

Standard Operating Procedure

Title:	Standard Operating Procedure For HC4-LM Cathodoluminescence Microscope
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SOP#	SOP-CLM-YANG-001
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1.0 Purpose:

The aim of this guideline is to inform all personnel who use the HC4-LM Cathodoluminescence Microscope (CLM) about the proper procedures, safety concerns and to maximize the degree of efficiency.

2.0 Scope:

The purpose of HC4-LM CLM is to reveal the true color luminescence features of samples under an optical microscope. This document applies to any personnel who will be using the HC4-LM CLM.	
	Department, Lab or Center: Cathodoluminescence Microscope Laboratory
	Research Group:
	Lab Bldg., Room(s): Science Building, Suite 437
	Operation/Experiment:

3.0 Responsibilities:

The user shall perform the following procedure within the CLM Lab (S437) regarding the use of the HC4-LM CLM. Only trained personnel should operate this equipment.

4.0 Health, Safety and Environmental Considerations:

4.1 Materials and Hazards

Principal Materials Used	Flammable	Corrosive	Sensitizer	Mutagen	Teratogen	Biological Toxin	Acutely Toxic	Pyrophoric	Water-Reactive	Shock Sensitive	Carcinogen	Unstable	Other Comments
MSDS attached	Yes			If not, please explain: <u>Not Applicable</u>									
	No												

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Describe equipment/instrumentation used to monitor/control hazards:

	Permits:
	Mgmt. Approval:
	Training:
	Medical Surveillance:
	Other: <ol style="list-style-type: none"> 1. IN AN EMERGENCY, LEAVE THE SCENE AS SOON AS POSSIBLE. 2. The EHT voltages present in this instrument can be lethal. 3. Do not use electrical equipment in: <ul style="list-style-type: none"> • Rain or excessive moisture environment • The presence of flammable or explosive gases <p>The equipment is not designed to be water or splash proof, or to be used in area where there are flammable or explosive gases or fumes.</p>

4.3 Special Emergency Procedures

Fire/Evacuation:	
Chemical Spill:	
Medical Emergency:	Call security office at 420-5000 immediately, or any of the nearby first aiders. The names, room# and phone# of the nearby first aiders are posted in the lab.
Personal Exposure:	

5.0 Equipment and Supplies:

Equipment(s):	
X	HC4-LM Cathodoluminescence Microscope

Special PPE Required:

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	Goggles
	Face Shield
	Chemical Resistant Apron
	Protective Clothing
X	Gloves
	Butyl
	Nitrile
	PVC
	Latex
	Neoprene
	Silver Shield brand
	Kevlar
	X Other: Powder free gloves, lint free sleeves
	Respirator (If yes, contact EHS Office for additional assistance)
<p>Note: If special PPE and/or protective clothing is not required, standard PPE and protective clothing required in Part II of the Department Chemical Hygiene Plan must be utilized.</p>	

6.0 Terms and Definitions:

Not Applicable

7.0 Procedure:

Basic Instructions

- Power on all the instrument in the following order:
 - Turn on the power switch on the Power Transformer.
 - Turn on the camera power located on the black box labelled **Kappa**. **Note: The digital camera must be turned on before turning on the PC.**



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- Turn on the main power switch of the CLM power supply. Switch the toggle switch to “Vac. only”.



- Turn on the power supply of the lamp (the black switch on the left of the power supply). The on/off toggle switch (on the right front) allows you to turn on/off the transmitted light to switch between CL and transmission light photography.



- Turn on and log onto the computer by using the following username:

Username: **geology**

Password: **Cathod11**

2. Create a new folder on the desktop. Please use your name as the folder name – all your data will be saved under your name. You can organize your own data by creating subfolders (e.g. by thin sections).

WARNING:

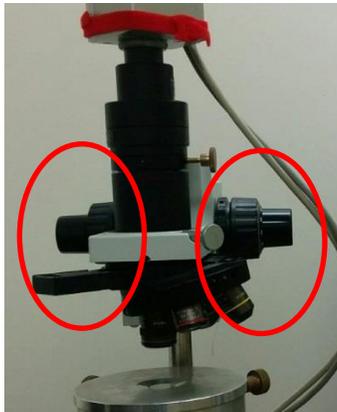
All users are responsible to backup (and remove) their own data on a timely manner. Due to limited hard drive space, the data will be empty on a regular basis. We are not responsible for the lost of data should this happen.

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3. Turn off the vacuum pump by switching the black switch on the left side of the control box from 1 to 0. It will take approximately 2-3 minutes for the system to reach atmosphere level.



4. Raise the microscope head completely (a) and carefully remove the cover of the sample chamber (b). **Do not let the lead glass window touch the objectives and avoid dirtying the rubber seal and seat of the sample chamber.**



(a)



(b)

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5. With powder free gloves on, carefully load your thin section without straining the stage clips. Make sure the thin section is placed upside down (sample side facing down). **Never reach into the chamber without gloves on!** Move the stage and locate the reference point on the thin section. Sample movement is done with the brass knobs at the back of the chamber. (Rotation of the right hand knob in a clockwise direction moves the sample to the right; rotation of the left hand knob in a counter clockwise direction moves the sample toward the front of the chamber. At low magnification, e.g. 5X, two complete revolutions of the knob will move the sample $\sim 2/3$ of the monitor screen. This leaves enough overlap for stitching multiple images together.)



WARNING: Any bare hand operation during the sample loading/unloading will result in the loss of privilege of using the system.

6. Replace the chamber cover and switch the Vacuum button to start pumping down the chamber. You can rotate the cover slightly to ensure a good seal. It would take approximate 2-5 minutes to reach the workable vacuum. Wait until the vacuum in the chamber is ready (the gauge has a reading between 5×10^{-5} and 2×10^{-4} mbar and the left LED1 lights up).

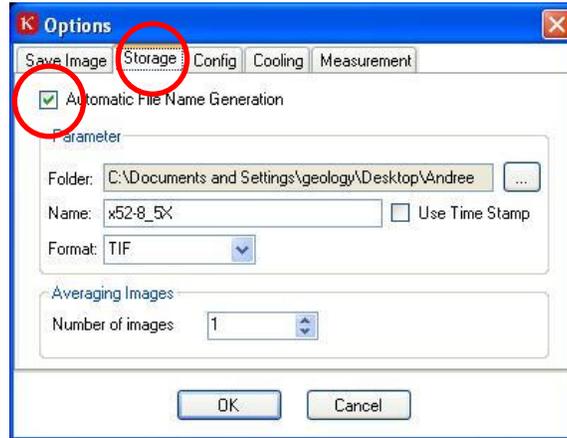


7. Open the Kappa program by double clicking the Kappa shortcut  on the desktop. Go to menu bar and select **Options** → **Storage**.
- Check “**automatic file name generation**”.
 - Folder: navigate to the proper project folder on the desktop.
 - Name: enter the sample ID.

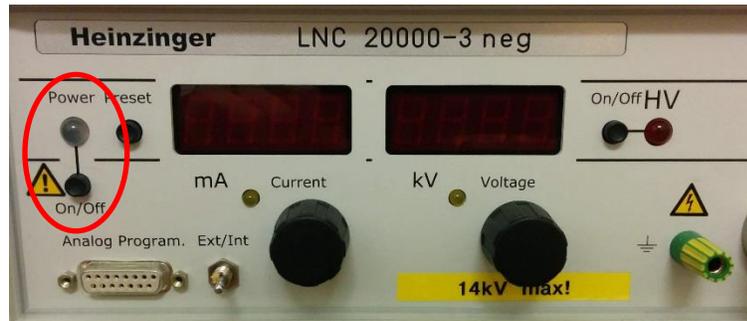
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- Format: **TIFF**.

Click **OK** to close the options window.



- When vacuum is ready, turn the switch from “Vac. only” to “beam”. The power supply for the cathode and the high voltage are now activated. Switch on the high voltage by pressing the “ON/OFF” button on the “Heinzinger” power supply, the “Power” LED will also change from orange to green.



- Press the “HV On/Off” button to turn on accelerating voltage. The red LED should light up.



- Turn on the filament by switching the toggle switch. Check if the Wehnelt is set around 280. Adjust the filament current slowly until the desired intensity of luminescence is achieved. You are now ready to view and collect CL images.

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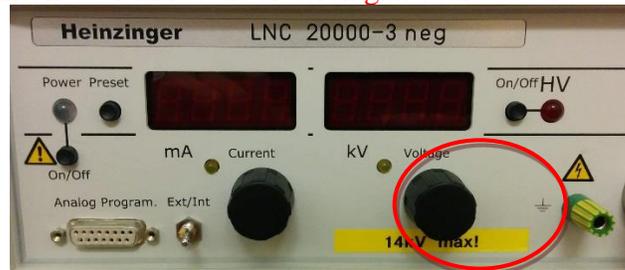
Note: Proper beam current should be between 0.1-0.4 mA. Never exceed 1.5 mA. The filament current should typically fall within 1.6-2.7A or <1.6A. The filament may burnt down if the current is too high.



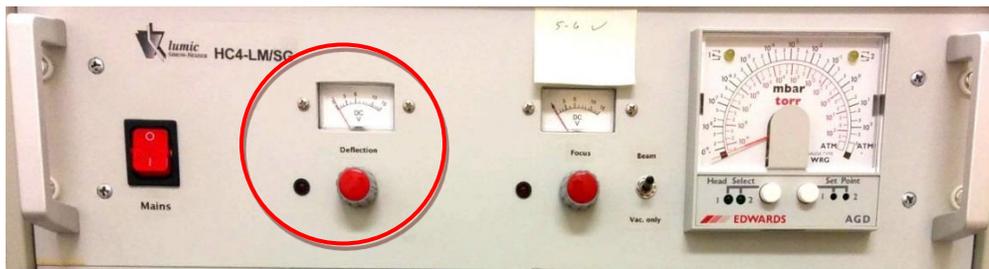
11. You can now fine tune the following parameters to obtain the best result if necessary.

- Adjust the voltage control knob to set your desired accelerating voltage.

Warning: The kV value should not exceed 14 kV as this may damage the unit and a bad vacuum flash-over inside the electron gun could occur.



- Tune the “Deflection” knob to move the luminescence spot to the right or left. The default value is set at about 8 V.

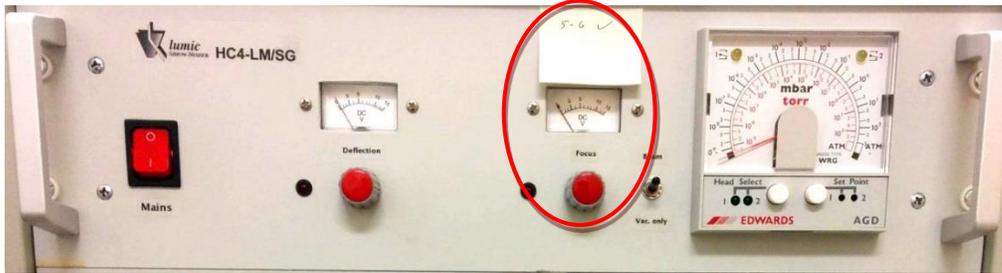


- Tune the “Focus” knob to expand or contract the electron beam. The default value is set about 5-6 V.

Warning: Do not try to focus the beam to small. Too strong contraction of the electron beam may cause partial overheating of the sample and could result in a

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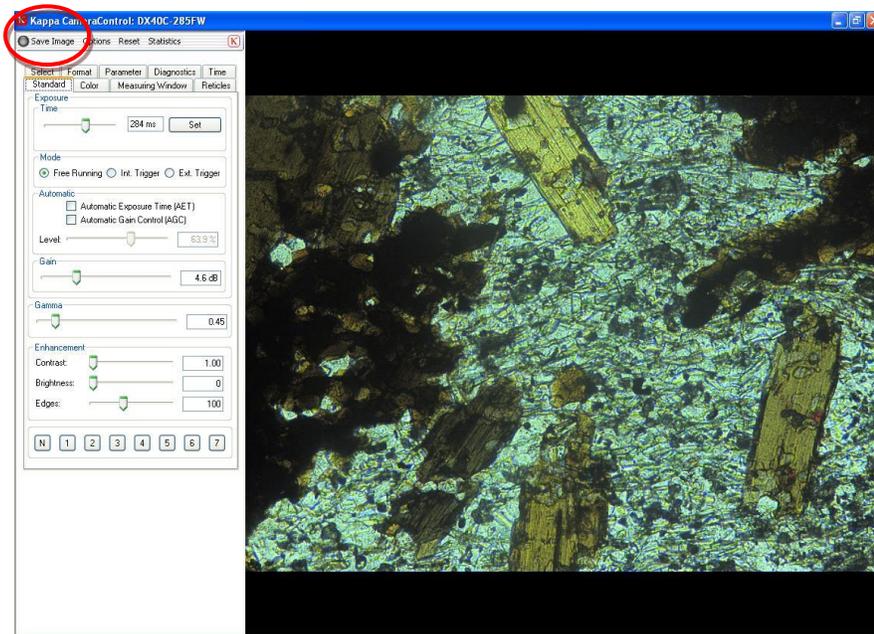
damage of the thin section. At worst case, the vacuum window of the sample chamber may break.



- Adjust the Wehnelt dial until the beam spot is homogeneous at maximum intensity. The value should be between 200 and 360. The default value is set about 280.



12. To save an image, set the proper exposure time and click on the **Save Image** button on the toolbar. **Note: For CL imaging, you have to turn off the room light to get rid off light contamination.**



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13. When done with collecting data, turn off the system in the following order: main switch and power to the lamp.
 - Turn off the KV
 - Switch off the filament
 - Switch the mode from “beam” to “Vac. only”
 - Switch off the Kappa power
 - Switch off the lamp
 - Close the Kappa software.
 - Turn of PC.
14. Vent the chamber and remove specimen from chamber, replace the chamber cover and pump the chamber.

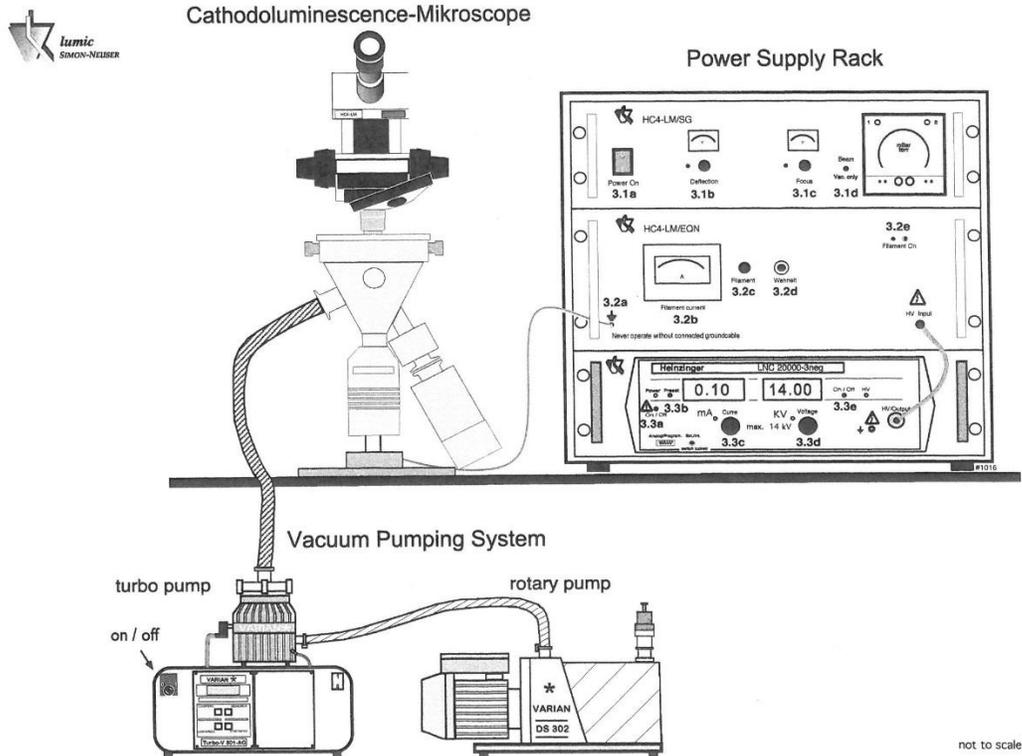
WARNING: Fail to pump the specimen chamber after you retrieve your samples will result in the loss of your privilege of using the system.

15. Fill the log book.

WARNING: Fail to fill the log book will result in the loss of your privilege of using the system.

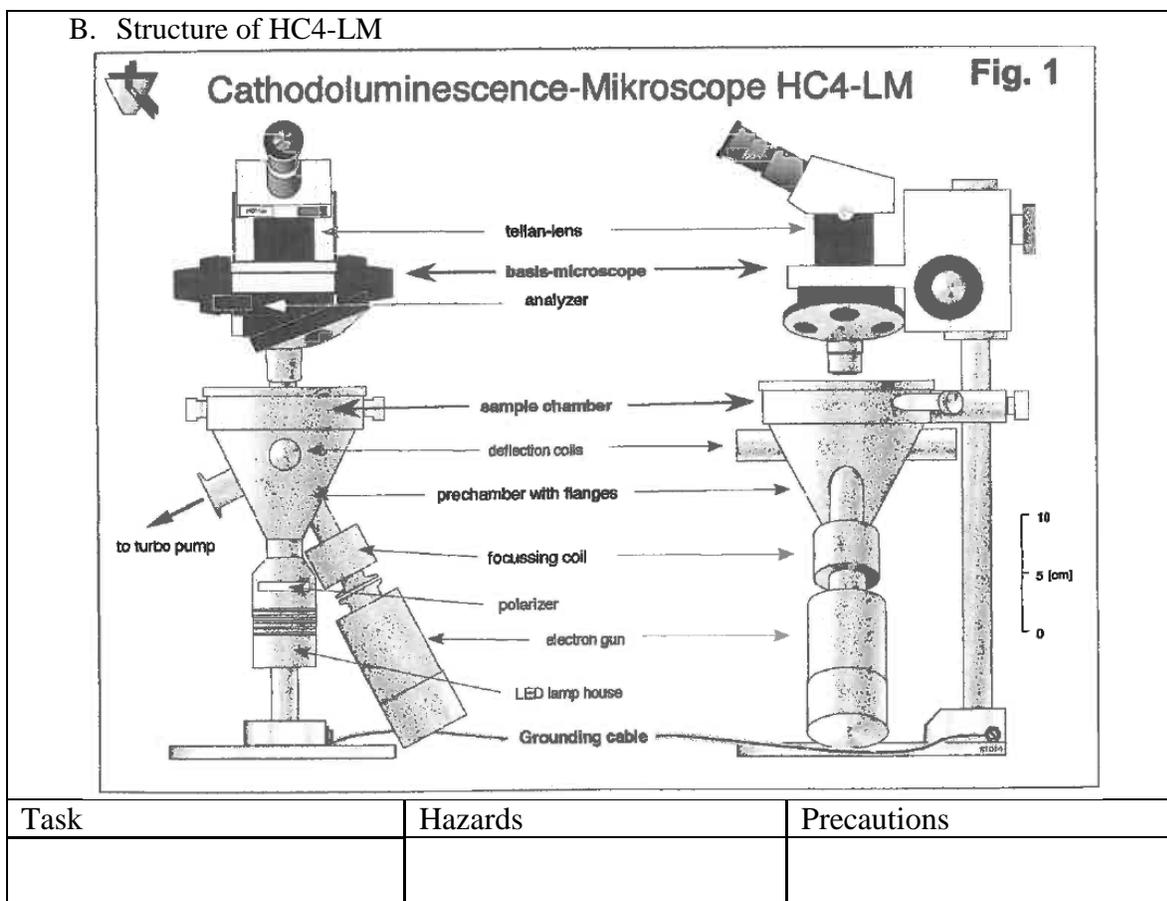
Appendix:

A. HC4-LM System diagram



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B. Structure of HC4-LM



8.0 References:

1. Maria Simon-Neuser. Cathodoluminescence-Microscope HC4-LM User and Service Manual. Lumic. 2008. Germany.
2. Kappa Opto-electronics GmbH. Kappa CameraControl V2.1, Digital Cameras PS 4/40, Digital Camera Systems DX 4/40 Operating Manual. 2008. Germany.
3. Randy Corney. Basic Operation of the HC4-LM Cathodoluminescence Microscope, Department of Geology, Saint Mary's University, 2014.

9.0 Applicable regulations and/or legislation:

10.0 Revision History:

Rev #	Revision Date	Review Date	SOP Section(s)	Revision Description	Revised By
0	Sep 15, 2018			SOP-CLM-YANG-001 created	Xiang Yang
1	Sep 20, 2018			Revise the power on procedure to simply the work.	Xiang Yang
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