

# Oxford INCA EDS

## Operating Check List for SEM

- 1) Follow the SEM checklist and obtain an image of your sample. Some special conditions for EDS analysis apply such as:
  - Operation Mode: Analysis
  - Working Distance: 12.0 mm
  - $V_{acc}$ : 2-3 times the critical ionization energy of the elements of interest (see table of critical ionization energies)
- 2) Turn off the camera.
- 3) Log onto the EDS computer as “INCA Operator”, and start the INCA software.
- 4) Chose a mode to operate in and follow the Navigation flow chart on the left side of the screen:
  - a) Analyzer: Generates spectra from the entire area of the SEM image
  - b) Point & ID: Generates spectra from user defined spots or areas
  - c) Mapping: Generates chemical maps and line scans
- 5) Site of Interest: In this window, make sure the two computers are talking (if not, restart INCA software and/or computer).
- 6) Project: Give your project a name (this will also be the file name).
- 7) Sample: Give your sample a name (see Data window for data organization)
- 8) Microscope Setup: Optimize conditions. Dead time should be as close to 30-40% as possible without going over. (Change process time, beam current ( $I_e$ ), and objective aperture on SEM.)
- 9) Quant Optimization: Acquire data from your sample and perform quant optimization for one of the available elements. (If you change  $V_{acc}$ , you will need to redo the Quant Optimization.) Although this isn't in the Mapping flow chart you still need to perform this under either Analyzer or Point & ID.

### Analyzer

- 10) Acquire Spectrum: Choose Livetime and input a time for spectrum collection. Press Start.
- 11) Compare: Compare multiple spectra if necessary.
- 12) Confirm Elements: Identify and confirm the elements present in each spectra.
- 13) Standardize: You can enter new spectra for use as standards. Do NOT perform without consulting Carrie.

- 14) Quant Setup: Adjust the quantitative analysis parameters if necessary.
- 15) Quant: Quantify the elemental analysis, and view your data in different formats (charts, tables).
- 16) Report: Prepare a report that can be exported as a Word file or as an HTML. Alternatively you can export individual spectra and images as text or image files (Right click on them and select export).

### **Point & ID**

- 10) Image Setup: Set the parameters for image acquisition (resolution, scan speed, number of frames).
- 11) Site of Interest: Check the magnification and voltage to ensure computers are talking (if not restart INCA software and/or computer). Choose an area to image with the SEM, create a new site of interest if necessary, and press Start to collect the image.
- 12) Acquisition Setup: Choose Livetime and input a time for spectrum collection. Also choose spectrum range and number of channels.
- 13) Acquire Spectrum: Choose tool and collect (multiple) spectra.
- 14) Compare: Compare spectra if necessary. Also allows you to subtract spectra.
- 15) Confirm Elements: Identify and confirm the elements present in each spectra.
- 16) Quant Setup: Adjust the quantitative analysis parameters if necessary.
- 17) Quant: Quantify the elemental analysis, and view your data in different formats (charts, tables).
- 18) Report: Prepare a report that can be exported as a Word file or as an HTML. Alternatively you can export individual spectra and images as text or image files (Right click on them and select export from most of the above windows).

### **Mapping**

- 10) Image Setup: Set the parameters for image acquisition (resolution, scan speed, number of frames).
- 11) Site of Interest: Check the magnification and voltage to ensure computers are talking (if not restart INCA software). Choose an area to image with the SEM, create a new site of interest if necessary, and press Start to collect the image.
- 12) SmartMap Setup: Choose map or linescan resolution, spectrum range, number of channels, and acquisition time.
- 13) SmartMap: Choose linescan, full map, or partial map and press Start.
- 14) Element Maps: View map data.
- 15) Element Linescans: View linescan data

- 16) Element Setup: Analyze sum spectra for elements, and add or remove elements from the map or linescan windows.
- 17) Report: Prepare a report that can be exported as a Word file or as an HTML. Alternatively you can export individual spectra and images as text or image files (Right click on them and select export from most of the above windows).

### **Shut down procedure**

- 1) Save your data file (d:/Your Advisor/Your name/)
- 2) Close the INCA software.
- 3) Return the SEM to “Normal SEM” imaging parameters:
  - a) Working mode: Normal
  - b) Working distance: 12.0 mm in Column window and on manual adjustment knob
  - c)  $V_{acc} = 2 \text{ keV}$
  - d) Condenser lens strength: 5.0
  - e) Objective Aperture: 3
- 4) Shut down the SEM following SEM checklist procedures (turn off HV, press “Go to Home”, remove your sample, turn off camera, and sign out of log book).