu. The database list shows the database files present in the CHANNEL5 directory and will also include user defined databases if present.

When you have selected a database all phases in the database will be listed. Highlight the desired phase in the phase list to view information about the unit cell of the selected phase, the phase details and the list of calculated reflectors to the right hand side of the work area. The calculated expected intensities of the Kikuchi bands or reflectors are shown in the Reflectors tab. Note that these intensities are given for families of reflectors.

You can also scroll to a phase in the database by entering the start of its phase name into the space provided. It will then highlight the best matching phase in the list.

Once you have found the phase that you are looking for, press the 'Add Phase for Acquisition' button. This will now load the phase into list of 'Phases for Acquisition' and it will be used for indexing EBSPs. You may repeat the process and include more phases if you wish.

#### 3-D Phase View

The 3-D Phase view may be shown with or without the unit cell within the spherical simulation of the Kikuchi bands. Use the context menu to select different view modes of the 3-D simulation.

The Kikuchi Map gives a spherical simulation of the Kikuchi bands for the unit cell orientation visible. This can be freely rotated using the mouse. You may also adjust the appearance and the number of Kikuchi bands using the options on the 3-D Phase View context menu by selecting a different kV and Simulator Reflectors.

The unit cell visualization gives a 3-D simulation of the unit cell of the selected phase, with the a, b and c axes marked respectively in red, green and blue. Using the mouse you can freely rotate this simulation.

### **Phases for Acquisition**

In the list of 'Phases for Acquisition' it is possible to include/exclude individual phases. When the phase is not included, that phase will not be used by the software for indexing EBSPs.

The number of reflectors per phase used during indexing can be adjusted by selecting the appropriate number from the drop down list. This number tells the software how many theoretical Kikuchi bands to utilise during the indexing process.

Lower numbers generally result in faster indexing, but may also give a lower hit rate (% of EBSPs indexed) and more wrong or misleading solutions.

If you wish to add a phase from a cry or hkl file to the "Phases for Acquisition" list, press the 'Add from File' button.



Once you have acquired the EBSD data into a Specimen, you can not remove phases, you can only add them. However you can disable them (this is to ensure data coherent in a Specimen for future applications).

#### See Also

Configuring groups of phases below

### **Configuring groups of phases**

During EBSD analysis, there are some situations when more than one phase with the same crystal structure can co-exist in a given sample. In many cases, these can be discriminated from each other using chemical information acquired from EDS data, but not directly from their EBSD patterns. In such cases, it is possible to look at the difference in lattice parameter in order to identify such phases of similar crystal structure.

The software enables phases of the same crystal structure to be discriminated from each other using their respective EBSD patterns using band widths, provided their lattice parameters differ by more than 10%. A good example of this is Platinum – Nickel, which have the same crystal structure but have a difference in lattice parameter of 14%.

The Phase Group Configuration window allows you to group materials together which have similar crystal structures.

To create groups of phases:

- 1. At the "Describe Specimen" step, select the Phases tab.
- 2. Under "Phases for Acquisition", click "Configure Grouping" to open the "Phase Group Configuration" dialog.
- 3. In the dialog under "Ungrouped Phases", select two or more phases for the group.
- 4. Click "Create Group". The new group appears under "Phase Groups".
- 5. Create any other groups, if required. To improve the view of large phase groups, you can drag the bottom right corner of the dialog outwards.
- 6. To remove a group, click the X in its top right corner. To delete all groups, click "Remove All Groups".
- 7. Click OK to close the dialog.

The name of each group appears next to its phases under "Phases for Acquisition".

When these materials have been grouped together, band width information will automatically be used to distinguish phases within a group.

### **Scan Image**



In the Scan Image step, you can acquire an electron image into a 'Site'. A 'Site' is like a folder, which contains images and analyses for a particular area on a specimen.

For EDS, if you do not want to collect an image and just want to acquire spectra, you can skip this step and go straight to the Acquire Spectra step.

You can have any number of images in a site. Just ensure that the images you want to keep are padlocked in the data tree to stop them being overwritten, as shown in the screen shot below:



You can toggle between saving or replacing the current image with successive image acquisition.

If your specimen is drifting, click the Settings cog and activate AutoLock.

The Scan Image step has several tools for manipulating and enhancing electron images:

- The acquisition toolbar above the electron image and other nearby controls.
- Scan Image toolbar (a vertical toolbar on the left) for manipulating and annotating the image.
- If a forward-scatter electron detector (FSD) is fitted, extra controls are available for combining the signals from each diode into a mixed image.

### Acquisition toolbar and other nearby controls

The acquisition toolbar, above the electron image and below the Navigator, has buttons for starting and stopping the image acquisition, the Settings cog for selecting the image acquisition parameters and a button to link/unlink images for manipulation.





Control	Description
(FSD only)	Displays up to three combinations of mixed image and individual images. Your selections are retained in your user profile.
► START	Click to start the image acquisition according to the current acquisition parameters.
■ STOP	Click to stop image acquisition. Acquisition stops at the end of the current frame. Click again to stop immediately. If you navigate away from the step, acquisition stops at the end of the current frame.
Settings	To change the acquisition parameters, click the Settings cog on the Acquisition Toolbar to display a dialog.
	You can select Image Scan Size, Dwell Time (µs), Input Signal the labels here reflect whatever was set during the installation for example SE, BSE or FSD), either Continuous Scan or Number of Frames and Frame Time (secs).
	If your specimen is drifting, you can activate AutoLock to ensure that any analysis corresponds to the true location on your image.

Control	Description
Mixing Mode (drop-down list)	Combines signals from the diodes to form a mixed image. Some options are available only if the required diodes are installed and configured.
(FSD only)	<ul> <li>FSD Z Contrast uses upper and side FSD detector channel images. Select this mode if you are inter- ested in seeing density/atomic Z contrast signal.</li> </ul>
	<ul> <li>FSD Topo/Orientation uses lower FSD detector chan- nel images. Select this mode if you are interested in seeing orientation contrast signal.</li> </ul>
	<ul> <li>Custom - include and exclude FSD detector channel images of your choice.</li> </ul>
	When you select either of the first two modes, the software automatically uses the FSD diode channels associated with that mode. For example, FSD Z mixing mode uses the upper and side FSD diode channels. The FSD Topo/Orientation mixing mode always uses the lower FSD diode channels. Custom mode allows you to mix any FSD diode channels.
Select Second Image	Selects further images to compare with the electron images, for example, a forward-scattered electron image. This control is available only when the map display is for an image only:
(FSD only)	Sets the number of images per row in the Standard and Interactive displays.
Standard •	Offers a choice of image display :
(FSD only)	<ul> <li>Standard - you can add individual images or remove them from the mixed image.</li> </ul>
	<ul> <li>Interactive - similar to Standard. You can also change the weighting and color contributed by each image.</li> </ul>
	<ul> <li>Summary - similar to Interactive and in a more compact display.</li> </ul>
<b>3C</b>	Links images. You can simultaneously manipulate all the layers using the pan or zoom controls.



Control	Description
	Unlinks images. You can manipulate individual layers using pan or zoom controls.
	Use the mouse wheel to zoom in and out of the image.
	Use these tools (near the bottom right of the screen) to adjust manual and automatic brightness, contrast and color.
Context Menus	Right-click the electron image to display context menus for copying, exporting and printing images.

### See Also:

Scan Image Toolbar on page 424

Scan Image - Settings below

FSD diode controls on page 432

Context Menus - Image Viewer on page 157

Export - Settings on page 132

AutoLock on page 133

### Scan Image - Settings

The selectable parameters that control image acquisition (Image Scan Size, Dwell Time and Number of Frames) should be chosen according to your specific requirements. Both the time taken and the data storage size of the image are dependent on these parameters.

For a quick look at the specimen select the lowest image scan size and the fastest speed. This will enable you to decide whether you require either a higher pixel density, in order to observe finer detail such as small features, or a longer dwell time in order to improve the image quality by reducing the noise.

The available acquisition parameters are:

- Image Scan Size
- Dwell Time (µs)
- Mains Synchronize
- Input Signal
- Software Tilt Correction
- Continuous Scan

- Number of Frames
- Frame Time (secs)
- FSD Control
- AutoLock

### **Image Scan Size**

In general, the resolution of an image or Image Scan Size is defined as the number of picture points or pixels along the x and y axes e.g., 256 x 256, 512 x 512 or 1024 X 1024. The quality of the image improves as the resolution at which an image is acquired is increased. However, a microscope monitor/CRT is usually a rectangular display (rather than square), so the resolution is displayed as a rectangle i.e., 256 x 200 in order to take into account the aspect ratio. The y dimension is set at installation, when imaging is calibrated. It will vary with each system.

Select the Image Scan Size for image acquisition from the following drop down options available: 64, 128, 256, 512, 1024, 2048, 4096, 8192

### Dwell Time (µs)

Images can be acquired using different speeds. The beam dwells on each pixel for a specified length of time while the signal is collected and then it moves to the next pixel. So the speed at which an image is acquired depends on the dwell time.

Speed	Dwell time
Fastest	1 μs
Fast	5 μs
Normal	10 µs
Medium	35 µs
Slow	65 µs
Slowest	400 µs

### **Mains Synchronize**

Selecting Mains Synchronize on the Image Setup window, synchronizes the start of each scanned line to the mains supply. This will help to reduce mains borne interference in the image. Note that the acquisition time will be marginally longer than when mains synchronize is not selected.

Note that Mains Synchronize will only be visible if the appropriate hardware is installed.

### **Input Signal**

Select the signals from the detectors on the microscope.



For EDS, secondary electron imaging is generally most appropriate if you are imaging a sample which has topography whereas backscattered imaging is a very useful means of identifying areas of different composition on flat samples. Secondary electron imaging is the most common form of imaging and for a first look at your sample, choose this mode.

If you are analyzing a flat, polished sample and you can see weak contrast, switch to back-scattered imaging which will tend to enhance this contrast by showing up areas of different phases.

For EBSD, Forward Scattered Imaging is often used. Because of the high angle of tilt dictated by the collection geometry required for EBSD, many electrons are scattered forward and down towards the bottom of the phosphor screen. Using Forward Scattered Electron (FSE) imaging diffraction contrast is enhanced and the resultant signal makes the presence of individual grains easy to identify. The forward scattered electron signal produced is therefore ideal for EBSD investigations. However, the user may use any electron signal as required for the reference image.

If you select the Auto checkbox before you start a new FSE acquisition, the software automatically adjusts the signal from each diode for optimal brightness and contrast. The optimized electron image then appears after a delay of a few seconds. When you first start the software, the Auto check box is already selected for you.

### **Software Tilt Correction**

Enables the use of imaging tilt correction. This is an important function when working with tilted samples. If no tilt correction is done, images and areas will be distorted. If the software tilt correction is enabled, it will be possible to correct scanned images and areas based on information about the tilt angle and the scanning tilt axis.

### **Continuous Scan**

If the Continuous Scan option is checked, you will see the image start to scan down the window and it will continue to refresh after each frame. If there are any instabilities in your specimen (e.g., charging or drifting problems) then these will be apparent as the image may shift slightly after each scan.

In order to stop the continuous scan, press the Stop button.

- Click Stop once and the scan will stop when the current frame is complete.
- Click Stop twice and it will stop immediately.

If you navigate to a different step, the scan will stop at the end of a frame.

### **Number of Frames**

Enter the number of times you wish the beam to scan the site of interest for image acquisition.

### Frame Time (secs)

The frame time is displayed in seconds. The value of frame time depends on the resolution, speed and mains synchronize if available.

### **FSD Control**

This section appears only if a forward-scatter detector is correctly configured. To control settings for the signals from the forward-scatter detection diodes, click the Settings button to open another dialog.

If you select the Auto checkbox before you start a new FSD acquisition, the software automatically adjusts the signal from each diode for optimal brightness and contrast. The optimized electron image then appears after a delay of a few seconds. When you first start the software, the Auto check box is already selected for you.

#### See Also:

AutoLock on page 133

Scan Image on page 418

### **Scan Image Toolbar**

The Toolbar is located near the top left side of the Scan Image window. Tools are provided to manipulate and annotate the image:



### **Pan and Zoom**

You can move the image using the Pan tool. Use the wheel mouse to zoom in and out.



### **Annotation**

There are five different tools to add annotations on the current image as shown in the screen shot below:





To edit an annotation double-click on it to select it, the editing handles will be displayed. Use the handles to edit the annotation.

To delete an annotation, select it by double-clicking on it and then press the Delete key on the keyboard.

To delete all annotations on an image, choose Select All from the Annotations context menu on the image viewer and then press the Delete key on the keyboard.

#### Information

Select the Show Data Values tool from the toolbar and click anywhere on the image to display the Intensity value at that point.



### **FSE** area optimization

If an FSD system is currently fitted, this tool is available. It helps you to improve the brightness and contrast of any area of the FSD mixed image.



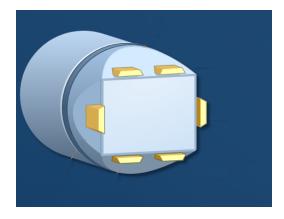
Normally, the software calculates optimized brightness and contrast values for the mixed image over the *whole* area. Some areas of the image might be very dark or very light, so that features within an area of interest might not be well defined.

- 1. Select the rectangle tool from the toolbar.
- 2. On the mixed image, find the area of interest.
- 3. Click and drag to form a rectangle around the area.

When you release the mouse button, the software calculates new values of brightness and contrast, then applies them to your area of interest. If necessary, you can repeat this step or reacquire the image and try again.

### Configuration of diodes on the FSE detector

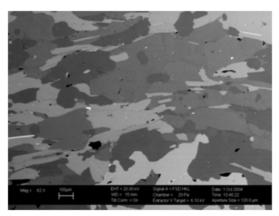
The EBSD detector can accommodate up to six forescatter diodes around the phosphor screen. The diodes can be positioned in a number of configurations because each diode can be removed and refitted.

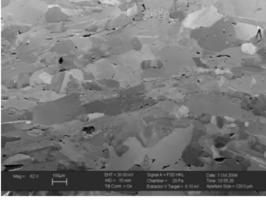


The diode positions provide different signals:

- Upper positions (above the phosphor): density/atomic number (Z) contrast signal.
- Side positions, coupled with the upper diodes to provide a stronger signal.
- Lower positions (under the phosphor screen): orientation contrast signal

The following example is from a polyphase rock sample:





Density/atomic number (Z) contrast

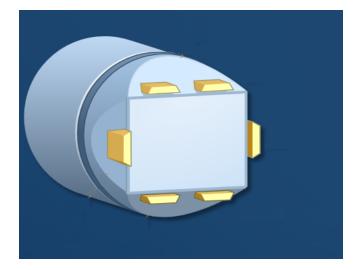
Polyphase rock sample: Orientation contrast

### How to obtain good FSD images

The forescatter detector (FSD) system is a complementary imaging system to the EBSD technique. The FSD system is a backscatter detector that uses multiple silicon diodes around the EBSD detector phosphor screen to produce microstructural images. The same phenomenon that generates a diffraction pattern generates imaging crystallographic orientation contrast. The FSD system provides orientation and phase (density/Z) contrast using up to six silicon diode detectors.

With a highly tilted sample, a typical backscatter detector mounted on the SEM pole-piece yields low orientation contrast. The FSD system has lower diodes and is well positioned for orientation contrast imaging. For some materials, sample topography will be observed where the upper and side diodes are positioned for optimal density/Z contrast imaging. You can enable, invert or disable each diode detector signal to achieve the best gray level image.





A weak signal channel is a common problem with forescatter systems, and can usually be fixed by one of the following changes:

- Increase the probe current or accelerating voltage.
- Adjust the sample-to-detector geometry.
- Check the polish of the sample. If the sample gives good EBSPs, it will generally also give good forescatter orientation contrast (OC) images but not always.
- Change samples. Some materials give better orientation contrast signals than others.

FSD images with strong orientation contrast will yield high-quality EBSPs for EBSD analyses. To optimize your FSD and get the best possible images, you need to consider:

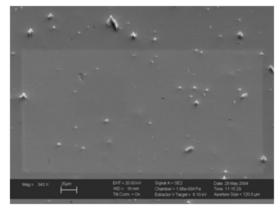
- sample preparation,
- SEM settings,
- sample-to-detector geometry.

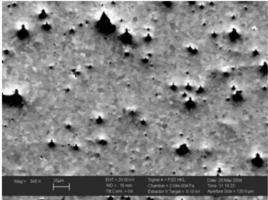
### Sample preparation

The quality of your FSD images is highly dependent on the preparation of the sample's surface. The forescatter signal is not very strong and is easily dominated by topography (roughness). Therefore, sample preparation is critical. For the best forescatter images, the specimen must be completely flat and featureless.

Mechanical polishing with colloidal silica is recommended to remove relief in the sample. It is an easier and similar polishing process across most materials, making it ideal for polyphase samples and harder particles in a softer matrix. With care, a quick electropolish gives the best surface finish without introducing too much topography. However, electropolishing can produce rough surfaces and highlight grain boundaries.

Any surface topography or dirt particle is exaggerated, casting shadows on the surface, affecting the FSD image quality. For example:





SE image of a Mg alloy with dirt particles on the surface

FSD image of the same area showing the effect of the dirt particles

Coating the sample has the same effect, weakening the signal significantly or possibly removing the signal altogether. If coating is necessary, keep the coat thickness very thin (typically 2-5 nm). If the coating is too thin, the conductive material will not be sufficient to dissipate the charge. If the coating is too thick, the signal-to-noise ratio will decrease significantly, resulting with poor EBSPs.

### **SEM** settings

FSD imaging generally requires relatively high probe currents and accelerating voltages. The backscatter diodes are less sensitive at lower accelerating voltages (<5kV), and work best in the range 15-30kV.

The FSD image signal increases significantly when the probe current is increased, and ideally must be above 2-5nA. You might need to increase the probe current for forescatter imaging, and then decrease the probe current for EBSD analyses.

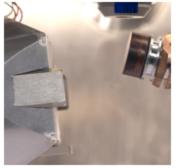
In variable-pressure (low-vacuum) mode, FSD imaging is possible at low chamber pressures (e.g. below 40 Pa / 0.3 Torr).

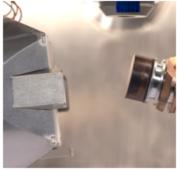
### Sample detector geometry

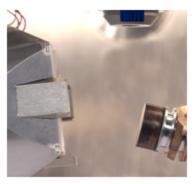
One of the most important factors for getting high-quality forescatter images is the sample-to-detector geometry. The ideal geometry allows all silicon-diode detectors to collect electrons that have been reflected off the surface of the sample.

To achieve the best FSD image, you need to vary the focal working distance and the detector position. If the geometry is incorrect, the signal might be too weak for the diode detectors to be useful even if the sample surface is well prepared.









Sample is too high

Sample is correctly placed

Sample is too low

If the sample is too close to the diode detector, the signal is shadowed by the sample. If the sample is too far from the diode detector, the signal is weak. A small change in geometry can often significantly improve the image quality.

### Collecting an FSE image

1. In the acquisition toolbar (above the electron image and below the Navigator), click Settings.



- 2. On "Scan Image Settings", select your electron image's scan size, dwell time and input signal.
- 3. At "Input Signal", tick a box to capture the forward scatter electron (FSE) image.
- 4. Enable Auto to automatically optimize the gray levels of the FSD diode channels.
- 5. Click Start to collect the electron images.

After your FSE image has been collected, the data tree creates the FSD Data folder, where it stores all data related to your FSD system.



The FSD Channels folder stores each collected image channel. The number of channels on the data tree depends on the physical number and position of FSD diode detectors for your FSD system.



### See Also:

Scan Image - Settings on page 421

Data Tree on page 85 (FSD data)

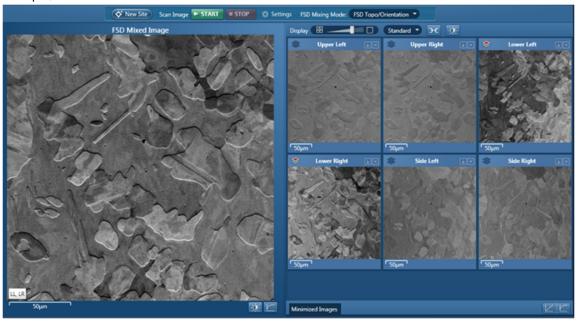
Scan Image on page 418

### Optimizing the FSD mixed image

1. On the Data Tree, highlight the FSD Mixed image.



The FSD mixed image and FSD image channels become visible in the image viewer. The FSD mixed image displays short labels for the selected FSE image channels. For example, LL indicates Lower Left.



2. Select a mixing mode:



- FSD Z Contrast uses the upper and side FSD detector channel images.
   Select this mode if you are interested in seeing a density/atomic Z contrast signal.
- FSD Topo/Orientation uses the lower FSD detector channel images. Select this mode if you are interested in seeing an orientation contrast signal.
- Custom. Include and exclude FSD detector channel images of your choice.
- 3. In the image viewer, do any of the following to obtain a good image:
  - Include or exclude individual channels to be displayed in the FSD mixed image.
  - Manually optimize the gray image levels to achieve the best contrast and brightness in each channel.
  - Add color to the lightest and darkest point in the channel image.

# Optimizing the signal from a single FSD diode

- 1. In the FSD image, find the darkest and lightest features of the image.
- 2. Below the image, click the cog icon to open a dialog box:



- 3. Adjust the Offset control to make the darkest features in the image appear black.
- 4. Adjust the Gain control to make the lightest features in the image appear white.
- 5. Continue making adjustments as in the previous steps until you obtain a good image.
- 6. Adjust the Weight control to include this image in the Mixed image.

### How the FSD gain and offset controls work

The FSD diodes generate a range of signals as they detect electrons emitted from the sample during scanning. The signals are processed by electronics before reaching the software for further processing.

Depending on conditions of the microscope and the sample, the *range* of diode signals might be small. When the image is subsequently viewed, its correspondingly small range of grays might make some features difficult to see. Although you can adjust the contrast and brightness using the software, the results might be disappointing.

For a clear view of the features in the final image, you need a wide range of signals from the detector to give with a wide range of grays - from nearly black to nearly white.

For example, the diode output gives only a small range of signals, somewhere between zero and a maximum.



The equivalent range of grays is small:



The electronics can apply an offset, forcing the same small range of signals to start at zero.



Although the equivalent range of grays is still small, it now originates with black:



The electronics can also apply a gain to increase the range of signals:



This increases the equivalent range of grays, such that the maximum signal is equivalent to white:



Starting from the original narrow range of signals, the electronics has produced a wider range of signals, which start at zero, and reach the maximum. The small range of grays is now fully black to white.

### **FSD** diode controls

You can generate a FSD mixed image by enabling the required diode detector channels. For example, you can choose the diode images to add to the mixed image, the color for each image, and their intensities. The controls are around the image from each diode.



Control	Description
<b>\$</b>	Adds or removes an image in the mixed image.
	Your changes to the number of images might affect the choices in the Mixing Mode dropdown list.
(Title)	States the source of the image such as the Top Left or Bottom Right diode.
¥	Minimizes an image.
	This is useful if you have too many images in view. The image moves to the Minimized Images tab, below the displayed images. The mixed image is not affected.
	Deletes an image from the mixed image completely.
	To restore a deleted image, start a new image acquisition.
<b>*</b>	Restores a minimized image to its normal size. The minimized images are on the Minimized Images tab, below the displayed images.
-100 ———————————————————————————————————	Sets the weighting of the image within the mixed image. For example:
	100 - Uses the full signal from the diode.
	0 - Does not use the signal from the diode.
	-100 - Uses a full, inverted signal from the diode.
100 100 100	Opens a dialog where you can adjust the gain and offset of the signal from the diode. This control is effective only while data is being acquired.
	Adjusts the level for the darkest and brightest parts of the image.
* *	Offers a choice of color for the image.

Control	Description
Context menus	Right-click the image to display context menus for copying, exporting and printing images.
	Exported data includes the mixed image and the images from each diode. This data is not supported by some earlier versions of the software.
	Use the mouse wheel to zoom in and out of the image.
Minimized Images	Any minimized images are stored on this tab, which is below the displayed images.
	Allows you to change the Brightness/Contrast and Gamma for all the images. The Auto Brightness button optimizes the images to give the best Mixed Image. The Auto Gamma button enables you to see all the image data including background noise.

### **FSD Control Dialog**

This dialog appears when you click FSD Settings in the Scan Image settings.

Here you can select the signals from preset combinations of diodes, or make your own selection from any number of diodes, to construct a forward-scattered image. The choices on the dialog depend on which diodes are fitted on the detector. Use the following controls:

Control	Description
FSD Topo/Orientation	Uses the signals from the bottom two diodes to accentuate topography and orientation.
FSD Z Contrast	Uses the signals from the top two diodes to accentuate the atomic-number contrast.
Custom	Allows you to create any combination of signals using the controls labeled: + - O
+	Uses the signal from the diode.
-	Uses an inverted signal from the diode.



Control	Description
0	Does not use the signal from the diode.

Your selections are retained in your user profile.

### **Optimize Pattern**



This step contains the necessary functions to allow optimal EBSP acquisition and processing.

### **Monitoring the EBSP**

The electron image shown in the top left quadrant is the image collected in the Scan Image step. As you enter the Optimize Pattern Step the position beam tool is highlighted on the toolbar. Positioning the beam around the image allows you to check the quality of the EBSPs from different points on the specimen. The EBSP is shown in the top right quadrant (unprocessed EBSP). When the tool is selected the beam can be controlled by the mouse as well as by the keyboard (Shift will give a coarse shift and Ctrl will give a fine shift with the arrow keys). Alternatively, press Center to position the beam at the center of the image. Press the Off button to release the beam.

### **Adjusting the Camera Settings**

Select the binning and gain for the camera, suitable for the analysis, and then set the exposure time to get suitable illumination. Alternatively press Auto to automatically adjust the exposure time to achieve a signal strength between 85 and 95 (see below).

The Nordlys detector (whether NordlysNano or NordlysMax) allows you to adjust the quality of the diffraction pattern to suit the particular type of analysis.

### **Binning**

By clustering groups of pixels (binning), it is possible to collect lower resolution EBSP's at higher speeds. Depending on the detector used, various binning options are available. The table below illustrates the general trend for binning levels and when to use them:

Binning	Suggested Use
None	High resolution EBSPs for posters and publications.
2x2	Regular EBSD analyses where accuracy is more important than extreme speed – e.g. phase identification.



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Binning	Suggested Use
4x4	Regular EBSD analyses where speed is an important issue (e.g. routine grain size & texture analyses).
8x8	High speed analyses where a slight loss in accuracy is not important (e.g large area texture analyses).

#### Camera Gain

Adjust the gain level to suit your experiment. High gain will increase the sensitivity of the camera but may introduce more noise as it is the total signal (signal + noise) that is amplified. The noise level indicator (see below) is provided in the application to allow you to look at the effect of adjusting the gain on the quality of unprocessed EBSP and hence which setting works best for your specimens.

### **Exposure Time**

Once you have selected the level of binning, enter a camera exposure time (also known as the integration time). Even with low signal strength values, the software copes very well with such EBSPs.

If the exposure time is set too low, there will be insufficient signal in the image and it will appear too dark. If the exposure time is set too high, part of the image may be over-saturated forming a white area.

The exposure time (for a given binning level) will depend on the probe current you have used to collect your electron image. With some microstructures, it will be necessary to work with low probe currents to achieve the spatial resolution necessary to resolve the desired features. In this case you will need to adjust the exposure time and binning accordingly.

### **Histogram of the Unprocessed EBSP**

This histogram allows you to look at the distribution of pixel intensity in the unprocessed EBSP. You may use the mouse wheel to adjust the size of the histogram.

### Signal Strength

The signal strength is a value which lies between 0-100. It corresponds to the position of where the end tail of the histogram lies on the x axis of the histogram. We recommend that you use a value between 85 and 95. If the signal strength is too low or too high, it will appear

in the red region. If the signal strength is an acceptable value, it will appear in the green region.

Anything above 95 is strongly not advised as it could mean over exposure.

#### Noise Level

This is a useful tool to monitor the noise level as you change parameters such as gain, binning and exposure time. The value is the percentage of noise in the image where 100 corresponds to no signal but all noise whereas 0 corresponds to all signal.

#### • Time per Frame

The time per frame is the time taken for the camera to read out one EBSP. Depending on the camera settings this time can differ slightly from the requested integration time.

### **Image Processing Settings**

Initially, any small clusters of pixels arising due to defects or scratches on the phosphor will be masked out and disregarded in further processing of the EBSPs such as background correction. Background correction methods as well as frame averaging are then available to use to improve the quality of the EBSPs. The processed EBSP is displayed below the unprocessed EBSP to show this improvement. Once the selection is made, these settings will be used whenever an EBSP is acquired. It should also be noted that automatic stretching of the processed image takes place in order to make full use of the dynamic range in the image.

### **Background method**

An EBSP consists of a series of relatively weak Kikuchi bands on a strong, non-linear background. Removing this background produces a much clearer EBSP. When a background correction method is applied, the EBSP pattern is divided by a model of the background, and the ratio that is obtained is then multiplied by a pixel whose value is mid range. Note that division is used because the strength of the pattern signal correlates with the variation in the background.

There are two methods commonly used when applying a background correction 'Static' and 'Dynamic'.

- A static background is a model of the background generated by averaging a large number of pattern images based on the signal from many different orientations.
- A dynamic background is a model of the background extracted from and used on the same pattern image. It can be derived by fitting a suitable mathematical model of the background to the image, or by filtering out the high frequency pattern, taking the low frequency residual as the background.

### Static background

In order to use a Static background, press the Collect button and select the number of frames. A higher number of frames will give cleaner EBSPs but will take longer to collect. Note that the default value is 64 frames. You are then able to use the Static background correction by checking this option.



The simplest way to define a background is to rapidly scan an area of the specimen that contains many grains and while acquiring an average EBSP. Backgrounds for single crystals may be produced by heavily defocusing the electron beam on the specimen.

### **Advantages:**

- A static background can compensate for sensitivity variations even scratches in the phosphor.
- The background does not have to be calculated for each EBSP acquired.

### **Disadvantages:**

- The background is likely to change position and magnitude when the
  - Position of the detector changes.
  - Position of the Specimen changes in the chamber.
  - Mean atomic number (Z) changes significantly in a multi-phased material.
  - Position of the electron beam changes on the specimen surface at low magnification.
  - Accelerating voltage is changed.
- Stage tilt angle is changed.
  - Note that any changes to gain, binning or exposure time mean that a new static background should be collected.

### **Auto Background**

Check the Auto Background routine option to background correct the acquired EBSP's on the fly.

The Auto Background process is very efficient at preserving the original shape and features of the data such as the Kikuchi bands whilst removing the unwanted background.

### **Advantages:**

- Seamless process requiring no user intervention.
- Independent of acquisition conditions.

#### **Disadvantages:**

- The dynamic correction is, per EBSP, slower than the static one because the background has to be calculated for each pattern.
- It cannot compensate for local irregularities in the sensitivity of the phosphor.

#### Frame Averaging

Enter the number of frames you wish to acquire. These frames are then averaged to reduce the noise level in your EBSP. A higher number of frames will give cleaner EBSPs but will take longer to collect. The time per frame displayed under the histogram includes the frame averaging.

Please note that there is a slight difference between the frame averaging that is applied to the live monitoring of EBSPs and the frame averaging that is applied when snapping an EBSP or doing mapping. The frame averaging method for monitoring uses a rolling average buffer so that the view can be refreshed at the rate of the camera. Other EBSP acquisitions will collect precisely the number of frames from the camera, as specified in the "Frame Averaging" setting for each beam position, and average those frames together to form a single processed EBSP.

### **Magnetic Field Correction**

This section is available only if this feature has been licensed and activated. To setup the correction, click Setup and use the dialog that appears. To apply the correction, select the check box. If the correction has been applied to maps or patterns, it is indicated in their details.

#### See Also:

Optimize Pattern - Toolbar on page 442

Context Menus - Image Viewer on page 157

Brightness, Contrast and Gamma Controls on page 214

Magnetic Field Correction Setup below

### **Magnetic Field Correction Setup**

This window is launched from the Optimize Pattern step. A correction factor is calculated, which will subsequently be applied to all distorted EBSPs if enabled.

A pair of distorted and undistorted EBSPs need to be collected:

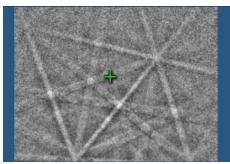
- From as near the same place as practically possible,
- From the same phase and orientation,
- From the center of the field of view,
- Under the same operating conditions such as acceleration voltage (kV), insertion distance (ID) and binning.

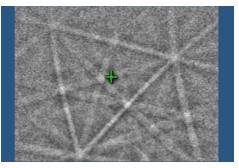
The correction factor that you acquire here is applicable only for the current conditions of working distance (WD), kV and ID, and only during the current session.

We recommend that you collect this data from a silicon single crystal specimen, if possible. Keep the specimen at the same position (or same grain if it is a polycrystalline material) if at all possible to keep the same geometry conditions. If a single crystal is used, there is then therefore no reason to move it.

- 1. On the SEM, set the mode with the field mode on.
- 2. Degauss the SEM.
- 3. Without moving the specimen, focus the image.
- 4. In the Distorted EBSP area, click SNAP to snap the EBSP. Information about kV, WD and ID appears in the status bar below the EBSP.

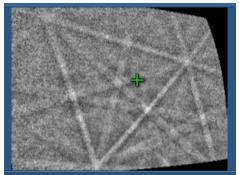
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  - 5. Click Save As and save the .oimfc file to a suitable location. This saves the distorted EBSP into the .oimfc file.
  - 6. On the SEM, set the field-free mode on.
  - 7. Without moving the specimen, refocus the image.
  - 8. In the Undistorted EBSP area, click SNAP to snap the EBSP. Information about kV, WD and ID appears in the status bar below the EBSP. Note that the reported WD in the field-free mode might be different compared to the WD displayed below the EBSP in the field mode.





The undistorted and distorted EBSP

9. Click Calculate. This calculates values for the dipole field strength and an X-offset. A correction factor based on these values is then applied to all patterns if the option is selected. The corrected image appears below the undistorted image and should closely resemble the undistorted image. Note that the black parts in some of the corners of the EBSP are ignored by any pattern solving and will not affect your analysis.



The corrected EBSP

10. Click OK to return to the Optimize Pattern step, where you have the option to apply the correction factor to all subsequent EBSPs.

### **Additional notes**

If this correction has been applied to your data, you can see this by checking the details of the data.

During the calculation, if both EBSPs are identical (for example the distorted EBSP has been snapped twice in error), the software reports an error and a correct pair of EBSPs should be collected.

## **Optimize Pattern - Toolbar**

Tool	Description
*	Moves the image. Click the Pan tool, then click and drag the image. Use the mouse wheel to zoom in and out.
T	Adds annotations to the current image. The tools include Caliper, Angle, Text, Rectangle and Ellipse.
	Click the tool, then click on the image to add annotation. For example, to add text, click the Text annotation tool, then click on the image where you want the text and start typing.
	To delete annotation, click it to select it. Press the Delete key on the keyboard, or use the context menu to delete the annotation.



Tool	Description
*	Positions the beam. Click the tool, then click on the image to position the beam around the image and check the quality of the EBSPs from different points on the spec- imen. The EBSP appears in the top right quad- rant (unprocessed EBSP).
	You can control the beam by the mouse. Alternatively, you can use the arrow keys on the keyboard. Then you can use the Shift key for a coarse shift, and Ctrl for a fine shift.
	The On, Off and Center buttons are linked with this tool. If you click On, the tool is automatically selected.
i	Shows the intensity and beam position for any pixel in the image.
	Click the tool and then hover on the image. The values appear as you move from pixel to pixel.

### **Optimize Solver**



This step contains the necessary functions to allow you to acquire, index and refine EBSPs.

### **Positioning the Beam**

As you enter this step, the 'Acquire an EBSP from a point' tool is selected by default. When this tool is selected the beam can be positioned on the image and its position can be controlled by the mouse as well as by the keyboard (Shift will give a coarse shift and Ctrl will give a fine shift). Note that you can also center the beam by pressing the Center button.

Once the beam is positioned, release the mouse and you are ready to snap a pattern. As described below, if Auto is checked, a pattern will automatically be snapped but if it is not, you should then press the 'Snap' button.

If you wish to release the beam back to SEM control, press Off.

In addition to acquiring a live EBSP by positioning the beam, you may wish to optimize the solver settings based on EBSPs from previously acquired EBSD Maps. It is often very useful to check the solver settings before setting up an area for reanalysis. In the same manner as described above, select the 'Extract EBSP from EBSD Map' tool on the toolbar. This tool will allow you to place the cursor on the map and extract the EBSP from the specified position. When the cursor is placed the EBSP is automatically extracted. If the Auto function is enabled then the band detection and indexing will automatically take place.

### Band detection and Indexing of the Processed EBSP

#### **Auto**

By enabling Auto, the system will automatically snap a live EBSP, detect the bands and try to index with the selected settings. It will also automatically update whenever a change is made to beam position, the EBSP, the detected bands or the analysis settings and phases.

If you do not select Auto, you must press Snap, Detect and Index in order to go through the steps of snapping a pattern, detecting the bands and indexing the pattern.

#### **Load EBSP**

This function allows you to load an EBSP from file together with its calibration and acquisition conditions. You will need to fill in the acquisition conditions when loading the EBSP in order for the software to be able to analyze the EBSP.

Depending on the calibration information available with the EBSP, the calibration conditions will either be those based on the system calibration or a previously acquired calibration file. In the latter case you will need to add additional information into the required fields.

### **Settings**



Under Settings, adjust the Hough resolution, number of bands and band detection method to ensure you aid the identification of the acquired EBSP. You should also ensure that you have included the phases you wish to use and their corresponding number of reflectors.

#### **Band Detection Area**



Selecting the 'Edit Band Detection Area' tool on the Optimize Solver toolbar will allow adjustment of the band detection area. When the tool is active the Area of Interest will be displayed on the EBSP and by use of the mouse, the area can be moved or resized.

#### **Edit EBSD Bands**



The application will automatically detect a number of Kikuchi bands, depending on the number of bands you have set it to detect. However, you have the ability to edit any existing band, delete bands or manually add bands. In order to interact with the detected bands select the 'Edit EBSD Bands' tool on the Optimize Solver toolbar. This will allow you to delete bands, draw new bands or modify existing bands.

#### Context menu

Right click on the EBSP to access various options in the context menu such as blinking the solution and removing the display of detected bands. It is also possible to switch on/off the display of the Pattern Center, marked with a green cross. Further options are available under Preferences from the main menu.

#### Status bar beneath the EBSP

## **Cycle Times**

Two cycle times are shown below the EBSP. The Analysis time is the time it takes for the software to perform the analysis of the acquired EBSP and the Detector time is the time it takes for one EBSP to be acquired by the detector and transferred to the PC.

The Analysis time will depend on the selected analysis parameters, and the Detector time will depend on the selected detector settings. In order to acquire data at high speed it is important to keep both the Analysis time and the Detector time low. If the analysis time is highest then the system will acquire at a lower speed than indicated by the detector time.

## **Number of Bands**

This is the number of bands detected on the EBSP.

#### **Band Contrast**

Band contrast is an EBSP quality number. The higher the number, the better the EBSP quality.

## **Band Slope**

Band slope is an EBSP quality number. The higher the number, the better the EBSP quality.

# **Hough Viewer**



The Hough space display, accessed by pressing X, shows the EBSP after transformation into Hough space. The Hough transformation converts Kikuchi bands into peaks which are used to find the edges or centers of bands during automatic band detection. Depending on the number of bands you have entered under Settings, these maxima will be marked with numbers to show which ones correspond to the detected bands. The highest number corresponds to the strongest peak. You may toggle between the two icons on the Hough Space display window to switch between showing all bands or currently selected band.

Move the mouse over Hough Space and the corresponding location on your EBSP, will be displayed. Hover over the maxima to see how it relates to the band in the EBSP.

#### **Solutions**

This area shows the results of the EBSP indexing process. A simulation of the selected solution will be overlaid on the EBSP allowing a visual comparison between the simulation and the original EBSP. Note that the order of the solutions can be changed if you select 'Use Band Widths in Sorting' under Settings in the Processed EBSP area if you are using the edges of the bands during band detection (See later).

The following parameters are displayed for each solution:

#### **MAD**

MAD or Mean Angular Deviation is the goodness of fit of the solution. The smaller the number, the better the match between the detected Kikuchi bands and the simulation. A number less than 1° is acceptable for most systems.

If Advanced Fit has been switched on, then the Advanced Fit index ("AFI") will also be displayed instead of the MAD. This, like the MAD value, is also a goodness of fit parameter. However, it is independent of the band detection process, and larger AFI values indicate better solutions. The AFI value is also affected by the quality of EBSP.

#### Orientation

These are the three Euler angles that describe the orientation of the crystal lattice as determined by the indexing procedure.

# Refinement



The refinement routine tries to compensate for any instability in the readout of WD or ID. You may choose to refine your calibration based on a selected solution. You should ensure that this solution is correct as this refinement will potentially produce an offset in Working Distance (WD) and Insertion Distance (ID) that will subsequently be applied when indexing any subsequent patterns.

## **Phases for Acquisition**



You should ensure that you have included phases you wish to solve with within the Phases for Acquisition area. If you wish to add or delete any phases, this can be done in the Describe Specimen Step. However, you can include or exclude individual phases from the list within the Optimize Solver step or change the number of reflectors and color can be set per Phase in this step.

In addition, details about any highlighted phase can be viewed in the Phase area by selecting the 3D Phase View, Phase Details and Reflectors tabs.

#### See Also:

Optimize Solver - Settings below

Context Menus - Image Viewer on page 157

Brightness, Contrast and Gamma Controls on page 214

# **Optimize Solver - Settings**

You can change the settings for the solver on this dialog.

Field	Description
Detect	Select Edges when the edges of the Kikuchi bands are sharp and clearly visible. The band edges are detected based on an initial detection of the peak position in the Hough space and then fine tuning the edge positions based on actual EBSPs.  Select Centers for blurred or narrow bands.

Field	Description
Number of Bands	Determines the number of bands that will be used for initial indexing.
	Increase the number of bands used and this should aid in finding the right solution. It is often helpful to look at the Hough and the peak intensities as this will assist in choosing the number of bands.
	The relation between the number of bands and the number of reflectors is critical in order to get the best performance of the system. It is therefore important to optimize these two numbers. Note that too many reflectors can result in more than one solution as can too few bands. Experiment with lowering the number of reflectors to optimize your settings.
Hough Resolution	Sets the radius of the area of interest in Hough space in pixels. Try different Hough Resolutions to find the best results for your specimens. Use the following guidelines:
	<ul> <li>40-45: For fast data collection, where small angular errors (&lt;2°) are not a problem.</li> </ul>
	<ul> <li>60-65: A good compromise between speed and angular res- olution.</li> </ul>
	<ul> <li>75-80: For more accurate, but slower indexing.</li> </ul>



Field	Description
Use Advanced Fit	Improves the angular accuracy of the indexing procedure, but significantly slows down the indexing process. Select an Advanced Fit Level (1 is low, 4 is high). Advanced Fit goes back to the full EBSP and optimizes the solution fit.
	For standard use, do not select Advanced Fit.
Band Detection Area X and Band Detection Area Y	Shows the X and Y coordinates of the center of the Band Detection Area. To move the area, select the 'Band Detection Area' tool on the Optimize Solver toolbar and drag the displayed area with your mouse. As the area moves, the coordinates change automatically.
	Alternatively, you can type the coordinates here.
Band Detection Area Radius	Shows the radius. To resize the area, select the Band Detection Area tool on the Optimize Solver toolbar and drag the displayed perimeter with your mouse.
	Alternatively, you can type the radius here.
Apply Refinement	Applies a previous refinement to the solver.

# **Acquire Map Data**



A Map is a regularly spaced grid of automatically acquired and solved EBSPs. In this step you can setup acquisition of EBSD or combined EBSD & EDS data from either the full area or a selected area.

Generally, candidate phases are selected before collecting data. This enables EBSPs to be solved during acquisition. However, you may choose to store EBSD patterns without solving them in order to analyze them later. Once this saving option is selected under Settings, you may proceed to acquire Map data without having to first select the candidate phases.

# **How to Acquire Maps**

The default way of selecting an area to map is by using the Map Area Calculator tool which is selected by default as you enter this step. This tool allows you to link the acquisition time, step size and area of a rectangular map region.

Alternatively, to acquire map data from a region, select the required map acquisition tool from the standard region Rectangle, Ellipse and Freehand tools available from the toolbar. If you wish to do a full area map, select any of the region tools and press start. If you wish to select a reduced area map, select one of the standard region tools, click on the image and drag with the left mouse to outline a region on the image. Maps will then be automatically acquired from the scanned region on releasing the mouse.

## **Using The Map Area Calculator**

If you cannot see the calculator above the image, click the Map Area Calculator tool icon on the toolbar on the left of the image:



The shaded box on the electron image shows the location and size of the area to be mapped. The Map Area Calculator tool works with the settings that appear above the image. You can position and resize the area using your mouse and the following controls.

Icon	Description
<b>a</b> Duration	Locks the acquisition time. While you define the area, the calculator changes the step size. While you define the step size, the area changes to keep the time constant.



Icon	Description
3 Step Size	Locks the step size. While you define the area, the calculator changes the duration. While you define the duration, the acquisition time changes to keep the step size constant.
a Area Width	Locks the width of acquisition area.  While you define the acquisition time, the calculator changes the step size.  While you define the step size, the acquisition time changes to keep the area constant.
<b>□</b>	Fixes the width-to-height ratio of the map area. Any change in one direction automatically causes a change in the other direction. If you maximize the area to be mapped, the current aspect ratio setting is ignored, and then recalculated for this new area.
	Fills the field of view with as many points as possible using the defined step size.
	Repositions the area to be mapped to the center of the field of view.
<b>*</b>	Moves the acquisition area. Click in the middle of the area and move the mouse.
⇔ Ĵ	Resizes the area. If you have not locked the area, you can resize it by clicking and dragging the edges.

Select the acquisition parameters from the Settings cog on the acquisition toolbar and click Start to acquire the map.

# Defining a map region using the standard region tools

By default, map data is acquired from the full area. To acquire map data from a region:

1. In the toolbar on the left of the layered image, click an icon, then outline the region.

Icon	Description
	Outlines a rectangular or square feature or area on the image.
8	Outlines an irregular shaped feature on the image.
8	Outlines an oval or elliptical feature on the image.

- 2. Click and drag the mouse on the image to outline the region.
- 3. Release the mouse button.

Map data will be acquired according to the current resolution. If you wish to use a different step size to the one determined by the resolution, type in the required step size.

# **Map Display**



If you have chosen to view the maps in addition to your acquisition area, you can see the layered image, maps (both EBSD and EDS, if selected) and electron images. You can also choose how to view your data from Standard, Interactive or Summary view from the drop-down list above the maps.

Map data will be acquired according to the resolution currently set under Settings. If you wish to use a different step size to the one determined by the imaging resolution, type in the required step size.

#### **Layered Images**

Layered images are composite images that show you the X-ray or EBSD maps overlaid on the electron image. If you chose to acquire both EDS data and EBSD data, two layered images are automatically created:

- EDS layered image consists of an electron image and X-ray maps whose selection is defined according to the current settings.
- EBSD layered image by default, consists of the electron image and the Phase Color Map if you have more than one phase. However you can change this configuration. If you have only one phase, the IPF Z is shown.

You can manipulate the layered image in several ways:

You can add or remove a layer from the layered Image, minimize some maps, or delete a map from the analysis completely. In the case of EDS, if you remove an X-ray map, this element will not be identified automatically (by AutoID) and will be excluded from the current analysis. If an element is present in a specimen, deleting or excluding it will affect the TruMap results.



You can choose the color for your maps, adjust intensities and decide which maps to add to the layered image. The AutoLayer feature automatically scales and colors all the maps, and in the case of EDS, selects the best maps to provide an effective color image that delineates regions of different composition. Maps are automatically corrected for brightness, and those that show similar structure are assigned the same color. Maps that are very noisy are shown in gray. The most significant map for each assigned color are added to the layered image.

# **Map Tools**

You can change the appearance of the maps and layered image with these tools.

Icon	Description
	Sets the number of maps per row in the Standard and Interactive displays.
Standard •	Offers a choice of map display:  Standard Interactive Summary
	Links images for manip- ulation of all layers using the Pan or Zoom controls.
	Unlinks images. You can manipulate individual layers using Pan or Zoom controls.
	Changes the brightness and contrast of the currently selected image or map.
	Adds or removes a map in the layered image. The Layered Image icon is in the top left hand corner of the map. A symbol representing the map appears on the layered image.

Icon	Description
	Minimizes a map. The Minimize icon icon is in the top right hand corner of each map. This is useful if you have too many maps in view. The map moves to the Minimized Map tab, below the displayed maps.
	Deletes a map from the analysis completely. This icon in the top right hand corner of each map.
	Restores a minimized map to its normal size. The mini- mized maps are on the Mini- mized Map tab, below the displayed maps.
Map   TruMap	Sets the mapping mode for X-ray Element Maps.
	Automatically adds maps to the layered image. This is the Autolayer feature.
	Changes the brightness and contrast and gamma for all the maps.

# **Data from Map Acquisition**

Once data acquisition starts, the Data Tree is populated with new items as shown below:





# **Electron image**

This can be a secondary electron image (SE) or backscatter electron image (BSE). For EBSD analysis, it is generally a forescatterred electron image (FSD) depending on the detector hardware installed.

## Map Data

EDS and EBSD map data are in their respective Map Data folders. As data acquires, EBSD maps acquire into the EBSD Data folder. If you selected the option to acquire EDS data in addition to your EBSD data, EDS maps acquire into an EDS Data folder.

The EBSD Data folder contains the following EBSD images:

- Band Contrast
- Phase Color
- Euler Color
- IPF X Color
- IPF Y Color
- IPF Z Color

The EDS Data folder contains:

- Map Sum Spectrum
- X-ray Element Maps

# X-ray Element Maps

Two modes of mapping are available, Window Integral Maps and TruMaps. Click the appropriate button at the bottom of the maps to select the mapping mode.

Standard Window Integral maps (counts in the energy window) are acquired for the element list chosen for analysis. These are raw X-ray maps, which are not corrected for background or peak overlaps.

You can process the map data as TruMaps, which are corrected for background and peak overlaps.

# **Post Acquisition Image**

You can acquire a new scan of the electron image with the current settings after the map is finished by selecting this option under Map acquisition settings. This is useful for checking drift of the specimen.

# Tips

Hover the mouse over the Map Data entry on the Current Site to see the acquisition progress and information such as speed and hit rate.

To delete a map from the analysis completely, right-click it to access the context menu on the Data tree or Current site.

## **Reanalyzing Map regions**

If you have acquired an EBSD Map with stored EBSPs, you can reanalyze a map region with new settings. Setup the required phase and solver settings (in previous steps), then use one of the reanalyze tools to select a smaller area.

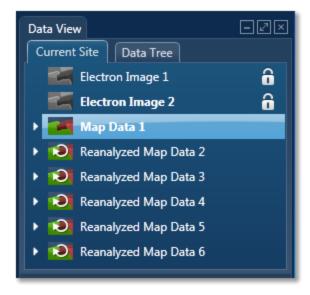
1. In the toolbar on the left of the layered image, click an icon, then outline the region.

Icon	Description
	Outlines a rectangular or square feature or area on an image.
	Outlines an irregular shaped feature on an image.
8	Outlines an oval or elliptical feature on an image.

2. Click and drag the mouse on the image to outline the area.



3. Release the mouse button. The map data will be reanalyzed from it. Reanalyzed map data is automatically stored in the data tree as shown below



#### See Also:

Context Menu - Map Viewer on page 223

Acquire Map Data - Settings below

Display Modes and Map View Settings on page 460

AutoLayer on page 222

Storing EBSD patterns without solving on page 459

# Acquire Map Data - Settings

#### Resolution

The resolution of a map is defined as the number of picture points or pixels along the x and y axes e.g.,  $256 \times 256$ ,  $512 \times 512$  or  $1024 \times 1024$ . The quality of the image improves as the resolution at which an image is acquired is increased. A microscope monitor/CRT is usually a rectangular display (rather than square), so the resolution is displayed as a rectangle i.e.,  $256 \times 200$  in order to take into account the aspect ratio. The y dimension is set at installation, when imaging is calibrated and it will vary from system to system.

Select a resolution for Map acquisition from the following options available

64 x 64

128 x 128

256 x 256

512 x 512

1024 x 1024

2048 x 2048

#### Step Size (nm)

This is the size of the spacing between beam acquisition points. If you wish to use a different step size to the one determined by the imaging resolution, type in the required step size. The software will not permit the step size to be smaller than the step size corresponding to  $4096 \times 4096$  resolution. Similarly it must be larger than the step size corresponding to  $64 \times 64$  resolution.

#### **Store Patterns**

During map acquisition, you can choose the storage conditions for the EBSD patterns. Select an option:

- All Patterns solves and stores EBSPs in the project.
- Zero Solutions stores only those EBSPs where no solution was found.
- All Patterns without Solving Stores all the EBSPs s but they are not solved.

In addition, you may select the type of Storage Format: compressed or uncompressed. Note that storing the EBSPs significantly increases the file size, however it is necessary for later reanalysis of the Map Data.

## **Include EDS**

Check this option if you wish to acquire EDS data with your EBSD Data.

If selected the EDS mapping settings will appear.

## **Number of Channels**

Select number of channels from the drop down list of Auto, 1024, 2048 or 4096 (4K) with which you wish to display the spectrum. The number of eV/channel will depend on both the energy range and the number of channels you select:

Energy Range (keV)	Number of Channels	eV/channel
0-10	4096	2.5
0-10	2048	5
0-10	1024	10
0-20	4096	5
0-20	2048	10
0-20	1024	20
0-40	4096	10
0-40	2048	20
0-40	1024	40



In the Auto mode, the software checks for the energy range selected and sets the appropriate number of channels.

## **Energy Range (keV)**

Select a spectrum energy range from the available options of Auto, 0-10, 0-20 or 0-40 keV from the Energy Range drop down list.

An appropriate energy range should be selected in conjunction with the current microscope accelerating voltage. If the accelerating voltage is above 10 kV, in order to view lines which may be excited above 10 keV, the 20 keV range should be chosen. Below 10 kV, it may be more appropriate to choose the 10 keV range since no lines above 10 keV will be excited.

In the Auto mode, the software checks for the accelerating voltage set on the microscope and selects a suitable energy range in the software.

#### **Process Time**

Select the Process Time from the drop-down list of Process Times: Default and 1 to 6. The Process time is the length of time spent reducing noise from the X-ray signal coming from the EDS detector during processing. By selecting different Process times it is possible to reduce differing amounts of noise. The longer the Process time, the lower the noise.

If noise is minimized, the resolution of the peak displayed in the spectrum is improved, the peaks are narrower and it becomes easier to resolve the peak from another peak that may be close by in energy.

If Default is selected, the Process Time is automatically set to a suitable value.

# **Post Acquisition Image**

If this option is selected, a new scan of the electron image with the current settings after the map is finished can be acquired. This can be used for checking any drift of the specimen.

**Exporting Unprocessed Patterns** 

# Storing EBSD patterns without solving

You have the option to store EBSP patterns and solve them later using the reanalysis tools.

- 1. In the Acquire Map Data toolbar, click Settings.
- 2. In the dialog, select "Store Patterns".
- 3. At "Storage Conditions", select "All Patterns without Solving".
- 4. Select a storage format.
- 5. Close the Settings dialog.
- 6. In the toolbar, click Start.

Map data is acquired and an EBSD data folder appears in the Data Tree. However, the folder does not contain images such as Band Contrast and Layered images, nor details such as the solver settings and hit rate because no solved patterns have been stored.

Note that you cannot export this project data to CHANNEL5 for analysis because the project contains no solved data.

## See Also:

Acquire Map Data - Settings

# **Display Modes and Map View Settings**

The settings are described below.

# **Display Modes**

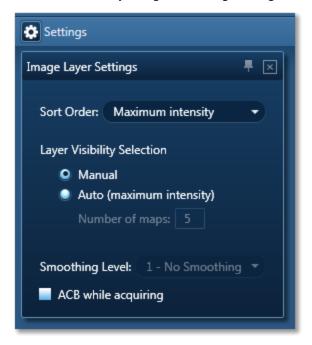
You can view maps in several different modes, using the drop-down list on the Display tool-bar:

- Standard
- Interactive
- Summary

In the Summary view, you can see details of the energy window and X-ray line used for each EDS map if you have selected to acquire EDS maps with your EBSD data. Other details include the Layer Name, Map Color (where appropriate) and whether the map has been selected for the Layered Image.

# **Map View Settings**

You can manipulate and view the data by using the Settings dialog:





Field	Description
Sort Order (EDS)	Sorts the map in several ways:  Alphabetically  By atomic number  By maximum intensity in map - sorts on the value of the bright- est pixel in cps.
Layer Visibility Selection (EDS)	Selects how layers are selected in the layered image:  Manual - you select the X-ray maps to include in the layered image.  Automatic - the software chooses maps with the maximum intensity up to the number that you type here.
Smoothing Level	Filters out some noise by applying a smoothing level. If there is not enough data, the maps might contain a lot of noise, which masks the distribution of the elements. This operation applies a low pass filter to an image to smooth the data.
	The smoothing level, low pass filter uses the following 3x3 kernel:  1/9 1/9 1/9  1/9 1/9 1/9  1/9 1/9 1/9  The smoothing level, low pass filter uses the following 5x5 kernel:  1/25 1/25 1/25 1/25 1/25  1/25 1/25 1/25 1/25

Field	Description
ACB while acquiring	Applies automatic brightness or automatic gamma to maps during acquisition depending on your previous selection of Auto Brightness or Auto Gamma.



# **Construct Maps**



In this step, you work more closely with your acquired maps and construct new EBSD and layered images. If you have acquired an EBSD map with stored EBSPs, you can also reanalyze a map region with new settings such as new solver settings or by including different phases.

This step displays a layered image, maps and electron images. You can choose how to view your data, and you can add or remove maps to build the layered image. You can also minimize some of your maps to give you a clearer view.

# **Map Tools**

You can use these tools to change the appearance of the maps.

Icon	Description
	Sets the number of maps per row in the Standard and Interactive displays.
Standard •	Offers a choice of map display.
<b>3€</b>	Links images for manip- ulation of all layers using the Pan or Zoom controls.
	Unlinks images. You can manipulate individual lay- ers using Pan or Zoom con- trols.
	Changes the brightness and contrast of the currently selected image or map.

Icon	Description
	Adds or removes a map in the layered image. The Lay- ered Image icon is in the top left hand corner of the map. A symbol rep- resenting the map appears on the layered image.
	Minimizes a map. The Minimize icon is in the top right hand corner of each map. This is useful if you have too many maps in view. The map moves to the Minimized Map tab, below the displayed maps.
	Deletes a map from the analysis completely. This icon in the top right hand corner of each map.
<b>不</b>	Restores a minimized map to its normal size. The mini- mized maps are on the Minimized Map tab, below the displayed maps.
Map   TruMap	Sets the mapping mode for X-ray Element Maps.
	Automatically adds maps to the layered image. This is the Autolayer feature.
	Changes the brightness and contrast and gamma for all the maps.

# **Constructing new EBSD images**

To construct a new EBSD image:

1. Expand the Construct EBSD Images panel and click the required image. A new EBSD image appears under the EBSD Data node in the Data Tree.



2. To display any image in the image viewer area, click on the smaller image on the Data Tree.

## **Deleting EBSD Images**

To delete a map from the analysis completely:

 On the Data tree or Current site, right-click the image, and in the context menu, select Delete.

#### Reanalyzing a Map region

If you have acquired an EBSD map with stored EBSPs, you can also to reanalyze a map region with new settings such as new solver settings or even solving by including different phases.

The reanalyze toolbar allows you to reanalyze a region of the complete map or entire map by selecting a rectangular, elliptical or free-hand region. Select your desired shape region to reanalyze your EBSD map. Click Start to start reanalysis.

Please take note that your total Area number of pixels will be dependent on the selected region.

## **Viewing Orientation information**

It may be helpful to view the orientation information from a point. To view how the indexing has changed with different acquisition settings, you can click to show orientation infor-

mation from point in map icon from the map area. Click on the electron image and place the crosshair on the pixel desired.

Orientation information such as EBSP, unit cell and pole figures, can now be monitored. The orientation information will be available during live map acquisition and reanalysis. Simply select Stored or Live in the toolbar to switch between live monitoring and stored mode. Stored pattern mode will be used when you are doing your offline reanalysis.

For live orientation, you will need to specify in settings how often you would like to refresh the rate of the orientation values.

#### **Phase Fraction**

The phase fraction percentage indicates how many pixels have been indexed to a particular phase in your specimen. The percentages of the phases and zero solutions are displayed and each will have a color, fraction percentage and total pixel area count.

Full map and region statistics are available now to compare results. These absolute numbers will change after noise reduction in the post-processing software, Tango because zero solutions will likely be replaced by neighboring phases.

# Reanalysis

## Introduction

Data reanalysis allows the reanalysis of a map region with different acquisition settings (i.e. phase, Hough, number of bands and number of reflectors). It also allows you to add and/or remove phases in case you may not know all the crystallographic phases present or different regions on the specimen require reanalysis with different phase lists. In addition it is possible to reanalyze data multiple times in order to standardize acquisition settings for specimens or until

When reanalysis of map data containing both EDS and EBSD data is performed, your EDS data is also carried into your reanalyzed map data folder. This functionality is yet another powerful tool to ensure successful phase identification and mapping. In addition, it allows you to extract both EBSP and EDS data point information for phase ID.

## **Setting up for Data Reanalysis**

In order to do any kind of reanalysis you need to be working with an EBSD Map that has stored patterns with it. Either highlight a previously acquired Map with stored Patterns or create a new Map:

To acquire a new Map with Stored Patterns:

- Create and save an Aztec Project.
- Go to the Acquire Map Data step and create a Map. Under Settings, ensure that you tick the Store Patterns option. Without the stored patterns option enabled, reanalysis will not be possible.
- Go to the Data Tree tab and confirm that under Map Data EBSD Data folder the
   Processed Patterns is visible.

## **Optimize Acquisition settings**

- Go to Phases tab add and/or remove phases. Select the phase database of your choice and add phase for acquisition. In the phases for acquisition quadrant, select the desired number of reflectors for each phase listed.
  - Use the Phase ID navigator step to identify any unknown phases and/or confirm composition using existing EDS and EBSD data. Search the phase databases for all phases using the composition ranges of the listed elements.
    - Go to the Acquire Data step and locate EBSP of interest to collect both EDS and EBSD data simultaneously.
    - Go to the Search Phases step to confirm the elements present by selecting the spectra and/or map sum spectrum in the spectrum viewer. Set the phase search criteria to generate the candidate phase list.



- Go to the Identify Phase step to index the EBSP using the list of candidate elements generated. The EBSP should index with a good fit and an acceptable MAD value. Select the best solution and click 'Add Phase for Acquisition'.
- Go to the Optimize Solver step to acquire an EBSP from a point on the map. If Auto is selected in the processed EBSP quadrant, the software will automatically snap, detect and index the EBSP. Go to settings and change desired number of bands, Hough resolution, etc. and the solution will be updated. Alternatively, you can manually click index after each setting is changed. Please note that you if you are interested in saving a pattern and

solution you will need to use the Extract an EBSP from a point toolbar. The extracted point data will be saved under an Extracted Point Data node in the data tree.

# Reanalysis of a Complete Map

Go to the Construct Maps step when your map with stored patterns has been collected or highlight your chosen map in the Data Tree. Optimize any acquisition settings (i.e. phases, Hough resolution, number of bands and number of reflectors.) However, note that you cannot change the step size to this map.

The reanalyze toolbar allows you to reanalyze a region of the complete map or entire map by selecting a rectangular, elliptical or free-hand region. Select your desired shape region to reanalyze your EBSD map. Click Start to start reanalysis.

Please take note that your total Area number of pixels will be dependent on the selected region.

During reanalysis, your Data Tree will display your data and map statistics under the Reanalyzed Map folder. Note that the processed patterns folder will also be stored under the Reanalyzed Map Data folder each time a map is reanalyzed.

The reanalyzed map in the data tree is the original project map area plus the selected region of reanalyzed pixels. All reanalyzed EBSD and EDS map data will be displayed in the data tree.

# **Results of Data Reanalysis**

#### **EDS and EBSD data**

Reanalysis of reanalyzed maps with both EDS and EBSD data is possible and the EDS data can be viewed together with EBSD reanalyzed maps. Note that the EDS data under the Reanalyzed Map is a complete copy of the original EDS data. Select your reanalyzed map on the data tree and the electron image of your choice will display the phase key for phases only identified during the data reanalysis. You can then change additional acquisition settings and start reanalysis of the reanalyzed maps.

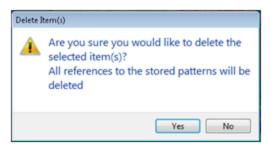
# **Different regions analyzed**

You can overlay all your reanalyzed map regions on the electron image. Simply right click on the electron image and show acquisition areas and select the highlight reanalyzed areas. The image viewer will display all the Reanalyzed maps (i.e. band contrast, Euler, IPF maps, Electron image, and post-acquisition image.)

It is worth noting that you can only reanalyze one map at a time. All reanalyzed map data will be saved in the data tree. To see all your map details highlight and right click on the EBSD data folder. Notice the Parent EBSD Map details will reference your reanalyzed map data project name.

## **Deleting Maps**

Reanalyzed maps are dependent on the processed patterns of the original map dataset. To delete a reanalyzed map from the analysis completely, simply go to the data tree, right-click and delete. Therefore, all folders located in the Reanalyzed Map data folder will be deleted. Therefore, deleting the processed patterns from the original map dataset with reanalyzed maps remaining in the data tree will prompt an error message. The error message will warn you 'All references to the stored patterns would be deleted'. Without patterns, it is not possible to reanalyze the data any further.



## **Creating a Pole Figure**

Pole figures represent the orientation distribution of lattice planes in crystals and textures of materials. You can construct a pole figure after you have acquired map data.

- 1. On the Orientation Information panel, select the Pole Figure tab.
- 2. Expand the Construct Pole Figures/Inverse Pole Figures tab, and click the Pole Figure icon to open a settings dialog.
- 3. Select the values such as phases and planes, then click OK to close the dialog. The pole figure appears in the Pole Figure tab. This might take a few seconds. You can watch its progress at the bottom of the tab.
- 4. To see details about the pole figure such as the number of projected points, find the object in the Data Tree. It has the label you gave in the settings dialog. Right-click and select Details.



- 5. If you have acquired EBSD data, you can select the Show Orientation Information tool, then click on a point on the map (or image) to display its solution, unit cell orientation or corresponding orientation on the pole figure in the Orientation Information tabs. If the data was acquired with stored EBSPs, you can display the solution with the EBSP, which helps you to interrogate the acquired data. If the phase you selected to create the pole figure matches the phase at the point you have selected, the corresponding orientation vectors blink on the pole figure in a color that matches the phase.
- 6. To make further changes to the pole figure, right-click on the Pole Figure tab to open a context menu. For example, you can change the direction and print the figure.

# Creating an inverse Pole Figure

Inverse pole figures represent the orientation distribution of lattice planes in crystals and textures of materials. You can construct an inverse pole figure after you have acquired map data.

- 1. On the Orientation Information panel, select the Pole Figure tab.
- 2. Expand the Construct Pole Figures/Inverse Pole Figures tab, and click the Inverse Pole Figure icon to open a settings dialog.
- 3. Select the values such as phases and planes, then click OK to close the dialog. The inverse pole figure appears in the Pole Figure tab. This might take a few seconds. You can watch its progress at the bottom of the tab. The data appears in the Data Tree, with the label that you gave in the settings dialog.
- 4. To see details about the inverse pole figure such as the number of projected points, find the object in the Data Tree. It has the label you gave in the settings dialog. Right-click and select Details.
- 5. If you have acquired EBSD data, you can select the Show Orientation Information tool, then click on a point on the map (or image) to display its solution, unit cell orientation or corresponding orientation on the inverse pole figure in the Orientation Information tabs. If the data was acquired with stored EBSPs, you can display the solution with the EBSP, which helps you to interrogate the acquired data. If the phase you selected to create the inverse pole figure matches the phase at the point you have selected, the corresponding orientation vectors blink on the inverse pole figure in a color that matches the phase.
- 6. To make further changes to the pole figure, right-click on the Pole Figure tab to open a context menu. For example, you can change the direction and print the figure.

#### Pole figure Settings dialog

This dialog appears when you click the Pole Figure icon.

Field	Description
Label	The label appears later on the object in the Data Tree.
	You can type your own text here. Otherwise, the text automatically consists of words that describe your selections in this dialog.
Phase	Select a phase from the drop-down list.
(radio buttons)	Select Direction or Plane.
Indices Use 4 Digit Indices	Select a 3-digit or 4-digit index notation, then type the required numbers in each box.  You can also select this option on the Preferences dialog on the Pole
	Figure tab.
Hemisphere	Select the hemisphere and
Projection Plane	projection plane.

# **Inverse Pole Figure Settings dialog**

This dialog appears when you click the Inverse Pole Figure icon.

Field	Description
Label	The label appears later on the object in the Data Tree.
	You can type your own text here. Otherwise, the text automatically consists of words that describe your selections in this dialog.
(Radio buttons: X, Y)	Select a direction such as X, or type a custom direction with alpha and beta angles.
Folded	When selected, creates a folded inverse pole figure. Otherwise, an extended (90-degree segment) inverse pole figure is created.

# See Also:

NanoAnalysis Encyclopedia, for "Pole figures" and "Inverse pole figures"



Export - Settings on page 132

Preferences on page 13

Context Menus - Pole Figure and Inverse Pole Figure Tab below

# Context Menus - Pole Figure and Inverse Pole Figure Tab

A number of useful shortcut menus are available when you right-click on the Pole Figure tab and it is displaying a pole figure or inverse pole figure:

Menu option	Description
Settings	Opens the Settings dialog, so that you can change the phase or direction.
Select Projection Type	Offers a choice of the type of projection - equal area or stereographic. When you select this display setting, it is applied to all your inverse pole figures.
	You can also select this option on the Preferences dialog on the Pole Figure tab.
Select Symbol Size	Offers choices of the pixel size. Normally one scatter point is represented by only one pixel. if you select Large, each point is represent by many more pixels, making smaller features easier to see.
Draw Great Circles (for Pole Figure only)	Shows the latitude and longitude lines on the sphere. When you select this dis- play setting, it is applied to all your pole figures.
	You can also select this option on the Preferences dialog on the Pole Figure tab.

Menu option	Description
Overlay on Sphere (for Inverse Pole Figure only)	Shows the location of the inverse pole figure as a segment within the sphere. When you choose this display setting, it is applied to all your inverse pole figures.
	You can also select this option on the Preferences dialog on the Pole Figure tab.
Export	Offers choices of output. For example, you can print the figure, or send it by email. Using the Settings option here, you can change the size of the figure.
Details	Shows details such as the number of projected points and the phase name.



# Phase ID

The Phase ID navigator has six steps.

Describe Specimen, Scan Image and Optimize Pattern are described in the Map section. Three steps which are unique to Phase ID are described in detail below:

Acquire Data	474
Search Phase	477
Identify Phase	480

# **Acquire Data**



The use of the EBSD to identify unknown phases in a material is called Phase Identification (Phase ID). For many years, EBSD has been used to discriminate between several phases in a specimen using Phase discrimination either by analyzing single patterns from various phases or by automated mapping of such phases.

Phase ID takes this approach a step further by utilizing chemical analysis in conjunction with a crystallographic phase database in order to produce a list of candidate phases. These phases are then used to index the EBSP from the 'unknown' phase.

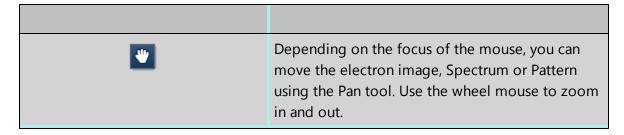
The steps in a phase ID analysis are typically:

- Locate a grain or particle of the unknown phase. Forward Scattered imaging will help to locate areas of different phases.
- Acquire both EDS and EBSD data simultaneously
- Determine the composition of the phase using EDS analysis.
- Search a crystallographic phase database for all phases using composition ranges of the elements present.
- Index the EBSP using the list of candidate phases.
- Determine the identification of the unknown phase.

# **Data Acquisition**

In this step, you can monitor both EBSD and EDS data coming from specific locations or collect and store data from specific points on your specimen.

## **Acquire Data toolbar**





T	To add text on your electron image, select the Text annotation tool, click on the object where you wish to enter the text and then start typing the text. To delete annotation double click on it to select it and then press the Delete key on the keyboard. To delete all annotations on the object, choose Select All from the Annotations context menu on the image viewer and then press the Delete key on the keyboard.
	Click on this icon to select the Point Tool from the toolbar and then click on the image to start monitoring as you hold down the mouse.
i	From the Spectrum  With this tool you can view the Energy (keV) and
	With this tool you can view the Energy (keV) and counts in any channel of the spectrum. Simply select the Show Data Values tool from the toolbar and then hover on spectrum. The values will be displayed as you move from channel to channel.
	From the Electron Image
	Clicking anywhere on the image will display the Intensity value at that pixel position.

# Monitoring the signals

Click on the acquisition tool and press the mouse down on the electron image to see the monitoring signals for both EDS and EBSD. Move the mouse over the image to monitor both the EDS and EBSD signals. Once the mouse is released, acquisition will take place according to the acquisition settings for EDS and EBSD.

Spectrum Monitor provides a dynamic way to see what X-rays are being detected at any given moment. It is useful for a quick survey of the specimen to find an area of interest for analysis. Spectrum Monitor uses the current spectrum acquisition settings with the additional setting of the refresh rate for monitoring the spectrum. This refresh time is referred to as the Buffer Size. The default is 20 but can be changed under the Settings for Spectrum Monitor in the Miniview. Increasing the Buffer Size corresponds to a longer refresh rate.

## **Data collection**

Click on the acquisition tool and click on a location on the electron image. Acquisition will immediately start under conditions currently set for both EDS and EBSD. EBSD acquisition will use the settings currently set in Optimize Pattern. EDS acquisition will use the parameters currently set under EDS Settings. Please select the link below for details on EDS Acquisition settings.

A Point Data node will immediately appear both in the Data Tree and Current Site views. The associated collected data (EDS Spectrum and EBSP) will be grouped together under this node with a corresponding label. Note that this label can be renamed by selecting the rename option from the context menu on the Data tree and Current Site Data Views.

Depending on the acquisition settings, EDS and EBSD could be acquiring for different lengths, however acquisition will continue until both spectrum and pattern acquisition are complete. The progress of acquisition is displayed on the Current Site tab on the Point Data node.

You may queue up several points to acquire data from by clicking with the acquisition tool on the electron image. Acquisition will continue until all the data has been acquired or the stop button is pressed. Note that the corresponding points are marked on the electron image.

#### See Also:

Acquire Data - EDS Settings

Context Menus - Image Viewer on page 157

Context Menus - Spectrum Viewer on page 321

Compare Spectra & MiniQuant Results on page 195

Current Site on page 29

Data Tree on page 85



# **Search Phase**



This step is divided into three main parts:

- Confirming the presence of elements in your spectrum.
- Setting up the search criteria
- Conducting the Phase Search

## Spectrum selection

The spectrum displayed in the Spectrum viewer corresponds to the one stored displayed under the currently highlighted Point Data node. Highlight a different Point Data node to display another Spectrum. Alternatively, select the required spectrum from the drop down list box positioned above the spectrum viewer, this will allow selection of not only spectra acquired from the Acquire Data step but also sum spectra or spectra acquired elsewhere in the application.

# Confirming the elements in your Spectrum

Part of this step is designed to help you confirm the elements that have been identified by AutoID in your spectrum. The options available are those found in the Confirm element step in the EDS part of the product. These elements are then used to create an element list for qualitative and quantitative analysis. Note that extensive tools including Element Series Markers, Overlays, Element Profiles and Show Candidate Elements are available to help you to manually confirm your elements.

If you wish to manually confirm the automatic peak identification:

- Press the question mark icon to select the Show Candidate Elements tool from the tool bar on the left hand side of the interface.
- Double click on a peak in the spectrum viewer.
- The candidate elements are displayed in a stacked spectra view on the right hand side of the window (you can double click on any of these elements to add or remove it from the confirm elements list).
- You can control what overlays you see in the Spectrum viewer via the 'Confirm Elements Settings'. These overlays can be very useful in helping you to interrogate complex spectra.
- Press Include/Exclude once you are satisfied with the identification of each element to build your list of confirmed elements.

## Setting up the Search Criteria

The section under 'Composition Used For Search' displays the Quant results for the currently displayed Spectrum in Wt% and At% calculated according to the currently selected Quant Settings. Note that the calculation of the composition takes into consideration the overall tilt of the specimen as entered in the Describe Specimen Step. This chemical information forms the basis of the search in the databases. However there are some extra settings provided which allow you to optimize the success of your search.

It is worth noting the following points:

- The EDS system may not be set up for obtaining optimized quantified data under EBSD Conditions as the Specimen is highly tilted. You should therefore allow large error margins on the chemical data.
- Light elements may not be detected. Therefore, if you suspect carbides, borides, hydrides to be present in your sample, you should add these manually to the periodic table.
- You should also be aware of the limitations of your databases. For example, database entries may include minor elements (e.g. rare earth elements), hydrous phases (with OH2) or end members of solid solution series. Even in steels, databases will often include the Fe end member, rather than including Cr, Mn, Al etc.
- You should be aware of the difference in spatial resolution of the EBSD and EDS
   Techniques. Particles < 200nm across may give good EBSPs, but the EDS signal will
   come mainly from the surrounding matrix.</p>

The following options apply to all elements listed in the table.

## Uncertainty

The Uncertainty controls how large the error margins on the quantified EDS data should be. The default for this parameter is 100%. The default value is set high to ensure that phases are not missed during a search due to inaccuracies in the chemical information in the structural databases or errors related to quant on highly tilted samples.

# **Threshold**

The Threshold controls the minimum (allowed) percentage of any element to be automatically included. Note that 10% is the default value for this. If the percentage found of the particular element is less than the threshold, the element is optionally included during the search.

# **Search Condition**

These options apply to the individual entries in the table.

#### **Include**

This means include this element and its search range in the Phase Search if the minimum concentration of the element is greater than the threshold value set.

# **Optional**

This means optionally include this element and its search range in the Phase Search if the minimum concentration of this element is less than the threshold value set.

#### **Exclude**



This means exclude the element and its search range in the Phase Search.

#### Wild Cards

A number of wild cards can also be used in the search which means that you are including 0, 1, 2...unknown elements during the search.

You should also note that adding wildcards can significantly slow down the search process of the database.

As an example a search using Ti and one wildcard will find Ti as well as all combinations of Ti and one other element such as TiC and TiN.

## **Conducting the Phase Search**

A list of available databases is shown. Check the databases you wish to search through and press Search. The results of the Phase Search are shown in the 'Phase Search Results' table.

The name of the phase, the database from which it was found, the Space Group and the Composition are all reported in this table. If you wish to limit the search to fewer databases, uncheck the database and press Search again. After performing search, the list of found phases are now stored associated with the highlighted spectrum. This will be used in the next step.

#### **Databases**

User created databases can also be used for the phase search process; however it requires that the databases are first copied into the C:\CHANNEL5 directory.

#### See Also:

Confirm Elements - Settings on page 171

Confirm Elements - Tools on page 174

Element Lists on page 191

Peak Labels on page 158

Compare Spectra & MiniQuant Results on page 195

Quant Settings on page 187

# **Identify Phase**



The purpose of this step is to use the phases in the 'Phase Search Results' area to index the EBSP shown in the image viewer and display the solution(s). The phase that gives the best solution to the EBSP can then be manually added to the list of phases for acquisition. This phase is then available to use, for example, during mapping.

#### **Phase Search Results**

This list of phases comes from the spectrum and is generated from the previous step. You can now prepare it for indexing. The list of phases together with the number of reflectors and phase color is shown in this table. You may wish to review the details of a particular phase. To do this, highlight the phase from the list and select the tab you wish to use from the Phase viewer below.

#### **Phase Color**

The colors for the phases are picked automatically with the same color being used for the phase as used in the 3D Phase View and the simulation used on the EBSP. You can manually change the color if you wish, but the software will attempt to pick a unique color for each phase.

#### **Maximum reflectors**

Rather than having to set the number of reflectors for each phase, you can set a maximum. This means that the closest value to this maximum while still being lower will be automatically found for the number of reflectors for each phase. You can then manually change it afterwards. Note that changing the maximum value will automatically trigger another update of the reflector values for each phase.

## **Exclude phases from list**

In situations where there are a number of phases found it may be useful to exclude some of the phases from the list. Uncheck the phases you wish to exclude when indexing the EBSP.

In order to make a potentially long list of phases more readable, you may wish to display only the included phases. This can be controlled from the 'Show excluded' check box.

#### Choice of EBSP

The EBSP displayed in the image viewer corresponds to the current EBSP – the one displayed under the highlighted Point Data node. Highlight a different Point Data node in order to display another EBSP. Alternatively, select the required EBSP from the drop down list box positioned above the image viewer. This EBSP might have been acquired at a different time or from a reconstructed point. Use the drop down list box to pick an EBSP that is acquired at a different time/point than the highlighted spectrum.



#### Indexing the EBSP

There is no limit to the number of phases that can be used for indexing. However, the greater the number, then the slower the Phase ID process becomes. For example, if you have over 100 phases, then it would be advisable to try and restrict your search criteria before indexing the EBSP.

You may check the Auto box which means that band detection and indexing will automatically take place if you change any settings or adjust any of the parameters in the Phase Search Results list. Alternatively, you can manually detect and index the EBSP by pressing the respective buttons. Since indexing is generally slower than normal because of the high number of phases, it is advisable not to use Auto.

#### **Suggested Settings**

Adjust the Maximum number of reflectors to a suitable value before indexing. This value is dependent on the quality of the EBSP from the unknown phase, but typically you may want to use more reflectors (e.g. 75-100) than for normal EBSD analysis. Note that increasing the maximum number of reflectors will also slow down the indexing process.

To optimize band detection, it is recommended that you use a high Hough resolution, for example 70, and a greater number of bands than for standard EBSD analysis. If the EBSP has well defined, sharp Kikuchi bands, use the Band Edge detection. Alternatively, use Center detection. Careful manual band detection is often the preferred method for accurate Phase ID.

It is advisable in the first instance to switch off the Advanced Fit mode.

The software will now try to index the EBSP using all of the possible phases you have included in the Phase Search lists. This may take a few seconds if you have many potential phases. Note that solving does not involve the 'Phases for Acquisition' list in Describe Specimen.

When the indexing process is complete, any solutions will be displayed below the EBSP in the Analysis area. If there is more than one solution, scroll through the different solutions and visually check the accuracy of the indexing by viewing the simulations. The MAD value displayed in the solution list will provide a good estimate of how well the simulation matches the EBSP. Whilst higher MAD values indicate a better match, it might not be correct if the wrong phase or settings have been used in the process so always check that the solution matches the EBSP. Select the solution from either the phase list or solution list.

## **Multiple Solutions**

In an ideal situation, there will be one solution which is clearly correct. However, there may be several different phases which appear to give a good match. In such cases one or more of the following steps may help to identify the correct solution:

- Check the simulation using more reflectors, by adjusting the number of reflectors in the Phase Search results view.
- Adjust the database search by adjusting the compositional ranges for individual elements if necessary (previous step).
- Optimize the band detection by manually adding more bands if necessary.

• Switch on Advanced Fit. Note that if you had many solutions (>10) then this may take a long time.

## **Adding Phase for Acquisition**

The phase from the solution that you consider the 'correct' solution can then be added to the usual list of phases for acquisition (in Describe Specimen) by pressing the 'Add Phase for Acquisition' button. You may wish to experiment solving with this full list of phases and a link to the Optimize Solver step is provided.

#### See Also:

Optimize Solver on page 444



# **Hardware Control**

In this section various software tools that control the hardware are described.

Detector Control	484
EBSD Detector Control	489
Microscope Control	493

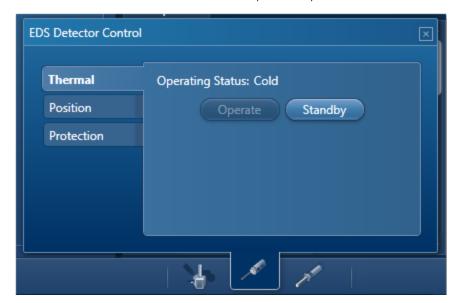
## **Detector Control**

There is a separate control for EDS and EBSD detector hardware which provides information about the hardware status. Each control is accessible from the Detector Control icon in the right-hand side of the Status bar. The Detector Control is displayed as a pop out dialog.

The EDS Detector Control software is described under three current EDS detectors:

#### X-Max Detector

In the case of X-Max detectors there are three tabs, Thermal, Position and Protection:



#### **Thermal**

The Thermal tab displays the current operating status of the detector. The possible states are shown in the table below:

State	Description
Standby	Cooling is powered down. Allows power save and detector longevity  (Steady orange LED)
Warm	Detector is not ready for data acquisition (Steady orange LED)
Cool	Detector is ready for data acquisition (Steady blue LED)
Warming	Detector is warming after selecting the Standby mode
Cooling	Detector is not ready for data acquisition (Flashing blue LED)
Fault	A fault has developed (Red LED)



There are two buttons in the Thermal Control tab, **Operate** and **Standby**. Pressing the Operate button initiates the cooling process. When the detector is not in use, pressing the Standby button puts it in the Standby state.

#### Note

If the detector is not cold or it is in the process of cooling, the spectrum acquisition will be disabled.

#### **Position**

The Position tab displays the current slide state of the detector:



There are three buttons on the Position tab, **In**, **Out** and **Stop**. Pressing the **In** button will start moving the detector into the chamber. The **Out** button will start retracting the detector from the chamber and **Stop** will stop the detector at the current position. The possible detector states are shown in the table below:

State	Description
Fully Inserted	The detector is fully inserted into the microscope chamber and ready for acquisition
Fully Retracted	The detector is fully retracted from the microscope chamber
Indeterminate Position	The detector is at some position in between the end stops
Fault	A fault has developed

The possible activities that may be displayed are Stopped, Moving in, Moving out and Autoretracting.

#### **Protection**

Depending on the software licenses that you have, two options are available to protect your detector from damage by X-rays:



## **Low Mag Protection**

This option is enabled on the X-Max detector provided the system has the relevant license (Low Mag Protection, 06) installed. The Low Mag Interlock status is displayed on the Protection tab as shown in the screen shot above. Putting the microscope into the low magnification initiates auto-retraction of the detector to protect it from the X-rays.

Note that the microscope needs to have hardware interlock for Low Mag Protection to work.

When the user switches out of low magnification mode, the detector will need to be moved back into the chamber by pressing the **In** button.

#### **Flux Protection**

The Flux Protection is only available if appropriate license (Detector Flux Protection, 05) is installed. A checkbox for enabling/disabling this option is available on the Protection tab on the TEM system. You can enter the values for Delay in seconds and Threshold in MeV per second (Mega electron volts per second). When the X-ray flux exceeds the threshold value for longer than the specified delay time, the detector will auto-retract.

The user will need to move the detector back into the chamber by pressing the **In** button. If the overload condition persists, the detector will auto-retract again.

You can restore the default values for Threshold and Delay by pressing the Restore Defaults button if you have overwritten them.

#### X-act Detector

The Detector Control has Thermal tab:

#### **Thermal**



There are two buttons on the Thermal control tab, **Operate** and **Standby.** Pressing the Operate button initiates the cooling process and pressing the Standby button puts the detector in the standby mode.



The Thermal control tab displays the current operating status of the detector:

State	Description
Standby	Cooling is powered down. Allows power save and detector longevity
	(Steady orange LED)
Warm	Detector is not ready for data acquisition (Steady orange LED)
Cool	Detector is ready for data acquisition (Steady blue LED)
Warming	Detector is warming after selecting the Standby mode
Cooling	Detector is not ready for data acquisition (Flashing blue LED)
Fault	A fault has developed (Red LED)

The Thermal control tab displays the current state of the vacuum in the chamber:

- Under Vacuum the chamber is under vacuum
- Vented The chamber has been vented
- Fault A fault has developed

The Thermal control tab also displays the extended mode state.

#### Note

The start button for spectrum acquisition is disabled if the detector is not cold or it is cooling down.

#### LN2 Detector

The Detector Control has a number of tabs depending on the type of detector. The tabs are described below:

#### **Thermal**

The Thermal tab displays the LN2 status:

State	Description
Ok	The liquid nitrogen level is ok
Low	The liquid nitrogen level is low
In Air	The liquid nitrogen sensor is exposed to the air
Sensor missing	The liquid nitrogen sensor is not plugged into the x-stream

If the LN2 level is low, there will be an audible alarm. You can silence the alarm for one hour by pressing the Mute button.

#### **Position**

If the detector has a motorized slide, the Detector Control will show the Position tab.

This tab shows the current slide state of the Si(Li) detector as shown in the table below:

State	Description
Fully Inserted	The detector is fully inserted into the microscope chamber and ready for acquisition
Fully Retracted	The detector is fully retracted from the microscope chamber
Indeterminate Position	The detector is at some position in between the end stops
Fault	A fault has developed

The Position tab displays the activity, Not Moving, Moving In and Moving Out.

There are three buttons on the Position tab of the Si(Li) Detector Control, In, Out and Stop. These controls are used to move the detector in and out of the chamber, and stop it at the current position.

## **Shutter**

If the detector has a pneumatic shutter, there will be a tab for the shutter control.

The Shutter tab has two buttons, **Open** and **Close** to allow the shutter to be opened and closed.

The status of the shutter is either Open or Closed.



## **EBSD Detector Control**

The EBSD detector control sets the position of the EBSD detector, and displays its position and movement. You can access this control from the status bar.

#### **States**

State	Description
Fully Inserted	The detector is moved as far into the chamber as the mechanical end stop allows. This position is set by the service engineer and cannot be changed by the customer.
Fully Retracted	The detector has moved as far out of the chamber as it can.
Intermediate Position	The detector is at an inserted position that is not the fully inserted position or the reference position.
Reference Position	The detector is at the position that has been saved as a ref- erence position by use of the handset.
Auto Retracted	The Touch Sensor has been triggered and the detector has moved all the way out to the home position.

## **Activity State**

State	Description
Not Moving	The detector is not moving.
Moving In	The detector is moving into the chamber.
Moving Out	The detector is moving out of the chamber.
Homing	During calibration of the hardware, the detector goes through a homing routine, which ensures that it finds the correct zero position for the counter mechanism.
Auto Retracting	The Touch Sensor has been triggered and the detector has automatically started moving out of the chamber.

## **Position**

The insertion distance from fully retracted position is shown in mm (and can also be shown in the status bar). This is important because it always shows the position of the detector and constantly updates when the detector is moving.

## **Touch Sensor**

State	Description
Not Touching	The front part of the detector has Touch Sensors and none of these sensors are currently triggered.
Touching	One of the Touch Sensors on the front of the detector is triggered.



#### **Interlock State**

Some systems have an interlock between the scanning electron microscope (SEM) and the EBSD electronics. The option is not available for all SEMs, and the function also varies between systems. On some SEMs, the interlock does not limit the detector movement. On other SEMs, the interlock does not allow the detector to be inserted into the chamber, which means that the detector cannot move in, and if it is already inserted, the detector will be retracted.

State	Description
Allow Movement	This is the default state. It is shown when no interlocking takes place because no interlock is configured or because the interlock allows the detector to be moved.
Prevent Movement	This state appears only if the interlock is configured and the SEM is in a state in which the interlock does not allow the detector to be inserted.

## **Detector Positioning**

The buttons on the Position tab control the movement of the detector.

Button	Description
In	Starts moving the detector into the chamber until either the movement is stopped by the operator or the detector reaches its fully inserted position.

Button	Description
Out	Starts moving the detector out of the chamber until the movement is stopped by the operator or the detector reaches its fully retracted position.
Stop	Stops the detector movement immediately at any time.
Move To	Moves the detector to a specific position. Type the insertion distance in the box to the right, then click Move To.
Step In, Step Out	Moves the detector a fixed distance inwards or outwards. Type the distance in the box to the right, then click Step In or Step Out to move the detector by the required distance.



## Microscope Control

The Microscope Control application is provided for controlling and reading the microscope

parameters. When you press located on the right hand side of the Status Bar, the Microscope Control is displayed as a pop up window.

The current value of each parameter is displayed in the appropriate entry box in the Microscope Control as shown in the screen shot below. In this example the Column tab is selected:



The exact way in which each of its functions operates depends on the facilities available on the microscope on which the EDS system is installed. Where possible, parameters can be changed either from the electron microscope controls or the Microscope Control and both systems will be updated accordingly. In some cases it may be possible that the parameters from the microscope are read automatically but can not be changed in the Microscope Control.

If Microscope conditions can not be read automatically, you will have to manually enter the values (see below). It is important that the correct values are entered such as WD (working distance) and magnification as these will be used during quantitative analysis and image calibration.

Column parameters such as working distance, magnification and high voltage are also important for the EBSD systems performance, so if these are not read automatically then they must be entered manually.

## How to change parameters in Microscope control

Enter a new value into the entry box adjacent to the parameter and press the Set button. The column parameters are Magnification, Working Distance (mm) and High Voltage (kV) which are described below:

#### Magnification

Magnification is used when calculating the length of the scale marker bar which can be superimposed on an image or the length of a linescan. You can change this in the Microscope Control window.

## Working Distance (mm)

Working distance is the distance between the point of focus of the electron beam and the final lens, i.e., when this is changed, the current through the lens is changed. You should ensure that the working distance set on the microscope, in millimeters, is the desired value for performing X-ray microanalysis. The recommended value for your instrument is displayed in the Mini View.

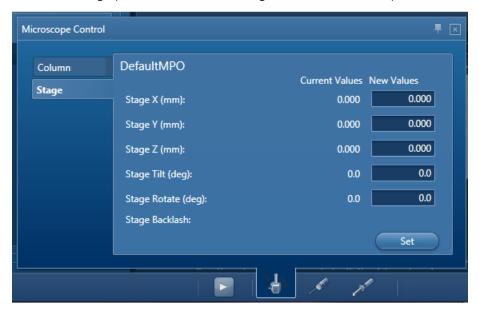
Note that if you change the working distance on the microscope, your image may then be out of focus, in which case you will need to adjust the Z of the stage to bring it into focus.

#### High Voltage (kV) or Accelerating voltage

It is particularly important to have the accelerating voltage set to the correct value, since it is used by the quantitative calculation when calculating intensity corrections. You can change this in the Microscope Control.

#### Stage

You can access the stage parameters from the Stage tab in the Microscope Control window:



#### Stage tilt

To display or change the stage tilt value, select the Stage tab on the Microscope Control window.

If your stage tilt is motorized, the current value can be displayed and changed. More commonly, the stage tilt is not motorized but, if you are working with tilted samples, it is necessary to enter the current tilt value. If you are working with tilted specimens and you are going to use the spectrum for quantitative analysis, it is important to enter the correct value since it is used by the quantitative analysis program to calculate the specimen geometry.



EBSD orientation data relies on knowing the sample tilt, so if the stage tilt can not be read automatically then it must be entered manually in order for the orientation data to be correct.

## See also:

Microscope Parameters on next page

## **Microscope Parameters**

- Microscope Control reads and controls the microscope parameters. Ensure that the values in the Microscope Control window correspond to the current microscope parameters.
- You can change the values displayed by changing the appropriate controls on the microscope. This will automatically update the displayed values in the Microscope Control window.
- If microscope parameters can not be read automatically, you will need to manually enter the values of kV and magnification directly into the spaces provided in the

Microscope Control. Access the Microscope Control by pressing located on the right hand side of the Status Bar. All images subsequently collected will be acquired with these parameters. Note that if you change the magnification on the microscope and forget to enter this new value into the Magnification box in the Microscope Control window the scale marker will be incorrectly calculated.

## Accelerating voltage (kV)

#### What is it?

The voltage applied to the electron gun that causes electrons to accelerate down the electron optic column. The higher the accelerating voltage, the greater the energy and the shorter the wavelength of the electrons striking the specimen.

#### Why is it important?

- The energy of electrons striking the specimen dictates what X-ray lines can be excited and their relative intensities.
- For quantitative calculations, the software needs to have an accurate value for kV.
- At higher kV, the higher energy incident electrons penetrate deeper and scatter more widely into the specimen so the excitation volume for X-rays is larger.

#### What value should I use?

Choosing 20kV is a good starting point particularly if the specimen is unknown. At this kV at least one series of X-rays from every element will be excited.

- Choose a lower kV if you are concerned about:
  - Accuracy of quantification of light elements since the lower penetration at low kV will reduce the absorption correction.
  - Analysis of a small particle, inclusion or a film less than 10 µm in depth since a smaller excitation volume will enhance the contribution from these features.
- Reducing the kV may reduce the options for easy element identification. A higher kV will excite the higher energy lines. The Br K line will be excited by 20kV but at 10kV, only the Br L line will be excited which overlaps with the Al K line.

#### Magnification



#### What is it?

The magnification of an image is formally defined as the ratio of the length of one line of the electron beam on the monitor to the width of the area scanned on the specimen. However, since monitors vary in size and the image may be printed, magnification on its own is not enough to work out the size of a feature.

## Why is it important?

Magnification is used by the software when calculating the length of the scale marker bar to display beneath all electron and X-ray images. The marker bar will scale with the size of the image and is a more reliable indication of feature size.

## Which Magnification should I use?

#### **Electron Imaging**

In order to see greater detail in your electron image; increase the magnification of the microscope. This effectively means that the electron beam rasters over a smaller area of the specimen. Depending on your microscope, magnifications of up to 300,000 can be attained. However, above these magnifications, no greater detail is observed because of the size of the scanning probe. Any greater magnification is often referred to as 'empty magnification'.

#### **Element Mapping**

If you are acquiring X-ray data from a point, choose a magnification so that you see sufficient detail to allow you to position the beam.

Choosing a magnification greater than 1000 times will usually ensure that you are analyzing in the center of the field visible to the X-ray detector and will allow you to scan over this area to obtain a map.

Note that the beam diameter is usually much smaller than the excitation volume so that even if you position the beam on a small feature in an electron image, the X-ray data that you obtain may originate from the surrounding material.



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