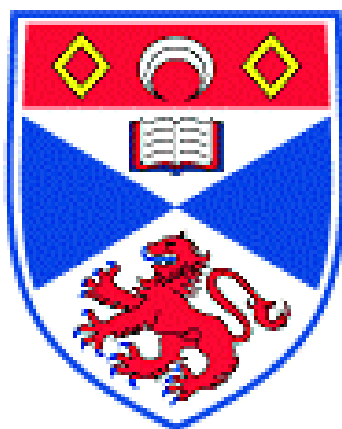


**CATHODOLUMINESCENCE**  
**AT THE**  
**UNIVERSITY OF ST ANDREWS**



**Instruction Guide**

by

**Dr Adrian Finch**

## **General Notes:**

# **N.B. NO-ONE SHOULD OPERATE THE CATHODOLUMINESCENCE EQUIPMENT WITHOUT EXPLICIT CLEARANCE FROM DR ADRIAN FINCH**

### **Technical Specifications of the Machine**

The School of Geography and Geosciences has a Technocyn 8200 Mk V Luminoscope fitted with an Alcatel Vacuum Pump. The chamber is mounted on a Nikon Optiphot Microscope which has had the normal stage replaced by the CL chamber. A digital camera is mounted on the third ocular of the microscope.

### **Layout of the Guide**

This guide is in three parts. The first is a SHORT version (summarised to one page) for those students 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 students using the equipment for the first time. The third part of the guide is a series of instructions to take photographs.

### **Problems**

If any problems arise during operation of the machine, these should be addressed immediately to Dr Adrian Finch, extension **2384**. There is an internal telephone in this room.

### **References**

A folder containing references relevant to cathodoluminescence is available. In addition, Mineralogical Magazine and American Mineralogist (held in the periodicals section of the Library) contain many articles about cathodoluminescence and its uses.

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

1. Switch on plugs at Wall (Microscope, Luminoscope and Photo control)
2. Turn on Microscope Light  
Turn on Power switch on luminoscope
3. Pull out Chamber door on left of CL chamber
4. Load samples and push door closed.
5. Turn **OPERATE** switch to "2" and wait for vacuum
6. Turn **ADJUST kV** switch 1 full turn to the left
7. Turn **OPERATE** switch to "3"
8. Using **ADJUST kV**, allow current to settle down to 800  $\mu\text{A}$
9. Keep at optimum conditions with **VACUUM CONTROL** knob

### **When you've finished...**

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

### **More samples? Go to 3**

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

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

## 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.

### **On the Chamber --**

- CHAMBER DOOR pushed closed.
- TOP PLATE (including electron gun) in place.

### **On the Luminoscope --**

- OPERATE switch set to "1".
- Green POWER switch off (points to the left).
- When POWER is on, ENABLE light on, all others in array are off.

### **On the Microscope --**

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

### **Photographic Control unit --**

- Control knob turned to AUTOMATIC on dial on front.

## Operation

1. Switch on all plugs at the wall (Microscope, luminoscope and photo control 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. Pull left-hand side of CL chamber (loading door).

*Door slides out revealing samples on a moving carriage*

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

6. Load your samples onto the tray (you will look at the right-hand sample first).
7. Push chamber door securely closed.  
Turn the **VACUUM CONTROL** knob a full turn clockwise.
8. Turn **OPERATE** switch on luminoscope to “2”.  
*Vacuum pump comes on*  
*Vacuum pump rhythm starts to change*  
*Vacuum gauge responds and starts to fall*
9. Wait for vacuum to go down to at least 0.1 Torr.  
Check: **ENABLE** and **READY** lights are on, all others off.

### **When Vacuum is Good...**

10. Turn the **ADJUST kV** knob a full turn anticlockwise.  
Turn the **VACUUM CONTROL** knob a full turn clockwise.
11. Turn luminoscope **OPERATE** switch to “3”.  
*kV ON light comes on*  
*kV gauge responds*  
*CATHODE CURRENT (digital display) responds*
12. 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.
13. Optimum conditions are 14 kV for feldspars, apatites, carbonates, sodalites and fluorites, 14kV+ for quartz. Don’t be frightened to use lower kVs if what you want to see is luminescing, since this minimises damage to the slide.

### **The Operation**

- You will probably find at first that you cannot achieve the required kV for a **CATHODE CURRENT** of 600  $\mu$ A. 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 **ADJUST kV** and **VACUUM CONTROL** to provide the required conditions.
- At first as the vacuum slowly increases, the current will drop and so you must slowly increase the kV to compensate. You are now trying to keep the **CATHODE CURRENT** constant (digital display at 600). Eventually the kV will reach the required values. However, to keep it there, you must now 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, exactly compensating for the amount of air drawn out by the vacuum pump. **THIS IS AN ART FORM!** Sometimes tiny differences in the **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 then 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. To take photographs, see the separate section on photography.

### **When you have finished...**

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

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

15. 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. Switch off **POWER** switch on luminoscope and plugs at the wall.

### **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 immediately wind it 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.

## Noddy's Guide to Photography at St Andrews

- Make sure that the photographic unit is connected to the camera, and that it is switched on.
- Find the view that you want to photograph in the normal binocular microscope.
- Pull the black handle on the right-hand side of the eye piece to the out position - the view in the binocular microscope darkens.
- Check that there is an image in the single eyepiece at the top of the column - check focus in this eyepiece!
  
- For **AUTOMATIC** exposures, click the photographic unit to **AUTO** and select the relevant film speed. The light-meter on the top of the column should register an exposure time (the red light is out).
- For **MANUAL** exposures, set the exposure time as required.
  
- Press the **EXPOSE** button on the photographic unit and the shutter opens (green light comes on)
- When you have finished, remove the film from the camera.

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