SU3900N Checklist:

1. Put the sample in the chamber
    a. Set the sub-screen display area to show the chamberscope
    b. Click on “Exchange Specimen” in the SU3900 GUI. The chamber will start to vent and a pop-up window will appear to guide the user through the sample exchange process – follow the process
        1. Select “Exchange Specimen”
        2. Choose the size of the sample stub
        3. Open the chamber door all the way and remove the sample holder, then thread the stub onto the holder. Measure the height of the highest point on the stub using the height gauge, then set the sample height in the window and click “Next.”. The height of the sample holder can be adjusted to suit.
        4. Install the sample holder (with sample) on the stage and click on “Stage Motion” while the chamber door is fully open. The stage will move to the maximum height allowed for the sample height. Keep hands away while the stage is moving.
        5. Carefully close the door (observe in chamberscope). The top of the sample should not touch as the door is closed. If the sample hits the safety check gauge, the sample is too tall and the height must be adjusted. Once the door is closed, click “Next.”
        6. Choose the vacuum mode desired and click on “EVAC”
2. Once the EVAC process starts, the system will automatically take a navigation camera image of the sample
    a. The stage will drive under the nav cam and an image will be collected. If this image is not good, “Re-capture,” if the image is good, “OK.”
    b. A closeup image of the sample holder will appear with a red circle, which can be adjusted to the actual sample size. Once any desired adjustment is complete, click on “OK”
    c. A pop up window will appear that allows the user to set the capture mode (Slow/Css recommended), pixel density, and data collection rate
    d. Within this window, one can name the (first) sample, set which folder the data will be saved in and the file format, and the starting number for the series
3. After vacuum gauge indicates < $5.0 * 10^{-4}$ (Torr), the beam can be turned on
    a. It is possible to set the starting beam conditions in the electron beam settings menu
4. Open the electron beam settings menu and choose the desired accelerating voltage (kV), spot intensity (aka beam current), and working distance
    a. Low kV will show more surface detail, high kV is typically required for EDS or variable pressure operation
    b. Low beam current will produce higher potential resolution!
    c. Short working distances are good for higher resolution, EDS working distance = 10mm
    d. Click on “Start” to turn on the beam

5. Double click on region of interest on the sample in the nav cam to set that position under the electron beam.
6. Coarse focus (don't forget astigmatism correction! Don't try to make the image perfect yet!)
7. Align the electron column
    a. The electron gun alignment should be pretty stable and not adjusted by the user
    b. There may be an aperture inserted (Aperture 2 is recommended for most use). If the aperture needs to be changed, this is the time to do so. Small apertures are good for improving depth of field and resolution, large apertures are for low magnification and EDS.
    c. Click on the mode button in the center of the knob set to first align the aperture. The aperture adjustment is a physical adjustment on the side of the instrument
    d. Click on the mode button to cycle through the electronic alignments (StigX, StigY, AFC, and Low Vacc). Each of these alignments are done using the mouse in the operation panel
8. Perform fine focus and astigmatism correction on region of interest
    a. Focus at higher magnification than desired for the image (by at least a factor of 2) – try to make the image perfect at this point.
    b. Zoom out to take image – the image is still in focus don't touch the focus knob.
9. Set the contrast (signal gain) and brightness (signal offset) so that the image is not oversaturated and not overly dark. Unfortunately, there is no signal monitor, so the image brightness and contrast are set by eye.
    a. Compose the image so it is pretty!
10. Save images by clicking on "Capture"
    a. The capture is finished when the scan rates at the top of the page become active
    b. Two channels of data can be collected simultaneously
    c. SE data will mostly show topography
    d. BSE data will mostly show compositional contrast (BSE data requires 5 kV or more and a spot of 30 or more)

Finishing your session:

1. Pause the acquisition
2. Turn off the beam by clicking on "Stop"
3. Set the sub screen display to the chamberscope
4. Click on "Exchange Specimen." The chamber should start to vent and the stage should be observed going to the bottom of the Z-range
5. Follow the flow chart for sample exchange (not removal). Choose any stub diameter and place the empty sample holder back on the stage. Set the vacuum mode to High. Please finish the process leaving a nav cam image of the empty sample holder.
6. Data can be collected from the internet access computer

For EDS: 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 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).
3. Choose a beam current that will allow for reasonable data collection rates
    a. The Ultim max detector is very fast with up to 40% deadtime allowed (20% or less is recommended)
    b. A spot intensity in the range of 50 - 70 will produce good results quickly, especially for mapping
4. Choose a working distance of 10 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 in the range from about 8 - 12 mm, but the efficiency is best at 10mm
5. Align the electron column and obtain a reasonable image of the region of interest.
6. Confirm the ChamberScope is off in the sub screen display area
    a. The flux of IR photons from the chamberscope will overwhelm the X-ray photon detector!
    b. If the chamberscope is on when X-rays are being collected, one will only see the noise peak from the IR photons striking the detector!
7. AZtec software should be open. Recommend that EDS data is collected through AZtec and not using the integrated EDS in the SEM software
8. 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.
9. 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.

10. 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.
11. 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.
12. Linescan: Collects X-rays along a user defined line within an acquired image.
    a. Why not a complete map? See Chuck for linescan recommendations.
13. 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!

14. 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.