

# INSTRUCTIONS FOR INCA EDS SYSTEM

## A. TURNING ON THE SYSTEM:

1. Make sure SEM is turned **ON** (**GREEN** button lit and EVO computer is up and running). Refer to section A (steps 1-10) of the EVO instructions if necessary.
2. Insert samples in to the chamber. Be sure to also include a EDS Copper (Cu) standard along with your unknown samples. Extra stubs with Cu tape mounted on to them are located next to the chamber in a white cardboard storage box. Refer to section **B** of the EVO instructions if necessary for assistance with specimen insertion.
3. Pump down chamber.
4. Turn **ON** the blue/grey **INCA X-Stream-2** box. This box is located on top of the **EDS computer**. Its switch is located on the backside of the box about mid-way down on the left-hand side of the box as you face it from the front. Two small **LED** lights should be lit on the front - top **LED** light will appear solid **ORANGE** and the lower **LED** light will be blinking **ORANGE**. Wait until the lower **LED** goes out before proceeding to next step.
5. Turn on the **EDS computer** which is located on top shelf of the black UPS/Battery Backup cart. You may also have to turn on the **EDS monitor** (far right-hand monitor).
6. At some point during startup, a “**RemCon break**” box may appear on the left-hand monitor. If this box appears, click on **Retry** until this warning box disappears. You may have to do this 2-3 times.
7. After a few moments, the SEM computer should recognize the EDS computer. If things load properly, the SEM mouse will now move across all three monitors. If it does not, wait another minute or two. It sometimes takes a few moments for the computers to recognized each other over the internet. If the SEM does not see the EDS computer, go ahead and use the extra EDS mouse and keyboard which is also located on the top shelf of the Battery Backup cart.
8. With the SEM mouse, left click on the **INCA** icon located on the desktop of the right-hand most monitor (EDS computer).
9. When prompted for the password, type in **INCA** (all caps, not bold) using the SEM computer keyboard. Allow Windows XP to load completely on the EDS computer.
10. Double click on the **INCA** icon (located on the EDS monitor desktop) and allow the INCA software to load. Note: You may receive a “**Detector Control Unit**” warning box or a “**Detector Coolant Low**” warning box at this point. Just select “**OK**” to bypass this box. During this time you may also hear an audible series of warning beeps which indicate low coolant levels. This should stop shortly after loading INCA.
11. Make sure the **LED** lights on both the **INCA mics-2** box and the **INCA x-stream-2** box are lit **GREEN**. These are both located on top of the EDS computer.
12. Turn on **SEM beam and saturate the beam** as you would do normally. See sections **C** and **D** of the SEM instructions if necessary. For most EDS work, the beam should be set on **20kv**.
13. Adjust **working distance to 8.5 or 9mm**. See section **E** of the SEM instructions if you need help with this.

## **B. ANALYZING SAMPLES by EDS**

1. Make sure INCA is set to **Point & ID** mode as indicated at the bottom of the flow chart when viewing the **Navigator** tab.
- 2 Move through the flow chart with the following boxes:

**Project** box - use this box to assign a project name and other information specific to the overall project. You may click on the 'bubble' icon at any time for help in knowing what to do next or in what order to do them.

**Sample** box - use this box to assign sample names and other information to the sample.

**Microscope Setup** box - used to set up percent dead time on your sample as shown with the meter at the bottom of this window.

- a. Locate and focus on your sample. Be sure to set the **working distance (WD)** to between **8.5 and 9 mm**.
- b. Click on the **green** dot (●) at the top of the window to start scan. Percent dead time should be between **20-30%**.
- c. Adjust % dead time by increasing or decreasing **iprobe current** found under the **Gun** tab.
- d. Once % dead time is set, click on the **red** square (■) to stop scan.

**Quant Optimization** box - do this at the beginning of your session to calibrate the system for accurate quantification of your sample.

- a. Move stage to the stub containing the copper tape standard. Focus on the tape but do not adjust the working distance or the probe current.
- b. Make sure **Copper** appears as the element in the **Optimization Element** box.
- c. Start the scan by clicking on the **green** dot (●).
- d. Allow scan to proceed as indicated by the **Acquisition Time** meter.
- e. Once the scan is complete, click on the now active **Measure** button.

**Site of Interest** box - use this box to transfer a SEM image to the INCA software.

- a. Click on the **green** dot (●). An image of your sample should now appear in the INCA viewing window. If the image is not suitable, make the necessary adjustments to the image in SmartSEM (SEM computer) and then recopy the adjusted image to INCA by clicking on the **green** dot (●) again. Another copy of the SEM image should now appear in the INCA viewing window.

**Acquire Spectra** box - use this box to define an area of interest and begin analysis of that area.

- a. Select either the **plus** + icon to analyze a single point on your sample OR select the **box** □ icon to analyze a user-defined area on your sample.
- b. If the single point **plus** + icon was selected, click on a point of interest on your sample image. Analysis and spectral collection will begin immediately. If you wish to analyze additional points of interest, click on another point during this

time to place those in the analysis queue. Once collection of the first spectrum is complete, analysis of the next item in the queue will begin.

c. If the **box**  icon was selected, place your cursor on the upper left-hand most corner of the area of interest, hold down on the left mouse button and drag the cursor to create a boxed area of interest. Once you release the left mouse button, analysis will begin and a spectrum will be collected of that area. Additional areas can be selected during this time with each being placed in a queue.

d. In either case, wait until analysis and spectrum collection is complete as indicated by the **Acquisition Time** meter. Progress bars will be greyed out when scan is complete.

**Confirm Elements** box - use this box to confirm the list of elements detected. Elements showing up in the **Confirmed** list will be included in the quant calculation. If you prefer not to include a listed element, either click on the element within the list and click the **Remove** button OR you can left button double click on the highlighted element in the periodic table to remove it from the list.

**Quant** box - use to display the quant summary results (weight % and atomic %)

**Report** box - use to display results in a variety of user-selected styles.

a. From the **Template** drop-down menu, select the appropriate results display. The most popular display type is “**Image/Spectrum/Summary of Results**”.

b. Now save your raw data. Click on **File** in the upper left-hand corner of INCA and then select “**Save Project As...**”. All data is being saved on the **D drive** in the **INCA Data** folder. Before saving, be sure to give your project a name. Notice that the file will be saved as a **.ipj** file which is specific to INCA.

c. Once you have saved your raw **.ipj** data file, you will be returned to the main **Report** page.

d. Click on the **W** icon to create a Microsoft Word document.

e. Click on the ‘**Office**’ button in the upper left-hand corner and select **Save As**. Open your INCA Data folder and assign your Word doc a name before clicking on the **Save** button. Once saved, click on the X in the upper right-hand corner to exit Microsoft Word.

To analyze another area on the same site of interest then go back to the **Acquire Spectra** box.

To go to another site of interest on the same sample, move stage slightly and go back to **Site of Interest** box.

To analyze another sample, move stage to a new sample, check working distance on sample, then go to **Microscope Setup** box to check % dead time. Once that is set, go to **Site of Interest** box.

### C. SHUTTING DOWN INCA and EDS SYSTEM:

1. After saving all data, click on **red X** in the upper right-hand corner to close INCA window. INCA will close and you will be returned to the main Oxford Instruments desk top.
2. Before shutting down the EDS computer, transfer your data to a disk or other portable storage device.
3. Shut down computer by clicking on the **Start** button and then select **Turn Off Computer**. Confirm by clicking on **Turn Off** button when prompted.
4. Allow computer to shut down. In the process, you will lose connection with the SEM computer. A error message will appear on the EDS monitor but will disappear shortly. You may also close this error message box using the EDS computer mouse.
5. Turn off EDS monitor.
6. **Turn off the blue X-Stream analyzer box with the toggle switch located on the rear of the box.**