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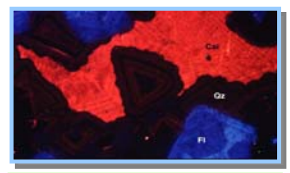
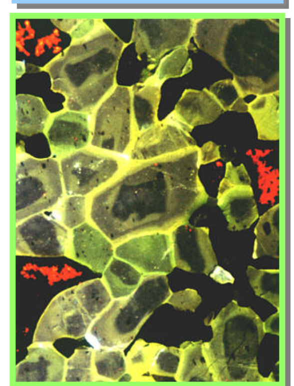
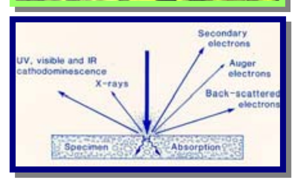
UPR-NSF Earth X-ray Analysis Center (EXACt!)

Standard Operating Procedure for:

Operating the Relion Cathodoluminescence Microscope







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

Revision Log

Revision No.	Effective Date	Prepared By	Description of Changes	Affected Pages
R-0	February 27, 2007	Miguel Santiago	All New Procedure	All
R-1	March 26, 2007	Miguel Santiago	X-rays Warning for Pregnant and Cancer patient 8.0 - Procedure	One Page -11

Operating the Relion Cathodoluminescence Microscope

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Operating the Relion Cathodoluminescence Microscope

1.0 PURPOSE

This procedure provides instructions for the operation of the Relion Industries, Cathodoluminescence Microscope Model Nikkon OPTIPHOT-2. The UPRM – Department of Geology has a cold cathode, CITL 8200 Mk 3A mounted on a Nikon Optiphot-2 petrological microscope available for optical CL studies. The Nikon Optiphot-2 is set up as a digital fluorescence acquisition microscope. This system should be used to observe and record images prior to use of other, higher resolution, such as Cameca – SX50. This is a general instruction guide for use with the Cathodoluminescence (CL) Apparatus, which uses a cold cathode electron gun. It covers basic operation, safety rules, which must not be avoid. This SOP states the responsibilities and describes the methods, procedures and documentation used to obtain Relion Cathodoluminescence Microscope's image at the UPR-NSF Earth X-ray Analysis Center (EXACt), Geochemistry Facilities, Department of Geology, UPR-Mayagüez Campus.

2.0 SCOPE

This SOP is a mandatory document and shall be implemented by all Researcher, Faculty member, Technician and/or students participants when using the Relion Industries, Cathodoluminescence (CL) Microscope for the collection of CL image. This SOP covers elementary physical operation of the instrument and radiation safety compliance requirements. Image interpretation is beyond the scope of this manual.

3.0 TRAINING

3.1 All users of this SOP will be 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 *GLP's* - Good Laboratories Practices

4.2 *SOP* - Standard Operational Procedure

4.3 *Machine Custodian* — The Machine Custodian is responsible for Relion Industries, Cathodoluminescence Microscope maintenance and User instruction. This includes calibration, basic repairs, and image taken and electronically-stored system backups, and all instruction and training of Procedure Users.

4.4 *Cathodoluminescence* - (CL) is the emission of light from material when it is irradiated with electrons. The technique is used for imaging spatial variations in the trace element composition of natural (e.g. calcite, diamond, feldspar and apatite) and synthetic materials.

4.5 *X-ray* - Electromagnetic radiation of very short wavelength (0.01 to 100 nm) produced when an electron hits a piece of metal in an evacuated tube. An electron beam of 5 to 15 kV or higher energy will produce X-rays. The CL chamber design should shield against these and viewing window should be made of a special X-ray leaded glass. **NOTE**: Never replace this with anything except the same type of glass.

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 THEORETICAL BACKGROUND

6.1 Introduction - Luminescence is the emission of light from a solid which is 'excited' by some form of energy. The term broadly includes the commonly-used categories of fluorescence and phosphorescence. Fluorescence is said to occur where emission ceased almost immediately after withdrawal of the exciting source and where there is no thermal cause, whereas in phosphorescence the emission decays for some time after removal of excitation.

The distinction between these so-called types of luminescence is somewhat arbitrary and confusing; for example, many minerals have very long post-excitation decay times. Confusion is avoided by using the term luminescence, and specifying the activating energy as a descriptive prefix. Thus roentgenoluminescence is produced by X-rays, photoluminescence by light (e.g. ultra-violet) and cathodolminescence(CL) results from excitation by electrons and thermoluminescence results from heating.

Cathodoluminescence petrography is now a routine technique that can provide essential information on provenance, growth fabrics, diagenetic textures and mineral zonation, in addition to enabling more precise quantification of constituents and fabrics. Without the support of CL spectroscopy, however, CL petrography can only remain a fabric analysis technique.

Although subtle variations in CL color recorded on film give important information, describing luminescence intensity and color from a photographic record is a dubious and subjective affair. The actual CL color is determined by the number and type of emission and quenching centers present. Superposition of several luminescence bands of different intensities can provide quantitative data on the wavelength and intensity of luminescence and the nature of the luminescing centers. CL spectroscopy should become a standard technique used by the luminescence petrographer becuase it is the only means of recording CL colors and emission intensity objectively and quantitatively, in addition to providing unique information on the nature of luminescence centres.

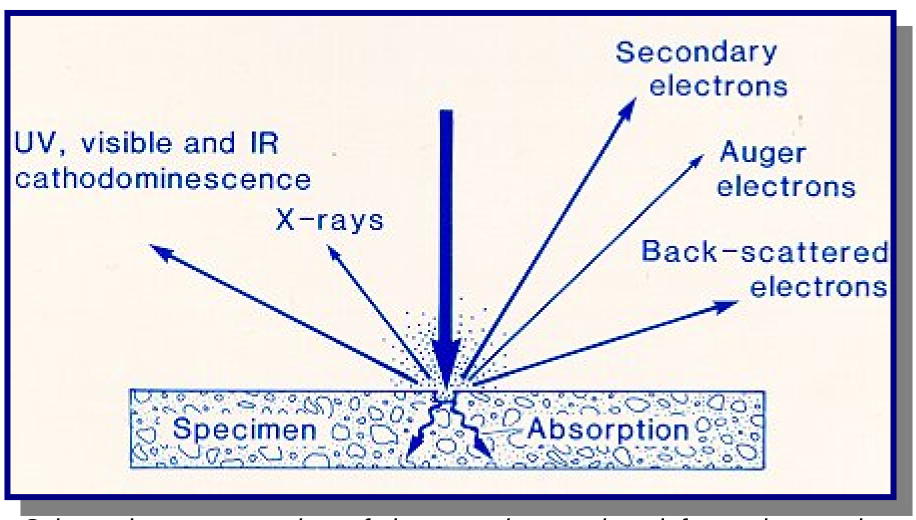


Fig. Schematic representation of the energies produced from electron beam interaction with solid matter.

6.2 **Cathodoluminescence** is an optical and electrical phenomenon whereby a beam of electrons is generated by an electron gun (e.g. cathode ray tube) and then impacts on a luminescent material such as a phosphor, causing the material to emit visible light. The most common example is the screen of a television. In geology, mineralogy and materials science a scanning electron microscope with specialized optical detectors, or an optical cathodoluminescence microscope, is used to examine internal structures of semiconductors, rocks, ceramic, glass etc. in order to get information on the composition, growth and quality of the material.

Cathodoluminescence occurs because the impingement of a high energy electron beam onto a semiconductor will result in the promotion of electrons from the valence band into the conduction band, leaving behind a hole. When an electron and a hole recombine, it is possible for a photon to be emitted.

The energy (color) of the photon, and the probability that a photon and not a phonon will be emitted, depends on the material, its purity, and its defect state. In this case, the "semiconductor" examined can, in fact, be almost any non-metallic material. In terms of band structure, classical semiconductors, insulators, ceramics, gemstones, minerals, and glasses can be treated the same way.

In materials science and semiconductor engineering, cathodoluminescence will mostly be performed in either a scanning electron microscope or a scanning transmission electron microscope. In these cases, the highly focused beam of electrons impinges on a sample and induces it to emit light from a localized area. This light will be collected by an optical system, such as an elliptical mirror. From there, a fiber optic will transfer the light out of the microscope where it will be separated by a monochromator and then detected with a photomultiplier tube.

By scanning the microscope's beam in an X-Y pattern and measuring the light emitted with the beam at each point, a map of the optical activity of the specimen can be obtained. The primary advantages to the electron microscope based technique is the ability to resolve features down to 10-20 nanometers, the ability to measure an entire spectrum at each point (hyperspectral imaging) if the photomultiplier tube is replaced with a CCD camera, and the ability to perform nanosecond- to picosecond-level time-resolved measurements if the electron beam can be "chopped" into nano- or pico-second pulses.

However, as the abilities are improved, the cost of the electron-microscope based techniques becomes very high.

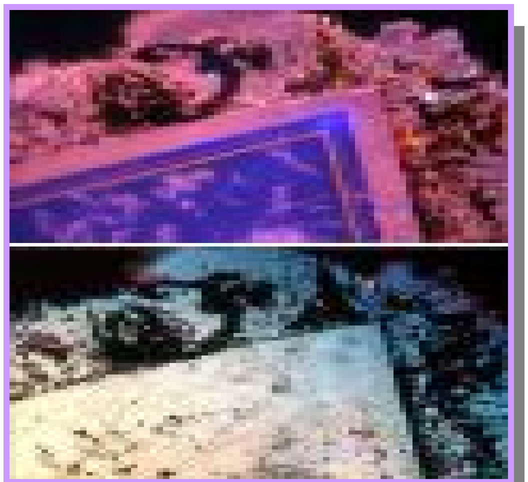
These advanced techniques are useful for examining low-dimensional semiconductor structures, such a quantum wells or quantum dots. Although direct bandgap semiconductors such as GaAs or GaN are most easily examined by these techniques, indirect semiconductors such as silicon also emit weak levels of light, and can be examined as well. In particular, the luminescence of dislocated silicon is different from intrinsic silicon, and can be used to map defects in integrated circuits.

Except of the much higher magnification and good versatility, an electron microscope with a cathodoluminescence detector will be more complicated and more expensive compared to an easy to use optical cathodoluminescence microscope, which benefits from its ability to show actual visible color features immediately through the eyepiece. In short, cathodoluminescence is a technique that can be implemented in an optical or electron microscope with the proper accessories, and allows the optical properties of non-metallic materials to be examined.

6.3 **Cathodoluminescence (CL) Microscope** combines methods from electron and regular (light optical) microscopes. It is designed to study the luminescence characteristics of polished thin sections of solids irritated by an electron beam. To prevent charging of the sample, the surface must be coated with a conductive layer of gold or carbon. This is usually done by a sputter deposition device or a carbon coater. Using a cathodoluminescence microscope, structures within crystals or fabrics can be made visible which cannot be seen in normal light conditions.

Thus, for example, valuable information on the growth of minerals can be obtained. CL-microscopy is used in geology, mineralogy and materials science (rocks, minerals, volcanic ash, glass, ceramic, concrete, fly ash et.). CL color and intensity are dependent on the characteristics of the sample and on the working conditions of the electron gun. Here, acceleration voltage and beam current of the electron beam are of major importance. Today, two types of CL microscopes are in use.

One is working with a "cold cathode" generating an electron beam by a corona discharge tube, the other one produces a beam using a "hot cathode". The advantage of a hot cathode is the precisely controllable high beam intensity allowing to stimulate the emission of light even on weakly luminescence materials (e.g. quartz - see picture).



Thin section of quartz from a hydrothermal vein - upper in CL and lower in transmitted light

7.0 EQUIPMENT

Descriptions of equipment constituting the Relion Industries, (CL) Cathodoluminescence Microscope system are provided below.

7.1 *Chamber* – the CL chamber is a vacuum chamber on which are mounted the electron gun and controls for moving the sample in the X and Y directions,. The X-direction, by convention, is left-to-right, as viewed by the operator seated at the microscope, and the Y-directions is toward or away from the operator.

7.2 *Gun / Deflection Magnet / Focus Coil* – The cold cathode electron gun consists of a cathode, which is held at the negative high voltage, an insulating discharge tub, and the anode, which is held at or near the electrical ground. The negative high voltage is typically in the range of 5 to 10 to 20 kV or higher and the available beam currents are typically 0.5 mA to 1.0 mA or greater. A discharge is created within the electron gun and this discharge is made up of positive ions and electrons. The electrons are attracted towards the anode and pass through a hole in the anode into the chamber.

WARNINIG: High Voltage supplies used can cause severe ***electric shocks*** to the operator incomes into contact with the cathode, cable or power supply internal components during operation or test. They should never be operated with gun covers off or with electronics cabinets removed.

7.3 *Cold Cathode Electron Gun Power Supply* – The CL Apparatus power supply provides a means for varying the beam energy (voltage) by a front panel control and may have a meter readout for the voltage and the beam current. The beam current is set by the conditions of a cold cathode discharge in the electron gun and hence depends on the pressure in the CL chamber. The basic power supply is a power pack with an AC input and DC output. Varying the input voltage with adjustable transformer on the front panel between 0 and 115 volts -AC causes the output to vary between 0 to 15 kV – DC. The beam power is the product of the beam voltage (V) and the beam current (I);

$$P = V \cdot I$$

This will be the power developed in the gun. However, not all of the beam current will actually strike the sample so the power dissipated on the sample will be less than this.

NOTE: When a overloaded circuit fires, the beam will be turn off and a front panel light should come on. This light is usually labeled “reset”. The procedure to restart the beam is to turn off the HV switch (high voltage), adjust the voltage to a slightly lower value, and then turn the HV on again. Repeat this procedure and set the HV at an even lower value every time if the current still too high.

7.4 *Vacuum System* – The usual cold cathode electron gun operates with the pressure in the system in the range of 20 to 100 millitorr.

Torr is a unit of pressure:

1 atmosphere = 760 torr.

1 torr = 1,000 millitorr.

1 torr = 1 mm Hg.

1 millitorr = 1 micron

This pressure range is established with a two-stage mechanical pump. Such a pump, in good operating conditions, will pump to less than 10 millitorr and could even produce too good a vacuum.

7.4.1 *Pump* – The vacuum pump is a mechanical pump with a speed in the range between 1 and 3 CFM. Such a pump will pump the chamber to a pressure less than 40 millitorr. This is actually lower than the pressure normally required for cold cathode discharge to be established so no additional pumping is required

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 CL microscope. 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 SOP's 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 CL, Relion Industries, Cathodoluminescence Microscope (Purchase Date: month/year, Relion Industries Serial Number: 145177 and UPRM-Property Number: 053996) allows, in cathodoluminescence microscopy, an electron beam is focused onto the sample. In certain samples, the electron-sample interaction results in visible luminescence. Luminescence in materials results from either the trace element content of a material or the nano-scale structure.

8.2 *Sample Preparation* — The samples to be examined with the CL Apparatus, must be in a form or size that can be inserted into or attached to the stage mount and very well compacted. Acquiring image process and microanalysis routines assume that all samples for analysis will be relatively flat and that analysis sites will be normal to the beam incidence.

NOTE: Samples should be well-ground in a mortar and pestle. This creates a uniform particle size and ensures that all possible crystallite orientations are present in the sample. A special problem that can arise in sample preparation is called **preferred orientation** which usually occurs with rod or plate-like crystals. For example, plate-like crystals tend to lie flat on the sample holder; very few will have a perpendicular orientation..

8.3 ***Performing the Standard Operation Procedure for Relion Cathodoluminescence Microscope – Image Scanning Sample.***

The eXact! CL Nikon Optiphot-2 is set up as a digital fluorescence acquisition microscope. This system should be used to observe and record images prior to use of other, higher resolution microscope.

8.3.1 *Environmental Considerations* – Maintain the room temperature between 17ºC (62ºF) and 26ºC (78ºF), at a gradient less than 0.5ºC/hr and a fluctuation of no greater than 2ºC. Relative humidity should not exceed 80% and should be no less than 20%. Ensure that the heat produced from the generator, spectrometer, computer and vacuum pump is adequately dissipated by ventilation or air conditioning and located so that the air flow is not directed at the instrument. A clean, dust-free environment is also necessary. Avoid the direct sunlight to CL microscope.

8.3.2 Power Up Procedure (STEP-1)

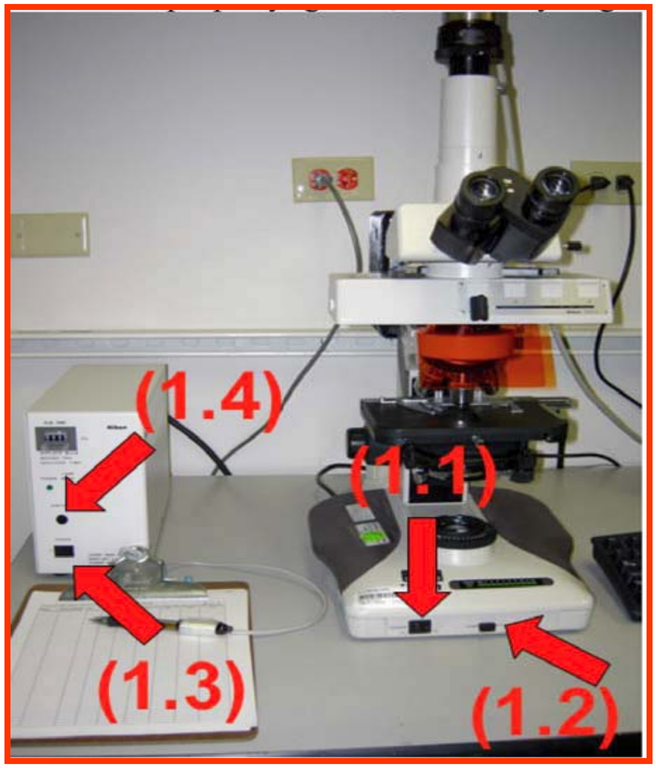
8.3.2.1 For your protection as well as the equipment, please dawn gloves provided to youby the eXact! at no charge.

8.3.2.2 Carefully remove the protective dust cover from the microscope ensuring that the back of the cover does not catch or move the mercury supply adjustments.

8.3.2.3 The Nikon Optiphot-2 has 2 power supplies. The order in which these supplies are turned on is important. Begin by turning on the main power supply (1.1). The slider (1.2) located next to the power switch is used for white light source intensity adjustment.

8.3.2.4 Should you desire fluorescence excitation, you will need to turn on the mercury light source. It is imperative that the mercury source is used for duration of at least 15minutes. Likewise, it is also imperative that the mercury source not be switched on after prior use for at least 30 minutes.

8.3.2.5 In order to illuminate the mercury light source, turn on the power supply (1.3). Next, in order to ignite the bulb, press and hold the “ignition” button (1.4). Once the bulb is properly ignited, the “ready” light will illuminate.

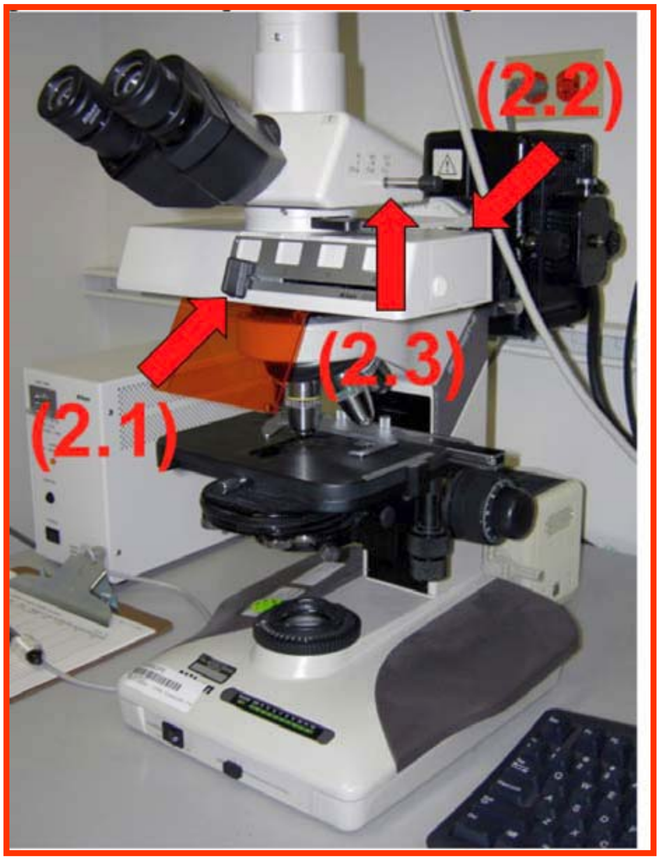


8.3.3 Image Acquisition (STEP-2)

8.3.3.1 Select the appropriate excitation wavelength using the filter cube selection slider (2.1). Further compose your image using the focus and translation stage. When you are not actively observing or imaging your sample, it is important to engage the shutter (2.2) and block the excitation light to your sample. When the shutter is depressed, the light path is undisturbed and excitation can occur. Subsequently, when the shutter is pulled out, the light path is disturbed.

8.3.3.2 In order to set the scope for digital capture, it is necessary to manipulate the “eyepiece/camera” selector (2.3). When depressed, the eyepieces are in the light path and when pulled out, the digital camera is in the light path.

8.3.3.3 Having configured the microscope properly as discussed herein, it is now time to configure the computer system for digital image capture. Please refer to the separate instruction document entitled “Computer Login/Image Acquisition Instructions.”



8.3.4 Shutdown Procedure (STEP-3)

8.3.4.1 After properly restarting the computer system as discussed in the “Computer Login/Image Acquisition Instructions,” attention must be paid to the microscope.

8.3.4.2 Begin by returning all settings on the scope to their respective “off” positions. This includes moving the filter slider (2.1) left to the first position. Next make sure the shutter (2.2) is pulled out from the scope and blocking the light path. Lastly, move the “eyepiece/camera” selector (2.3) inward towards the scope to “eyepiece” position.

8.3.4.3 Having returned the microscope settings to their respective “off” positions, you may now shut down the power supplies for the microscope and mercury lamp. Again, the order in which you shut down the supplies is important.

8.3.4.4 Begin moving the light intensity slider (1.2) to the left or lowest intensity.

8.3.4.5 Next, turn off the microscope power supply (1.1).

8.3.4.6 Lastly, switch off the mercury lamp supply (1.3).

8.3.4.7 After both power supplies have been shutdown and the computer has been restarted, please fill out the log sheet located to the left of the microscope. MAKE SURE TO FILL IN THE MECURY LAMP COUNTER.

8.3.4.8 Allow 15 minutes for the microscope and lamps to cool prior to replacing the dust cover. Again, take care to ensure the dust cover is draped over the scope without impeding the rear lamp housings (see below).

8.4 *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. Procedure Users may define different acceptance criteria.

8.5 *Equipment Malfunctions* - Malfunction of the CL Apparatus. 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.

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

8.7 *Environmental Conditions* - Normal interior building temperature and humidity are acceptable for the operation of the CL Apparatus. The pump and electronics chassis is supplied by the building maintained in the range of 55 to 65 degrees Fahrenheit. Ambient air temperature for the CL Apparatus. should range between 60 and 80 degrees Fahrenheit.

NOTE: *If environmental conditions move out of range during operating the CL Apparatus, Procedure Users should take extra precaution to ensure system stability by checking standards often.*

8.8 *Calibration of Magnification* - The Machine Custodian (or delegated individual) will check the accuracy of the image at the CL Aparratus.

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-D.

9.1 Log-Book or Notebook records of the sample handling and results of analysis relevant to Production of XRD data.

9.2 Data submittals will be storage in the electronic database.

9.3 It is obligation of the user to backup the data obtained.

9.4 Data will be held by the laboratory for thirty (30) days, after which it may be eliminated from departmental files.

10.0 REFERENCES

The following documents have been cited within this procedure by:

- Basic Operation and Maintenance Instructions for (CL) Cathodoluminescence Instruments, Using Cold Cathode Electron Guns, Relion Industries, PUB037F/0697
- http://www.wikipedia.org/
- http://www.answers.com/topic/cathodoluminescence
- http://geology.uprm.edu/facilities/exact.html
- http://geology.uprm.edu/facilities/labrules/exact.doc