EDS Checklist

These are not detailed instructions and assume some familiarity with the system. For more detail, please see Chuck or Roberto.

General: X-rays with less than 1keV of energy are almost always underestimated in quant results! Li X-rays require a UHV SEM and a windowless detector that is optimized for low energy! Please do not bring any elements higher than atomic number 92 (non-enriched) to the lab!

1. Insert a sample into the chamber. Take care that the sample is not too tall by using the height gauge as described above.
2. Choose an accelerating voltage that is >2X the X-ray energy that one is looking to excite
    a. For example, if one is looking to observe Cl and Na in a sample, the accelerating voltage should be ~10kV. The Cl Ka X-ray has an energy of 2.62 keV. A 10 kV beam has ~4X the energy of the X-ray to be excited.
    b. The X-ray excitation has a maxima between 2-3X the energy of the beam and starts to fall off as the beam energy exceeds 5X of the X-ray line that is to be excited.
    c. The energy should be chosen based on the highest energy X-ray that is to be excited (30keV max).
    d. If you don't know what is in your sample, choose 20keV
3. Choose a beam current that will allow for reasonable data collection rates
    a. The X-max detector is very fast with up to 40% deadtime allowed (20% or less is recommended)
    b. Beam currents >1.6 nA will produce good results quickly, especially for mapping
4. The optimum working distance range for EDS is 6.0 – 6.5 mm
    a. This aims the X-ray detector at the beam impingement point and makes for the most efficient X-ray collection
    b. The collimator has a large opening, so X-rays can be collected over a large range of working distances, but the efficiency is best between 6.0 and 6.5 mm
5. Run Tidy Up
    a. Tidy up reboots the hardware and restarts the firmware
    b. Run Tidy up if there is any question about how the system is operating. Three hardware items should be observed: Mics, XStream2, XMax. Mics is used for imaging, XSteam2 and XMax for X-ray analysis.
6. Open AZtec software

7. Create a new project. It is recommended that sample names are placed on all the data as it is collected. It is recommended that a new site is created for different data collection modes.
8. Confirm that Aztec is set for EDS data collection instead of EBSD
9. Insert the EDS detector. (Be sure you are not inserting the EBSD detector as this will break the EBSD detector! $125k to replace!)
10. Align the system and obtain a reasonable image of the region of interest.
11. Choose the mode of X-ray data collection one desires (Analyzer, Point&ID, Linescan, Mapping) and follow the flow chart. For all:
    a. Describe the specimen: This can be as simple as a sample name or more detailed notes can be added as desired.
        i. Summary allows one to enter detailed notes about the sample, if desired.
        ii. Pre-defined elements allows one to pre-define what is to be observed. In general, it is best to leave the periodic table blank, that is, find what is there from the collected spectrum instead of telling the system what you think/want to be there. Perform AutoID is generally recommended.
12. Analyzer mode: Collects X-rays from what is being imaged on the SEM imaging monitor.
    a. Analyzer mode is useful when the sample is charging and the image is drifting during acquisition. In this case, the operator should adjust the stage or image shift to keep the region of interest in the field of view while X-rays are being collected.
    b. Check the settings to be sure that they are what is desired. Recommendations: Energy range = beam energy, Number of channels = Auto, Process time = 4, Acquisition mode = Livetime, Acquisition time = 60s, Pulse pile up correction on.
    c. The acquisition time can be adjusted as needed. Short acquisition times are often sufficient for qualitative ID and may be necessary if the sample is drifting very quickly. Longer times will give higher confidence in quantitative results.
    d. Go to Confirm Elements to confirm peak shapes and eliminate overlaps. Use your brain!
    e. Calculate composition will allow a table of quant results to be observed and exported.
13. Point&ID mode: Collects X-rays from a region of a user acquired image.
    a. Collect an image. See imaging notes above. Be sure that there is no apparent image drift!
    b. Check the settings to be sure that they are what is desired. Recommendations: Energy range = beam energy, Number of channels = Auto, Process time = 4, Acquisition mode = Livetime, Acquisition time = 60s, Pulse pile up correction on.

c. Choose a point, box, circle, or free-hand area on the collected image. Once the first point or area is chosen the first acquisition will begin. Multiple points/areas can be chosen and will queue in the order they are drawn. The active acquisition area is yellow.
d. The acquisition time can be adjusted as needed. Short acquisition times are often sufficient for qualitative ID. Longer times will give higher confidence in quantitative results.
e. Go to Confirm Elements to confirm peak shapes and eliminate overlaps. Use your brain!
f. Calculate composition will allow a table of quant results to be observed and exported.

14. Linescan: Collects X-rays along a user defined line within an acquired image.
    a. Why not a complete map? See Chuck for linescan recommendations.
15. Mapping: Collects X-ray maps of the elements in the sample.
    a. Collect an image. See imaging notes above. Be sure that there is no apparent image drift!
    b. Check the settings to be sure that they are what is desired. Recommendations: Resolution = 1024, Acquisition time = until stopped, Energy range = beam energy, Number of channels = Auto, Process time = 4, Pixel dwell time = 10us → Frame time = 8s.
    c. Collect the map until it is sufficiently detailed. The map will continue to collect until the user presses stop. After stop is pressed the system will finish the current frame (hence the recommended short frame time). The quality of the map will increase with increasing number of frames, assuming that there is no apparent drift in the maps. Drift in the image will degrade the map! The image is pre-saved and not updated, so it is smart to collect a before and after image if drift is suspected. High drift = bad maps!
    d. Go to Construct Maps to confirm the elements that are being mapped. Beware of overlaps! Use your brain! The color of the elements can be changed and element maps can be added or removed. The observed maps can be resized to make them easier to observe. A sum spectrum from the entire map region can be observed here too. Construct map recommended settings: Sort order as desired, Visibility selection = auto, Smoothing = 1 no smoothing, ACB (auto contrast and brightness) while acquiring = checked.
    e. It may be desirable to do *exactly* the same acquisition for more than one map. If this is the case, count frames on the first map to determine how many frames to set and then set the Acquisition time to the number of frames. Use your brain!

16. Export your data.
    a. It is smart to export data as it is collected.
    b. A report can be generated, which will be a MS Word document showing the data from a particular site.
    c. Images can be batch exported by right clicking on the site or specimen.
    d. Reports can be batch exported right clicking on the site or specimen.