

Oxford EDS Instruction Manual

— Digital Imaging and Spectra Collecting with INCA

In emergency, please contact:

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1. In SEM system:
 - (a) Select the desired Accelerating Voltage: for EDS, the recommended accelerating voltage is 1.5 to 2 times the highest energy line in the spectrum. *For example: the Cu K line appears at 8.04keV, so the proper accelerating voltage for a sample in which copper is the highest energy line would be 12 – 16kV. For unknown sample, 20kV is a good starting point, which will excite most X-ray lines from most elements for analysis.*
 - (b) Choose the Objective Aperture 3 for EDS.
 - (c) Set the working distance (WD) to 10 mm.
 - (d) Get a high quality sample image by fine alignments and adjustments.
 - (e) **Make sure the IR camera is turned off** by minimizing or closing the IR camera window before collecting any spectra. Otherwise the deadtime will 100% during spectrum collection.
 - (f) When collecting EDS spectrum, adjust the Spotsize so that the deadtime is about 30-40%.
2. Log in to EDS computer.
3. Open the INCA software by double-clicking the **[INCA]** icon on the desktop.
4. There are three analysis options for the program:
 - (a) **[Analyzer]** – allows for EDS acquisition and quantitative analysis but no imaging acquisition.
 - (b) **[Point & ID]** – allows image acquisition and site specific EDS analysis.
 - (c) **[Mapping]** – allows for EDS mappings and linescans using a data mining approach (i.e. a full spectrum is stored from each pixel)
5. Within each project file you may have multiple samples. For each sample, you may have multiple sites of interest, and each site of interest is an image with various associated spectra.
6. Following are basic instructions, but more detailed information can be readily obtained from the **[Show bubble]** help icon (upper right corner of the screen).
7. Image capture:
 - (a) Get high quality SEM image and select [Scan 2].
 - (b) Click the **[Image Setup]** → select the desired Image Resolution (512×384), Speed (medium) and Data (8 bit), and input the number of Frames (1). In the parentheses are recommended normal settings.
 - (c) Click the **[Site of Interest]** menu and click on the green button to acquire an image.
8. EDS acquisition:
 - (a) Click **[Acquisition Setup]** → set the desired Livetime, Process time (1-6, 5 and 6 for high resolution, 3 or 4 for normal spectra and mapping), Spectrum range (determined by the accelerating voltage), and Number of channels. *A lower process time allows for faster collection with higher acquisition rates at the expense of energy resolution (i.e. wider peaks).*

- (b) Click **[Acquire Spectra]** → select region(s) of the sample to analyze. In Point & ID you can use the point, box, etc. tools, but in Analyzer mode there is no scan control so the system collects X-rays from the whole area.
- (c) Click **[Confirm Elements]** to verify the peaks.
9. Quantitative analysis:
- (a) Click **[Quant setup]** → set Processing Option (All elements), check Normalize Quantitative Results, set Element List (Current spectrum).
- (b) Click **[Quant]** to get the quantitative results, choosing different tabs to select different display methods.
10. EDS mapping:
- (a) In the **[Mapping]** option, collect an image as per the image capture instructions above.
- (b) Click **[SmartMap Setup]** → set Map or Linescan Resolution (256×192); Conditions: Process time (3), Spectrum range (0-20 keV), and Number of channels (2k); and Acquisition Time: choose fixed duration, Frame (20), map dwell (100μs), linescan dwell (2000μs).
- (c) Click **[Element Setup]** to select the elements for mapping or linescan.
- (d) Click **[SmartMap]** → select a region to map or draw a line to scan. Click the green button to begin acquisition.
- (e) Click **[Element Maps]** or **[Element Linescans]** to show the mapping or linescan.
11. Save the project file to D:\data\. You can create your own folder **only** under this directory. To save any images, maps or EDS spectra, right click on them and select Export. For image format, please choose JPG for smaller file size. And EDS spectra can be exported as EMSA file (a text file) which can be plotted using software such as Excel or Origin. The linescan and mapping data can be exported as “.TSV” file (right click and select “Export Data Only”, then select “.TSV” format) to be plotted using other software.