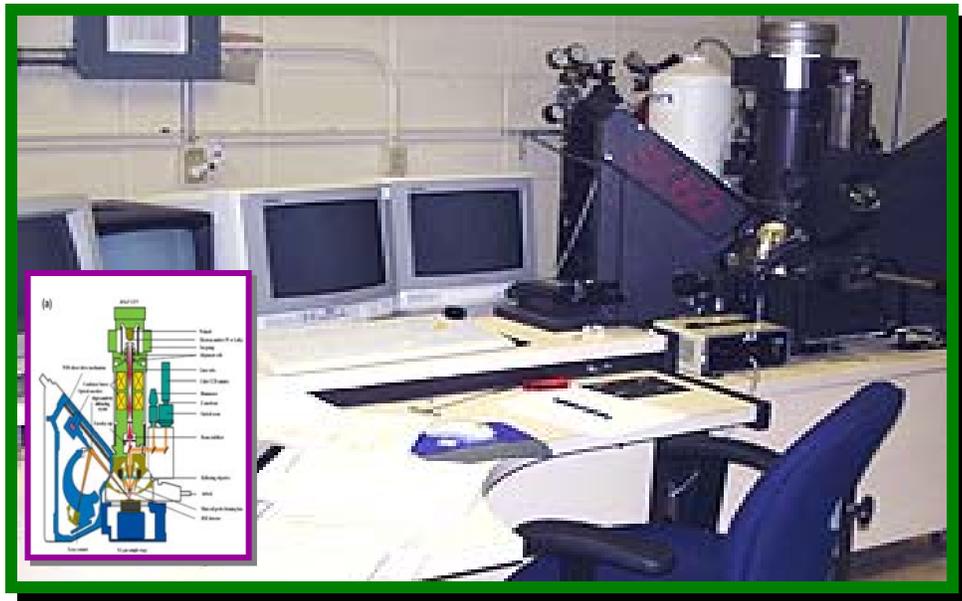


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Geology Dept. Document No.: EXACT - 003				
Author: <b>Miguel A. Santiago Rivera</b>				

**UPR-NSF Earth X-ray Analysis Center (EXACT!)**  
**Standard Operating Procedure**  
for:

# **Operating the Cameca SX-50 Electron Microprobe**



**UPR-NSF Earth X-ray Analysis Center (EXACT!)**  
X-Ray LABORATORY

DEPARTMENT OF GEOLOGY  
F- 304 Geochemistry Facilities at Physics Building  
University of Puerto Rico – Mayagüez Campus

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## Revision Log

Revision No.	Effective Date	Prepared By	Description of Changes	Affected Pages
R-0	September 11, 2006	Miguel Santiago	New Procedure	All
R-1	March 26, 2007	Miguel Santiago	X-rays Warning for Pregnant and Cancer patient 8.0 – Procedure	One Page -15
R-2	January 20, 2009	Miguel Santiago	8.2.3 – New Carbon Coater Procedure	<b>Page 16-20</b> of 33, were deleted
	January 20, 2009	Miguel Santiago	10.0 – Reference	<b>Page 32</b> , now page 29

# Operating the Cameca SX-50 Electron Microprobe

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# Operating the Cameca SX- 50 Electron Microprobe

## 1.0 PURPOSE

This procedure provides instructions for the operation of the Cameca Instruments. Incorporated for Model SX-50, electron microprobe system.

## 2.0 SCOPE

This SOP is a mandatory document and shall be implemented by all Researcher, Faculty member, Technician or Students participants when collection electron microprobe data.

## 3.0 TRAINING

3.1 All users of this SOP are trained by reading the procedure, and the training is documented in accordance with the GLP's.

3.2 The Geology Task Leader will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with GLP's.

## 4.0 DEFINITIONS

- 4.1 *SOP* - Standard Operational Procedure
- 4.2 *GLP's* - Good Laboratories Practices
- 4.3 *Machine Custodian* —The Machine Custodian is responsible for Cameca SX-50 maintenance and User instruction. This includes calibration of magnification marker and Energy Dispersive System (EDS) gain, EDS resolution check, basic repairs, and software, data taken and electronically-stored system backups and all instruction and training of Procedure Users.
- 4.4 *SEM* – Electron Microprobe
- 4.5 *SX-50* — SX-50 is the model name of the electron microprobe manufactured by Cameca Instruments, Incorporated (Purchase Date: month/year and Serial Number: )
- 4.6 *SX-50 system* — The SX-50 System includes the Cameca SX-50 electron microprobe.
- 4.6 *WDS* — The SX-50 contains four Wave -length Dispersive Spectrometers (WDS).

## 5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

5.1 Scientific Instrumentation Specialist

5.2 Faculty Member

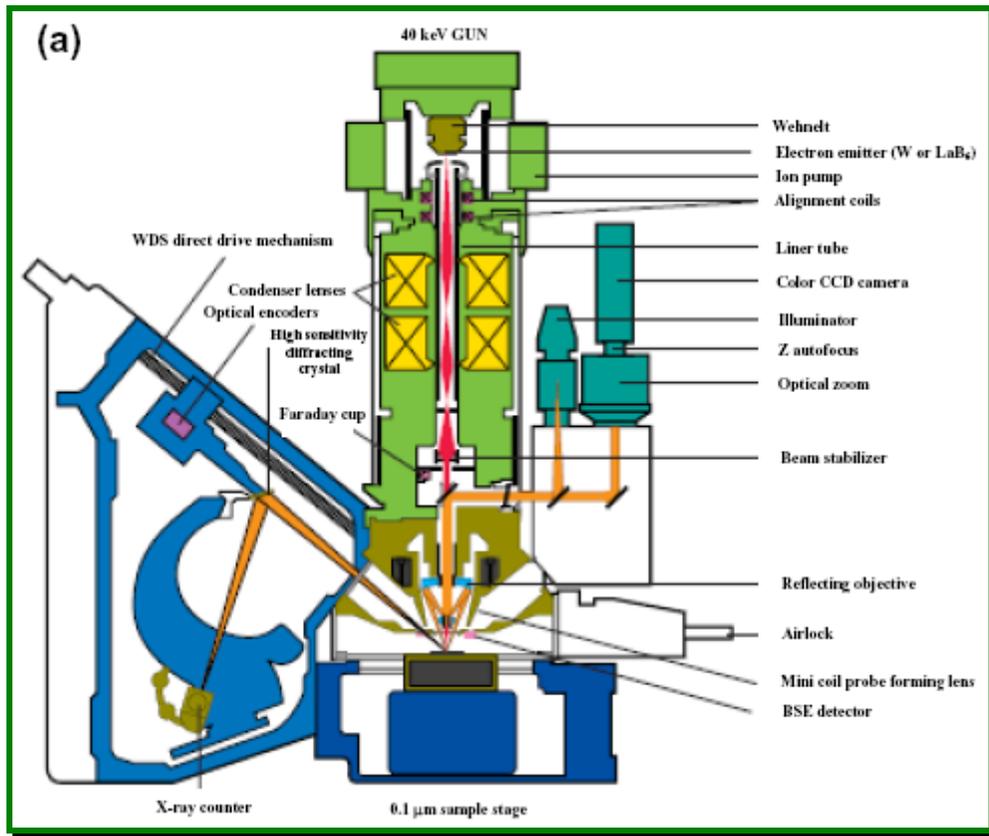
5.3 Researcher

5.4 Student Participant

## 6.0 EQUIPMENT

Descriptions of equipment constituting the SX-50 system are provided below.

6.1 *SX-50* — The Cameca SX-50 (Purchase Date: month/year and Serial Number: **057119** ) is an integrated computer controlled electron microprobe with capabilities for secondary and backscattered electron imaging, wavelength- and energy dispersive elemental acquisition, and image processing.



## **6.2 The CAMECA SX-50 counts with;**

- **Wehndt grid**
- **Electron emitter**
- **Ion pump**
- **Alignment coils**
- **Liner tube**
- **Color CCD camera**
- **Illuminator**
- **Zoom auto-focus**
- **Optical zoom**
- **Beam stabilizer**
- **Reflecting objective**
- **Air lock**
- **Mini coils probe forming lens**
- **BSE detector**
- **WDS direct drive mechanism**
- **Condenser lens**
- **Optical encoders**
- **High sensitivity diffracting crystal**
- **Faraday's cup**
- **X-ray counter**
- **0.1  $\mu\text{m}$  sample stage**
- **40keV gun**

## **6.3 Major components of an electron microprobe**

### **6.3.1 Electron optic system.**

- Electron gun
- focussing lenses and apertures
- associated electronics

### **6.3.2 Optical microscope and sample stage**

- optical/video apparatus for viewing sample
- high-precision stage that holds samples and standards

### **6.3.3 X-ray optic system**

- spectrometers
- secondary electron detector
- backscatter electron detector
- flow-proportional gas counters

6.3.4 **Vacuum system**

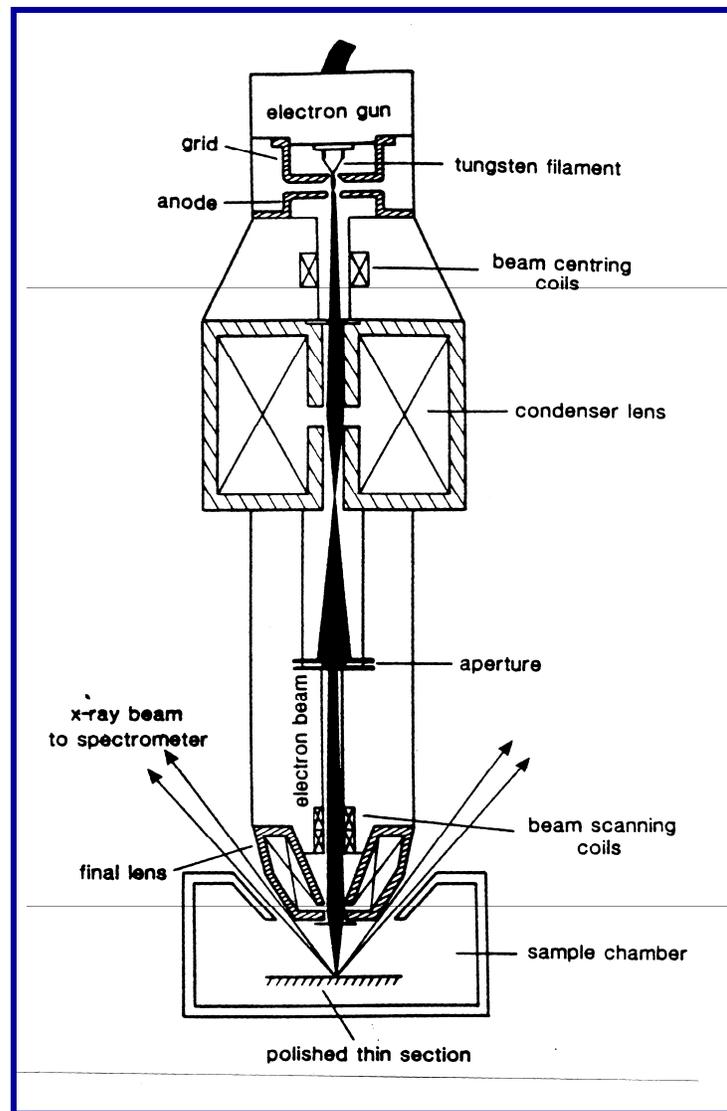
- roughing pumps
- diffusion pump
- ion pump

6.3.5 **Amplification/electronics**

- amplification of signals

6.3.6 **Computer Controller**

- interface between instrument and operator
- image collection and processing systems
- quantitative data reduction programs



## 6.4 Components of Initial EMP

- 6.4.1 **Electron Gun** - Turning on beam, setting beam current, acceleration, saturation level.
- 6.4.2 **Focusing Lenses** - Permit focused beam to hit sample.
- 6.4.3 **Sample Stage** - Allow precise positioning of sample under beam.
- 6.4.4 **Optical System** - Allows visual positioning of sample and selection of sample sites.
- 6.4.5 **Spectrometers** - Allow collection of X-rays emitted from the sample.

## 6.5 Distinguishing Electrical currents and voltages

- 6.5.1 **Accelerating Voltage**
  - The voltage used to accelerate the electrons towards the sample.
  - For geological samples, typically 15 kV. The higher the accelerating voltage, the larger volume of sample will produce X-rays. Ranges from 0.2-50 kV on the SX-100.
- 6.5.2 **Filament current**
  - Current used to heat the filament and generate electrons
- 6.5.3 **Beam Current**
  - The total current emitted by the filaments. Shown as “I emission” on the SX-100. Units in microamps ( $\mu\text{A}$ ).
- 6.5.4 **Probe Current (often referred to as “beam current”)**
  - The current actually delivered to the sample after the beam has passed through apertures and lenses

- For good analyses, this must be a very stable value. The SX-100 has a “beam stabilization” aperture with a feedback to the condenser lenses in order to maintain a very stable probe current.
- The current actually delivered to the sample after the beam has passed through apertures and lenses.
- For good analyses, this must be a very stable value. The SX-100 has a “beam stabilization” aperture with a feedback to the condenser lenses in order to maintain a very stable probe current.
- Beam sensitive samples should be analyzed with a lower probe current. Typical geological samples should be analyzed at 20 nanoamps.
- Glass can be analyzed at 10 nanoamps. Less counts will be obtained with lower probe current.
- High resolution SEM work can be done with 0.1 nanoamps (100 picoamps) beam current, see below and compare the size against each other.

· 0.1 nA

• 1 nA

● 10 nA

## 7.0 THEORETICAL BACKGROUND

### 7.1 ***What is an electron microprobe?***

An electron microprobe is a high-powered microscope that uses electrons, rather than light, to examine a sample. Because electrons are charged particles, they can be accelerated towards, and focused onto, a sample surface.

### 7.2 ***What can an electron microprobe do?***

Electron microprobes can be used to produce morphological (roughness and shape) or chemical images of a sample surface. The microprobe can also be used to qualitatively or quantitatively determine the chemical composition of a very small spot (1 micron) on a sample surface. The electron microprobe can be used to chemically characterize samples so small that they can be analyzed by virtually no other analytical technique, and also allows spatial resolution of chemical variability on a sample surface.

The capabilities of an electron microprobe are fundamental to many types of research, including many aspects of geology, such as ore deposition, environmental geochemistry, hydrology, petroleum research, igneous and metamorphic geochemistry, soil science, and sedimentology, as well as various aspects of biology and metallurgy.

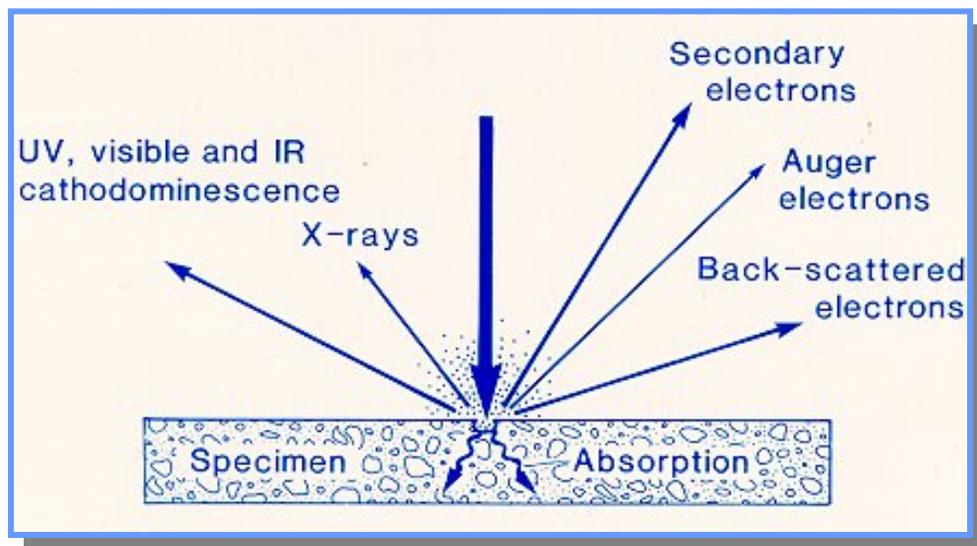
### 7.3 ***How does an electron microprobe work?***

- Electrons are produced by heating a tungsten filament to high temperatures (in the gun) and those electrons are accelerated away from the filament and gun towards the sample surface. Because the electrons are charged particles, they can be directed and focused using electrostatic lenses.
- The electron beam is directed onto the sample surface, and can produce secondary electrons, backscattered electrons, cathodoluminescence, or X-rays that can be analyzed to determine the sample's composition.
- As electrons from the primary beam hit the sample, they produce a number of secondary particles.

- **These include:**

1. *Secondary electrons* - Secondary electrons are produced when the microprobe electron beam interacts with the sample surface, producing low energy secondary electrons that are detected by the secondary electron detector. A secondary electron image appears 3-dimensional. A well tuned instrument can produce secondary electron images at magnifications of 100,000x, see the figure below.
2. *Backscattered electron* - Backscattered electron images can be generated from samples with relatively smooth surfaces, as opposed to secondary electron image that rely on sample roughness to produce an image. Backscattered electrons are high-energy electrons that rebound from the sample surface. The quantity of electrons backscattered from a given surface is proportional to the mean atomic number (Z) of the sample material. Hence, a high mean Z material will produce more backscattered electrons than a low mean Z one, see the figure below.
3. Auger electrons-When an atom is ionized in an inner shell, energy may be released by ejecting another bound electron, instead of producing an x-ray. These electrons can have characteristic energy. Most effective for low atomic number elements (<10), see the figure below.
4. Cathodoluminescence - Visible light emitted by minerals bombarded by electrons. Mechanism depends on the mineral in question. This property of certain minerals, particularly diamond and some types of feldspar, allow the beam to be viewed optically, which is useful for focusing, see the figure below.
5. Continuous X-rays - Most interactions between the primary beams do not produce the characteristic x-rays discussed earlier. Many electrons from the incident beam are simply decelerated by the interactions with outer orbital shell electrons from atoms within the sample. The energy released during these decelerations produces a continuous (or bremsstrahlung) x-ray spectrum. These x-rays are noise, and are undesirable, but unavoidable, see the figure below.

- Interaction between the electron beam and the sample also produces characteristic X-rays, that have a wavelength and energy proportional to the atom from which they are produced. An electron from the primary beam causes an electron from the target atom to be ejected from its orbital shell. In response to the empty energy shell, another electron will cascade down to fill the lower energy position, in the process releasing energy in the form of a characteristic X-ray.
- These characteristic X-rays can be detected by spectrometers to determine something about the sample composition.



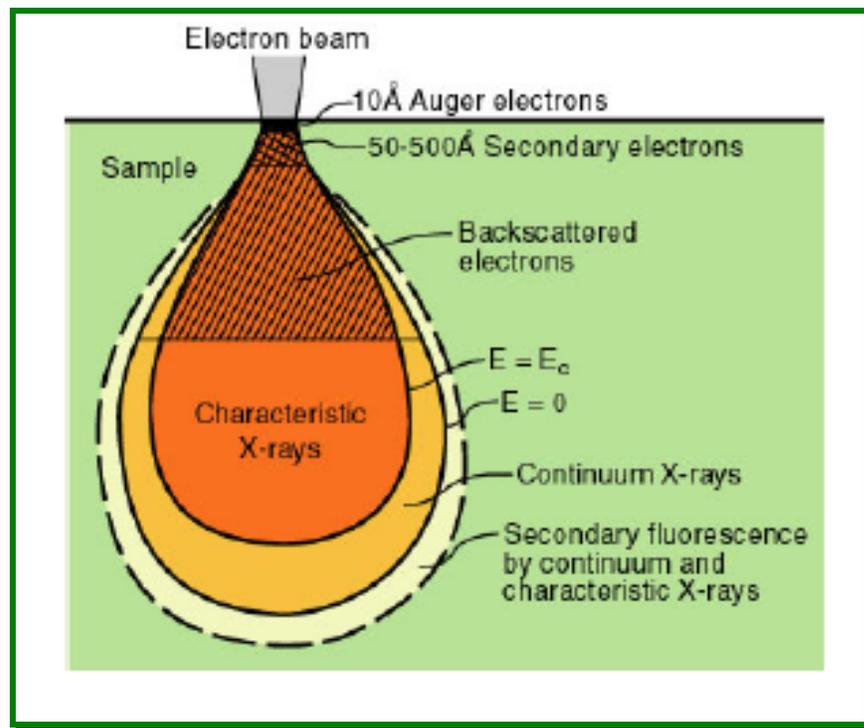
#### 7.4 ***Electron Probe Micro-Analysis (EPMA)***

The electron microprobe is an instrument designed to determine the elemental composition of small regions of solid samples, of several cubic micrometers. The principle of operation consists on the detection of X-rays generated by bombarding the sample with a focalized beam of energetic electrons. This beam ionizes the inner layers of the atoms, and consequently atomic vacancies are generated. Thus, electrons from outer layers occupy these vacancies through the emission of X-ray photons, among other phenomena, which correspond to the ionized species due to electron bombardment. An EPMA instrument is essentially a Scanning Electron Microscope (SEM) equipped with a set of X-ray spectrometers. Figure (a) reproduces the scheme of an EPMA similar to the employed in the measurements.

It is used in the identification and quantitative analysis of a wide range of chemical elements. It can also register in digital images the different processes due to the electron interaction.

As in SEM, the technique probes the sample a few microns in depth. Electrons can be absorbed, and then emit X radiation; backscattered (BSE) and directly collected by the detector; and finally, they can give rise to Auger secondary electrons, which are emitted by the sample surface as a result of electron bombardment.

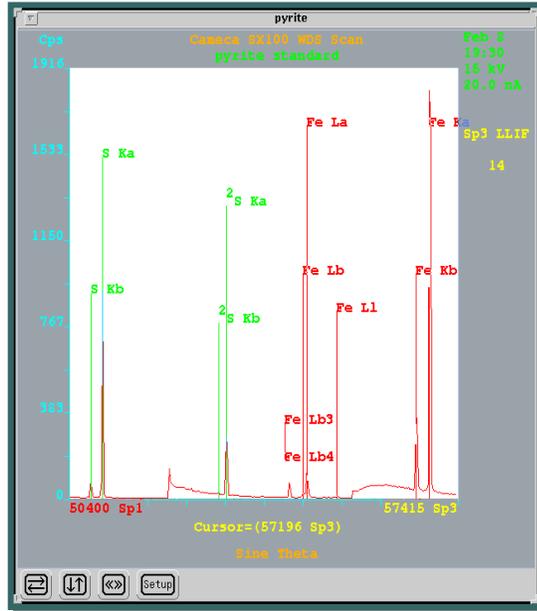
In the Figure (B) shows an example of the interaction in the volume along with some of the mentioned electron processes. X-ray spectra from the sample are obtained at different electron beam energies, which can be modified by controlling the voltage of the filament. This procedure provides the chemical composition averaged in different interaction volumes. The detected X-rays come from both the film and the substrate. The EPMA measurements can be performed by a Cameca SX-50 microprobe, which is equipped with four WDS (wavelength dispersion spectroscopy) and one EDS (energy dispersive spectroscopy) spectrometers.



*In the figure - B, there is an interaction volume in a sample irradiated by any electron beam.*

7.5

**Qualitative Analysis:** The elements present in an unknown sample can be determined qualitatively by analyzing the characteristic X-ray production from the sample. The following diagram shows a characteristic X-ray scan collected on a sample of pyrite, a mineral that contains Fe and S. X-ray peaks corresponding to those elements are labeled on the scan.



7.6

**Quantitative Analysis:** The electron microprobe can be used to make accurate determinations of the chemical composition of a very small (1-micron) spot on a sample surface. Quantitative analysis is accomplished by comparing the number of characteristic X-rays for a given element generated by an unknown sample with the number generated by a standard of known composition. An average geochemical analysis takes between 2 and 10 minutes to complete.

Quanti Report		
Point #3	an-3	
	Concen.	Cations
SiO2	44.183	2.048
Al2O3	35.447	1.937
CaO	19.223	0.955
FeO	0.380	0.015
SrO	0.105	0.003
BaO	0.005	0.000
Na2O	0.551	0.049
K2O	0.017	0.001
<b>Total:</b>	<b>99.911</b>	<b>5.008</b>

## 8.0 PROCEDURE

**Note:** All personnel performing work under the UPR-NSF Earth X-ray Analysis Center (EXACT!) quality program may follow this standard operating procedure (SOP) for electron microprobe analysis. May use their own procedure(s) as long as the substitute, only if, meets the minimum requirements prescribed by the UPR-NSF Earth X-ray Analysis Center (EXACT!) Plan or are better in the performing, and have been approved by the Director of the Department of Geology, UPR-Mayagüez Campus, before the commencement of the activitie(s).

**Note:** UPR-NSF Earth X-ray Analysis Center (EXACT!) personnel may produce paper copies of this procedure printed from the controlled-document electronic file. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear. Deviations from SOPs are made in accordance with Department Director approval and documented in accordance with the GLP's.

**WARNING:** X-rays equipments may produce radiation out of the equipment. Pregnant may risk in injury to the unborn child and/or may interfere with the treatment for cancer patient. If you are pregnant or cancer patient, consult your physician before to use and/or be exposed to x-rays equipments.

- 8.1 *Introduction* - The SX-50 system is capable of analytical work in the following general areas: elemental qualitative and semi-quantitative analysis using no standards, semiquantitative and quantitative elemental analysis using standards, image analysis, particle analysis, elemental mapping, elemental line profiles, and composite analysis of X-Ray maps and images. Procedure Users are responsible for determining what methods are appropriate for their work.
- 8.2 *Sample Preparation* - Samples to be examined with the SX-50 must be in a form or size that can be inserted into, or attached to, an SX-50 stage mount. Quantitative microanalysis routines assume that all samples for analysis will be relatively flat and that analysis sites will be normal to beam incidence.
  - 8.2.1 Before being placed in the SX-50, non-conductive samples may be given an electrically conductive coating. Carbon is most often used, however specific applications may benefit from other types of conductive coatings.
  - 8.2.2 For accurate quantitative analysis, the sample must be well polished and flat mounted. If the sample is not conductive, it has to be coated with a conductive layer. In most cases, a 20nm carbon layer is used as conductive layer, but in principle, any conductive layer could be deposited.

- 8.2.3 Carbon coating procedures are outlined in the **SOP Denton Desk IV – Carbon Coater** at the web in the X-ray Lab for the UPR-NSF Earth X-ray Analysis Center (EXACT), F-304-D, UPRM – Department of Geology.
- 8.3 *Selection of Standards* - The Procedure User may employ standards traceable to National Institute of Standards and Technology (NIST, formerly National Bureau of Standards, NBS) or well characterized materials published in credible technical journals and widely used by microanalysts for calibration of quantitative X-Ray analysis routines. Procedure Users may use standards from other sources for specific applications but must document the basis for usage of these standards in their notebooks.  
(**For example:** reagent-grade NaCl may be used as a Cl standard by stating the material source and noting the stoichiometric nature of this material even though it is not NIST-traceable).
- 8.4 *Sample Insertion and SX-50 Operation* - Detailed operating instructions for the SX-50 system are given in the SX-50, IMIX, and VISILOG (VISIVIEW) Manuals stored in the laboratory near the instrument. After training, the Procedure User should refer to the manuals when questions arise or consult with the Machine Custodian to solve specific problems.
- 8.4.1 *Sample Handling* – Mount the sample in the desired shuttle. If polished specimen is being analyzed, push the sample to the top reference plane in order to have a non-inclined analyzed plane with a sample surface at the correct Z altitude. The sample must be hold firmly in the shuttle. Use the screws available on the shuttles or use the CuBe springs to push the sample from their backside.
- IMPORTANT: Be careful that the sample does not exceed the shuttle plane level. If it does, the sample may hit an accessory (SE and BSE detector) in the chamber and may create severe damages. If you have a doubt on the conductivity between the coating layer and the sample holder, put some silver paint to ensure the conductivity.
- 8.4.2 *Sample Control* – Sample identification will be based on the unique identifier marked on the sample. This will typically be

an etched identification on the thin section that has been coated for analysis.

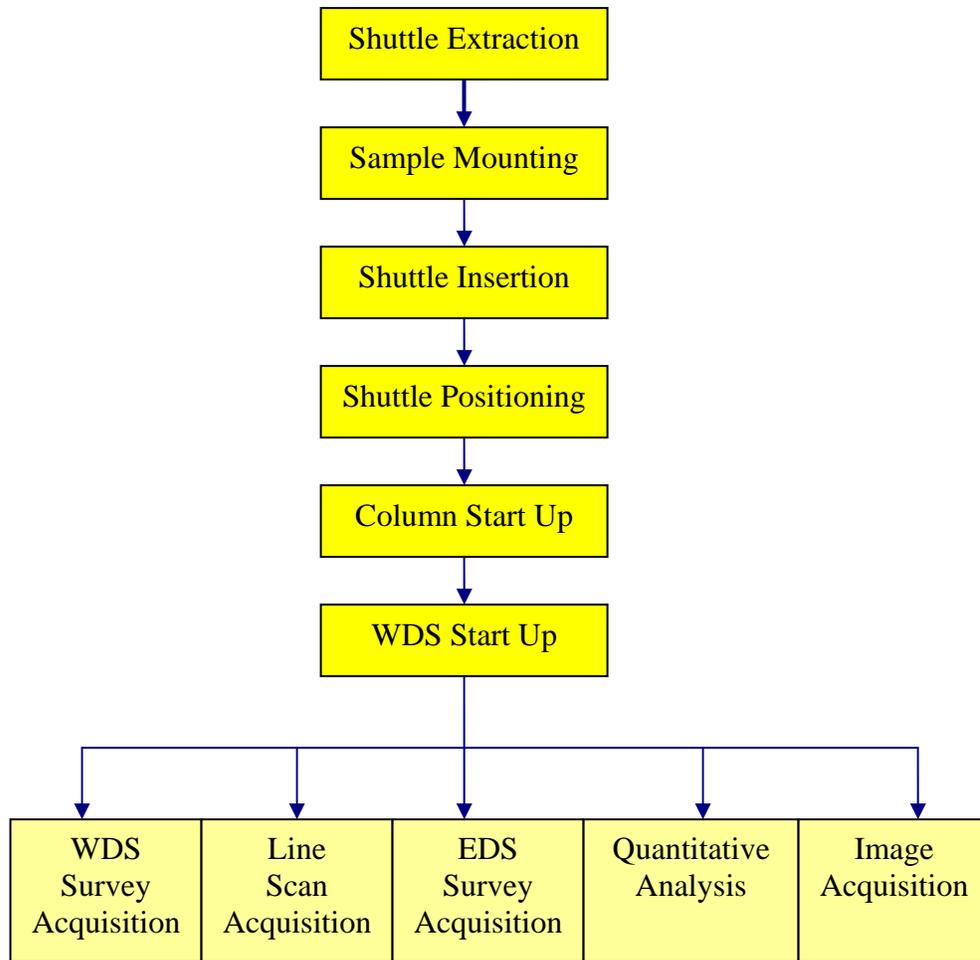
8.5 *Safety Considerations* - Normal-operating conditions as performed by trained Procedure Users present no safety hazards.

8.6 *Performing the Standard Operation Procedure for Electron Microprobe Cameca SX – 50.*

8.6.1 *Environmental Conditions* - Normal interior building temperature and humidity are acceptable for the operation of the SX-50 System. Cooling water for the SX-50 diffusion pump and electronics chassis is supplied by the building chilled-water system maintained in the range of 55 to 65 degrees Fahrenheit. Ambient air temperature for the SX-50 System should range between 60 and 80 degrees Fahrenheit. If environmental conditions move out of range during operating the SX-50 system in WDS mode, Procedure Users should take extra precaution to ensure system stability by checking standards often.

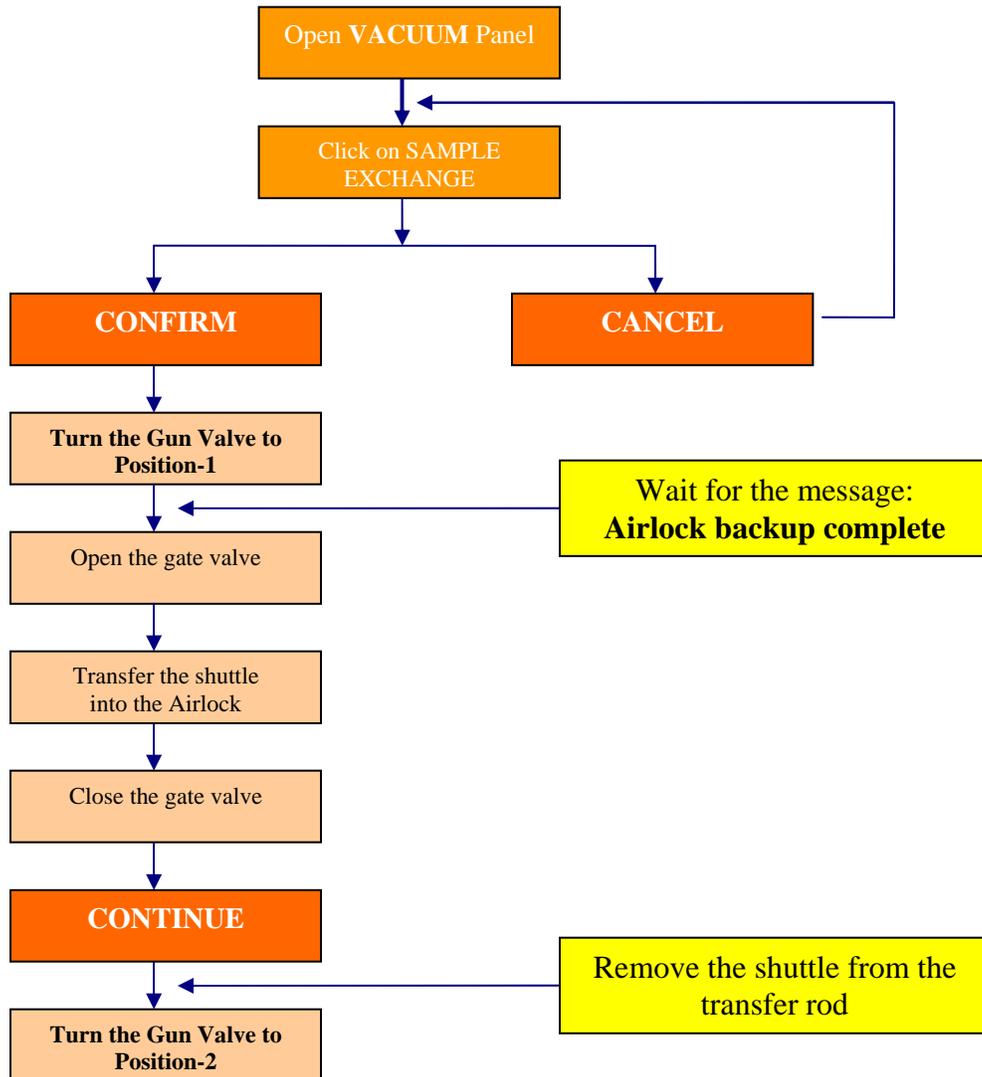
**IMPORTANT:** *The following flowcharts describe the sequence, which is executed when a new sample has to be introduced and analyzed in the SX-50 and for starting the machine only. You must accompany this SOP for Cameca-SX50 Electron Microprobe with the SX50: User's Guide Reference: 45-404-901 for more specific details or options.*

8.6.2 *General Analysis Sequence*



### 8.6.2.1 Shuttle Extraction

- *Shuttle Extraction – Sequence Synoptic*



- *Shuttle Extraction – Sequence Description*

- Click the left mouse button on the **Vacuum** button located in the SX-100 set up window.
- Click the left mouse button on the **Sample Exchange** button located in the **Vacuum Setup** window and confirm the sequence as shown on the figure. The sample stage is automatically driven to the transfer position.

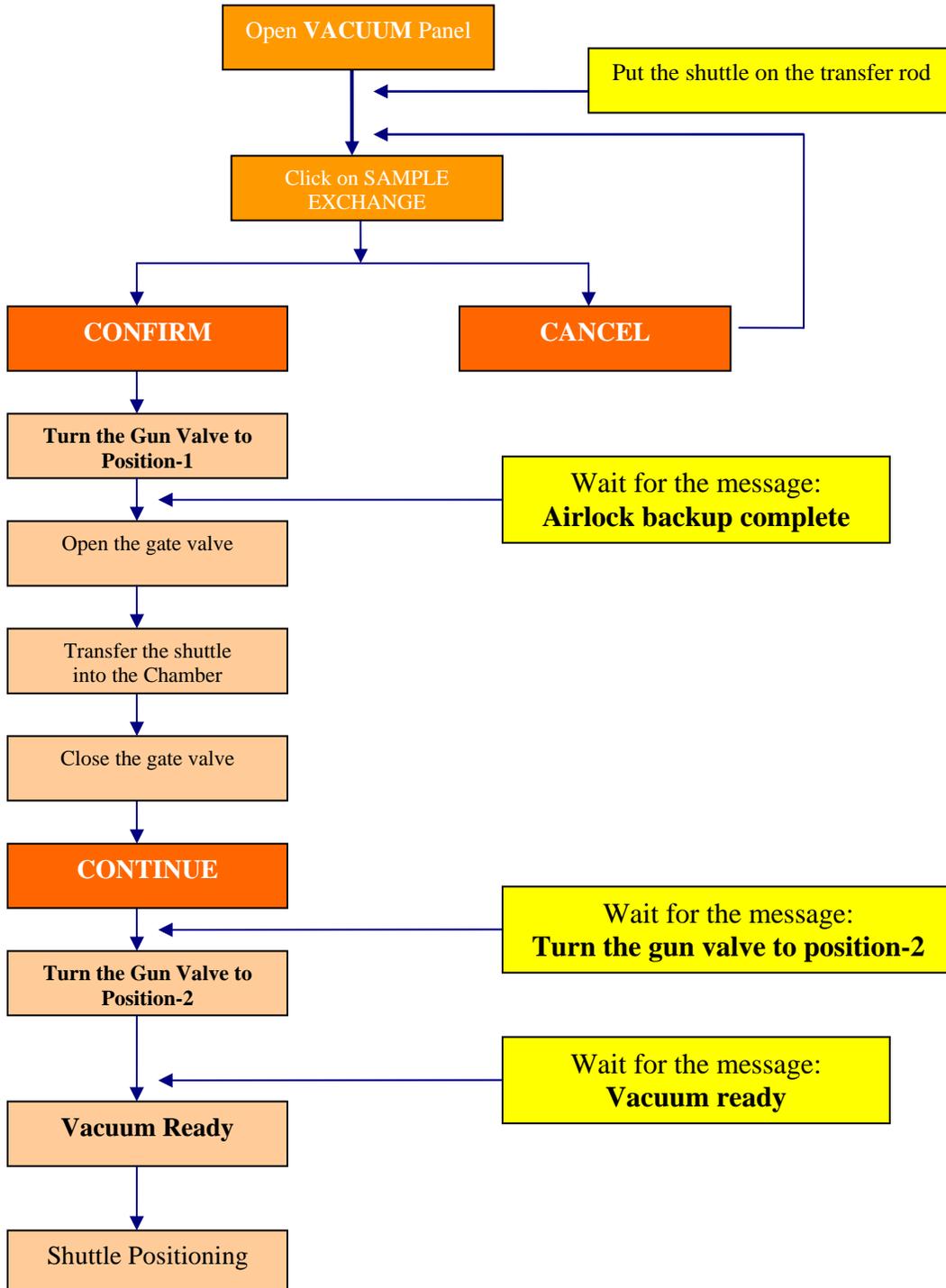
**Important:** The transfer position is stored and validated in the Configuration panel as the Airlock position in the **Stage Configuration** panel.

- Follow the Instructions displayed in the Vacuum Setup window:
  - **Turn the gun valve to position-1** to isolate the gun from the sample chamber. As soon as the gun valve is turned to position 1, the secondary valve (Diffusion pump/Analysis chamber) automatically closes and the airlock volume is automatically evacuated.
  - When the message **Airlock backup complete** is displayed in the **Vacuum Setup** window, open the valve between the airlock chamber and the sample analysis chamber.
  - Rotate the ring located on the transfer rod to the **Extraction position** (white arrow in the extraction position).
  - Push the transfer rod until it's maximum position and pull it fully back to move the shuttle out of the analysis chamber in the airlock chamber.
  - Close the valve between the airlock chamber and the sample analysis chamber.
  - Click the left mouse button on the **Continue** button located in the **Vacuum Setup** window. The airlock is automatically vented and the analysis chamber is pumped again.
  - Open the airlock and remove the shuttle from the transfer rod.

8.6.2.2 **Sample Mounting-** Place the sample.

### 8.6.2.3 Shuttle Insertion

- *Shuttle Insertion – Sequence Synoptic*

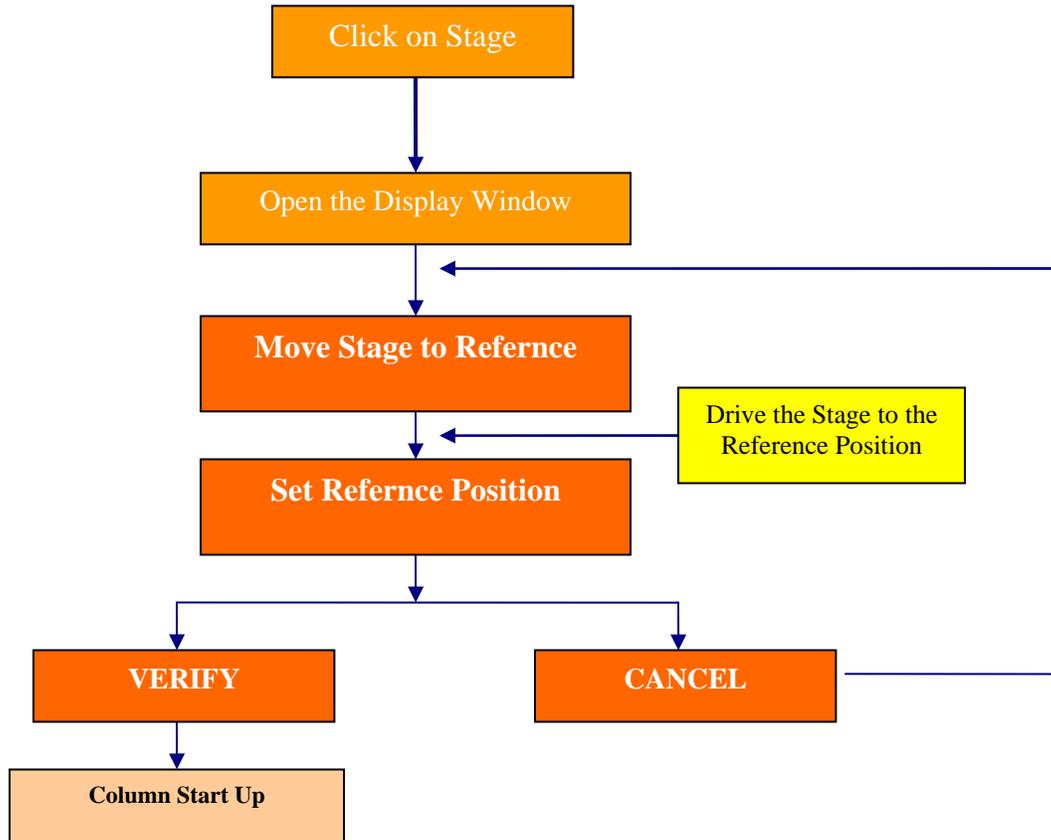


- *Shuttle Insertion – Sequence Description*
  - Put the shuttle on the transfer rod.
  - Click the left mouse button on the **Vacuum** button located in the SX-100 set up window.  
Click the left mouse button on the **Sample Exchange** button located in the **Vacuum Setup** window.
- Follow the Instructions displayed in the **Vacuum Setup** window:
  - **Turn the gun valve to position-1** to isolate the gun from the sample chamber.
  - Open the valve between the airlock chamber and the sample analysis chamber when the message **Airlock backup complete** is displayed in the **Vacuum Setup** window.
  - Rotate the ring of the transfer rod to the Insertion position (white arrow in the insertion position).
  - Push the transfer rod until it's maximum position and move it fully back.
  - Close the valve between the airlock chamber and the sample analysis chamber.
  - Click the left mouse button on the **Continue** button located in the **Vacuum Setup** window.
  - **Turn the gun valve to position-2** when the message is displayed in the Vacuum Setup panel.
  - Wait for the message **Vacuum ready** as shown on the figure before starting the electron column.

8.6.2.4 **Shuttle Positioning** – After its introduction and before any analysis, the shuttle must be moved to the Reference position and if necessary, the Reference position must be updated.

The Reference position must be as accurate as possible because all the stage positions (in the Stage position panel for example) are calculated with the Reference position as being the 0,0 coordinates.

- *Shuttle Positioning – Sequence Synoptic*



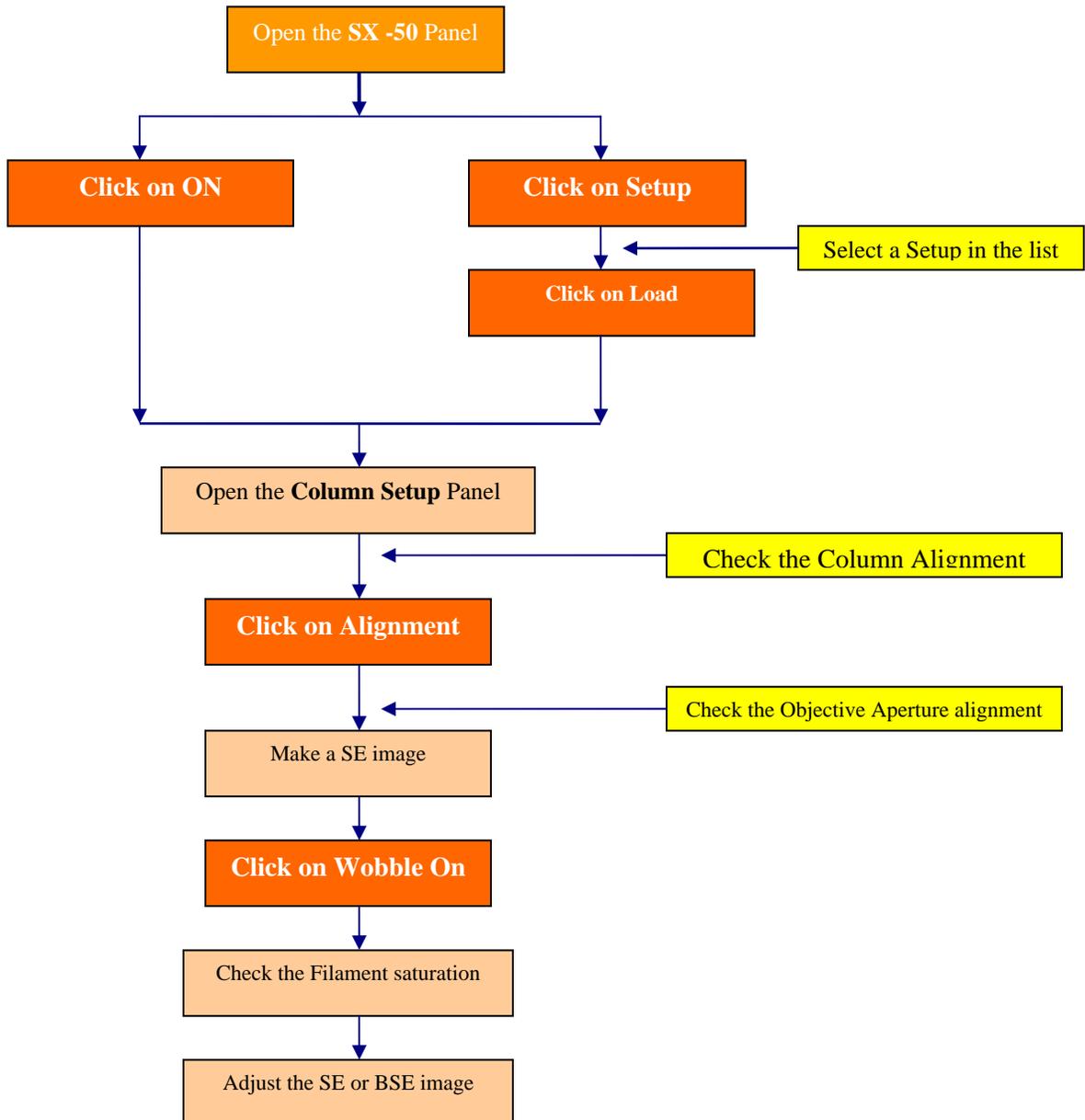
- *Shuttle Positioning – Sequence Description*

- Click the right mouse button on the **Stage** button available in the SX-50 main synoptic.
- Click on the **Display** icon to open the Stage Display window.
- Click the left mouse button on the **Stage to Reference** button. The stage will be driven to the position with  $X = Y = Z = 0$ .
- Focus the optical image and drive the sample stage to the point, which is always used as the reference point.
- Click the right mouse button on the **Stage to Reference** button to open the pull right menu shown on figure.
- Select set Reference position item and click the left mouse button on verify to reset the Stage Reference position. All the current stage coordinates are forced to 0 (check in the Roller Wheel Control window) when the verify button has been depressed.

8.6.2.5 **Column Start Up Procedure** – Before starting any analysis (Quantitative, Qualitative or Image acquisition), the column has to be switched on by using preset conditions or conditions used before the shuttle insertion.

**NOTE:** At least once a week, the column alignment and the filament saturation must be checked.

- **Column Start-Up – Sequence Synoptic**

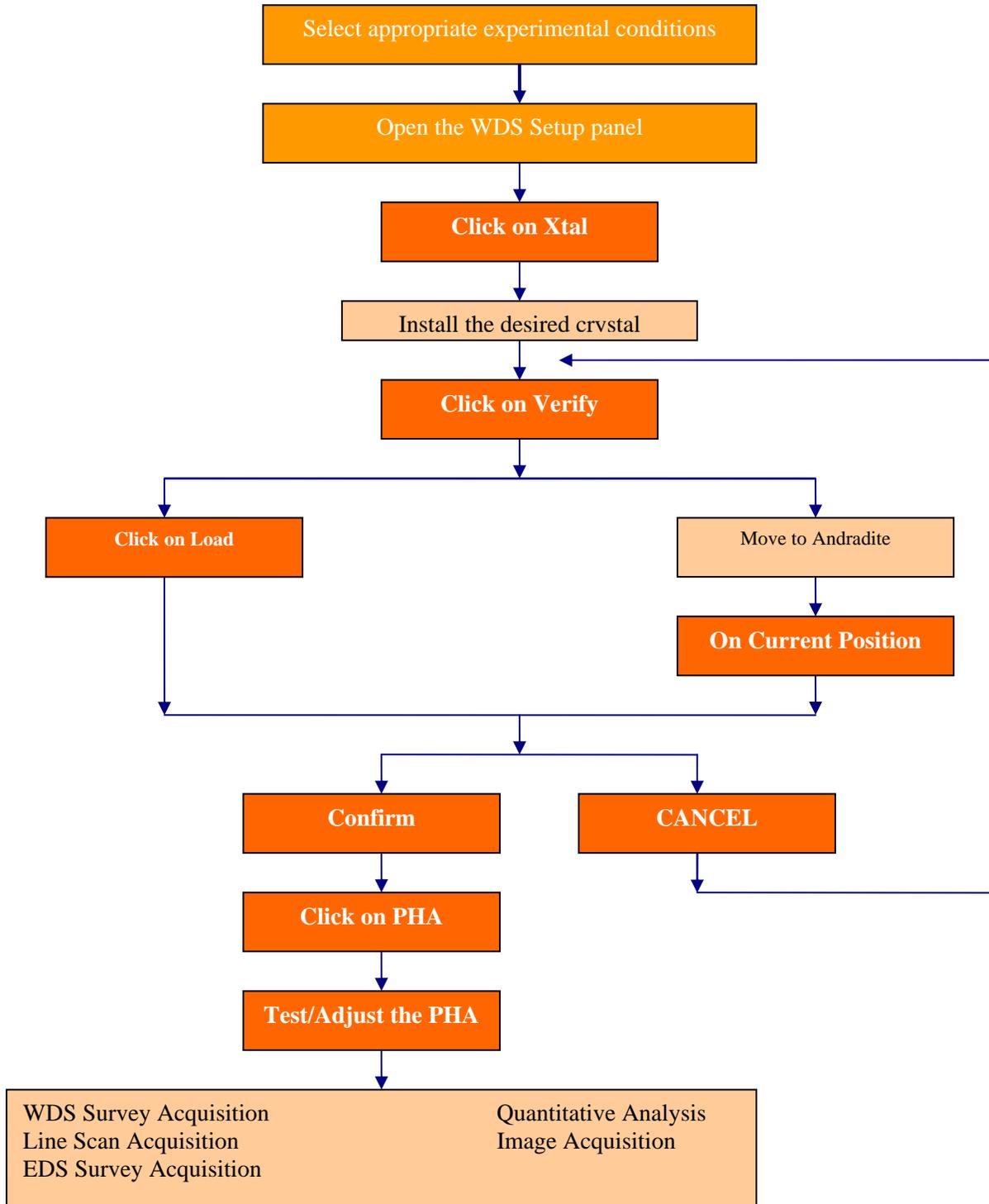


- **Column Start-Up – Sequence Description**
  1. Starting the Electron Column – There are two ways for starting the electron column.
    - Loading a stored Setup file
    - Using the previous column conditions
  2. **LOADING A STORED SETUP FILE**
    - Click the left mouse button on the **Setup** button located in the **SX-50 Setup** window.
    - Select the desired Setup file in the displayed list.
    - Click the left mouse button on the **Load** button and wait for the “setup ready” message displayed in the Status Window. All the parameters stored in the setup file will be applied again on the column.
  3. **USING THE PREVIOUS CONDITIONS**
    - Click the left mouse button on the **ON** button located in the **SX-50 Setup** window. The last conditions used before the sample exchange will be applied again to the column.
  4. **COLUMN ALIGNMENT** – The column alignment must be done each time a filament is exchanged and periodically checked during the life time of a filament. It is recommended to check the column alignment for given working conditions at least once a week. It is also recommended to check it when the accelerating voltage is changed. The alignment procedure is not necessary at every sample exchange.
    - **Automatic Column Alignment**
    - **Manual Column Alignment**
  5. **OBJECTIVE APERTURE ALIGNMENT** – The objective alignment must be checked periodically. For each accelerating voltage, it might be necessary to optimize the aperture position.
    - **Alignment Using the SEM image**
    - **Alignment Using the Beam Impact**

6. FILAMENT SATURATION – The so called filament saturation point corresponds to a filament temperature which gives the optimum working conditions for the electron source:
  - The cross over has a minimum diameter
  - The beam brightness is maximum
  - The beam stability is optimum
7. BEAM INTENSITY MEASUREMENT
8. CROSS OVER VISUALIZATION
9. SE Image / BSE Image
10. SAVING A SETUP
  - Click the left mouse button on the **Setup** button located in the **SX-50 Setup** window.
  - Enter the desired setup name in the Name field and validate it with <CR>
  - Click the left mouse button on the **Store** button.

8.6.2.6 **WDS Spectrometers Start Up** – Click the left mouse button on the **WDS** button located in the **SX-50 Setup** window.

- *WDS Spectrometer Start-Up – Sequence Synoptic*



- *WDS Spectrometer Start-Up – Sequence Description*

- 8.7 *Data Acquisition and Reduction* - Data may consist of image information and/or elemental information. Image and elemental information may be processed following instructions in the SX-50, IMIX, and VISILOG Manuals. Image and elemental information may be photographed and/or printed on paper following instructions in the SX-50 reference Manual if an electronic copy or a hard copy is desired. The Cameca software package “QuantiView” comprises software used for acquisition and reduction of quantitative elemental data. “QualiView” is the Cameca software package used for acquisition of qualitative elemental information and “VisiView” is the image processing software package.
- 8.8 *Potential Sources of Error and Uncertainty* - Criteria for recognizing and evaluating potential sources of error and uncertainty will be indicated by the Procedure User’s inability to obtain a quality image or to generate a semiquantitative or quantitative analysis within tolerance limits. Acceptance criteria for quantitative analysis of Samples are based on acceptable analyses of appropriate standards. Procedure Users may use as a general guide a value of two sigma. That is, if standard analyses are within two sigma (based solely on counting statistics) of the list or published values, then the analysis is acceptable. Procedure Users may define different acceptance criteria (e.g. charge balance ratios).
- 8.9 *Equipment Malfunctions* - Malfunction of the SX-50 System is readily detectable by the Machine Custodian during operation of the instrument. If a trained Procedure User has doubts concerning his/her ability to detect equipment malfunction during operation of this equipment, he/she should consult with the Machine Custodian.
- 8.10 *Calibration of Magnification* - The Machine Custodian (or delegated individual) will check the accuracy of the computer-generated micrometer marker annually against NIST Standard Reference Material Catalog, SEM magnification Standard. Tolerance is + 10%. If out of tolerance, it will be the Machine Custodian’s responsibility to arrange for repair of the instrument so that it will be within tolerance.
- 8.11 *Calibration of Gain* - The Machine Custodian (or delegated individual) will check the gain on the EDS annually by following the energy calibration instructions in the IMIX Instruction Manual using Copper  $K\alpha$  and  $L\alpha$  X-Ray lines. Tolerance is +20 eV. If found to be out of tolerance, it will be the Machine Custodian’s responsibility to recalibrate the gain on the EDS system as described in the IMIX Manual.

If the gain cannot be calibrated it will be the Machine Custodians responsibility to initiate repair for the instrument so that it will be within tolerance.

- 8.12 *Energy Resolution Check* - The Machine Custodian (or delegated individual) will check the energy resolution of the EDS detector annually by measuring the full width at half maximum (FWHM) of the Manganese K-alpha X-Ray line. This is a check for degradation of the detector ONLY: no adjustment or calibration will be made.

## 9.0 RECORDS

The **Procedure User** is responsible for submitting the following records to the Machine Custodian/Lab. Instrumentation specialist to be storage at the UPR-NSF Earth X-ray Analysis Center (EXACT), F-304-A and F-304-D.

- 9.1 Log-Book or Notebook records of the sample handling and results of analysis relevant to Production of electron microprobe data.
- 9.2 Data submittals will be storage in the electronic database.

## 10.0 REFERENCES

The following documents have been cited within this procedure by:

- <http://erproject.lanl.gov/docs/Quality/SOP/SOP-09.02R1T.pdf>
- <http://geology.uprm.edu/facilities/exact.html>
- <http://geology.uprm.edu/facilities/labrules/exact.doc>
- **Theory and Practice of Electron Microprobe Analysis** – Power Point Presentation: University of Puerto Rico – Mayagüez Campus, Department of Geology, Geochemistry Facilities, X-ray / Microanalysis Laboratory - F304
- **Cameca SX-50 Operation Manuals:** User's Guide, Reference Guide, Quantitative Reference Guide, and X-Ray Microanalysis, Geo-Multilabel, Wavelength Table Reference Manuals, 1991, Cameca, Inc. Paris, France.
- **SX-50 User's Guide;** Reference: 45-404-901, Reference Manuals, 1991, Cameca, Inc. Paris, France
- **VISILOG User's Guide**, 1988, Noesis, Jouy en Joses, France
- Potts, P.J., 1987. *A Handbook of Silicate Rock Analysis*. Blackie: Glasgow

- Potts, P.J., Bowles, J.F.W., Reed, S.J.B., and Cave, M.R., 1995. ***Microprobe Techniques in Earth Sciences***. Chapman and Hall: London
- Reed, S.J.B., 1993. ***Electron Microprobe Analysis***. Cambridge University Press: Cambridge
- Reed, S.J.B., 1996. ***Electron Microprobe and Scanning Electron Microscopy in Geology***. Cambridge University Press: Cambridge