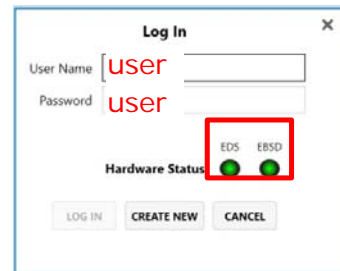


Guide to EDAX EDS and/or EBSD Data Collection using APEX Suite

I. Launching APEX

- From the windows desktop double click on the APEX™ application icon to start the software: APEX EDS, APEX EBSD, or APEX Suite which is combined EDS and EBSD.
 - Type username: **user** and password: **user** to log in to the desired application.

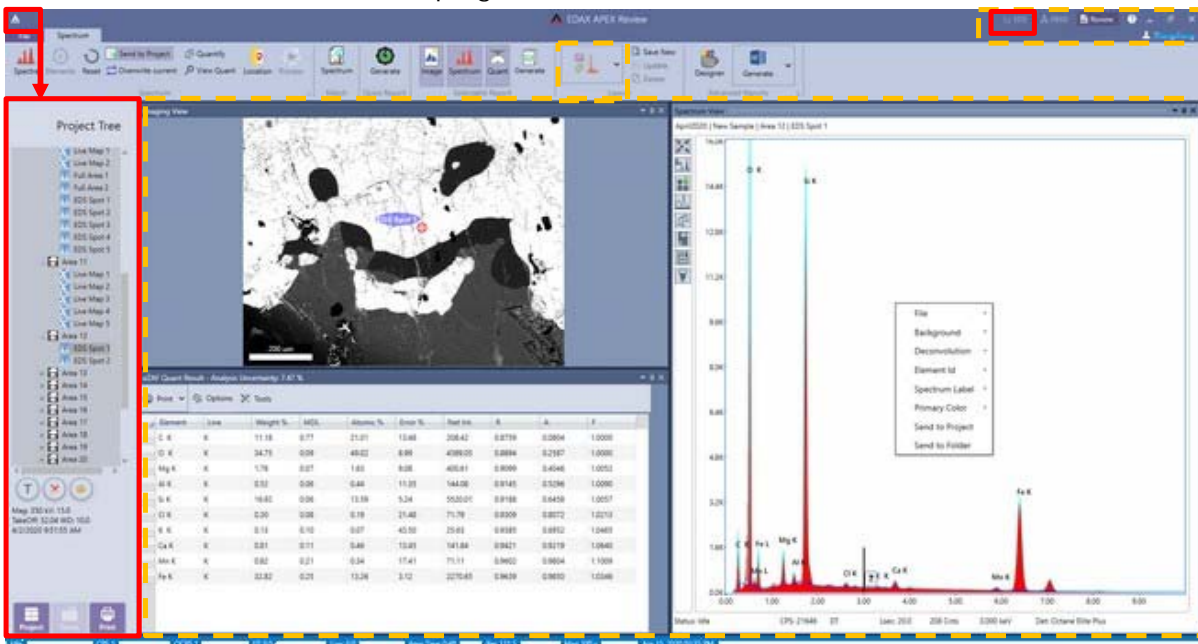


- When the application is ready, an existing project can be selected to review or examine data, or a new project can be created.
 - To create a new project, click **Create>Project**. Then, the project can be named or left as the default: "New Project," then click OK. (Note: choose drive **(UserFile)G:\User-Data**, **DO NOT** select drive C:\ or F:\ as your data destination)
 - To change modes between EDS, EBSD and Review, click on the desired button in the top right of APEX window title bar.
- If you run EDS analysis and the EDS detector is not cooled down, the cooling must be turned on by clicking the **Detector Cooling** icon on the Collection Toolbar.



II. Layout Overview

The title of the window displays which mode APEX™ EDS is in. It has two states: a live collection window, and a review window. EDAX APEX™ EDS is the data collection window, while EDAX APEX™ Review is where you can review all types of data and produce reports. Click the EDS icon or Review icon on the top right corner to switch between the two modes.



III. Image and Data Collection

1. Image Collection

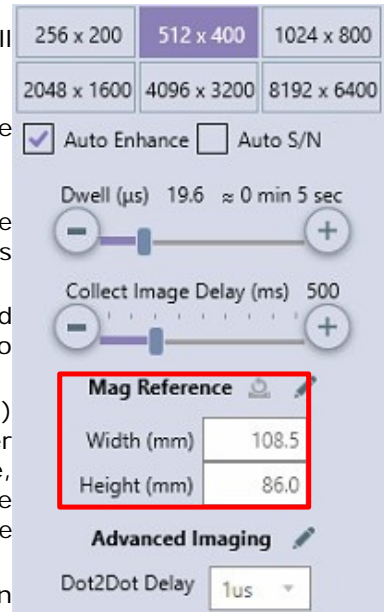


1.1 Click the camera icon, , on the image toolbar starts or stops image collection.

Clicking on the down arrow selection (on the image icon) will display the Image Collection Parameters pop up window.

The quality of the image can be adjusted further using the following settings:

- Auto Enhance: This option automatically adjusts the image brightness and contrast after the image is collected. It yields nicely contrasted images.
- Auto S / N: This option pre-scans the image area and automatically determines the Image Dwell Time number to acquire a low noise (High Quality) image.
- Dwell(us): The imaging dwell time (in microseconds per pixel) can be selected by the user. The larger the number, the longer the collection time, but this yields lower noise and therefore, better quality. Clicking or sliding the scroll bar shows the time per point and estimated frame time update. Note: When using the Auto S / N feature, dwell time is selected automatically.
- Collect Image Delay (ms): This setting is the delay time (in ms), set by the user, for the microscope to settle between the external control of the beam and image acquisition.
- Mag Reference: **DO NOT ATTEMPT TO ADJUST IT!** The adjustment is done at initial installation and checked as part of a preventive maintenance visit.



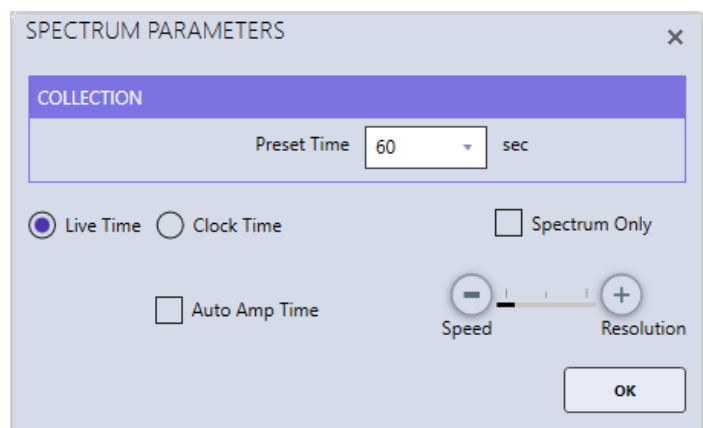
2. Spectrum Collection and Settings

After collecting an image, click on the image area to define a spot, or area(s) over which the spectrum will be collected. If multiple areas are defined, the software will collect spectra at each

of them sequentially. Click the **Collect Icon**, , to start the spectrum acquisition.

Spectrum Parameters:

The right-side drop-down menu, next to the Spectrum Quality Icon, allows the user to select one of the four quality modes for data collection: Quick, SD (standard definition) HD (high definition) and Manual. The collection parameters can be customized by the user for each collection mode. When in spectrum mode, the settings for each quality mode can be modified by clicking the arrow next to the word Spectrum on the bottom of the collection tool bar. A pop-up window appears in which the user can set a collection time and choose between Live and Clock time.

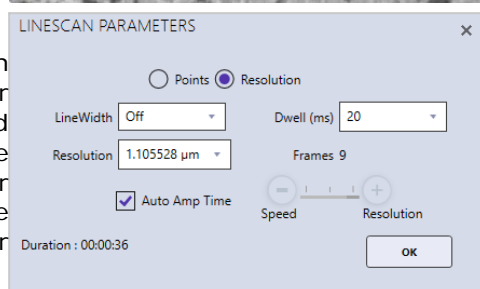
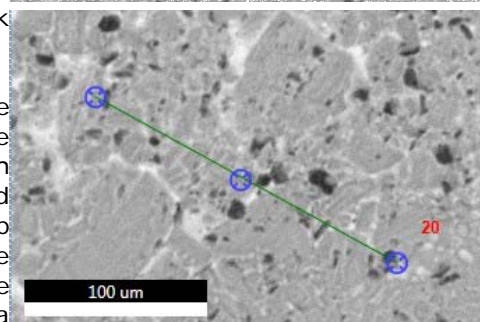
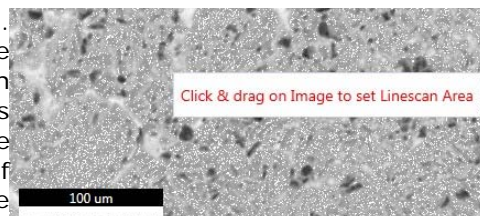


3. Linescan Collection and Settings

Clicking on the **Linescan** Icon enters the linescan mode. A message will pop up over the image area asking the user to draw a line on the image to define the linescan profile for collection. After drawing the line, the end points of the line can be dragged to change the length of the line. To move the line, drag the blue circle in the middle of the line. To delete the line, move the cursor onto the line (outside of the blue circles) and a stop sign appears. Click the stop sign to delete the line.

Once the line is defined, click the Collect Icon to start the linescan acquisition. The software will pre-scan the line selected and determine which elements are present, which will be shown in a pop-up box along with the estimated collection time. Clicking Confirm Elements in the pop-up box will immediately display the detected elements on the periodic table where the user can add and remove elements as desired. Note: If the element list is locked, a spectrum preview will not be displayed.

The right-side drop-down menu, next to the Linescan Quality Icon, allows the user to select one of the four quality modes for data collection: Quick, SD, HD, and Manual. In quick mode the software will aim to acquire 1,000 X-rays per point on average while the numbers for SD and HD are 10,000 and 20,000 respectively. The collection parameters can be customized by the user for each collection mode.



Linescans can be acquired in two possible stepping modes: Points or resolution. In Points mode, the number of points over which data will be collected is manually entered by the user. Enter the number of points and the click Enter. In Resolution mode, select a resolution value from the drop-down list or type in a number and number of points used will be calculated automatically.

- **Line Width:** To scan a line perpendicular to the linescan instead of individual point locations, change the line width. This is useful for obtaining a more accurate intensity average for the elements. If the desired line width value is not in the drop-down list, type in the value and press enter.
- **Dwell (milliseconds):** The data collection time per point per frame. If the desired dwell time is not in the drop-down list, type in the value and press enter.
- **Resolution:** When using the Resolution radio button selection, this will select the distance between each point collected.
- **Frames:** The number of integrated frames that will be collected. In manual mode, the user can select the number of frames to collect. In the other three modes (Quick, SD, or HD), the software will automatically calculate the number of frames to acquire the pre-defined level of data quality. For High, the software will collect an average of 20,000 X-rays per point, for standard 5000 X-rays and for quick 1000 X-rays. If the desired frame number is not in the drop-down list, type in the value and press enter.
- **Amp Time Selection:** In the automatic quality modes (Quick, SD, HD) there is a check-box for Auto Amp Time which automatically sets the amp time based on the input counts per second in order to balance throughput and resolution. When the Manual quality icon is selected or when Auto Amp Time is deselected in the automatic modes, the pop-up window displays a slider bar with Speed and Resolution. Drag the slider bar or click the plus and minus symbols to adjust the amp time.
- **Duration:** The estimated time required to collect the linescan based on the given parameters.

4. X-Ray Map Collection and Settings

4.1 Map Collection and General Settings

After collecting an image, click on the Mapping Icon to enter mapping mode. By default the entire image will be mapped. You can also change the area, which is discussed below. Once the area is defined, click the Collect Icon to start the map acquisition. The software will pre-scan the area selected and determine which elements are present, which will be shown in a pop-up box along with the estimated collection time, shown below. Clicking Confirm Elements in the pop-up box will immediately display the detected elements on a periodic table where the user can add and remove elements as desired. Additionally, the red delete icon next to each element map can be used to remove an element. Note: If the element list is locked, a spectrum preview will not be displayed.

Map Starting

Mapping duration approximately 1 Mins / 38 Frames, but you can stop the data collection at any time.

Surveying collection region before commencing. This will take no longer than 30 seconds. Click Stop button to abort collection.

Elements found so far: C K α , O K α , Na K α , Mg K α , Br L α , Si K α , Ca K α , Fe K α

Confirm elements after preview

During the collection, the X-ray maps will be displayed in real-time, illustrated in the figure below. Clicking the arrow icon in the top right corner of the Multi-map window will toggle the display to show the default layout or the custom zoom layout. In the custom zoom layout the slider control beneath the arrow icon changes the amount of zoom. The status bar provides useful information about the collection, including: the frame number being collected, the time to collect a frame, the time remaining in the map collection, and a progress bar. A line showing the current frame progress can be turned on by right clicking the main map window and selecting Show Progress Line.

The screenshot displays the software interface during map collection. At the top, there's a toolbar with various icons. Below it, a 'Multi-map' window shows several small X-ray maps of different elements. A larger 'Spectrum View' window is open, showing an 'Area Spectrum from Manual | Sample1 | Area 9 | Live Map 1 | Pixels:2332 Location 191,113 - 244,157'. The spectrum plot shows intensity versus energy (keV) with peaks labeled for various elements like O K, Al K, P K, Zr L, Mo L, K K, Ca K, Sc K, Ba L, V K, Mn K, and Er L. Below the spectrum, an 'Imaging View' window shows a grayscale image of a sample with a green box highlighting a region of interest labeled '2332 Pixels'. A 200 μm scale bar is visible at the bottom of the imaging view.

During collection it is possible to extract from a spot or an area by interacting with the Imaging View. The extracted spectrum is overlaid in the summary spectrum, shown below. The summary and extracted spectra will be updated on periodic basis. To extract from a location, just click for spot and click-drag for area.

To stop the map data collection, click the Stop icon next to the collect image icon. This can be done at any point during the collection and the following menu of options will appear.

The right-side drop-down menu next to the Map Quality Icon, allows the user to select one of the four quality modes for data collection: Quick, SD, HD, and Manual. In Quick mode the software will aim to acquire 300 X-rays per pixel on average while the numbers for SD and HD are 600 and 1,000 respectively. The collection parameters can be customized by the user for each collection mode. To set the map parameters, click the arrow next to the word Mapping on the bottom of the collection toolbar in mapping mode and a pop-up box will appear.

MAPPING PARAMETERS

Resolution 512 x 400 Dwell (μs) 200

Frames 67

Auto Amp Time

Speed
-
+
 Resolution

Duration : 00:53:44

OK

- Resolution: There are five different collection matrices that can be used for mapping: 64x 50, 128 x 100, 256 x 200, 512 x 400, and 1024 x 800 pixels. Use the drop-down menu to select a resolution value.
- Dwell (μseconds): The data collection time per pixel per frame. If the desired dwelltime is

not in the drop-down list, type in the value and press enter.

- **Frames:** The number of integrated frames that will be collected. In manual mode, the user can select the number of frames to collect. In the other three modes (Quick, SD, or HD), the software will automatically calculate the number of frames to acquire the pre- defined level of data quality. If the desired frame number is not in the drop-down list, type in the value and press enter.
- **Amp Time Selection:** In the automatic quality modes (Quick, SD, HD) there is a check-box for Auto Amp Time which automatically sets the amp time based on the input counts per second in order to balance throughput and resolution. When the Manual quality icon is selected or when Auto Amp Time is deselected in the automatic modes, the pop-up window displays a slider bar with Speed and Resolution. Drag the slider bar or click the plus and minus symbols to adjust the amp time.

5. Data Analysis and Display

5.1 Quant View Window

While data is collecting, the quant results will automatically appear and update in the quant window, designated by the user in layout settings. There are multiple options for viewing quant results including: numerics only, graphs, and statistics. Be aware that any accurate quantitative analysis requires the spectra to originate from a flat, homogenous bulk sample.

The numerics only view displays the quant results in a table which can be customized by the user. Right click on the Quant Results Window to turn on or off (via a check mark) the quant values that will be used for display and reports. The Single Decimal Quant option sets the weight and atomic percentages to just a single decimal point of accuracy. The default display setting is two decimal points, but be aware that this does not imply that the quant results have two decimal points of precision.

Element	Line	Weight %	Net Int.	R
K K	K	8.26	1331.09	0.9
Si K	K	19.39	5637.21	0.9
Al K	K	7.06	2024.76	0.9
O K	K	41.90	2713.40	0.9
Na K	K	0.67	105.60	0.9
C K	K	22.64	307.80	0.9
Ca K	K	0.08	10.89	0.9

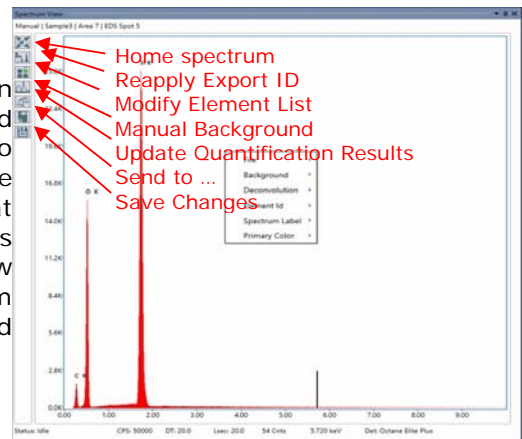
Context Menu Options:

- Weight %
- Atomic %
- Error %
- Net Int.
- RAF factors
- Single Decimal Quant
- Auto-hide R zero values

- **ID Confidence:** You can see in the top right corner of the spectral collection window; the ID Confidence % value is displayed during the collection.
- **MDL:** The Minimum Detectable Limit for each element. This is displayed on the QuantWindow during the collection and in review mode.

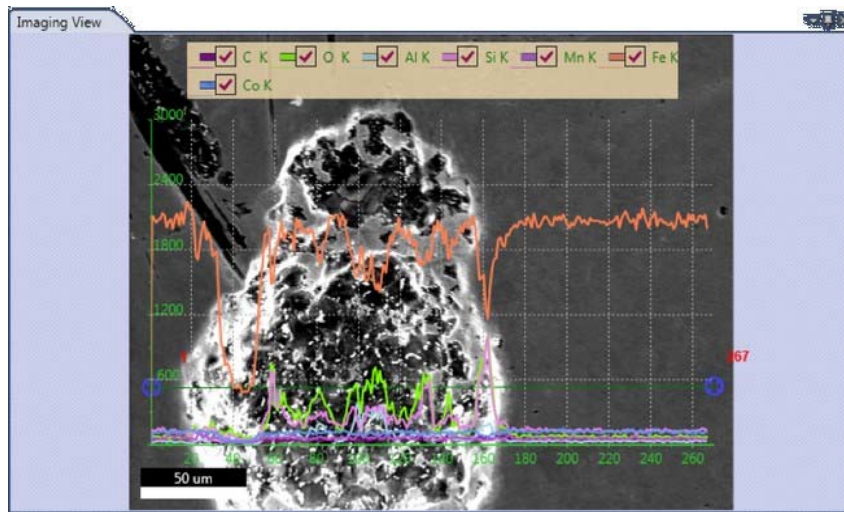
5.2 Spectrum View

During or after a spectrum is collected, the user can alter the Peak ID in order to identify trace elements and other elements of interest or to remove elements. To Add an elemental label, left click on the peak in the spectrum, hover the cursor over the question mark that appears, and a window containing the possible elements at that energy will be displayed. This Peak ID window can be dragged to different locations on the spectrum view window and will be displayed until manually closed using the X icon in the top right corner.



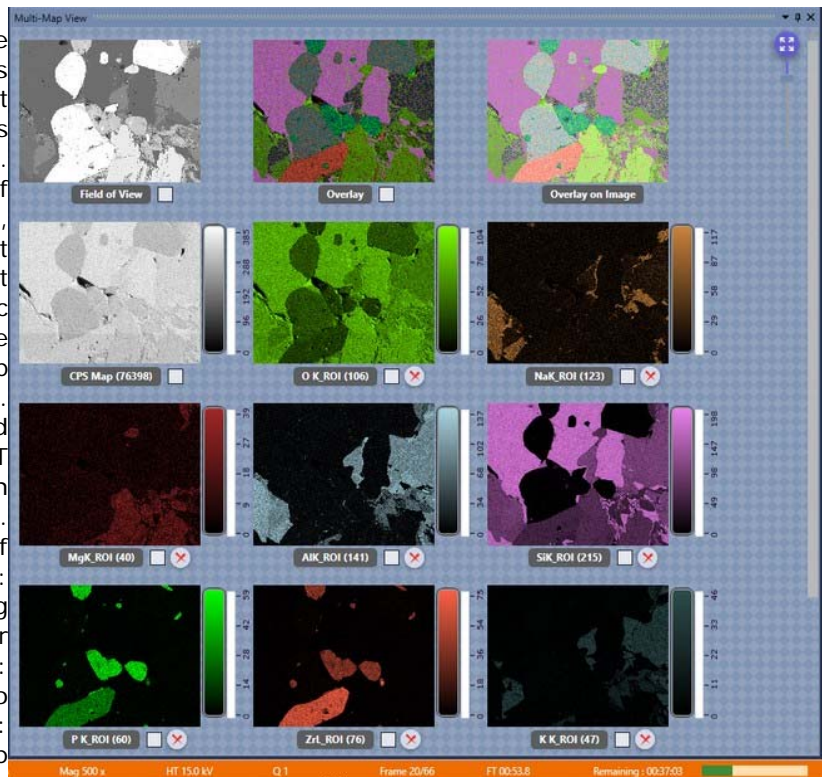
5.3 Linescan View-> Image Window

When in linescan mode, the spectral lines of each element appear in the image window, overlaid on the image. The user can check or uncheck the boxes on the top of the image to display the profiles of each element.



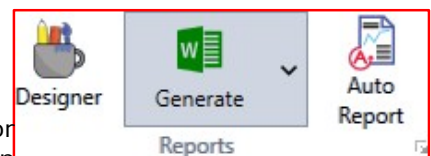
5.4 Multi-map View Window

When in mapping mode, the Multi-Map View window displays the different types of maps that the software compiles, as thumbnails in the same window. The name, data type (Field of View/SEM image, CPS map, region of interest (ROI), net intensity (NET), weight percentage (Wt %), or atomic percentage (At %)), and the maximum intensity of each map is displayed below the map. Maps are automatically collected in ROI mode or optionally NET but can be post processed in ROI, NET, Wt%, or At% mode. The status bar, on the bottom of the screen, displays the Frame: frame number currently being collected over the total number of frames, the frame time (FT): how long each frame takes to collect, the Remaining dialogue: the time remaining for map collection, and a Progress Bar: a visual representation of how much of the map is completed.



6. Reporting and Printing

APEX™ allows users to easily create reports in Microsoft Word or PowerPoint, while still in livemode. The report icons in the top



right corner of the collection toolbar, enable the user to generate a report using pre-existing templates or design a template. To use a pre-existing template, select one of the options from the drop down menu. A preview of the report will be displayed as the mouse is hovered over each option. Clicking the Report Icon will start report generation and orange dots will appear under

the icon while the report is loading.

7. After Data Collection

- 1) Retract the EDS detector by clicking Move Out button from the Spectrum drop down menu.
- 2) Set the EDS detector to Standby by clicking Detector Cooling button.
- 3) Vent SEM chamber and remove sample.
- 4) Pump the SEM chamber to high vacuum.
- 5) Log off your SEM session.