# STANDARD OPERATING PROCEDURE NO. 26

# ELECTRON MICROPROBE ANALYSIS

| REVISION LOG | | |
|---|---|---|
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## 1. PURPOSE AND SCOPE

This document outlines the procedures for sample analysis using the electron microprobe. The electron microprobe will be used to generate images, qualitative and quantitative chemical data for polished thin sections of waste rock pile materials, as well as material from alteration scars. The types of images will include backscatter electron images, which show the mean atomic number of sample material, as well as X-ray maps that show distribution of specific elements. Qualitative chemical analysis will indicate all elements present in a given 1-20 micron spot of a sample to levels of 1-5 wt.%.

Quantitative analysis will allow determination of the exact chemical abundance of chosen elements in a 1-20 micron spot on a sample surface.

# 2. RESPONSIBILITIES AND QUALIFICATIONS

The Team Leader and Characterization Team will have the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate staff to implement this SOP and for ensuring that the procedures are followed.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP, and receive specific training regarding these procedures, if necessary. Personnel trained on the CAMECA SX-100 electron microprobe will undertake the procedures outlined in this protocol. Qualified personnel include either the electron microprobe lab manager or senior research associate.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Team Leader.

# 3. DATA QUALITY OBJECTIVES

The electron microprobe will be used to address objectives 2, 3, 4 and 5 in the data quality objectives outline by Virginia McLemore for the "Geological and Hydrological Characterization at the Molycorp Questa Mine, Taos County, New Mexico".

- Determine how mineralogy, stratigraphy, and internal structure of the rock piles contribute to weathering and stability.
- Determine if the sequence of host rock hypogene and supergene alteration and weathering provides a basis to predict the effects weathering can have on mine rock stability.
- Determine if cementation forms in the rock piles and how does the cementation contribute to the stability of the rock piles.
- Determine how reactive are pyrite and carbonate minerals so that a representative sample goes into the weathering cells.

# 4. EQUIPMENT LIST

- Cameca SX-100 electron microprobe
- Denton DV-502 carbon coater

# 5. RELATED STANDARD OPERATING PROCEDURES

The procedures set forth in this SOP are intended for use with the following SOPs:

- SOP 1 Data management (including verification and validation)

- SOP 2 Sample management (chain of custody)
- Sop 24 Petrographic analyses
- Sop 29 Clay mineralogy analyses
- SOP 38 DI leach

# 6. PREPARATION

## A. SAMPLE SELECTION

Only a limited number of samples can be examined by electron microprobe. Therefore, samples will be carefully selected after petrographic analysis (SOP 24) and clay mineralogy (SOP 29) are completed. A DI leach (SOP 38) also will be performed on each sample to provide information on water soluble phases.

## B. SAMPLE PREPARATION

- Decide which type of sample preparation mount will be best suited to each sample. The options include standard petrographic thin section size, 1" round section, or grain mounts. If one of the first two is selected, prepare billet accordingly. For the third, place sample in labeled vial.

- For petrographic thin section of 1" round mounts send billets to commercial lab for probe-grade polishing.

- For grain mounts, prepare and polish according to the following procedure:

Equipment**:** Round slides or leucite/epoxy sample mounts, leucite slide holder, small piece of tin foil, 100, 220, 600 grit, and 12 micron magnetic grit laps (in MSEC thin section lab), 6, and 1 μm diamond powder (in probe prep lab), distilled water, cloth laps and glass mounting plates.

For soil samples: Spray red molds with silicon. This is important because it is very difficult to remove samples from molds without silicon spray. Write sample number on side of mold with Sharpie. Place soil sample in mold. Include fine fraction and larger pieces if possible, and break up some pieces with a metal scoop. Try to spread sample uniformly on bottom of mold. Pour epoxy into mold so that it covers entire sample. It is acceptable for larger pieces to stick up out of epoxy. Stir epoxy and soil sample with a toothpick to ensure that epoxy has mixed with sample. Smaller sized particles have a tendency to move to the outside of the mold, so try to prevent this at this stage. Place mold in an aluminum boat and write sample number on boat. To cure, place in oven for at least 4 hours at setting 3. Remove sample from oven and remove base of mold. Push sample out of the bottom of mold. Remove sample number from mold using acetone.

Polishing Procedure: It is difficult to write a generalized polishing procedure, because the length of time needed for each step is variable from sample to sample. However, here are some general guidelines that may be helpful.

1) Once sample has cooled to room temperature following epoxy curing, mount in leucite sample holder. Sample may need to be held in with a small piece of tin foil.

2) Find the 100 grit diamond magnetic grinding lap and the wheel. Put the wheel on the polishing mount and set the lap in place.

3) Grind the sample down until most of the grains are exposed, but not ground down too far. Less than half of the average grains should be exposed or they could be ground to nothing on the next grinding step. Take care at this step to keep the sample flat. Look at the sample occasionally, and if it has become wedged or domed, attempt to correct the problem. Once the sample is done, rinse the sample (still in the sample holder) in tap water. Dry the lap carefully and put away.

4) Find the 220 grit diamond lap, and mount onto the wheel. Remount the sample, and grind for about 3 min. on the 220 wheel. Again, try to keep the sample flat. Rinse the sample when done, and dry lap.

5) Find the 600 grit diamond mesh. Grind the sample for 3 minutes on this wheel. Once this step is complete, the sample should be removed from the sample holder, placed in a small beaker of water and ultrasonified for about 20 seconds. Rinse the sample holder in tap water. Dry lap and wheel.

6) Find the 12 micron diamond mesh wheel. Grind the sample for 3 minutes on this wheel. Once this step is complete, remove the sample from the sample holder, place in a small beaker of water, and ultrasonify for about 20 seconds. Rinse the sample holder in tap water. Dry lap and wheel.

7) Find the 6 micron polishing cloth. This should be stuck to a glass backing plate and stored in a ziploc bag. Remount the sample in the sample holder. Put a pinch of 6 micron diamond powder onto the cloth, and wet it with distilled water. Polish the sample for 3 minutes, changing the sample orientation occasionally, and applying moderate pressure. Check the sample now and then with the microscope to see how the polish is progressing. The sample surface should have few pits and an even network of small scratches. If the sample is still pitted at the end of 3 minutes, keep polishing for another 3 min. If, at the end of the second 3 minutes the sample doesn't appear to have progressed at all, get the 15 micron polishing cloth and again do 3 minutes on. Ultrasonify and go back to the 6 micron cloth. When you have achieved the no-pit/even-scratch surface, remove the sample from the holder and ultrasonify for about 20 seconds. Return the 6 micron cloth to the ziploc bag.

8) Find the 1 micron polishing cloth. Prepare in the same way as the 6 micron cloth, using the 1 micron diamond powder. Polish the sample for 3 minutes on this wheel. Rinse the sample. Return the 1 micron polishing cloth to the ziploc bag.

Notes:

- If polishing a number of samples, it is easier to go through each polishing step with all of the samples before beginning the next step.
- The 6 and 1 micron polishing cloths are easily contaminated and once contaminated will not give a good polish. Always ultrasonify the sample and rinse the sample holder before using these cloths, be careful not to spill anything on them, and cover or bag them when not in use.

## C. CARBON COATING

Prior to carbon coating, the sample surface is cleaned with petroleum ether under a hood. A sputter coat of carbon corresponding to a thickness of 20nm of carbon rod (monitored by the color produced on a brass plate within the coater) is applied to the sample using a DV-502 carbon coater according to the following procedure:

1) Turn on chiller (silver toggle on front of chiller).
2) Check that **MP VENT** toggle is in a closed position, (parallel to front of machine).
3) Check that **MECH and DIFF PUMP** toggle switches are **OFF**.
4) Turn on **MAIN POWER**.
5) Turn on **MECH PUMP**.
6) Turn **Thermocouple gauge** on**.**
7) On main pressure gauge, monitor Foreline pump pressure.
8) Open **BACKING VALVE** 3 turns. Make sure that MAIN VALVE and ROUGHING VALVES are shut.
9) Turn **DIFFUSION PUMP ON**. The diffusion pump must warm up for ~20 min before it can be connected to the main chamber (bell jar).
10) Re-check to be sure that the **MAIN VALVE** is shut.
11) **Vent Chamber** by moving the black toggle labeled "**Chamber Vent**" just below the bell jar from a position parallel to the front of the machine, to a position at a right angle to the front of the machine.
12) Carefully lift the bell jar and rotate the assembly until it catches on the shaft key and remains elevated.
13) Prepare the carbon rods. Sand the end of one rod flat. Sharpen the other using the metal die to form a tapered end approximately mm in length with a rounded tip. Replace the old carbon rods with the new ones.The flat rod is inserted on your left and the pointed one on your right.
14) Place the sample(s) on a clean brass plate under the carbon rods, in a position

forward of the flat rod. If the bell jar is dirty, clean off a small spot so that the rods can be viewed (use welding goggles) while the carbon is being deposited.

15) Check the sealing surface of the bell jar to make sure that it is clean. Rotate the bell jar and slide it back down onto the sealing surface.

16) **Close MAIN CHAMBER VENT.**

17) **Close BACKING VALVE** (this disconnects that roughing pump from backing the diffusion pump).

18) **Slowly open ROUGHING VALVE** (this allows the roughing pump to begin pumping the bell jar chamber). Open slowly while you hear a gurgling noise, and then when the gurgling noise stops, open completely.

19) Wait until the **chamber pressure** of the main gauge reads 100 millitorr. Be sure that the gauge is reading CHAMBER pressure, not FORE pressure.

20) **Close roughing valve**. **Change** the pressure gauge over to "**FORE**" (foreline pressure), and wait until the needle on pressure gauge to drops to below 40 millitorr.

21) **Open backing valve**. This reconnects the roughing pump to the diffusion pump, where it acts as a backing pump. DO NOT leave the diffusion pump running for a long period of time unpumped by the backing pump (ie. roughing valve open, backing valve closed).

22) **Open main valve** (opening the diffusion pump to bell jar chamber).

23) Allow the chamber to pump for 30 min to 1 hr.

24) Use the **Discharge Gauge** to check the chamber vacuum. Turn the **Range Selector** to Zero. Push the red **Vacuum Read** button. Light should come on and stay on and needle should go to zero. Change the **Range Selector** to $10^{-4}$. Needle should go to low numbers (1-4).

1.

25) Turn on **FILAMENT POWER** and **FILAMENT GLOW** switches.

26) Turn up **"FILAMENT ADJUST"** wheel until filament current is 35 amps or until thinned carbon rod glows. Leave the current at this point for ~10 sec. Then increase the current until you see small glowing particles being emitted from the carbon rod. Allow evaporation to take place for between 10 and 15 seconds until the brass plate on which the sample(s) rest appears blue. This is equivalent to a 20nm thick carbon coat. An equivalent carbon coat is applied to the standard blocks.

27) Turn off **FILAMENT POWER and FILAMENT GLOW** switches. Allow system to cool for 5-10 min.

28) Close **MAIN VALVE**. This disconnects the diffusion pump from the bell jar chamber.

29) **Vent bell jar** as described in step 10-12. Open bell jar and remove sample.

30) Close bell jar. Pump system down as described in steps 15-21. Leave the bell jar at 100 millitor.

31) Shut system down as follows:

32) Turn **DIFFUSION PUMP** off and allow pump to cool for 20-30 min.

33) Turn cooling water off.

34) Close **BACKING VALVE.**

35) Close **ROUGHING VALVE.**

36) **MECHANICAL PUMP** off.

37) **MAIN POWER** off.

38) Open **MECHANICAL PUMP VENT. Toggle labeled MP VENT should be moved** into the open position (away from machine). Wait until hissing noise stops. Close **MP VENT** toggle.

Note: Once samples have been carbon coated, they should be placed in a dessicator and handled only with gloves.

# 7. PROCEDURES

## A. QUALITATIVE ANALYSIS:

- Samples will be examined using backscattered electron microprobe imaging (see procedure below).
- The number and distribution of major phases, trace phases and phases present as cements will be noted.
- Zonations within phases will be noted.
- Phases will be examined qualitatively using short collection time X-ray maps and qualitative X-ray scans (see procedure below).
- Backscattered electron images will be collected and annotated to illustrate the above observations.

Qualitative data will be acquired utilizing the ***Imaging*** and ***Quali*** software embedded within the main ***SX100*** program. The image programs allow the acquisition and processing of X-ray maps and backscattered electron (BSE) images. All imaging subprograms are loaded and started using the ***Image*** icon. Qualitative subprograms used for the acquisition and processing of WDS survey and line scans are loaded and started using the ***Quali*** icon. The ***Acquisition*** icon within ***Image*** accesses the Image Acquisition setup window. The ***Acquire*** icon within ***Quali*** accesses the Quali Acquisition setup window. The respective windows are used to set the specifics of a given scan such as scanning mode, signal(s) selection, scanning parameters and to

assign a unique filename to the scan. The windows also display the current status of the SX100 machine conditions: beam accelerating voltage (kV), current intensity (nA), selected beam spot size or diameter (μm), and current stage position (x,y,z). When the desired scan and setup conditions are stored the current machine conditions are recorded and can be recalled or reloaded from the ***Setup*** pull down menu at the bottom of the screen (right hold (RH) on setup icon).

- **Experimental or Machine conditions:** The SX100 typically operates at an accelerating voltage of 15kV and a beam current of 20nA. Higher or lower values may be selected depending on the purpose of the scan. For instance, a higher beam current is required to detect elements present in concentrations less than a few percent (trace elements). For qualitative scans a point beam is generally utilized. A specific beam diameter may be applied when a line scan is performed through a beam sensitive sample or when large stage steps (>2□) are used. Machine conditions will be optimized based on the desired scan output.

- **Signal selection:** Options for selection of signals that will be acquired are located in the lower left quadrant of the Image (or Quali) Acquisition setup window. Types of signals selected include **BSE** and/or **WDS signals**. The number of Video and/or WDS channels available for simultaneous acquisition depends on the selected definition (see scanning parameters). Typically the BSE signal will be collected on the VS2 Video channel and up to three WDS channels will be selected and set to collect X-ray signal(s) of the desired element(s). An associated Display window opens at the same time as the Image (or Quali) Acquisition setup window and is used to display scans in real time during acquisition.

- **Scanning Mode:** Options for the selection of scanning mode are displayed at the top of the Image (or Quali) Acquisition setup window. Applicable modes include:

  - **Beam scanning mode *(Beam)***:
    In this mode the stage is held stationary while the beam is digitally moved from pixel to pixel. Acquisition by beam motion is utilized for images of small areas at high magnification (scan width < 100□m) or for short line scans (scan length < 100□m). At high magnification spectrometer defocalization is negligible. Although not preferred, beam scanning can be employed at lower magnification if a dynamic correction of the selected spectrometer is performed. With dynamic correction, the crystal(s) move at the same time as the beam to compensate for the change in Bragg angle. This requires a restricted spectrometer configuration (single spectrometer or two opposite spectrometers).

  - **Stage scanning mode *(Stage)***:
    In this mode the stage moves digitally from pixel to pixel while the beam is kept in a static or "fixed probe" position. Because spectrometer

defocalization is not an issue, this is the preferred mode for scans across large areas of the sample (scan width > 100□m). An ***"On The Fly"*** motion in which the stage moves continuously during acquisition will be utilized. "On The Fly" motion is selected for pixel times of 500 ms or less. A ***"Step By Step"*** mode in which the stage stops and counts at each pixel is mainly used for mapping at low definition and for long pixel count times (i.e. trace element mapping).

- **Scanning Parameters** are selected based on the type of qualitative analysis (i.e. ***Image*** or ***Quali*** acquisition (see below).

**1) IMAGE ACQUISITION (BSE and X-ray Images)**

Scanning parameters selected within the ***Image Acquisition*** setup window for the collection of BSE and X-ray images include pixel time (ms), number of accumulations, current scan and definition. Values for these parameters are selected on a case by case basis depending on the desired output of the scan. The **Pixel Time** (ms) corresponds to the counting time per each individual pixel of the desired scan. Alternatively, a **Frame Time** (s) equivalent to the total acquisition time of the scan can be selected. The program automatically computes the frame time based on the selected pixel time and vice versa. The selected pixel time is dependent on the detected signal intensity. Increasing the pixel time improves the signal intensity and counting statistics and consequently the resolution and accuracy of the scan. However the total acquisition time (frame time) will also increase. If only the Video signal (BSE) is selected, the minimum pixel time is 1ms. Selection of WDS X-ray signals increases the minimum pixel time to 5 ms. The **Accumulations** designates the number of frames of the scanned area that will be accumulated. If low signals (elements with low concentrations) are expected, either the number of accumulations or the pixel time will be increased to improve the signal to noise ratio.

The parameter related to the magnification or mapped area size is designated by selecting either current scan, raster length or pixel step. If **Current Scan** is selected, the width of the current scan is the length in X of the scanned field and the effective mapped area corresponds exactly to the area displayed on the real time monitor. This option is preferred for images acquired by beam scan. Selecting **Raster Length** allows the selection of a specific width of the scan area in X. **Pixel Step** is selected when designating a specific step size (distance between two adjacent pixels). When current scan or raster length options are selected the program automatically calculates the step size. The **Definition** parameter allows selection of the number of pixels in the X and Y direction. The values displayed in the menu (right hold; RH) correspond to common values. Scanning parameters will be set to optimize conditions such that maximum instrument efficiency is maintained while still obtaining the desired output.

Two types of images can be collected:

**a) Backscattered Electron Images (BSE)**

BSE images of the sample will be acquired by rastering the source pixel by pixel and line by line over a two-dimensional area and collecting the signal intensity of backscattered electrons versus the beam or stage position. The resulting information is stored and provides an image showing differences in average atomic number (Z), with brighter areas corresponding to more intense signal (higher average Z) and darker areas to less intense signal (lower average Z). The observed contrast between phases of differing mean Z is purely qualitative. For general reconnaissance of the sample the beam is rastered using a frame time of 1.07s and a 1024*768 definition. To collect an image the frame time is increased to 12.8s and the image is pinned (frozen) and transferred to the SUN monitor where it is typically saved in a TIF format.

**b) X-ray maps**

X-ray maps are acquired by collecting characteristic X-ray signals emitted by the sample under electron bombardment. A total of three spectrometers are available to collect the characteristic X-rays of up to three elements of interest simultaneously (one element/spectrometer). The X-ray signal intensity reflects the elemental concentration in the specimen (the higher the signal, the higher the elemental concentration). A 2-dimensional map that shows the elemental distribution and concentration across the scanned area is generated for each element selected. Images can be processed in gray scale in which bright areas (hi intensity per pixel) indicate high concentrations of the element and regions with low concentrations appear dark gray. Alternatively, a false color scheme can be utilized to enhance the contrast within the sample. Typical short collection maps will be set to a pixel time of 7ms, 1 accumulation, and a 512*512 definition. The scan size will vary depending on the area of interest.

**2) QUALI ACQUISITION (WDS Survey and Line Scans)**

Scanning parameters selected within the ***Quali Acquisition*** setup window for the collection of WDS surveys and line scans include the dwell time, number of channels, and accumulations. The total scan time is the product of the channels * dwell time * accumulations. The **dwell time** (ms) corresponds to the counting time per each individual channel of the desired scan. Alternatively, a **Line time** (s) equivalent to the total acquisition time of the scan can be selected. The program automatically computes the line time based on the selected dwell time and vice versa. Increasing the dwell time improves the counting statistics and consequently the resolution of the scan. However the total acquisition time (line time) will also increase.

The number of **Channels** corresponds to different crystals in the instrument**.** The **Accumulations** represents the number of repetitions acquired of the scanned area. Increasing the accumulations improves the signal to noise ratio. This may be desired if extremely low signals have to be acquired. Scanning parameters will be set to optimize conditions such that maximum instrument efficiency is maintained while still obtaining the desired output.

Two types of qualitative scans can be acquired:

**a) WDS Survey Spectrum *(WDS Scan)***

Reconnaissance technique used to determine what elements are present in an unknown sample. When the WDS mode is selected within the ***Quali Acqusition*** setup window, the WDS spectrometers are scanned over their respective wavelength ranges and a record of X-ray intensity versus spectrometer position is recorded. For cursory examination of the sample a short collection time, typically 1-3 minutes is selected. This will detect all elements present in concentrations >1-5 wt% (lower limit is dependent on the element selected). The scan is set to 1000 channels and one accumulation. The resulting spectrum exhibits the characteristic X-ray peaks of the elements present at a specific point on the sample.

**b) Qualitative Line Scans**

A line scan generates a profile of the distribution of a selected element by scanning the probe along a single line. Line scans will be utilized to determine whether or not selected elements are homogenous along a designated cross-section of the sample. To set up a line scan, parameters specific to the scan are selected from the ***Quali Acquisition*** setup window. Qualitative line scans can be acquired utilizing either ***stage*** motion or ***beam*** motion. The stage scanning mode (stage is moved digitally while the spectrometer positions and beam remain fixed) will be used for most scans. For typical short collection scans, the BSE signal will be collected on video channel VS2, up to three WDS X-ray lines corresponding to specific elements of interest will be selected, and stage positions (x,y,z) corresponding to the starting and ending (stop) points of the desired line scan stored (***valid*** icon). A typical line scan will be acquired using 1000 channels, a 100-200 ms dwell time, and one accumulation. Once all fields have been entered the scan is given a unique filename and the scan parameters and experimental conditions are saved via the ***store*** icon. The programmed line scan can be acquired immediately or a series of line scans can be stored and run in sequence.

## B. QUANTITATIVE ANALYSIS

### CALIBRATION for Quantitative Analysis

1) From the main SX-100 toolbar, **LC *QUANTITOOL***. A quantitool window will open. This takes up to a minute.

2) On the left-hand side of the **QUANTITOOL** icon, **LC *BULK***

3) From the **QUANTITOOL** window, **LC *CALIBRATE***.

4) Make a list of the elements that you want to calibrate, decide on the standard, crystal, background positions and slope factors that you will need to use. In order to do this, you can use the **UTILITY** tool. To access **UTILITY**, from the

**QUANTITOOL** window, **LC *UTILITY***. Under **FILES,** select "**calib WDS Peak-Background**". You will now see a list of previously-done calibrations for specific elements. Use these to give you an idea of how to proceed with your calibration. Also, you can use the list of standards in the "standards" section of the instruction binder to help decide which standards and reference materials may be useful. Keep in mind that to save time, you can calibrate more than one element on a single standard.

5) Calibrating

a) Decide which element to do first, and in the **CALIBRATE** tool, choose the appropriate standard.
b) Deselect the spectrometers that you do not need.
c) To select standard, ***RH*** on the arrow next to **STANDARD**. Select the standard that you want.
d) Select the appropriate accelerating voltage (normal 15 kV), and appropriate probe current (usually 20 nA)
e) **RH** on the **CONDITION** line. Select "Recall calib" and then slide right to choose the appropriate setup for the element that you want to calibrate.
f) **LC *ACQUIRE***. Once the stage moves to the standard, optically focus the sample using the Z wheel. When message "**Adjust column and stage conditions**" appears, **LC *OK***.
***g)*** A peak search will take place now. When the peak search is done, verify that the peak looks OK by opening the WDS window, and looking at the display. If peak looks OK, **LC *OK*** when you get the "**Verify Peak Position**" message
h) When you get the "**Set PHA**" message, **LC *YES***
i) When message appears "Adjust Stage for Measurement", move the stage slightly and refocus using the Z wheel. **FOCUS VERY CAREFULLY!!!** When done, **LC *READY***.
j) Counting will begin. At least 3 peak and background counts must be done on the standard, and you will be prompted to move the stage each time. You must **LC *OK*** when a new position is selected.
k) Do enough count cycles so that the "**meas. dev.**" is equal to, or less than, the "**theo. dev**."
l) Once the "**meas. dev**." is similar to the "**theo. dev**.", LC "***stop calib***"
m) Next, you have a chance to remove any counts that you don't want. **LC "*confirm*"** to go ahead and remove counts.
n) **LC *STORE*** when done.
o) Go through this process for all standards.

**PREPARING the DEFINITION FILES**

Set up **DEFINITION** for analysis. This selects that elements that you want to analyze, sets the analytical conditions and the order of analysis.

1) On the **QUANTITOOL** bar, **LC *DEFINE***.

2) **RH *TYPE***- select ***GEOLOGIC***

3) **RH *SPECIES***- select appropriate species.

4) **LC *LABEL***. Change the label to a unique identifier. But, you must keep the first 4 letters of the name. For instance OLIV-7411 could be a label for olivine analysis set up on April 11, 1997. An alternative approach is to label the file with the beam size, such as OLIV-10 for an olivine file with a 10 micron beam.

5) Set accelerating voltage. Usually 15 kV

6) Set probe current. Usually 20 nA

7) Select beam size on the ***COLUMN*** window.

8) **RH *APPEND*** and select elements that you want to analyze in order of increasing Z. If you make a mistake, you can use the ***"DELETE"*** and ***"INSERT"*** commands.

9) Select beam size on the ***COLUMN*** window.

10) Go to scanning mode and increase the magnification to a high value.

11) Return to ***FIXED PROBE*** mode**.**

12) When done, **LC *STORE***.

- **Updating a DEFINE file**

The "**DEFINE**" file saves all of the instrumental conditions at the time that it is saved. Therefore, before starting an analytical session, you need to re-open and re-store the **DEFINE** file.

1) **RH *LABEL***, choose appropriate label.

2) If necessary, correct the number of oxygens, and the number of $H_20$ (particularly if you're using "misc")

3) Make sure that the beam current, HV, and beam size are set correctly on the ***BEAM SETUP*** panel

4) Set scanning magnification to a high value

5) If you've recalibrated any element, and want to incorporate the new calibration information in your file, go to ***UTILITY*** and see if the old calibration file, and the new one both exist. If not, the information in the old and new were sufficiently similar that the old one was automatically overwritten by the new. Identify the files that you want.

**To proceed with an INDIVIDUAL SPOT ANALYSIS**

In single point mode (not actually used much, but try this first to get used to the steps that you'll need to follow on a regular basis).

1) **LC *ANALYZE*** on the **QUANTITOOL** tool bar.

2) RH *ACQUIRE*

3) Select ***GEO SINGLE POINT***.

4) **RH *CURRENT*** area. Select appropriate current.

5) **RH** in the **LABEL** area. Select the appropriate label.

6) Add comment.

7) **LC *CONTINUE***

8) Find the spot that you want to analyze using the optical microscope and/or **BSE** ( here you will need to switch from ***FIXED PROBE*** to ***SCANNING*** mode). I virtually always use BSE, and only use the optical microscope for the final optical focus

9) In **BSE**, with the beam on, increase the magnification until your analysis spot fills the entire screen

10) Blank the beam by putting in the Faraday cup.

11) ***Optically focus*** in the SX-100 TV window, using the Z wheel. You'll have to turn the light on if you've just been in BSE. **BE SURE TO NOT FORGET THIS STEP.**

12) For the next point, continue from step 7e.

**To proceed with STORING POINTS**
This is the more typical format to use for quantitative analysis. You can store a large number of individual points, line scans or gridded points, and the probe will run them for you automatically.

1) ***** Before***** beginning to store points, decide on which **DEFINITION** files you are going to be using, and run a GEO Single Point on reference materials using each **DEFINE** file to make sure that all definition files are working properly. This is particularly important if you've just updated the **DEFINE** files. This may seem tedious, but will save you time and trouble in the long run.

2) ***LC*** **XYZ** in the **QUANTITOOL** window.

3) Decide if you want to program points, line scans or grids. Select the appropriate choice. The following instructions are for **POINTS**.

4) If in the lower right corner of the XYZ window, the line has a number higher than 1 associated with the number of points, ***RH*** **DELETE**, and select **DELETE ALL**.

5) Find the spot that you want to analyze using the optical microscope and/or **BSE** ( here you may need to switch from ***FIXED PROBE*** to ***SCANNING*** mode, depending on what was being done last). I virtually always use **BSE**, and only use the optical microscope for the final optical focus

6) In **BSE**, with the beam on, increase the magnification until your analysis spot fills the entire screen

7) Blank the beam by putting in the Faraday cup.

8) *****Optically focus***** in the SX-100 TV window, using the Z wheel. You'll have to turn the optical light on if you've just been in BSE. **BE SURE TO NOT FORGET THIS STEP.**

9) ***RH*** on the bar next to **LABEL**, and select the label that is appropriate for the spot that you've selected for analysis.

10) Put in a unique comment for this analytic point

11) ***LC*** **ADD**

12) You can see all of your saved points by opening **VIEW**.

13) Record the point in your lab book and sample map. In my lab book, I normally record the analysis point number, the label file, the sample name and point number, and then some comments about the spot.

14) To save the next point, go back to step e and start there.

15) Once all of your points are stored, ***LC*** **DONE**.

- **RUNNING YOUR STORED POINTS AUTOMATICALLY.**

Once you've stored in all of your points, you can have the SX-100 run them automatically while you go off and do something else. There are two ways to doing this, either **Geo Multipoint** acquisition, or acquisition from within **TASK**. I recommend using **TASK**.

1) ***** BEFORE ***** starting an automated analytical run, you should always verify the spectrometers. To do this, go to **STEP 9** in the **"GETTING STARTED"** instructions, and go through the steps described there.

2) **Using TASK.**

3) ***LC*** **TASK** from the SX100 icon bar.

4) Delete any tasks on the list

5) RH QUANTI, select **xyzpos.dat** (or line, or grid file)

6) If you want the beam to turn off when the acquisition is complete (for instance, an overnight run) ***RH*** **TASK**, select **stop.tsk** Now xyzpos.dat and stop.tsk should both be on the list, with xyzpos.dat first.

7) *LC* START.

## STORING AND PRINTING DATA

Once you're done with a set of analyses, you will want to print the data, and also store the data in a format that you can access using a spreadsheet program.
You will need to store the data after:
1. Finishing a set of "**Geo Single Point**" analyses
2. Finishing a single XYZ multipoint run, either run by **TASK** or **GEOMULTIPOINT**

**THIS IS VERY IMPORTANT!!** If you do not store the data, it will be overwritten by the next set of analytical data.

**Storing the master file for analyses obtained in Geosinglepoint or Geomultipoint.**

1) Hold down the **RM** button within main window that contains all of the data.

2) Select ***FILE***, then ***STORE AS NEW FILE***.

3) Type in your directory, type in the filename, and click ***STORE.***

**Storing the master file from TASK.**

1) Hold down the **RM** in the window area.

2) Select ***HISTORY***

3) Slide right

4) Select ***STORE LOG AS NEW FILE***

5) Type in a filename. The directory cannot be changed at this point. Use the file manager to move this file to your home directory.

**Storing and printing the individual label files.**

Once you've done the previous step, all of your raw and processed data is safely stored. Now, you can use the **REPORT** tool to print your data in a nice format and also to save your data to a file in a format that can be read by Excel, or another spreadsheet.

1) On the open ***QUANTI*** menu, **LC** ***BULK*** on the ***PROCESS*** side of the window. A window called **Quanti Process** will open.

2) **LC** ***REPORT*** in the lower right corner.

3) **RH** on ***NAME***, select the label of the file that you want to print or save as a file You must make separate reports for each label used, ie. FELD, PYRO, OLIV, etc...

4) Now you can experiment with different scripts, which will give you different output formats. You can view the output on the screen when the device selected is ***SUNDISPLAY***

5) To print your output, change the device to ***LINEPRINTER***, select the script that you want, and **LC** ***REPORT***.

6) To save your output as an ascii file, change the device to ***FILE***, select the script that you want, change format from ***UNIX*** to ***DOS***, select your directory, type in a filename, and **LC** ***SAVE.***

I would recommend printing out your data using a script that includes the ID label of individual analyses (such as NORMAL or SIMPLE). The script titled

COMPOUND-COLUMN is a convenient format for importing the data into a spreadsheet. I would recommend printing data and saving data to a file.

# 8. SAMPLE HANDLING

Once samples have been run, they should remain in a dessicator and only be handled with gloves. Samples will be archived at least until the end of the project.

# 9.0 DATA REPORTING

The electron microprobe will be used to generate images, qualitative and quantitative chemical data for polished thin sections of waste rock pile materials, as well as material from alteration scars. The types of images will include backscatter electron images, which will be as tif files. Qualitative and quantitative chemical analysis will be in excel spreadsheets. Interpretative reports will be provided to the team.

# 10. QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance and quality control procedures are outline above in the procedures. Certified standards are used to calibrate the electron microbe.

# 11. REFERENCES

Cameca manuals