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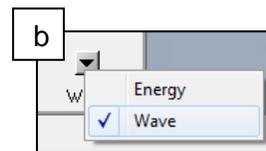
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I. Start Up

1. Turn on P10 gas
 - a. The pressure should be at 10 psi
 - b. P10 gas should be flowing for 30-60 minutes before doing any quant
2. Load samples on regular sample holder. Include the minerals or metals standard plate
 - a. Make sure the height different between samples and standard plate is no more than 5 mm
3. Follow the Sample Loading steps in the Hitachi S-3400 manual to assemble, measure, and insert the sample into the chamber.

4. WHILE THE CHAMBER IS PUMPING!

- a. Open INCA on the Oxford computer and select **Full Acquisition Mode**
- b. At the bottom of the analysis column, click the drop down arrow and select **Wave** from the list

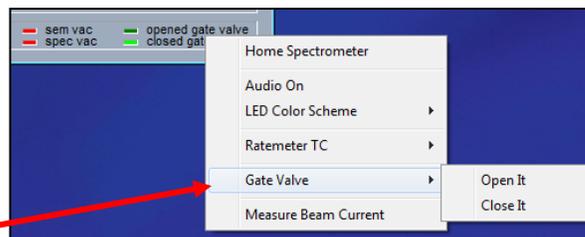


- c. Go to **Option > Wave Options > Spectrometer Direct Control**

- d. Select **Motor**



- e. Right click on the bottom right hand corner > **Gate Valve > Open it**



5. Back out EDS detector by rotating knob at the end counterclockwise until it stops moving
6. Change the BNC cable attached to the right side of the chamber door to the cabled labeled WDS.
7. Set the following parameters:
 - a. Accelerating Voltage – Usually between 15 to 30 kV
 - b. Probe Current – Set to 70 – 100 (depending on count rate)
 - c. No aperture (pull the aperture knob while turning it counter clockwise to 0)

8. Turn on Beam
9. Focus on a particle on the faraday cup
10. Press Analysis under the Stage tab to bring the sample to 10 mm working distance
 - a. When the stage stops moving, ensure the sample is as close to 10 mm as possible
11. Go through alignments (See 3400 Manual)
 - a. Be sure to manually adjust the **Gun Tilt** and **Shift** to the brightest image



12. Turn on **Focus Monitor**

13. Adjust the **Filament** slider until the waveform stops going up

- a. **NOTE:** if the waveform goes so high that it leaves the frame, adjust the brightness knob on the knob panel.



- b. Once the slider stops going up, turn the slider down 1 notch

14. Set the preset magnifications to x50 and x10k



15. Take an image of the **Faraday Cup** and **all standards** at 10 mm working distance

- a. Take images at 50x magnification.
 - b. **NOTE:** double check the number, location, and shape of the standard you're looking for in the standard block booklet.

16. Take image of Fe metal standard for calibration and Co standard if doing combined EDS and WDS

17. Open INCA Energy and do an EDS analysis if there are unknown major or minor elements in your sample

- a. Take pictures of additional standards if necessary
 - b. **NOTE: Make sure chamber camera is off before doing any EDS analysis**

II. Beam Stabilization

1. Open INCA (if it's not already open)
2. Move to the Faraday Cup and zoom in on the center
3. In the Wave analysis flow chart on INCA, select **Microscope Setup**
4. Start the Beam Current Monitor
 - a. Set the difference to 1% variation to start. This will change as you adjust the beam current

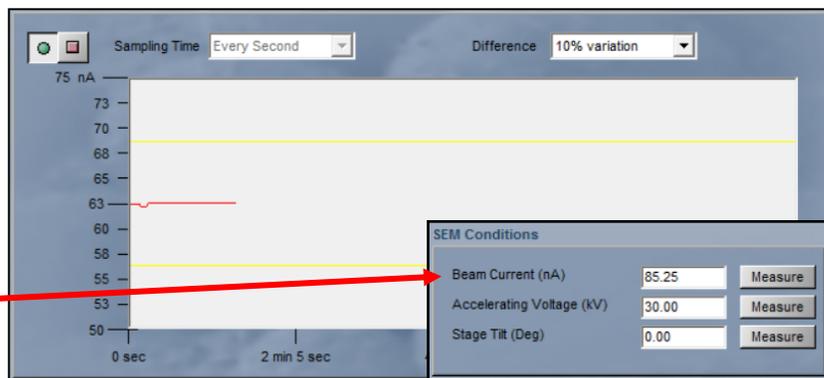


5. Watching the beam current monitor, adjust the gun bias up or down to get the maximum current on the SEM computer



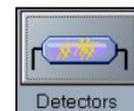
6. Once you achieve the highest current possible, let beam stabilize for 20-40 minutes

7. Measure the beam current



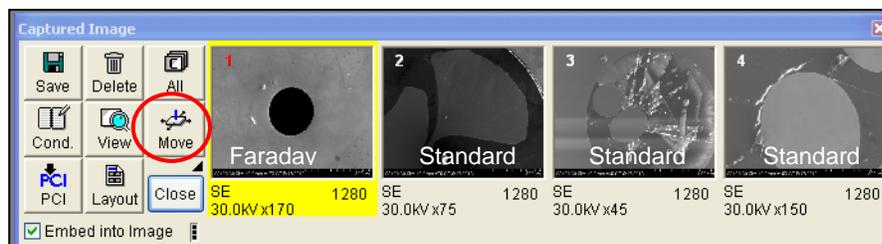
III. Detector Calibration

1. Move to the iron standard, focus at 10 mm at a magnification of x10k
 - a. Use the metal standards block guide book to ensure it's the right standard
2. Go to the **Wave > Options > Wave Options > Spectrometer Direct Control** window
3. Select the **Detectors** button and hit **Calibrate**.
 - a. Make sure the "Calibration Element" is set to **IRON**



IV. Element Peak Search

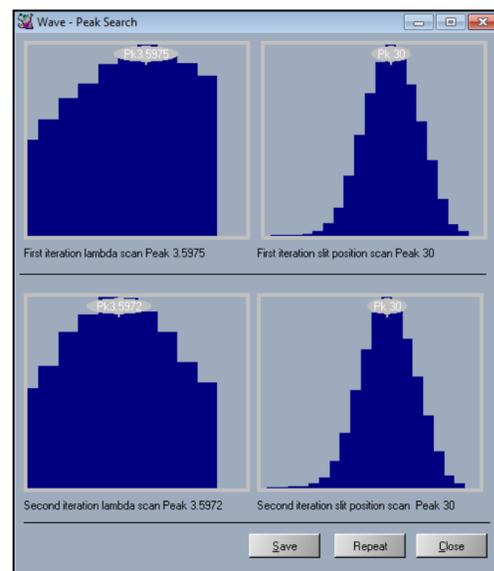
1. Center the first standard at 10mm under the beam at x10k magnification
 - a. **NOTE:** Use the images collected on the SEM screen to navigate to the standards that were previously imaged by clicking on the image and selection the “Move” button



2. Go to **Wave > Options > Wave Options > Spectrometer Direct Control > Motor**
3. Perform a **Peak Search** on the standard under the beam



- a. Select the element from the list
 - b. Select **Peak search on element**
 - c. Click the **Setup** button
 - d. Click the **Peak search** button
4. The energy peak will be identified during the scan and can be recorded for the analysis by pressing the **Save** button after the peak search has completed
 5. If the difference between the first and second lambda peak scans are more than 0.0005, hit **Repeat** until they are closer



- a. Lighter elements tend to be further apart but should be as close to 0.0005 difference as possible
 - b. The Lambda scan moves the crystal and detectors along Rowland circle in the diffraction range of the selected element.
 - c. The slit position moves the detector slit to the optimum detection point.
6. Repeat these steps for all of the standards required for the analysis
 7. **NOTE:** if you can't find the peak, repeat the search once

- a. If still no peak is found, go to **Wave > Spectral Scan > Find Element Peak** (slit size = 2.5, slip pos. = 0, scan speed ~6)
- b. Choose element, add to scan, start
- c. Write down peak energy
- d. Edit peak energy with **Wave > Options > Wave Options > Line Table Editor**
- e. Back under Motor, type peak in A, do **Peak Search** in **Current Location**

V. Standard Measurements

1. Go back to the **INCA** interface and select Standardize at the bottom of the **Navigation** panel



2. Select Choose Elements from the flow chart



3. Select the elements to be measured on each standard by double clicking the element on the periodic table. The elements will be added to the list in **Standardization details**

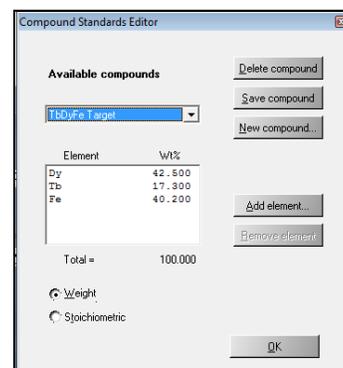
4. In **Standards Setup**, go through each standard in the list and make sure the **Standardization name** corresponds to the correct standard plate. Mineral = composition or Metal = pure standard



5. For mineral standards, select “The standard composition is specified by the (Standard name) compound”
6. For all metal standards, select “Pure Standard”

7. Adding a standard

- a. Select Edit > New Compound
- b. Enter Standard Name and composition one element at a time by weight (if measured using ICP-MS or similar) or by Stoichiometry



8. Adjust scan time for each element
 - a. Make sure ‘Peak search before acquisition’ is unchecked

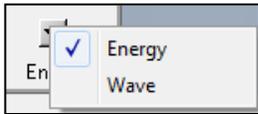
Elements of interest	Peak (sec)	Background (sec)
Major	10	5
Minor	20	10
Trace	40	20

9. Move back to faraday cup and measure beam current under **Microscope Setup**
10. Select **Measure Standards**

11. Make sure all standard elements are checked in the list
12. Center the beam on the standard used for the element you have selected (The software should tell you which one to start on)
13. Select the start button and allow the detector to measure on the standard 
14. Once one standard is measured, you will be prompted to move to another
15. Accept the measured standard value and repeat steps 8 and 9 for every element standard measurement

VI. WD Energy Plus

Energy Plus allows you to search for element peaks in selected energy ranges in an EDS spectrum. This allows you to determine if an element that EDS is unable to detect is present in the sample. This is not a quantitative technique.

1. Ensure chamber camera is turned off
2. In INCA, open the Energy window 
3. Go to Microscope Setup
 - a. Hit the green start acquisition button and check count rates
 - b. Lower the process time, if necessary, to achieve a dead time at or below 50%
4. Acquire an image under **Site of Interest**
5. In **Acquire Spectrum**, select a **Point** on the image to collect a spectrum
 - a. **NOTE:** Energy Plus only works on **Point** or **Analyzer** acquisitions

6. Select **Confirm Elements**

- a. Locate the energy range of the trace elements or elements of interest
7. Click the Wave button in the top right hand corner of the window



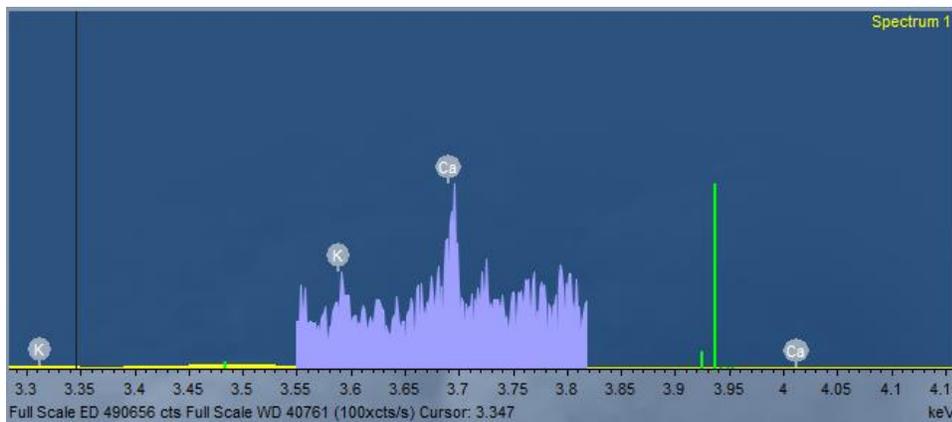
8. Adjust the following settings:
 - a. Slit size



- i. A smaller slit will produce higher resolution but take longer to scan
- b. Slit Position – Leave at 0
- c. Scan Speed
 - i. A higher scan speed will make the acquisition go faster, but will sacrifice resolution

9. While holding down on the Shift key, highlight the energy range you want to scan

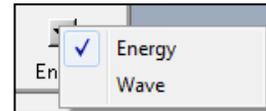
10. Once scan is complete, open confirm elements and select elements from periodic table if they are not already auto identified



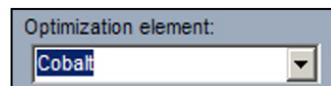
VII. Combined WD and EDS

Wave data can be acquired in conjunction with EDS data for trace elements in the sample. This allows you to do EDS on major elements samples of unknown composition while assigning trace element compositions to WDS.

1. Open the INCA **Energy** window
2. Select **Analyzer** or **Point and ID** analysis
3. Move to the Cu or Co standard on the metals standard plate
4. Select **Microscope Setup**
 - a. Ensure dead time is ~45% by adjusting the process time
5. Select **Quant Optimization**
 - a. This step is important for doing unnormalized EDS Quant. It normalizes any beam current difference between the standard and sample, resulting in 100 wt%.
 - b. QO is normalizing factor for EDS, Measured beam current for WDS. These two must be associated



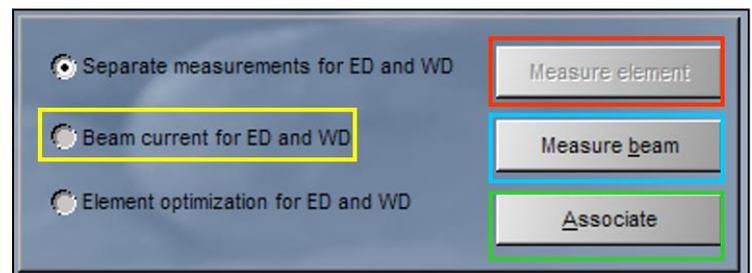
6. Select **Acquisition Setup**
 - a. Set live time to at least 1 minute



7. Go back to **Quant Optimization**
8. Select **Copper** or **Cobalt** from the dropdown menu
9. Once scan complete, select "Separate measurements for ED and WD" and click **Measure**

Element

10. Move stage to faraday cup and hit **Measure Beam** button
11. Click "**Associate**" button
12. Select the second option "Beam current for ED and WD" only when done associating



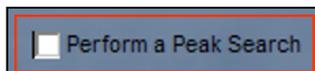
- a. From now on, only the beam current needs to be measured

13. Select **Point and ID** or **Analyzer**
14. Create a new site of interest at 2000x magnification or higher

15. Open **Acquisition Setup**

16. Select elements to be analyzed by WDS and define appropriate **peak and background times** using the table

17. Uncheck "Perform Peak Search" for each element



18. Define live time for EDS analysis

- a. Use a time roughly that of WDS analysis so they finish around the same time

19. Make sure magnification at least 2000x or higher

20. Select point or start Analyzer

21. Results should range from 98%-102%

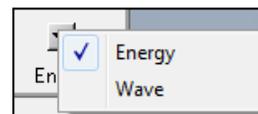
- a. Low totals (<80%) indicate presence of unanalyzed element

Element Estimated Wt %	Peak (sec)	Background (sec)
> 50	20	10
10 to 50	40	20
< 10	60	30
< 1	100	50

VIII. **WD Mapping**

1. Select **Energy** from the drop down list in the bottom right hand

corner of the acquisition flow chart



2. Select **Mapping** at the bottom of the screen

3. Find an area of interest on the sample, magnify to x10k and focus

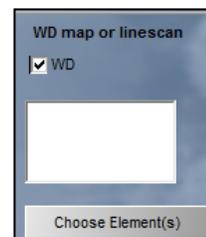
4. In **Site of Interest**, capture and image for the site

5. Select **Smart Map Setup**



6. Check the WD box in the WD map or linescan box at the bottom of the screen

7. Click Choose Element(s) and select the first element to analyze using WDS



8. Select Element Maps again and start the acquisition

- a. WD Mapping with collect data for one element at a time using the WD detector while simultaneously collected EDS data
- b. You can collect 1, 5, or 10 frames at a time

IX. WD Quant Analysis

Wave Quant analysis provides high resolution qualitative results of whole sample composition. This analysis requires some previous knowledge of the sample. Running EDS analysis prior to WDS quant is recommended.

1. On the SEM computer, move so that the sample is under the beam
2. Focus at 10 mm and at x5k to x10k magnification

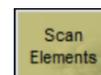
3. Select **Analyze** at the bottom of the INCA window



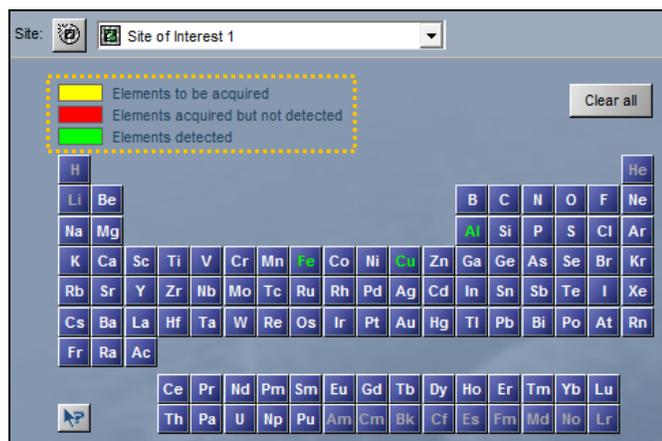
4. Make notes in the **Projects** tab about the project (not necessary)

- a. You can also create a new Site (under **Sample**) and name and make notes on each specific sample

5. **OPTIONAL:** Do an Element scan on unknown sample to determine if trace elements are detectable



- a. Select **Scan Elements**
- b. Select the desired elements from the periodic table and start the element scan
- c. The elements on the period table will change color to indicate whether or not the select elements are present in that area of the sample



6. Open **Measurement Setup**

7. Select appropriate peak and background times for each element

- a. Click on each element individually and adjust these times (Use times listed in table of Combined WDS and EDS section)
- b. **NOTE:** This depends on various factors (e.g. beam current, sample matrix, etc.). Shoot for 10,000 cps on each element

Element Estimated Wt %	Peak (sec)	Background (sec)
> 50	20	10
10 to 50	40	20
< 10	60	30
< 1	100	50

8. Move to faraday cup and measure beam current

9. Select **Measure Elements**

10. Confirm that all elements of interest are in the list to be analyzed

11. Move to the unknown sample and click the green button to start WDS acquisition



12. Once all of the elements have been measured, select the **Quant**

- a. This shows the quant in Wt. % and Atomic %
- b. Under **Quant Setup**, uncheck the box to normalize results
 - i. Quant results should be close to 100.00 ± 2.0
 - ii. Quant results <80% might indicate the presence of un-analyzed elements (carbon, lithium, etc.)

1. If elements in sample in the form of oxides (e.g. minerals in geological samples) go to Quant Setup > Elements by Stoichiometry > Oxygen

13. Select the **Reports** tab and export the quant data as a Microsoft Word document

- a. Leave the document open so subsequent reports are added directly to the same document.

14. Go to **Scan Elements** and select **New Site** to analyze a different location on the sample



X. Shut Down Procedures

1. Turn off the Beam
2. Close the WDS gate while the SEM is still under vacuum
 - a. **Go to Option > Wave Options > Spectrometer Direct Control**
 - b. Select **Motor**



- c. Right click on the bottom right hand corner >
Gate Valve > Close it

3. Reinsert the EDS detector
4. Change the BNC cable on the chamber door
5. Close P10 gas tank
6. Vent the chamber and remove the sample

