EDS with AZtec
Operating Procedure

NUANCE
Atomic and Nanoscale Characterization Experimental Center
I. Start Up
   1. Set up for EDS analysis following the EDS with AZtec procedure in the microscope user manual
   2. Open the AZtec software on the Oxford PC
   3. Save the project locally on the computer in the AZtec Projects Folder or D: Drive
      a. NOTE: Remember to migrate data saved locally onto the computer to the EPIC_SEM (S:) drive at the end of your session
   4. For Quantitative analysis, see section VIII and IX for Calibrations and Standardizing before section II
   5. For Qualitative analysis, continue to section II

II. Describe Specimen and Scan Image
   1. Select the type of analysis to perform from the drop-down menu
      a. Point and ID (Section V) – analyze points or areas
      b. Mapping (Section VI) – spatial distribution of elements in field of view or selected area
      c. Linescan (Section VII) – composition along a transect
      d. Layer Probe (Section VIII) – calculate thickness and composition of multilayered structures or substrates
2. **Summary** Tab
   a. Make notes about the **Project** and the **Specimen**. This is also where you can add new specimens
   b. Select the **Specimen** in the **Data Tree**
   c. Indicate sample coating material and thickness
      i. The software will automatically indicate the density
      ii. **NOTE**: This is important for calculation of quantitative results

3. **Pre-Defined Elements** Tab
   a. Double click the elements in the periodic table until they turn green (optional)
   b. Select the check box next to **Auto ID During Acquisition** to have software identify the elements in your sample
   c. Select **Save to Profile**
   d. **NOTE**: The predefined elements will only be applied to the selected specimen in the data tree. New Specimens will need to be redefined.
   e. **Fixed List of Elements** for comparative analysis
      i. Select the **Quant Element List Details** tab
      ii. Select **Fixed List** and identify the elements you wish to include in your quant
4. Data Tree  
   a. The data collected will appear as icons in the list on the right hand corner of the display window.  
   b. Each sample is labeled “Specimen” and each area of interest in the sample will be a “Site”  
   c. NOTE: Right click and select Rename to name each Specimen and Site  
5. Select the Scan Image tab  
6. Determine signal strength using the Ratemeter  
   a. The ratemeter should appear in the bottom right hand corner of the screen. If the ratemeter is not present, select it from the drop-down menu in the Mini View screen  
   b. Ratemeter display  
      i. Input Count Rate (cps) – total number of x-ray counts analyzed by detector  
      ii. Output Count Rate (cps) – number of x-ray counts being processed and incorporated in spectrum (Ideal is at least 5000 cps)  
      iii. Deadtime (%) – the ratio between input and output; the percentage of cps not being processed (Ideal is between ~30-50%)  
7. Select the Ratemeter Setting cog and make adjustments  
   a. Select Energy Range  
      i. This is based on your Accelerating Voltage and will determine the highest energy shown on the x-axis of the spectrum  
   b. Select Number of Channels (divisions of the X-axis in eV/channel)  
      i. More channels = higher spectral resolution  
      ii. Use high number of channels for larger energy range (see table)
c. Adjust **Process Time** - the length of time spent reducing noise from the X-ray signal coming from the ED detector during processing
   
   i. Select Process time between 1-6
      
      1. Longer process time = better spectral resolution, lower noise, longer acquisition
      2. Shorter process time = lower spectral resolution, high noise, quicker acquisition

### III. **Point and ID**

1. Select the **Point and ID** analysis from the drop-down menu
2. Go through **Describe Specimen** and **Scan Image**
3. Select the **Acquire Spectra** tab
   
   a. Select the **Setting** cog in the **Acquire Spectra** tab
      
      i. Select the energy range, number of channels, and process time (if not already set)
      
   ii. Select the **Acquisition Mode**
      
      1. Auto – acquisition will continue until enough counts are collected in the spectrum for quantification
      2. Live time – a set time. The actual live time will vary depending on the process time
      3. Counts – Set number of counts to collect. Use at least 300000 counts in the count limit
   
   iii. Select **Pulse Pile Up Correction**
      
      1. Corrects for pulses arriving at the same time resulting in sum peaks (e.g. Fe kα at 6.4 sum peak will show up at 12.8 kV)
      2. This works best with point analysis and has a harder time with any beam rastering analyses (e.g. Mapping and Linescans)
   
   b. Select a point ✗, rectangular area ☐, circular area ☁, or free-hand drawn area ☖ from the menu on the left side of the screen
   
   c. Click the area of interest on the image or click and drag to select an area
d. A spectrum should appear at the bottom of the screen. If it does not, select the split screen option on the top right of the user interface.

e. **MiniQuant and Compare Spectra**

   i. The MiniQuant will show composition as a bar graph or in wt.%

   ii. Change to At% by selecting the setting cog

   iii. **NOTE**: Normalized quant should be used if energy calibration not performed

   iv. Select the compare drop down list to select a spectrum to compare to the currently displayed spectrum

    1. Both spectra quants and lines will be shown in the MiniQuant

4. **Confirm Elements** – confirm elements identified in spectrum

   a. **Candidate elements**

      i. Double click on element peaks until they are highlighted in brown

      ii. A list of candidate elements will appear to help identify or confirm that peak
IV. Mapping

1. Select Map from the analysis drop-down menu

2. Select the Acquire Map Data tab
   a. Select the Setting cog on the top of the Aquire Map Data screen
      i. Select the image resolution (pixels in the x direction)
      ii. Select an acquisition time
         1. Until Stopped – Scanning will run until STOP is manually selected
         2. Fix number of frames to scan
      iii. Select the energy range, number of channels, and process time (see Section II)
      iv. Select the pixel dwell time (default is 100 µs)
      v. Frame Live Time (s) depends on the map resolution and the pixel dwell time

3. Select the Construct Maps tab
   a. Open the periodic table by expanding the Map Details tab
      i. Select the element maps you would like to display if not displayed already
   b. OPTIONAL: in Map Details, select the Selected Elements Details option
      i. Here you can manually define the width of the energy window (Specify Energy Window) or energy line being analyzed (Specify Line Series)
         1. Example: when looking at lower energy peaks while doing low kV EDS
   c. Select the Reconstruction tools on the left side of the screen to reconstruct a spectrum from a selected area on the map
4. Open the **Analyze Phases** tabs (If available)
   a. Allows the software to automatically converts X-ray maps into phase maps to I.D. constituent elements of the phase, and phase distribution in sample
   b. **Find Phases**
      i. In the acquisition toolbar, click **Find Phases** button to start processing
      ii. Open the Setting cog icon to change processing setting and reprocess if necessary
         1. **Boundary Tolerance** – low tolerance removes pixels that has influence of many phases. High assigns closest possible phase
         2. **Grouping Level** – Low grouping level = larger number of phases. High = smaller number of phases displaced (combines phases)

V. **LineScans**

1. Select LineScan from the analysis drop-down menu
2. Describe specimen and Scan Image
3. Open the **Acquire Line Data** tab

   a. Select the **Acquire Line** tool from the toolbar on the left
   b. Click the image on the line starting point then drag the mouse to the end of the line and unclick
   c. Open the line data **Settings** cog
      i. See Section II for Energy Range, Number of Channels, and Process time information
      ii. Select the Pixel Dwell Time in ms
      iii. Indicate the Line Definition (Points or Segments)
         1. Identify the number of points along the line or segments
   d. Press the Start button to start the acquisition
4. Construct Linescan
   a. Define energy windows and configure x-ray line series using
      Selected Element Details
   b. AutoID elements in the spectrum and add or remove any
      you wish to see

VI. Reporting Data

1. Open the data tree menu and minimize each site

2. Select the data you wish to report (e.g. a spectrum, map data, linescans)

3. Select the Report Results dropdown menu

4. Select Report Templates from the menu
   a. Select the report template
      i. Check Favorites to display a list of the most commonly used reports
   b. Suggested reports
      i. Spectrum: Electron Image/Spectrum/Quant
      ii. Map Data: Electron or Layered Image and Maps
      iii. LineScan:

5. Select Save As and save to the Reports folder of your saved project

6. A Word or Excel document will open. Leave this document open

7. Select another spectrum/map/linescan from the data tree and follow steps 3 and 4

8. Select Append instead of save as
   a. This will add your data as another page or sheet to the existing Word/Excel Document

9. Continue this for remaining data to report

10. Saving Spectral data in spread sheet format
    a. Select the spectrum of interest
    b. Open the confirm elements tab
    c. Right click on the spectrum and select Export > EMSA

11. Save all project data locally on the computer (in Aztec Projects or D: drive)

12. Open the local folder, copy your data and paste it to your folder (Named with NetID) on the
    EPIC_SEM (S:) drive
VII. Calibrations

1. Energy Calibration
   a. Energy Calibration measures the shift in the position of the spectral peaks and resolution of the system
      i. Select Optimize from the analysis dropdown menu
      ii. Select the Calibrate tab
      iii. Select Energy Calibration from the Calibration Routine drop-down list
      iv. Select element (copper tape on a stub) from drop down list
      v. Adjust working distance to optimal (See instrument manual)
      vi. Press Start to start acquisition of the calibration spectrum
      vii. A window will appear asking if you wish to perform Energy Calibration.
         1. Press Yes
   b. Energy Calibration should be recalculated if you change the process time, number of channels, or energy range of spectrum
   c. NOTE: Energy Calibration can be found in Spectrum Details dialog in Calculate Compositions when spectrum has been quantified

2. Beam Measurement
   a. Quantitative analysis requires a Beam Measurement for accurate, un-normalized quant
      i. Beam Measurement must be performed when any beam conditions changed (e.g. probe current, accelerating voltage, I.e., etc.)
   b. Perform Energy Calibration
   c. Select Beam Measurement from Routines dropdown menu
      i. Use pure element standard
         1. Use Co for 15 kV and above
      ii. Change acquisition settings in Setting menu
         1. Energy range should be selected in conjunction with microscope accelerating...
2. Number of channels depends on energy range. Set to auto for system to check energy range and select appropriate number of channels

3. Process time – length of time spent reducing noise from X-ray signal
   a. Longer PT – lower noise, better spectral resolution (narrow peaks)

4. Total Counts in Spectrum – set to 600000 counts
   iii. Select Calibration routine from drop-down list of Energy Calibration and Beam Measurement
   iv. Select element for calibration
   v. Put element in field of view on microscope
   vi. Press Start
   vii. Save beam measurement

VIII. Standardization

Standards are materials which are used to relate the intensity of a peak in a spectrum to the concentration of that element in the specimen. They are materials in which the concentrations of all the elements are accurately known. Standards may be pure elements or compounds. High quality reference standards are essential to perform accurate quantitative microanalysis in the SEM. Not only must their chemical composition be well characterized, but they must also be microscopically homogeneous, stable under the electron beam and prepared with a flat, polished surface.

1. Open the Optimize from the analysis drop-down menu
2. Select the Standardize Tab
3. Click Add to add a block in Standards Compositions area
   a. Enter the name of the block (e.g. Metals standards block/Mineral Standard block)
4. Click Add to add a standard
   a. Enter name and composition of standard
5. Enter the composition
   a. Number of atoms
      i. If you enter it in a wrong format for example feS2 or FES2, it will not be accepted.
   b. Weight %
      i. Enter element symbol followed by weight percent
1. Fe 46.55 S 53.45
   ii. Wt. % must be between 95% and 105%

6. Open the Analyzer or Point and ID analysis window and collect a spectrum on the standard
7. Open the Optimize option from the drop-down menu and select standardize
8. Ensure that the spectrum measured on the standard is in the spectrum viewer
9. Select the standard block from the standard block drop-down list
10. Select the standard from the drop-down list
11. Select Pure Element or Element from Standard (compound/mineral standard)
12. Select the line you wish to standardize from drop-down
13. Press Standardize to perform standardization.
   a. The current and new values of standardization will be displayed
14. Press Accept to update the standards.
   a. Old standard values will be overwritten!
   b. You will be asked if you wish to use the new standardization for Quant analysis
15. Standard blocks can be selected in the Calculate Composition tab of Point and ID analysis
   a. Select the settings cog and indicate the standard block from the drop-down menu