

### X-Ray Acquisition and Analysis Software

# **Pathfinder**

### **User Guide**

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## **Pathfinder System Overview**

The basic Thermo Scientific<sup>™</sup> Pathfinder system is a high-performance X-ray energy dispersive spectroscopy (EDS) detector with supporting electronics and controlling/analysis software installed on a dedicated computer. The optional MagnaRay add-on system is a wavelength dispersive spectrometer (WDS) that is fully integrated into the Pathfinder software. All Pathfinder detectors can be mounted on a variety of scanning electron microscopes. In addition, the EDS detectors can be mounted on transmission (TEM) and scanning transmission (STEM) electron microscopes.

Thermo Scientific EDS detectors are available with various crystal sizes. Here are some examples:

- UltraDry. Silicon drift detector (10, 30, 60 & 100 square millimeter crystals)
- NORVAR. Ultra-thin windows
- MagnaRay (WDS). Parallel beam wavelength spectrometer with supporting electronics

The Pathfinder software lets you acquire, analyze and quantify X-ray microanalysis data and create reports showing the results of your work.

Several advanced options are available, including <u>Stage Control</u>, <u>Beam Current Measurement</u>, <u>Analysis Automation</u>, <u>Compass</u> statistical analysis, <u>Drift Compensation</u>, <u>Feature Sizing</u> with Chemical Typing, <u>Direct-to-Phase</u>, <u>Phase Analysis</u>, <u>XPhase</u> and <u>Spectral Match</u>.

These hardware options are also available: <u>Synergy</u> mouse and keyboard, <u>Multiple Detectors</u> and <u>WDS Automation and Analysis</u>.

**Note** The sections that follow provide a complete list of the optional software and hardware available for Pathfinder systems.

For purchasing information, contact your sales representative.

### **Optional Software**

The Pathfinder software includes optional features to enable advanced operations. These features will not appear in the software unless they have been purchased and installed.

To obtain any of these software options, please contact your sales representative.

#### **Software Options**

- **Beam Current Measurement** (PF-Beam). Software option for measuring beam current automatically and entering the measurement into the microscope parameters. (Can include associated hardware if not included with your microscope.)
- **Image Filters** (PF-Filters). Provides a range of filters that can be applied to an X-ray image.
- <u>Spectrum Match</u> (PF-Match). Allows you to identify a spectrum from an unknown material by comparing it to a database of previously acquired spectra from known materials.
- **Quant** (PF-Quant). Full standards quantitative analysis. Allows you to use your own standards to quantify X-ray data. (Standardless quant, which uses standards stored with the software, is always included).
- Quantitative Mapping (PF-Extr-Elem). Allows you to generate maps using quantified data, for example in Atomic % or Weight % or K-ratio %. (The ability to generate X-ray count maps is always included.)
- Topographical maps (PF-Topo). Allows topographical representation of X-ray maps.
- **3D** (PF-Jeol-3D). Ability to collect and process image stacks for 3D analysis.
- **Compass** (PF-Compass). Allows statistical (principle component) analysis of a spectral imaging data set. Includes Direct-To-Phase extraction of phases during acquisitions.
- **Phase Analysis** (PF-XPhase). Creates phase maps from conventional X-ray maps.
- **Drift Compensation** (PF-Drift). Automatically corrects for image movement during acquisitions.
- **Analysis Automation** (PF-Auto). Lets you automatically perform repetitive acquisitions on large scale analyses without the need for user intervention or input (requires stage control).
- Feature Sizing and Chemical Typing software (PF-Particle). Measures morphological parameters of particles or features in a sample and determines their chemical makeup.
- WDS Automation and Analysis (PF-WDS). Allows wavelength dispersive spectrometer acquisitions and analysis with Pathfinder EDS acquisitions (requires MagnaRay spectrometer). WDS software is fully integrated with Pathfinder software.
- 3rd Party Remote (PF-Portal). Remove control from 3rd party software packages.

### **Optional Hardware**

The Pathfinder system includes optional hardware for enhanced integration or operation. These features will not be available unless they have been purchased and installed.

To obtain any of these hardware options, please contact your sales representative.

#### **Hardware Options**

- **Synergy**. Allows control of microscope and Pathfinder software from a single mouse and keyboard.
- Multiple Detectors. Two or more detectors controlled by one front end.
- **MagnaRay**. Allows wavelength dispersive spectrometer acquisitions and analysis with Pathfinder EDS acquisitions (requires WDS Automation and Analysis software).

### **Contact Us**

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### **Software Overview**

The Pathfinder software lets you acquire, analyze and quantify X-ray microanalysis data and create reports showing the results of your work.

Pathfinder features a common, multi-pane user interface containing a wide assortment of analytical views and controls. As you switch <u>analytical modes</u>, the software provides tools to assist in your analysis. Data is saved within projects, and the current project's files for the mode you are using are displayed in the <u>file list pane</u>. Data files are easily transportable to other microanalysis systems and to graphics, word processing and spreadsheet programs.

Most operations are performed by clicking <u>toolbar</u> buttons. Each analytical mode has an associated toolbar that provides the various functions performed in that mode. Some toolbars, such as the acquisitions toolbar, are common to all modes.

General, program-wide operations are performed through <u>menu commands</u> or message boxes. Menu and submenu entries may be different for each analytical mode.

One <u>Controls Panel</u> provides access to all the setup parameters for each analytical mode.

Depending on the selected analytical mode, other panes may contain an electron image, <u>map</u>, <u>spectrum</u> or <u>periodic table</u> to show associations with specific chemical elements.

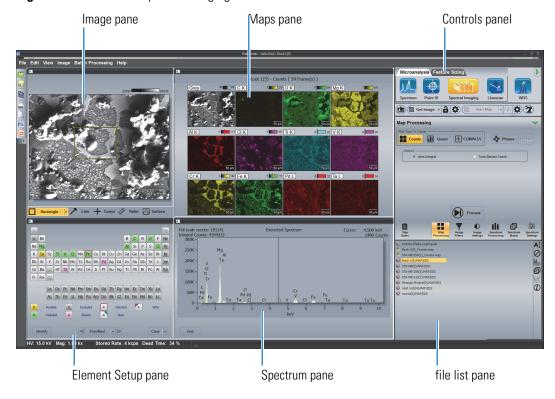


Figure 1. Pathfinder Spectral Imaging Mode Interface

Data are saved and managed within **Projects**. As you switch analytical modes, the project's files for that mode are displayed in the file list pane in the Controls Panel. Data files are in industry standard formats (TIF for images, EMSA (CSV format) for spectra). X-ray maps can be saved individually in TIF format.

### **Start Pathfinder Software**

#### ♦ To run Pathfinder from the Windows<sup>™</sup> Start menu

1. Choose Start > All Programs > Thermo Scientific > Pathfinder > Pathfinder.

The Project Explorer window is displayed.

2. Create or select a project folder and choose OK.

The Pathfinder <u>Home screen</u> is displayed in the last <u>analytical mode</u> used.

#### \* To run Pathfinder from the Windows desktop

1. Double-click the Pathfinder icon icon on the Windows desktop.

The Project Explorer window is displayed.

2. Create or select a project folder and choose OK.

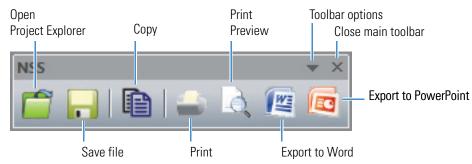
### **Toolbars, Status Bar and Buttons**

The toolbars, status bar features and buttons available in each <u>analytical mode</u> vary depending on that mode's functions. Some toolbars are available only when optional software or hardware is installed.

To view a button's name or function, hover the mouse pointer over the button.

#### **Main Toolbar**

The main toolbar is common to all the <u>analytical modes</u>. It contains buttons for general functions such as <u>opening</u> and <u>saving</u> projects (through Project Explorer), <u>copying</u>, <u>printing</u>, and <u>exporting</u>. You can add and remove the standard buttons on this toolbar and hide or show the tool tips.



#### Figure 2. Pathfinder Main Toolbar

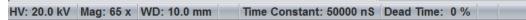
The main toolbar may be docked or displayed in a floating palette.

- To dock the main toolbar, grab the toolbar by its title bar and drag it to an edge of the Pathfinder window. When the toolbar flattens and adheres to the window, release the mouse button and the toolbar will dock to the pane.
- To use the main toolbar from a floating palette, drag the toolbar by its edge into any visible pane and drop it.
- To close the floating main toolbar, click the X in the title bar.
- To reopen the main toolbar, choose **View** (menu) > **Restore Toolbars**.

#### **Status Bar**

The status bar along the bottom of the Pathfinder window appears in all analytical modes. It provides information about the current acquisition or idle condition and a variety of <u>data</u> <u>acquisition</u> settings. The status bar can also include microscope settings if those settings are selected for display in the <u>Microscope Parameters and Status Bar Selection</u> box.

Figure 3. Pathfinder status bar



- To display or hide the status bar, from the Home screen, choose **View** (menu) > **Status Bar.**
- To change the status bar information, choose **Edit** (menu) > **Microscope Parameters**. The <u>Microscope Parameters And Status Bar Selection</u> box appears. Select the features to be displayed in the status bar and choose **Close**.

### **Controls Panel**

The Controls Panel contains the tabs, buttons, slider bars and icons to control all integrated analytical modes associated with Pathfinder software. The Controls Panel can be hidden or displayed using the hide/show button.

Figure 4.Pathfinder Controls Panel

Microanalysis Feature Sizing	<ul> <li>Show/hide Analytical mode toolbar</li> <li>Analytical mode toolbar</li> </ul>
📺 🛧 Get Image 🗸 👌 🔅 Start Map 🖌 🖓 🤔	_ Acquisitions toolbar
Spectral Image Setup	<ul> <li>Show/hide Advanced View</li> </ul>
Active Detector(s)          Detector One       Detector Two         SI Acquisition	<ul> <li>Analytical Mode Setup pane</li> </ul>
Custom	
Step Map Image Spectrum Spectrum Setting Notes Processing Filters Settings Processing Match Setting Rock 1(5)_Counts.map	<ul> <li>Setup pane toolbar</li> </ul>
Kock 1(5)_LOUNTS.map         STA-NB-256(1)_Counts.map         Rock 1(5).MAP.EDS         STA-NB-526(1).MAP.EDS         STA-NB-526(1).MAP.EDS         STA-NB-526(1).MAP.EDS         Strange Mineral(2).MAP.EDS         Ineat rock(4).MAP.EDS         Inuone(3).MAP.EDS         Inuone(3).MAP.EDS	_ file list pane

The Controls Panel includes these main features:

- Analytical Mode Toolbar
- Acquisitions Toolbar
- Analytical Mode Setup Pane
- File List Pane

### **Analytical Mode Toolbar**

Use the large toolbar at the top of the Controls Panel to select the analytical mode.

```
Figure 5. Analytical Mode Toolbar
```



The toolbar is divided into two groups of analyses, Microanalysis and Feature Sizing. <u>Microanalysis</u> contains the modes for X-ray data acquisition and analysis. <u>Feature Sizing</u> contains modes for measuring, counting and identifying particles.

#### **Microanalysis Modes**

There are five analytical modes for the microanalysis technique:

- **Spectrum**. Performs an X-ray analysis of an area of interest on a sample, including element identification and quantification if desired, using a selected detector.
- **<u>Point ID</u>**. Acquires an electron image. Then lets you specify numbered points and areas on the image, and then acquire spectra at those points and areas. Each spectrum is stored along with its x, y location on its associated electron image.
- <u>Spectral Imaging</u>. Acquires an electron image. Then lets you acquire a spectrum at every pixel in the electron image (The spectra may be acquired at a different resolution than the electron image).

These optional features may also be also available:

- Acquire <u>SEM</u> image at same resolution as spectral image
- Acquire <u>WDS</u> image
- <u>Linescan</u>. Acquires an electron image. Then lets you acquire a series of spectra along a linescan (vector).
- **WDS**. Lets you acquire WDS data (quantitative, qualitative and images) using an image using an installed parallel beam wavelength spectrometer.

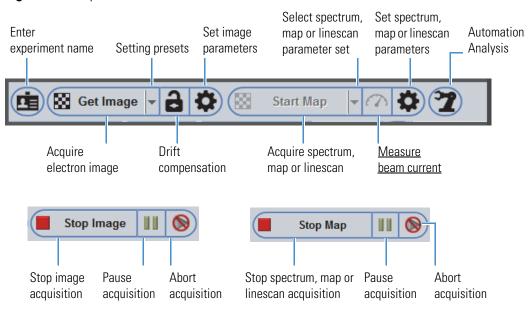
#### **Feature Sizing Mode**

Feature Sizing is an optional software module (PF-Particle) that lets you measure, count, and identify particles and subparticles (particles touching or surrounded by another particle) of interest in a sample.

#### **Acquisitions Toolbar**

The acquisitions toolbar is common to all the microanalysis analytical modes. This toolbar contains buttons for starting, stopping, and pausing data acquisition for images, spectra, maps and line scans. It also includes buttons for editing acquisition settings.

The Drift Compensation. Beam Current, and Automation Analysis controls appear only when the optional Drift Compensation, Beam Current measurement and/or Automation Analysis software are installed.





### **Analytical Mode Setup Pane**

The analytical mode setup panel changes with the selected analytical mode. The setup panels include all the available processing and display settings for the selected analytical mode.

Use the icons in the toolbar at the bottom of the Setup panel to select a setup tab. The settings on that tab are displayed in the space above. The example below shows the settings on the Spectrum Settings tab.

ectrum Setting	js 🗸	Typical/Advanced View toggle
Spectrum Color: Background Color: Cursor Color:	Log     ✓ Cursor       Smooth     Qutine       Grid       ✓ Histogram	
Peak	✓ Lines On ✓ Smart Labels  Short (Fe)  Font Tahoma 11	Spectrum Settings
<ul> <li>Show KLMs</li> <li>Scale Peaks</li> <li>Escape Peaks</li> <li>Sum Peaks</li> </ul>	Include Shells: All  Transition Type: All Intensity above % of max All	
Step otes	Map Image Image Spectrum Spectrum Spectrum Spectrum Spectrum Spectrum Spectrum Spectrum Spectrum Strings	Click a tab to display its settings

Figure 7. Analytical Mode Setup Panel, Spectral Imaging Mode, Spectrum Settings, Typical View

Click to display step-by-step instructions for Spectrum Settings

The toggle button in the upper right switches between typical settings and advanced settings.

The Step Notes button displays step-by-step instructions for the selected analytical mode.

#### **File List Pane**

The file list pane at the bottom of the Controls Panel lists all files in the open project. The file types listed depend on the selected analytical mode. Click a filename to view its data.

Figure 8. file list pane

<ul> <li>turbine-blade-coating.csi</li> <li>Rock 1(5)_Counts.map</li> <li>STA-NB-256(1)_Counts.map</li> <li>Mineral(8).MAP.EDS</li> <li>Mineral(9).MAP.EDS</li> <li>Rock 1(5).MAP.EDS</li> <li>STA-NB(2).MAP.EDS</li> <li>STA-NB-256(1).MAP.EDS</li> <li>STA-NB-256(1).MAP.EDS</li> <li>STA-NB-512(1).MAP.EDS</li> </ul>	File management panel
--	-----------------------------

The file list pane includes a panel of file management options described in the table below.

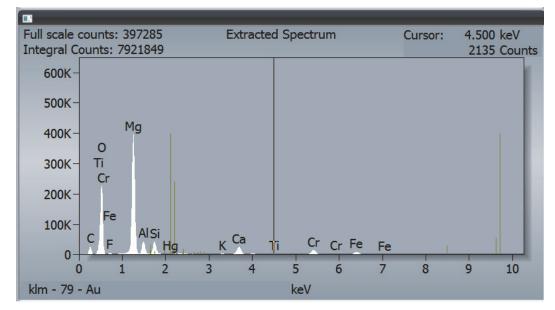
	Table 1.	file list pane,	file management	panel
--	----------	-----------------	-----------------	-------

lcon	Feature	Description
AI	Rename	Enables editing of a selected filename
$\oslash$	Delete	Permanently deletes the selected file
M <sub>200</sub>	Compare	Overlays the selected spectrum with other spectra in the folder for visual comparison
Ð	Match	After running a spectral Match, overlays the original spectrum with the best matched spectra from the spectral library for visual comparison
M.	Spectra Check	Overlays primary spectra with spectral check results (synthetic spectrum/background spectrum/residual spectrum) for visual comparison)
(i)	Information	Displays basic information about the selected spectrum, such as acquisition date/time, voltage, etc. Click <b>Details</b> to show all file attributes for the selected file.

### **Spectrum Pane**

The spectrum pane appears in all the analytical modes except Linescan and shows the most recently acquired spectrum. The pane size and location depend on the selected analytical mode. If you change the analytical mode, the displayed spectrum remains in the spectrum pane until you select a new sample file.

Use <u>Spectrum Settings</u> on the Controls panel to adjust the appearance of the spectrum pane or displayed spectra.

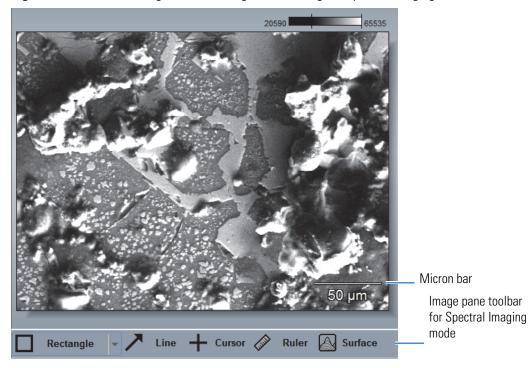


**Figure 9.** Pathfinder Spectrum Pane Showing Extracted Spectrum in Spectral Imaging Mode

### **Image Pane**

The image pane shows the acquired electron image of the microscope field of view for the selected analytical mode and sample. The image pane appears in the upper left portion of all the analytical modes except Spectrum mode, which does not use an electron image. It also appears in the Feature Sizing Analysis mode. In Spectral Imaging mode, you can perform extractions on images for further analysis.

Use Image Settings on the Controls panel to adjust the appearance of the image pane or the displayed electron image.



The features on the image pane toolbar are different for each analytical mode.

### **Figure 10.** Pathfinder Image Pane Showing Electron Image in Spectral Imaging Mode

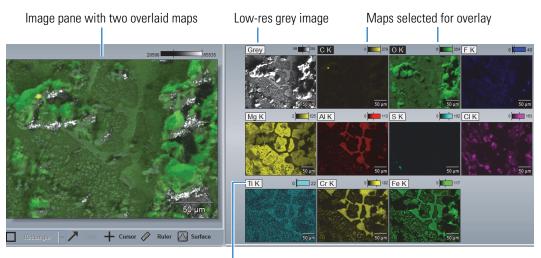
### **Maps Pane**

The maps pane appears in the upper middle portion of the screen in Spectral Imaging mode only. It shows any <u>X-ray maps</u>, <u>COMPASS components</u>, or <u>phase data</u> that have been extracted from the <u>electron image</u>.

Use the <u>Element Setup pane</u> to specify which elements will be mapped and assign them colors. Use the tools on the image pane toolbar to create the maps.

Below is an example of the maps for seventeen elements. Each map shows the distribution of the associated element over the analyzed region. The map labeled "Grey" in the upper left corner of the map pane shows a low-resolution black and white version of the image displayed in the image pane. The low-resolution image is acquired simultaneously with the spectral imaging data.





Mapped element

### **Element Setup Pane**

The Element Setup pane allows you to select elements for automatic analysis in all the analytical modes. (Right-click an element to see the available options, or left-click to scroll through them.) You can also manually identify elements in a spectrum.

The Element Setup pane appears in the lower left portion of all the analytical modes. The elements are presented in the format of a standard periodic table for easy identification.

H																	He
Li	Be											В	С	Ν	0	F	Ne
Na	Mg											A	Si	Р	S	Cl	Ar
К	Са	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Те	Ι	Xe
Cs	Ba	L	Hf	Та	W	Re	Os	Ir	Pt	Au	Hg	TI	Pb	Bi	Po	At	Rn
Fr	Ra	A															
		La	Ce	Dr	Nd	Dm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	
		AC	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr	
x		Possib	ble	)	<	Exc	duded		x		Select	ed	5	•	٧	VDS	
x		Indud	led		<	Alt	ways		x		Nea	r					
Id	lentif	y					P	'rev/N	ext	<b>•</b>					С	lear	

Figure 12. Pathfinder Element Setup Pane Showing Periodic Table of Selectable Elements

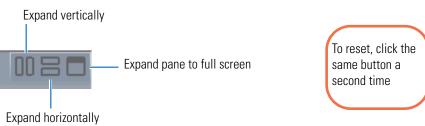
### Adjust the Sizes of the Panes

The Pathfinder Home screen can be customized to suit your preferences by resizing the panes.

To resize a pane, drag its border up, down, right or left.



Hover the mouse cursor over the upper right corner of each pane for additional sizing options. For example, the maps pane has these options:



#### Note

- Pathfinder software is designed for a wide screen (16/9) monitor. For best viewing, maximize the Pathfinder application window.
- Each analytical mode has a different display configuration.

### **Overview of the Menus**

Menu and submenus may change when you select a different analytical mode, or change the active pane within a mode. For example, in Point ID, Spectral Image, or Linescan mode, click the image pane to show the Image menu on the menu bar. Then click the spectrum pane and the Image menu switches to the Spectrum menu.

Menu entries are active (selectable) only when applicable.

Figure 13.	Changing Menus E	xample
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File Edit View Batch Processing Help

File	Edit	View	Spectrum	Batch Processing Help
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**Table 2.**Overview of Menus

Menu Name	Option	Function
File	Project Explorer	Used to open, create or edit a project and its associated files
	Close Project	Closes the current project
	Create Template From Project	Creates a new template from the current project
	System Security	Password protects project settings and service screens from unauthorized changes
	Open	Allows you to select a file to open (not a project)
	Close	Closes the current file
	Save	Saves the current file under its current name
	Save Map File	Available for Spectral Imaging mode only. Can be used to save an extracted set of maps and their element IDs along with the electron image but not the associated spectra. Map information is saved with the same filename as the original data and a .map extension.

Menu Name	Option	Function
	Import/Export	<i>Export Image as CSV</i> Exports an electron image as a Comma Separated Values file
		<i>Export Image as Bitmap</i> Exports the electron image as a bitmap file. The image is exported exactly as displayed in the image pane and at the same resolution.
		<i>Export Image as Full-Res TIF</i> Exports an electron image as a full-resolution TIF image
		<i>Export Linescan as CSV</i> Exports the selected linescan image as a Comma Separated Values file
		<i>Import Gatan File</i> Allows you to import a Gatan spectral image file. Appears in the Import/Export menu only when the optional <u>3rd Party Remote</u> (portal) software is installed.
	Page Setup	Opens a tabbed series of boxes to define how data will be printed for each analytical mode
	Compress SI File	Allows you to select a folder of acquired data for additional compression, resulting in file sizes that are reduced by a factor of 10. (All acquired data are partially compressed during acquisition.)
	Exit	Closes Pathfinder software
Edit	Сору	Copies image in selected Image, Map or Spectrum pane to the Windows Clipboard so it can be pasted into other applications. (To select the Image, Map or Spectrum pane, click once just inside the pane border so that the pane is temporarily highlighted.)
	Сору То	Used to save image in selected Image, Map or Spectrum pane in jPEG (*jpg) format to selected folder
	Erase All Points	In Point ID mode, erases all points on the image (before acquisition)

Menu Name	Option	Function
	Microscope Parameters	Displays the Microscope Parameters and Status Bar Selection box, which lets you set microscope parameters and select which settings will appear on the Pathfinder status bar
	Properties	Opens a tabbed box that contains all the available properties for the selected Microanalysis or Feature Sizing mode. These are the same property settings available in the Controls Panel setup panes.
	Language	Allows you to select an available display language for Pathfinder software. The default setting (Default Operating System Language) displays the software in the same language as the Windows Operating System software, if that language is available.
	Set Faraday Cup Stage Position	Sets x, y stage position for the Faraday cup when it is on the stage (requires stage automation)
View	Restore Toolbars	Redisplays the (floating) main toolbar after it has been closed
	Status Bar	Shows or hides the Pathfinder <u>status bar</u>
	Drift Diagnostics	Displays drift compensation diagnostics window
	Attributes	Available for Microanalysis Spectral Imaging mode only. Displays attributes of the microscope and maps of displayed SI data.
	Application Look	Sets overall color scheme for Pathfinder Home screen
	Customize	Allows you to customize Pathfinder menus and toolbars and specify display options for tool tips and shortcut keys
	Service Mode	Allows accesses to user service functions including System Configuration, EDS energy calibration, and image calibration.
	<u>Electron Imaging</u> <u>Mode</u>	Used to acquire, save, and load electron image file
	Preferences	Lets you <u>set user preferences</u> for panels and display panes. User preference settings are maintained even after Pathfinder software is reinstalled.

Table 2.         Overview of Menu	S
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Menu Name	Option	Function
Image	Return To Stage Position	Image menu appears on menu bar when image pane is selected in Point ID, Spectral Image, or Linescan mode.
		Return To Stage Position option moves stage to sample location where the currently displayed spectrum was acquired (requires stage automation). Assumes the sample is still mounted on the microscope stage.
Spectrum	Remove Escape Peaks	Spectrum menu always appears on menu bar in Spectrum mode and when spectrum pane is selected in Point ID, Spectral Image, or Linescan mode.
		Use Remove Escape Peaks to toggle any found <u>escape peaks</u> on and off for the displayed spectrum.
	Remove Sum Peaks	Toggles any found <u>sum peaks</u> on and off for the displayed spectrum
	Append Notes To Spectrum Label	Adds first line of spectrum notes to spectrum label when it is displayed
	Element X-Ray Lines	Lets you add or adjust X-ray line energies for elements and their relative intensity values for known KLM lines
	Reference Subtract/Add	Allows peaks in an unknown spectrum to be added or removed after performing quantitative analysis. Used to ensure all elements have been accounted for in the quantification.
	Spectrum Math	Lets you perform mathematical operations on spectra
	Region Tool	Used to calculate gross, net and ratio values for selected elements in a spectrum
	Return to Stage Position	Moves stage to sample location where previous spectrum was acquired (requires stage automation). Assumes sample is still mounted on microscope stage.
Batch Processing	Create Montage	Stitches a selection of images, X-ray maps or COMPASS components acquired using the optional Analysis Automation software so they can be viewed together

#### Table 2. Overview of Menus

Menu Name	Option	Function
	Spectrum Analysis	Available in Spectrum mode only. Used to perform spectral analyses (qualitative, quantitative and spectrum match) on a series of selected spectra. Results are saved in user-defined file with .csv extension.
	Linescan Analysis	Available in Linescan mode only. Allows you to reanalyze a Linescan data set using the current Linescan mode processing settings.
	Point ID Analysis	Available in Point ID mode only. Allows you to reanalyze a Point ID data set using the current Point ID processing settings.
	Extract Quant Maps	Available in Spectral Imaging mode only. Allows you to automatically extract quantitative maps, compass components, or phases from a series of spectral imaging files in the current project as defined in the Batch Extract Quant Maps settings box. Can also be used process a series of files and associate them into a tomography data set.
	Process Tomography Dataset	Provides a method to combine component or phase images into one image. The processing is on all slices of a tomography data set.
Specials	Displayed menu options depend on the application	Specials menu appears only when special versions of Pathfinder hardware and/or software are installed. See the documentation that came with your specialized system for details on Specials menu commands.
Help	Help Topics	Displays this Help system for learning Pathfinder software
	What's This	Displays Help information in the Analytical Mode Setup panel. Information is relevant to the selected analytical mode.
	About Pathfinder	Displays version numbers for Pathfinder software

#### Table 2.Overview of Menus

### **System Security**

Use System Security in the File menu to protect selected Pathfinder features (including data acquisition) with an administrator password. This is helpful in a multi-user environment with established protocols to ensure that all EDS acquisitions are performed under the same conditions. When project templates, acquisition properties and/or service views are protected and locked, those system parameters are displayed but cannot be changed by the user.

#### \* To specify a password and select which Pathfinder features to lock

1. Choose File > System Security.

A message prompts for the administrator password.

Lock Projects	8
Pathfinder Administrator Password Required	
Password:	
OK Cancel	

2. Enter the administrator password (or choose OK for the default password).

**Note** Pathfinder software is installed with one user name and no administrator password, and all security settings are off. Additional users and passwords may be added at your discretion—consult your Windows documentation for more information.

In the Security Setup box, select the features to protect, enter and confirm your password and choose **OK**.

System Security Setup
✓ Lock Templates
Lock Acquisition Properties
✓ Lock Service Views
Administrator Password — You may leave the existing password if you so desire.
New Password: Confirm New Password:
OK Cancel

Protection options:

• Lock templates. Locks the current project templates for all the analytical modes. Project templates remain available for use but they cannot be changed or reset.

- Lock acquisition properties. Locks the acquisition settings for all the analytical modes. Users can view acquisition settings defined in project templates but not change them.
- Lock service views. Locks the user service mode (View > Service Mode). Users cannot display or change the user service mode settings, which include <u>System</u> <u>Configuration</u>, <u>EDS energy calibration</u>, and <u>image calibration</u>)

#### \* To lock the system

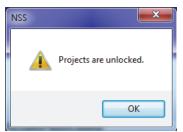
- 1. Choose File > System Security > Lock System.
- 2. Enter the administrator password and choose OK.

A message indicates the system features that are selected for protection (i.e., templates, acquisition settings and service views in the previous example) are locked. (If templates and acquisition properties are selected in the System Security Setup box, these features are locked for all projects.)

#### ✤ To unlock the system

- 1. Choose File > System Security > Unlock System.
- 2. Enter the administrator password and choose OK.

The system features selected for protection are unlocked. (If templates and acquisition properties are selected in the System Security Setup box, these features are unlocked for all projects.)



#### ✤ To remove password protection

- 1. Choose **File > System Security > Unlock System**, enter the administrator password and choose **OK**.
- 2. Choose **File > System Security > Security Setup**, enter the administrator password and choose **OK**.
- 3. In the System Security Setup box, clear all the check boxes, delete the passwords and choose **OK**.

### **Customize the Pathfinder Interface**

The Pathfinder color scheme, toolbars, buttons and background colors can be customized to suit your preferences.

#### **Choose Theme**

- \* To change the overall color scheme of the Pathfinder Home screen
- 1. From the Home screen, choose **View > Application Look**.
- 2. Select a color scheme from the submenu.

#### **Customize Toolbars and Menus**

#### \* To customize Pathfinder toolbars and menus

1. From the Home screen, choose View > Customize.

The Customize box is displayed.

Customize	
Customize           Commands         Toolbars         Menu           Categories:         File         Edit           Edit         View         EBSD         Batch Processing           Specials         Help         Help         Help	Options Commands: Close Project Create Template From Project
New Menu All Commands	Lock System
	Unlock System
Description:	
	Close

2. Customize Commands, Toolbars, Menus and Options as needed.

### **Set User Preferences**

You can select specific colors for borders and backgrounds, border widths and styles and whether tool tips are displayed. These settings are maintained even after you reinstall the software.

#### \* To set user preferences for the Pathfinder interface

1. From the Home screen, select **View > Preferences**.

The Preferences box is displayed.

Preferences	
Properties for All Displays	
Background Fill :	
<ul> <li>Solid Fill</li> </ul>	
Themed Fill	
Themed Gradient	
Border	
Border width 5	
Show Shadow	
Control Panel Options	
Show text labels on toolbars	
Periodic Table Options	
Show Legend	
OK Apply Cancel	

Available options:

- **Background**. Sets the background color for the Image, Maps, Element Setup and Spectrum panes on the Pathfinder Home screen.
  - **Solid fill**. Lets you choose the background color.
  - Themed Fill. Uses the selected theme (View > Application Look) for the background color.
  - **Themed Gradient**. Uses a gradient version of the selected theme for the background color.
- **Border**. These options affect the color and width of the selection box that shows up briefly when you click inside a selectable pane, such as the image, map or spectrum pane. Click just inside a pane border to select the pane, for example, to <u>copy</u> the pane's contents to the Clipboard.
  - Color. Allows you to define a color for the selection box.

- **Width**. Allows you to specify the width of the selection box.
- **Show Shadow**. Adds a shadow to the displayed image in the image pane and to the displayed spectrum in the spectrum pane.
- **controls panel**. These options affect only the <u>controls panel</u> at the right side of the Pathfinder interface.
  - **Show text labels on toolbars**. Displays or hides text labels below button icons on the toolbar in the Analytical Mode Setup pane.
- **Periodic Table Options**. These options affect only the <u>Element Setup pane</u> in the lower left portion of the Pathfinder interface.
  - **Show Legend**. Displays or hides the current color assignments for selecting elements for analysis in the periodic table.

#### **1** Pathfinder System Overview

Customize the Pathfinder Interface

2

## **Project Management**

All Pathfinder data is stored within projects. Each project can consist of several sub-projects. A project includes the element lists, identification parameters and acquisition properties from the project's previous use.

When you start the Pathfinder software, Project Explorer appears allowing you to select or create a project. When you create a new project, the software automatically creates a new project folder.

Figure 14. New project folder

Organize 🔻 Share	with  Burn New fold	ler		= •
★ Favorites ★ Eavorites	Documents library	1	Arrang	ge by: Folder 🔻
Downloads	Name	Date modified	Туре	Size
Sector Dropbox	- 📔 ~temp	3/21/2016 9:18 AM	File folder	
Recent Place:	AcqProps.tnp	3/9/2016 3:02 AM	TNP File	7 KB
🔁 Libraries	NSS Lock.txt	3/21/2016 9:18 AM	Text Document	1 KB
Documents Documents Music Pictures Videos	🖭 NSSProject.xml	3/21/2016 9:18 AM	XML Document	1 KB

Template file

Temporary data folder

Project settings are stored in a template file in the project folder. The template consists of the acquisition properties, periodic table settings (including the history), and the spectrum processing setup. Templates are stored with a .tnp extension. A template can be applied to other projects.

When you acquire new data (or process existing data), the data and its associated parameter settings are saved in a temporary folder using a base filename and inherit the project's settings. When you create a child project under a parent project, the child project inherits all of the parent project's settings.

When you close a project or exit the software, you are asked whether to permanently save any unsaved data with the current project. If you choose to save a file with the project, the file is moved from the project's temporary folder to the main project folder. The temporary folder is then removed along with any non-selected data.

E' 4E	0 ()	. 1	1 4 1	
Figure 15.	Software message to	nermanently save	data when y	In close a project
ingano ioi	oonthato moodago to	pormanonici, ouro	aata milon	

Save Modified Project Data	J
Some project data has been modified. Select the files you wish to save. All other changes will be lost.	
I Strange Mineral(2).MAP.EDS	
Clear All OK Cancel	

**Note** If you are working with data stored in the temporary folder (not yet saved in a project) and experience a power failure, the data will be in the temporary folder when you restart Pathfinder software.

### **Project Explorer**

Similar to Windows Explorer, Project Explorer lets you manage projects and their data files. You can create new projects and folders and move (drag) or copy (drag + Ctrl) data files from one project to another.

When you start the Pathfinder software, Project Explorer appears allowing you to select or create a project.

Use the **Properties pane** to enter information about the project. Use the **Explorer pane** for project manipulations.

**NOTICE** Always perform data and file manipulations using Pathfinder Project Explorer. (Do not use the Windows Explorer.)

Project Explorer pane		Properties pane
Project Explorer		
File Edit		
Image: Second		OK Cancel
		Author:
My eBooks		Person that created new project.
My Music		Key Words:
My Pictures		Put searchable words here.
My Videos		Client:
New Project		Put the origin of the sample(s) here.
		Due Date:
		Put date the project was created here.
		Project Template:
	=	default.tnp   Reset
		Notes:
		Put any notes that are relavent to the project here.
	Ψ.	<u></u>

### **Figure 16.** Pathfinder Project Explorer Window

**Note** Right-clicking a folder in the Project Explorer pane brings up a menu containing all the Project Explorer <u>File menu</u> and <u>Edit menu</u> commands.

### **Project Explorer Properties**

Table 3.	Project Explorer Prop	erties Panel Entr	y Descriptions
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Field Name	Description
Author	Enter your name
Key words	Enter any key words to associated with this project to help you identify this project using <u>Project Search</u>
Client	Enter client's or recipient's name if applicable
Due Date	Enter a date (typically the project creation date)
Project Template	Select an existing project settings template (*.tnp) file, or the default template file (default.tnp)
Notes	Enter any relevant notes about the project

Use the **Reset** button to restore the project to the settings used when the project was created.

### **Project Explorer Menus**

You can access the following commands through the File and Edit menus within Project Explorer:

#### File

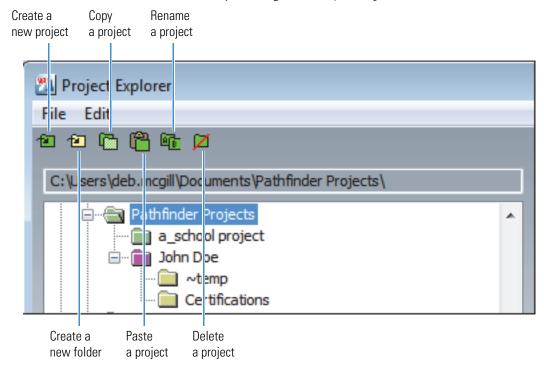
- New Project (creates a new project folder)
- New Folder (creates a new folder that is not considered a project)
- Open in Windows Explorer (displays project or folder contents in Windows Explorer)

#### Edit

- Project Search (search all projects for entered key words)
- Remove Project Lock (unlock a project folder)
- Copy (a project or folder)
- Paste (a project or folder)
- Rename (a project or folder)
- Delete (a project or folder)

#### **Project Explorer Toolbar Buttons**

You can access the menu commands by clicking these Project Explorer toolbar buttons:



### **Create a New Project**

#### ✤ To create a new project

1. Open the Project Explorer window.

From the Home screen, do one of the following:

- Click the Project Explorer button an the main toolbar.
- Choose File (menu) >Project Explorer.
- 2. Select the folder where you want to create the project folder (typically the Documents library).
- 3. Create a new folder by right-clicking the parent folder and choosing **New Project** from the shortcut menu.

A folder named New Project is added to the parent folder.

- 4. Rename the folder by doing one of the following:
  - Click the folder name to enable editing (as in Windows Explorer) and enter a new project name.
  - Right-click the folder, choose **Rename** from the shortcut menu and enter a project name.
- 5. Enter the project information in the Properties pane.
- 6. Choose OK.

The project folder is created under the folder you selected. The name of the new (active) project appears in the Pathfinder title bar.

**Note** You can also use New Folder in the Project Explorer <u>File menu</u> to create new folders that are not considered projects.

### **Change to a Different Project**

There are two ways to change projects:

• Open Project Explorer (click Project Explorer button on main toolbar or choose **File** (menu) > **Project Explorer**).

Locate and select the project you want to open (project folders are green):

• If you know the project folder's location, navigate to the folder, click it and choose **OK**.

• If you do not know the project's location, right-click the parent folder and choose **Project Search**. Enter a keyword or select one from the drop-down list and choose **OK**. The project folders that include that keyword are displayed in bright pink. Click the desired folder and choose **OK**.

If there is unsaved data from the previous project, a message asks whether you want to save or discard the data. After you respond, the previous project closes and the new one opens.

When the new project opens, its name appears in the Pathfinder title bar.

• From the Pathfinder Home screen, choose **File > Close Project**. If there is unsaved data, a message asks whether you want to save or discard the data. After you respond, the previous project closes and the software opens Project Explorer. Locate and select a new project and choose **OK**.

### **Create a New Project Template**

You can save the current project settings, including acquisition, processing and quant settings, as a template that can be applied to a new project for the same sample type. This saves time and ensures consistent experiment setup and analysis.

- \* To save the current project settings as a new template
- 1. From the Pathfinder Home screen, choose File (menu) > Create Template From Project.
- 2. Navigate to the Pathfinder Project Templates folder (default).

**Note** You can save templates in other folders. However, only those templates stored in the Pathfinder Project Templates folder will be visible when you create a new project.

3. Enter a name for the new template and choose Save.

The acquisition properties, quant settings, periodic table settings with the history, spectrum processing setup, and Feature Sizing parameters are saved in the template. The new template is stored with a .tnp extension.

### **Protect Project Templates**

Use <u>System Security</u> in the File menu to prevent unauthorized modification of project templates.

- To protect project templates with a password
- 1. From the Pathfinder Home screen, choose **File > System Security**, enter the administrator password (or leave the Password box blank for the default password) and choose **OK**.

- 2. In the <u>Security Setup</u> box, select **Lock Templates**, enter and confirm your password and choose **OK**.
- 3. From the Pathfinder Home screen, choose File > System Security > Lock System.
- 4. Enter the administrator password and choose OK.

A message indicates that the project templates are locked.

Pathfinder		×
<b></b>	Projects have been locked.	
	ОК	

5. Choose **OK** to close the message box.

### **Reset a Project Template**

Resetting a project restores the template settings for that project.

#### To reset a project

1. Open the Project Explorer window.

From the Home screen, do one of the following:

- Click the Project Explorer button 🔲 on the <u>main toolbar.</u>
- Choose File (menu) >Project Explorer.
- 2. Select the project in the left pane.
- 3. Click the **Reset** button in the right pane.

The project settings are returned to the settings from the template file used to create the project.

### **Pathfinder Files**

Most Pathfinder files are stored in industry standard formats, such as EMSA for spectra files, TIFF for image files, etc. Due to their unique nature, Spectral Imaging files are stored in a Thermo Fisher Scientific proprietary format. You can save data in an industry standard format by switching to a mode that supports the file format. For example, switch from Spectral Imaging to Spectrum mode to save an EMSA spectrum file.

You can open many of the files by using a text editor like Notepad.

You can save a file at any time by clicking the Save button on the <u>main toolbar</u> or choosing **Save** from the File menu. To change the file's name, select the file in the file list pane, click **A**] on the file management panel and type the new name. You can also select a pane and use Copy To in the Edit menu to save the pane in a JPEG file.

The table below describes the Pathfinder file types.

**Note** All TIFF files contain both an 8-bit image and a 16-bit image. The 8-bit image is used by third-party programs such as Microsoft Word. The 16-bit image is used within Pathfinder software.

.apf .chem .clib	Automation points fileFeature sizing chemical type fileFeature sizing chemical library file
.clib	Feature sizing chemical library file
.csi	Spectral Imaging control file. Contains the names of files used for the Spectral Imaging acquisition.
.em	Element map if phase data came from map input in TIFF format
.emsa	Spectrum files stored in industry-standard EMSA format
.fss	Feature Sizing setup file
.fzm, .fzma or .fzme	Map from optional XPhase processing in TIFF format
.fzs, .fzsa or .fzse	Spectrum from optional XPhase processing in EMSA format
.grid	Automation grid file
.jpg	Image file saved in JPEG format (Edit > Copy To)
.lscan	Linescan file
.lsctl	Linescan control file
.lsmsa	Spectrum files for each pixel in a linescan stored in industry-standard EMSA format
.lsref	Linescan reference image in TIFF format
.map	Element maps control file
_map.tif	X-ray map file in TIFF format
.mtdb	Spectral match database
.p_s	Point ID control file
.pcm or .pcma	Compass image file in TIFF format
.pcs or .pcsa	Compass spectrum file in EMSA format
.psmsa	Point ID spectral file in EMSA format

**Table 4.**Pathfinder File Types

File Type (Extension)	Description
.psref	Point ID reference image in TIFF format
.ref	EMSA-compliant file containing standardless references
.si	Spectral Imaging data file
.simcs	Spectral Imaging concurrent external input map file in TIFF format
.siref	Spectral Imaging reference image file in TIFF format
.sitif	Spectral Imaging concurrent video image file in TIFF format
.std	EDS standards spectral data file stored in EMSA format
.stn	Standards reference file in EMSA format
.tif	Image file in TIFF format
.tnp	Project template file
.wstd	WDS standards spectral data file stored in EMSA format

Table 4.	Pathfinder	File	Types
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### **Import Files into the Current Project**

- To import a file into the current project
- 1. Select the analytical mode that corresponds with the file type you want to import.
- 2. From the Pathfinder Home screen, choose File > Open.
- 3. Navigate to the project folder that contains the file.
- 4. Select the file and choose **Open**.

The data is copied to the current project's temporary folder and inherits the current project's settings. To permanently save the file in the project, click the **Save** button on the main toolbar or choose **File > Save**.

### Save a File in a Project

When you close a project, Pathfinder displays a list of files that have been acquired or modified in the current session. Deselect those files you do NOT want saved. Choose OK to save the changes, or choose Cancel to close the Save dialog box and return to the project without saving any changes.

#### \* To save a file without closing the project

Click the Save button **main toolbar** to save the file in the currently open project.

Or:

From the Pathfinder Home screen, choose **File > Save**.

The file is moved from the temporary folder to the project folder.

### **Export Image or Map Data**

Use one of these options in the File menu to save an electron image or map to a different file format:

- Export Image as CSV. Saves the currently displayed or extracted image or maps (component, phase or X-ray) as a a series of CSV (.csv) files (one file for each element specified as Included or Always Included, plus one for the low-res grey image). The CSV files are saved in a location you specify with a base name you specify followed by the element name.
- **Export Image as Bitmap**. Saves the currently displayed electron image as a bitmap (.bmp) file. If the micron bar is set to be overlaid on the electron image, it will be exported as such in the bitmap. If the micron bar is set to be displayed in the header, it will not be exported in the bitmap. The bitmap file is saved in the corresponding Point ID or Spectral Imaging project folder with the sample name (displayed in the file list pane) followed by "export" as the filename.
- **Export Image as Full-Res TIF**. Saves the currently displayed electron image at the full resolution of the acquired data as a TIFF (.tif) file. If the micron bar is set to be overlaid on the electron image, it will be exported as such in the .tif image. If the micron bar is set to be displayed in the header, it will not be exported in the .tif image. The TIFF file is saved in the project folder with the sample name (displayed in the file list pane) followed by the resolution (24bit) as the filename.
- **Export Linescan as CSV**. Saves the currently selected linescan as a CSV (.csv) file. You can enter a filename for the CSV file and select the folder where the CSV file is saved. The default folder is the current project folder, and the default filename is the sample name (displayed in the file list pane).
- **Import Gatan File**. Allows you to import a Gatan spectral image file. Appears in the Import/Export menu only when the optional <u>3rd Party Remote</u> (portal) software is installed.

### **Search For a Project**

You can search for specific projects using keywords. The same keyword or words can be used in multiple projects.

#### \* To add keywords to a project

- 1. Open Project Explorer (click Project Explorer button **button** on main toolbar or choose **File** (menu) > **Project Explorer**).
- 2. Select the project folder.
- 3. In the Properties panel, enter one or more keywords in the Key Words box, separating each word or phrase with a comma.

1	-Properties Author:	
	John Doe	<b>-</b>
	Key Words:	
	School, 2011, final data	-

4. Choose OK.

#### To find projects using keywords

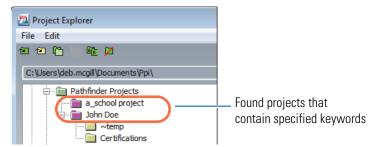
1. Open Project Explorer (click Project Explorer button on main toolbar or choose **File** (menu) > **Project Explorer**).

There are three ways to start a project search:

- In the Project Explorer window, right-click a high level folder (for example, Pathfinder Projects) and choose **Project Search**.
- In the Project Explorer window, select a high level folder and choose **Edit > Project Search**.
- In the Project Explorer window, select a high level folder and press **F3** on the keyboard.
- 2. Enter a key word or words (with a comma separator) or select a key word from the list box and choose **OK**.

Key Word Search
Enter key words:
school, 2011 💌
OK Cancel

3. The software searches all projects in the selected folder. Found project folders are highlighted in pink.



### **Open a Project in Windows Explorer**

Use Project Explorer's Open In Windows Explorer menu option to display the contents of a selected Pathfinder project or other folder in Windows Explorer, for example, to copy an exported data file.

**NOTICE** Be careful not to accidentally delete a Pathfinder data file, especially an associated file, or you may render the data unusable.

- \* To open a project folder in Windows Explorer
- 1. Open Project Explorer (click Project Explorer button **button** on main toolbar or choose **File** (menu) > **Project Explorer**).
- 2. Right-click a Pathfinder folder (project or other) and choose **Open in Windows Explorer**.

The software opens a Windows Explorer window and displays all the files in the selected Pathfinder folder.

# 3

# **Spectrum Mode**

This section discusses the Spectrum analytical mode and its associated components. It provides an in-depth look at the tools for setting up, acquiring and analyzing X-ray spectral data (qualitative and quantitative) and all associated features. These tools described here are common to any analytical mode that includes a spectrum, including Spectrum, <u>Point ID</u>, and <u>Spectral Imaging</u>.

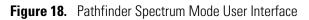
### **Select Spectrum Mode**

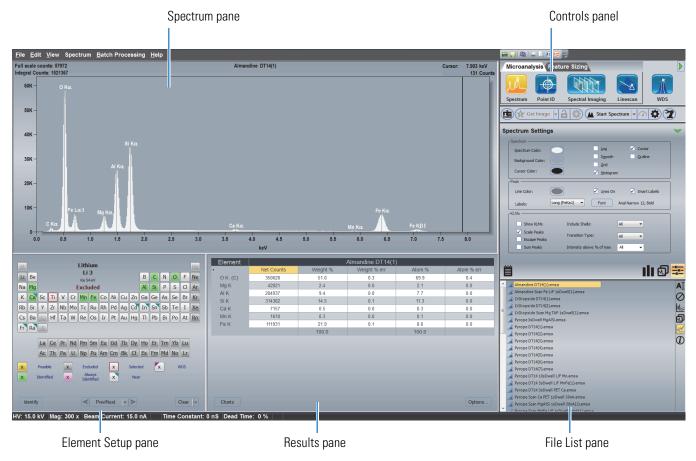
Use Spectrum mode to acquire and analyze an X-ray spectrum. To select Spectrum mode, click the Spectrum button on the analytical mode toolbar.

Figure 17. Select Spectrum mode



In Spectrum mode, the software displays all the features and tools for X-ray analysis of a sample.





### **General Steps for Spectral Acquisition and Analysis**

### **Pre-analysis Considerations**

Sample preparation, microscope operating conditions, and detector geometry all affect the accuracy of any EDS analysis. This section provides general guidelines to consider before you use Pathfinder software to perform an EDS analysis for the first time.

### **Sample Preparation**

The first step with any analysis is sample preparation. The instrument works well with samples that are flat, well polished (1 micron or less final polish), and electrically conductive. For some applications, such as failure analysis, polishing the sample would be counter-productive. For unpolished samples, some topography may affect the line of sight to the detector.

If your samples are non-conductive, apply a conductive coating or use variable pressure if your SEM has that feature. If you apply a conductive coating such as carbon or gold, make sure any quantitative measurements account for the added element in the spectrum. This can be accomplished by identifying the element (i.e., setting it to Identified in the periodic table) but excluding it from the quantitative analysis (i.e., setting its <u>Quant Lines</u> to Absent in the periodic table). The thinnest useful coating of carbon is best for the majority of samples. Be aware that coatings with high atomic number materials will absorb X-rays of low atomic number elements within the sample.

#### **Microscope Considerations**

Position the sample at the optimal (or analytical) working distance for the selected detector. This value is specific to the microscope and is included in the documentation provided with the EDS detector. To do this, first adjust the microscope working distance so the readout (usually labeled "WD" or Focus Point") matches the detector's optimal working distance. Then use the stage Z controls to raise or lower the sample surface to bring the sample into visual focus.

Make sure the detector is fully inserted before you acquire data. The insertion position is determined and set during installation. The detector position affects the efficiency of X-ray collection. Some sample conditions may preclude full insertion of the detector. However, keep in mind that this can cause the system to detect stray X-rays from areas not associated with the sample.

The microscope accelerating voltage determines the energy range of X-rays that are fluoresced and the rate of excitation from a sample. Estimate the highest order of element X-ray lines of interest and set the accelerating voltage 1.5 to 2 times higher than the energy of that line. This is known as the "over-voltage" for that line. For example, if you are analyzing the Copper K $\alpha$ line (8.047 keV), use a minimum accelerating voltage of 13 KV. However, a good starting point is 15 or 20 KV.

The microscope beam current reaching the sample affects how much data can be acquired over a given period of time. Higher beam currents lead to higher count rates, which are useful for mapping large areas in a reasonable amount of time. A lower beam current produces lower count rates and higher quality spectra, which are useful for quantitative analysis. These higher quality spectra have narrower peaks, better peak separation and fewer spectral artifacts such as sum peaks.

To control the beam current reaching the sample, adjust the beam current (referred to as "probe current" or "spot size" on some microscopes). The microscope aperture, located in the beam path in the microscope column, can also be used to limit the amount of beam current that reaches the sample.

"Dead time" is a measure of how busy the pulse processor is when measuring the X-ray events. For mapping applications, a dead time of 50% or less is best. For quantitative applications, keep dead time between 20% and 30%. The chances of inducing spectral artifacts (<u>sum peaks</u>) increases with count rate for a given time constant. Using a lower count rate may require a longer acquisition time to produce sufficient X-ray counts for statistically good analyses.

### **Detector Geometry**

Accuracy depends on many things, including sample preparation and how the analysis area is chosen. Precision mostly depends on how many X-ray counts are acquired, up to a point. The detector characteristics such as the takeoff angle and the window material affect the efficiency of X-rays reaching the detector. This changes as a function of energy (soft X-rays, those with energies less than 1.5 keV, are absorbed more easily than higher energy X-rays).

Depending on the microscope, the takeoff angle for the detector ports can vary from 20 to 40 degrees. The higher the takeoff angle for a given microscope/detector installation the more efficient is the measurement. If your detector has a window, the window material may absorb certain X-rays, which can affect the measurement results if not accounted for in the analysis.

### Acquire and Analyze a Spectrum

This section provides an overview of the steps to perform a spectral acquisition and element identification and quantification.

#### To acquire and analyze a spectrum

1. Prepare the sample.

Place the sample in the microscope and obtain an image. Adjust the stage to the optimal working distance for the EDS detector. Obtain a good electron image of an area of interest on the sample.

- 2. Fully insert the detector.
- 3. Start Pathfinder software.

The Pathfinder Project Explorer window is displayed.

4. Select or create a project and choose OK.

The Pathfinder main screen is displayed.

- 5. Click the Spectrum button on the <u>analytical mode toolbar</u>.
- 6. Enter an experiment name (used as a base name for automatically saving acquired data).

On the acquisitions toolbar, click the 💷 button and enter the experiment name (or leave the default name).

	Experiment Name			x	Start M
_	Base name		ОК		
Ma		_			_

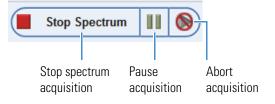
7. Click the Experiment Setup 😧 button on the acquisitions toolbar (normal view toggle setting) to display the available acquisition settings for spectral analysis.

Microanalysis Feature Sizing	Experiment Setup
Experiment Setup	Acquisitions toolbar Normal View
Active Detector(s)  Detector One Detector Two Termination Criteria	toggle setting
Live Time Limit (s): 10   Max Peak Counts: 0 -Energy Range	
Time Constant : Auto  (projected max throughput) User Defined	

- 8. In the Active Detectors group (appears only when multiple Pathfinder-compatible detectors are installed on the microscope), select one or both Pathfinder-compatible detectors for the analysis.
- 9. In the Termination Criteria group:
  - Set Live Time Limit (acquisition time) to 10 seconds.
  - Set Max Peak Counts to 0.
- 10. In the Energy Range group:
  - Set Low Energy Cutoff to 100 eV.
    - Set High Energy Cutoff to Auto.
- 11. Set Time Constant to Auto.
- 12. Adjust the beam current, (spot size or condenser lens) on the microscope to obtain a detection rate of 50% or less dead time. (UltraDry detectors have a high count rate with low dead time.)
- 13. On the acquisitions toolbar, click Start Spectrum to begin the spectral acquisition.



The button changes to Stop Spectrum and the acquisition continues until the termination condition (such as the Live Time Limit) is met.

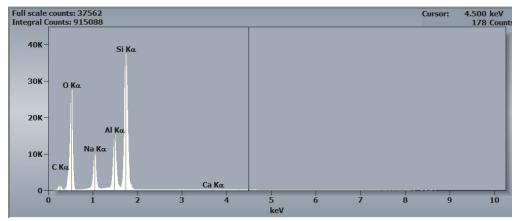


• To pause the acquisition, click the **Pause** button.

To continue the acquisition, click the pause button again.

- To end the acquisition and save the data, click Stop Spectrum.
- To end the acquisition without saving the data, click the Abort button

The live spectrum is displayed in the spectrum pane.



The progress bar at the top of the Pathfinder window shows the progress of the acquisition.



The real acquisition time shown on the progress bar is always longer than the specified <u>Live Time Limit</u> due to <u>dead time</u> when the pulse processor is busy measuring the X-ray events. This is a normal part of X-ray sampling.

14. Wait until the acquisition is completed.

15. If <u>Auto ID</u> and/or <u>Auto Quant</u> are selected (default), element identification and quantification results are displayed and updated at defined intervals during the acquisition and final results are displayed after the acquisition completes.

The qualitative (identification) results are shown as peak labels on the spectrum displayed in the spectrum pane. The quantitative results are displayed in the Results Pane. Here is an example:

Element		A CONTRACTOR OF A CONTRACT OF	lbite(1)20KV_origir		
	Line Type	Intensity	Net Counts	Weight %	Weight % en
СК	K	33.96	6235	3.39	0.08
ОК	K	998.95	183407	45.46	0.23
Na K	K	389.14	71447	8.37	0.05
AI K	K	662.04	121551	10.60	0.06
Si K	K	1833.28	336591	31.93	0.12
SiL (D)	L	0.00	0		
Са К	K	8.70	1597	0.25	0.03
CaL(D)	L	0.00	0		
		3926.08	720828	100.00	0.56

Figure 19. Quantitative results displayed in the results pane

### **Spectral Acquisition Settings**

This section explains the tools and settings for spectral acquisitions that are common to all the analytical modes that involve spectra (all but Linescan), including:

- microscope settings
- X-ray acquisition settings
- settings for element identification
- settings for element quantification
- spectral display properties
- advanced analytical methods such as Spectral Match.

The tools and settings are discussed in logical order for setting up a spectral analysis.

### **Microscope Settings**

For any analysis, the software requires the current microscope accelerating voltage, working distance and magnification values.

### **Enter Microscope Settings**

Most modern microscopes allow the software to read the microscope accelerating voltage, working distance and magnification values automatically when they are needed. If your microscope does not include column automation, enter these values before you start an analysis (see the instructions below) and update the values whenever the Microscope Parameters box is displayed.

Skip this section if your microscope includes column automation, which allows our software to read these values automatically when they are needed.

#### To enter microscope settings manually

1. From the Pathfinder main window, choose Edit (menu) > Microscope Parameters.

The Microscope Parameters and Status Bar Selection box is displayed.

Figure 20. Microscope Parameters and Status Bar Selection Box

Microscopic Parameters and Status Bar Selection						
Operating Parameter	Value	Operating Parameter	Value			
✓ Accelerating Voltage (KV)	15.000	Stage (X,Y) Coord (mm)	0.000	0.000		
Magnification	1000	Stage <u>Z</u> Coord (mm)	0.000			
Beam <u>C</u> urrent (nA)	0.000	Stage <u>T</u> ilt (°)	0.000			
Working Distance (mm)	15.000	Stage <u>R</u> otation (°)	0.000			
EDS Slide Position (mm)	60.000	Stage <u>B</u> ank (°)	0.000			
✓ EDS Dead Time	32	✓ <u>S</u> torage Count Rate	4800			
Time Constant		Take Off Angle	30.000			
Column Stage Beam Current Faraday Cup Close						
* Use check boxes to select items to be displayed in the status bar. Display On Acquisitions Start						

- 2. Enter the accelerating voltage in KV.
- 3. Enter the magnification.
- 4. Enter the microscope working distance in mm.

#### Note

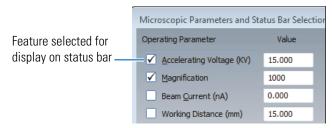
- If Column Communications software is installed, these parameters are updated automatically.
- The Microscope Parameters can be displayed automatically at the start of an acquisition by selecting **Display On Acquisition Start** (see image above). This option is helpful if Column Communications are not installed.

#### **Display Microscope Settings on the Status Bar**

The status of various microscope parameters can be monitored on the status bar, which appears in all analytical modes. Good choices include Accelerating Voltage, Magnification, Working Distance, Time Constant and Dead Time.

#### \* To display microscope settings on the status bar

- 1. From the Pathfinder main window, choose **Edit** (menu) > **Microscope Parameters** to display the Microscope Parameters and Status Bar Selection box.
- 2. Click the check box adjacent to the settings you want displayed so the check box is selected. Here is an example:



**Note** To show or hide the status bar, from the Pathfinder main window, choose **View** (menu) > **Status Bar** so the menu command is selected.



### **X-ray Spectral Acquisition Settings**

#### **Experiment Name**

Before you begin taking measurements, enter an experiment name by clicking the button on the acquisitions toolbar and entering a name for your experiment (or leave the default name). The experiment name is used as a base filename for automatically saving acquired data.



### **Spectral Acquisition Presets**

In Spectrum mode, there are four setting presets available for acquiring X-ray spectra without having to set individual parameters. To select a preset, click the down arrow at the right of the Start Spectrum button.

Figure 21. Acquisitions toolbar, X-ray spectral acquisition presets

Get Image 🗸 🔒 🔇		Start Spectrum 🔻 🤇	<b>\$</b> 7
Spectrum Processing		High Resolution	
opeen unit rocessing		High Throughput	
R Ident Setup	☆	User Defined 1	
Auto ID	*	User Defined 2	

These selections are available:

- **High Resolution**. Longest (and slowest) Time Constant setting for high resolution spectral acquisition.
- **High Throughput**. Shorter (and faster) Time Constant setting for high throughput spectral acquisition.

**Note** The High Resolution and High Throughput options are defined by the software and their settings cannot be changed.

• User Defined 1 (and User Defined 2). Two sets of customized conditions that have been previously saved. See below for details.

#### \* To set up customized conditions for X-ray spectral acquisition

1. Click the Experiment Setup 😧 button next to the Start Spectrum button on the Acquisitions Toolbar and set individual parameters as desired.

Microanalysis Feature Sizing	
Spectrum Point ID Spectral Imaging Linescan	WDS
💼 🔯 Get Image 🔻 🔒 🌍 🏔 Start Spectrum 💌 🖓	<b>\$</b> 7
Experiment Setup	~
Active Detector(s) Detector One Detector Two	
Termination Criteria	
Live Time Limit (s): 10    Max Peak Counts: 0	•
-Energy Range	
Low Energy Cutoff (eV): 100 - High Energy Cutoff (keV): Auto	
Time Constant : Auto	
User Defin	ned

2. Click User Defined > Save User Defined Settings 1 (or Save User Defined Settings 2).



The icon on the toolbar changes to User Defined 1 (or User Defined 2) to show which preset is selected.

Spectrum	Point ID	Spectral Imaging	Linescan	WDS
GE CA GE	et Image 🔽	🔒 🗐 🖈 Star S	pectrum 👻 🙆	

3. Click **Start Spectrum** to start the acquisition.

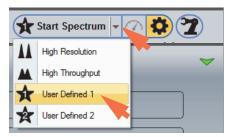
**Note** In the future, use the arrow next to the Start Spectrum button on the Acquisitions toolbar to select your custom conditions.

#### To update custom settings

1. Click the Experiment Setup 😧 button next to the Start Spectrum button on the Acquisitions Toolbar.



2. Click the Setting Presets button and select the custom parameter set you want to modify (User Defined 1 or User Defined 2).



- 3. Modify the individual parameters as desired.
- Choose User Defined > Save User Defined Settings 1 (or Save User Defined Settings 2).



#### **Spectral Acquisition Experiment Settings**

You can set acquisition parameters manually or use a <u>spectral acquisition preset</u>. This section provides general considerations for X-ray spectral acquisition and gives brief descriptions of all the available settings for acquiring X-ray spectra.

#### **Spectral Acquisition Considerations**

Consider these points before acquiring X-ray data for the first time.

• **Termination Criteria.** There are two criteria for terminating an acquisition: by time or by counts in the spectrum.

**Note** If values, other than 0 or blank, are entered in the Termination Criteria group, the first criterion reached will terminate the acquisition (zero equates to infinite).

• **Terminate By Time**. To terminate by time, enter a **Live Time Limit** value in seconds in the Termination Criteria group. The length of time should be sufficient to acquire a statistically valid spectrum. See below for more information.

- Terminate By Counts. There are two ways to terminate by counts in the spectrum:
  - Maximum peak counts. If any channel in a spectrum reaches the specified number of counts, the acquisition is terminated.
  - **Region of interest by element and line max counts**. If the **gross counts** in the element peak reaches the specified Max Counts value, the acquisition is terminated.

**Note** In general, there should be sufficient counts in a peak to achieve a counting error of less than 1%. For most applications, this can be achieved with a Live Time Limit of 60 seconds and a Max Peak Count of 10,000. (Make sure you use Time Constant and beam current settings that will achieve 30% dead time or less.)

- Energy Range. The Energy Range group includes two selections:
  - Low Energy cutoff (eV). Clips the energy acquisition range from 0 eV to the value entered in the edit box (i.e., it clips off the noise peak). The Auto selection adjusts the cutoff value depending on the estimated noise at a given pulse processor time constant. For most time constant settings, using a Low Energy Cutoff of 100 to 120 eV eliminates the noise peak at the low energy range.
  - **High Energy Cutoff (keV)**. Determines the number of channels allocated in memory (each channel represents 10 eV). For example, if a maximum range of 10 keV is selected, 1024 channels of memory are allocated. At this range, any peak greater than 10.240 keV is not saved or displayed.

Generally, the accelerating voltage in an SEM is limited to 30 KV. At this voltage the highest energy X-ray line that can be fluoresced and displayed is Mo K $\alpha$  line at about 17.4 keV. Thus, on an SEM, the maximum keV range is 20 keV; whereas, on a TEM at 200 KV, a 40 keV range would be appropriate.

The Auto selection adjusts the cutoff value to the accelerating voltage of the microscope. If the microscope accelerating voltage setting is incorrect, the high energy cutoff may also be incorrect.

**Note** Fixing the high energy cutoff at 20 keV for an SEM provides consistent spectra for comparison.

• **Time Constant**. The time constant is the time allowed for the pulse processor to estimate the energy of an X-ray. The longer the time allowed, the better the estimate of the energy and the higher the peak resolution. However, the longer the time constant, the higher the dead time for a given count rate. High resolution spectra (long time constant) require a lower count rate and a longer acquisition time to produce statistically good spectra (low counting errors).

For high resolution detectors, there are 10 fixed time constants plus the Auto selection. Non-extreme detectors have 8 or 9 fixed time constants plus the Auto choice. All time constants have estimated through-puts (counts per second). (To see the counts per second for the selected time constant, add Storage Count Rate to the status bar display by selected its check box in the <u>Microscope Parameters and Status Bar Selection box</u>.)

When selecting a time constant, consider the resolution of the spectrum and the length of time it takes to acquire the spectrum. Generally, select the longest time constant and acquire at a lower count rate to obtain a high resolution spectrum. For general samples, using a faster time constant (for example, Rate 3) increases throughput (i.e., shortens acquisition times) while still yielding a quality spectrum. For UltraDry detectors, a time constant of Rate 2 or Rate 3 yields quality spectra.

**Note** When Time Constant is set to Auto, the software adjusts the time constant to achieve between 30% and 50% dead time. At high beam currents, the time constant is shorter and spectral resolution degrades. At low beam currents, the time constant is longer and spectral resolution improves.

Do not use the Auto setting for spectral comparisons or quantification with standards because spectra acquired with different time constants will have different spectral resolutions.

### **Experiment Setup (Normal View)**

To access the spectral acquisition settings, click the Experiment Setup button in the right side of the acquisitions toolbar. For normal view, click to toggle the green arrow so it looks like this:

Microanalysis Feature Sizing Spectrum Point ID Spectral Imaging Linescan	Experiment Setup
Get Image   Get Image   Start Spectrum   Factive Detector(s)   Detector One   Detector Two	Normal View toggle setting
Live Time Limit (s): 10   Max Peak Counts: 0  Energy Range Low Energy Cutoff (eV): 100  High Energy Cutoff (keV): Auto Time Constant : Auto (projected max throughput) User Defined	

The following spectral acquisition settings are available in normal view. For more information about setting acquisition parameters for sample acquisition, see <u>X-ray Spectral Acquisition</u>. <u>Considerations</u>.

Table 5.	Spectrum M	de Experimen <sup>.</sup>	t Settings,	Normal View
----------	------------	---------------------------	-------------	-------------

Group	Setting	Description
Active Detector(s)	Detector One/ Detector Two	Available only when multiple Pathfinder-compatible EDS detectors are installed on the microscope. When multiple EDS detectors are installed, use the Detector One/Detector Two buttons to select which detector(s) you want to use for the analysis.
Termination Criteria <b>Note</b> : If all termination criteria have entered values, the first met termination criterion terminates the acquisition.	Live Time Limit(s)	<ul> <li>"Live time" refers to the period during acquisition when the system is waiting for new X-rays (whereas <u>dead time</u> is the period when the pulse processor is measuring the energy of an X-ray). Live Time Limit stops the acquisition when the system has acquired live data for the specified time period.</li> <li>A general rule for spectral acquisitions is 100 seconds of live time collection at 25–30% <u>dead time</u> with Time Constant set to Rate 2 or Rate 3. With SDD (UltraDry) detectors, the dead time will be lower.</li> </ul>
		<b>Note</b> : Set Live Time Limit to 0 for no live time limit (acquisition terminates based on other criteria).
	Max Peak Counts	Terminates the acquisition when the vertical full-scale of the centroid channel of <b>any</b> peak in the spectrum (including a noise peak) reaches this value.
		<b>Note</b> : Set Max Peak Counts to 0 for no count limit (acquisition terminates based on other criteria).
Energy Range	Low Energy Cutoff (eV)	X-ray counts below the specified Low Energy Cutoff are ignored during an EDS acquisition. Can be used to eliminate the "noise" peak in the low energy region. Typical options:
		• 100 eV. Clips the noise peak in the spectrum
		• Zero. Includes the noise peak
		• Auto. Adjusts the energy value according to the specified Time Constant. Each Time Constant setting has a unique noise level. In general, Auto includes about 3 channels of noise in the spectrum. Each time constant has a unique noise level.
	High Energy Cutoff (keV)	X-ray counts below this limit are included in an EDS acquisition. Typically, a High Energy Cutoff setting of 20 keV will include an X-ray line from all elements using an SEM. For TEM applications, 40 keV is appropriate. The Auto option changes the energy limit according to the microscope kV.

Group	Setting	Description
Time Constant		The amount of time the software measured the energy of an X-ray. Consider these options:
		• Rate 1 and Rate 2 produce statistically good, high-resolution spectra
		• Rate 3 and Rate 4 produce quality spectra for most analyses
		• Auto adjusts the time constant to keep the dead time between 30% and 50%. Depends on the beam current and accelerating voltage.
		<b>Note</b> : Do not use the Auto setting for spectral comparisons or quantification with standards.
		To see the time associated with each Rate setting, add Time Constant to the Pathfinder <u>status bar</u> .
User Defined	Save User Defined Settings 1 Save User Defined Settings 2	Allows you to save up to two instances of the experiment settings for spectral acquisitions (User Defined 1 and User Defined 2) that can be easily recalled later by clicking the <u>Setting Presets</u> button on the <u>Acquisitions toolbar</u> and choosing an option from the list box.

### Table 5. Spectrum Mode Experiment Settings, Normal View

### **Experiment Setup (Advanced View)**

To access the spectral acquisition settings, click the Experiment Setup button in the right side of the acquisitions toolbar. For advanced view, click to toggle the green arrow so it looks like this:

Microanalysis Feature Sizing	
Spectrum Point ID Spectral Imaging Linescan	Experiment Setup
Get Image - 2 2 Start Spectrum - 7 2 2	
	Advanced View
Experiment Setup	toggle setting
Active Detector(s)  Detector One Detector Two	
Termination Criteria	
Live Time Limit (s): 10 V Max Peak Counts: 0 V	
Energy Range	
Low Energy Cutoff (eV): 0 - High Energy Cutoff (keV): 20 -	
Time Constant : Rate 1 Shaping time 71400 cps (projected max throughput)	
Custom	
-Region of Interest	
Element: Line: VMax Counts:	
User Defined Low (eV): 0 High (eV): 0	
Stores /s: 3900	
Dead Time %: 25	

The following spectral acquisition settings are available in advanced view.

Table 6. Spectrum Mode Experiment Settings, Advanced View

Group	Setting	Description
Region of Interest	Element	To define termination criteria based on the integrated counts in the energy region for a particular element, enter the atomic symbol for the element and select an X-ray line (see below).
	Line	If you entered an element (see above), select the X-ray line for that element.

Group	Setting	Description
	Max Counts	Terminates the acquisition when the <b>integral</b> counts in the selected region of interest (element or user-defined) reaches this value.
	User Defined	To modify the default region of interest used for terminating an acquisition for a given element and line, set Element and Line, select the User Defined check box, and enter new high and low values for the region (see below).
	Low (eV)	When modifying the default region of interest for an element and line (see User Defined above), enter the low energy cutoff value in eV. X-ray counts below this value are ignored during an EDS acquisition.
	High (eV)	When modifying the default region of interest for an element and line (see User Defined above), enter the high energy cutoff value in eV. X-rays of energy up to this limit are included in the EDS acquisition.
Readouts	Detects (/s)	Shows the number of X-rays entering the EDS detector per second.
	Stores (/s)	Shows the number of X-ray counts that are measured and stored in the spectrum per second.
	Dead Time (%)	Shows the percentage of dead time for the current measurement settings. Dead time refers to the time the pulse processor is busy measuring X-ray events and cannot process another X-ray event. Rate 1 has the longest measurement time and produces the highest dead time for a given count rate. Rate 10 has the shortest measurement time and produces the lowest dead time for the same count rate.
		A dead time of 25-30% is recommended for quantitative analysis, and up to 50% for a spectral imaging (mapping) analysis.

#### Table 6. Spectrum Mode Experiment Settings, Advanced View

### **Set Up Automatic Spectral Processing**

The Spectrum Processing settings control how spectrum identification and quantification are performed. To perform spectrum processing automatically for each acquired spectrum, set the spectrum processing parameters before you acquire a spectrum and make sure Auto ID and/or Auto Quant are selected.

Spectrum processing settings apply to all the analytical modes and are not included in the presets, so you only need to set them once.

### ✤ To set up automatic spectral processing

1. Click the Spectrum Processing icon n the controls panel.

The following options are displayed.



2. To set up automatic element search and identification, choose <u>Ident Setup</u>, set the <u>identification parameters</u> as desired and make sure **Auto ID** is selected.



3. To set up a automatic quantitative analysis, choose **Quant Setup**, set the <u>quant</u> <u>parameters</u> as desired and make sure **Auto Quant** is selected.

Spectrum Processing		~
R Ident Setup	<b>Quant Setup</b>	
	Auto Quant	

Preliminary identification and quantitative results are displayed during the acquisition with a final update after the acquisition is completed.

### **Display Quant Results**

The software provides many options for displaying the results of a quantitative analysis. This section explains how to customize the quant results pane.

### **Select Quant Results Display Mode**

### **Grid Display Mode**

The default display mode for quant results is called grid mode. Each row in the grid represents a quantified element. The columns included in the grid are defined by the **Options** button in the quant results pane. Here is an example of Intensity, Net Counts, Weight% and Weight% Error displayed in grid mode:

Element	Line Type	Intensity	Ibite(1)20KV_origin Net Counts	Weight %	Weight % err
ск	K	33.96	6235	3.39	0.08
ок	К	998.95	183407	45.46	0.23
Na K	К	389.14	71447	8.37	0.05
AI K	К	662.04	121551	10.60	0.06
Si K	К	1833.28	336591	31.93	0.12
SiL (D)	L	0.00	0		
Ca K	К	8.70	1597	0.25	0.03
CaL(D)	L	0.00	0		
		3926.08	720828	100.00	0.56
Charts					Options

Figure 22. Quant Results Displayed in Grid Mode

Click **Charts** to display quant results in <u>chart format</u>.

### **Chart Display Mode**

To see quant results in chart format, click the **Charts** button in the quant results pane. Here is an example of a pie chart display:

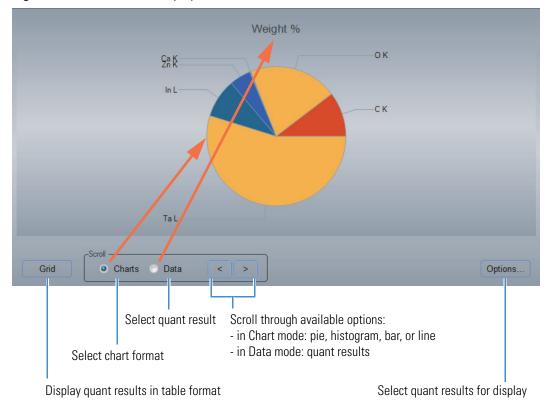


Figure 23. Quant Results Displayed as Pie Chart in Chart Mode

To change the chart format, select **Charts** and then use the right and left arrow buttons to scroll through the available chart formats:

- pie chart (each color represents a quantified element)
- histogram (each vertical bar represents a quantified element)
- bar chart (each horizontal bar represents a quantified element)
- line chart (each data point represents a quantified element)

To change the quant result displayed, select **Data** and then use the right and left arrow buttons to scroll through the available results.

Use the **Options** button to select the <u>quant results</u> for display.

Click Grid to display quant results in table format (default).

### **Select Quant Results for Display**

The software calculates a number of parameters during the quantification routine. Use the Options button on the results panel to select which results you want displayed after a quantitative analysis.

### How to Select Quant Results for Display

- \* To select the quantitative results for display
- 1. Click **Options** in the results pane to display the Selectable Columns box.

Selectable Columns		
<ul> <li>✓ Line Type</li> <li>✓ Intensity</li> <li>✓ Intensity err</li> <li>✓ Net Counts</li> <li>Net Counts err</li> <li>✓ Weight %</li> </ul>	Atom % Atom % err Source K-ratio K-ratio err Z Factor	K Factor Norm. Wt. % Norm. Wt. % err Chemical Formula Compound % Norm. Compoun
Veight % err ZAF Factor	A Factor	No. of Cations Standard Name
Error Measures (Sigma):	Decimal Place:	2 3
	Total Weight A	OK

2. Select the quantitative results you want displayed.

Weight%, and Weight% Error are typically included.

3. Select Line Type to display the X-ray lines used for Quantification.

Brief descriptions of all the available options are provided below.

4. Choose **OK** to save and close the Selectable Columns box.

The features you selected are added to the columns displayed in Grid mode and to the <u>scrollable Data</u> in <u>Chart mode</u>.

#### Note

- Right-click the title bar in the results pane to quickly select or deselect commonly used column headings for quant results.
- If an "(S)" appears next to an element quantification result, this indicates the result was calculated by stoichiometry. In the Selectable Columns box, select **Compund%** to display the compound result. Select **Chemical Formula** to display the formula used.

### **Available Quantitative Results for Display**

The table below briefly explains the available quantitative results.

Table 7. Available Quantitative Results for Display in Results Pane

Result Name	Description
Line Type	X-ray line type (K $\alpha$ or L $\alpha$ ) used for quantification
Intensity	Intensity value for each reported line (net counts divided by live time and then divided by beam current)
Intensity err	Error value for intensity
Net Counts	Number of X-rays received and processed by the detector with background characteristics removed
Net Count err	Error value for net counts
Weight %	The mass of the quantified element in the sample expressed as a percentage
Weight % err	Error value for measured weight%
ZAF Factor	Correction factor for "matrix effect" (includes atomic number, X-ray absorption and X-ray fluorescence corrections combined).
Atom%	The atomic concentration of the element in the sample expressed as a percentage
Atom% err	The error in the atomic percentage value based on the counting error of the net count measurement
Source	Source of the net count measurement (EDS, EDX STD, WDS, or FILE)
K-ratio	Measured K-ratio (intensity sample/intensity from pure element) for the X-ray line
K-ratio err	Error value for K-ratio (based upon the error in the net counts)
Z Factor	Correction factor for atomic number effect as calculated by the Proza or ZAF matrix correction routine
A Factor	Correction factor for X-ray absorption effect as calculated by the Proza or ZAF matrix correction routine
F Factor	Correction factor for X-ray fluorescence effect as calculated by the Proza or ZAF matrix correction routine

Result Name	Description
K Factor	Correction factor applied to element intensity ratios when calculating quantitative results from thin film samples, usually in STEM analysis. Uses ratios of intensities taken from all the elements in a sample. The samples are assumed to be thin enough to ignore X-ray absorption effects.
	K factors can be measured from standards or the software can calculate theoretical K factors for standardless analysis.
Norm. Wt.%	The element's weight concentration value normalized so that the total weight percentage from all elements equals the current Total Weight% setting.
Norm. Wt.% err	Error value for normalized weight%
Chemical Formula	Chemical formula of the compound containing the element used for stoichiometric calculations for Compound% results
Compound %	Percentage by mass of the quantified compound in the sample
Norm. Compound %	The element's compound percent value normalized so that the total from all compounds is 100 percent.
No. of Cations	Number of cations for the element based on the number of oxygen atoms defined in Quant Setup (see <u>Normalize cations</u> ) based on the following number of oxygen atoms)
Standard Name	Name of the standard, if any, used to quantify the element or compound
Error Measures (Sigma)	<ul> <li>Defines the confidence level of the calculated results. Available options:</li> <li>1 Sigma (~68% confidence)</li> <li>2 Sigma (~95% confidence)</li> <li>3 Sigma (~99% confidence)</li> </ul>
	For example, if the Wt% of an element is 10.0% with a 2 Sigma error of +/- 1.0%, we are 95% confident that the true Wt% of the element lies somewhere between 9.0 and 11.0 Wt% (i.e., 10% +/- 1%).
Decimal Places	Defines the number of decimal places reported for the calculated results. Available options: 1, 2 (default), or 3.
	<b>Note</b> : Because of the inherent error in EDS measurements, 1 decimal place is warranted. However, some protocols have specific decimal place reporting requirements.

Result Name	Description					
Show Matrix Only	Displays only the X-ray lines used for quantification in the results pane.					
	Selecting this op results grid or cl		ices the n	umber of	entries in	the quan
	When this option is cleared, all X-ray lines in the displayed spectrum (Spectrum pane) are included in the quant results pane. The following example shows both K $\alpha$ and L $\alpha$ lines for iron (Fe). Only the dominant (K $\alpha$ ) line has a quantification result; the L $\alpha$ line has dashes for Wt% and Wt% error.					
	Element					Ag Stand
	Þ	Line Type	Intensity	Net Counts	Weight %	Weight % err ±
	СК	K	3.64	3513	0.97	0.07
	Fe K	K	2.17	959	0.50	0.07
	FeL (D)	L	1.39	33		
	Ag L	L	26.88	3210	98.11	0.54
	Ag M (D)	М	4.65	5098		
Total Weight %	Defines the tota (default is 100%	U	value use	d to norm	alize the d	lata
	Leave this set to sample and wan 100%.				-	
	<b>Note</b> : For stand default total we		•		end using	the

### **Table 7.** Available Quantitative Results for Display in Results Pane

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## **Display Quantification Information About an Acquired Spectrum**

There are several ways to view quantification information about an acquired spectrum.

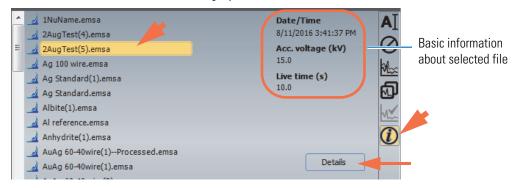
#### \* To view quantification information from the results pane in grid mode

Move the mouse pointer over the title bar:

Element				A	uAg wire(	1)		
Þ	Line Type	Intensity	Net Counts	Thu Aug ( Filter Fit ( Correction	04 18:14:30 201 Chi <sup>2</sup> value: 53.3 n Method: Pro ge: 20.0 kV	1 325 za (Phi-Rho-Z		)
Ag L	L	10.67	4532	68.91	0.32	80.19	0.37	
Ag M (D)	М	1.57	16244					
Au L	L	6.24	31769	31.09	1.21	19.82	0.77	
Au M (D)	М	7.86	3225					

#### \* To view basic spectrum information from the file list pane

- 1. Select the file in the file list pane.
- 2. Click the information button to display basic information about the selected file.



3. Click **Details** (see the previous image) to open the Details box for the selected file.

Here is an example:

NSS 20	1					
20	;			-		
C.C.						
65						
10						
1						
						-
	0.3	3				
:		None	0	Alumir	num	
s (um):	: 0.0	)3				
	•	None		Water	C	Oil
(um):	0					
		1		2	•	3
	ОК			(	Cano	el
			О 1			

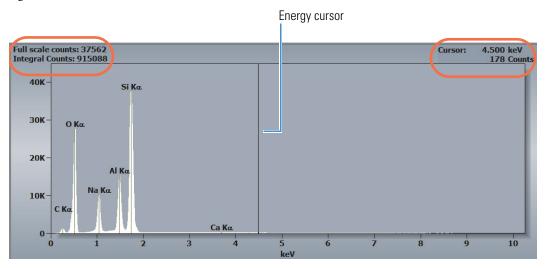
# **Customize the Spectrum Pane**

The spectrum pane shows the acquired or selected spectrum, with energy in keV along the X-axis and counts along the Y-axis.

If the <u>Energy Cursor</u> is displayed, it appears as a vertical line.

The vertical scale and integral counts are displayed in the upper left corner.

The cursor location and number of X-ray counts at that location are displayed in the upper right corner.



## **Change Appearance of Spectrum Pane**

You can change the appearance of the spectrum pane, such as the background color, to suit your preferences.

### ✤ To change the appearance of the spectrum pane

1. Click **Spectrum Settings** on the controls panel.

Microanalysis Fea	ture Sizing		
Spectrum Point ID	Spectral Imaging	Linescan WDS	
Get Image 🗸	A Start Sp	ectrum 🗸 🕜 🐼 🤇	7)
Spectrum Settings			
Spectrum Color:	Log Smooth Grid Histogra		
Peak	Lines Or	n 🗹 Smart Labels	
Labels: Sho	rt (Fe) 🔻 Font	Tahoma 11	
-KLMs			
Show KLMs	Include Shells:	K, L, & M 🔻	
Scale Peaks Escape Peaks	Transition Type:	1To 3 🔹	
Sum Peaks	Intensity above % of max	All 👻	
			-
Step Notes			pectrum Settings

## **Customize How Spectra are Displayed**

You can change the colors of displayed spectra and define the appearance of spectra displayed in the spectrum pane.

Microanalysis Fea	ture Sizing		
Spectrum Point ID	Spectral Imaging	Linescan	WDS
Get Image 🔻	Start Sp	ectrum 🔻 🕻	
Spectrum Settings			~
-Spectrum			
Spectrum Color:		V Cur	
Background Color:	Smooth	<u>O</u> ut	line
Cursor Color:	Grid	200	
Line Color:	Lines Or	n 🗸 Sma	art Labels
Sho	rt (Fe)	Tahoma 11	
Labels: Sho			
Show KLMs Scale Peaks	Include Shells:	K, L, & M ▼	
Escape Peaks	Transition Type:	1 To 3 🔹	
Sum Peaks	Intensity above % of max	All 👻	
444		-1-	
Stan		Spectrum \$	
Step Notes		Processing	Match Settings

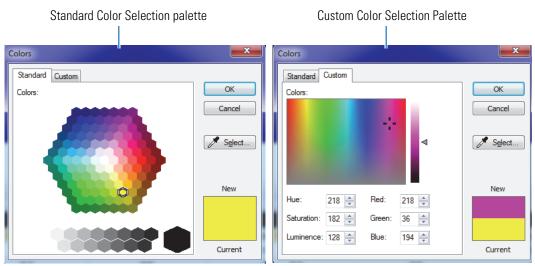
## **Adjust Colors of Spectra**

Use these features to change the colors of various features of the display pane, such as the spectrum, background, cursor, line and font.

Microana	ysis Featu	ire Sizing		
	Point ID	Spectral Imaging	Linescan	WDS
	et Image 🔻		pectrum 🔻 C	
Spectrum S	Settings			~
-Spectrum				
Spectrum	Color:	Log	✓ Cu	
		S <u>m</u> oot		rsor tline
Spectrum ( Backgroun	d Color:	S <u>m</u> oot	n <u>Q</u> u	
Spectrum	d Color:	S <u>m</u> oot	n <u>Q</u> u	

To change the color of a feature, such as the spectrum, click its color selection button, then select a color from the standard color palette or from the custom color palette.





## **Display Spectra in Log Scale**

Checking Log displays the spectrum in a logarithmic scale (useful for seeing small peaks).

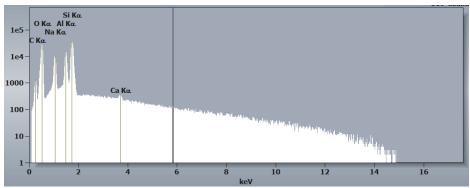
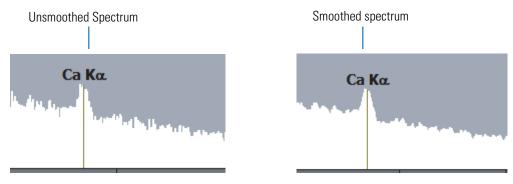


Figure 25. Spectrum displayed using log scale

## **Smooth Displayed Data**

Select Smooth to smooth the spectrum. In some instances, the irregularities may be enhanced. Smoothing is only done in the display. The raw data are preserved.

Figure 26. Spectrum displayed with and without smoothing



## **Display Grid in Spectrum Pane**

Select Grid to display a grid in the spectrum pane to the display (useful for comparing peak counts).

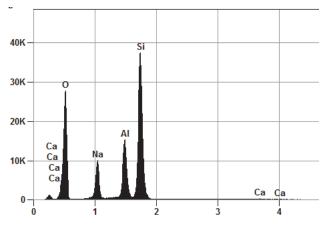
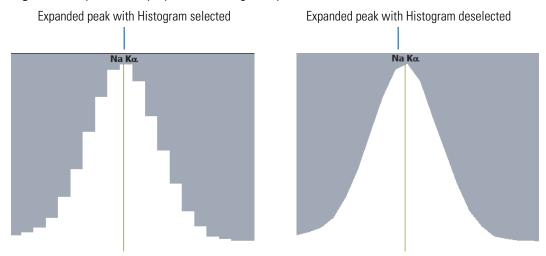


Figure 27. Spectrum displayed with background grid

### **Display Spectra as Histograms**

Select Histogram to view the spectral data as it was acquired (no processing). Deselecting Histogram applies a smoothing function to the spectral data.

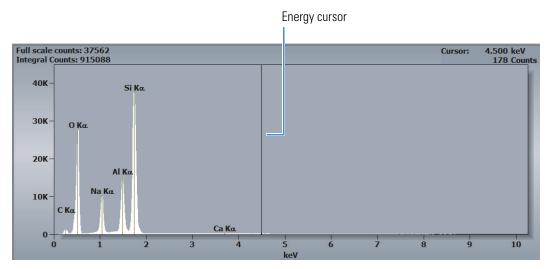
Figure 28. Spectrum displayed with Histogram option turned on and off



## **Display Energy Cursor in Spectrum Pane**

Select Cursor to display the energy cursor in the spectrum pane.

Figure 29. Spectrum pane with energy cursor displayed

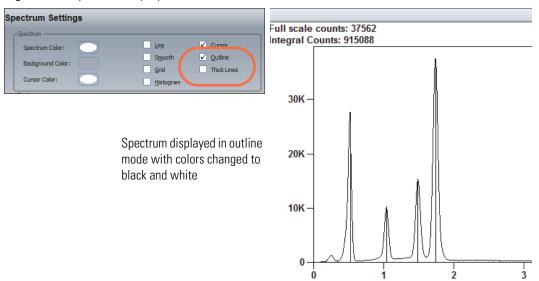


#### **Display Peaks as Outlines**

Select Outline to display peaks without the solid fill color. When Outline is selected, an additional Thick Lines option appears in Spectrum Settings. Use it to switch between thick and thin outlines.

**Note** With a simple black and white outline, you may also need to change the font color to see the peak labels.

**Figure 30.** Spectrum displayed in Outline mode (with colors set to black and white)



## **Customize How Peak Lines and Labels are Displayed**

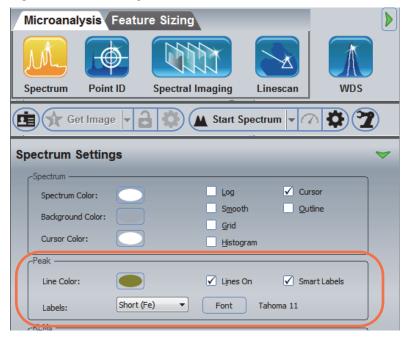
Peak lines are the vertical lines in the center of each labeled peak.

Peak label Peak line Full scale counts: 37562 Integral Counts: 915088 4.500 keV 178 Counts Cursor: 40K **Si K**α **30K** Ο Κα 20K-ΑΙ Κα Na Ka 10K-C Ko **Ca K**α 0-7 9 i 2 3 4 5 6 8 10 ò

Figure 31. Spectrum with peak lines displayed

You can display or hide peak lines and peak labels and change several properties that affect how peak lines and labels appear in displayed spectra. These settings are in the Peak group in Spectrum Settings.

Figure 32. Peak settings



### **Display Peak Lines**

Select Lines On to display peak lines in the spectrum pane (default is selected).

#### Note

- We recommend leaving peak lines displayed. They indicate the measured centroid location and provide information about the status of the <u>energy calibration</u>.
- The software does not display Peak Lines and KLM markers at the same time. If Show KLMs is selected in the Spectrum Settings KLMs group when you select Lines On, the peak lines will not be displayed.

### **Select Peak Line Color**

To change the peak line color, click the Line Color selection button, then select a color from the <u>standard color palette</u> or from the <u>custom color palette</u>.

### **Use Smart Labels**

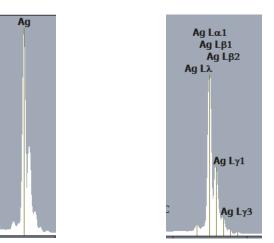
This selection controls the number of element X-ray lines that are labeled for a given element. When Smart Labels is selected, the software displays a limited number of element labels when space is limited to keep the spectrum pane from looking cluttered.

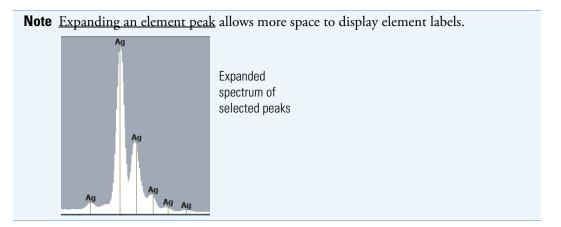
Deselect Smart Labels to display peak labels for all identified X-ray lines.

Figure 33. Spectrum with Smart Labels option turned on and off

Smart Labels selected

Smart Labels deselected

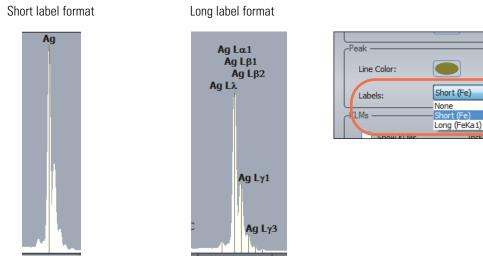




## Select Peak Label Format (Short/Long)

Element labels can be displayed in a short form (element symbol only) or long form (element symbol and its X-ray line).

Figure 34. Spectrum with short and long format peak labels displayed



The long label format is useful for viewing which lines can be fluoresced at a given SEM accelerating voltage.

Note Setting Labels to None turns off peak labeling.

### **Select Peak Label Font**

You can select the font, point size and color of displayed peak labels by clicking the Font button in the Peak group.

Figure 35. Select peak label font



The currently selected font and point size are displayed next to the Font button.

#### **Customize How KLM Lines are Displayed**

Characteristic KLM lines represent the energy of electron transitions between the K shell, L shell and M shell of an atom (or between their subshells) during X-ray analysis. You can display or hide KLM lines and change several properties that affect how KLM lines and labels appear in displayed spectra. Note that there can be several KLM lines in a single peak. Here is an example:

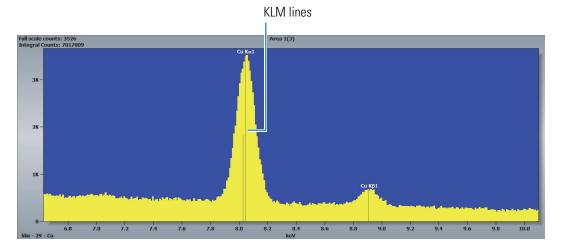


Figure 36. Spectrum with KLM lines displayed

Microanalysis Feature Sizing
Spectrum Point ID Spectral Imaging Linescan WDS
💼 🛧 Get Image 🗸 🔒 🔅 🛦 Start Spectrum 🗸 🏠 🤦
Spectrum Settings
Spectrum Color:          Background Color:          Grid          Cursor Color:          Histogram
Peak       Line Color:       ✓       Lines On       ✓       Short (Fe)       Font       Tahoma 11
KLMs       Show KLMs       Scale Peaks       Transition Type:       1To 3
Sum Peaks Intensity above % of max All -

These settings are in the KLMs group in Spectrum Settings.

Here are brief descriptions of the options available for defining the appearance of KLMs displayed in the spectrum pane.

Feature	Description
Show KLMs	Show or hide K, L and M markers in displayed spectra.
	<b>Note:</b> The software does not display KLM markers and peak lines at the same time. If <u>Lines On</u> is selected in the Spectrum Settings Peak group when you select Show KLMs, the KLM markers will not be displayed
Include Shells	Specifies which markers (K, L, or M), or which combinations of markers, to display.
Scale Peaks	Scales the height of the K markers such that the tallest K marker (usually K $\alpha$ 1) is the same height as the corresponding peak in the spectrum. It repeats this scaling for the L markers and M markers. Useful for finding hidden K, L, or M peaks.
Escape Peaks	Marks the locations of any <u>escape peaks</u> for all elements in the displayed spectrum using a short marker bar
Sum Peaks	Marks the locations of any <u>sum peaks</u> for all elements in the displayed spectrum using a short marker bar

Feature	Description
Transition Type	Specifies which transition types between energy states to include.
Intensity Above % of max	Lets you define a minimum intensity value for display. Useful for filtering out lines with lower intensity values.

# Adjust the Size of a Displayed Spectrum

Use these tools to expand or contract a displayed spectrum in order to see features of interest.

## **Using the Mouse**

**Table 8.** Using the mouse to size a displayed spectrum

То	Do this
Expand the spectrum	Click the spectrum in the low energy range and drag it right Or Use the scroll wheel on the mouse to expand the spectrum around the current cursor position
Contract the spectrum	To reduce the magnification of the spectrum and show a greater energy range, double-click the spectrum pane Or Use the scroll wheel on the mouse to contract the spectrum around the current cursor position
Scroll the spectrum	Drag the spectrum left or right to scroll horizontally; drag it up or down to scroll vertically
Zoom in on a peak	Hold down the Ctrl (or Shift) key on the keyboard while you drag the mouse cursor across the peak
Restore full spectrum view	To reset the X- and Y-axes to full spectrum view, double-click the spectrum pane

### **Using the Numeric Keypad**

Table 9. Using the keyboard to size a displayed spectrum (action centers around energy cursor)

То	Use this keypad key
Display the full spectrum	Home (7)
Scroll left	NumLock + Left Arrow (4)
Scroll right	NumLock + Right Arrow (6)
Expand	Page Up (9)
Contract	Page Down (3)
Full vertical scale	End (1)

То	Use this keypad key
Increase vertical scale	NumLock + Up Arrow (8)
Decrease vertical scale	NumLock + Down Arrow (2)

Table 9. Using the keyboard to size a displayed spectrum (action centers around energy cursor)

# **Manage Spectrum Files**

After you acquire a spectrum—either from an acquisition in Spectrum mode or from a Spectral Imaging mode extraction viewed from Spectrum mode—you can change the file name of the spectrum, and delete a spectrum file. Elements can be identified and labeled automatically or manually.

# **Rename a Spectrum File**

The current <u>Experiment Name</u> is automatically used as the base filename for each acquired spectrum. Subsequent spectra are given the same base name followed by the acquisition number.



To rename a spectrum file, select the file in the file list pane, click the **Rename** button on the <u>file management panel</u>, type a new filename and press Enter.

Figure 37. Rename a spectrum file



**Note** If you enter the name of an existing spectrum file when renaming a file, the filename will not be changed.

## **Delete a Spectrum File**

To delete a spectrum file, select the file in the file list pane and click the Delete button on the file management panel.

Figure 38. Delete a spectrum file

📩 🤟 1NuName.emsa	A
🔬 2AugTest(4).emsa	
E 🔬 2AugTest(5).emsa	$\bigcirc$
🔬 Ag 100 wire.emsa	kı
🔬 Ag Standard(1).emsa	
📝 Ag Standard.emsa	
🔬 Albite(1).emsa	
🔬 Al reference.emsa	

**NOTICE** Deleting a spectrum file permanently removes the data from the project and moves it to the computer's Recycle bin.

## **Open a Spectrum File**

Files opened in Spectrum mode are handled as standard spectrum files. You can open files acquired using Pathfinder software, or EMSA format files acquired using a third-party EDS system.

#### \* To open a spectrum file within a project

- 1. Open the project.
- 2. Click the Spectrum icon <u>Management</u> on the <u>analytical mode toolbar</u>.
- 3. Select the file in the file list pane.

^	🔟 1NuName.emsa
	🔬 2AugTest(4).emsa
Ξ	🔬 2AugTest(5).emsa
	Ag 100 wire.emsa
	🔬 Ag Standard(1).emsa
	🔬 Ag Standard.emsa
	🔬 Albite(1).emsa

The file is displayed in the spectrum pane.

#### \* To open (import) an EMSA file into the current project

- 1. Click the Spectrum icon <u>seetum</u> on the <u>analytical mode toolbar</u>.
- 2. From the Pathfinder Home screen, choose File > Open.
- 3. Navigate to the project folder that contains the file.
- 4. Select an EMSA (.emsa) file to import and choose **Open**.

The spectrum is copied to the current project's temporary folder and inherits the current project's settings.

5. To permanently save the file in the project, click the **Save** button **[]** on the main toolbar, or choose **File > Save**.

## Save a Spectrum

Spectra acquired in Spectrum mode are automatically stored in the current project's temporary folder with an EMSA (.emsa) filename extension. When you close a project, Pathfinder displays a list of files that have been acquired or modified in the current session. Deselect those files you do NOT want saved. Choose OK to save the changes, or choose Cancel to close the Save dialog box and return to the project without saving any changes.

#### \* To save a displayed spectrum without closing the project

Click the Save button **Main toolbar** to save the file in the currently open project.

# **Qualitative Analysis**

Qualitative analysis is the identification of elements that make up a specimen by recognizing the characteristic X-ray energy peaks associated with those elements in an X-ray spectrum. A qualitative analysis may be initiated automatically, immediately after an acquisition, or started manually at any time on any acquired spectrum. This section provides an overview of peak identification, guidelines to perform an accurate analysis, and general qualitative analysis procedures.

When qualitative analysis is done automatically, the software uses internal algorithms based on peak shape and location to identify the peaks in a spectrum. It is a good idea to manually inspect and refine the results in order to incorporate information about the sample and to resolve uncertainties due to overlapping peaks. The software also provides tools for identifying spectral peaks manually using KLM markers, which show the K, L, and M lines associated with each element. Both methods are explained in this section.

To perform an accurate qualitative analysis, acquire spectral data long enough to achieve sufficient counts for a good statistical analysis. A general rule is to acquire at least 5,000 counts (vertical full scale) at 25 to 30 percent dead time at the longest time constant. Be aware that some peak energies are shared by more than one element. That fact, and the inherent resolving power of the EDS detector, results in the overlap of energy peaks in the spectra.

## **Set Up Automatic Element Search and Identification**

#### \* To set up automatic element search and identification

1. Click the <u>Spectrum Processing</u> icon **u** on the controls panel.

The following options are displayed.



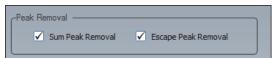
2. Click **Ident Setup** and then click the green arrow to show the Advanced view.

Spectrum Processing Quant Setup Auto ID Peak Removal Sum Peak Removal Sum Peak Removal Sum Peak Removal Comparison of the setup of the s	Advanced View toggle setting
Ident Sensitivity: 5 Overvoltage: 2.0	

3. Make sure Auto ID is selected.



4. In the <u>Peak Removal group</u>, select Sum Peak Removal and Escape Peak Removal.



5. Set <u>Overvoltage</u> to 1.5 and set the <u>Ident Sensitivity</u> bar between 5 and 10.

The sensitivity setting determines the minimum peak height for element identification. A lower sensitivity value increases the number of peaks that are identified, especially the smaller peaks. (A higher sensitivity value reduces the number of peaks that are labeled and leaves only the larger peaks.)

Ident Sensitivity:	5	-0
Overvoltage:	1.5	

When Auto ID is selected, preliminary identification results are displayed during the acquisition with a final update after the acquisition is completed. Sum peaks and escape peaks are removed after the acquisition is completed and before the final results are displayed.

**Note** When Auto ID is off, use the **Process** button on the controls panel to start automatic qualitative analysis using the displayed spectrum or the selected file.



The Identify button below the periodic table starts automatic qualitative AND quantitative analysis.



## **Element Search and Identification Settings**

Use **Ident Setup** on the Spectral Processing tab to define how element search and identification works for an acquired spectrum. You can set up the software to perform the analysis at run time or you can run it manually after an acquisition is completed.

The following element search and identification settings are available in advanced view.

To access these settings, click the Spectrum Processing icon **under Setup** on the controls panel, click **Ident Setup** and then click to toggle the green arrow so it looks like this:

Spectrum Processing	Advanced View toggle setting
Auto ID	
-Peak Removal	
Sum Peak Removal Escape Peak Removal	
Ident Sensitivity: 5	
Overvoltage: 2.0	

Group	Setting	Description
	Auto ID	Automatically performs element search and identification during a spectrum acquisition.
		When Auto ID is selected, preliminary identification results are displayed during the acquisition with a final update after the acquisition is completed. Sum peaks and escape peaks are removed after the acquisition is completed and before the final results are displayed.
Peak Removal	Sum Peak Removal	Automatically removes any sum peaks from the spectral data. A sum peak occurs when two or more X-ray events reach the detector at exactly the same time. The two events are processed as a single pulse with a magnitude equal to the sum of the individual pulses.
		When Sum Peak Removal is selected, the software determines which X-ray events generated sum peaks and transfers those counts into the appropriate component X-ray lines.
		Sum peak removal is an additional processing step that occurs immediately after the acquisition is completed. The software always saves the originally acquired spectrum. It is always possible to return to the original, uncorrected data. Sum peak removal affects both qualitative and quantitative analysis.
		<ul> <li>Notes:</li> <li>To toggle sum peak removal on and off for a displayed spectrum, use Spectrum (menu) &gt; Remove Sum Peaks.</li> <li>You can reduce the magnitude of sum peaks by reducing the count rate.</li> </ul>

## Table 10. Spectrum Mode Ident Setup, Advanced View

Table 10.	Spectrum	Mode I	dent Setup,	Advanced	View
-----------	----------	--------	-------------	----------	------

Group	Setting	Description
	Escape Peak Removal	Escape Peak Removal automatically eliminates the effects of escape peaks from the spectral data. An Escape peak is an artifact peak that occurs when an Si K X-ray photon is produced near the surface of the detector as a result of the interaction between an incoming X-ray photon and the detector. If the Si K X-ray photon escapes from the detector, the energy measured for the original incoming X-ray photon will be reduced by the energy of the Si X-ray photon. The artifact peak always has an energy that is 1.74 keV less than the original X-ray photon.
		When Escape Peak Removal is selected, the software calculates the probability of escape peaks in the spectrum, removes the escape peaks from the spectrum and adds the counts to the parent peaks.
		Escape peak removal is an additional processing step that occurs immediately after the acquisition is completed. The software always saves the originally acquired spectrum. It is always possible to return to the original, uncorrected data. Escape peak removal affects both qualitative and quantitative analysis.
		<ul> <li>Note:</li> <li>To toggle escape peak removal on and off for a displayed spectrum, use Spectrum (menu) &gt; Remove Escape Peaks.</li> <li>Escape events are relatively rare under most conditions. Usually the magnitude of an escape peak is, at most, a few percent of that of its parent peak.</li> </ul>

Group	Setting	Description
	Ident Sensitivity	Determines the minimum peak height for element identification. A lower sensitivity value increases the number of peaks that are identified, especially the smaller peaks. (A higher sensitivity value reduces the number of peaks that are labeled and leaves only the larger peaks.)
		Available settings range from 1 to 100. Typical settings are between 5 and 10.
	Overvoltage	In electron microscopy, overvoltage is the ratio of the accelerating voltage of the electron beam relative to the ionization energy for a given X-ray emission line. The ionization energy is always slightly higher than the corresponding X-ray emission line. Thus, a simple rule of thumb is that the overvoltage is the ratio of the accelerating voltage to the energy of the X-ray emission.
		Available settings are 1 to 2. To obtain adequate emission for most analyses, we recommend using an overvoltage setting of at least 1.5.
		<b>Note</b> : The peak identification software uses the Overvoltage setting to determine which peaks should be considered for labeling. If the accelerating voltage is 20 kV and the Overvoltage is 2, then peaks above 10 keV will not be considered for labeling. If the Overvoltage is reduced to 1.5, then the peaks above about 13 keV will not be considered for labeling. Keep in mind that the probability of generating an X-ray emission decreases rapidly in the low overvoltage range (below 1.5).

#### Table 10. Spectrum Mode Ident Setup, Advanced View

## **Identify Elements Using the Periodic Table**

When element identification is performed automatically, the software creates a list of possible elements identified from the spectrum and highlights them on the periodic table in the element setup pane. Depending on the display settings and the peak label settings, the spectral peaks associated with these elements may also be labeled in the spectrum pane. You can refine these results to search for or identify specific elements or to exclude other elements from the analysis.

If you know the sample composition and want to limit the analysis to specific elements, you can turn off automatic element identification and use the periodic table to specify elements for identification.

**Note** Your selections in the periodic table do not change when you select a different analytical mode. This lets you analyze a spectrum in one mode, then switch to another mode and use the same analysis results.

#### **\*** To identify selected elements using the periodic table

- 1. In the element setup pane, right-click the symbol for the element you want to identify.
- 2. In the shortcut menu, choose Identified (or Always Identified).

H Li Na	Be Mg	Aluminium Al 13 Ka 1.49 keV Always Identified										C		0	F	He Ne	
К	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	G		Identif	ied		
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	I	_	Inactiv	/e		
Cs	Ba		Hf	Та	W	Re	Os	Ir	Pt	Au	Hg	T	_	Exclud	ded		
Fr	Ra	A											~	Always Identified			
		La Ce Pr Nd Pm Sm Eu Gd Tb Dy H								H		Quant Lines			•		
		Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	E		Group			+
x		Possił	ble		<	Exc	duded		x		Select	ted		Advar	nced		
X Identified X Always X Near																	
lo	dentif	iy 🛛					P	rev/N	lext	-					CI	ear	-

The symbol for that element changes to green (identified) or pink (always identified).

3. If there are found elements you want to exclude, right-click each element symbol and choose **Excluded** (their symbols change to dark grey).

#### Note

• You can also left-click an element to scroll through the available settings (Identified, Inactive, Excluded, or Always Identified). The color of the element indicates its current setting. The legend shows the color assignments.



If the legend is not displayed in the software, choose **View** (menu) > **Preferences** and, in the Periodic Table Options, select **Show Legend**.

• Hydrogen (H) and Helium (He) do not generate X-rays that are detectable using EDS and their element names are unavailable in the software. Lithium (Li) and several groups of elements including the noble gases, the lanthanides and the actinides, are set to Excluded by default. However, you can reset Li or individual elements in these groups if they are present in the samples.

To toggle all the elements in a group between Excluded and Inactive, right-click any element and, from the shortcut menu, choose **Group** and then select the group name.

4. Click the **Process** button to start automatic qualitative analysis using the selected elements.

The button is located at the bottom of the Ident Setup tab in the Spectrum Processing group on the controls panel.



**Note** The Identify button below the periodic table starts automatic qualitative AND quantitative analysis.



## **Exclude Elements from Analysis**

#### \* To exclude an element from any analysis

- 1. In the element setup pane, right-click the element you want to exclude.
- 2. In the shortcut menu, choose Excluded.

Н					A	lumi		n				_				He	
Li	Be	Ka 1.49 keV						B				= Ne					
Na	Mg							A									
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Со	Ni	Cu	Zn	G		Identified	1		
Rb	Sr	Y	Zr	Nb	Мо	Тс	Ru	Rh	Pd	Ag	Cd	I		Inactive			
Cs	Ba		Hf	Та	W	Re	Os	Ir	Pt	Au	Hg	Т		Excluded	ł		
Fr	Ra												~	Always Identifie			
		La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dv	Н		Quant Li	nes	I	·
			Th	Pa	U			Am			Cf	E		Group		,	,
x		Possib	ole	)	<	Exc	duded		x		Select	but		Advance	ed		
x	X Identified X Always Near				_												

The symbol for that element changes to dark grey.

## **Clear Identification Results**

Use the Clear button at the bottom of the element setup pane to reset the element selections in the periodic table.

La Ce Pr Na Pm Sm Eu Ga ID Dy Ho Er In	YD LU	<u>C</u> lear All
AC Th Pa U Np Pu Am Cm Bk Cf Es Fm Md	No Lr	Reset to Default
X         Possible         X         Excluded         X         Selected         X           X         Identified         X         Always         X         Near	WDS	Reset ROIs
		Clear All WDS
Identify Prev/Next 💌 🕨	Clear 🔻	

There are several options described below:

Table 11.         Element Setup Pane, available Clear option
--

Setting	Description
Clear All	If an element is set to Identified (green) or Always Identified (pink), Clear All resets it to Inactive (light grey). If an element is set to Excluded, it will be unchanged.
Reset to Default	Reset all elements to their default settings. Elements that are excluded by default, for example Li, the noble gases, the lanthanides and the actinides, are set to Excluded (dark grey). Everything else is set to Inactive (light grey).
Reset ROIs (Regions Of Interest)	Reset the Mapping Energy Region Of Interest (ROI) for all elements to their default values. These ranges can be set manually by right-clicking an element, choosing <b>Advanced</b> and entering new values.
Clear All WDS	Element that are selected for WDS have a purple triangle in the upper left corner of the element symbol in the periodic table). Clear All WDS resets these elements to Inactive (light grey).

## **Identify Elements in a Spectrum**

The automatic peak identification function labels each peak that the software identifies. When KLM markers are not displayed, the software can display lines, called "peak markers," that indicate the locations of the found peaks. Use these markers as a starting point for manual peak identification.

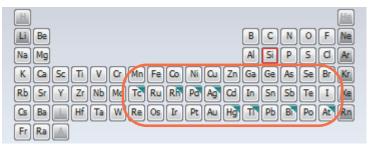
The software provides several tools for refining element identification including peak markers, KLM markers and peak labels.

### Use Energy Cursor to Identify Elements in a Spectrum

#### \* To identify elements using the energy cursor

To view a list of possible elements associated with an identified spectral peak, place the energy cursor over the centroid of the peak of interest. Elements with a K, L or M X-ray line near the energy at the cursor position are highlighted with a dark colored triangle in the upper right corner of the element name in the periodic table.

**Figure 39.** Elements near energy cursor are highlighted with dark triangle in upper right



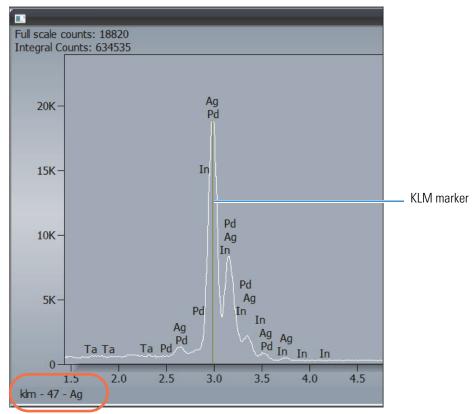
You can change any of them to Identified if they match the element you are seeking. Some knowledge of the sample and other element peaks is useful in determining which elements should be identified manually.

#### Use KLM Markers to Identify Elements in a Spectrum

#### ✤ To display KLM markers

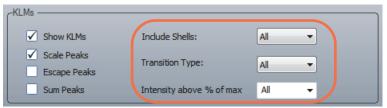
You can use KLM Markers for a selected element to aid in positively identifying an element. First, make sure KLM markers are displayed in the software (in <u>Spectrum Settings</u>, select **Show KLMs**).

Click an element in the periodic table to display the X-ray lines associated with that element. The symbol for the element that has its KLM markers displayed appears in the lower left corner of the spectrum pane, along with the element's Z number. Here is an example:



#### ✤ To adjust the number of KLM lines displayed

1. Use the features in the KLMs group of Spectrum Settings to adjust the number of KLM lines displayed for all identified peaks in a spectrum.



- 2. Set **Include Shells** to the shells you want displayed (K, L, M or All). We recommend setting Include Shells to All.
- 3. Set **Transition Type** to the number of transitions you want to include (1 to 3, 1 to 2, 1, or All). We recommend setting Transition Type to All.
- 4. To adjust the number of lines that appear at various levels of X-ray intensity, select the intensity level to display. We recommend setting **Intensity Above % Of Max** to All.

These selections will show all KLM Lines, including minor lines such as La and Ma.

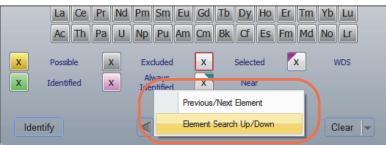
#### \* To scroll through the KLM markers in a displayed spectrum

Use the Previous and Next arrow buttons at the bottom of the element setup pane (or click the spectrum pane and use the left and right arrow keys on the keyboard) to scroll through element KLM markers by atomic number, starting with the selected element.



#### **\*** To search for elements using their KLM lines

You can position the energy cursor at an energy location on the displayed spectrum and search for elements with KLM lines that are higher or lower than that energy location by using the Element Search Up and Element Search Down buttons in the element setup pane. (You can also click the spectrum pane and use the up and down arrow keys on the keyboard.)



The Element Search Up/Down buttons use the energy reading at the energy cursor to search the software's internal database of element lines and displays candidate KLM markers for each element found.

A high pitched beep means Pathfinder found an element X-ray line near the current cursor location. The found element is outlined in red in the periodic table and its KLM lines are shown on the spectrum. The elements with KLM lines near the cursor have cyan triangles in the element. A low pitched tone means you have reached the end of the possible elements for that cursor location.

## **Label Identified Peaks Manually**

After you have identified a peak, you may want to label it on the spectrum for reference and reports. To label a peak, right-click its associated element in the periodic table and choose **Identified** (green) or **Always Identified** (pink).

### **Change How Peak Labels are Displayed**

The features in the Peak group in Spectrum Settings specify how peak labels appear in displayed spectra.

Figure 40. Peak settings

Microanal	ysis Featu	ure Sizin	g			
Spectrum	Point ID	Spectra	I Imaging	Linescan	WDS	
E A Ge	t Image 🔻	30	Start Sp	ectrum 🔻 C	707	)
Spectrum S	Settings	_	_	_	_	~
-Spectrum						
Spectrum (	Color:		Log	V Cu		
Background	d Color:		S <u>m</u> ooth	<u> </u>	utline	
Cursor Cole	or:	Ĵ	<u>G</u> rid <u>H</u> istogra	am		
-Peak		_			]	
Line Color:			🖌 Lines O	n 🗹 Sm	nart Labels	
Labels:	Short	(Fe) 🔻	Font	Tahoma 11		J
-RLMS						

Set **Labels** to Short to display element symbols only, or Long to display element symbols and lines.

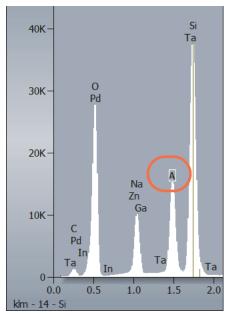
**Note** Setting **Labels** to None turns off peak labeling. If you try to label an individual peak, the label will not appear on the spectrum until Labels is reset to Short or Long.

You can also change the font and color of peak labels using features in the Peak group in Spectrum Settings.

#### **Delete Peak Labels**

To delete all the peak labels for one element, right-click the element in the periodic table and select **Inactive** from the shortcut menu.

To delete a single peak label, click the peak label so it is outlined and then press the Delete key on the keyboard. The element symbol for that peak is removed.





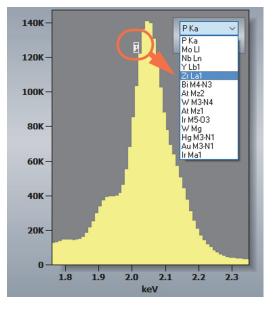
#### **Add Peak Labels**

You can label specific elements for reference and reports.

To label all the peaks for a particular element, right-click the element in the periodic table and choose **Include**. If you just want to label an individual peak, manually delete the unwanted peak labels.

### **Select the Element Assigned to a Peak**

To view possible overlaps for a found element, double-click the element label. A selection box lists the elements that emit in that energy range. To change the element assigned to the selected peak, select an element in the list and choose **OK**. The peak is relabeled with the new name.





## **Use Spectra Check to Verify Peak Identification**

Spectra Check is a tool for confirming peak assignments in a displayed spectrum. Use it after you have finished reviewing and adjusting peak assignments in a spectrum.

When the Spectra Check took is selected, it generates three synthetic spectra:

- **Synthetic Spectrum**. The software uses reference spectra from its internal database to calculate a synthetic spectrum based on the elements selected in the periodic table (i.e., set to Identified or Always Identified).
- Background. The calculated background for the displayed spectrum.
- **Residual**. The software subtracts reference spectra for the elements selected in the periodic table from the original spectrum to create a residual spectrum, which shows the peaks that are not accounted for in the original spectrum.

**Note** A theoretical model of the background shape and spectrum can be created if the following sample characteristics are known:

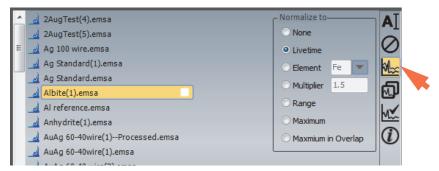
- approximate mass of the sample
- energy of the excitation electrons
- geometry of the system
- characteristics of the detector

Synthetic models provide visual evidence of the accuracy of a qualitative analysis and indicate whether a given sample is suitable for quantitative measurements.

#### To activate Spectra Check and display the synthetic spectrum

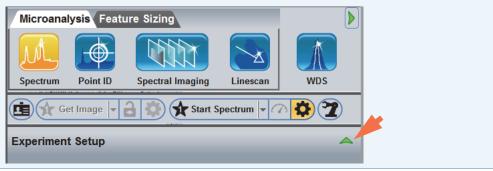
1. Click the **Compare** button on the file management panel (along the right edge of the file list pane).

You can skip the normalization setting.



The button stays highlighted to show that it is active.

**Note** If the file list pane is not visible, click the green arrow to collapse Advanced View.

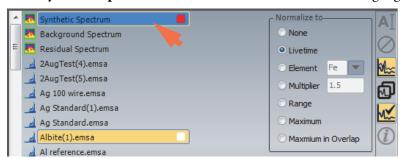


2. Click the Spectra Check 🚾 button on the file management panel.

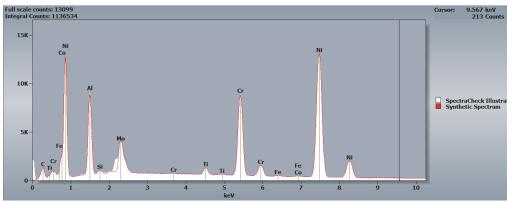
The button stays highlighted, to show that it is active, and the software adds 3 spectral files to the top of the file list (synthetic, background and residual).

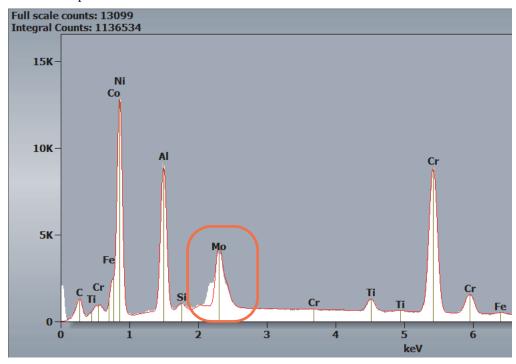
Albite(1).emsa 🛛 🖉 Maxmium in Overlap		Synthetic Spectrum Background Spectrum Residual Spectrum 2AugTest(4).emsa 2AugTest(5).emsa Ag 100 wire.emsa Ag Standard(1).emsa Ag Standard.emsa Albite(1).emsa	Normalize to None Livetime Element Fe Multiplier 1.5 Range Maximum Maxmum in Querlap	
---------------------------------------	--	---	---	--

3. Click Synthetic Spectrum in the file list so its selection box is highlighted (red).



The synthetic (red) spectrum is displayed in the spectrum pane, overlaid on the original (white) spectrum. The synthetic spectrum is based on the selected elements in the periodic table and is normalized to the displayed spectrum using the <u>Maximum</u> normalization algorithm.





In this example, there is an element near Mo that is not identified.

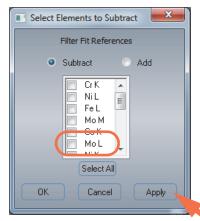
Hover the mouse cursor over the title bar in the results pane to view the "goodness of fit" value  $(\chi^2)$  for the two spectra.

Element	Albite(1)20KV_original							
Þ	Line Type	Intensity	Net Counts	Weight %	Weight % err			
СК	К	33.96	6235	3 39	0.08			
ОК	К	THE FULLO IT	Fue Jul 26 17:02:19 2016 Filter Fit Chi <sup>2</sup> value: 16.793 Correction Method: Proze (Phi <sup>-</sup> Rho-Z) Acc.Voltage: 20.0 kV Take Off Angle: 34.8°					
Na K	К							
AI K	К							
Si K	К	1033.20	220291.	31.93	0.12			

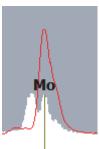
- 4. Use one of the following methods to identify the missed peak:
  - Reference subtraction
  - Nearest neighbor

## **\*** Using reference subtraction to identify a missed peak

- 1. From the Home screen, choose **Spectrum** (menu) > **Reference Subtract/Add** to display the Elements to Subtract selection window.
- 2. In the Select Elements To Subtract box, select the **Mo** (L) line and click **Apply**.

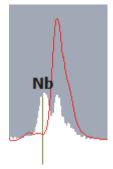


The software subtracts a reference spectrum for the Mo (L) line from the original spectrum. The resulting (white) spectrum has the Mo (L) line removed.



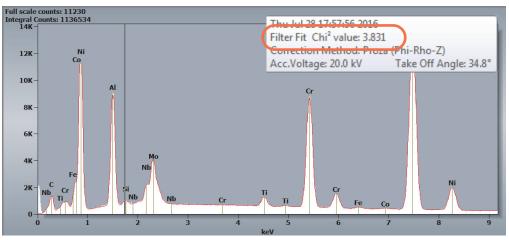
3. Click the **Identify** button in the element setup pane.

The element Nb is now identified.



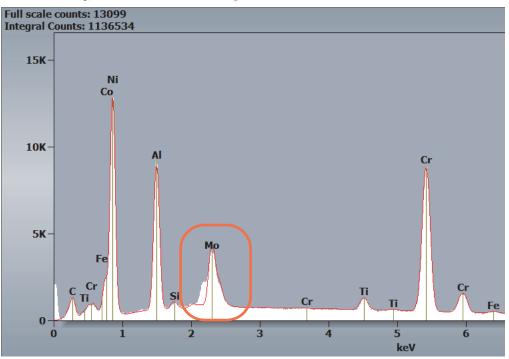
4. Right-click the element Nb in the periodic table and set it to **Identified**.

The synthetic spectrum now outlines the missed peak and the  $(\chi^2)$  value has decreased from 16.8 to 3.8.



## **Solution** Using the "nearest neighbor" method to identify a missed peak

1. Move the energy courser over the missed peak.



Elements with a K, L or M X-ray line near the energy at the cursor position are highlighted with a dark colored triangle in the upper right corner of the element name in the periodic table.

H																	He
Li	Be											В	С	N	0	F	Ne
Na	Mg											A			S	Cl	Ar
к	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Мо	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Те	Ι	Xe
Cs	Ba		Hf	Та	W	Re	Os	Ir	Pt	Au	Hg	TI	Pb	Bi	Po	At	Rn
Fr	Ra	A															

2. Add the elements to the spectrum one at a time and view the fit to the overlaid synthetic spectrum.

Start with the element with the lowest Z number (Zr in this example). To add an element to the spectrum, right-click the element name in the periodic table and choose **Identified**. To remove an element, right-click its name and choose **Excluded**. When the peaks in the synthetic spectrum match the peaks in the original spectrum, you have correctly identified the missed element.

## **Compare Spectra**

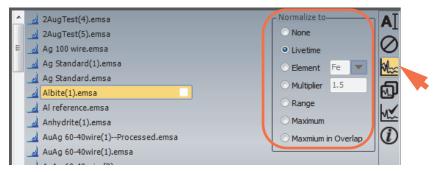
Use the Compare tool to overlay two or more spectra in the spectrum pane for comparison.

#### To select multiple spectra for display

1. Display the spectrum you want to compare.

We will call this spectrum the "original" spectrum.

2. Click the **Compare** button on the file management panel (along the right edge of the file list pane)



The button stays highlighted to show that it is active.

**Note** If the file list pane is not visible, click the green arrow to collapse Advanced View.

Microanal	ysis Featu	ıre Sizing				
M	Ø					
Spectrum	Point ID	Spectral Imaging	Linescan	WDS		
💼 🛧 Get Image 🔻 🔒 🎲 🛠 Start Spectrum 🗸 🏠 🏠						
Experiment	t Setup					

3. Select a normalization setting for the comparison spectra.

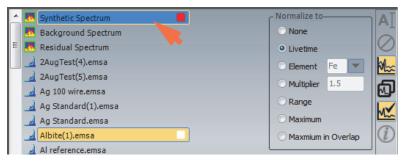
The normalization setting has no effect on the original spectrum. The available options are described below.

Setting	Description
None	No normalization factor is applied
Livetime	Normalizes the overlapped spectra based on the live time of the overlapped spectrum compared to the live time of the original spectrum. This simulates the same acquisition times for both spectra.
Element	Adjusts the overlapped spectra to the height of the selected element peak or line in the original spectrum. The selected peak or line is based on accelerating voltage and the energy range of the spectrum.
Multiplier	Enter a value that will be used to scale all overlapped spectra
Range	Scales the overlapped spectra to the original spectrum based on a selected energy range. When Normalize To is set to Range, two dashed vertical lines are added to the displayed spectra. (If you don't see the vertical lines, double-click to expand the spectrum pane.) Drag the lines left and right to define the left and right boundary of your selected range.
	<b>Note</b> : Defining a range in a background region (between element peaks) sometimes gives the best results.

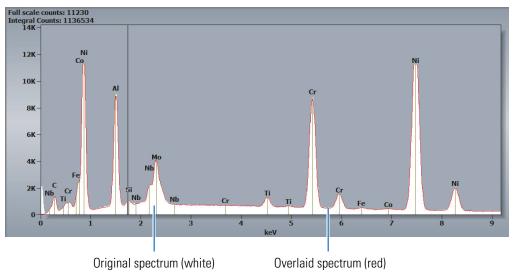
Setting	Description
Maximum	Scales the overlapped spectra to the original spectrum based on their maximum peak values in the displayed region
Maximum in Overlap	If the overlapped spectrum intersects the original spectrum, the software scales the maximum peak value in the overlapped spectrum to match the maximum peak value in the original spectrum.
	<b>Note</b> : This setting is intended for WDS data where the spectra may not cover the same energy range but still overlap in some energies.

4. Select one or more spectra to overlay by clicking their names in the file list pane so their selection boxes are highlighted (red).

In this example, the synthetic spectrum generated by a Spectra Check is selected for overlay.



Each overlaid spectrum selected in the file list pane is displayed in the spectrum pane in a unique color in outline mode.



# **Quantitative Analysis**

Quantitative analysis determines the chemical composition of a sample by calculating the relative concentrations of chemical elements in the sample from the relative X-ray counts and applying matrix corrections. The matrix corrections adjust for atomic number, absorption, and secondary fluorescence effects. The general steps are outlined below:

- 1. The background characteristics are removed from the sample spectrum. When using Filter Fit, the background is removed using a "top hat filter." When using Gaussian peak fitting, the peaks and background are fit using a model.
- 2. The software determines the net X-ray counts in each peak.
- 3. An iterative process is used to calculate a matrix correction by simultaneous equations with the element concentrations as unknowns.
- 4. Element concentrations are calculated by multiplying a measured K-ratio by the matrix correction.
- 5. The data are normalized to 100% and the results are reported.

**Note** Before you quantify a new sample, run a qualitative analysis and verify that the list of elements present in the sample is accurate. See <u>Qualitative Analysis</u> and <u>Using</u> Spectra Check to Verify Peak Identification for more information.

This section provides guidelines for setting up and performing an accurate analysis.

## **Specify the Element Lines Used for Quant**

The software assigns the element lines used for quantitative analysis based on the element and the accelerating voltage. This is typically the dominant line for each element. However, if the accelerating voltage is not high enough to excite the line properly, the software may select a line that is lower in energy instead.

You can specify the element lines used for quantitative measurements by right-clicking the element symbol in the periodic table. From the shortcut menu, choose **Quant Lines** and select an available line.

K Ca Sc Ti V Cr Mn Fe Co Ni Cu	
Rb Sr Y Zr Nb Mo Tc Ru Rh Pd A	Cd To So Sh Te I Xe
Cs Ba 📙 Hf Ta W Re Os Ir P 🖌	Identified Po At Rn
Fr Ra	Inactive
	Excluded
La Ce Pr Nd Pm Sm Eu G	Always Identified
Ac Th Pa U Np Pu Am Cr	Quant Lines Absent
X Possible X xcluder	Group V L Lines
X Identified X Iontifie	Advanced M Lines
Identify Prev/Next	Clear 💌

Figure 43. Select Element Lines Used for Quant

#### Note

- Some elements may have only one line available for quantitative measurements.
- The Absent setting excludes the selected element from the quantitative results. For more information, see Exclude an Element from Quant Results.

## **Exclude an Element from Quant Results**

You can include an identified element, such as a coating, in the matrix correction calculation but not report quantitative results for that element by right-clicking the element symbol in the periodic table. From the shortcut menu, choose **Quant Lines** and select Absent. When Quant Lines is set to Absent, a series of dashes (-----) appear in any displayed quantitative results cell for that element.

**Note** This change is applied to all future analyses in the current project until the element line for that element is changed.

## Set Up Automatic Quantitative Analysis

Use Spectrum Processing on the controls panel to set up a quantitative analysis. Auto Quant with no standards are sufficient for most analyses. For information about the standards manager or running quant with standards, see Quant with Standards.

## \* To set up automatic quantitative analysis

1. Click the <u>Spectrum Processing</u> icon **becken** on the controls panel.

The following options are displayed.

Spectrum Processing	-	Normal/Advanced View toggle	
R Ident Setup	Quant Setup		view toggio

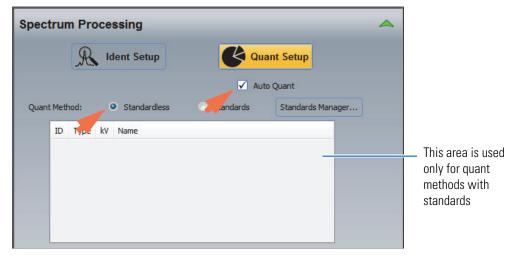
2. Click Ident Setup and make sure Overvoltage is set to 1.5 or higher.

Overvoltage is in the advanced view. Typically, the accelerating voltage should be 1.5 to 2 times greater than the highest X-ray energy line. The software uses this value to select a major X-ray line for quantitative measurements.

3. Click **Quant Setup** and then click the green arrow to show the Advanced view.

Spectrum Processing			<b>A</b>	Advanced View toggle setting
R Ident Setup	C Qu	ant Setup		
Quant Method: <ul> <li>Standardless</li> </ul>	✓ Aut	to Quant Standards Manager		
ID Type kV Name				

4. Make sure **Auto Quant** is selected and set **Quant Method** to Standardless.



5. Set <u>Peak Fit Method</u> to the algorithm you want to use to determine the peaks and remove the background.



These options are available:

• **Filter Fit** (default). Applies a digital "top hat" filter to remove the background and determine the peaks.

- **Gaussian**. Uses Gaussian shapes to model the peaks. Uses a theoretical model to fit the background.
- 6. In the <u>Correction Method</u> group, select a matrix correction that suits the samples to be analyzed.

Correction Method	
O None	🔘 Proza (Phi-Rho-Z)
🔿 ZAF	Oliff-Lorimer (MBTS)
	Absorbance

These options are available:

- None. Only net counts are reported.
- **ZAF**. Used primarily in metallurgical SEM applications.
- **Proza**. Used in SEM applications, especially for light elements. This is the default setting and the recommended setting for most sample analyses.
- **Cliff-Lorimer (MBTS)**. Used in TEM/STEM applications only.

**Note** If you want to specify the ratio element for the Cliff-Lorimer correction, see Select Ratio Element for Cliff-Lorimer Correction.

When Auto Quant is selected, preliminary quantitative results are displayed during the acquisition with a final update after the acquisition is completed. Sum peaks and escape peaks are removed after the acquisition is completed and before the final results are displayed.

**Note** When Auto Quant is off, use the **Process** button on the controls panel to manually quantify the displayed spectrum or the selected file.



## **Quantitative Analysis Settings**

Use **Quant Setup** on the Spectral Processing tab to define quantitative analysis for an acquired spectrum. You can set up the software to perform the analysis at run time or do it manually after an acquisition is completed.

The following quantitative analysis settings are available in advanced view.

To access these settings, click the Spectrum Processing icon **processing** on the controls panel, click **Quant Setup** and then click to toggle the green arrow so it looks like this:

Spect	trum Processing		_	Advanced Vie toggle setting
	R Ident Setup		t Setup	
		🖌 Auto C	Quant	
Quant	Method: O Standardless	Standards	Standards Manager	)
	ID Type kV Name			
	-Peak Fit Method			
	Filter Fit	🔘 Gaussian		
	Correction Method			
	🔘 None	🔘 Proza (Phi-Rho	o-Z)	
	🔘 ZAF	Cliff-Lorimer (N	MBTS)	
		Absorbance		
	✓ Calculate a	all elements as compounds		
	Normalize cation	ns based on the		
	following numbe	er of oxygen atoms: 0		

Group	Setting	Description
Advanced View	Quant Method	Defines the type of quantitative method that will be used for quantitative measurements. Available options:
		• <b>Standardless</b> . A quantitative method without standards. This is the typical setting for most X-ray analyses.
		• <b>Standards</b> . A quantitative method that uses one or more standards for calculating sample results. Use the <u>Standards</u> <u>Manager</u> to select a standards database and to define standards for this analysis.
	<u>Standards Manager</u>	Used to set up a database of standards for any analysis and to define standards for the current analysis.
	Peak Fit Method	Defines the algorithm use to determine the peaks used for quantitative measurements and also to remove the background. Available options:
		• <b>Filter fit</b> . Applies a digital "top hat" filter to remove the background and determine the peaks.
		• <b>Gaussian Fit</b> . Uses Gaussian shapes to model the peaks. Uses a theoretical model to fit the background.
		If the quant method includes standards, both peak fit algorithms will use the selected standards.

## Table 12. Spectrum Mode Quant Setup, Advanced View

Table 12.	Spectrum	Mode	Quant	Setun	Advanced	View
10010 12.	opeenum	IVIOUC	Quant	ootup,	Auvunccu	1010

Group	Setting	Description
	Correction Method	Defines a matrix correction method for the analysis. These options are available:
		• <b>None</b> . No matrix correction is applied. The quantification results include only the intensity and net counts. Results such as weight percent are not reported.
		• ZAF. Reports net counts and weight percentages. Uses classical formulas to adjust for average atomic number (Z) (Duncumb and Reed), absorption (A) (Heinrich), and fluorescence (F) factors. These factors influence spectrum differences between elements in pure form and elements in composition. Used in metallurgical SEM applications.
		• <b>Proza</b> (Phi-Rho-Z). Reports net counts and weight percentages. Similar to ZAF but uses the Phi-Rho-Z technique to calculate the atomic number (Z) and absorption (A) corrections. Used in SEM applications, especially for light elements in a heavy matrix.
		Proza is the default setting and the recommended setting for most sample analyses.
		• <b>Cliff-Lorimer</b> (MBTS). Reports net counts and weight percentages. Provides metallurgical and biological thin-section (MBTS) corrections based on relative elemental K factors (Cliff-Lorimer factors). Assumes samples are infinitely thin (i.e., no absorption or fluorescence). Used for TEM and STEM applications only.
		<b>Absorbance</b> . Select this option to use the Cliff-Lorimer correction described above with an absorption correction for thicker samples. Enter the sample thickness in nm and its density in g/cc).
		Note:
		<ul> <li>If you want to specify the ratio element for the Cliff-Lorimer correction, see Select Ratio Element for Cliff-Lorimer Correction.</li> <li>Bulk matrix corrections (Proza or ZAF) do not work with</li> </ul>
		TEM thin sections.

## Table 12. Spectrum Mode Quant Setup, Advanced View

Group	Setting	Description
	Calculate all elements as compounds	Selecting this option changes all reported elements to their associated compounds. The associated compound is the compound that is currently selected in the Compound Formula list in the Advanced Element box (default is the first compound in that list). To open the Advanced Element box, right-click the element in the Element Setup pane and choose <b>Advanced</b> . Here is an example:
		Advanced Element Compound Formula Quantification of Weight % Calculated By difference Fixed % Use Derivative References Use Compound
		<b>Note</b> : To add measured compounds to the results pane, click the <b>Options</b> button in that pane and make sure <b>Compound%</b> is selected for display. To add the compound name to the results table, make sure <b>Chemical Formula</b> is also selected for display.
Normalize cations based on the following number of oxygen atomsThis setting enables the software to an oxide, typically a mineral, from example, if you suspect the analysis has a chemical formula (Mg,Fe)_2Si Based On" to 4. When viewing t results, if the Mg and Fe values sun stoichiometry consistent with an ob	This setting enables the software to calculate the stoichiometry of an oxide, typically a mineral, from a quantitative analysis. For example, if you suspect the analysis was done on an olivine, which has a chemical formula $(Mg,Fe)_2SiO_4$ , set "Normalize Cations Based On" to 4. When viewing the numbers of cations in the results, if the Mg and Fe values sum to 2, and Si=1, the analysis has stoichiometry consistent with an olivine. If not, the results likely represent some other oxide, or they indicate a poor analysis.	
		<b>Note</b> : To add a No. of Cations column to the results pane, click the <b>Options</b> button in that pane and make sure <b>No. of Cations</b> is selected for display.

## **Quantify Elements Manually**

When quantitative analysis is performed automatically, the software quantifies all the elements listed as Identified (green) or Always Identified (pink) in the periodic table.

If you know the sample composition and want to limit the quantitative results to specific elements, you can turn off automatic identification and use the periodic table to specify the elements to quantify.

**Note** Your selections in the periodic table do not change when you select a different analytical mode. This lets you analyze a spectrum in one mode, then switch to another mode and use the same analysis results.

#### \* To quantify the selected elements manually

1. Acquire a spectrum for a sufficient amount of time to allow for a good statistical analysis.

For quantitative measurements, use more than 5,000 counts vertical full scale and less than 30% dead time. Recommended acquisition settings:

Feature	Recommended Setting
Live Time Limit	30 seconds
Max Peak Counts	0
Low Energy Cutoff	100 eV
High Energy Cutoff	20 keV for SEM applications 40 keV for TEM or STEM applications
Time Constant	Rate 1

2. Run a qualitative analysis on the spectrum and confirm the list of found elements.

**Note** The accuracy of the qualitative results impact the quantitative analysis. For more information, see Qualitative Analysis and Using Spectra\_Check to Verify Peak Identification.

- 3. If there are found elements you want to exclude from the quantitative results, right-click each element symbol in the periodic table and choose **Excluded** (their symbols change to dark grey).
- 4. Click the Spectrum Processing icon icon on the controls panel, click **Quant Setup** and then click to toggle the green arrow to open the advanced view.

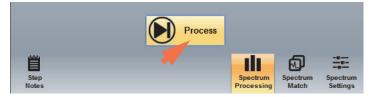
Recommended quant settings:

Feature	Recommended Setting
<u>Auto Quant</u>	Selected
Quant Method	Standardless (adequate for most analyses)

Feature	Recommended Setting
<u>Peak Fit Method</u>	<ul> <li>Select a peak fit algorithm that suits the samples to be analyzed. Available options:</li> <li>Filter Fit. Applies a digital "top hat" filter to remove the background and determine the peaks.</li> <li>Gaussian. Uses Gaussian functions to calculate and subtract a theoretical background and determine the peaks.</li> </ul>
Correction Method	<ul> <li>Select a matrix correct that suits the samples to be analyzed.</li> <li>Available options: <ul> <li>None. Reports net counts only.</li> <li>ZAF. For metallurgical SEM applications.</li> <li>Proza. For SEM applications, especially light elements.</li> <li>Cliff-Lorimer (MBTS). For TEM and STEM applications.</li> </ul> </li> </ul>

- 5. Click the **Options** button in the results pane and <u>select column headings for the reported</u> <u>results</u>.
- 6. Click the **Process** button to manually quantify the spectrum.

The button is located at the bottom of the Quant Setup tab in the Spectrum Processing group on the controls panel.



When the analysis is completed, the results are displayed in the results pane.

**Note** You can change the elements used for quantitative analysis at any time or any of the settings in Quant Setup. After you change an element or setting, click the **Process** button to rerun the analysis and update the results pane.

## Set an Element Weight Percent to a Fixed Value

#### \* To set the reported weight percentage for an element to a fixed value

1. Right-click the element and choose Advanced.

2. In the Advanced Element box, set **Quantification of Weight%** to Fixed and type a weight percentage in the associated entry box.

Advanced Element	
Quantification of Weight % ~ Calculated By difference Fixed 18 %	Compound Formula SeO SeO3
Use Compound -Mapping Energy Range From: 1278 T Is Ratio Element	(eV) o: 1480 Close

The software sets the weight percentage for this element to the entered value and takes that into account when calculating the quantitative results for other elements.

## **Report Compounds by Stoichiometry**

You can associate specific compounds with individual elements in the periodic table, or configure the software to use the associated compounds for all identified elements.

#### \* To associate a specific compound with an element

- 1. Right-click the element symbol in the periodic table and choose Advanced.
- 2. In the Advanced Element box, select Use Compound.
- 3. Select a item in the Compound Formula list.

Advanced Element	
Quantification of Weight % Calculated By difference Fixed	Formula
✓ Use Derivative References	
Mapping Energy Range (eV) From: 2887 To: 3081	Close

#### **\*** To use the associated compounds for all elements

1. Click the Spectrum Processing tab on the controls panel, click Quant Setup and toggle the green arrow to show the advanced view.

#### 2. Select Calculate All Elements As Compounds.

When this option is selected, the software reports the compound formula associated with each element instead of the pure element. This is true even if individual elements do not have their **Use Compound** boxes selected (see the previous section).

If you selected a compound for an element, the selected compound is reported. If you did not select a compound for an element, the first element in that element's compound formula list is reported.

#### Note

- Leave Normalize Cations Based On The Following Number Of Oxygen Atoms set to zero. The software calculates the number of oxygen atoms based on the most common valence. Change this value only when the number of oxygen atoms is known.
- To add the compound name to the results pane, click the **Options** button in that pane and make sure **Chemical Formula** is selected for display.

# **Additional Tools in Spectrum Mode**

## **Reference Subtract/Add**

You can subtract (or add) one or more element peaks or X-ray lines from (or to) the displayed spectrum using reference peaks from the software's internal database. The spectrum must be quantified before you can subtract or add a reference peak.

#### \* To subtract (or add) an element peak from (or to) the displayed spectrum

- 1. Display the spectrum you want to process.
- 2. From the Home screen, choose **Spectrum** (menu) > **Reference Subtract/Add**.
- 3. In the Select Elements To Subtract box, select Subtract or Add.
- 4. Select one or more element peaks or X-ray lines from the list or click **Select All** to select all the available lines.

The list includes all the elements found in the displayed spectrum.

5. Choose Apply (or OK) to complete the subtraction (or addition).

The resulting (displayed) spectrum has the selected peaks removed (or added).

**Note** These operations create a copy of the spectrum before making modifications. The original spectrum is not changed.

## **Element X-ray Lines**

Use the Element X-Ray Lines feature in the Spectrum menu to quickly find the energy or relative intensity value of an element and X-ray line from the software's internal database.

#### \* To reference the energy or intensity value for an X-ray line

- 1. From the Home screen, choose **Spectrum** (menu) > **Element X-Ray Lines**.
- 2. In the X-Ray Lines box, set **Element** to the element you want to reference.
- 3. Set Line Series to the type of X-ray lines for the selected element (K, L or M).

The energy in keV and relative intensity value for the select X-ray lines are displayed.

X-Ray Lines		×
File		
Element: Cu		Line Series: K
Line	Energy (keV)	Relative Intensity
Ka1	8.046	1000
Kb1	8.904	201
Ka2	8.026	514
-Selected Line		
Line:	<ul> <li>Energy:</li> </ul>	Intensity:
	Modify Database	-
Default Value	s	OK Cancel

**Note** The features in the Selected Line group allow you to edit the stored reference values for any element. However, these values are generally accepted physical constants and, thus, should not be changed.

4. Choose OK.

## **Batch Process**

You can quantify a number of spectra and save the results as a CSV (Comma Separated Values) file (.csv filename extension) which can be opened in a spreadsheet application, such as Microsoft Excel, for further mathematical analysis.

**Note** Batch processing can only be used on files that have been saved in the project folder (no temp files). To save files to the project folder, choose **File** (menu) > **Save** or click the **Save** button on the main toolbar.

#### ✤ To batch process spectra

- 1. From the Home screen, choose Batch Processing (menu) > Spectrum Analysis.
- 2. In the Select Type of Spectrum Processing to Perform window, select whether, as part of the batch process, you would like to Remove Escape Peaks, Remove Sum Peaks, Identify Elements, Quantify Elements or Match Spectrum.

Select Type of Spectrum Processing to Perform
Remove Escape Peaks
Remove Sum Peaks
☑ Identify Elements
Quantify Elements
Match Spectrum
OK Cancel

**Note** If you select Quantify Elements above, you may want to clear (deselect) Identify Elements and predetermine your element list before you start the batch process.

- 3. Then click OK.
- 4. In the Select Files window, locate and select the spectrum (.emsa) files to process (use Ctrl or Shift keys to select multiple files) and click **Open**.
- 5. In the Enter a Filename box, type a filename for the csv file, select a folder for saving the file and choose **Save**.

## **Region Tool**

Use the region tool to extract peak counts or rate data from a spectrum. You can also perform mathematical manipulations on element data in a spectrum. For example, you can add, subtract, multiply or divide peak counts or peak rates of elements in a spectrum. You can also determine the net and gross counts of a single peak or determine a ratio (divide) of two elements in a spectrum or X-ray line ratio.

#### \* To use the region tool to extract peak information

- 1. Display the spectrum you want to extract peak information for.
- 2. From the Home screen, choose **Spectrum** (menu) > **Region Tool**.

If the Spectrum menu is not displayed, click the spectrum pane.

- 3. Set **Element** to the desired element.
- 4. Set Line to an available X-ray line for the selected element.
- 5. Set **Data Type** to Counts or Rate.
- 6. Set **Operator** to None.
- 7. Click the plus (+) button to add the operation to the Region Operations list.

The gross count (Gross Rate) and net count (Net Rate) results are displayed in the Region Operations box (background is subtracted first).

Region Tool			×
Region Operations		Gross Rate	Net Rate
Al_K (1410:1562 eV)		594.37	390.01
Data Type Counts	Rate		+ •
Element: Line:	Operator:	Element:	Line:
Al 🔻 K 🗸	None 🔻	AI	K
	Close		

## Note

- To remove an operation, select the row in the Region Operations box and click the minus (-) button.
- Subtract net counts from gross counts to get the counts in the background.

#### To extract information for two element peaks

- 1. From the Home screen, choose **Spectrum** (menu) > **Region Tool**.
- 2. Set the left **Element** box to the desired element for the first peak.
- 3. Set Line to an available X-ray line for the selected element.
- 4. Set the right **Element** box to the desired element for the second peak.
- 5. Set Line to an available X-ray line for the selected element.
- 6. Set **Data Type** to Counts or Rate.
- 7. Set **Operator** to the operation to perform (divide, multiply, add or subtract).

8. Click the plus (+) button to add the operation to the Region Operations list.

The results are displayed in the Region Operations box. (For example, element1 gross counts/element2 gross counts and element1 net counts/element2 net counts.)

ion Operations		Gross Rate	Net Rate
K (1410:1562 eV) + Mg_K (1182:1326 eV)		739.74	449.65
-Data Type	Counts   Rate		+ -
Element: Line:		Element:	+ -
0		Element:	<u> </u>

## ✤ To extract information for a selected spectral region

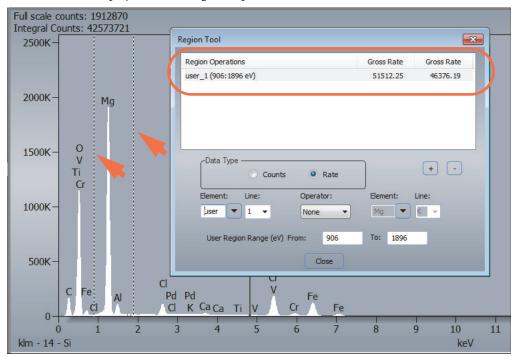
1. From the Home screen, choose **Spectrum** (menu) > **Region Tool**.

If the Spectrum menu is not displayed, click the spectrum pane.

- 2. Click the **Element** box and type "user."
- 3. In the User Region Range, type and region start and end in eV.

You can also drag the vertical region markers (dashed lines) right or left to define the region.

4. Click the plus (+) button to add the operation to the Region Operations list.



The results are displayed in the Region Operations box.

## **Spectrum Math**

Use Spectrum Math from the Spectrum menu to perform math operations on spectra. You can add, subtract, multiply and divide any or all of the spectra in a project by a constant or by another spectrum. In addition, you have the option of getting the average or summed spectrum from any of the project spectrum files.

#### \* To perform math operations on spectra

- 1. Display the spectrum you want to use for math operations.
- 2. From the Home screen, choose **Spectrum** (menu) > **Spectrum Math**.

If the Spectrum menu is not displayed, click the spectrum pane.

- 3. In the Spectrum Math box, click Select.
- 4. In the Spectrum Selection box, set **Files of Type** to an available file type, select a file in the current project (use the Ctrl or Shift key to select multiple files) and choose **OK**.

These file types are available:

Analytical Mode	File Type
Spectrum	.emsa
Point ID	.psmsa

Analytical Mode	File Type
Spectral Imaging	.fzs and .fzsa
Linescan	.lsmsa

You can also left double-click to select a file and close the Spectrum Selection box, or right double-click to add a file and close. The selected file(s) are listed in the Spectrum Math box.

- 5. Set **Operator** to the desired math operation (see below for details).
- 6. Set **Operand**. The following options are available:
  - **Constant**. Select the constant in the list box or enter a value.
  - Spectrum. Use the list box to select another spectrum in the current project.
- 7. Use the **Result Name** box to enter a filename for storing the math operation result.
- 8. Click **Calc. Result** to perform the operation.

A progress bar shows the progress of the selected math operation(s). When the operation is completed, the resulting spectrum is displayed in the spectrum pane.

#### Note

- When selecting Average or Sum operations, each of the selected files are read into memory and the average or sum spectrum is generated by averaging or summing the data on a channel-by-channel basis. Selecting any of the other choices result in the math operation being performed on each of the selected files with a new result spectrum being created for each.
- All math operations are performed on a channel by channel basis. The calculations are conducted with floating pint arithmetic. Upon completion of the calculation for a given channel, the floating point number is rounded to the nearest integer count value before the result is stored. If the calculation results in a negative number for the number of counts in a channel, the negative value is stored in the spectrum. All result spectra are stored in the project's temp folder.

#### **Available Math Operators**

Math Operator	Result of Operation	
+ (Add)	Add to each spectrum	
- (Subtract)	Subtract from each spectrum	
x (multiply)	Multiply each spectrum	
/ (divide)	Divide each spectrum	
Average	Average several spectra	
Sum	Sum several spectra	

Math Operator	Result of Operation
Normalize Beam Current	Normalize each spectrum by beam current
	<b>Note</b> : When you normalize a spectrum to a constant beam current, the constant beam current is stored with the attributes of the result spectrum. The normalization of a constant beam current is the same as livetime normalization. The counts of the result spectrum are normalized to what they would be if the constant beam current had been used for the spectrum acquisition.
	When you normalize a spectrum to another spectrum, the result spectrum has counts that would have been present if the selected spectrum had been counted with the same beam current as the Operand spectrum. Upor completion the result spectrum has attributes that reflect those of the Operand spectrum upon which the normalization is based.
Normalize Livetime	Normalize Livetime (same note above applies to Normalize Livetime)
Normalize Beam Curr. and LT	Normalize by both beam current and livetime
Normalize vertical full scale	Normalize each spectrum by the maximum counts

## **Reset the Spectrum Math Window**

To clear the selected file list and reset the Result Name to a default based on the current <u>experiment name</u>, click the **Reset** button in the Spectrum Math box. It also resets the Operator to Add, the Operand Type to Constant and the Operand to 1.0.

#### **Right-Click Menu**

If you right-click when the cursor is within the Spectrum Math box boundaries, a menu is displayed so that you can easily perform common actions. the menu items include:

- **Clear the Selected Spectra List.** Removes all spectra from the selected file list without performing a complete reset of the Spectrum Math box.
- Add Current (displayed spectrum) to List. Provides an easy way to add a currently displayed spectrum to the selected file list.
- **Calculate the Result Spectrum**. Alternate way to calculate the result using the current settings in the Spectrum Math box.

**3 Spectrum Mode** Additional Tools in Spectrum Mode

# 4

# **Electron Imaging Mode**

This section covers electron imaging mode, which can be used to acquire, save and load electron image files. The images are saved in the standard TIFF (.tif) file format. A ruler is provided for measuring objects in the image. Guidelines for setting up and acquiring images from a scanning electron microscope (SEM) are provided. With optional stage and column integration modules and the Analysis Automation software, you can also acquire and generate a single image from multiple images (montage).

# **Select Electron Imaging Mode**

To select electron imaging mode, from the Pathfinder Home screen, choose **View** (menu) > **Electron Imaging Mode**.

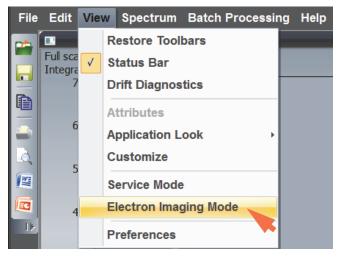
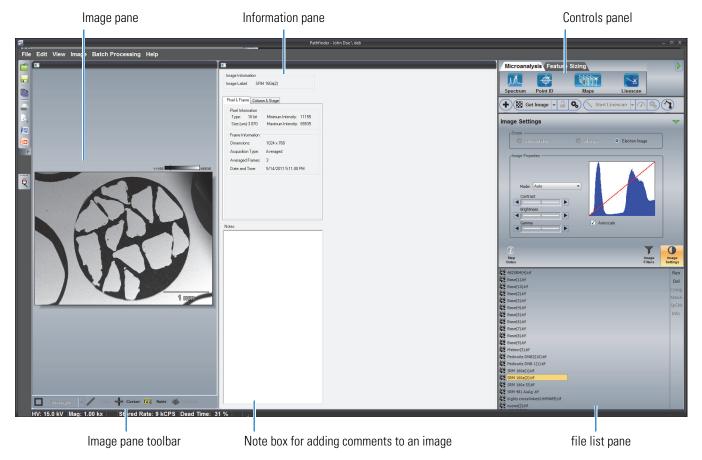


Figure 44. Select Electron Imaging Mode

Electron imaging mode contains all the necessary tools for collecting electron image data and for image data processing and file manipulations.





# **Acquire an Electron Image**

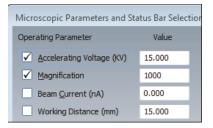
The software is configured to acquire an electron image of the current microscope location on the sample. You can acquire an electron image two ways, as an averaged, static image or as a live image with continuous image acquisition. This section explains how to acquire the image both ways.

## **Acquire Averaged Electron Image**

An averaged electron image is a static (on time) image of the microscope view. If you collect more than one frame (one scan per frame), the frames are averaged to produce the image.

#### To acquire an averaged electron image

- 1. Insert the sample on the microscope.
- 2. Set the basic microscope parameters:
  - From the Pathfinder Home screen, choose **Edit > Microscope Parameters**
  - Set these microscope parameters and then choose Close
    - Accelerating Voltage in kilovolts (kV)
    - Magnification
    - Working Distance in millimeters (mm)



**Note** If your SEM has Column Communications, these parameters are entered automatically.

- 3. Focus the sample (it should be in critical focus).
- 4. From the Pathfinder Home screen, choose View > Electron Imaging Mode.

- 5. Set the image parameters, or use one of the <u>acquisition presets</u> such as Fast Image or High Resolution or a <u>User-Defined</u> option (see the image below).
  - Click the Electron Image Setup in button on the acquisitions toolbar (normal view toggle setting).

	Acquisition
	presets
	Electron
Microanalysis Fe ture S zing	Image Setup
Spectrum Point ID Me ps Linescan Imaging	
+ 🔀 Get Image + 🔒 🍫 🗸 - 🖓 🥎	
Electron Image Setup	Normal/Advan ced View
Averaged Acquisition	toggle setting
Resolution: 512x384 💌	
Frame Time (s): 5.0 - Dwell Time (us): 25	
Number of Frames: 2	
Acquisition Time (s): 10	
Custom	
Use Input 1 Use Input 2	

- In the Averaged Acquisition group:
  - Set **Resolution** (image size in pixels) to 512x384. This setting provides a good trade off between acquisition time and resolution. (A 512 image can be digitally zoomed about 4 times before it becomes pixelated.)
  - Use the **Frame Time** box to enter or select the number of seconds to acquire each scan. Generally, a 5–10 second Frame Time produces a good pixel-to-noise ratio with few averaged frames.
  - Set <u>Number Of Frames</u> to 2. Acquiring two averaged frames (each frame is one complete scan) produces a good image under most conditions for the first acquisition; thus you can eliminate the need to change settings and reacquire the data.
- Make sure Use Input 1 is selected (selects the microscope input channel to generate the image).

6. Enter an Experiment Name, which will be used as a base name for saving the image files.

On the acquisitions toolbar, click the 🕮 button and enter the Experiment Name (or leave the default name).



7. Start the acquisition and wait for it to complete.

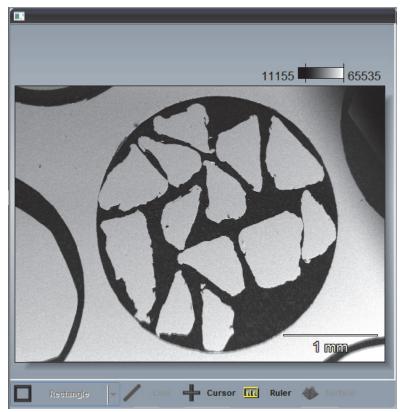
On the acquisitions toolbar, click **Get Image**. The Get Image button changes to Stop Image, and the Pause and Abort buttons appear.

	Stop Image Stop image acquisition	Abort image acquisition
Feature		Description
Stop Ima	ige	Stops image acquisition at the end of the current frame and save acquired data
Abort Im	nage	Stops image acquisition immediately without saving acquired data

The image progresses vertically in the image pane. The progress bar at the top of the Pathfinder window also shows the progress in real time.

9 secs

When the acquisition is completed, the acquired image appears in the image pane in the upper left corner of the Pathfinder window. Here is an example of a typical averaged electron image.



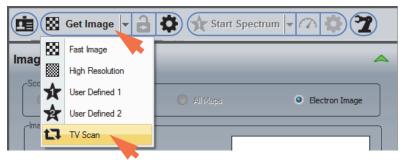


The software saves the image automatically in the open project using the experiment name entered in step 6 above, plus a numeric ID and a .tif filename extension.

## Acquire Live Electron Image (TV Scan)

TV Scan mode allows you to use Pathfinder software almost at a video rate to view selected locations on a sample and to optimize the image settings to match the microscope.

To acquire a live electron image using the default settings, set the acquisition preset to **TV Scan** and click **Get Image**.



**Note** You can set Resolution and Frame Time for TV Scan acquisitions. See Electron Image Settings for details.

# **Electron Image Settings**

## **Image Settings**

The following settings are available for creating and viewing an electron image in Electron Imaging Mode.

## Table 13. Electron Imaging mode settings

Feature	Group	Setting	Description
Electron Image Setup	Averaged Acquisition	Resolution	Image size for averaged image acquisitions in pixels. (Your computer's video adapter determines the number of Y pixels). A higher resolution produces greater image detail but may increase acquisition time. Recommended setting is 512.
		Frame Time (s)	Time in seconds to acquire one frame. A frame is one complete scan from the upper-left corner to the lower-right corner of the image. A long frame time reduces image noise but increases acquisition time. A short frame time can result in blurred, streaked or noisy images. Optimum values depend on beam current, microscope and detector type (BS or SE). Select Fastest to automatically select the fastest possible frame time for a given image resolution. Recommended setting is 5 seconds.
		Dwell Time (us)	Shows the length of time the beam will remain at each pixel location in µseconds (based on the current <u>Resolution</u> and <u>Frame Time</u> settings). A long dwell time reduces image noise but increases acquisition time. A short dwell time might cause a blurred, streaked or noisy image.
		Number of Frames	Total number of frames to be acquired or averaged using the Kalman averaging technique. A frame is one complete scan from the upper-left corner to the lower-right corner of the image.
			Recommended setting is 2 frames per second. For beam-sensitive samples, increase the number of frames.

## Table 13. Electron Imaging mode settings

Feature	Group	Setting	Description
		Acquisition Time	Estimated total acquisition time in seconds.
	Live Acquisition	Resolution	Image size for continuous (TV Scan) acquisitions in pixels. (Your computer's video adapter determines the number of Y pixels). Generally, a low resolution is best for continuous acquisition.
		Frame Time	Time in seconds to acquire one frame. Generally, a low frame time is best for continuous acquisition.
		Dwell Time	Shows the length of time the beam will remain at each pixel location in µseconds (based on the current <u>Resolution</u> and <u>Frame Time</u> settings).
	User Defined button		Allows you to save up to two instances of the Electron Image Setup screen (User Defined 1 and User Defined 2) that can be easily recalled later by clicking the <u>acquisition presets</u> button on the <u>Acquisitions toolbar</u> and choosing an option from the dropdown list.
	Channel	Use Input 1 Use Input 2	Specifies the channel from which the image data is acquired (for example, the SEI or BEI imaging detector of your microscope).
			<b>Note</b> : Your microscope may not be set up for using the Channel group.
	Compensation Interval		Applies to optional Drift Compensation software only. See <u>Drift Compensation</u> for more information.
	Advanced	Zoom factor	Applies to optional <u>Drift Compensation</u> software only.
Image Settings		Scope	Specifies where to apply the image settings (electron image, a selected map or all maps). In Electron Imaging mode, Scope is automatically set to Electron Image and cannot be changed.

## Table 13. Electron Imaging mode settings

Feature	Group	Setting	Description
	Image Properties	Mode	<ul> <li>Defines how to apply selected properties. Available options:</li> <li>Manual. Lets you manually adjust brightness, contrast and gamma encoding using the slider bars.</li> <li>Auto. Expands the histogram to average the image across the full range of intensities (0–65000 for a 16-bit image).</li> <li>Hot Pixel Suppression. Prevents unusually bright pixels (typically due to noise) from skewing an analysis results.</li> <li>Note: The Contour option in the Mode list box applies to Spectral Imaging mode only.</li> </ul>
		Contrast	Adjusts the contrast of the displayed image
		Brightness	Adjusts the brightness of the displayed image
		Gamma	Applies a gamma function to the <u>colorizer</u> used to display the selected image or map. Allows you to see detail at one end of the color ramp or the other (makes one end more sensitive than the other). Slide the Gamma bar to the positive (right) end to enhance the lower (darker) end of the color ramp; move it to the negative (left) end to enhance the upper (lighter) end of the ramp.
		Autoscale	Automatically scales the histogram and expands it to the grey levels from 0 to 65000.

## Table 13. Electron Imaging mode settings

Feature	Group	Setting	Description
		Show Legend	Displays or hides the element overlay legend for the image pane. The legend shows the maps (Spectral Imaging mode) or line scans (Linescan mode) that are overlaid on the electron image and their associated colors. Here is an example of a map legend: Mg K Overlay legend (Image pane)
			The legend is available only when one or more extracted maps (SI mode) or line scans (Linescan mode) are selected for overlay. To select a map or line scan for overlay, click its element name so that the element name is highlighted (Spectral Imaging mode) or checked (Linescan mode). Here is an example of an element map that is selected for overlay.
			Mg K 100 µm Map selected for overlay
		Transparency Factor	Adjusts the transparency of any maps that are overlaid on the electron image (so you can see the image features below the map)

### Table 13. Electron Imaging mode settings

Feature	Group	Setting	Description
		Marker Colors	<ul> <li>Lets you select colors for the following items in the image pane:</li> <li>In the Image Markers group</li> <li>Coordinated Cursor. Specifies the color of the intensity cursor when it is displayed in the image pane.</li> <li>Extraction Shape. Applies to Spectral Imaging mode only. Specifies the color of the drawn rectangle, spot, polygon or magic wand when these are displayed in the image pane. For extracting maps from an electron image.</li> <li>Micron Bar Foreground. Fill color for micron bar displayed in the image pane.</li> <li>Micron Bar Background. Outline color for micron bar displayed in the image pane.</li> <li>In the Map Markers group (applies to Spectral Imaging mode only):</li> <li>Coordinated Cursor. Specifies the color of the intensity cursors when they are displayed in the maps pane in Spectral Imaging mode.</li> </ul>
		Micron Bar	Specifies the position of the micron (scale) bar in the image pane

# **Image Presets**

In Electron Imaging mode, there can be up to five setting presets available for acquiring an electron image. To select a preset, click the down arrow at the right of the Get Image button.

Figure 47. Acquisitions toolbar, electron image acquisition presets

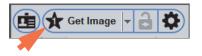


These selections are available:

- **High Resolution**. Use to quickly set up for high resolution (1024x768) image acquisition.
- Fast Image. Use to quickly set up for fast image acquisition.
- User Defined 1 (and User Defined 2). Two sets of user-defined conditions that have been previously saved. See below for details.

- **TV Scan**. Acquires a continuous (live) "video" image of the sample, which can be helpful for finding specific features on a sample.
- \* To set up user-defined conditions for electron image acquisition
- 1. From the Pathfinder Home screen, choose **View > Electron Imaging Mode.**
- 2. Click the Electron Image Setup 🔯 button on the acquisitions toolbar and set the parameters as desired.
- 3. Click User Defined > Save User Defined Settings 1 (or Save User Defined Settings 2).

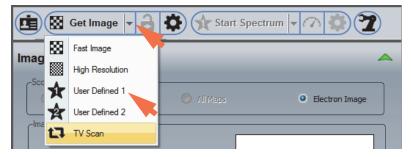
The icon on the toolbar changes to User Defined 1 (or User Defined 2) to show which preset is selected.



**Note** In the future, use the arrow next to the Get Image button on the Acquisitions toolbar to select your custom conditions.

### To update custom settings

- 1. In Electron Imaging mode, click the Experiment Setup 💭 button on the acquisitions toolbar.
- 2. Click the Presets button and select the custom settings set you want to modify (User Defined 1 or User Defined 2).



- 3. Modify parameters as desired.
- Choose User Defined > Save User Defined Settings 1 (or Save User Defined Settings 2) to save the new settings.

# **About the Image Pane**

The image pane shows the electron image that was most recently acquired or the stored image from the selected image file.

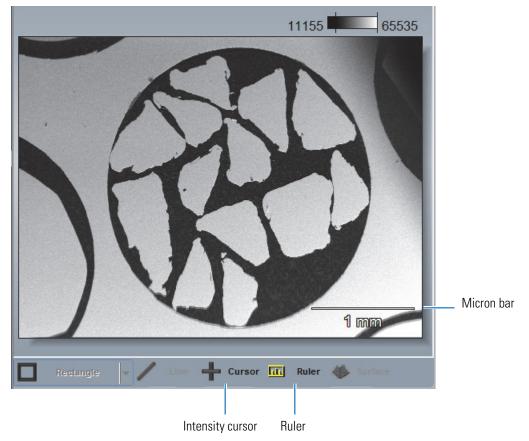


Figure 48. Example of typical averaged electron image displayed in Electron Imaging mode

In Electron Imaging mode, the following tools are available for interacting with the displayed electron image.

Tool	Description
Micron bar	Shows the scale of the displayed image in the appropriate unit. <u>Position and colors of micron bar</u> are selectable.

Tool	Description	
Ruler	Adds the ruler to the displayed electron image. The ruler can be used to measure features in the image. To shorten or lengthen the ruler, drag either end. To move the ruler, drag it from the middle.	
	761.042 um Ruler	
Intensity cursor	Adds the intensity cursor (+) to the displayed electron image. The cursor shows the pixel intensity and location of the current curpixel in the upper left corner of the image pane. You can drag cursor to another location on the image to update the pixel intensity and location information.	
	Pixel location Pixel intensity Intensity cursor	

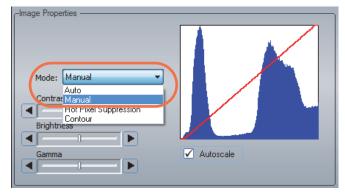
# **Post Acquisition Image Processing**

The following tools are available for working with the displayed image.

- Adjust Contrast, Brightness and Gamma Settings
- Measure Particles or Features

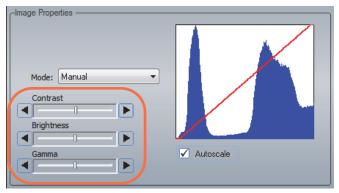
# **Adjust Image Contrast, Brightness and Gamma Settings**

- ✤ To adjust image contrast, brightness and gamma
- 1. From the Pathfinder Home screen, choose **View > Electron Imaging Mode**.
- 2. Acquire an electron image or open a stored one. (The image should be displayed in the image pane.)
- 3. On the Controls panel, click **Image Settings**. (The software automatically sets Scope to Electron Image when Electron Imaging mode is selected.)

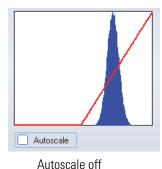


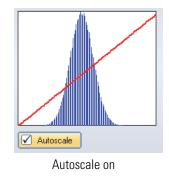
4. In the Image Properties group, set **Mode** to Manual, Auto or Hot Pixel Suppression.

- Manual. Lets you manually adjust brightness, contrast and gamma encoding.
- Auto. Automatically expands the histogram to average the image across the full range of intensities (0–65000 for a 16-bit image).
- Hot Pixel Suppression. Prevents bright pixels from skewing an analysis.
- 5. If Manual mode is selected, use the slider bars as needed to adjust the image contrast, brightness and gamma encoding.



6. To automatically scale the histogram and expand it to the grey levels from 0 to 65000, select **Autoscale**.





# Measure Particles or Features in an Image

If you have the optional <u>Feature Sizing</u> software, you can measure or count particles or features in an image and extract a number of parameters.

# **Manage Image Files**

# Save an Image File

Files saved in Electron Imaging mode are stored in TIFF (.tif) format with the experiment name and acquisition number.

When you close a project, the software displays a list of files that have been modified in the current session. Uncheck those files that you do NOT want saved. Choose **OK** to save the changes. Choose **Cancel** to close the Save dialog box and return to the project without saving any changes.

### To save without closing the project

1. Click the Save button \_\_\_\_\_ on the main toolbar to save to the currently open project.

Or

1. Choose File > Save.

# **Open an Image File**

### \* To open an electron image file within a project

1. Open the project.

The image files contained in the project are listed in the file list pane.

- 2. From the Pathfinder Home screen, choose View > Electron Imaging Mode.
- 3. Select the image file in the file list pane. The image appears in the image pane.

### \* To open an image file outside the current project

- 1. From the Pathfinder Home screen, choose **File > Open** and navigate to the directory that contains the file.
- 2. Select the file and choose **Open**.

The file is copied to the current project's temporary folder and inherits the current project's settings. and displayed in the image pane.

**Note** To save an imported file to the current project folder, from the Pathfinder Home screen, click the save button on the main toolbar or choose **File > Save**. If you close the project without first saving an imported file, the software displays a message asking if you want to save or discard the imported file.

# **Rename an Image File**

### To rename an electron image file

- 1. In the file list pane, select the file to rename.
- 2. On the File Management panel, click the Rename A button to enable filename editing.
- 3. Enter the new name and press the Enter key on the keyboard.

# **Delete an Image File**

- 1. In the file list pane, select the file to delete.
- 2. On the File Management panel, click the Delete 🖉 button.

The software displays a message confirming the deletion.

3. Choose Yes to delete the file.

**NOTICE** Deleted files cannot be recovered.

# **Batch Processing**

In electron imaging mode, you can stitch together a series of images to create one larger image, referred to as a "montage." The series of images must be collected using the optional Analysis Automation and <u>Stage Control</u> software.

# Create a Montage (Mosaic) Image

Use Create Montage in the Batch Processing menu to create a montage of electron images (and X-ray maps) by stitching together images acquired as adjacent fields in a grid automation sequence into a larger image.

**Note** You must use Analysis Automation to create the grid file. For more information, see <u>Acquiring Grid Files</u> in <u>Analysis Automation</u>.

### **\*** Creating a montage from a grid file

- 1. From the Pathfinder Home screen, choose **View > Electron Imaging Mode**.
- 2. Choose Batch Processing > Create Montage.

The Create Montage box is displayed.

Create Montage	×
Process Counts     O Process	ss Quant 💿 Process Compass
Compass Parameters	
Correlation Factor (01): 0.99	
🗖 Create Phases 📃 U	Jse image filters when creating phases
Select Files to Process Cer Brake Pad[Grid2]	87 229
Processing Progress	250 μm
Montage Progress	Close

Image files acquired in a grid pattern using Analysis Automation are listed in the Select Files to Process box. Grid files are named with the experiment name followed by "[Gridn]." (They have a .grid file extension in the project folder.)

- 3. Select a grid file to use for the montage.
- 4. Click the arrow button to start the montage.

The progress bars show the progress. When the montage is completed, it is displayed in the image pane. Here is an example of a montage image created from a grid file.

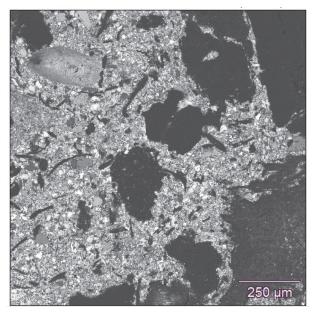


Figure 49. Displayed montage created from Analysis Automation grid file

The montage data are saved in the Grid folder of the open project with this filename:

[gridn,montage].tif

## 4 Electron Imaging Mode

Batch Processing

# 5

# **Point ID Mode**

This section covers Point ID mode, which can be used to acquire spectra from user-defined points or areas of an acquired electron image.

You can start with an existing electron image or acquire a new image. Each new electron image starts a new Point ID data file. Files are named with the experiment name and a numeric identifier, followed by "PS" (for "") and an .eds filename extension.

Spectral acquisition and analysis works the same way in this mode as it does in <u>Spectrum</u><u>mode</u>. You can acquire spectra two ways, instant or batch, using a mixture of five different shapes (point, rectangle, irregular, Fat Spot and polygon). An instant acquisition begins when you click a new analysis point or area. In a batch acquisition, you select a number of analysis points or areas and then begin an automated sequence of spectral acquisitions.

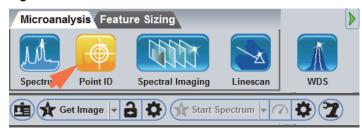
You can add analysis points or areas to the open Point ID file until the file is saved or closed by either acquiring a new image or selecting a different analytical mode.

**Note** You must acquire a new electron image before adding new analysis points or areas if you have switched the analytical mode, changed projects, or opened a Point ID file containing areas that were previously acquired. If you switch to another analytical mode or open a different project, acquired areas will be retained and areas that have not been acquired will be removed.

# **Select Point ID Mode**

To select Point ID mode, click the Point ID button on the analytical mode toolbar.

Figure 50. Select Point ID mode



In Point ID mode, the software displays all the features and tools for X-ray count analysis of the selected microscope field of view.

Figure 51. Pathfinder Point ID Mode User Interface

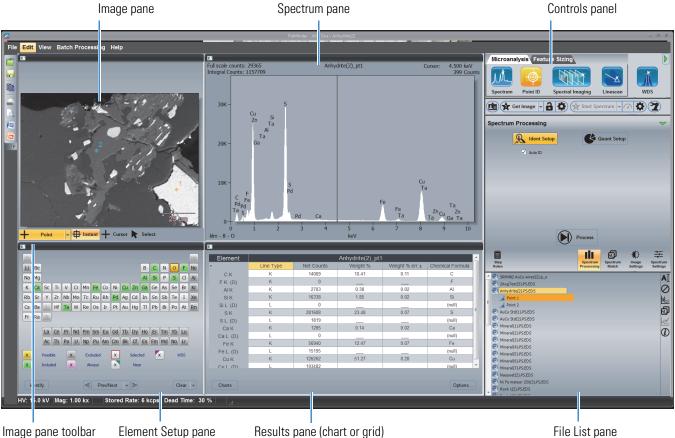
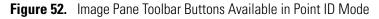
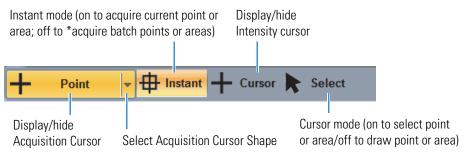


Image pane toolbar

Element Setup pane

File List pane





\* Use Start Spectrum button on Acquisitions toolbar to start a batch acquisition.

# **Acquire Electron Image for Point ID Measurements**

An electron image is required for all Point ID analyses. An average electron image must be used for the analysis, but a live image can be used for preliminary sample viewing. For more information, see Acquire an Averaged Electron Image.

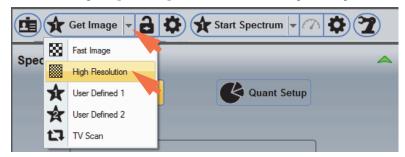
**Before you begin**, enter the microscope settings. Most modern microscopes allow the software to read the microscope accelerating voltage, working distance and magnification values automatically when they are needed. If your microscope does not include column communications, enter these values before you start an analysis and update the values whenever the Microscope Parameters box is displayed.

### \* To acquire an averaged electron image for a Point ID measurement

5. Click the Point ID icon on the Analytical mode toolbar.



6. Select an image acquisition preset such as Fast Image or High Resolutions.



7. Enter an experiment name, which will be used as a base name for saving the image file and any of its associated Point ID data.

On the acquisitions toolbar, click the 👜 button and enter the experiment name (or leave the default name).



8. Click Get Image on the Acquisitions toolbar and wait for the acquisition to complete.

The image is displayed in the image pane and saved along with any subsequently acquired Point ID data.

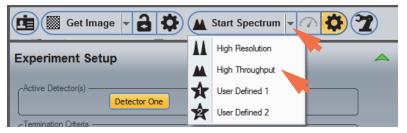
# **Set Point ID Spectral Acquisition Parameters**

Spectral acquisition and analysis in Point ID mode works the same way it does in <u>Spectrum</u><u>mode</u>. For more information, see Acquire and Analyze a Spectrum and Spectral Acquisition Settings.

For a Point ID analysis, set the acquisition parameters as you would for a single acquisition for your sample.

### \* To set the Point ID spectral acquisition parameters

Select a spectral acquisition preset such as High Resolution or High Throughput.

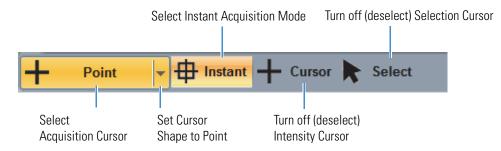


# Acquire and Analyze Data in Instant Acquisition Mode

You can do two different styles of Point ID acquisitions, instant and <u>batch</u>, using a mixture of five different shapes (point, rectangle, irregular, Fat Spot and polygon). This section covers instant acquisitions, which begin after you click a new analysis point or area. In a batch acquisition, you select a number of analysis points or areas and then begin an automated sequence of spectral acquisitions.

Use the image pane toolbar buttons to set up the analysis.

- \* To acquire and analyze data in instant acquisition mode
- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Select **Instant** acquisition mode
  - Turn off (deselect) Intensity Cursor
  - Turn off (deselect) Selection cursor

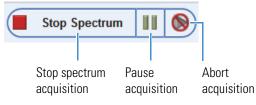


- 2. Set the **Cursor Shape** selector (see the previous image) to Point or one of the area options (rectangle, spot or polygon).
- 3. Use the mouse to locate the first acquisition point over a feature of interest on the electron image.
- 4. Click on the electron image at the location where you want to begin the analysis.

A Point acquisition starts when you click the point. For the area options, such as Rectangle and Spot, acquisition starts when you finish selecting the area.

**Note** See the sections that follow for step-by-step instructions to acquire data using each Cursor Shape option.

The progress bar appears at the top of the Pathfinder window shows the progress of the acquisition. The acquisition continues until a termination condition (such as the Live Time Limit) is met.

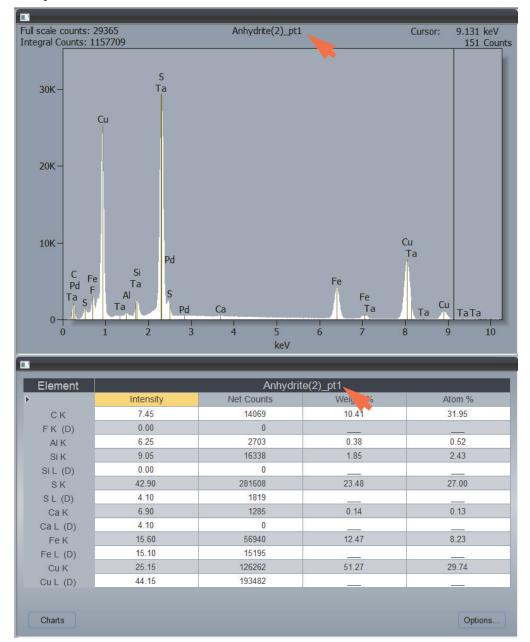


• To pause the acquisition, click the **Pause** button.

To continue the acquisition, click the pause button again.

- To end the acquisition and save the acquired data, click Stop Spectrum.
- To end the acquisition without saving the data, click the Abort button

When the acquisition is completed, the spectrum appears in the spectrum pane and any quantitative results appear in the results pane. For the area acquisitions, the spectrum and quantitative results are averaged over the defined area.



The filename and point (or area) number appear at the top of both panes. Here is an example:

The analysis results are based on the settings in <u>Quant Setup</u> and <u>Ident Setup</u> and the elements selected in the <u>Element Setup pane</u>.

5. To add another point or area, move the mouse to the new location, set Cursor Shape, and either click the new point or draw the new area. The new spectrum and results replace the previous ones in the spectrum and results panes.

The data are saved in a temporary folder with the current <u>measurement name</u> followed by the point or area number. The folder appears in the file list pane. Here is an example:

4	Stone(7).PS.EDS
-	Stone(8).PS.EDS
	🦼 Point 1
	🦼 Point 2
	🔬 Point 3
	🦼 Area 4
1	Stone(9).PS.EDS

You can select acquired points or areas for display two ways:

- by setting the Cursor Mode to Select (selected) on the image pane toolbar and then clicking the numbered point or area you want to view. The selected point or area is highlighted in the image pane and its associated spectrum and results are displayed in the spectrum and results panes, respectively.
- by selecting the measurement in the file list pane (look for the <u>measurement name</u>) to list the points or areas measured, and then selecting the numbered point or area you want to view.

When the acquisition is complete, the data set is automatically stored in the temporary directory and is ready for analysis. From here you can perform extractions to view spectral information or create different map sets and extract line scan data.

6. To save the data set from the temporary folder to the current project, click the **Save** button on the main toolbar, or choose **File** (menu) > **Save**.

After the data are saved, you can no longer add analysis points or areas to that measurement.

# **Acquire Data at Each Defined Point**

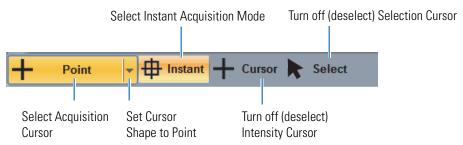
When Instant mode is selected and Cursor Shape is set to Point, you can actively acquire data at each selected point on an electron image. If you have acquired an electron image and set the acquisition parameters, an instant acquisition begins after you specify a new analysis point. (The default settings are for instant acquisitions with the beam in Point mode.)

When the acquisition is finished, you can repeat the procedure to acquire additional data using the same electron image.

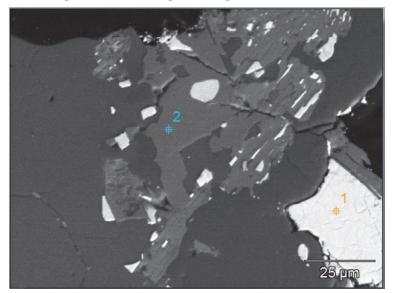
### To acquire data at each defined point on the electron image

- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Set Cursor Shape to Point
  - Select **Instant** acquisition mode

- Turn off (deselect) Intensity Cursor
- Turn off (deselect) Selection cursor



- 2. Move the mouse to a feature of interest on the electron image.
- 3. Click the point to start the spectral acquisition.



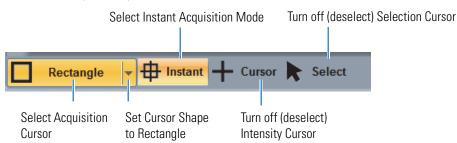
# Acquire Data at Each Defined Area

### Rectangle

When Instant mode is selected and Cursor Mode is set to Rectangle, you can actively acquire data in each defined area on an electron image.

### \* To acquire data in each defined area on the electron image

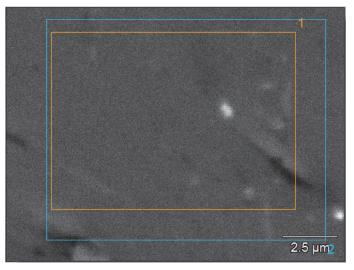
- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Set **Cursor Shape** to Rectangle
  - Select **Instant** acquisition mode
  - Turn off (deselect) **Intensity Cursor**



Turn off (deselect) Selection cursor

- 2. Move the mouse to a feature of interest on the electron image.
- 3. Click and drag to draw a rectangle around the area.

The spectral acquisition starts when you release the mouse button.

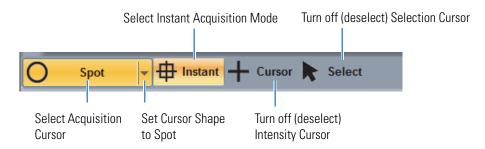


### Spot

When Instant mode is selected and Cursor Mode is set to Spot, you can actively acquire data in each defined spot on an electron image.

#### To acquire data in each defined spot on the electron image \*

- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Set Cursor Shape to Spot
  - Select Instant acquisition mode
  - Turn off (deselect) Intensity Cursor \_
  - Turn off (deselect) Selection cursor \_



- 2. Move the mouse to the center of a feature of interest on the electron image.
- 3. Click and drag to draw a circle around the area.

The spectral acquisition starts when you release the mouse button.

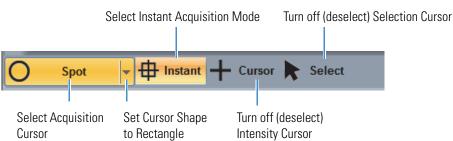


## **Polygon (Free-Hand Shape)**

When Instant mode is selected and Cursor Mode is set to Polygon, you can actively acquire data in each defined area on an electron image. The Polygon shape allows you to free-hand draw around an irregular-shaped object to scan the electron beam over that object.

### \* To acquire data at each free-hand defined area on the electron image

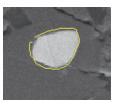
- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Set Cursor Shape to Polygon
  - Select **Instant** acquisition mode
  - Turn off (deselect) Intensity Cursor
  - Turn off (deselect) Selection cursor



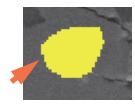
2. Move the mouse to an area of interest on the electron image.

- 3. Single-click an edge of the feature and then continue to click adjacent points to draw a boundary around the area.
- 4. Right-click to complete the polygon (it turns an opaque color).

The spectral acquisition starts when you release the mouse button.



Right-click to close the outline

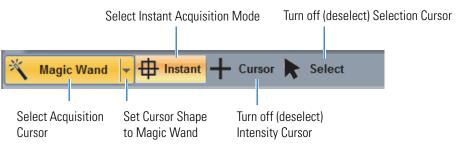


# Acquire Data in a Region Defined by Pixel Intensities

When Instant mode is selected and Cursor Mode is set to Magic Wand, you can actively acquire data in an irregular-shaped area that is defined by pixel intensities.

### \* To acquire data in each defined spot on the electron image

- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Select **Instant** acquisition mode
  - Turn off (deselect) **Intensity Cursor**
  - Turn off (deselect) Selection cursor



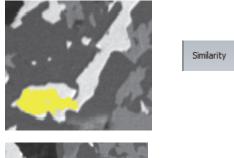
2. Set Cursor Shape to Magic Wand.

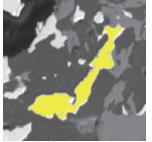
The software displays the Image Intensity Selection box.

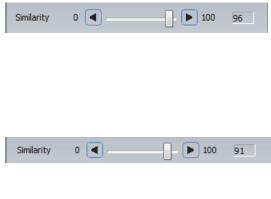
Guard Ring Width	10	um	
Cursor Diameter	10	% of image width	= 409.6 nm
	Diam	eter in microns	
Similarity 0	<		95
Smoothing	4	* *	
Brightness	28562	×	
			Close

- 3. Move the mouse to a feature of interest on the electron image.
- 4. Set Cursor Diameter to the diameter of your selection area around the tip of the mouse cursor as a percentage of the image.
- 5. Adjust Similarity.

Start with a high similarity value and then gradually reduce it to include the intensities that select features of interest.

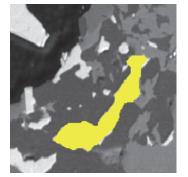






6. Increase Smoothing to better define the image.

Here is the same image with additional smoothing.



Smoothing	4	×.

- 7. Adjust the Brightness value as needed.
- 8. When you are finished, choose **Close** to start the spectral acquisition and analysis.

The progress bar appears above the spectrum pane and shows the progress of the acquisition. When the acquisition is completed, the spectrum appears in the spectrum pane and its analysis results appear in the results pane.

Table 14.         Image Intensity Selection Box Feature	ires
---	------

Feature	Description	
Guard Ring Width	Defines a perimeter width of the selected area that will not be sampled. Used to keep the excitation volume within the selected area when there are unwanted pixels next to the area boundary. Default setting is 5 um.	
Cursor Diameter	Sets the size of the area (number of pixels) around the tip of the mouse pointer as a percentage of the image width to select pixel grey level intensity. The higher the percentage value, the more pixels are evaluated.	
Similarity	Adjust the slide bar to include the intensities that select the object of interest.	
Smoothing	Select the number of smoothing passes to fill in holes.	
Brightness	This number is entered automatically based on the current mouse location. Adjust the value to change the image brightness.	

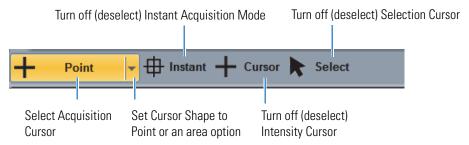
# Acquire and Analyze Data in Batch Acquisition Mode

You can do two different styles of Point ID acquisitions, <u>instant</u> and batch, using a mixture of five different shapes (point, rectangle, irregular, spot and polygon). This section covers batch acquisitions, where you select a number of analysis points or areas and then begin an automated sequence of spectral acquisitions.

Use the image pane toolbar buttons to set up the analysis and the acquisition toolbar to start it.

### \* To acquire and analyze data in batch acquisition mode

- 1. Use these Image toolbar settings:
  - Select Acquisition cursor
  - Set **Cursor Shape** to Point or one of the area options (rectangle, spot or polygon)
  - Turn off (deselect) **Instant** acquisition mode
  - Turn off (deselect) Intensity Cursor
  - Turn off (deselect) Selection cursor



- 2. Move the mouse to a feature of interest on the electron image.
- 3. Click a point (or select an area) for the first acquisition.
- 4. Set the Cursor Shape selector for the second acquisition (you can mix points and areas).
- 5. Click another point (or select an area) for the second acquisition.

Each point or area is numbered sequentially.

6. Continue adding your remaining acquisition points or areas.

You can delete a point or area without affecting the remaining drawn points or areas.

7. When you are ready to start the acquisition, click Start Spectrum on the acquisitions toolbar.



The software acquires the spectral data sequentially during one pass of the progress bar, starting with the first defined location and ending with the last one. When all the acquisitions are completed, the spectrum from the last point or area measured is displayed in the spectrum pane and its associated analysis results are displayed in the results pane.

The spectra can be analyzed individually or as a group (see <u>Batch Processing</u> for details) using the same functions as in <u>Spectrum mode</u>.

# **Delete a Defined Batch Point or Area**

### \* To delete a defined batch point or area before you start the batch acquisition

- 1. Click the Select button on the image pane toolbar so the button is highlighted.
- 2. Click the point or area you want to delete.
- 3. Press the Delete key on the keyboard.

The selected point or area is removed from the image pane and the remaining points and areas are renumbered.

# **Batch Process Point ID Data**

Point ID spectra can be quantified at one time using commands from the Batch Processing menu in Pathfinder software. You can select multiple files for batch processing.

Batch processing uses the current Identify and Quant settings to perform the analysis. The analysis results can be saved in table format to a comma separated values (CSV) file (.csv filename extension) in a selected folder. The data are organized to enable further analysis, such as calculating average Weight %.

**Note** Files must be saved so they exist in the Project folder (not just the Temp folder) before they can be batch processed.

### \* To quantify multiple Point ID spectra acquired in Batch mode

- 1. Select Point ID mode on the analytical toolbar.
- 2. Select the file you want to batch process in the file list pane.
- 3. On the main menu, select **Batch Processing > Point ID Analysis**.

A list of available measurement data is displayed with the selected measurement listed first.

4. Select one or more files to process and choose OK.

A Save box is displayed.

5. Select a folder for storing the CSV file (default setting is the current project folder), enter a filename and choose **Save**.

### Or...

Click **Cancel** to batch process the selected data without writing it to a CSV file. (This can be useful for creating reports.)

**Note** To locate and open the csv file, click the <u>Project Explorer button</u> on the main toolbar. In the Project Explorer window, right-click the project and choose **Open in Project Explorer**. This opens an Explorer window that shows the contents of the current project folder with all the files visible. Double-click to open the CSV file.

# Save Individual Spectra From a Point ID File

A Point ID file contains the electron image, the numbers and locations of the acquired points or areas and their associated spectra. To save an individual spectrum from a selected point or area, a copy of the spectrum must be separated from the Point ID file.

### \* To save a Point ID spectrum

- 1. Select **Point ID** mode on the analytical toolbar.
- 2. Select the desired point or area measurement to display its associated spectrum in the spectrum pane.

To select a point, use the <u>Create/Select button</u> in the image pane toolbar or select the measurement from the <u>file list pane</u>.

3. Select **Spectrum mode** on the analytical toolbar.

The selected Point ID spectrum remains in the spectrum pane.

4. Choose **File** (menu) > **Save**.

The spectrum is saved to the file list pane with its measurement name followed by the point or area number and an <u>.emsa filename extension</u>. Here is an example:

Stone\_pt1.emsa

# 6

# **Spectral Imaging Mode**

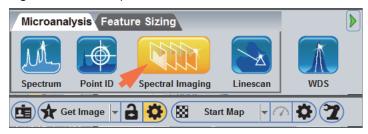
In Spectral Imaging mode, a data set of dead time corrected spectra are acquired at every point (pixel) in the microscope field of view. This data set is displayed in the image pane and referred to as the reference electron image. Once acquired and stored, you can analyze the data set and generate spectra for qualitative and quantitative analysis of selected areas, or the entire electron image, create a set of X-ray maps, extract line scans, and print reports.

Spectral imaging mode is the only way to collect X-ray maps. The optional <u>Compass</u> (Principle Component Analysis) software uses a spectral imaging data set to calculate and display principle component maps. If you purchased the optional Direct-to-Phase software, you can also extract and display phase maps during acquisition.

# **Select Spectral Imaging Mode**

To select Spectral Imaging mode, click the Spectral Imaging button on the analytical mode toolbar.

Figure 53. Select Spectrum mode



In Spectral Imaging mode, the software displays all the features and tools for X-ray analysis of the selected microscope field of view.

Figure 54. Pathfinder Spectral Imaging Mode User Interface

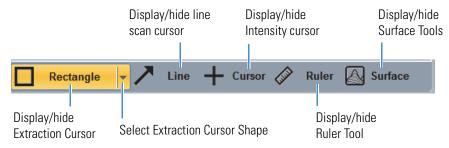


Element Setup pane

Spectrum pane

File List pane

Figure 55. Image Pane Toolbar Buttons Available in Spectral Imaging Mode



# **Overview of Spectral Imaging Data Acquisition**

The following steps outline the acquisition parameters that need to be set up before starting a Spectral Imaging (SI) acquisition. These steps are more or less in the sequence of setting the parameters but they can be set up in any sequence as long as all parameters are addressed.

### ✤ To set up spectral imaging data acquisition

1. Prepare sample.

Place into microscope, acquire image and adjust stage height (Z axis) to optimal working distance for EDS detector. Obtain good microscope image in critical focus.

- 2. Fully insert EDS detector to dead stop.
- 3. Select Spectral Imaging Mode in Pathfinder.
- 4. Set Microscope Parameters.
- 5. Set up electron image acquisition.
- 6. Set up spectral image acquisition.
- 7. Enter SI file name in Experiment Name box.
- 8. Acquire electron image.
- 9. Acquire spectral imaging data.

**Note** See Chapter 3, Spectrum Mode, for acquisition and sample preparation considerations for EDS best practices.

# **Acquisition Settings**

# **Microscope Settings**

For any analysis, the software requires the microscope accelerating voltage, working distance and magnification. To set microscope parameters, from the Home screen, choose **Edit** (menu) > **Microscope Parameters**. Enter the accelerating voltage, magnification and working distance in the appropriate edit boxes. For more information, see <u>Microscope Settings</u>.

**Note** If you have the optional Column Communications software, the microscope parameters are updated automatically.

# **Electron Image Acquisition Settings**

For any analysis, the software requires you to first acquire an electron image. You can start with a Livetime electron image. For more information, see Acquire Live Electron Image and Electron Image Settings in Electron Imaging Mode.

# **Spectral Acquisition Settings**

You can set spectral acquisition parameters manually or use a <u>spectral acquisition preset</u>. This section provides general considerations for X-ray spectral acquisition and gives brief descriptions of all the available settings for acquiring X-ray spectra in Spectral Imaging mode.

## **Spectral Acquisition Considerations**

Consider these points before acquiring X-ray data in Spectral Imaging mode for the first time.

- **Map Resolution**. When selecting a map resolution size, be aware that the larger the size, in pixels, the larger the SI data file and a longer time required for data acquisition. A 256 resolution map is a good trade-off for length of acquisition time, SI file size and map detail. A 256 SI file may be as large as 100 Mb. However a 256 map will have reasonable resolution when printed. A 128 map is about the size of a postage stamp when printed and is not very useful for map detail. However, 128 maps have short acquisition times (<5 minutes). On the other hand a 512 map takes 4 times as long to acquire as a 256 map. Be aware that when a 256 map is overlaid on the 512 gray reference image, there is some intercalation that must be calculated for the overlay.
- **Energy Range**. Within the Energy Range group there are two selections: Low Energy Cutoff (eV) and High Energy Cutoff (keV).
  - Low Energy Cutoff. Clips the energy display range from 0 eV to the value entered in the edit box (clips off the noise peak). The Auto selection adjusts the cutoff value depending on the estimated noise at a given pulse processor time constant. Generally, a fixed value of 100 to 120 eliminates the noise peak at the low energy range for most time constants.
  - **High Energy Cutoff**. Determines the number of channels allocated in memory (each channel represents 10 eV). For example, if High Energy Cutoff is set to 10 keV, 1024 channels of memory are allocated. At this range, any peak greater than 10.240 keV is not saved or displayed.

Generally, the accelerating voltage in an SEM is limited to 30 KV. At this voltage, the highest energy X-ray line that can be fluoresced and displayed is Mo K $\alpha$  line at about 17.4 keV. Thus, the high energy cutoff keV for an SEM would be 20 keV; whereas, for a TEM at 200 KV, a 40 keV range would be appropriate.

The High Energy Cutoff list box has the following options: Auto, 5, 10, 20, 40, and 80 keV. Select Auto to automatically adjust the high energy cutoff to the accelerating voltage of the microscope. If the accelerating voltage is not entered correctly, the range may be incorrect. Fixing the high energy cutoff to 20 keV for an SEM provides consistent spectra for comparison.

• **Time Constant**. The pulse processor time constant is the time allowed for the pulse processor to estimate the energy of an X-ray. The longer the time allowed, the better the estimate of the energy and the better the estimate, the higher the resolution of the peak. However, the longer the time constant, the higher the dead time for a given count rate. High resolution spectra (long time constant), translate to lower count rates and longer acquisition times.

There is a trade-off of the length of time for SI data acquisitions and the resolution of the spectra. However, for mapping, X-ray counts are taken from the center of the peak. As a result, resolution plays a much lower importance over the time required for an acquisition. Good data sets take about 10 minutes to acquire even at very high count rates. Thus, use a shorter time constant (Rates 6 to 10, depending on the available beam current from the microscope) when acquiring spectral image data sets.

Set Time Constant to Auto to allow the software to adjust the time constant rate to keep the dead time between 30% and 50%. To switch from a high resolution (longer time constant) to a faster, high count rate SI acquisition, just increase the beam current and the software will adjust the time constant. This is very convenient when shifting from fast acquisitions to high resolution acquisitions and vise versa.

- **Termination Criteria**. Essentially, there are two criteria for terminating an SI acquisition: by number of total frames or by statistics.
  - **Termination by frames**. You can set up termination by frames two ways: manual termination or total number of frames. The acquisition terminates when the total number of frames reaches the entered value, or it can be terminated manually at any time when the maps reach a "quality" level of counts (i.e., the maps "look good").
  - Termination by statistics. There are three criteria for termination by statistics:
    - **By average pixel counts**. Terminates the acquisition when the average X-ray counts in all pixels from all acquired maps reaches the specified value.
    - By total spectrum. Terminates when any peak reaches a specified vertical full scale value)
    - By element peak. Terminates when the specified element peak reaches a specified vertical full scale value)

If values, other than 0 or blank, are entered in a termination section, the first criterion reached terminates the acquisition (zero equates to infinite). Generally, spectral acquisitions are terminated by number of frames or manually.

# **Experiment Setup (Normal View)**

To access the spectral acquisition settings, click the Experiment Setup button in the right side of the acquisitions toolbar. For normal view, click to toggle the green arrow so it looks like this:

Microanalysis Feature Sizing	Experiment Setup
Spectral Image Setup  Active Detector(s)  Detector One Detector Two  SI Acquisition  Resolution: 512x384	Normal View toggle setting
Frame Time (s):     50.0     ▼     Dwell Time (µs):     254       Number of Frames:     500     ▼     Acquisition Time (s):     25000	
Energy Range Low Energy Cutoff (eV): 0    High Energy Cutoff (keV): Auto Time Constant : Auto    (projected max throughput)	
Custom	

The following spectral acquisition settings are available in normal view. For more information about setting acquisition parameters for sample acquisition, see <u>Spectral Acquisition</u> <u>Considerations</u>.

Group	Setting	Description
Active Detector(s)	Detector One/ Detector Two	<ul> <li>Available only when multiple Pathfinder- compatible detectors are installed on the microscope. When multiple detectors are installed, use the Detector One/Detector Two buttons to select which detector(s) you want to use for the analysis.</li> <li>Note: The Detector 1 and Detector 2 settings in the software refer to the detector cables attached to the Detector 1 and Detector 2</li> </ul>
		Preamp ports on the Pathfinder chassis.
SI Acquisition	Resolution	Map image size in pixels. A higher resolution provides greater image detail but increases acquisition time. Recommended setting is 256x195.

Group	Setting	Description
	Frame Time	Time in seconds to acquire one frame. A frame is one complete scan from the upper-left corner to the lower-right corner of the map. A long frame time reduces image noise but increases acquisition time. A short frame time can result in blurred or streaked images. Optimum values depend on the microscope and detector type.
		Select Fastest to automatically select the fastest frame time. Recommended setting is 5 seconds.
	Dwell Time	The length of time the beam remains at each pixel location in µseconds (based on the current <u>Resolution</u> and <u>Frame Time</u> settings). A long dwell time reduces image noise but increases the acquisition time. A short dwell time might cause a blurred or streaked image.
		Minimum dwell time should be about 4-5 times longer than the pulse processor <u>time constant</u> . Recommended setting is 50 seconds. The shortest dwell time is 50 µsec.
	Number of Frames	Total number of frames to be acquired or averaged using the Kalman averaging technique. A frame is one complete scan from the upper-left corner to the lower-right corner of the map.
		Recommended setting for first acquisition is 200-300 frames. For beam-sensitive samples, increase the number of frames. Select Infinite if you want to stop the acquisition manually by pressing Stop or Abort on the <u>acquisitions toolbar</u> .
	Acquisition Time	Estimated total acquisition time in seconds.
Energy Range	Low Energy Cutoff	X-ray counts below the specified Low Energy Cutoff are ignored during an EDS acquisition. Can be used to eliminate the "noise" peak in the low energy region. Also used to determine where the noise peak ends and the baseline begins, which helps determine light elements such as the Be peak.
		These options are available:
		• 100. Clips the noise peak in the spectrum
		• Zero. Includes the noise peak
		• Auto. Adjusts the energy value according to the specified Time Constant. Each Time Constant setting has a unique noise level. In general, Auto includes about 3 channels of noise in the spectrum. Each time constant has a unique noise level.

# Table 15. Spectral Imaging Mode Experiment Settings, Normal View

Group	Setting	Description
	High Energy Cutoff	X-ray counts below this limit are included in an EDS acquisition. Typically, a High Energy Cutoff setting of 20 keV will include an X-ray line from all elements using an SEM. For TEM applications, 40 keV is appropriate. The Auto option changes the energy limit according to the microscope kV.
Time Constant		The amount of time the software integrates the energy during the measured portion of the peak. Consider these options:
		• Rates 1 and 2 produce statistically good, high-resolution spectra
		• Rate 3 and Rate 4 produce quality spectra for most analyses
		• Auto adjusts the time constant to keep the dead time between 30% and 50%. Depends on the beam current and accelerating voltage.
		<b>Note</b> : Do not use the Auto setting for spectral comparisons or quantification with standards.
		To see the time associated with each Rate setting, add Time Constant to the Pathfinder <u>status bar</u> .
Custom	Save Custom Settings 1 Save Custom Settings 2	Allows you to save up to two instances of the Experiment Setup screen for spectral acquisitions (C1 and C2) that can be easily recalled later by clicking the <u>Setting Presets</u> button on the <u>Acquisitions toolbar</u> and choosing an option from the list box.

 Table 15.
 Spectral Imaging Mode Experiment Settings, Normal View

# **Experiment Setup (Advanced View)**

To access the spectral acquisition settings, click the Experiment Setup button in the right side of the acquisitions toolbar. For advanced view, click to toggle the green arrow so it looks like this:

Microanalysis Feature Sizing				
	Experiment Setup			
Spectrum Point ID Spectral Imaging Linescan WDS				
💼 🛧 Get Image 🖵 🔒 🏟 🔯 Start Map 🖵 🖓 🔅 🍸				
Spectral Image Setup	Advanced View toggle setting			
Active Detector(s)  Detector One  Detector Two				
SI Acquisition				
Resolution: 512x384				
Frame Time (s): 50.0 - Dwell Time (µs): 254				
Number of Frames: 500 - Acquisition Time (s): 25000				
Energy Range				
Time Constant : Auto  v (projected max throughput)				
Custom				
Terminate by Statistics				
Average Counts per Pixel Limit: No Limit 👻				
Element Map Vertical Full Scale: No Limit 👻				
Element: 💌 Line: K 🗸				
Options				
Concurrent Image				
Detects /s:				
Stores /s:				
Dead Time %:				

The following spectral acquisition settings are available in advanced view.

Table 16. Spectrum Mode Experiment Settings, Advanced View

Group	Setting	Description
Terminate by Statistics	Average Counts per Pixel Limit	Terminates the acquisition when the average total X-ray counts in a pixel for all maps reaches this value.
	Element Map Vertical Full Scale	Terminates the acquisition when the vertical full scale of the accumulated spectrum reaches this value.
	Element	To define termination criteria based on max counts in the energy region for a particular element, enter the atomic symbol for the element and select an X-ray line (see below).
	Line	If you entered an element (see above), select the X-ray line for that element.
Options	Concurrent Image	Acquires a gray level image at the same time as the maps. This image defaults to single color and is displayed in the map view pane at the same resolution as the maps.
Readouts	Detects (/s)	Shows the number of X-rays entering the EDS detector per second.
	Stores (/s)	Shows the number of X-ray counts that are converted, pass through the noise discriminator and are stored in the spectrum per second.
	Dead Time (%)	Shows the percentage of dead time for the current measurement settings. Dead time refers to the time the pulse processor is busy and cannot process another X-ray event. The higher the Time Constant setting (where Rate1 is the highest), the higher is the dead time for a given count rate.
		A dead time of 25-30% is acceptable for a typical X-ray analysis.

# Acquire and Analyze a Data Set

This section provides an overview of the steps to perform a spectral imaging acquisition and element identification and quantification.

### ✤ To acquire and analyze a data set

1. Prepare the sample.

Place the sample in the microscope and obtain an image. Adjust the stage height (Z-axis) to the optimal working distance for the EDS detector. Obtain a good microscope image of an area of interest on the sample. See <u>Microscope Considerations</u> for details.

2. Fully insert the EDS detector.

3. Start Pathfinder software.

The Pathfinder Project Explorer window is displayed.

4. Select or create a project and choose OK.

The Pathfinder Home screen is displayed.

- 5. Set the basic microscope parameters:
  - From the Home screen, choose **Edit > Microscope Parameters**
  - Set these microscope parameters and then choose Close
    - Accelerating Voltage in kilovolts (kV)
    - Magnification
    - Working Distance in millimeters (mm)

Microscopic Parameters and Status Bar Selection				
Operating Parameter	Value			
✓ <u>A</u> ccelerating Voltage (KV)	15.000			
Magnification	1000			
Beam <u>C</u> urrent (nA)	0.000			
Working Distance (mm)	15.000			

**Note** If your SEM has Column Communications, these parameters are entered automatically.

- 6. Focus the sample (it should be in critical focus).
- 7. Click the Spectral Imaging icon second on the <u>analytical mode toolbar</u>.
- 8. Enter an experiment name (used as a base name for automatically saving acquired data).

On the acquisitions toolbar, click the 🛄 button and enter the experiment name (or leave the default name).

	Experiment Name	x	Start M
	Base name	ОК	
Ma			

9. Acquire an averaged electron image to use as the reference image.

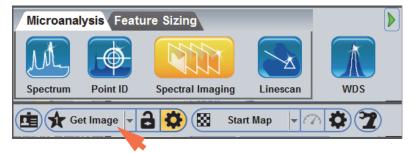
Click the Experiment Setup button at the right of the Get Image group on the acquisitions toolbar (normal view toggle setting) to display the available image acquisition settings.

Microanal	ysis Featu	ire Sizing		
Spectrum	Point ID	Spectral Imaging	Linescan	WDS
	t Image 🔻		t Map 🚽 C	

Use these settings in the Averaged Acquisition group:

Feature	Setting
Resolution	512x384
Frame Time	5 seconds
Number of Frames	1

To start the image acquisition, click Get Image on the acquisitions toolbar.



The button name changes to Stop Image. Wait until the acquisition is completed.

10. Acquire the X-ray data.

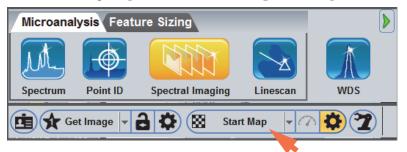
Click the Experiment Setup 😥 button at the right of the Start Map group on the acquisitions toolbar (normal view toggle setting) to display the available map acquisition settings.



Use these settings in the Averaged Acquisition group:

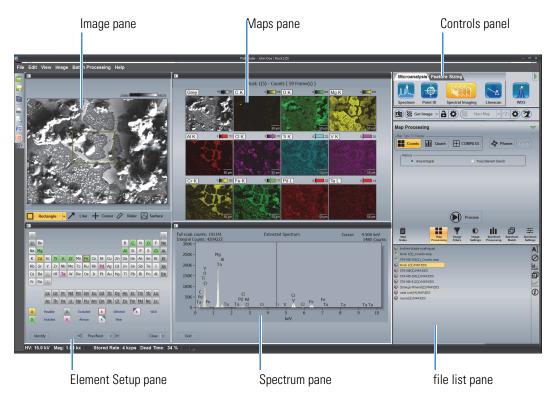
Feature	Setting
Resolution	256x192
Frame Time	20 seconds
Number of Frames	200
Low Energy Cutoff	100
High Energy Cutoff	Auto
Time Constant	Rate 3

To start the image acquisition, click **Start Map** on the acquisitions toolbar.



The button name changes to Stop Map. Wait until the acquisition is completed.

The cumulative spectrum from all pixels in the electron image is displayed in the spectrum pane. An Auto ID is performed periodically during the acquisition and X-ray maps are periodically updated in the maps pane. Here is an example:



When the acquisition is complete, the data set is automatically stored in the temporary directory and is ready for analysis. From here you can perform extractions to view spectral information or create different map sets and extract line scan data.

11. To save the data set in the current project, click the **Save** button **[]** on the main toolbar, or choose **File > Save**.

**Note** If you have the optional COMPASS software, during an acquisition you can switch to COMPASS mode and acquire principal component maps. To open COMPASS mode, select the **Map Processing** tab and click **COMPASS**. The component maps are updated and displayed periodically during the acquisition. You can also switch back to X-ray maps.

Map Processing	$\checkmark$
Map Type To Display Counts Quant	COMPASS Freeze
-Method	Spectral
-Background Calculate using data	Use Internal Model
Low energy cutoff (eV): 120	Number of Components: 0 (Auto)
Step Notes	

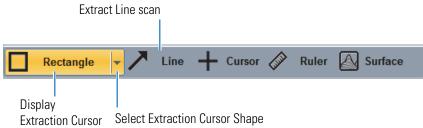
# **Extract and Analyze Data**

After the spectral imaging data set is acquired, use these tools to extract and analyze spectral data from specific areas of the electron image.

# **Extraction Tools**

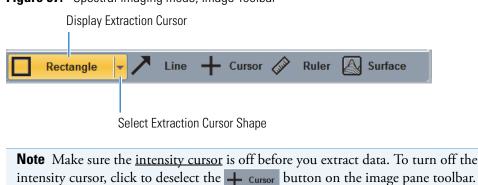
The toolbar at the bottom of the image pane contains buttons to specify an area of the electron image for analysis. Auto extraction is always selected in the software. This means X-ray maps are extracted automatically based on the elements identified from the extracted spectrum. The maps are extracted from the entire field of view; whereas the displayed cumulative spectrum is only from the area defined by the extraction tool.





# **Extract and Analyze from Defined Points or Areas**

Use the extraction cursor on the image pane toolbar to extract and analyze X-ray counts from each selected point or area on the electron image. Select a cursor shape from the dropdown list box. Click the cursor button to turn extraction on and off.



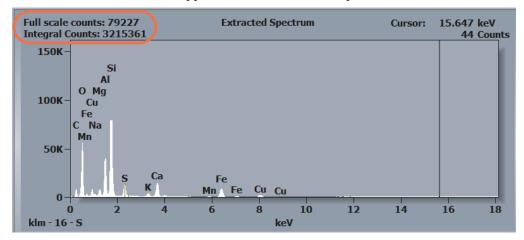
**Figure 57.** Spectral Imaging mode, Image Toolbar

When the extraction cursor button is selected on the image pane toolbar, the Map Processing tab opens on the controls panel with the Counts Map Type selected.

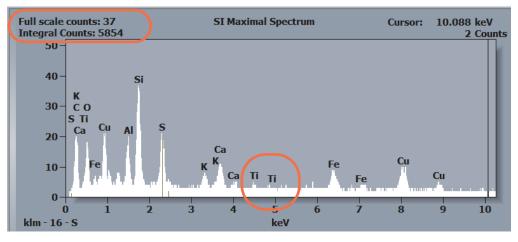
Map Processing						~
Counts	Quant	c	OMPASS	🏘 PI	hases	reeze
-Method						
<ul> <li>Area Integ</li> </ul>	gral		Trace	e Element Searc	h	
			Process			
Ш			Ö	- du	ā	-=
Step Notes	Map Processing	u Image Filters	Image Settings	Spectrum Processing	Spectrum Match	Spectrum Settings

There are two algorithms for extracting X-ray counts:

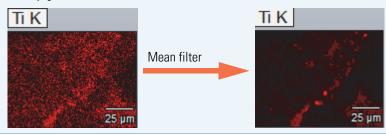
• Area Integral (default). Extracts the spectral data and displays total X-ray counts from the selected area. Useful for most applications. Here is an example:



• **Trace Element Search**. Extracts the spectral data and reports the maximum counts for each channel in the selected area. Useful for finding small concentrations of an element, or small, localized areas of an element that would otherwise be lost in the total background. Here is the sample processed using the Trace Element Search algorithm:



**Note** Notice that Ti shows up when the trace element algorithm is used but not for the area integral algorithm. If we filter out the noise from the Ti map, this element is clearly present.

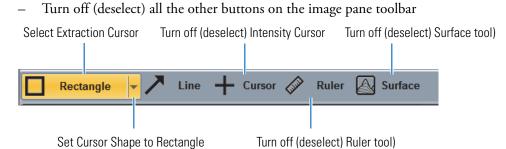


# **Single Pixel**

Extract from a single pixel in the electron image. This is useful for evaluating noise in the spectral data.

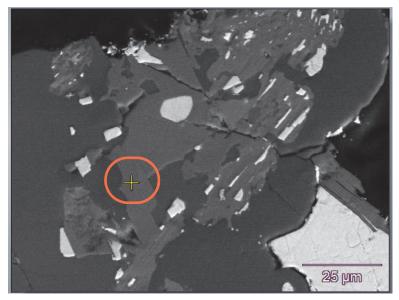
## **\*** To extract and analyze data from a single pixel

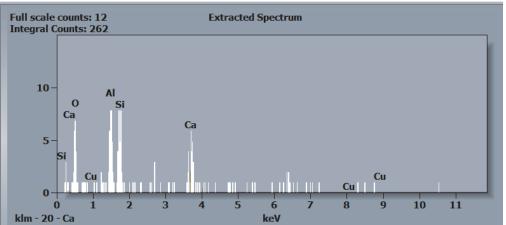
- 1. Use these image pane toolbar settings:
  - Select the extraction cursor
  - Set Cursor Shape to Rectangle



2. Click the desired location on the electron image.

The location is marked with a plus (+) sign on the electron image and the extracted spectrum is displayed in the spectrum pane. Here is an example:



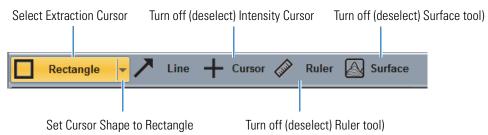


# Rectangle

Use the rectangle cursor shape to extract and analyze data from each defined area on an electron image.

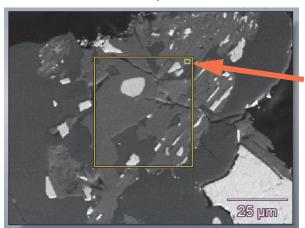
#### \* To extract and analyze data from each defined area on the electron image

- 1. Use these image pane toolbar settings:
  - Select the extraction cursor
  - Set Cursor Shape to Rectangle
  - Turn off (deselect) all the other buttons on the image pane toolbar



- 2. Move the mouse to a feature of interest on the electron image.
- 3. Click and drag to draw a rectangle around the area.

The extraction starts when you release the mouse button.



Click here to expand the shape to fill the image pane. Click again to return to the previous size and location

To move the shape, place the cursor inside the shape, then drag it to the new location

To resize the shape, drag any edge or corner

To delete the shape and redraw the area, drag a new rectangle over the image. The rectangular area can be resized and moved.

The software uses all pixels in the defined area to create a cumulative spectrum. The cumulative spectrum is displayed in the spectrum pane. The appearance of the spectrum and the identified elements are dependent upon the settings on the Map Processing tab on the controls panel. X-ray maps are extracted from the entire image.

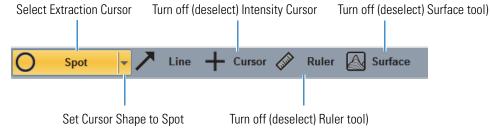
# Spot

Use the Spot cursor shape to extract and analyze data from each defined spot on an electron image.

## \* To extract and analyze data from each defined spot on the electron image

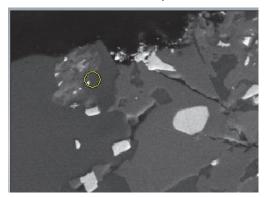
1. Use these image pane toolbar settings:

- Select the extraction cursor
- Set Cursor Shape to Spot (a predefined circle is displayed on the electron image)
- Turn off (deselect) all the other buttons on the image pane toolbar



- 2. Move the mouse to a feature of interest on the electron image.
- 3. Click to center the spot over the area.

The extraction starts when you release the mouse button.



The software uses all pixels in the defined area to create a cumulative spectrum. The cumulative spectrum is displayed in the spectrum pane. The appearance of the spectrum and the identified elements are dependent upon the settings on the Map Processing tab on the controls panel. X-ray maps are extracted from the entire image.

## ✤ To change the spot size

- 1. From the Home screen, choose **Edit** (menu) > **Properties**.
- 2. In the Properties box, click the Image Extract tab.

3. In the Spot group, enter the Radius for the spot size (as a percent of the image) and choose **Apply** to see the new spot size on in the image pane.

Properties	? 🗙
Image View       Image Extract       Linescan Overlay       Map Colors       Map Filters         Line       Thickness (% of image):       2       •         Thickness for last extract:       2       •         Number of Points:       5       •         Spot       Radius (% of image):       15	
Spectrum Type Extract Image Size: 512 x 384 pixels	
OK Cancel Apply	

4. Click **OK** to keep the new setting and close the Properties box.

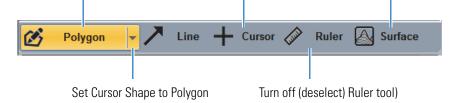
# Polygon (Free-Hand Shape)

Use the polygon cursor shape to extract data from a user-defined area on an electron image. The Polygon shape allows you to free-hand draw around an irregular-shaped object to extract from that object.

## \* To extract and analyze data from each user-defined area on the electron image

- 1. Use these image pane toolbar settings:
  - Select the extraction cursor
  - Set **Cursor Shape** to Polygon
  - Turn off (deselect) all the other buttons on the image pane toolbar

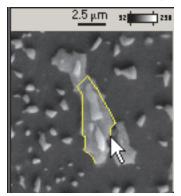
Select Extraction Cursor Turn off (deselect) Intensity Cursor Turn off (deselect) Surface tool)



2. Move the mouse to an area of interest on the electron image.

- 3. There are two ways to draw a shape:
  - Click the image at the points where you want to begin your shape. Hold down the left mouse button and draw a closed shape around the object of interest.
  - Single-click a corner of the shape and then continue to click adjacent corners to draw a boundary around the area.
- 4. Right-click to complete the polygon (it turns an opaque color).

The extraction starts when you release the mouse button.



To delete the shape and redraw the area, turn the cursor shape button off and back on. Then draw a new polygon over the image.

To draw multiple polygon shapes, hold down the Ctrl key on the keyboard while you draw all the shapes. Press the mouse button and draw the first shape. Then release the mouse button, move to a new location, press the mouse button again and draw the next shape. Right-click the image pane to close and fill all the drawn shapes.

The software uses all pixels in the defined area to create a cumulative spectrum. The cumulative spectrum is displayed in the spectrum pane. The appearance of the spectrum and the identified elements are dependent upon the settings on the Map Processing tab on the controls panel. X-ray maps are extracted from the entire image.

# **Extract and Analyze From Region Defined by Pixel Intensities**

Use the Magic Wand cursor shape to select an irregularly-shaped area on the electron image based on grey-level intensity values. This is similar to drawing a polygon but it uses intensities within specified values to define the area. The magic wand tool is easier than hand drawing a polygon.

#### To extract and analyze data using pixel intensities on the electron image

- 1. Use these image pane toolbar settings:
  - Select the extraction cursor
  - Set **Cursor Shape** to Magic Wand

Select Extraction Cursor Turn off (deselect) Intensity Cursor Turn off (deselect) Surface tool) Magic Wand Line – Cursor 🖉 Ruler Surface Set Cursor Shape to Magic Wand Turn off (deselect) Ruler tool)

Turn off (deselect) all the other buttons on the image pane toolbar

The software displays the Image Intensity Selection box.

Image Intensity Selec	tion		
Cursor Diameter	10	% of image width	= 27.741 um
	📃 Diam	eter in microns	
Similarity 0	<	• 100	83
Smoothing	2	×	
Brightness	46546		
🗹 Spectrum au	to extract		Close

- 2. Click within an area of interest on the electron image.
- 3. Set Cursor Diameter to the diameter of your selection area around the tip of the mouse cursor as a percentage of the image, or select Diameter In Microns and enter a value in microns.

The cursor diameter is the size or number of pixels around the mouse pointer that will be evaluated.

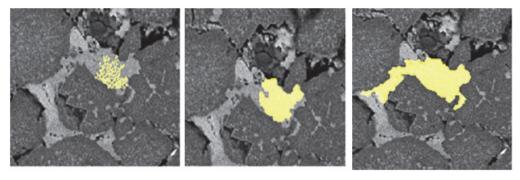
\_

#### 4. Adjust Similarity.

Start with a high similarity value and then gradually reduce it to include the intensities that select features of interest. A lower similarity value will include more pixels.

5. Set **Smoothing** to the number of passes for a smoothing function.

One or two passes will decrease the noisy pixels around the mouse cursor.



95% similarity 0 smooth 95% similarity 2 smooth 85% similarity 2 smooth

- 6. Adjust the Brightness value as needed.
- 7. Select **Spectrum Auto Extract** to automatically extract the spectrum after an intensity area is selected.
- 8. When you are finished defining the magic want area, choose **Close** to start the spectral extraction and analysis.

The software uses all pixels in the defined area to create a cumulative spectrum. The cumulative spectrum is displayed in the spectrum pane. The appearance of the spectrum and the identified elements are dependent upon the settings on the Map Processing tab on the controls panel. X-ray maps are extracted from the entire image.

Feature	Description
Cursor Diameter	Sets the size of the area (number of pixels) around the tip of the mouse pointer as a percentage of the image width to select pixel grey-level intensity (or in microns if Diameter in Microns is selected). The higher the percentage (or diameter) value, the more pixels are evaluated.
Diameter in Microns	Changes the Cursor Diameter entry box from percentage of image to diameter.
Similarity	Adjust the slide bar to include the intensities that select the object of interest.
Smoothing	Select the number of smoothing passes to fill in holes.

**Table 17.** Image Intensity Selection Box Features

Feature	Description
Brightness	This number is entered automatically based on the current mouse location. Adjust the value to change the image brightness.
Spectrum Auto Extract	Select this option to automatically extract and analyze the spectrum after an intensity area is selected.
	When Spectrum Auto Extract is cleared, the Extract Spectrum button is displayed. When you are finished defining the magic wand area, click Extract Spectrum to perform the extraction and analysis.

**Table 17.** Image Intensity Selection Box Features

# **Extract and Analyze Line Scan Data**

Line scan extraction allows you to investigate and analyze elemental transitions from region to region on the sample. You can specify the direction, width and number of points of the extracted area.

## ✤ To extract and analyze line scan data

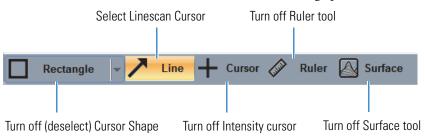
1. Select the elements to include in the line scan.

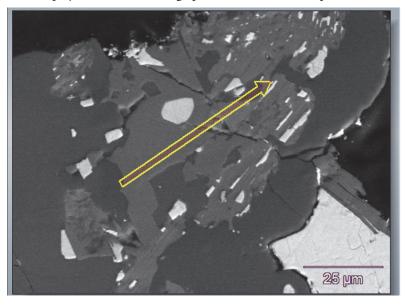
There are two ways to do this:

- In the element setup pane (periodic table), set individual elements to Identified
- To populate the periodic table automatically, select Auto ID in Ident Setup (controls panel > Spectrum Processing > Ident Setup) and use the rectangle cursor shape to extract a large area.

**Note** If you are unsure whether to include a particular element, include it in the initial line scan. You can exclude elements that are not of interest later.

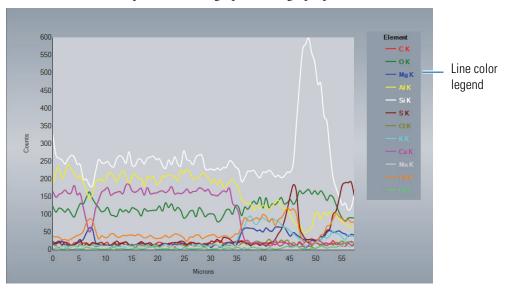
- 2. Use these image pane toolbar settings:
  - Select Line
  - Turn off (deselect) all the other buttons on the image pane toolbar





This displays a line in the image pane and extracts the spectral data along the line.

The line scan data are presented as a graph in the graph pane.



## ✤ To move the line

- To move the entire line, position the mouse cursor in the middle of the line. The cursor changes to a 4-way arrow. Hold the left mouse button and drag the line to a new location.
- To lengthen, shorten or redirect the line, position the mouse cursor at either end of the line. The cursor changes to a 2-headed arrow. Hold the left mouse button and drag the end to a new location.
- To draw a new line, move the mouse cursor to a new location and drag and drop a new line.

## **\*** To remove an element from the analysis

- 1. Right-click the element symbol in the element setup pane (periodic table) and choose **Inactive**.
- 2. Click the extraction line in the image pane to update the extraction.

# ✤ To add an element to the analysis

- 1. Right-click the element symbol in the element setup pane (periodic table) and choose **Identify**.
- 2. Click the extraction line in the image pane to update the extraction.

The element is added to the graph and legend.

## **\*** To change the number of points and width of the line scan

1. In the Linescan Processing group on the controls panel (opens automatically when you select Line on the image pane toolbar), use Number of Points in the Line group to change the number of points in the line scan.

Linescan Processing	~
Linescan Data Type Counts	
Line — Ihidkness (% of image): 2 ▼ Number of Points: 100 ▼	
-Data Options	

- 2. Enter a Thickness (% of image) value to change the width of the line scan.
- 3. Click the extraction line in the image pane to update the extraction.

# **Linescan Settings**

Use the features in the Linescan Processing group on the controls panel to define the line scan graph output. The Linescan Processing group opens automatically when you select the Line button on the image pane toolbar in the image pane.

Figure 58. Linescan Processing Tab

Linescan Processin	g	_	_	~
Linescan Data Type	Counts 🔻			
Line	2 🗸	Number of Points:	100	•
-Data Options	Smooth	3	Spline	
-Linescan Display	Symbols	Erro	r Bars	
Line Profiles	On Axis			
Overlayed	Stacked	_	_	
Step Notes	Linescan			age Spectrum ters Settings

The line scan settings are described below.

Table 18.         Linescan Processing Settings	Table 18.	Linescan	Processing	Settings
--	-----------	----------	------------	----------

Group	Setting	Description
Linescan Data Type		<ul> <li>Defines the format of the extracted quant data displayed in the line scan graph. These options are available:</li> <li>Counts. Number of X-rays received and processed by the detector.</li> <li>Atomic%. The atomic concentration of the element in the sample expressed as a percentage.</li> <li>Weight%. The mass of the quantified element in the sample expressed as a percentage.</li> <li>K-ratio%. Measured k-ratio (net counts sample/net counts from pure element) for the X-ray line.</li> <li>Net counts. Number of X-rays received and processed by the detector with background characteristics removed</li> </ul>
Line	Thickness	Width of the line scan expressed as a percentage of the electron image
	Number of Points	Divides the length of the line scan into sections where each point is a section. If you include enough points, each section is equivalent to one pixel. With lower Number of Points settings, the data in each section are summed.
Data Options	Log Scale	Displays the acquired data using a logarithmic scale
	Smooth	Applies the selected level of smoothing to the line scan graph
	Spline	Applies a spline function to the line scan graph
Linescan Display	Grid	Displays a grid in the line scan graph background
	Symbols	Marks each data point in the line scan graph with a symbol

Group	Setting	Description
	Error Bars	<ul> <li>When Linescan Data Type is set to one of these options, adds error bars to the graph to indicate the error level for the measured value:</li> <li>Atomic%</li> <li>Weight%</li> <li>K-ratio%</li> <li>Net counts</li> </ul>
Line Profiles	Line Profiles	<ul> <li>Determines how line scan data for selected elements are overlaid on the electron image. These options are available:</li> <li>On Linescan. Displays line scan data for the selected elements adjacent to the line scan.</li> <li>On Axis. Displays line scan data for the selected elements on the electron image axis.</li> <li>Overlaid. When line scan data for multiple elements are selected for overlay, all of the element line scans are overlaid on the electron image.</li> <li>Stacked. When line scan data for multiple elements are selected for overlay, the element line scans are stacked and scaled according to the scale setting (see below).</li> <li>Scaled Independently. When selected, scales each overlaid line</li> </ul>
		scan independently. When deselected, scales all overlaid line scans the same.

# Table 18. Linescan Processing Settings

# Examples:

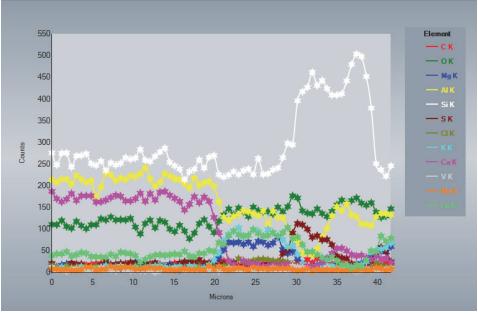
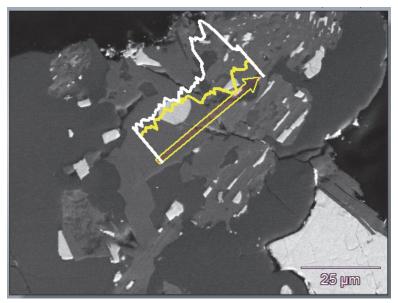


Figure 59. Linescan Display with Symbols Feature Selected

Figure 60. Linescan with Overlaid Element

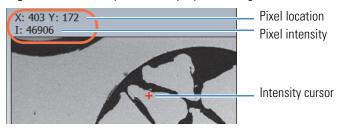


# **Analyze Line Scan Data**

# **Coordinated Intensity Cursors**

Click the **Intensity Cursor** button on the image pane toolbar to display the intensity cursor in the image pane. The cursor shows the pixel intensity and coordinates of the current cursor location in the upper left corner of the image pane.

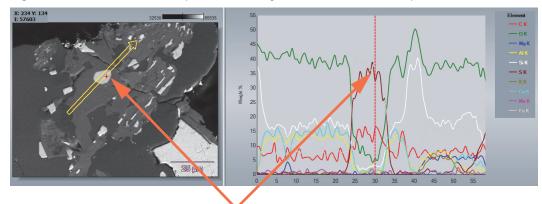
Figure 61. Intensity Cursor Displayed in Image Pane



You can click or drag the cursor to another location on the image to update the pixel intensity and location information.

If you click a point on a line scan in the image pane, a coordinated intensity cursor (vertical line) shows the location of that point on the line scan graph. You can also click points on the line scan graph to update the cursor location and information in the image pane.

Figure 62. Coordinated Intensity Cursors in Image Pane and Line Scan Graph

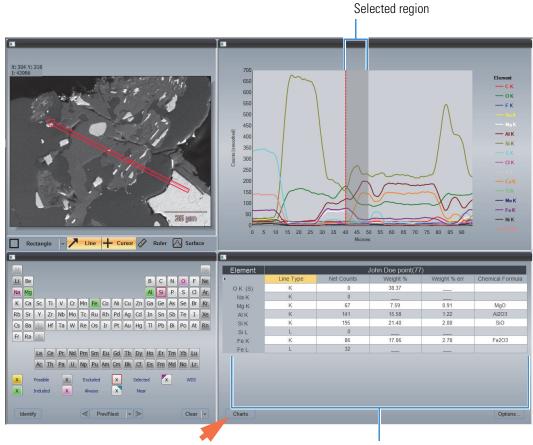


**Coordinated Intensity Cursors** 

# **Results Pane**

The results pane shows the quant data at the current cursor location. If you drag the cursor line right or left to select a region of the graph (gray area), the results pane shows the averaged results for the selected region. Here is an example:

Figure 63. Quant Results for Selected Region



Quant Results for Selected Region

• To display the spectrum for the current cursor location, click **Charts** (see above) and then click **Spectrum**.

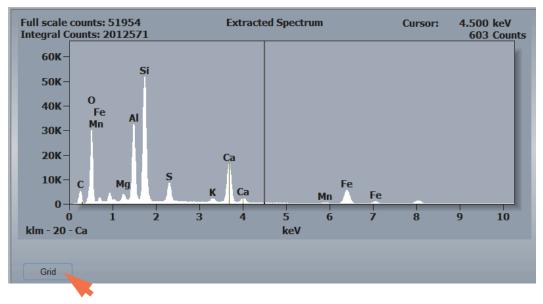


Figure 64. Results Pane with Spectrum Displayed

• To redisplay the quant results, click **Grid** (see above).

# **Compare Multiple Extracted Spectra**

To compare spectra from multiple extraction regions, extract and then manually save each spectrum file to the project folder. Perform the spectral comparison in Spectrum mode. See Compare Spectra for more information.

# **Specify Which Elements are Mapped**

- To specify which elements are mapped
- 1. Open or acquire a spectral imaging data set.

If Auto ID is selected, the software extracts the spectra from the electron image and identifies the found elements. A map for each found element is displayed in the maps pane. The cumulative spectrum from the electron image is displayed in the spectrum pane.

2. Perform an area extraction.

The software extracts the spectra in the selected area and identifies the found elements.

- 3. From the controls panel and select the Spectrum Processing tab.
- 4. Click Ident Setup and turn off (deselect) Auto ID.
- 5. Click Quant Setup and turn off (deselect) Auto Quant.

- 6. Right-click each element you want to analyze for and choose Identify.
- 7. Right-click each element you want to exclude from the analysis and choose Inactive.
- 8. Select the Map Processing tab and click the **Process** button.

Map Processing					~
Map Type To Display — Counts	Quant 🕂	COMPASS	🍂 P	hases	reeze
Method Area Integ	jral	Trace	e Element Searc	h	
	_				
		Process			
Ë	H Y	¢	ılı	Ø	-8
Step Notes	Map Image Processing Silters	Image Settings	Spectrum Processing	Spectrum Match	Spectrum Settings

The new map set is created and displayed in the maps pane. The cumulative spectrum for the selected area is displayed in the spectrum pane.

## \* To change which element X-ray line is mapped

**Note** The software automatically identifies which lines are used for mapping based on their associated elements and the accelerating voltage setting in <u>Microscope Settings</u>.

- 1. Right-click the element symbol in the periodic table.
- 2. Choose Mapping Lines and select a line type.

$\checkmark$	Included		Sn	Sb	Te	I	X
	Inactive				Po	At	R
	Excluded		P	-			
	Always Included				14		
	Mapping Lines	(	1		nes		
	Quant Lines	۲		LLi	nes		
	Acquire WDS	۲		Ali			
	Group			K+L	. Lines	;	
	Advanced				0	Clear	

3. Select the Map Processing tab and click the **Process** button.

#### To specify which energy range is mapped

- 1. Right-click the element symbol in the periodic table.
- 2. Choose Advanced.
- 3. In the Advanced Element box, specify the energy range in eV.

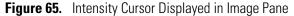
Advanced Element	
Quantification of Weight % Calculated By difference Fixed %	
Use Derivative References	
-Mapping Energy Range (eV) From: 2466 To: 2650	

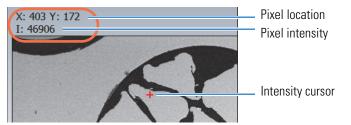
4. Select the Map Processing tab and click the **Process** button.

# **Quantify Map Data**

# **Coordinated Intensity Cursors**

Click the **Intensity Cursor** button on the image pane toolbar to display the intensity cursor in the image pane. The cursor shows the pixel intensity and X-Y coordinates (in pixels) of the current cursor location in the upper left corner of the image pane.





A coordinated intensity cursor shows the location of that point in every map in the maps pane. The X-Y coordinates (in pixels) of the current cursor location is displayed in the upper right corner of the maps pane.

# Figure 66. Coordinated Intensity Cursors in Image and Maps Panes

File Edit View Batch Processing Help

Current Cursor Location

Coordinated Intensity Cursors

**Note** If the resolution of the electron image is different from the resolutions of the element maps, the current cursor locations will be different since those locations are reported in pixels.

You can click points, or drag the cursor to another location, on the image or any map to update the pixel intensity and location information.

#### Note

- To change the colors of the intensity cursors, choose Image Settings on the controls panel and click Marker Colors.
- If the Intensity Cursor and the Cursor (Extraction) Shape are both selected, the Intensity Cursor takes preference. (Turn off (deselect) the Intensity Cursor when you are extracting spectra and generating maps.)

# **Quantify Selected Elements**

Follow these steps to specify quant settings and quantify the elements that are selected in the element setup pane (periodic table).

# ✤ To quantify the selected elements

1. On the controls panel, select the Map Processing tab and click Quant.

Get Image 🗸 🔒 🐼 🧾 Start Map 🗸 🏹	
Map Processing	~
Map Type To Display Counts Quant COMPASS Phases	Freeze

2. Set Map Data Type to the type of quant data to extract from the maps.

Get In	nage 🗸 👌 💭 🛛 Start Map 🗸 🏠 🤦
Map Processi	ng 🗸 🗸
Map Type To Display	Quant COMPASS Phases Freeze
Map Data Type	Atomic %
Threshold	Atomic % Weight % K-Ratio %
-Output Resolution	Net Counts

3. Clear the **Threshold** option for the initial quantitative analysis to quantify all the extracted element maps.

	💼 🛧 Get Image 🗸 🔒 🏟 🔯 Start Map 🛛 🗘 🏹
	Map Processing 🗸 🗸
	Map Type To Display Counts Quant COMPASS Freeze
	Map Data Type Weight %
	Threshold
2	-Output Resolution High Speed (Tiled Kernel)  Partial Shift Kernel (Full Shift Kernel)
	Processing Accuracy High Precision Kernel Size Auto
	Process
	Step Notes Map Processing Filters Stepsortum Notes Step

**Note** If the noise level of your data is high, you can set up a count or intensity based threshold to minimize it. See <u>Threshold</u> for more information.

- 4. Set **Output Resolution** to Partial Shift Kernel.
- 5. Set **Processing Accuracy** to High Precision.
- 6. Set Kernel Size to Auto.
- 7. To start the analysis, click the Process button on the Map Processing tab.

	I	1		
Rock	( 1(5) - Weight % ( 59	9 Frame(s) Kernel Size	= 5)	
Grey 109 255 C K	0 <b>—</b> 37 <mark>O K</mark>	0 Mg K	5 <b>5</b> 1 AI K	0
50 um	50 µm	50 µm	50 µm	50 µm
	0 📫 1 Ca K	0 💻 15 Ti K	0 📫 1 🗸 K	0 1
50 um	50 µm	<u>50 µт</u>	50 µm	50 µm
Cr K 1 33 Fe K		0 4		

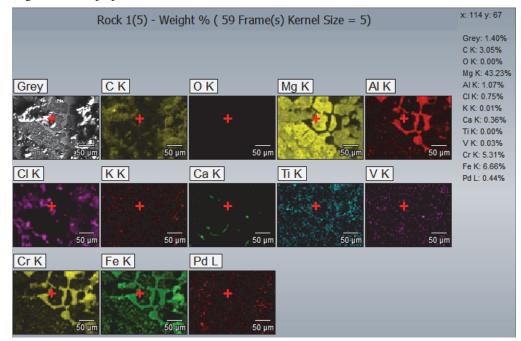
The quant results are displayed in the maps pane. The data type, number of frames and kernel size used are listed above the maps. Here is an example:

8. You can refine the analysis based on your knowledge of the sample by trying other <u>quant</u> settings and then clicking the **Process** button to reanalyze the data.

Keep in mind that the High Detail resolution setting takes longer to process.

9. To see the quant data, select the **Intensity Cursor** + <sup>Cursor</sup> on the image pane toolbar and click a point on the electron image or any extracted map.

The quant results for each element at the current cursor location are displayed at the right edge of the maps pane.



# **Map Processing Quant Settings**

To see the quant settings in Spectral Imaging mode, select the **Map Processing** tab on the controls panel and click the **Quant** button.



These options are available:

Setting	Description
Map Data Type	<ul> <li>Defines the format of the extracted quant data displayed in the maps pane. These options are available:</li> <li>Atomic%. Atomic concentration of the element in the sample expressed as a percentage.</li> <li>Weight%. Mass of the quantified element in the sample expressed as a percentage.</li> <li>K-Ratio%. Measured k-ratio (net counts sample/net count from pure element) for the X-ray line expressed as a percentage.</li> <li>Net Counts. Number of X-rays received and processed by the detector with background characteristics removed (useful for cleaning up noisy maps)</li> </ul>
Threshold	<ul> <li>Allows you to set a threshold value for the quantitative analysis.</li> <li>When Threshold is selected, you can enter threshold values for on <b>peak counts</b> or <b>pixel intensity</b> or both of those. To determine an appropriate peak count or intensity setting, perform a <u>single pixel extraction</u> at a representative location on the sample and assess the noise level on the spectrum.</li> <li>Clear the Threshold option to quantify all peaks and pixel intensities in the extracted element maps.</li> </ul>
Output Resolution	<ul> <li>Defines the resolution of the extracted element maps used for quantitative analysis. These options are available:</li> <li>High Speed. Uses tiled (adjacent) kernels with no overlap and provides the lowest level of detail.</li> <li>Partial Shift Kernel. Kernel areas are overlapped by less than half a kernel width (provides medium detail).</li> <li>High Detail. Kernel areas are overlapped by one pixel (provides the highest detail).</li> </ul>
Processing Accuracy	<ul> <li>Defines the accuracy of the quantitative analysis. These options are available:</li> <li>High Speed. Provides reasonable filter fit results at high processing rates (makes some assumptions about the data).</li> <li>High Precision. Provides the most accurate filter fit results at slower processing rates (no assumptions are made).</li> </ul>

Setting	Description
Kernel Size	<ul> <li>Divides the maps into squares based on pixels. The data in each section is summarized and used for quantitative analysis. These options are available:</li> <li>Auto. The software selects the optimal kernel size based on the total number of counts within the kernel.</li> <li>Manual (from 1x1 to 19x19).</li> </ul>
	Note: Kernel size affects the accuracy of the quantitative results; the larger the kernel size, the less detail will occur in the maps.
Process button	Runs a quantitative analysis on the displayed data using the current Map Processing settings. Displays the results in the maps pane.
	Select the <b>Intensity Cursor</b> — Cursor on the image pane toolbar to see the quant results for selected points on the electron image or element maps.

# **Overlay Maps and Electron Image**

You can overlay individual (or multiple) element maps (X-ray count, quant or principal component) on the electron image to see where found elements overlap visible features on the sample.

# ✤ To overlay an element map on the electron image

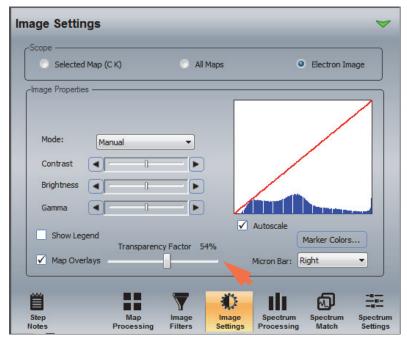
1. Click to select the element label above the map.

Here is a map of magnesium K line overlaid on the electron image.

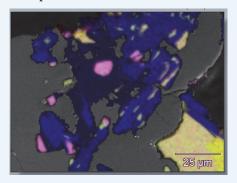
Mg map overlaid on electron image

Mg map selected for overlay

To adjust the transparency of the overlaid map, select the **Image Settings** tab on the controls panel, set **Scope** to Electron Image, and adjust the Transparency Factor slider bar.



**Note** To help identify elements that are found together, assign the related element maps primary colors. For example, when a red map and a green one are overlaid on the electron image, the areas where they overlap will be yellow. In the same way, red plus blue equals magenta; blue plus green equals teal; red plus green plus blue equal white. Here is an example:



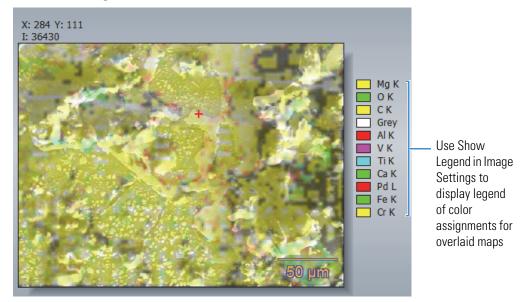
To assign map colors, select the Image Settings tab on the controls panel. See Adjust Map Type, Intensity and Color for more information.

#### ✤ To overlay all element maps on the electron image

- 1. Select the **Image Settings** tab on the controls panel.
- 2. On the Image Settings tab, set **Scope** to Electron Image.
- 3. Clear and then reselect Map Overlays.

Selected	Мар (СК)	All Maps		<ul> <li>Electron Image</li> </ul>
Image Properties	3			
				/
Mode:	Manual	•		
Contrast				/
Brightness				
Gamma				
Show Lege	end		<ul> <li>Autoscale</li> </ul>	Marker Colors
	and the second	ncy Factor 54%		
🖌 🖌 Map Overl	lays		Micron Bar	Right 🔻

Here is an example:



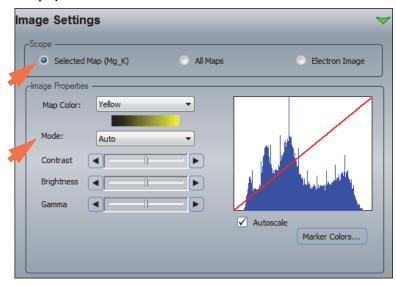
**Note** To display the legend of color assignments for the element quant maps, select **Show Legend** on the Image Settings tab:



# Adjust Map Type, Intensity and Color

## **Select Map Type**

Element maps (and the electron image) can be displayed as graduated maps or contour maps. All of the maps use color variations to show the locations and concentrations of elements on the sample. Use the **Scope** and **Mode** settings in **Image Settings** to select which type of maps to display.



Use Scope to select the map (electron image, all element maps, or a selected element map). Then use Mode to select the mode (Auto, Manual, Hot Pixel Suppression or Contour). All of these options are described below:

**Note** The software displays all maps with hot pixel suppression during acquisition. After that, it may switch to Auto if the maps contain sufficient data to produce good definition with a linear color ramp.

Map Mode options:

- Auto. Displays the selected map(s) or image as a single color with a linear ramp between the lowest and highest intensity values. Color intensity is directly related to X-ray intensity. Works well for electron images or element maps that have lots of statistical data (high number of X-ray counts).
- **Manual**. Lets you manually adjust brightness, contrast and gamma encoding for the electron image, a selected element map, or all displayed element maps. (If you change a Contrast, Brightness or Gamma setting, Mode automatically resets to Manual.)
- Hot Pixel Suppression. Displays the selected map(s) or image as a single color with a linear ramp that uses hot pixel suppression to assign intensity values. The pixel suppression algorithm eliminates unusually high intensity pixels that are typically due to random noise. The remaining pixel intensities represent X-ray counts that are due to the sample. It also expands the histogram to average the image across the full range of intensities. This helps ensure that pixels with the highest X-ray counts are set to a high intensity, and pixels with the lowest X-ray counts (i.e., due to noise) are set to a low intensity.
- **Contour**. Displays the selected element map with multiple colors where each color represents a step change in X-ray intensity. The relative spacing of the lines indicates the relative change in X-ray intensity. The Contour setting is available only when Scope is set to Selected Map.

## **Select Map Colors**

#### To change the color of a selected element map

- 1. Select the **Image Settings** tab on the controls panel.
- 2. Set **Scope** to Selected Map.
- 3. Click the element map in the maps pane to select the map you want to change.

Image Settings		-
Scope Selected Map (Mg_K) -Image Properties	All Maps	Electron Image
Map Color: Yellow Mode: Auto	<b>•</b>	
Contrast		
Gamma		Autoscale Marker Colors
		Marker Colors

The element symbol appears next to the "Selected Map" button in the Image Settings.

4. In the Image Properties group, set Map Color to the new color for the selected map.

The selected map changes to the new color in the maps pane.

5. Adjust the brightness, contrast and gamma settings as needed.

These settings affect only the selected map.

6. Select <u>Autoscale</u> to autoscale the histogram of the selected map.

## **Reset Colors for All Element Maps**

- ✤ To reset the colors for all displayed element maps
- 1. Select the Image Settings tab on the controls panel.
- 2. Set **Scope** to All Map.

#### 3. Click Reset Colors.

mage Settin	igs	4
Scope	Map (C K)   All Maps	C Electron Image
-Image Propertie	s ————	
	Reset Colors	
Mode:	Hot Pixel Suppression	
Contrast		
Brightness		
Gamma		
		Marker Colors

The software's default color scheme is applied to all displayed element maps.

## **Select Colors for Contour Maps**

We recommend using the default color palette for contour maps. If you want to change the palette for a contour map, follow these steps.

#### \* To change the color palette for a contour map

- 1. Select the **Image Settings** tab on the controls panel.
- 2. Set **Scope** to Selected Map.
- 3. Click the element map in the maps pane to select the map you want to change.

Image Settings	_	_	_	Click to select Advanced View
Selected Map (C K)	O All Maps		Electron Image	
Map Color: Contour Mode: Contour Contrast I I Brightness I I Gamma I		Autoscale	Marker Colors	

The element symbol appears next to the "Selected Map" button in the image settings.

4. In the Image Properties group, set **Mode** to Contour (see previous image).

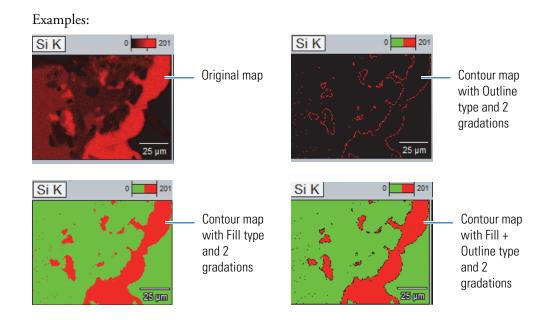
Map Color is automatically set to Contour, which is a custom color palette for contour maps.

- 5. Click the green arrow to select Advanced View (see previous image).
- 6. In the Contour Properties group, set Type, Gradations and High/Low.

-Contour Properties				
Type: Fill	✓ High:	279	279 Max	
Gradations: 2	Low:	0	0 Min	

These contour color options are available:

Feature	Setting
Туре	<ul> <li>Defines how contour areas are displayed. Available options:</li> <li>Outline. Contour areas are outlined with selected color.</li> <li>Fill. Contour areas are filled with selected color.</li> <li>Fill + Outline. Contour areas are outlined and filled with selected color.</li> </ul>
Gradations	Number of gradations (steps) used for the contour map where each step is a unique color. The higher the number of gradations, the finer the detail in the contour map.
High	Upper limit of intensity range that will appear in the contour map
Low	Lower limit of intensity range that will appear in the contour map



# **Image Filters**

Image filters can be used to improve the appearance of element maps and to selectively highlight specific information. The software offers a number of filtering techniques in Spectral Imaging mode. The best option depends on the image and how it will be used.

**Note** Image filters alter the appearance of displayed element maps but do not change the raw data.

Filtering uses a kernel with an odd numbered matrix array of MxM size (e.g., 3x3, 5x5, 7x7, etc.). For example, a 3x3 kernel looks like this:

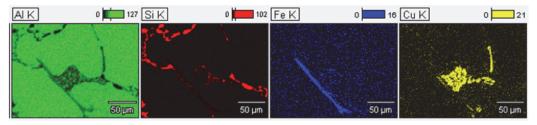
3 x 3kernel					
Α	В	С			
D	E	F			
G	н	I			

Filtering scales the pixel values that correspond with each kernel cell (A to I). The scaling factor for each cell is defined by the type of filter used (for example, the Sobel filter has different scaling factors than the Laplace filter). The results are always placed in the center pixel (cell "E" in the example above). The kernels are moved across the image at the full resolution of the image (i.e., one pixel at a time).

Using image filters to clean up element maps can facilitate interpretation. Here are some examples:

Figure 67. Raw Versus Filtered X-ray Maps

Raw X-ray Maps



Same Maps with 1 Pass or 3x3 Kernel, Mean Filter Applied

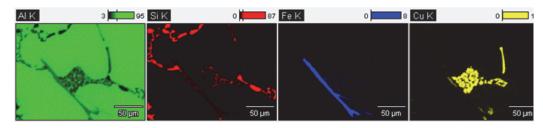
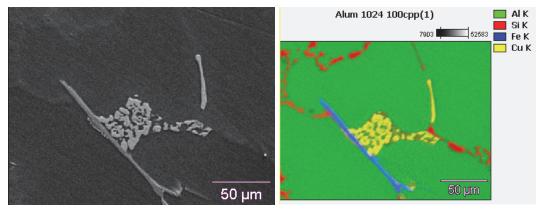


Figure 68. Overlay of Effectively Filtered X-ray Maps



**Note** Our software includes generally accepted filter types for processing image maps. Information about scaling factors and other setable parameters for these filters is available in any image processing text book or an equivalent resource.

## **Apply Image Filters**

Image filters can be used to enhance element maps that have low counts in the element distribution and are "noisy" due to background counts. For example, semi-conductors produce low element counts because they do not have a lot of material to fluoresce. Selecting a filter that lowers background but preserves edge definition can significantly improve the appearance of these types of maps.

#### \* To apply a filter on a single element map in Spectral Imaging mode

- 1. Select the Image Filters tab on the controls panel.
- 2. Select Use Filters.
- 3. On the Image Filters tab, set **Scope** to Selected Map.
- 4. Click the element map in the maps pane to select the map you want to apply a filter to.

The element symbol appears next to the "Selected Map" button in the image filters.

Image Filters	_	_	<b>~</b>	Click to select Advanced View
✓ Use Filters Scope ● Selected Map (O K)	i IA	Maps		
Mean		Selected Filters Mean		
Properties	3 •			
Normalize Output Negative Offset	0 🜩			
Update				

**Note** To apply image filters to all displayed element maps, set **Scope** to All Maps instead of Selected Map.

5. Select one or more <u>available filters</u> to apply by selecting the filter name in the list box and then clicking the right arrow **b** button to move it to the Selected Filters box (see the previous image).

We recommend that you start with a smoothing filter such as the **mean filter** and evaluate its effect on the element map before applying additional filters.

- 6. Change the <u>Kernel Size</u> and <u>Passes</u> if desired and click **Update** to adjust the effects of the selected filters.
- 7. Enter or select values for any other setable parameters for the selected filter.

For example, the Mean filter can be applied with or without <u>normalization</u> and <u>negative</u> <u>offset</u>.

The values are either 1 (on) or 0 (off).

# **Image Filter Settings**

Setting	Description	
Use Filters	Defines whether the filters listed in the Selected filters box have been applied to the selected element map or to all displayed element maps	
Scope	Specifies where the selected filters are applied (selected element map or all displayed element maps)	
Available Filters	List of industry standard filters available in the software. Select an available filter from the dropdown list and click the right arrow button to copy it to the Selected Filters list and apply it to the selected map(s).	
	<ul> <li>Filters fall into three general categories:</li> <li>Smoothing filters, which reduce noise but blur intensity level boundaries. Includes Mean, Adaptive Mean, Lowpass 1 or 2, Gaussian, Median, Rank, Erode, Dilate, SNN, K-N EPS, Extended MHN, Wiener.</li> <li>Sharpening filters, which enhance differences in intensity levels but increase noise. Includes Sharpen, Highpass 1, 2 or 3, Gated Rank, Min/Max.</li> <li>Edge filters, which smooth less effectively but preserve the divisions between intensity levels. Includes Sobel, Laplace 1 2, or 3, Horizontal or Vertical Derivative, Horizontal or Vertical Line, Positive or Negative Diagonal.</li> </ul>	
	If you add multiple filters to the list, use the up and down arrows to specify the order in which the filters are applied.	
Kernel Size	Divides each map into squares based on the specified odd number of pixels (e.g., 3x3, 5x5, 7x7, etc). This is the pattern used to apply the specified filter(s).	
Passes	Specifies the number of times the selected filters are applied	
Normalize Output	For mean filtering, set this option to 1 to normalize the filtered output. Normalization adjusts the histogram scaling using a predefined scaling factor based on the number of pixels.	
	Set Normalization Output to zero to turn off normalization.	

 Table 19.
 Image Filter Settings Available in Spectral Imaging Mode

Setting	Description
Negative Offset	For mean filtering, set this option to 1 to apply a negative offset to the data.
	Set Negative Offset to Zero to turn off the negative offset.
Update	Applies the current filter settings to the selected element map or to all displayed maps, depending on the <u>Scope</u> setting.

 Table 19.
 Image Filter Settings Available in Spectral Imaging Mode

# **Work With Spectral Imaging Files**

## **Open Spectral Imaging Files**

#### \* To open a spectral imaging file in the current project

1. Select the file in the file list pane.

This can be a CSI (.csi), MAP (.map), or EDS (.eds) filename extension.

#### \* To open a spectral imaging file from other project

- 1. From the Home screen, choose File (menu) > Open.
- 2. Navigate to the project that contains the file.
- 3. Select All Files to show all the available files in the folder.
- 4. Select the file click **Open**.

This can be a CSI (.csi) (full data) file or a noneditable MAP (.map) file that was previously saved. The data are displayed in the Spectral Imaging mode and take on the characteristics of the current project.

## **Compress Spectral Imaging Files**

Use this option to select a folder of acquired data for additional compression, resulting in file sizes that are reduced by a factor of 10. (All acquired data are partially compressed during acquisition.)

#### \* To reduce the size of a spectral imaging file

1. From the Home screen, choose File (menu) > Compress SI File.

**Note** If Compress SI File is unavailable in the File menu, all the files in the current project folder have already been compressed. This option is also unavailable during acquisition.

- 2. Select a folder and select one or more SI files to compress.
- 3. Click **OK**.

A progress dialog box appears. Compression time might take several minutes per file.

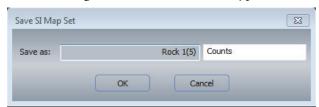
## **Save Element Maps**

Spectral imaging files that contain all the available data and information have an EDS (.eds) filename extension in the <u>file list pane</u>. You can also save just the map data that have been extracted or processed, or all the displayed element maps, to a MAP file (.map filename extension). This is helpful if you want map data in a specific format without having to extract it each time you open the file.

**Note** Once saved, the .map file is a fixed image of the final map state. It can no longer be processed or manipulated other than changing image filters or image settings such as brightness, contrast and gamma. If you want to apply more advanced processing steps, you will need to re-extract the original spectral imaging data set.

#### \* To save just the map data from a spectral imaging file

- 1. In Spectral Imaging mode, select a spectral imaging file that contains all the acquired data, such as an EDS (.eds) file.
- 2. Extract and configure the electron image (image pane) and element maps (maps pane) as you want them saved, including any map processing or image filters.
- 3. Choose File (menu) > Save Map File.
- 4. In the Save SI Map Set box, enter a two-part filename for the map file in the two edit boxes (the original SI filename and data type are the defaults).



5. Choose OK to close the Save box.

The map file appears in the file list pane with the entered filename and a MAP (.map) filename extension. Here is an example:



**Note** When you save maps, the software also creates the following files which are visible in the file list pane in <u>electron imaging mode</u> and in <u>Windows Explorer</u>.

• [Filename]\_[element name]\_Map.tif (one file for each element map).

# **Additional Tools**

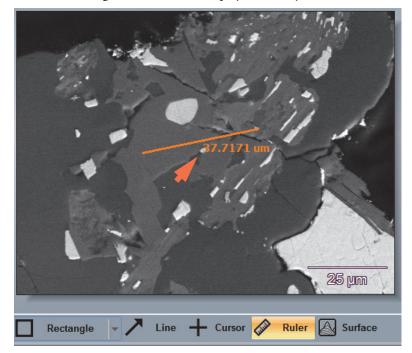
## **Measure Feature Sizes with the Ruler Tool**

Use the Ruler tool *Ruler* on the image pane toolbar to measure the size of features in the electron image.

#### \* To measure a feature in the electron image

1. Select the **Ruler tool** on the image pane toolbar to display the ruler in the image pane.

The current length of the ruler is displayed directly below it.



2. Drag the middle of the ruler to move it to the feature you want to measure. To lengthen or shorten the ruler, drag either end.

## **Display Topographical Images with the Surface Tool**

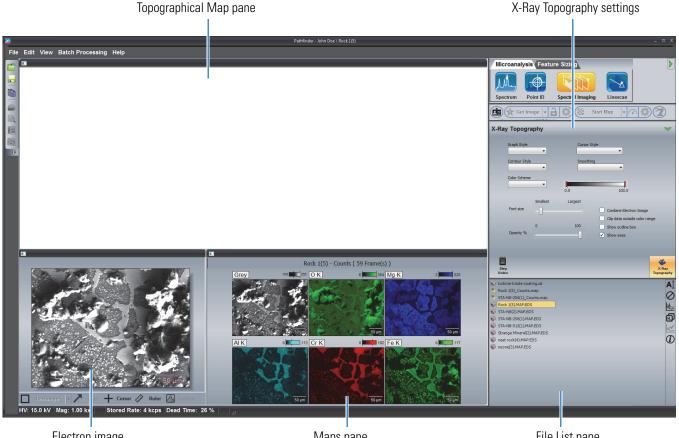
Use the Surface tool Surface on the image pane toolbar to create a topographical element map for the entire region displayed in the image pane. The topographical image shows the distribution of X-ray count values for the selected element over the measured sample surface.

#### **Display a Topographical Element Map**

#### \* To display a topographical element map

1. Select the **Surface tool** on the image pane toolbar.

The image pane and element map pane move to the bottom of the screen and a large topographical map pane appears. The X-Ray Topography settings are displayed on the controls panel.



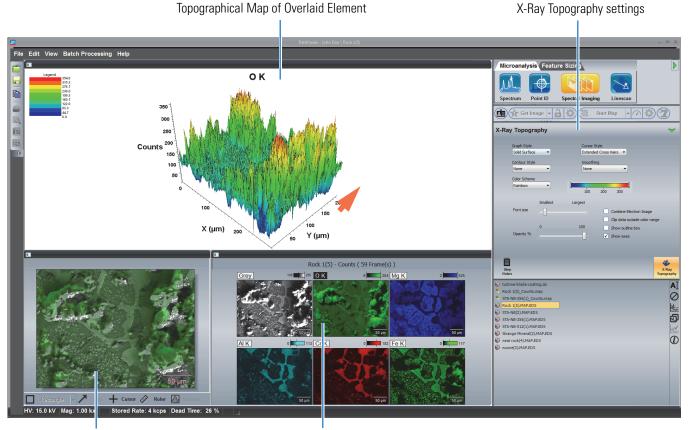
Electron image

Maps pane

File List pane

2. Select an element map by clicking it's symbol directly above the map.

The image pane shows the selected element map overlaid on the electron image. A topographical map for the selected element overlaid on the electron image appears in the topographical map pane. The X-Ray Topography settings are displayed on the controls panel.



Electron image with overlaid element map

Selected element

#### Change the Element Map Displayed in the Topographical Map Pane

#### \* To change the element map displayed in the topographical map pane

- 1. Click to deselect the symbol for the currently displayed element map.
- 2. Click to select the symbol for the new element map.

### X-Ray Topography Settings

Use the settings on the X-Ray Topography tab to specify the appearance of the displayed topographical map. These options are available:

#### Table 20. X-Ray Topography Settings in Spectral Imaging Mode

Setting	Description
Graph Style	Defines the overall appearance of the topographical graph. Available options:
	• <b>None</b> . Used for showing contour plots only. Here is an example:
	Sik Sik Sik Sik Sik Sik Sik Sik
	• <b>Solid Surface</b> . A warped, interpolated surface showing values at different locations on the sample. This is a conversion of the <u>contour</u> display to a three-dimensional form that makes it easier to see the values across the sample
	• Wireframe. Same as <u>Solid Surface</u> setting with black lines running along the intersecting edges of the imaginary plane of the surface plot but does not display the planes themselves.
Contour Style	Defines the appearance of the contour lines. Available options:
	• <b>Contour</b> . Uses contour lines to represent constant values. When Graph Style is set to Solid Surface, the contour lines are black. When Graph Style is set to Wireframe, values are indicated by the colors defined in the color bar (similar to the contour lines of a geographical relief map that indicate constant elevation).
	• <b>Profile (waterfall)</b> . Uses profile lines to represent constant Y-axis values. When Graph Style is set to Solid Surface, the profile lines are black. When Graph Style is set to Wireframe, the Z (vertical axis) values are indicated by the colors defined in the color bar.

Setting	Description
Color Scheme	Defines the color scheme for the topographical image. Available options:
	• <b>Gray Scale</b> . Uses shades of gray along a gradient to represen values.
	• <b>Rainbow</b> . Values are represented by a gradient of blended rainbow colors.
	• <b>Banded</b> . Values are represented by bands of distinct colors.
	• <b>Blue-to-red</b> . Values are represented by blended colors from blue to red.
Cursor Style	Specifies a style for the intensity cursor in the topographical map pane. (Use the image pane or map pane to select the cursor location.) Available options:
	• Wireframe Planes. Displays cursor as outlines of three imaginary orthogonal (perpendicular) planes that intersect at the cursor location. Perpendicular lines that pass through that location are also displayed.
	• <b>Extended Cross Hairs</b> . Displays cursor as perpendicular lines that pass through the cursor location along with extension lines that connect those lines to the axes.
Smoothing	Smooths the edges and blends color transitions in the topographical image.
	• <b>None</b> . No smoothing function is applied to the topographical image.
	• <b>Mesh</b> . Applies a smoothing function that emphasizes peak heights.
	• <b>Shading (Gouraud)</b> . Applies a (Gouraud) smoothing function that emphasizes shading.
Font Size	Increases or decreases the font size of axes markers in the topographical map pane.
Opacity %	Increases or decreases the opacity of the topographical image.
	Reducing the opacity makes high intensity peaks that are close together more translucent so you can see underlying peak shapes.

 Table 20.
 X-Ray Topography Settings in Spectral Imaging Mode

Setting	Description
Combine Electron Image	Creates a topographical grey scale image in which the grays match those in the electron image. Intensities are indicated by peak heights (Z values) rather than by colors.
Clip Data Outside Color Range	Clips peaks that are outside the color range of the selected <u>color</u> <u>scheme</u> .
Show Outline Box	Displays or hides the outline box in the topographical map pane. The outline box indicates the outer limits of the topographical image.
Show Axes	Displays or hides the X, Y, and Z axes in the topographical map pane.

 Table 20.
 X-Ray Topography Settings in Spectral Imaging Mode

# **Extract and Analyze Principal Components with the COMPASS Tool**

COMPASS is an optional software application that allows you to extract compositionally distinct components from a spectral imaging data set. COMPASS is a multivariate statistical analysis with no user input.

Principal Component Analysis (PCA) is a powerful technique to determine the relationship between variables within a data set. PCA uses advanced matrix algebra (linear transformations) to transform a number of possibly correlated variables into a smaller number of variables called principal components. The results are based on eigenvectors and eigenvalues. The eigenvector with the highest eigenvalue has the highest variance and is principal component number 1. The next highest eigenvalue is principal component number 2, and so on.

## **Map Processing COMPASS Settings**

**Map Processing** -Map Type To Display Counts -+-Phases Quant COMPASS -Method Area Spectral -Background Calculate using data Use Internal Model Low energy cutoff (eV): 120 Number of Components: 0 (Auto) 🔻 Process O ΠΓ 函 Step Notes Map Processing Spectrum Processing Spectrum Settings Image Filters Spectrur Match Image Settings

To see the COMPASS settings in Spectral Imaging mode, select the **Map Processing** tab on the controls panel and click **COMPASS**.

The following settings are available for performing a COMPASS analysis.

Setting	Description
Method	Specify a data set to evaluate. Available options:
	• Area (default). Weights the principal component analysis such that the pixels for a given component are generally located close together (i.e., weights by nearest neighbor). Recommended for most sample types.
	• <b>Spectral</b> . Does not add any nearest neighbor weighting. Intended for data sets that contain many small phases or when the spectral imaging resolution is such that the interaction volume is much smaller than the step size.
Background	Defines how the background is removed. Available options:
	• <b>Calculate Using Data</b> . Recommended for data sets that contain sufficient X-ray counts.
	• Use Internal Model. Recommended for data sets that have low numbers of X-ray counts.

Description
Allows you to remove the noise peak from the data set to remove the noise component from the analysis results by entering a low energy cutoff value in eV. (The noise peak is a component and is calculated along with the elements.)
Defines the number of expected components (minus the noise peak if the Low Energy Cutoff is set up to remove it from the data set). Available options:
• <b>0</b> (Auto). Recommended for the first pass of a COMPASS analysis. This allows the software to calculate the maximum number of eigenvectors (covariance pairs) in the data set.
• 1-15. You can decrease the number of found components to exclude components you don't need. For example, some components are similar and can be combined. You may also want to eliminate components that represent the interaction volume between two phases.
<b>Note</b> : You can also use the <u>Phases</u> and <u>image filter</u> tools to help with interpreting COMPASS maps.
Runs a COMPASS analysis on the displayed data using the current Map Processing settings. Displays the results in the maps pane.

#### Table 21. COMPASS Settings in Spectral Imaging Mode

## **Perform a COMPASS Analysis**

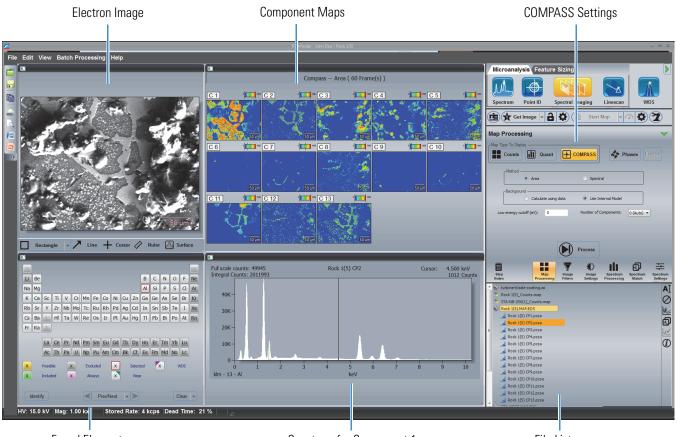
- \* To perform a COMPASS analysis on the selected elements
- 1. On the controls panel, select the Map Processing tab and click COMPASS.

Map Proce	Display —	Quant		OMPASS	n Pi	hases F	reeze
-Backg	• Ar	ea		Spectr	al		
		alculate using	data	Ose In	iternal Model		
Low energ	y cutoff (eV	): 120		Number of (	Components:	0 (Auto) 🔻	
				Process			
Ë			7	٢	ılı	Ð	
Step Notes		Map Processing	Image Filters	Image Settings	Spectrum Processing	Spectrum Match	Spectru Setting

- 2. Select the <u>method</u> and <u>background</u> options.
- 3. If you want to remove the noise component from the analysis, enter an appropriate <u>Low</u> <u>Energy Cutoff</u> value for the data set in eV.
- 4. Set Number Of Components to 0 (Auto) for the initial analysis.

#### 5. Click **Process** to start the analysis.

When the analysis is completed, the component maps are displayed in the maps pane. Here is an example:



Found Elements

Spectrum for Component 1

File List pane

Each component map represents a unique found component in the sample. The first component (C1) represents the elements with the smallest covariance value. The map labeled C2 represents elements with the next highest covariance, and so on. The spectrum displayed in the spectrum pane (CP1) is associated with the first component map (C1).

- 6. To display the spectrum for another component, click its map image in the maps pane.
- Reduce the Number Of Components setting if desired to eliminate unwanted found components, or adjust other settings as needed, and click **Process** to rerun the analysis.

## **Change Component Map Colors**

- To change the colors of the component maps
- 1. Select the **component map** you want to change by clicking its image.
- 2. Select the **Image Settings** tab on the controls panel.

3. In Image Settings, set **Scope** to Selected Map.

The map name appears next to the "Selected Map" button in the image settings.

Image Settir	ngs					~
Scope Selected	I Map (C 2)	🔘 All M	laps	6	Electron Im	nage
Image Propertie	35					
Map Color:	Earth	-				
Mode:	Hot Pixel Suppress	sion 🔻				
Contrast				/		
Brightness						
Gamma						
				Autoscale	Marker Colors	ş
Ë				. III	តា	-=
Step	Map	T Image	Image	Spectrum	Spectrum	-= Spectrum
Notes	Processing	Filters	Settings	Processing	Match	Settings

- 4. Set **Map Color** to the new color for the selected component map.
- 5. Adjust Mode, Brightness, Contrast or Gamma or Autoscale settings if needed.

## **Reset Color Palettes for All Component Maps**

- \* To reset the color palettes for all displayed component maps
- 1. Select the Image Settings tab on the controls panel.
- 2. Set **Scope** to All Map.

3. Click Reset Colors.

Image Settir	ngs		<b>^</b>
-Scope	i Map (C K)	All Maps	Electron Image
-Image Propertie Mode: Contrast Brightness	Reset (	pression	
Gamma			Marker Colors

The software's default color scheme is applied to all displayed component maps.

# **Extract and Analyze Phases with the Phases Tool**

The Phases tool is an optional software application that allows you to generate phase maps from X-ray count maps or COMPASS (component) maps. Phase maps are binary maps that indicate the locations of areas with similar composition.

## **Map Processing Phase Settings**

To see the phase settings in Spectral Imaging mode, select the **Map Processing** tab on the controls panel (Map Type To Display can be set to <u>Counts</u> or <u>COMPASS</u>), click the **Process** button to extract the maps and then click **Phases**.

Map Processing	_	~	
Map Type To Display Counts Quant	COMPASS	Phases Freeze	
	Combinations		
Phase Map Type Maximum Intensity X Phase Minimum Phase Area % 1.5	Name           Grey           ✓<		
Auto Manual	Image       Filters	Spectrum Processing	

**Note** After you have generated phase maps for a set of element maps (count maps or COMPASS maps), the Phases tab only allows you to combine selected phase maps. To regenerate the phase maps, first turn off (deselect) the Phases tab and click the **Process** button on the Map Processing tab to re-extract the element maps. Then click the Phases button to display the Phases tab shown above.

The following settings are available for performing a phase analysis.

Setting	Description
Phase Map Type	<ul> <li>Defines the type of phase map to generate. Available options:</li> <li>Maximum Intensity. Uses Maximum intensity values in each pixel of the X-ray lines to create phase maps. When this option is selected, the resulting phase maps look similar to <u>COMPASS</u> maps that have been extracted maps from the same sample, except the phase maps are binary. There are always the same number of found maximum intensity phases than found principal components.</li> <li>X Phase. Uses a proprietary algorithm to create phase maps (based on relative concentrations of the elements in the entire electron image). When this option is selected, the resulting phase maps may or may not resemble the COMPASS maps (There are typically more found phases than found components).</li> </ul>
Minimum Phase Area %	The software does not create phases that are smaller than the entered minimum phase percent value. If a phase falls below the entered value, it is combined with the nearest phase. A setting of 100% always produces one phase. A setting of 50% produces at most two phases.
Combinations	Use this box to select the element count maps (COUNTS) or component (COMPASS) maps used to generate phases.

 Table 22.
 Phase Settings in Spectral Imaging Mode

Setting	Description
Auto	Runs a phase analysis on the selected elements where the software identifies the phases based on peaks and valleys in the data. Results are displayed in the maps pane.
Manual	Allows you to manually set the thresholds used to generate phases for selected elements. Click <b>Manual</b> to set up the thresholds. In the Manual Phase Editor box, use the list box to select an element (Counts) or principal component (COMPASS) and then click the plus (+) button to add it to the setup box. Drag the vertical markers in the element spectrum to define low and high thresholds based on the displayed element map.
	C Manual Phase Editor
	Full scale counts: 2032
	C1 +-
	Segment Name     Low     High     Area %       C 1 Segment 1     100     1000     39.91
	Auto Segment Images         Auto Create Phases         Close

**Table 22.** Phase Settings in Spectral Imaging Mode

The Low and High Thresholds and Area % are updated automatically each time you move a marker. These additional options are available:

- Auto Segment Images. Automatically defines thresholds for all the element maps. May define multiple low/high threshold sets (segments) for each element.
- Auto Create Phases. Creates phase maps based on the elements and segments listed in the Manual Phase Editor box.

# **Extract Phases From X-ray Count Maps**

You can generate phase maps from X-ray count maps and combine selected phases. Select the element maps to include before you generate the phases. You can also include the low-resolution electron image.

#### ✤ To extract phases from X-ray count maps

- 1. Acquire a spectral imaging data set or open an existing spectral imaging file.
- 2. Select the Map Processing tab on the controls panel and click Counts.
- 3. Extract and analyze X-ray count maps for the elements selected in the element setup pane (periodic table).
- 4. Click **Phases** on the Map Processing tab.
- 5. In the Select Elements For Phase Analysis box, select the element maps to include.

Map Processing	<b>~</b>
Map Type To Display	COMPASS Freeze
	Select Elements For Phase Analysis
Phase Map Type	Name
<ul> <li>Maximum Intensity</li> </ul>	Grey
C X Phase	СК
	OK
Minimum Phase Area %	<ul> <li>✓ FK</li> <li>✓ MgK</li> </ul>
1.5	
	✓ CIK
	🔽 Ca K
Auto Manual	🖉 тік
	Cr K
	V Fe K

**Note** If you don't see the Phase tab settings shown above, turn off (deselect) the Phases tab and click the **Process** button on the Map Processing tab to re-extract the element maps. Then click the **Phases** button to display the Phases tab shown above.

6. Set the **<u>Phase Map Type</u>** and **<u>Minimum Phase Area%</u>** if desired.

7. Click Auto to start the analysis.

 ■
 FausSympledtile=BigMap127us30nAtate4DT31(2) (160 Frame(s))

 Phase1
 Phase2

 ●
 Phase3

 Phase3
 Phase4

 ●
 Phase3

 ●
 Phase3

 ●
 Phase3

 ●
 Phase4

 ●
 Phase3

 ●
 Phase3

The phase maps are generated and displayed in the maps pane. Here is an example:

8. To combine phases, select the phases you want to combine in the Combinations box.

Hold down the Ctrl key on the keyboard to select additional phases.

Map Processing Map Type To Display Counts Quant	Combinat	DMPASS ions	)	Phases Freez	<b>*</b> e	
Combine selected phases into this Phase:	Name Phase1 Phase2	Color Custom Custom	Area % 16.72 79.97	Combinations O K Mg K	]	Combine these
Combine	Phase3	Custom	3.31	Cr K		phases

9. In the Combine Selected Phases Into This Phase box, select the phase to combine into (default is the first phase selected).

#### 10. Click Combine.

The phases maps are updated with the selected phases combined.

**Note** The phase maps are stored in the project's temp folder and are moved to the project folder when the data are saved. This allows you to switch between COMPASS phase maps and X-ray count phase maps for comparison.

# **Extract Phases From COMPASS Maps**

You can generate phase maps from component (COMPASS) maps and combine selected phases. Select the component maps to include before you generate the phases.

#### \* To extract phases from principal component maps

- 1. Acquire a spectral imaging data set or open an existing spectral imaging file.
- 2. Select the Map Processing tab on the controls panel and click Counts.
- 3. Extract and analyze X-ray count maps for the elements selected in the element setup pane (periodic table).
- 4. On the Map Processing tab, click COMPASS.
- 5. Adjust the COMPASS settings as needed and click Process to extract and analyze component maps for the selected elements.
- 6. Click Phases on the Map Processing tab.
- 7. In the Select Elements For Phase Analysis box, select the component maps to include.

Map Processing		<b>\</b>		
Map Type To Display Counts Quant	COMPASS Phases Fr	eeze		
Select Elements For Phase Analysis				
-Phase Map Type	Name	*		
Maximum Intensity	V C1			
🔿 X Phase	✓ C2			
	<ul> <li>✓ C3</li> <li>✓ C4</li> </ul>			
Minimum Phase Area %	▼ C4 ▼ C5	E		
1.5	✓ C6			
	▼ C7			
	✓ C8			
Auto Manual	▼ C9			
	<ul> <li>✓ C 10</li> <li>✓ C 11</li> </ul>	- 1		
	▼ C 12	-		

**Note** If you don't see the Phase tab settings shown above, turn off (deselect) the Phases tab and click the **Process** button on the Map Processing tab to re-extract the element maps. Then click the **Phases** button to display the Phases tab shown above.

- 8. Set the Phase Map Type and Minimum Phase Area% if desired.
- 9. Click **Auto** to start the analysis.

The phase maps are generated and displayed in the maps pane.

10. To combine phases, select the phases you want to combine in the Combinations box.

Hold down the Ctrl key on the keyboard to select additional phases.

- 11. In the Combine Selected Phases Into This Phase box, select the phase to combine into (default is the first phase selected).
- 12. Click Combine.

The phases maps are updated with the selected phases combined.

**Note** The phase maps are stored in the project's temp folder and are moved to the project folder when the data are saved. This allows you to switch between COMPASS phase maps and X-ray count phase maps for comparison.

#### 6 Spectral Imaging Mode

Extract and Analyze Phases with the Phases Tool

# 7

# **Linescan Mode**

This section covers Linescan mode, which can be used to acquire X-ray spectra at each point on a user-defined line drawn on an acquired <u>electron image</u>. Line scan data can provide detailed information about the transitions between distinct features on a sample. For example, line scan analysis is sometimes used to evaluate the integrity of a coating material or to investigate the migration of a coating into the base material and vice versa.

Acquired line scan data can be easily reprocessed and displayed in different formats.

# **Select Linescan Mode**

To select Linescan mode, click the Linescan button on the analytical mode toolbar.

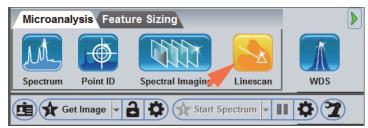
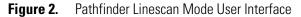


Figure 1. Select Linescan mode

In Linescan mode, the software displays all the features and tools for X-ray count analysis of the transitions in a user-defined axis across the selected microscope field of view.



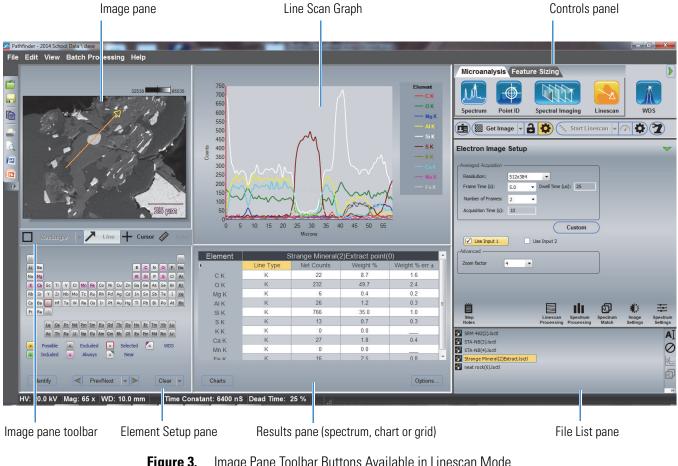
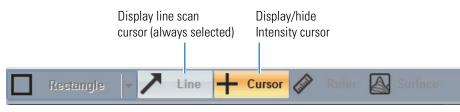


Figure 3. Image Pane Toolbar Buttons Available in Linescan Mode



# **Overview of X-ray Line Scan Acquisition**

The basic steps to set up and acquire X-ray line scan data are outlined below. All of these steps are described in more detail in the sections that follow.

**Note** The sample composition is assumed to be known. If the sample elements are unknown, start with a spectral analysis (<u>Spectrum mode</u>) or spectral imaging analysis (<u>Spectral Imaging mode</u>) to determine the sample composition before proceeding with the line scan analysis.

#### \* Basic steps to perform a line scan analysis

- 1. Start Pathfinder software and create or select a project.
- 2. Select the Linescan analytical mode.
- 3. Set the basic microscope parameters.
- 4. Prepare the sample.
- 5. Enter an experiment name.
- 6. Acquire an averaged electron image to use as the reference image.
- 7. Set the line scan acquisition and processing parameters.
- 8. Set the known elements to Always Identified in the periodic table.
- 9. Acquire and save the line scan data.

# **Line Scan Spectral Acquisition Settings**

To access the spectral acquisition settings for Linescan mode, click the Experiment Setup button on the right side of the acquisitions toolbar.

Microanalysis Feature Sizing				
Spectrum Point ID Spectral Imaging Linescan	Linescan Acquisition Settings			
Get Image - 2 0 1. Start Linescan - 7 0.2				
Linescan Setup  Active Detector(s)  Detector One Detector Two	~			
Linescan       Number of Points in Scan:       10       Number of Scans:       1				
Dwell Time per Pixel (s): 1.00				
Acquisition Time (s): 75 Total Line Time (s): 75				
Options				
Custom				

The following spectral acquisition settings are available.

**Table 1.** Linescan Mode, Spectral Acquisition Settings

Group	Setting	Description
Active Detector(s)	Detector One/ Detector Two	Available only when multiple Pathfinder- compatible detectors are installed on the microscope. When multiple detectors are installed, use the Detector One/Detector Two buttons to select which detector(s) you want to use for the analysis.
		<b>Note</b> : The Detector 1 and Detector 2 settings in the software refer to the detector cables attached to the Detector 1 and Detector 2 Preamp ports on the Pathfinder chassis.
Linescan	Number of Points in Scan	Defines the number of measured points in the line scan (typically at least one point per micron).
		<b>Note</b> : The extract properties for line scan points apply only to <u>Spectral Imaging Line Scans</u> and do not affect the acquired number of points in X-ray Linescan mode.

Group	Setting	Description
	Number Of Scans	The number of times the electron beam scans the line. To increase element intensities, acquire more scans. For sensitive samples, increase the scan count and reduce the dwell time.
	Dwell Time Per Pixel	The length of time the beam remains at each measured location in seconds. A long dwell time reduces image noise but increases the acquisition time. A short dwell time might cause a blurred or streaked image.
		For sensitive samples, increase the scan count and reduce the dwell time.
	Acquisition Time	Estimated total time in seconds to acquire all requested scans of the drawn line
	Total Line Time	Estimated time in seconds to acquire one scan of the drawn line
Options	Acquire Video	Acquires a grey-scale intensity profile concurrently with each line scan. The profile provides a reference intensity for each line scan point.
	Scan Stage	When the Stage Automation option is installed, selecting this option moves the microscope stage to generate the line scan, resulting in a higher resolution scan. Typically used for WDS acquisitions to ensure the WDS stays in focus throughout the line scan.
		When this option is cleared, the software moves the electron beam to generate the line scan. This is the faster option.
Custom	Save Custom Settings 1 Save Custom Settings 2	Allows you to save up to two instances of the Experiment Setup screen for spectral acquisitions (C1 and C2) that can be easily recalled later by clicking the <u>Setting Presets</u> button on the <u>Acquisitions toolbar</u> and choosing an option from the list box.

#### **Table 1.** Linescan Mode, Spectral Acquisition Settings

# **Linescan Processing Settings**

Inescan Process	Counts		
	Number of Points:	-	
-Data Options	Smooth 3 Spi	ine	
Linescan Display	Symbols Error Bars		
-Line Profiles	On Axis		
	<ul> <li>On Axis</li> <li>Stacked</li> </ul>		

To access the processing settings for Linescan mode, click the Linescan Processing tab on the controls panel.

The following Linescan processing settings are available.

Table 2.	Linescan Mode, I	linescan	Processing	Settings
----------	------------------	----------	------------	----------

Group	Setting	Description
Linescan Data Type	Linescan Data Type	<ul> <li>Defines the format of the extracted quant data displayed in the line scan graph. These options are available:</li> <li>Counts. Number of X-rays received and processed by the detector.</li> <li>Atomic%. The atomic concentration of the element in the sample expressed as a percentage.</li> <li>Weight%. The mass of the quantified element in the sample expressed as a percentage.</li> <li>K-ratio%. Measured k-ratio (net counts sample/net counts from pure element) for the X-ray line.</li> <li>Net counts. Number of X-rays received and processed by the detector with background characteristics removed</li> </ul>
Number Of Points	Number Of Points	Defines the number of measured points in the line scan (typically at least one point per micron).
Data Options	Log Scale	Displays the acquired data using a logarithmic scale
	Smooth	Applies the selected level of smoothing to the line scan graph

Group	Setting	Description
	Spline	Applies a spline function to the line scan graph
Linescan Display	Grid	Displays a grid in the line scan graph background
	Symbols	Marks each data point in the line scan graph with a symbol
	Error Bars	<ul> <li>When Linescan Data Type is set to one of these options, adds error bars to the graph to indicate the error level for the measured value:</li> <li>Atomic%</li> <li>Weight%</li> <li>K-ratio%</li> <li>Net counts</li> </ul>
Line Profiles	Line Profiles	<ul> <li>Determines how line scan data for selected elements are overlaid on the electron image. These options are available:</li> <li>On Linescan. Displays line scan data for the selected elements adjacent to the line scan.</li> <li>On Axis. Displays line scan data for the selected elements on the electron image axis.</li> <li>Overlaid. When line scan data for multiple elements are selected for overlay, all of the element line scans are overlaid on the electron image.</li> <li>Stacked. When line scan data for multiple elements are selected for overlay, the element line scans are stacked and scaled according to the scale setting (see below).</li> <li>Scaled Independently. When selected, scales each overlaid line scans the same.</li> </ul>

 Table 2.
 Linescan Mode, Linescan Processing Settings

## **Line Scan Spectral Processing Settings**

We typically recommend that you use one of the other analytical modes, such as <u>Spectrum</u><u>mode</u> or <u>Spectral Imaging mode</u>, to identify the elements in the microscope field of view before you perform a line scan analysis. You should also specify the spectral processing settings (Ident Setup and Quant Setup) you want to use.

Once you know the sample composition, select the Linescan analytical mode and set the known elements to Always Identified before you start the line scan analysis.

If you want to run the line scan analysis with automatic spectral processing (qualitative and quantitative) using only the data points measured in the line scan, follow these steps.

#### \* To set up automatic spectral processing (not recommended)

1. Click the Spectrum Processing tab not the controls panel.

The following options are displayed.

Spectrum Processing		~
R Ident Setup	Quant Setup	

2. To set up automatic element search and identification, choose <u>Ident Setup</u>, set the <u>identification parameters</u> as desired and make sure **Auto ID** is selected.



3. To set up a automatic quantitative analysis, choose <u>Quant Setup</u>, set the <u>quant</u> <u>parameters</u> as desired and make sure **Auto Quant** is selected.

Spectrum Processing		~
R Ident Setup	Quant Setup	
	Auto Quant	

4. In the element setup pane, right-click the C (carbon) symbol and choose **Always Identified**.

Since carbon is present in most samples, this allows the line scan to run without any other elements identified. You can then identify the elements of interest and manually set each one to Always Identified.

## **Acquire a Line Scan**

This section walks you through a line scan analysis.

**Note** The sample composition is assumed to be known. If the sample elements are unknown, start with a spectral analysis (Spectrum mode) or spectral imaging analysis (Spectral Imaging mode) to determine the sample composition before proceeding with the line scan analysis.

#### To acquire and analyze a data set

- 1. Start Pathfinder software and create or open a project.
- 2. Click the Linescan icon 🔀 on the <u>analytical mode toolbar</u>.

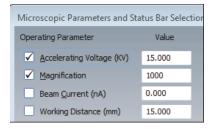
3. Prepare the sample.

Place the sample in the microscope and obtain an image. Adjust the stage height (Z-axis) to the optimal working distance for the EDS detector. Obtain a good microscope image of an area of interest on the sample. See <u>Microscope Considerations</u> for details.

- 4. Fully insert the EDS detector.
- 5. Set the basic microscope parameters:

**Note** If your SEM has Column Communications, these parameters are entered automatically.

- To set the microscope parameters manually, from the Home screen, choose **Edit** > **Microscope Parameters.** Set these parameters and then choose **Close** 
  - Accelerating Voltage in kilovolts (kV)
  - Magnification
  - Working Distance in millimeters (mm)



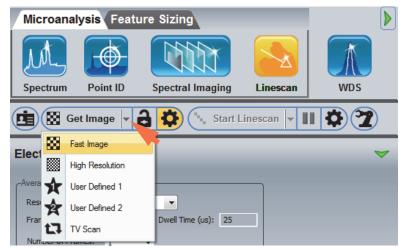
6. Enter an experiment name (used as a base name for automatically saving acquired data).

On the acquisitions toolbar, click the is button and enter the experiment name (or leave the default name).



7. Acquire an averaged electron image to use as the reference image.

- Click the image acquisition **presets** button on the acquisitions toolbar and choose **Fast Image** or **High Resolution** (see <u>Electron Imaging Mode</u> for more information).



- Click **Get Image** to acquire an electron image.

Microana	l <mark>ysis</mark> Featu	ire Sizing		
	Point ID	Spectral Imaging	Linescan	WDS
	et Image 🔻		t Map 🔻 C	

The button name changes to Stop Image. Wait until the acquisition is completed.

8. Set the line scan spectral acquisition parameters.

- Click the Linescan spectral acquisition **presets** button on the acquisitions toolbar and choose **Fast Line** or **High Resolution** (see Line Scan Spectral Acquisition Settings for more information).



9. Set the line scan processing parameters.

- Click the Linescan Processing tab on the controls panel and set Linescan Data Type.

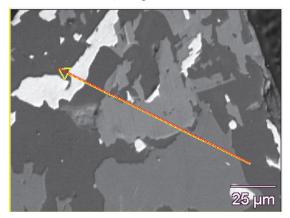
Linescan Proces	sing
Linescan Data Type	Weight % Counts Atomic % Weight % K-Ratio % Net Counts

10. Select the known elements for analysis.

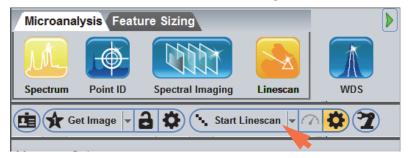
In the element setup pane (periodic table), right-click each element that is present in the sample and choose **Always Identified**.

11. Acquire the line scan data.

- In the image pane, click and drag the mouse cursor to draw the line you want to scan. The line can be any length and direction. Here is an example:



- Click **Start Linescan** to start the line scan acquisition.

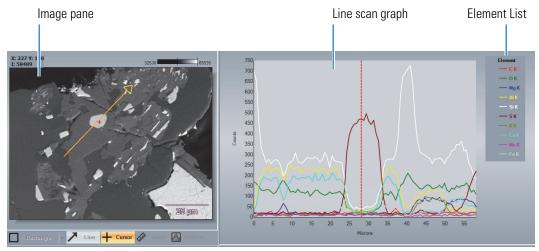


The button name changes to Stop Linescan.

- To end the acquisition and save the data, click Stop Spectrum.
- To end the acquisition without saving the data, click the Abort button

Wait until the acquisition is completed.

The results are displayed in a line scan graph. A legend shows the color assigned to each identified element. Here is an example:



When the acquisition is completed, the data set is automatically stored in the temporary directory and is ready for analysis. From here you can view the spectral or quant information for selected points or regions, or change the quant data type or the line scan display options.

12. To permanently save the data set in the current project, click the **Save** button **\_\_\_\_** on the main toolbar, or choose **File > Save**.

Linescan data are saved with a LSCTL (.lsctl) filename extension. If you open a LSCTL file from Windows Explorer, it contains the following individual files:

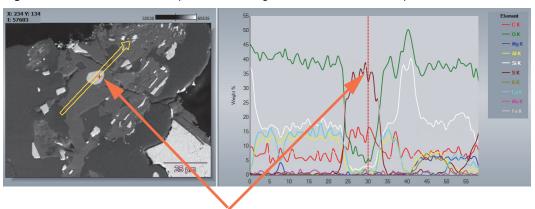
- .lsmsa (spectra)
- .lsref (reference image)

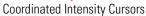
# **Analyze Line Scan Data**

#### **Coordinated Intensity Cursors**

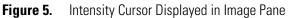
Click the **Intensity Cursor** button on the image pane toolbar to display intensity cursors in the image and graph panes.

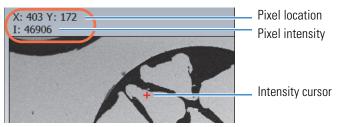
Figure 4. Coordinated Intensity Cursors in Image Pane and Line Scan Graph





The cursors show the pixel intensity and coordinates of the current cursor location in the upper left corner of the image pane.





You can click or drag the cursor to another location in either pane to update the pixel intensity and location information.

The results pane shows the quant data at the current cursor location. If you drag the cursor line right or left to select a region of the graph (gray area), the results pane shows the averaged results for the selected region. Here is an example:

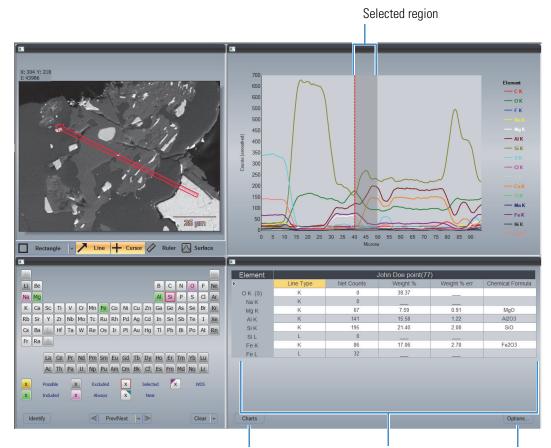


Figure 6. Quant Results for Selected Region

Charts button Quant Results for Selected Region

Options button

- To select which quant results to include in the table, click the **Options** button (see the previous image).
- To display the spectrum for the current cursor location, click the **Charts** button (see the previous image) and then click **Spectrum**.

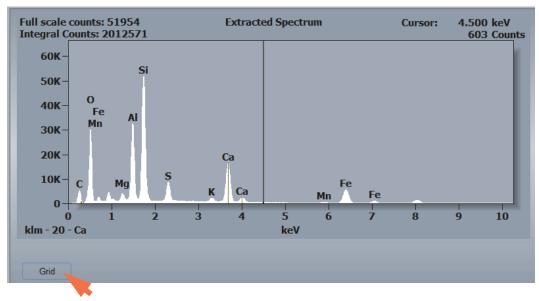


Figure 7. Results Pane with Spectrum Displayed

• To redisplay the quant results, click **Grid** (see above).

## **Select Elements For Line Scan Graph**

When the line scan acquisition is completed, a list of found elements appears at the right edge of the line scan graph.

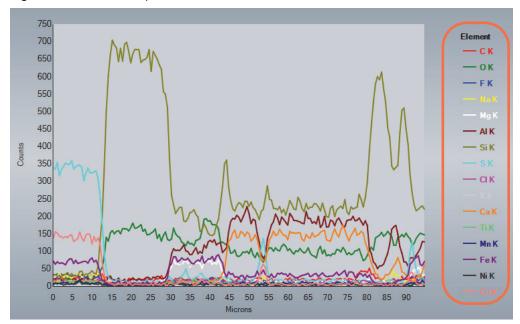


Figure 8. Linescan Graph Element List

#### To add an element to the line scan graph

1. Right-click the element in the element setup pane (periodic table) and choose **Always Identified**.

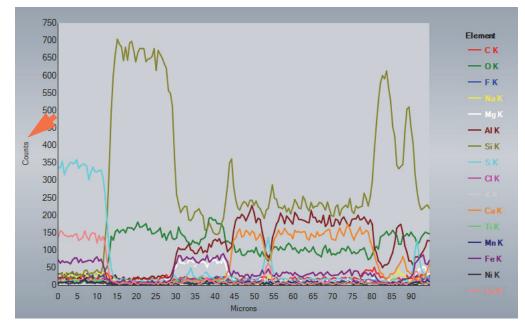
The data are reprocessed automatically and the added element appears in the element list.

#### \* To remove an element from the line scan graph

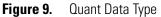
1. Right-click the element in the element setup pane (periodic table) and choose **Excluded**.

The data are reprocessed automatically and the excluded element is removed from the element list.

## **Change Line Scan Data Type**

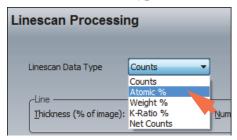


The Y-axis label shows the data type for the line scan graph.



#### ✤ To change the line scan data type

- 1. Click the Linescan Processing tab on the controls panel.
- 2. Reset Linescan Data Type.



The data are reprocessed automatically. The updated results are displayed in the graph pane.

# **Change the Appearance of the Line Scan Graph**

## Add Log Scale, Smoothing, Symbols or Grid

Use the features in <u>Data Options</u> and <u>Linescan Display</u> on the Linescan Processing tab to change the appearance of the line scan graph.

Figure 10. Line Scan Graph Display Options

Linescan Process	ing	~
Linescan Data Type	Counts	
	Number of Points:	
Data Options	Smooth 3 🔔 Spline	
Linescan Display Grid	Symbols Error Bars	
-Line Profiles     On Linescan	On Axis	
Overlayed	Stacked	
Step Notes	Linescan Processing Processing	Spectrum Settings

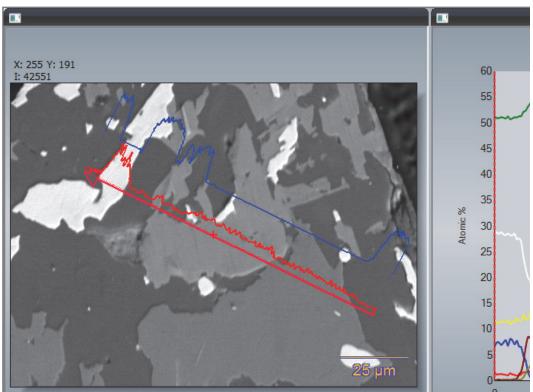
## **Overlay Line Scans on Electron Image**

- \* To overlay element line scan profiles on the electron image
- 1. Select the elements to overlay by clicking their symbols in the element list.



- 2. Select the position of the overlaid element profiles (On Linescan or On Axis).
- 3. If multiple elements are selected, choose whether their profiles are overlaid or stacked on the electron image.

Figure 11. Line Scan Profiles Overlaid On Electron Image (On Linescan and Stacked)



# **Export Line Scan Data**

#### \* To export line scan data as a CSV (comma separated values) file

- 1. In the Linescan analytical mode, select the file to export in the file list pane.
- 2. From the Home screen, choose File (menu) > Import/Export > Export Linescan as CSV.
- 3. Navigate to or create a folder for the exported data and choose Save.

The CSV file can be opened in any data processing application.

# 8

# **Spectrum Match**

Use the optional Spectrum Match software to identify a spectrum from an unknown material by comparing it to a database of previously acquired spectra from known materials. Spectrum Match can also be used in conjunction with Phase calculations in Spectral Imaging mode. Spectrum Match features are available in the software only when the option is installed.

Before you can use Spectral Match, you must create a Match library of spectra from known materials that are relevant to your field of study. You can then use the Match library to create smaller, application-specific databases that can be searched individually or in groups.

All of the features for creating a Match library and databases and for setting up and performing a search are on the Spectrum Match tab on the controls panel. Spectrum Match is available in all the <u>analytical modes</u>.

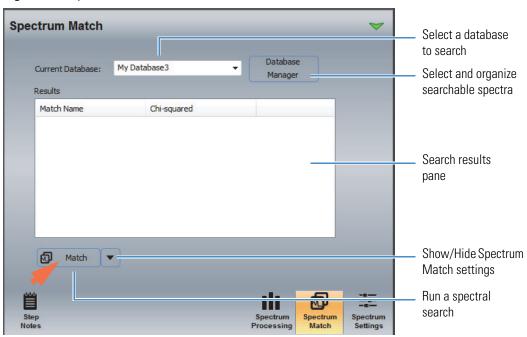


Figure 12. Spectrum Match features

Use **Database Manager** (see the previous image) to add spectra to the Match library and to create personal databases of searchable spectra that can be used with any project. Use **Current Database** to select a database to search. Use the **Match** button to search the selected spectrum. Use the **down arrow** button to access additional settings for customizing the search results.

## Add Spectra to the Match Library

Before you can use Spectral Match, you must create a Match library of spectra of known materials that are relevant to your field of study. You can then use the Match library to create smaller, application-specific databases that can be individually searched.

**Note** It is good practice to search using spectra acquired with the same microscope <u>accelerating voltage</u> setting as the unknown sample spectrum. As a result, you may want to acquire spectra that will be added to the Spectrum Match library at several microscope accelerating voltage settings.

This section explains how to add spectra to and delete spectra from the Match library. You can add spectral files immediately after you acquire them, import spectra from any project, or add synthetic spectra created using the software's internal database of element spectra. Synthetic spectra can be created manually by entering elements and composition values or using CSV (comma separated values) data from an analyzed spectrum.

#### **Add Acquired Spectra to Match Library**

#### To add acquired spectra to the Match library

- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Acquire the first spectrum so that it is displayed in the spectrum pane in any analytical mode.

**Note** The file will be added to the Match library with the filename shown in the file list pane. If you want to edit the filename before adding the spectrum to the library, use the Rename AT button on the file management panel.

3. Click Database Manager.

Spectrum Match	_	_	_	-
Current Database:	My Database3	-	Database Manager	
Deculte		C	Hundger	

- 4. In the Database Manager box, click the down arrow next to New Match File and select **From Spectrum**.
- 5. Enter a filename for the spectrum or use the default name and choose **OK**.

Current Database	My Database 1	-	Create Da	tabase	Delete Database
Available Match Files					
Name	kV	Date	Label	×	New Match File
					Delete
	Spectrum Albite(1				Add To Database

The spectrum is added to the end of the Available Match Files list. Here is an example:

Database Manager					×
Current Database	My Database	1	•	Create Database	Delete Database
Available Match Files					
Name		kV	Date	Label	New Match File 👻
Albite(1)20KV	1	15.0	17 May 2011	Albite(1)20KV	Import From Spectrum
					Manual From CSV

## **Import Spectra to Match Library**

- ✤ To import spectra to the Match library
- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager.

Spectrum Match		_		~
Current Database:	My Database3	-	Database Manager	
Deculte		C		

3. In the Database Manager box, click the down arrow next to **New Match File** and select **Import**.

Current Database	My Database 1	•	Create Database	Delete Database	
vailable Match Files					
Name	kV	Date	Label	New Match File	-
Albite(1)20KV	15.0	17 May 2011	Albite(1)20KV	Import	
				From Spectru	n, A

- 4. Navigate to the project and folder where the spectrum (.emsa) file is stored.
- 5. Select the file and choose **Open**.

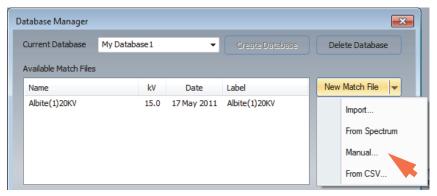
The spectrum is added to the end of the Available Match Files list.

#### **Create Synthetic Spectrum For Match Library**

- \* To create a synthetic spectrum for the Match library
- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager.



3. In the Database Manager box, click the down arrow next to **New Match File** and select **Manual**.



- 4. In the Create Simulated Spectrum box, enter the following:
  - use Match Name to enter a name for the synthetic spectrum
  - enter the desired Acceleration (accelerating voltage) in kV
  - enter the desired Zero Width (width of the zero peak) in eV
  - select a **Composition Type** (weight%, atomic% or atom count, which is the number of atoms from a stoichiometric formula)

Create Simulated Mat	ch Spectrum From Comp	osition	×
Match Name:	Synthethic SS Spectrum 20	kV	Show Spectrum
Acceleration kV:	20		Define For Match
Zero Width (eV):	34		
Composition type:	weight %		Clear
-Composition			
Element	weight %	Element	weight %
Na 💌	10		
К 💌	1.2		

5. For each element you want to add, use the down arrow to select the element symbol from the periodic table and enter its composition in the specified format.

The software uses its internal element database to create the simulated spectrum.

- 6. Click **Show Spectrum** to display the synthetic spectrum in the spectrum pane.
- 7. Click **Define For Match** and click **OK** to add the spectrum to the Match library.
- 8. Create and add additional synthetic spectra if desired.

9. When you are finished, click the **Close** button to close the Create Simulated Match Spectrum box.

The added spectra are listed at the end of the Available Match Files list.

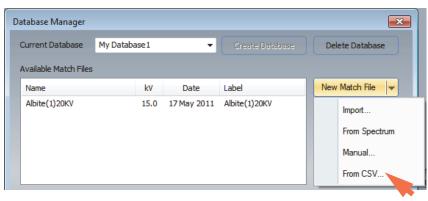
Current Database My Da	atabase 1	•	Create Database	Delete Database
Available Match Files				
Name	kV	Date	Label	New Match File 💌
Albite(1)20KV	15.0	17 May 2011	Albite(1)20KV	
Au Standard 30 kv(1)	30.0	13 May 2011	Au Standard 30 kv(1)	Delete
Synthethic SS Spectrum 20	)kV 20.0	26 Sep 2016	Synthethic SS Spec	
				Add To Database

#### **Use CSV Data To Create Synthetic Spectrum For Match Library**

- **\*** To use CVS data to create a synthetic spectrum for the Match library
- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager.

Spectrum Match	_		<
Current Database:	My Database3	Database     Manager	
Danulta		Manager	

3. In the Database Manager box, click the down arrow next to **New Match File** and select **From CSV**.



4. In the Create Simulated Match Spectrum From CSV File box, enter the **Acceleration Voltage** in kV and **Zero Width** (width of the zero peak) in eV for the simulated spectrum.

Compounds for Matc	h Analysis ———									
Acceleration Volta	ge (kV): 15		Zero Width (	eV): 34		Show Spectrum	Define For	Match		
Compound Name	Elmt.1 Wt.%	Elmt.2 Wt.%	Elmt.3 Wt.%	Elmt.4 Wt.%	Elmt.5 Wt.%	Elmt.6 Wt.%	Elmt.7 Wt.%	Elmt.8 Wt.%	Elmt.9 Wt.%	Elmt.10 Wt.2 *
20155	Cr 17.00	Mn 6.50	Fe 72.00	Ni 4.50						
	Cr 22.75	Mn 1.00	Fe 62.00	Ni 13.50	Mo 0.35					
30955	Cr 22.75 Cr 25.00	Mn 1.00 Mn 1.11	Fe 62.00 Fe 53.50	Ni 13.50 Ni 20.50	Mo 0.35 Cu 0.40	Mo 0.35				
309SS 310SS						Mo 0.35 Mo 2.35				
30955 31055 31655 31755	Cr 25.00	Mn 1.11	Fe 53.50	Ni 20.50	Cu 0.40					E.

5. To select one or more reference CSV files provided with the software, such as the stainless-steel spectra shown above, select the files in the list (use Ctrl or Shift keys to select multiple files).

The software uses its internal element database to create the simulated spectra.

Click **Show Spectrum** to display the selected synthetic reference spectrum in the spectrum pane. (This button is active only when one spectrum is selected.)

Click Define For Match and click OK to add the selected spectra to the Match library.

**Note** To add CSV data to this list in order to recreate the associated spectra and import them to the Match Library, open the list and enter the CSV values manually. The list is stored in this location:

C:\Program Files (x86)\Thermo Scientific\NSS\Match Library.csv

- 6. Create and add additional synthetic spectra if desired.
- 7. When you are finished, click the **Close** button to close the Create Simulated Match Spectrum box.

The added spectra are listed in the Available Match Files list.

## **Delete Spectra From the Match Library**

#### ✤ To delete spectra from the Match library

- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager.



3. In the Available Match Files box, select the file(s) you want to remove and click Delete.

Hold down the Shift key to select multiple contiguous files; hold down the Ctrl key to select multiple independent files.

urrent Database		-	Create Databa		Delete Database
vailable Match Files					
Name	kV	Date	Label	-	New Match File 📼
17-4 PH	15.0	27 Sep 2016	17-4 PH	=	
19-9DL	15.0	27 Sep 2016	19-9DL		Delete 🔪
19-9DX	15.0	27 Sep 2016	19-9DX		
254SMO	15.0	27 Sep 2016	254SMO	7	
31655	20.0	26 Sep 2016	31633		Add To Database
			Albite(1)20KV_ori		

# **Create a Searchable Match Database**

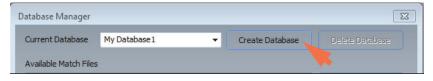
Once you have the needed spectra in the Match library, you can use them to create application-specific databases that can be individually searched in any project.

**Note** It is good practice to search using spectra acquired with the same microscope <u>accelerating voltage</u> setting as the unknown sample spectrum. As a result, you may want to set up searchable databases that include spectra acquired at specific accelerating voltage settings.

- \* To create a searchable Match database
- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager.



3. In the Database Manager box, select the **Current Database** box, enter a new database name and click **Create Database**.



**Note** The Create Database button is available in the software only when the Current Database box is empty or contains a new database name.

The Available Match Files box shows all the files in your <u>Match library</u> (use the scroll bar to review them). Those files are available for use in any searchable database you create. The Currently In Database box is empty.

Current Database My Datab	ase1	•	Create Database	Delete Database	
vailable Match Files					
Name	kV	Date	Label	New Match File 💌	
19-9DX	20.0	26 Sep 2016	19-9DX		
316SS	20.0	26 Sep 2016	316SS 🗄	Delete	
Albite(1)20KV	15.0	17 May 2011	Albite(1)20KV		Match libra
Au Standard 30 kv(1)	30.0	13 May 2011	Au Standard 30 kv(		spectra
Synthethic SS Spectrum 20kV	20.0	26 Sep 2016	Synthethic SS Spec	Add To Database	эреспа
Ni Al sample(4)	20.0	16 May 2011	Ni Al sample(4)		
•			•		
Currently in Database					
Currently in Database Name	kV	Date	Label	Remove	
	k۷	Date	Label	Remove	Creature in
	kV	Date	Label	Remove	Spectra in
	kV	Date	Label	Remove	Spectra in current
	kV	Date	Label	Remove	•

4. In the Available Match Files box, select one or more files to include in the new database and click **Add To Database**.

**Note** All the spectra you select should have been acquired using the same accelerating voltage setting. Use the kV column in the Available Match Files group to verify the accelerating voltage associated with each spectrum.

Hold down the Shift key to select multiple contiguous files; hold down the Ctrl key to select multiple individual files.

urrent Database My Databa	ase 1	•	Create Database	Delete Database
vailable Match Files				
Name	kV	Date	Label	New Match File
19-9DX	20.0	26 Sep 2016	19-9DX	
316SS	20.0	26 Sep 2016	316SS ≡	Delete
Albite(1)20KV	15.0	17 May 2011	Albite(1)20KV	
Au Standard 30 kv(1)	30.0	13 May 2011	Au Standard 30 kv(	
Synthethic SS Spectrum DkV	20.0	26 Sep 2016	Synthethic SS Spec	Add To Database
li Al sample(4)	20.0	16 May 2011	Ni Al sample(4)	
	111		•	
irrently in Database				
Name	kV	Date	Label	Remove
Albite(1)20KV	15.0	17 May 2011	Albite(1)20KV	
Au Standard 30 kv(1)	30.0	13 May 2011	Au Standard 30 kv(1)	
Synthethic SS Spectrum 20kV	20.0	26 Sep 2016	Synthethic SS Spec	
			Ni Al sample(4)	

The files remain in the Match library box and also appear in the Currently In Database box.

- 5. Continue adding as many spectra as you need.
- 6. When you are finished, click the **Close** button to save the new database and close the Database Manager box.

The new Spectrum Match database is now ready for use.

## **Remove Spectra From a Match Database**

- To remove spectra from a Match database
- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager.

Spectrum Match	_	_	_	~
Current Database:	My Database3	•	Database Manager	
Deculto			Manager	

3. Set **Current Database** to the database that contains the file(s) you want to remove.

4. In the Currently In Database box, select the file(s) you want to remove and click **Remove**.

Hold down the Shift key to select multiple contiguous files; hold down the Ctrl key to select multiple independent files.

	New Database2	-	Create Databa		Delete Database
vailable Match Files					
Name	kV	Date	Label	-	New Match File
17-4 PH	15.0	27 Sep 2016	17-4 PH	=	
19-9DL	15.0	27 Sep 2016	19-9DL		Delete
19-9DX	15.0	27 Sep 2016	19-9DX		
254SMO	15.0	27 Sep 2016	254SMO		
316SS	20.0	26 Sep 2016	316SS		Add To Database
Albite(1)20KV_origina	al 15.0	17 May 2011	Albite(1)20KV_or	igi. 🔻	
•				F.	
	kV	Date	Label	-	Remove 🔪
Name					Remove
Name 19-9DX	20.0	26 Sep 2016	19-9DX		Remove
Name			19-9DX 316SS	5	Remove
19-9DX	20.0	26 Sep 2016	19-9DX		Re

## **Delete a Match Database**

#### To delete a Match database

- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click Database Manager, set Current Database to the database you want to delete.
- 3. Click Delete Database.
- 4. click the **Close** button to close the Database Manager.

## **Use Spectrum Match**

After you have created one or more databases of searchable spectra, you are ready to run a Spectrum Match analysis, which searches the selected (displayed) spectrum against the selected spectral database. Spectrum Match can also be used in conjunction with Phase calculations in Spectral Imaging mode. Spectrum Match can be set up to run automatically after each spectrum is acquired, or it can be run manually on a displayed or stored spectrum.

## Identify an Unknown Material Using Spectrum Match

#### \* To manually identify an unknown material using Spectrum Match

- 1. In any analytical mode, acquire or select the spectrum of an unknown sample so the spectrum is displayed in the spectrum pane.
- 2. Click the **Spectrum Match** tab on the controls panel.
- 3. Set **Current Database** to the database you want to use.

Spectrum Match		_	<b>V</b>
Current Database:	My Database3	Database     Manager	
Results	My Database1 New Database2		

4. Click the arrow at the right of the Match button and specify the <u>Spectrum Match settings</u> (Auto Match can be on or off).

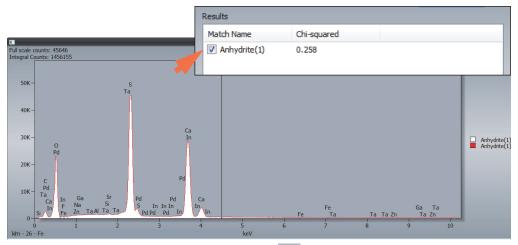
	Results          Match Name       Chi-squared         Image: Chi-squared       Image: Chi-squared         Image: Chi-squared <th>Match Name Chi-squared</th>	Match Name Chi-squared	Match Name Chi-squared	Match Name Chi-squared	Match Name Chi-squared	Match Name Chi-squared	Match Name Chi-squared
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Match Cutoffs (keV): Low High 50000	Match Match Cutoffs (keV): Low High 50000	Match	Match
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	<u>~</u>		<u></u> .	<u></u>		Match Cutoffs (keV): Low High 50000	
	<u>~</u>		<u></u> .	<u></u>		Match Cutoffs (keV): Low High 50000	
	Max. Number of Match Results: 5	Max. Number of Match Results: 5	Max. Number of Match Results: 5				
Low High 50000				Max. Number of Match Results:	Max. Number of Match Results: 5		<u>000</u>
sults: 5	Step Sp	Step Sp	Step Sp	Cha-		Max. Number of Match Results:	Max. Number of Match Results:
	Notes Chi-square Cutoff: 5	Notes S	Notes Sector Sec		Step Sp	Step Step Step	Step Sp
	Match Cutoffs (keV): Low High 5000	Match Cutoffs (keV): Low High 5000	Match Cutoffs (keV): Low High 5000	Match Cutoffs (keV): Low High 5000	Match Cutoffs (keV): Low High 5000		
	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000	Match Cutoffs (keV): Low High 50000		
	<u>~</u>		<u>~</u>	<u></u>		Match Cutoffs (keV): Low 0 High 50000	
	Max. Number of Match Results: 5	Max. Number of Match Results: 5	Max. Number of Match Results: 5				
Low I High 50000	Max, Number of Match Results:	Max, Number of Match Results:	Max. Number of Match Results:	Mary Number of Match Deputitor 5	Mary Number of Match Daviday 5		
	Stan	Sten	Step	Max. Number of Match Results:	Max. Number of Match Results: 5		

The results are displayed on the Spectrum Match tab. The best matched spectrum is listed first. Here is an example:

rrent Database:	My Database 1	Database Manager
sults		
latch Name	Chi-squared	
Anhydrite(1)	0.258	

Spectrum Match search results:

- Match Name is the filename of the spectrum in the selected database
- **Chi-squared** indicates the quality of the fit. The lower the chi-squared value the better is the fit. A chi-squared value of 0.0 is a perfect match. Try to keep the chi-squared value under 10.
- 6. To overlay a database spectrum from the Match Results box with the spectrum of the unknown sample, click the spectrum's associated check box. The overlaid spectrum is highlighted in red in the spectrum pane. Here is an example:



To scale the overlaid spectrum, click the **Match** button on the File Management panel and select a normalization setting. The normalization setting has no effect on the original spectrum.

Setting	Description
None	No normalization factor is applied
Livetime	Normalizes the overlapped spectra based on the live time of the overlapped spectrum compared to the live time of the original spectrum. This simulates the same acquisition times for both spectra.
Element	Adjusts the overlapped spectra to the height of the selected element peak or line in the original spectrum. The selected peak or line is based on accelerating voltage and the energy range of the spectrum.
Multiplier	Enter a value that will be used to scale all overlapped spectra
Range	<ul> <li>Scales the overlapped spectra to the original spectrum based on a selected energy range. When Normalize To is set to Range, two dashed vertical lines are added to the displayed spectra. (If you don't see the vertical lines, double-click to expand the spectrum pane.) Drag the lines left and right to define the left and right boundary of your selected range.</li> <li>Note: Defining a range in a background region (between element peaks) sometimes gives the best results.</li> </ul>
Maximum	Scales the overlapped spectra to the original spectrum based on their maximum peak values in the displayed region
Maximum in Overlap	If the overlapped spectrum intersects the original spectrum, the software scales the maximum peak value in the overlapped spectrum to match the maximum peak value in the original spectrum.
	<b>Note</b> : This setting is intended for WDS data where the spectra may not cover the same energy range but still overlap in some energies.

The available normalization options are described below.

## Identify an Unknown Phase Spectra Using Spectrum Match

Spectrum Match can be used in conjunction with Phase calculations in Spectral Imaging mode. If a database exists and if Auto Match is selected in the Spectrum Match settings, the software attempts to match each spectrum generated by a Phase calculation with the spectra in the database. If a match is found, it renames that phase with the name of the matched spectrum.

## **Configure Spectrum Match to Run Automatically**

- \* To configure Spectrum Match to run automatically
- 1. Click the **Spectrum Match** tab on the controls panel.
- 2. Click **Database Manager**, set **Current Database** to the database you want to use and click the **Close** button.
- 3. Click the arrow at the right of the Match button and select Auto Match.

		Database
Current Database:	My Database 1	Manager
Results		
Match Name	Chi-squared	
Anhydrite(1)	0.258	
භා Match	Match Cutoffs (keV): Low 0	High 50000
Match		
	Match Cutoffs (keV): Low 0	

4. In any analytical mode, acquire or select the spectrum of an unknown sample so the spectrum is displayed in the spectrum pane.

The results are displayed on the Spectrum Match tab. The best matched spectrum is listed first.

# **Spectrum Match Settings**

To display the settings for customizing a Spectrum Match search, select the Spectrum Match tab on the controls panel and click the arrow next to the Match button.

Figure 13. Spectrum Match Settings

pectrum Match		_	~
Current Database:	My Database1	Database Manager	
Results			
Match Name	Chi-squared		
Match			
Match			
	Match Cutoffs (keV): Low		
Ë	Match Cutoffs (keV): Low Max. Number of Match Results:	High 50000	-=
			Spectrur Setting:

The available options are described below.

#### Table 3. Spectrum Match Settings

Feature	Description
Match Cutoffs	Enter a low and/or high cutoff value in eV to limit the searched region of the unknown sample spectrum (where 1000 eV is equal to 1 keV)
Max. Number of Match Results	Defines the maximum number of search results to display

Feature	Description
Chi-Square Cutoff	Defines the maximum chi-squared value for the search results. Typical value is 5. Increase this number to include more spectra in the search results box. Generally, a chi-squared value of 10 or less indicates a reasonable match.
	<b>Note</b> : The number of background channels greatly influences the chi-squared values.
Auto Match	Select this option to automatically run a Spectrum Match after each acquired spectrum

 Table 3.
 Spectrum Match Settings

8 Spectrum Match Spectrum Match Settings

# 9

# **Analysis Automation**

Use the optional Analysis Automation software to automate analyses at pre-defined locations of the microscope stage without requiring operator input. Analysis Automation features are available in the software only when the software is installed. (The stage must be motorized and under digital control.)

You define the number of sampling points and their stage locations. The selected analytical mode specifies the type of data acquired and how the data are analyzed. When you start the automated analysis, the system moves the stage to the first sampling location and acquires and processes the data. Then it moves the stage to the next position and repeats the acquisition and analysis. The process continues until all specified points or area have been acquired and processed. Sampling locations can be saved for reuse.

Analysis Automation can be used in these analytical modes:

- <u>Spectrum</u>
- Spectral Imaging
- Point ID
- Linescan
- Electron Imaging
- Feature Sizing

#### Note

- In Spectrum mode, the Analysis Automation settings allow you to specify the beam position (center spot or the entire field of view). The Pathfinder chassis hardware controls the beam position for Analysis Automation in all other analytical modes.
- Analysis Automation is designed to work with simple drift compensation where the area used to track the electron image is the same as the area being measured. To run an automated sequence with drift compensation, before you acquire an electron image or automated spectral acquisition, make sure Drift Compensation is set to Drift On. (Do not use the Rect Drift On setting.) See <u>Drift Compensation</u> for more information.

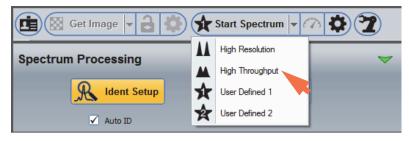
# **Analyze Pre-defined Locations In Spectrum Mode**

This section provides an overview of the steps to set up and run a basic X-ray spectral analysis of selected sampling locations with Analysis Automation. It is assumed that you are familiar with basic sample acquisition and analysis using the <u>Spectrum</u> analytical mode.

- \* To acquire and analyze a series of sampling locations with Analysis Automation
- 5. Click the Spectrum <u>U</u> button on the <u>analytical mode toolbar</u>.
- 6. Set up the microscope as you would for a Spectrum analysis (adjust beam current to desired count rate and dead time).
- 7. Click the button on the acquisitions toolbar and enter an experiment name (used as a base name for automatically saving acquired data).



8. Click the spectral acquisition presets button on the acquisitions toolbar and select a preset such as High Resolution or High Throughput.



9. Select the **Spectrum Processing** tab on the controls panel and set up any qualitative or quantitative analysis for the acquired data.

For qualitative analysis, click **Ident Setup** and make sure Auto ID is selected; for quantitative analysis, click **Quant Setup** and make sure Auto Quant is selected.

10. Click the Analysis Automation button on the acquisitions toolbar.



11. Set Acquisition Type to Selected Area.

Analysis Automation		~
Acquisition Type	Sequence Name	
Selected Area	Point	
	<b></b>	

12. Enter a Sequence Name (appended to the experiment name to identify each microscope location analyzed with Analysis Automation).

- 13. Specify any advanced automation settings.
  - Click the green arrow to show the advanced settings:

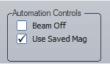
Analysis Automation		~
Acquisition Type	Sequence Name	· · · · · · · · · · · · · · · · · · ·
Selected Area 🔹	Point	

- Set Spectrum Acquisition:

1	-Spectrum Acquisition						
	<ul> <li>Center Spot</li> </ul>						
	Microscope Raster						
l		ļ					

- **Center Spot**. Acquires a spectrum in the center of the field of view for each specified location.
- **Microscope Raster**. Acquires spectra over the entire field of view and produces an average spectrum for each specified location.

- If you want the software to save the magnification settings with each sampling location, select **Use Saved Mag**.



- If you want the software to turn off the electron beam after the automated analysis is completed, select **Beam Off**.

- 14. Move the microscope stage to the first location you want to analyze on the sample.
- 15. Click Add Location.

The software adds the location to the Acquisition Locations box.

- <u>1</u>	ition Type red Area	2	•		equence N Point	ame	_	
Add Chang Locati	ge)	Remove	Go T Locat	-		Save Points File	Load Points File	
No.	X (mm)	Y (mm)	Z (mm)	Rot (deg)	Tilt (deg)	Mag		
V 1	6.92	51.92	10.00	56.14	0.00	3595		

- 16. Move the stage to the next location and click **Add Location** to add that point to the Acquisition Locations box.
- 17. Continue moving the stage and adding sampling locations as needed.

**Note** To remove a sampling location, click the row in the Acquisition Locations box and then click **Remove Location**.

- 18. When you are finished, click **Save Points File** to save the locations if you want to use them for future acquisitions.
- 19. Click Start Automation to start the analysis.



The button name changes to Stop Automation.

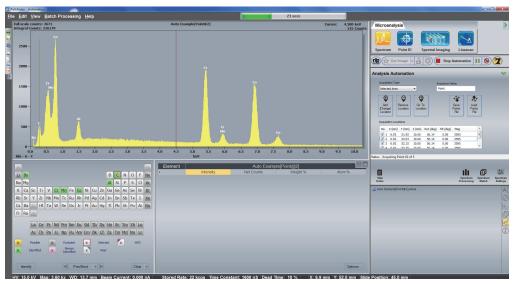
💼 🔄 Get Image 🗸 🔒 🔕 🔳	Stop Autom	ation	87
	Stop	Pause	Abort

• To pause the acquisition, click the **Pause** button.

To continue the acquisition, click the pause button again.

- To end the acquisition after the current point is completed and save the acquired data, click **Stop Automation.**
- To end the acquisition without saving the data, click the Abort button

The spectrum for the first sampling point is displayed in the spectrum pane.

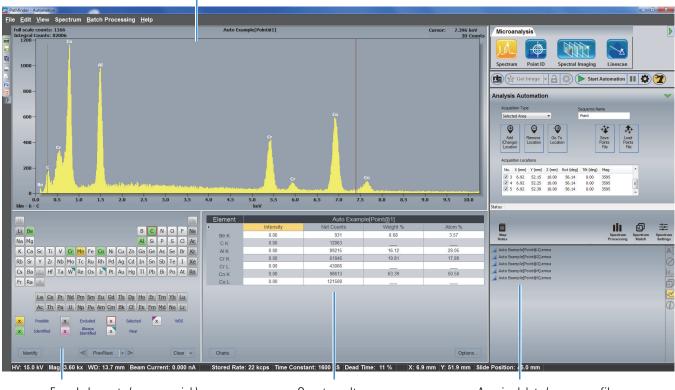


The progress bar at the top of the Pathfinder window shows the progress of the acquisition.

20. Wait until the acquisitions for all selected sampling locations are completed.

The results for the first sampling point are displayed. Here is an example:

Spectrum from the first analyzed location





Quant results

If Auto ID and Auto Quant are selected in Spectrum Processing, the found elements are displayed in the element setup pane (periodic table) and the quant results are displayed in the results pane.

The acquired data are listed in the file list pane (one spectral (.emsa) file for each analyzed location).

21. To save the data set in the current project, click the **Save** button **[**] on the main toolbar, or choose **File** (menu) > **Save**.

**Note** If Auto Quant is selected in Quant Setup on the Spectrum Processing tab, the quantitative results are automatically saved to a CSV (Comma Separated Values, .csv) file.

Acquired data (one .emsa file for each analyzed location)

## **Save and Load Selected Locations**

After you have located and entered a series of sampling location so they are listed in the Acquisition Locations box on the Analysis Automation tab, you can save the settings so they are available for repeated use.

## ✤ To save a series of sampling locations

1. On the Analysis Automation tab, add the sampling locations for automated analysis by moving the stage to each location and clicking the **Add Location** button.

nalysis Au	tomatic	n	_	_	-		~
Acquisition Ty	pe		5	Sequence N	ame		
Selected Are	a			Point			
<b>(</b>	Ø	<b>S</b>		ſ	+**	<b>1</b>	
Add (Change) Location	Remove	Go T Locat	-		Save Points File	Load Points File	
Acquisition Lo	cations						
No. X (mn	1) Y (mm)	Z (mm)	Rot (deg)	Tilt (deg)	Mag	<u> </u>	
1 6.92	51.92	10.00	56.14	0.00	3595	E	
2 6.92	52.03	10.00	56.14	0.00	3595		
3 6.92	52.15	10.00	56.14	0.00	3595		
4 6 07	57.75	10.00	56 14	0.00	2505	•	

- 2. Click Save Points File.
- 3. In the Save Points File box, select a folder, enter a filename and click Save.

The file is saved with an .apf (Automation Points File) filename extension.

#### To load a saved sampling series

- 1. On the Analysis Automation tab, click Load Points File.
- 2. Navigate to the project and folder that contains your saved .apf (Automation Points File), select the file and click **Open**.

The file's contents are displayed in the Acquisition Locations box on the Analysis Automation tab.

cquisi	tion Type			5	equence N	ame	
elect	ed Area		•		Point		
Add Chang Locatio	je) L	Remove location	Go T Locat	-		Save Points File	Load Points File
quisi	tion Loca X (mm)	tions Y (mm)	Z (mm)	Rot (deg)	Tilt (deg)	Mag	•
✓.	6.92	51.92	10.00	56.14	0.00	3595	E
<b>v</b>	6.92	52.03	10.00	56.14	0.00	3595	
<b>V</b> 3	6.92	52.15	10.00	56.14	0.00	3595	
	6.92	52.25	10.00	56 14	0.00	3505	-

If you want to use some locations and not others, use the check boxes (see the previous image) to select which points to sample.

If you want to remove a sampling location from the list, click its associated row in the table so the row is highlighted and then click **Remove Location**.

If you want to move the stage to a location to verify the settings, select (highlight) the row and click **Go To Location**.

## **Analyze a Grid at Each Location**

If you need to investigate a large region of the sample, use the Grid (rectangular) or Circular option for Acquisition Type to acquire a series of spectra. You can elect to have the software define a grid of adjacent areas based the current microscope field of view, or to define the grid manually by specifying the number of X and Y fields to include.

## \* To acquire and analyze a grid using analysis automation

- 1. Click the Spectrum button on the <u>analytical mode toolbar</u> and set up your spectral analysis.
- 2. On the Analysis Automation tab, set **Acquisition Type** to Grid or Circular.

Analysis Automation	~
Acquisition Type	

The following message is displayed for the Grid (rectangular) acquisition type:

ſ	Grid (Step 1)	
	Drive the stage to the upper left-hand corner of the grid. Focus the image (via the microscope's focus control). Click 'OK' to record this position.	
	OK Cancel	

3. Move the stage to the upper left-hand corner of the region you want to sample, focus the image by adjusting the stage Z-axis, and choose **OK**.

The next message is displayed:

Grid (Step 2)			×
the grid	d. Focus the im	lower right-hand co age (via moving the cord this position.	
	ОК	Cancel	

4. Move the stage to the lower right-hand corner of the region you want to sample, focus the image and choose **OK**.

A third message is displayed:

Grid (Step 3)		<b>—</b> ×	
the grid movem Focus f	d (i.e. upper-rig ent doesn't ne the image (via i	e of the remaining corners of ght or lower-left). This eed to be exact, just close. moving the Z-stage axis). Cli al grid position definition.	
	ОК	Cancel	

5. Move the stage to a third location inside the region you want to sample (in approximately the upper-right or lower-left corner) and focus the image.

This position allows the software to calculate a 3-dimensional plane that defines the stage Z-axis position for each point in the grid.

6. When you are finished, click **OK**.

The first grid location is displayed in the Acquisition Locations box.

7. Continue to define additional sampling grids as needed.

Here is an example:

	🔄 🔆 Get Image 👻 🔒 🔅 🕨 Start Automation 🔢 🔅 🏹					
Analysis A	utomatior	ı				~
Acquisition	Туре					
Grid		-				
Add Location	Remove Location	Edit Location		Save Points File	Load Points File	
No.			Label			
✓ 1			Grid1			
<ul> <li>✓ 2</li> <li>✓ 3</li> </ul>			Grid2 Grid3			

#### Note

- To remove a sampling location, click the row in the Acquisition Locations box and then click **Remove Location**.
- You can <u>edit the grid settings</u> (including the magnification and anchor point locations) by selecting its associated row in the Acquisition Locations box and choosing **Edit Location**.
- 8. When you are finished, click **Start Automation** to start the analysis.

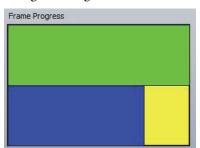
Depending on the number of sampled areas included, a series of files are created for each defined grid. For example, the following files are created for a 4x2 grid:

Basename[Grid@0 0].emsa Basename[Grid@0 1].emsa Basename[Grid@1 0].emsa Basename[Grid@1 1].emsa Basename[Grid@2 0].emsa Basename[Grid@2 1].emsa Basename[Grid@3 0].emsa Basename[Grid@3 1].emsa

To monitor the acquisition, click the green arrow to open the advanced view.

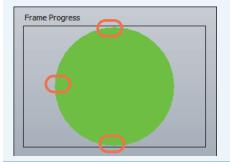
Analysis Automation		~
Acquisition Type	Sequence Name	•
Selected Area 🔻	Point	

The grid changes color as each new area is completed.



The spectrum in each file is acquired by rastering the beam over the field of view of the SEM at each point in the grid. If the SEM is set to focus the electron beam to a point or spot on the sample, then the EDS spectrum at each grid point will come from the point defined in each field of view.

**Note** The circular grid works the same way but uses round areas and is ideal for round samples such as a circular filter paper. The first two specified locations define the diameter of the circular region. The third location marks the Z movement to keep the image in focus over the defined region. The software skips the empty corners.



## **Clear a Grid List**

To clear the list of grid locations, right-click anywhere in the Acquisition Locations box on the Analysis Automation tab and choose **Clear All**.

cquisition	Гуре					
Circular		•				
Add Location	Remove Location	Edit Location		Save Points File	Load Points File	
cquisition l	ocations					
No.			Label			
<b>V</b> 1			Grid			
√ 2			Grid 1			
<b>V</b> 3			Grid	Clear All		

## **Grid Settings for Analysis Automation**

You can edit the grid location settings and then redo a grid by selecting its associated row in the Acquisition Locations box and choosing **Edit Location**.

Get	Image 🗸 🔒	<b>S</b>	itart Automa	tion	• 2
Analysis Aut	omation				~
Acquisition Typ	e				
Grid	•				
	Remove Location		Save Points File	Load Points File	
No.		Label			
☑ 1		Grid 1			
<ul><li>✓ 2</li><li>✓ 3</li></ul>		Grid2 Grid3		ノ	

The Edit Positions box is displayed.

I	Edit Position	s						
ſ	Number:	3	-					
	Label:	Grid	3		Ма	gnification:	3595	Update
	Frame Lay	outs						
	Adjacent	:	0.0	00000		Overlap %		
	O User Grid	ł	0			X Fields		
			0			Y Fields		
	Position	X (n	nm)	Y (mm)	Z (mm)	Rot (deg)	Tilt (deg)	
	First	7.2	3	52.54	10.00	56.14	0.00	
	Last	7.1	5	52.63	10.00	56.14	0.00	
	Z Correct	7.24	1	52.70	10.00	56.14	0.00	
				+ -	•			
							Accept	Cancel

The following Grid options are available in the Edit Positions box.

## Table 4. Grid Settings for Analysis Automation

Feature	Description
Number	Grid number displayed in the Acquisition Locations box on the Analysis Automation tab and used in the filename for the acquired data
Label	Label displayed in the Acquisition Locations box on the Analysis Automation tab and used in the filename for the acquired data
Magnification	Shows the magnification used for the selected grid. You can enter a magnification value or set the microscope to the desired magnification and click <b>Update</b> to read the new setting.

Feature	Description
Frame Layouts	Specifies how the grid is divided up. Available options:
	• <b>Adjacent</b> . The software defines a grid of adjacent areas based on the magnification of the microscope field of view.
	<ul> <li>Overlap%. Specifies the amount of overlap for adjacent areas of the grid.</li> </ul>
	<b>Note</b> : We recommend using a small overlap (2-3%) of adjacent frames, especially if you plan to use the grid to <u>creat</u> <u>a montage</u> .
	• User Grid. Allows you to enter the number of sampling areas to include along the X and Y-axes of the grid. The software divides the grid equally into the specified number of fields based on the magnification of the microscope field of view.
	<b>Note</b> : You may have unsampled areas in your defined region when using the Adjacent setting but not for the User Grid setting if you include enough fields to completely cover the defined region.
Position table	Shows the current locations of the two anchor points used to define the grid, and the location used to define the focus (Z Correct). Available options:
	• Add or change a point (+). Allows you to change the selected anchor point or redo all three anchor points.
	To change an anchor point, move the stage to the new location (or click <b>Move Stage To Point</b> and adjust from there), select the anchor point you want to change and click the <b>+</b> button.
	To add an anchor point, first delete all three anchor point and then add them in this order: upper-left corner, lower-right corner, upper-right or lower-left corner (for focus position).
	• <b>Remove Point</b> (—). Removes the selected anchor point from the Position table.
	<ul> <li>Return To Stage Point (-&gt;). Moves the microscope stag to the anchor point selected in the Position table.</li> </ul>

## Table 4. Grid Settings for Analysis Automation

14010 11	and obtained for Analysis Automation
Feature	Description
Accept	Saves your edits
Cancel	Closes the box without saving your edits

## **Table 4.** Grid Settings for Analysis Automation

## **Analysis Automation with Other Analytical Modes**

Analysis Automation can be used in these analytical modes:

- Spectrum
- Point ID
- Linescan
- Spectral Imaging
- Electron Imaging
- Feature Sizing

See the previous sections for details on how to use Analysis Automation in Spectrum mode. This section explains additional considerations for Analysis Automation when use with the remaining analytical modes.

## **Analysis Automation in Point ID Mode**

When Point ID mode is selected, the only Acquisition Type available on the Analysis Automation tab is Selected Area (no grid options). This is because Point ID is based on user-defined points or areas at each sampling location.

The automated Point ID analysis is similar to automated analysis in Spectrum mode except you need to acquire an electron image at each sampling location and add acquisition points or areas to the image before you add the location to the Acquisition Locations box on the Analysis Automation tab.

#### \* To acquire and analyze a series of sampling locations in Point ID mode

- 1. Click the Point ID button on the <u>analytical mode toolbar</u>.
- 2. Set up the microscope as you would for a Point ID analysis.
- 3. Enter an experiment name.
- 4. Move the microscope stage to the desired location on the sample.

- 5. Acquire an electron image.
  - Select an image acquisition preset on the acquisitions toolbar.

(			Get Image 🗸 🔒	Start Automation 🗥 🔅 🤦
	Anah	88	Fast Image	
ſ	niai;		High Resolution	
	Ac	¥	User Defined 1	Sequence Name
	S	*	User Defined 2	Point
		t7	TV Scan	save laad

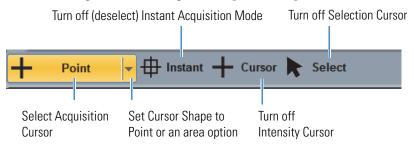
- Click **Get Image** to acquire the image.
- 6. Select a spectral acquisition preset on the acquisitions toolbar.

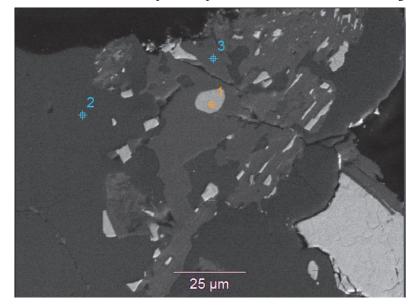
Get Image 🗸 🔒 🌑	🖈 Start Spectrum 💌 🏠 🏹
Spectrum Processing	High Resolution
	High Throughput
R Ident Setup	User Defined 1
✓ Auto ID	User Defined 2

7. Select the **Spectrum Processing** tab on the controls panel and set up any qualitative or quantitative analysis for the acquired data.

For qualitative analysis, click **Ident Setup** and make sure Auto ID is selected; for quantitative analysis, click **Quant Setup** and make sure Auto Quant is selected.

8. Use these Image toolbar settings to set up batch acquisitions:





9. Use the mouse to add acquisition points or areas to the electron image.

10. Click the Analysis Automation button on the acquisitions toolbar.



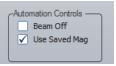
11. Enter a Sequence Name (appended to the experiment name to identify each microscope location analyzed with Analysis Automation).

Analysis Automation		~
Acquisition Type	Sequence Name	
Selected Area 🔹	Point	

- 12. Specify any advanced automation settings.
  - Click the green arrow to show the advanced settings:

Analysis Automation		~
Acquisition Type	Sequence Name	•
Selected Area 🔻	Point	

- If you want the software to save the magnification settings with each sampling location, select **Use Saved Mag**.



- If you want the software to turn off the electron beam after the automated analysis is completed, select **Beam Off**.

#### 13. Click Add Location.

If you see a message similar to the following, click **Proceed**.

Linescan Analysis Automation
Please make sure that you have acquired an image and placed a line over the area to be analyzed.
Proceed

The software adds the location to the Acquisition Locations box.

Acquisition Type Selected Area			Sequence Name Point				
elected Ar	ea	•		FUIIL			
Ð	Q	Ð			4	<b>1</b>	
Add Change) Location	Remove Location	Go T Locat	-		Save Points File	Load Points File	
cquisition L	ocations						
No. X (m	m) Y (mm)	Z (mm)	Rot (deg)	Tilt (deg)	Mag		
1 6.9	2 51.92	10.00	56.14	0.00	3595		

- 14. Move the stage to the next location, acquire a new electron image, add your acquisition points or areas to the image and click **Add Location**.
- 15. Continue moving the stage and adding sampling locations as needed.

**Note** To remove a sampling location, click the row in the Acquisition Locations box and then click **Remove Location**.

16. Click Start Automation to start the analysis.



If Auto ID and Auto Quant are selected in Spectrum Processing, the found elements are displayed in the element setup pane (periodic table) and the quant results are displayed in the results pane.

The acquired data are listed in the file list pane (one Point ID file for each analyzed location). Here is an example:

	Auto	Example[Point@1].PS.EDS
-	Auto	Example[Point@2].PS.EDS

17. To save the data set in the current project, click the **Save** button **[**] on the main toolbar, or choose **File** (menu) > **Save**.

**Note** If Auto Quant is selected in Quant Setup on the Spectrum Processing tab, the quantitative results are automatically saved to a CSV (Comma Separated Values) file with a .csv filename extension.

## **Analysis Automation in Linescan Mode**

When Linescan mode is selected, there are two Acquisition Type options on the Analysis Automation tab:

- Linescan Image. Similar to automated analysis in Point ID mode in that it is based on an electron image acquired at each sampling location. The main difference is that you draw a line scan on the image where you want to acquire data before you add the location to the Acquisition Locations box on the analysis Automation tab.
- Linescan Stage. Allows you to acquire a continuous line scan over a large area of the sample by defining two anchor points and automating the stage movement and acquisitions between them.

#### Note

- The sample composition is assumed to be known. If the sample elements are unknown, start with a spectral analysis (<u>Spectrum mode</u>) or spectral imaging analysis (<u>Spectral Imaging mode</u>) to determine the sample composition before proceeding with any type of line scan analysis. Before you start the line scan analysis, set the known elements to Always Identified in the element setup pane (periodic table). See <u>Linescan Mode</u> for more information.
- The speed of the microscope stage is the primary factor that determines the length of time needed to acquire a large line scan.

#### **Run an Automated Linescan Image Analysis**

The automated Linescan Image analysis is similar to automated analysis in Point ID mode in that it is based on an electron image acquired at each sampling location. The main difference is that you draw a line scan on the image where you want to acquire data instead of selecting points or areas.

To set up the analysis, set **Acquisition Type** in Analysis Automation settings to Linescan (Image) and follow the steps for Analysis Automation in Point ID mode with the exception of drawing a line scan on each acquired image instead of points or areas. The acquired data have the same filename extension as data acquired in normal Linescan mode, except the Analysis Automation Sequence Name is appended to the experiment name. One file is produced for each analyzed location. Here is an example: Auto Example[Point@1].lsctl Auto Example[Point@2].lsctl

**Note** If Auto Quant is selected in Quant Setup on the Spectrum Processing tab, the quantitative results are automatically saved to a CSV (Comma Separated Values, .csv) file. If you save the

#### **Run an Automated Linescan Stage Analysis**

The automated Linescan Stage analysis allows you to acquire a continuous line scan over a large area of a sample by defining two anchor points and automating the stage movement and acquisitions between them. The software uses a wizard to step you through setting up this analysis.

The wizard allows you to specify a step size between sampling points or a total number of points to sample, as well as the acquisition time in seconds at each sampling point. Each point can be one spectrum acquired in the center of the electron beam or an averaged spectrum representing the entire field of view. For example, if you select center point sampling with a 10 mm step size for a set of anchor points that are 1 mm apart, the sampling points will be 0.01 mm apart with a total of 11 points sampled (includes the anchor points). See Analysis Automation Linescan Settings for details.

#### To set up and run an automated Linescan stage analysis

1. Set up the Linescan analysis including spectral acquisition, Linescan Processing and Spectrum Processing.

If you are using Auto ID, make sure the known elements are set to Always Identified in the element setup pane (periodic table).

**Note** There is no need to manually acquire an electron image or draw a linescan in this analysis mode. The software performs these steps automatically.

2. Click the Analysis Automation button on the acquisitions toolbar.

💼 🗟 Get Image 🗸 🔒 🏟 🕨 Start Automation 🗥 🔅 🏹						
Analysis Automation						
Acquisition Linescan (		•				
Add Location	Remove Location	Edit Location		Save Points File	Load Points File	

- 3. Set Acquisition Type to Linescan (Stage) (see the previous image).
- 4. Click **Add Location** and follow the wizard to define the line scan.

nalysis A	utomatio	n			-
Acquisition 7	Туре				
Linescan (S	Stage)	-			
Ð	Θ	Ø	+#	<b>1</b>	
Add Location	Remove Location	Edit Location	Save Points File	Load Points File	
Acquisition I	Locations				
No.		Label			
<b>V</b> 1		Linescar	1		

The software adds the location to the Acquisition Locations box.

5. Continue adding line scans as needed.

#### Note

- To remove a sampling location, click the row in the Acquisition Locations box and then click **Remove Location**.
- You can <u>edit the wizard settings</u> (including beam control, stage control, acquisition time, magnification and anchor point locations) and then redo a line scan by selecting its associated row in the Acquisition Locations box and choosing **Edit Location**.
- 6. Click **Start Automation** to start the analysis.



If Auto ID and Auto Quant are selected in Spectrum Processing and the known elements are set as Always Identified in the element setup pane (periodic table), the quant results are displayed in the results pane.

The acquired data have the same filename extension as data acquired in normal Linescan mode, except the Analysis Automation Sequence Name is appended to the experiment name. One file is produced for each analyzed location. Here is an example:

Auto Example[Point@1].lsctl Auto Example[Point@2].lsctl

The acquired data are listed in the file list pane (one Linescan file for each analyzed location). Here is an example:

	Auto Example[Point@1].PS.EDS	
-	Auto Example[Point@2].PS.EDS	

7. To save the data set in the current project, click the **Save** button **[]** on the main toolbar, or choose **File** (menu) > **Save**.

**Note** If Auto Quant is selected in Quant Setup on the Spectrum Processing tab, the quantitative results are automatically saved to a CSV (Comma Separated Values) file with a .csv filename extension.

## Linescan (Stage) Settings for Analysis Automation

You can edit the settings and then redo a line scan by selecting its associated row in the Acquisition Locations box and choosing **Edit Location**.

Acquisition Linescan (S	12	•			
Add Location	Remove Location	Edit Location	Save Points File	Load Points File	
No.		Label			

The Edit Positions box is displayed.

Edit Positi	ions
Number: Label:	1 Linescan Magnification: 1000 Update
	Beam Control Acquisition Time Microscope Raster 1.0 seconds Center Point
	Stage Control Step Size Mumber of Points 10
Position	X Y Z Rot ( Tilt (deg) Bank (deg)
First Last	0.00 0 0 0.00 0.00 0.00 0.00 0 0 0.00 0.00
	+>
	Accept

The following options are available in the Edit Positions box.

-	
Feature	Description
Number	Line scan number displayed in the Acquisition Locations box on the Analysis Automation tab and used in the filename for the acquired data
Label	Label displayed in the Acquisition Locations box on the Analysis Automation tab and used in the filename for the acquired data
Magnification	Shows the magnification used for the selected line scan. You can enter a magnification value or set the microscope to the desired magnification and click <b>Update</b> to read the new setting.
Beam Control	Specifies how each location is sampled. Available options:
	• <b>Microscope Raster</b> . Acquires spectra over the entire field of view and produces an average spectrum for each specified location.
	• <b>Center Point</b> . Acquires a spectrum in the center of the field of view for each specified location.
Stage Control	Specifies where each location is sampled. Available options:
	• <b>Step Size</b> . Distance in mm between each sampling point (center point to center point). The software determines the number of points sampled based on the distance between the specified anchor points and the beam diameter.
	• <b>Number of Points</b> . You specify the number of sampling points to include between the specified anchor points.
	<b>Note</b> : If you specify the number of points to sample, consider the distance between the anchor points and the beam diameter to avoid duplicate sampling.
Acquisition Time	Length of time in seconds to acquire data at each sampling location. An acquisition time of 1 second is typical.

 Table 5.
 Grid Settings for Analysis Automation

<b>F</b> (	
Feature	Description
Position table	Shows the current locations of the two anchor points used to define the line scan (focus and magnification settings are saved with both anchor points). Available options:
	• Add or change a point (+). Allows you to change the selected anchor point or redo both anchor points.
	To change an anchor point, move the stage to the new location (or click <b>Move Stage To Point</b> and adjust from there), select the anchor point you want to change and click the <b>+</b> button.
	To add an anchor point, first delete both anchor points and then add them in this order: beginning of line scan, end of line scan (both positions should be in focus).
	• <b>Remove Point</b> (—). Removes the selected anchor point from the Position table.
	• <b>Return To Stage Point</b> (->). Moves the microscope stage to the anchor point selected in the Position table.
Accept	Saves your edits
Cancel	Closes the box without saving your edits

Table 5.         Grid Settings for Analysis Automat	ion
---	-----

## **Analysis Automation in Spectral Imaging Mode**

To use Analysis Automation in Spectral Imaging Mode, you specify the points or areas to be sampled and the acquisition and analysis settings, including spectral processing and map processing.

When Spectral Imaging mode is selected, all three Acquisition Type options are available on the Analysis Automation tab:

- <u>Selected area</u>. You specify the locations to acquire a spectral image by moving the microscope stage and clicking the Add Location button.
- <u>Grid</u>. You specify a rectangular sampling area for each stage location and how the area is divided up and measured.
- <u>Circular</u>. You specify a circular sampling area and how the area is divided up and measured.

**Note** There is no need to manually acquire an electron image when Acquisition Type is set to Grid or Circular. The software performs this step automatically. For details on using the grid options for Analysis Automation, see To acquire and analyze a grid using analysis automation.

- To acquire and analyze a series of sampling locations in Spectral Imaging mode
- 1. Click the Spectral Imaging button on the <u>analytical mode toolbar</u> and set up your analysis.

This includes experiment name, electron image acquisition, spectral image (map) acquisition, Spectrum Processing and Map Processing (Counts, Quant or COMPASS, with or without phases).

2. On the Analysis Automation tab, set Acquisition Type to Selected Area, Grid or Circular.

Ana	alysis Automation	~
	Acquisition Type	
	Grid	

- 3. Move the microscope stage to the first location you want to analyze on the sample.
- 4. If you selected the Selected Area option in step 2 above, acquire an electron image and use the extraction cursor on the image pane toolbar to specify the points or areas to be analyzed. Then click Add Location on the Analysis Automation tab to add those points to the Acquisition Locations box.

If you selected the Grid (rectangular) or Circular option, click Add Location and follow the steps in the wizard to define anchor points (boundaries) for the rectangle or circle you want to sample. For more information, see To acquire and analyze a grid using analysis automation.

- 5. Move the stage to the next location and repeat step 4 to add the new point, area or grid to the Acquisition Locations box.
- 6. Continue moving the stage and adding sampling locations as needed.

**Note** To remove a sampling location, click the row in the Acquisition Locations box and then click **Remove Location**.

7. Click **Start Automation** and wait until the automated acquisitions are completed.



If Auto ID and Auto Quant are selected in Spectrum Processing, the found elements are displayed in the element setup pane (periodic table) and the quant results are displayed in the results pane. The quant maps are displayed in the maps pane.

The acquired data are listed in the file list pane (one Spectral Imaging (.eds) file for each analyzed location). Here is an example:

For selected area:

Auto Example[Point@1].MAP.EDS

For grid and circular:

Auto Example[Grid@X Y ].MAP.EDS

8. To save the data set in the current project, click the **Save** button **[**] on the main toolbar, or choose **File** (menu) > **Save**.

**Note** If Auto Quant is selected in Quant Setup on the Spectrum Processing tab, the quantitative results are automatically saved to a CSV (Comma Separated Values) file with a .csv filename extension.

# **Create a Montage From Analysis Automation Data**

You can use Analysis Automation to create a montage of electron images, X-ray images, COMPASS maps or phase maps by stitching together images acquired as adjacent fields in a grid automation sequence.

## **Create Montage of Electron Images**

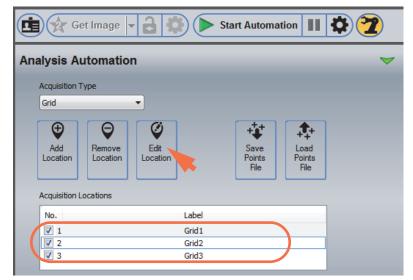
#### \* To create a montage of adjacent electron images

- 1. From the Pathfinder Home screen, choose **View** (menu) > **Electron Imaging Mode**.
- 2. In the Analysis Automation settings, set Acquisition Type to Grid.



3. Click **Add Location** and follow the steps in the wizard to define anchor points for the grid. For more information, see To acquire and analyze a grid using analysis automation.

4. Select the grid by clicking its associated row in the Acquisition Locations table and then click **Edit Location**.



5. In the Edit Positions box, set Frame Layouts to Adjacent and enter a small overlap (3% is a good start) and click **Accept**.

Edit Positions		
Number: 1		
Label: Gri	d	Magnification: 1000 Update
-Frame Layouts		
Adjacent	3	Overlap %
O User Grid	0	X Fields
	0	Y Fields

6. Click **Start Automation** and wait until the automated acquisitions are completed.



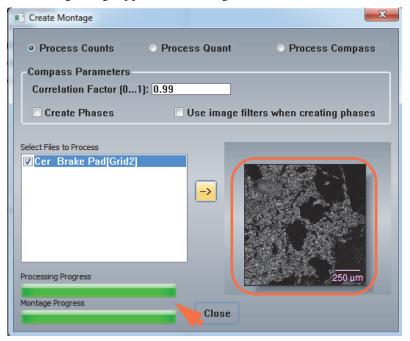
7. From the Home screen, choose **Batch Processing** (menu) > **Create Montage**.

8. In the Create Montage box, select your grid file in the Select Files to Process box (make sure its associated check box is selected).

Create Montage
• Process Counts • Process Quant • Process Compass Compass Parameters • Correlation Factor (01): 0.99
Create Phases Use image filters when creating phases
Select Files to Process Cer Brake PadJGrid2
Processing Progress
Montage Progress Close

- 9. Set Data Type to Process Counts (see the previous image).
- 10. Click the —> button to create the montage (see the previous image).

The two rate bars at the bottom of the Create Montage window indicate the progress and the montage image appears in the image box.



11. To save the montage in the current project, click the **Save** button **[]** on the main toolbar, or choose **File** (menu) > **Save**.

A montage consists of a .map file and a .tif for each element map and electron image. The map file is named:

BaseName[Grid,montage].map

Here is an example of how the elemental map TIFF files are named:

BaseName[Grid,Montage]\_Al\_K\_map.tif

Corresponding electron images are either

BaseName[Grid,Montage]\_Grey\_map.tif

or

BaseName[Grid,Montage]\_RefGrey.tif

## **Create Montage of X-ray Maps**

The steps to create a montage of X-ray maps are similar to creating a montage of electron images. You can use this technique to create a montage of X-ray count maps, X-ray quant maps, COMPASS components or phases. Start by creating a grid file in Analysis Automation. Then create the montage.

**Note** Keep in mind that grid files acquired in Spectral Imaging mode can be large. Each grid file is a complete spectral imaging (.eds) file.

#### ✤ To create a montage of X-ray maps

- 1. Click the Spectral Imaging button on the analytical toolbar.
- 2. Set up your spectral analysis, including acquiring the electron image and spectrum processing, and map processing.

Depending on the kind of montage you want to create, on the Map Processing tab, set **Map Type To Display** to Counts, Quant, COMPASS or Phases and set any associated parameters as needed.

3. In the Analysis Automation settings, set Acquisition Type to Grid.

Analysis Automation	▼
Acquisition Type	
Grid	

4. Click **Add Location** and follow the steps in the wizard to define anchor points for the grid. For more information, see To acquire and analyze a grid using analysis automation.

5. Select the grid by clicking its associated row in the Acquisition Locations table and then click **Edit Location**.

0	( + - )		
0	(++)		
dit ation	Save Points File	Load Points File	
	Label		

6. In the Edit Positions box, set Frame Layouts to Adjacent and enter a small overlap (3% is a good start) and click **Accept**.

Edit Positions			
Number: 1			
Label: Grid	ł	Magnification: 1000 Update	
Frame Layouts			
Adjacent	3	Overlap %	
🔘 User Grid	0	X Fields	
	0	Y Fields	

7. Click Start Automation and wait until the automated acquisitions are completed.



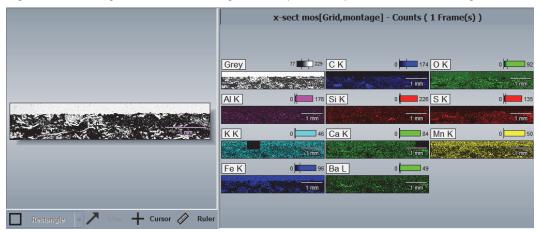
- 8. Choose Batch Processing (menu) > Create Montage.
- 9. In the Create Montage box, select your grid file in the Select Files to Process box (make sure its associated check box is selected).
- 10. Depending on the kind of montage you want to create, set **Data Type** to Process Counts, Process Quant or Process COMPASS.
- 11. If you want to create a montage of phase maps (Data Type must be set to COMPASS), select **Create Phases**.

12. Click the —> button to create the montage.

<ul> <li>Create Montage</li> <li>Process Cou Compass Para</li> </ul>	meters	<ul> <li>Process Compass</li> </ul>
	ctor (01): 0.99	
Create F Select Files to Pro	Spectral Imaging Resolution: 512 x 384 Kernel Size: 9 x 9 Level of Quant Map Detail: Medium Estimated Time Remaining: 10 seconds	ig phases
Processing Progress Montage Progress	Close	

The two rate bars at the bottom of the Create Montage window indicate the progress. The spectral imaging files are processed first and then added to the montage, which slowly appears in the image box as the montage is created.

- 13. To save the montage in the current project, click the **Save** button **[]** on the main toolbar, or choose **File** (menu) > **Save**.
- **Figure 14.** Montage created from a 7x2 grid of X-ray count maps and the electron image



	x-sect mo	s[Grid@7 1] - A	Atomic % ( 10 I	Frame(s) Kerne	l Size = 7)
	Grey	СК	<u>0 K</u>	Na K	Mg K
	4100µm	100.µm	100 µm	100 µm	<b>π</b> 100 μm
	AIK	Si K	<u>S K</u>	CIK	КK
	100 µm	ν 100 μm	100 µm	100 µm	100 µm
	Mn K	Fe K	Sb L	BaL	
100 µm					
Rectangle 🗸 👗 Line 🕂 Cursor 🔊 Ruler	100 µm	100 µm	— — 🦻 б 🔨 — 100 µḿ	100 µm	

Figure 15. Montage created from a grid of X-ray quant maps

# **Troubleshoot Analysis Automation**

## **Minimize Montage Edge Mismatch**

To minimize any mismatch at the edges of the images in a montage, double-check the following:

- Make sure the X axis of the microscope scan is aligned with the X-axis of the stage. Use the microscope's scan rotation to align these movements together. This can be done by putting a feature at the top of the scan and moving the stage back and forth until the feature remains at the same Y position as the stage is moved. Adjust the scan rotation if this is not the case.
- Make sure the Pathfinder X and Y scan are calibrated correctly for the selected magnification. If the pixel size and squareness of the pixels are incorrect, the amount of stage motion associated with the scans field width will be incorrect. Make sure the stage backlash correction has been set up properly (must be done by a qualified service engineer). Also, try increasing or decreasing the overlap percentage setting in the grid location settings on the Analysis Automation tab.

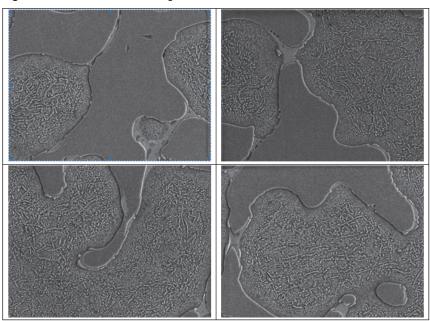
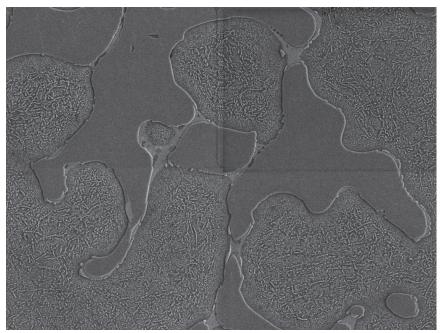


Figure 16. Individual Grid Images

Figure 17. 2x2 Montage Properly Aligned



## Problems with Point Registration for Point ID or Linescan Data

If you experience a problem with point registration when using Point ID or Linescan mode with Analysis Automation, one of the following may be the cause:

- The stage is not correcting for backlash. Make sure the stage supports backlash correction. If it does, consult the stage manufacturer to ensure backlash correct is set up properly. If the stage does not support backlash correction, contact our Technical Support for information about enabling backlash correction in the Pathfinder automation routines.
- Backlash was not corrected properly before acquiring the reference image and indexing the points. Index the stage location in the points table, tell the software to return to the previously defined stage position, acquire an image, set the desired location, and update the previously set locations.

**Note** If you combine Point ID or Linescan with Analysis Automation and Drift compensation, make sure Drift Compensation is enabled (set to **Drift On**) before you acquire the reference image at each stage location. You cannot mix stage locations that use drift compensation with stage locations that do not.

## 9 Analysis Automation

Troubleshoot Analysis Automation

# 10 -

# **Drift Compensation**

In an electron microscope, the image can shift during an acquisition, especially when using high magnifications. Drift can be caused by slight movement of the microscope stage or electronic drift in the lens and is dependent upon your microscope configuration. Depending on the severity, drift can significantly affect the integrity of the data and the accuracy of your results (qualitative and quantitative). Our optional Drift Compensation software uses precise hardware beam control to monitor image position over time and immediately correct any drift that occurs during an acquisition.

There are two Drift Compensation operating modes:

- Simple. The area used to track image drift is the same as the area being analyzed.
- **Rect Drift**. You define a rectangular area for image tracking, which is typically in a different location than the feature you want to analyze. This enhances drift compensation when the sampling location has low contrast. (The user can select an area with higher contrast to monitor and compensate for any drift.)

Drift Compensation can be used in the following analytical modes and works similarly in all of them:

- Point ID
- Spectral Imaging
- Linescan

# **Set Up Drift Compensation**

## Set Up Simple Drift Compensation

Simple drift compensation uses the same area to track image drift as the area being measured.

- \* To set up simple drift compensation in Point ID, Spectral Imaging or Linescan mode
- 1. Click the Drift Compensation icon on the acquisitions toolbar and select Drift On.



The icon changes from an "open lock" (drift off) to a "closed lock" to indicate that simple drift compensation is enabled.



- 2. Click the Electron Image Setup 😵 button on the acquisitions toolbar (see the previous image) and adjust the <u>image acquisition settings</u> and <u>drift compensation settings</u> as needed.
- 3. Click Get Image to acquire an electron image.

Image tracking starts immediately with a new electron image acquired after the specified interval. See Acquire Data Using Simple Drift Compensation for more information.

4. Click the Experiment Setup 😧 button and adjust the mapping, line scan or spectral acquisition settings as needed.



5. Click **Start Map** (Spectral Imaging mode), **Start Spectrum** (Point ID mode) or **Start Linescan** (Linescan mode) to begin the spectral (or spectral imaging) acquisition.

## Set Up Rectangular Drift Compensation

Rectangular Drift compensation uses a user-defined (rectangular) area for image tracking, which is typically in a different location than the feature you want to analyze. This enhances drift compensation when the sampling location is featureless or has low contrast.

#### \* To set up rectangular drift compensation

1. Click the Drift Compensation icon on the acquisitions toolbar and select Rect Drift On.



The icon turns green to indicate that Rect Drift is enabled.



- 2. Click the Electron Image Setup 😥 button on the acquisitions toolbar (see the previous image) and adjust the <u>drift compensation settings</u> and the other electron image acquisition settings as needed.
- 3. Click Get Image on the acquisitions toolbar.

The drift compensation icon changes to red and the Set Image button is displayed.

Click to exit Rect Drift setup mode

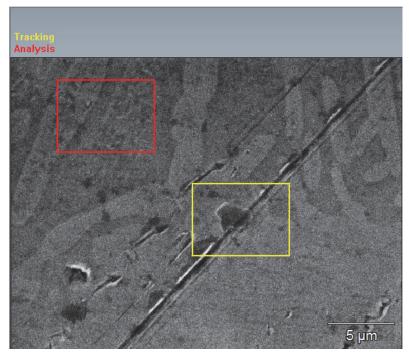


The software acquires an electron image and displays it in the image pane along with two rectangles (yellow and red).



4. Drag the red rectangle to the area you want to analyze; drag the yellow rectangle to the area you want to use for image tracking.

It is important to select an area that includes unique features and some contrast for image tracking. Here is an example:



**Note** Keep in mind that the boundaries for image tracking are defined by the boundaries of your current field of view. If your system has significant drift, position the yellow rectangle as close as possible to the center of the image. To increase the boundaries, increase the Zoom Factor setting in Electron Image Setup. However, keep in mind that this automatically decreases the maximum resolution that can be used in the analysis area. See Zoom Factor for more information.

5. Click Set Image on the acquisitions toolbar.

Image tracking starts immediately with a new electron image acquired after the specified interval. See Acquire Data Using Rect Drift Compensation for more information.

6. Click the Experiment Setup 😧 button and adjust the mapping, line scan or spectral acquisition settings as needed.

💼 🔯 Get Image 🗸 🔒 🏟 🔯	Start Map	- 7 0 7

7. Click **Start Map** (Spectral Imaging mode), **Start Spectrum** (Point ID mode) or **Start Linescan** (Linescan mode) to begin the spectral (or spectral imaging) acquisition.

## **Drift Compensation Settings**

To adjust the drift compensation settings, click the Electron Image Setup 😟 button on the acquisitions toolbar and then click the green arrow to show the advanced view.



SettingDescriptionCompensation IntervalDetermines how often the software should check the position of<br/>the electron image and correct for any movement.Available options:Available options:• Automatic with min time of \_\_\_\_\_ seconds. The software<br/>acquires the electron image used for drift compensation each<br/>time the minimum time is exceeded. If the software<br/>determines that the image is drifting at a faster or slower<br/>pace than the entered value, it adjusts the interval based on<br/>the actual drift rate to achieve a maximum of one pixel<br/>change per correction.

The settings that affect drift compensation are described below.

Setting	Description
	• Manual with fixed time of seconds. The user specifies how often to acquire the electron image used for drift compensation.
	• Current mapping resolution gives a minimum Map and SI interval of seconds. Reports the minimum time required to complete a Spectral Imaging acquisition based on the current settings for Resolution and Frame Time in Spectral Image Setup and the specified drift compensation interval (see above). For example, if Compensation Interval is 60 seconds and Frame Time is 5 seconds, the calculated interval is 69 seconds for one drift check. (Due to the complexity of the operation, the calculations for drift compensation require a few seconds to complete.)
	<b>Note</b> : In Spectral Imaging mode, the software can perform image correction only at the end of a frame. The entered minimum (automatic) or fixed (manual) value should always be higher than the reported "minimum Map and SI interval." (The software defaults to the reported minimum.) To compensate at a faster rate, decrease total frame time by decreasing the Resolution or Frame Time setting in Spectral Image Setup.
Zoom Factor	Defines the boundaries of the area used to track image shift by adjusting the magnification. The zoom factor ranges from 2 to 8 in powers of 2. Use a higher zoom factor setting if your system has a lot of drift. The higher the zoom factor, the larger the area used for drift compensation and the lower the resolution used for the sample data. For example, when Zoom Factor is 1, the maximum resolution setting available for the sample data is 4096. In the same way, when Zoom Factor is 2, 4, or 8, the maximum sample resolution is 2048, 1024 or 512 respectively.

# **Acquire Data Using Simple Drift Compensation**

Simple drift compensation uses the same area to track image drift as the area being measured. To turn on simple drift compensation, click the **Drift Compensation icon** on the acquisitions toolbar and select **Drift On**.

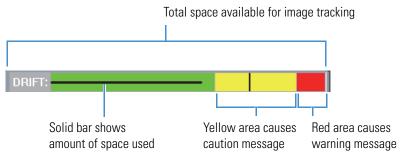


When Simple drift compensation is used, the software periodically acquires a new electron image and measures how far it has shifted from the original one. If the image has shifted at least one pixel, the software immediately uses beam control to correct it.

In Spectral Imaging mode, a concurrent gray level image is used for the simple drift correction if that option is selected in <u>Spectral Image Setup</u>. If the Concurrent Image option is not used, the Compensation Interval setting in Electron Image Setup determines when the drift correction is applied, as it does in Point ID and Linescan mode. When the Compensation Interval setting is used to trigger image tracking, the software pauses spectral acquisition while it acquires a new image and corrects for any shift that has occurred. Then spectral acquisition resumes. The frequency of image tracking affects the overall acquisition time.

The Zoom Factor setting in Electron Image Setup defines the boundaries of the area used to track image shift by adjusting the magnification. In the default configuration (Zoom Factor is set to 4), the image can drift off the screen at the top, bottom, left, or right and still be tracked. When the hardware reaches a boundary, drift compensation stops and the software displays a message that it can no longer correct for drift. To increase the boundaries, use a higher Zoom Factor setting.

The drift compensation status bar at the top of the Pathfinder Home screen indicates the amount of space remaining for drift correction. Here is an example:





# Acquire Data Using Rectangular Drift Compensation

Rectangular drift compensation uses a user-defined (rectangular) area to track image drift, which is typically in a different location than the measured feature or area. This enhances drift compensation when the sampling location has low contrast. (The user can select an area with higher contrast to monitor and compensate for any drift.)

To enable rectangular drift compensation, click the Drift Compensation icon on the acquisitions toolbar and select **Rect Drift On**, then acquire an electron image and select the areas used for image tracking and sample analysis. See Set Up Rectangular Drift Compensation for step-by-step instructions.



Rect Drift compensation works the same as simple drift compensation except it does not use the gray level image in Spectral Imaging mode, even when the Concurrent Image option is selected in Spectral Image Setup.

**Note** Do not use Rect Drift Compensation with <u>Analysis Automation</u>.

# **Turn Off Drift Compensation**

To turn off drift compensation (simple or rectangular), click the Drift Compensation icon on the acquisitions toolbar and select **Drift Off**.



**Note** If you combine Point ID or Linescan with <u>Analysis Automation</u> and Drift compensation, make sure Drift Compensation is enabled (set to **Drift On**) before you acquire the electron image at each stage location. You cannot mix stage locations that use drift compensation with stage locations that do not.

# **Drift Compensation Diagnostics**

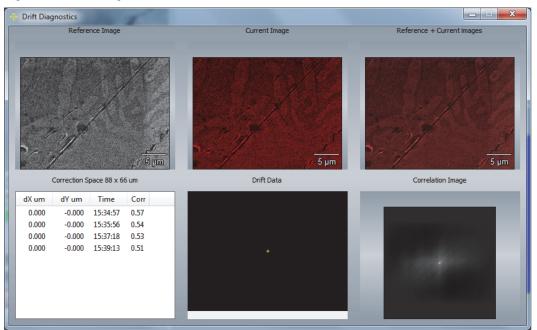
The software provides diagnostic information for drift compensation to help you monitor its activity during sample acquisitions. The Drift Diagnostics display information about drift compensation, such as the amount the electron image has drifted over time, how much space is available for image tracking, and how the most recently acquired correction image compares with the original one.

## To view drift diagnostics

1. From the Pathfinder Home screen, choose View (menu) > Drift Diagnostics.

The Drift Diagnostics window is displayed.

Figure 19. Drift Diagnostics Window

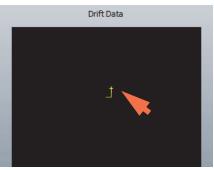


The six labeled sections are explained below:

- **Reference Image**. Original (gray) electron image used for sample acquisition and analysis.
- Current Image. Most recently acquired (red) electron image used for image tracking.
- **Reference + Current Image**. Most recently acquired tracking (red) image overlaid on the original (gray) image. When drift compensation is working properly, there should be very little difference between the two images at any time.
- **Correction Space**. Provides a detailed history of every correction since drift compensation was enabled. It shows the time of each image tracking measurement, the movement that occurred along the X or Y-axis in µm, and the correlation value. The correlation value indicates how well the current tracking image matches the reference (original) image, where 1.0 is a perfect match. If the two images are totally different, the reported correlation coefficient is 0.0 and Drift Compensation stops running.

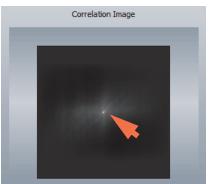
• **Drift Data**. Demonstrates how the image has moved over time. If the image has not moved, a white + appears in the center of the pane. If the image moves one or two pixels in any direction, the plus sign is extended with a small arrow indicating the direction of movement. Here is an example:





Drift Data also shows the remaining hardware space available for image tracking. For example, when the path reaches any edge, image tracking stops and the software displays a message that it can no longer correct for drift. To increase the boundaries, increase the <u>Zoom Factor</u> setting in Electron Image Setup and restart the analysis.

- **Correlation Image**. Shows the correlation between the current tracking image and the reference (original) image. When the two images are aligned perfectly, the brightest spot is in the center of the correlation image with no shift. Here is an example:
  - Figure 21. Correlation Image Showing Two Perfectly Aligned Images (brightest point is dead center)



If the image shifts by one pixel to the right, the brightest pixel in the correlation image will be one pixel to the right of the center pixel. Here is an example:

**Figure 22.** Correlation Image Showing Movement Along the X-axis (brightest point has shifted right)



**10 Drift Compensation** Drift Compensation Diagnostics