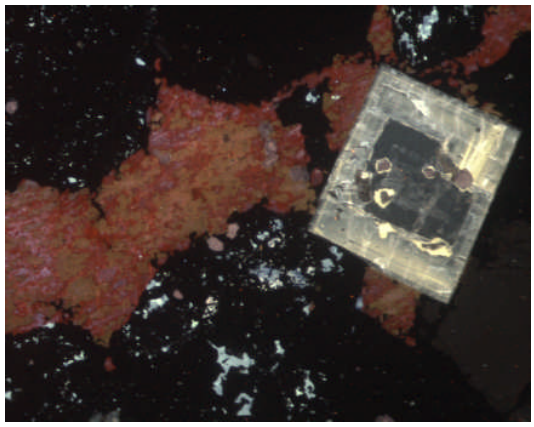
University of St Andrews

Facility for the Luminescence of Minerals

CATHODOLUMINESCENCE IMAGING

AT THE

UNIVERSITY OF ST ANDREWS



Instruction Guide by Adrian Finch

Noddy's Guide to Using the CL at St Andrews (SHORT version)

1. Switch on plugs at Wall (Microscope, Luminoscope and Camera Power)
2. Turn on Microscope Light
   Turn on Power switch on luminoscope
   Turn on Power switch to Camera
3. Boot Computer, Double-click on XCAP for Windows Icon
4. Pull out Chamber door on left of CL chamber
5. Load samples and push door closed.
6. Turn **OPERATE** switch to "2" and wait for vacuum
7. Turn **ADJUST kV** switch 1 full turn to the left
8. Turn **OPERATE** switch to "3"
9. Using **ADJUST kV,** allow current to settle down to 600 μA
10. Keep at optimum conditions with **VACUUM CONTROL** knob
11. Take photos…, make observations.

When you've finished...

12. Turn **OPERATE** switch to "2", then to "1", chamber vents automatically.

More samples? Go to 4

13. Remove your samples and close chamber door
14. Switch off **POWER** on luminoscope and plugs at wall.

Noddy's Guide to Using the CL at St Andrews (LONG version)

GENERAL NOTES

Layout of the Guide

This guide is in three parts. The first is a SHORT version (summarised in one page) for users who are familiar with the machine and only require prompts for successful operation. The second part is a LONG version of the operation guide, detailing each step and explaining the responses expected from the equipment. This section is aimed at those using the equipment for the first time.

Technical Specifications of the Machine

The Department of Earth Sciences has a Technocyn 8200 Mk III Luminoscope mounted on a Nikon Optiphot Microscope which has had the normal stage replaced by the CL chamber. A DVC high-sensitivity CCD camera using EPIX software is mounted on the third ocular of the microscope. Beam conditions are maintained at ~600 μA and ~15 kV corresponding to a power density of 50 kW m^{-2}. Typical exposure times are 8 s and the images can be enhanced after collection using Adobe Photoshop.

Vacuum system

One vacuum pump serves both the carbon coater and the CL. You may need to swap the vacuum pump between the two systems. Twist the Knob just above the pump fully counter-clockwise and also make sure that the glass chamber of the carbon coater is sealed (turning the knob on the top of the glass chamber fully clockwise will close it - light finger pressure only on this knob please).

Objectives

There are three objectives on the rotating head, but only two can be used for CL. The 4x and 20x can focus on the sample in the CL chamber, sadly the 40x will not focus on the sample in the chamber. The 20x is a long working-distance lens that can focus even though it is still a long way from the sample. Do not attempt to use it, since it will crack against the glass window as you try to movie it up and down.

Problems

If any problems arise during operation of the machine, these should be addressed immediately to Donald Herd (dah, 2018) or Adrian Finch, (aaf1, 2384). There is an internal telephone in the next room (next to Donald's desk).

STARTING CONDITIONS

These starting conditions are assumed by the following sets of instructions. Some of them are evident when the machine is switched off but others will require the control box to be switched on before you can check.

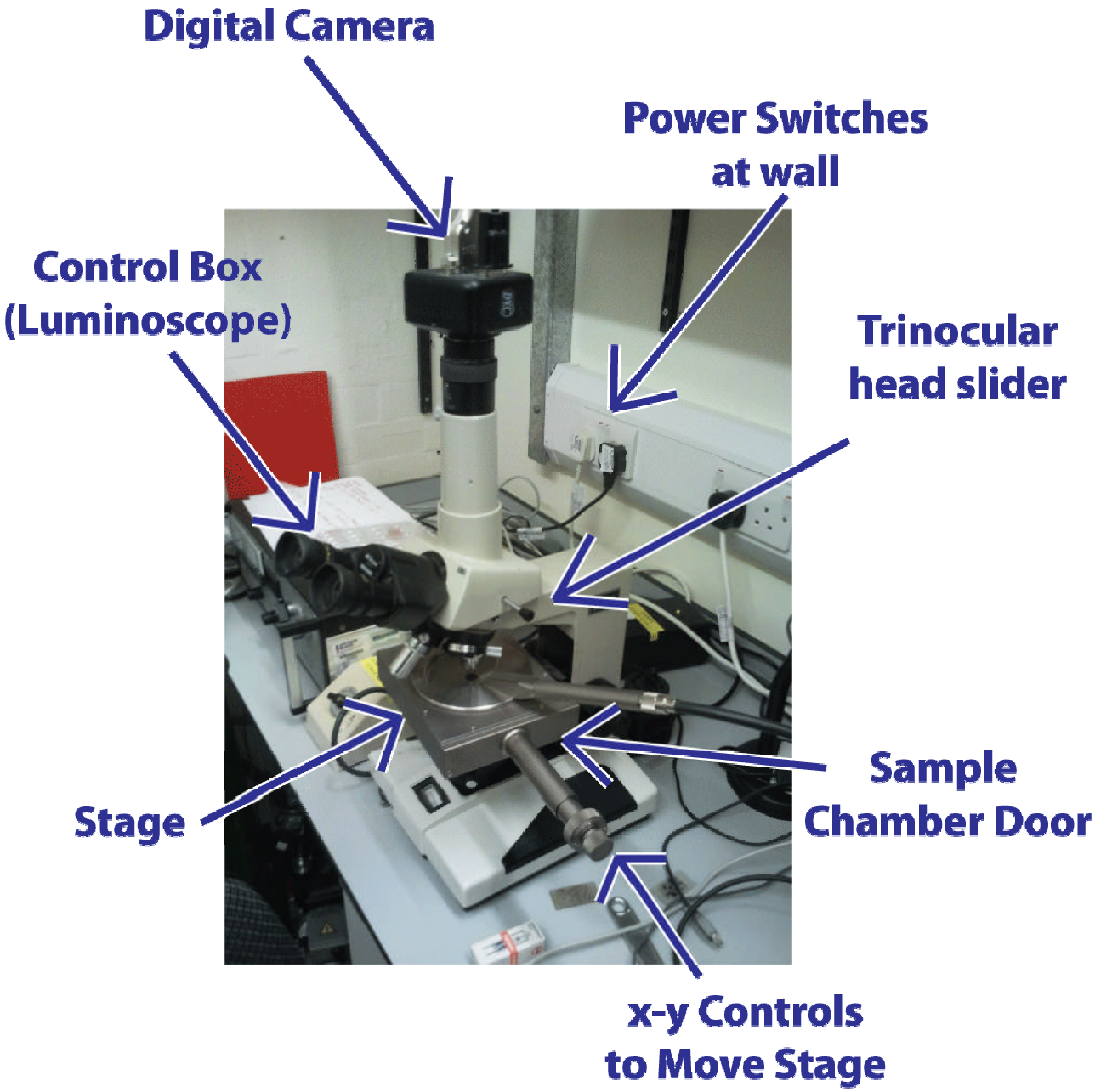


Figure 1: Overview of CL Petrography System.

START UP CONDITIONS

On the Chamber --

* SAMPLE CHAMBER DOOR pushed closed.
* TOP PLATE (including electron gun) in place and flat.

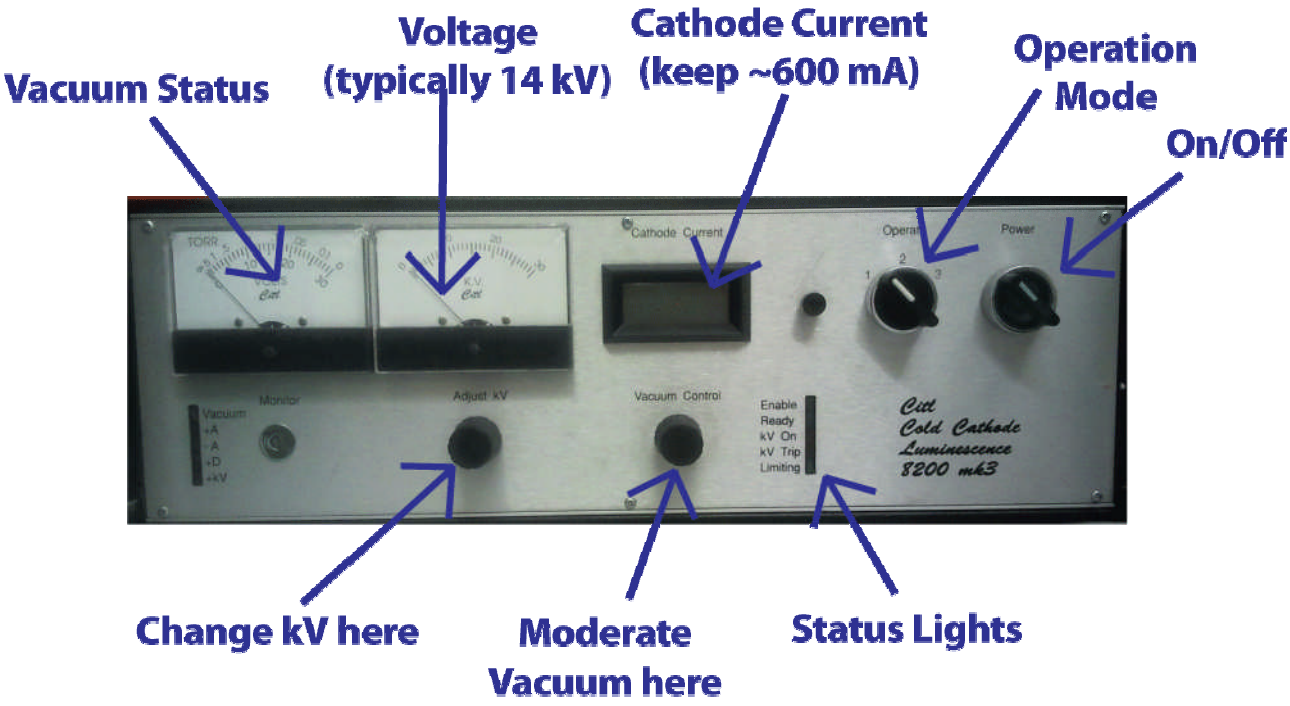


Figure 2: Front Panel of the Control System (Luminoscope)

On the Luminoscope (Control Panel) –

* **OPERATE** switch set to "1".
* Green **POWER** switch off (points to the left).
* When **POWER** is on, green **ENABLE** light on Status Lights; all others off.

On the Microscope --

* Sub-stage lighting off.
* Two very useful beer-mats to hand.

Camera Power Unit --

* Power Off.

OPERATION

1. Switch on all plugs at the wall: a) Microscope, b) Luminoscope and c) Camera Power unit.

2. Turn rotating sub-stage lighting switch on the microscope anticlockwise to minimum setting.

   Sub-stage lighting comes on

3. Turn green power switch on luminoscope clockwise.

   Luminoscope display lights up

4. Boot the Computer (138.251.88.68), click on the XCAP icon. Click on 'Live'. Pull the slider on the side of the trinocular head (Fig. 1) and you should see the ppl image of your slide.

5. Pull left-hand side of CL chamber (loading door).

   Door slides out revealing samples on a moving carriage

6. Using the x-y movement controls, move the sample carriage as far to the left as possible to allow loading of samples.

7. Load your samples onto the tray (you will look at the right-hand sample first).

8. Push chamber door securely closed.

9. Turn **OPERATE** switch on luminoscope to "2".

   Vacuum pump comes on
   Vacuum pump rhythm starts to change
   Vacuum gauge responds and starts to fall

10. Wait for vacuum to start to respond (vacuum gauge, Fig. 1)

    If the vacuum does not fall and there is a hissing noise, push the chamber door closed, press the top plate down.

    Wait until vacuum is <0.1 Torr...

    Check: **ENABLE** and **READY** lights are on, all others off.

11. Turn the **ADJUST kV** knob a full turn anticlockwise.

    Turn the **VACUUM CONTROL** knob a full turn clockwise.

12. Turn luminoscope **OPERATE** switch to "3".

    kV ON *light comes on*
    kV gauge responds
    CATHODE CURRENT *(digital display) responds*

13. If there is a 'click' and an alarm, the **kV ON** light goes off, the **kV TRIP** light is on and the kV gauge reads zero, then a safety circuit within the luminoscope has tripped. To restart:-

    a). Return **OPERATE** switch to "2"
    b). Turn **ADJUST kV** knob one full turn anticlockwise
    c). Turn **VACUUM CONTROL** knob one full turn anticlockwise
    d). Try again from 10.

14. Optimum conditions are 14 kV for feldspars, apatites, carbonates, sodalites and fluorites, 14 kV+ for quartz. Don't be frightened to use lower kVs if what you want to see is luminescent, since this minimises damage to the slide.

Comments

- You will probably find at first that you cannot achieve the required kV for a **CATHODE CURRENT** of 600 μA. The factor that determines which current is stable with which voltage is the state of the vacuum. The skill (!) of CL work is knowing how to juggle **VACUUM CONTROL** to provide the required conditions. Generally I keep the kV constant and adjust the vacuum.
- At first as the vacuum slowly increases, the current will drop and so you must slowly increase the kV to compensate. You are trying to keep the **CATHODE CURRENT** constant (digital display at 600) whilst the kV is at the required values. To keep it there, you must adjust the **VACUUM CONTROL** knob to keep the **vacuum** constant. The **VACUUM CONTROL** knob allows a small amount of air to seep into the chamber, compensating for air drawn by the vacuum pump. THIS IS AN ART FORM! Sometimes tiny differences in **VACUUM CONTROL** will significantly influence kV and cathode current.
- Turn the substage lighting on. To change from PPL to CL views, insert the beer-mats over the sub-stage light source – this provides a simple way of switching between PPL and CL images. (If you set the sub-stage lighting too high, you'll blind yourself going between the images).
- Move the sample using the x and y adjusting screws on the end of the position control arm on the chamber.
- The **focus position for the camera** may not be exactly the same as that for the eyepieces. Once you move to the CL view, set the acquisition time as 1 s and maximise the gain. Although this image is ugly, it refreshes quickly making it easier to focus. Try to get point luminescent sources (often grains of polishing powder) as sharp points rather than diffuse discs.
- **Set the XCAP exposure time** at around 8 s and the gain at ~10 dB. For most minerals, this gives a satisfactory result; remember you can extend the exposure times but going beyond ~16 s is not recommended.

When you have finished...

15. Turn the **OPERATE** switch to "2".

    ***kV ON** light goes out, CATHODE CURRENT goes to zero, kV goes to zero*

16. Turn the **OPERATE** switch to "1".

    Pump goes off, chamber vents automatically

MORE SAMPLES? Go to 4.

SHUT DOWN PROCEDURE

16. Open chamber and remove your samples.

17. Push chamber door securely closed.

18. On computer, close XCAP window, click Start >> Shut Down >> Shut Down

18. Switch off **POWER** switch for a) luminoscope, b) camera power and c) lighting to stage. Turn off plugs at the wall.

PHOTOGRAPHIC SYSTEM

Room lights: Stray light from the room will enter the CL producing light that will make the CL images less crisp. You can use the anglepoise lamp behind the CL for low level light if you find the room too dark to work. You may even plug it in on the far side of the room. However, always work with the room at low light levels.

Slider: The microscope has a trinocular head with a beam splitter to pass the light either all to the eyepieces or to split it between the camera and the eyepieces. If you look in the eyepieces and pull the slider out, you should see the image darken. When the slider is out (Fig. 1), light is passed between the eyepieces and the camera. If the slider is in, all light goes to the eyepieces and the camera gives a blank live image.

Gain: XCAP defaults to an automatic exposure and a gain of 0 dB. We have found the best CL images are at about 8 s exposure time and a gain of ~10 dB. Set the gain at the start you start.

Focus: The focus position of the camera is not necessarily the same as that of the binocular eyepieces, although it should not be far off. Look carefully for bright grains of polishing powder – are these points or diffuse discs? Set the exposure time to 1 s and the gain to maximum. This will give you visually unpleasant images, but they refresh sufficiently fast to allow tweaking of the focus. Once the focus is OK, drop the exposure time back to the original values, e.g. 8 s and ~10 dB.

Exposure Time: The exposure time is initially automatically controlled, but if you change this manually the automatic values are overridden. Typical exposure times for CL are 8 – 16 s, if you get good images with faster times, then fine. Longer exposures are possible but the stage sometimes sinks under its own weight making these difficult to keep in focus.

CL and ppl images: It is normal procedure to take both ppl and CL images of the same areas. This can be done easily by ***inserting beer mats*** between the light at the base of the microscope and the substage diaphragm. The camera does not like being wildly overloaded, so when flipping from CL to ppl images, reduce the exposure time to e.g. 81 ms ***before you remove the beer mats***.

Saving Images: To save the images, click off the live button (the 'unlive' indicator is checked) and then click File >> Save Image. This can only be done in 'unlive' mode.

Data Storage: There is a shortcut to the Data Archive on the Desktop. If you do not already have one, make a directory in Data Archive with your name as the title. Copy all files to here. Avoid odd files on the desktop or in the 'My Documents' folder.

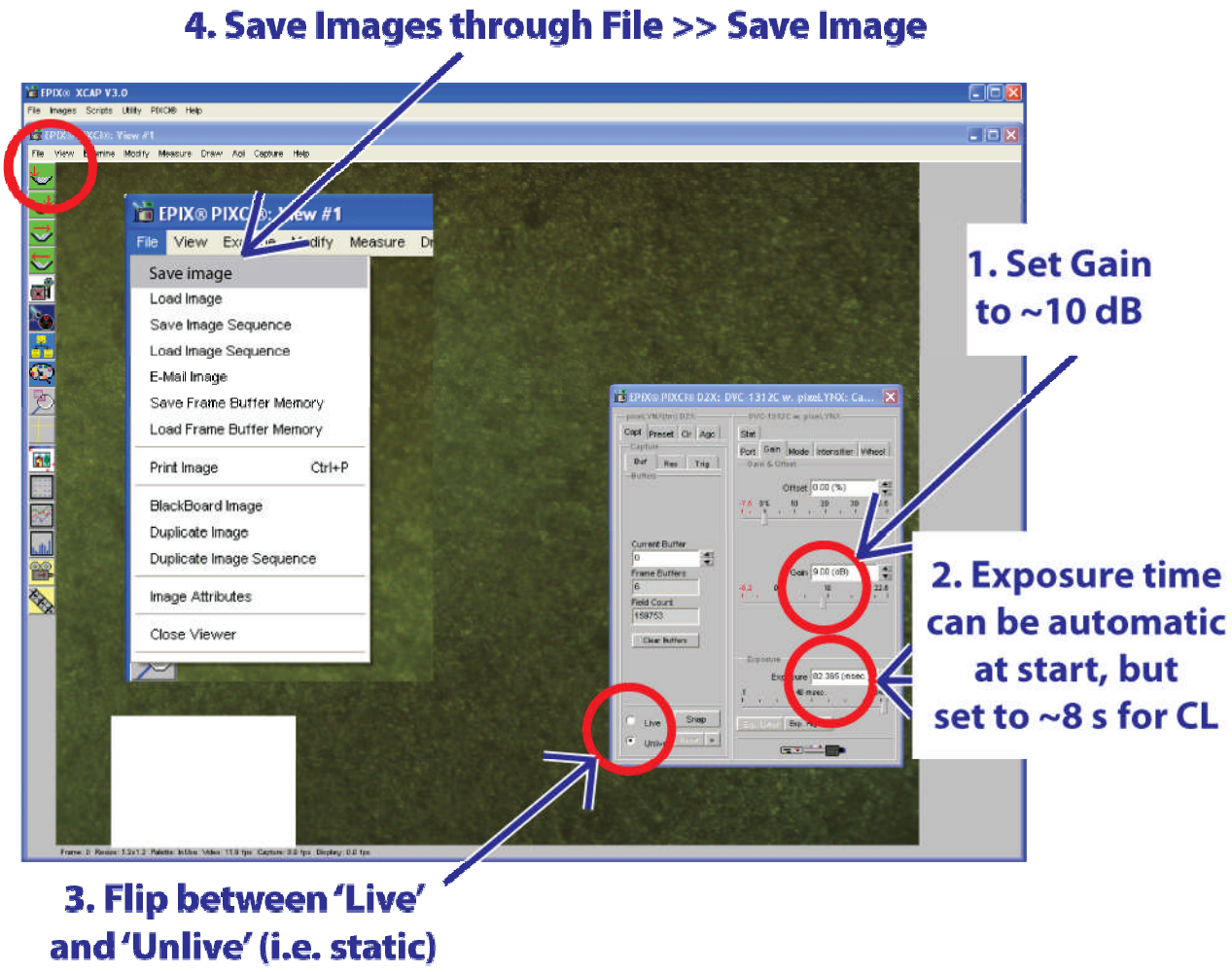


Figure 3: EPIX Screen Dump showing the main commands.

A Few Handy Tips

- ***Run the CL at the lowest kV*** at which you are sure that the minerals you want to study are luminescing. Araldite mounting becomes unstable at around 12 kV and at very high kVs can actually luminesce yellow or orange.
- ***Keep the sample on the move*** as much as possible. This reduces the likelihood of losing the slide or burning the mount. Use fast films (e.g. ASA 1000) to reduce exposure times during photography and move the sample immediately after a photograph is taken.
- When working at hard vacuums, ***don't move the x-y position controls jerkily*** - this allows gulps of air into the chamber, which ruins the state of the vacuum.
- Sometimes when you're starting, the vacuum is very unstable. This is because of ***adsorbed moisture and gases*** on the inside of the chamber. Allow a few minutes without the electron beam to let the vacuum settle down.
- Occasionally when you start, there is little current for the kV being applied. This is because there are no ions in rarefied atmosphere in the chamber to conduct the current. In you increase the voltage too much, the current suddenly gives (a lighting strike!) and immediately trips the safety control in the luminoscope. If you are having trouble in getting a current to flow, then slowly crank up the voltage but be prepared to wind it immediately down again, once you feel it is about to pass current. With practice, you can stop it tripping the safety switch. Once a chamber has passed a current, there are usually enough ions in the chamber to stop this problem arising again.
- Remember - Don't hang about - be swift and efficient.

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12 Dec 2011