Multi-Laboratory Results for the Cathodoluminescence Emission Spectrum from a Synthetic Zircon Standard

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MULTI-LABORATORY RESULTS FOR THE CATHODOLUMINESCENCE EMISSION SPECTRUM FROM A SYNTHETIC ZIRCON STANDARD

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Abstract

The Standards Committee of the Society for Luminescence Microscopy and Spectroscopy (SLMS) circulated doped zircon crystals as a standard for comparison of cathodoluminescence (CL) emission spectra obtained at different laboratories. Eleven laboratories have submitted spectra acquired from this standard. The crystals are synthetic zircons doped with 1.4 wt. % of Dy_2O_3.

The participating laboratories used a variety of CL instrumentation including cold cathode optical microscope attachments, hot cathode optical microscope attachments, and EMPA/SEM-based systems. Two laboratories provided both uncorrected and corrected spectra. All other spectra are uncorrected. A variety of different spectrometers/spectrographs and detector systems were used. Photomultiplier tubes (PMTs), photo diode arrays, intensified photo diode arrays, and charge coupled devices (CCDs) are represented among the final results. The laboratories with apparently the best resolving power reported 8 peaks in the multiplet at 485 nm. The spectra submitted by some laboratories showed only a single peak at this position.

The wavelength scale calibrations were compared by noting the wavelength of the most intense peak in the 485 multiplet. This varied from 476.3 nm to 489 nm among the eleven laboratories.

The systems have different overall transmission and detection functions: a combination of the influence of the transmission of the viewing window (typically leaded-glass), the microscope, the fiber optics coupling (if used), the response of the grating and spectrometer/spectrograph, and the detector response. As an initial test of this variation, the ratio of the peak maximum intensity at 575 nm to that at 485 nm was compared. This ratio varied from 0.6 to 3.93 among the eleven laboratories.

Key Words: Zircon, cathodoluminescence emission spectra, spectral standards, dysprosium.

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Introduction

Applications of cathodoluminescence (CL) in the earth sciences began in earnest in the early 1960s, after the papers by Smith and Stenstrom (1963, 1965) and Long and Agrell (1965). Much of the initial interest was in visual observations, and for several years, only a relatively small number of laboratories had spectrometers attached to their CL apparatus. Often, these investigators worked in conjunction with physicists. Now, spectrometers are much less expensive and easier to operate, and as a result, they are more frequently used.

Within the past few years, there has been an ever-expanding presentation of CL emission spectra in the earth science literature. The quality of published spectra varies widely, and there is often inadequate information given on important parameters such as the calibration of the wavelength scale and the calibration of the total system response as a function of wavelength. The idea was developed of asking different laboratories to provide CL emission spectra on a standard sample to try to develop more information on the variations that existed. This is not an original idea, and, among others, Dr. Remond suggested to one of the authors (DJM) that standards were needed in 1991 at the Scanning Microscopy Cathodoluminescence Conference in Bethesda (Maryland).

The standard selected is a batch of zircon crystals, prepared by John Hanchar of the Dept. of Earth and Environmental Sciences at Rensselaer. The program was done under the auspices of the Society for Luminescence Microscopy and Spectroscopy (SLMS). This Society has been in existence for about 10 years; as one of its goals, it has encouraged and helped the exchange of information on cathodoluminescence and its applications, especially among the earth science community.

The program was announced in the Spring 1993 Newsletter of the SLMS. To date, about 20 laboratories have requested one of the zircon crystals, and eleven laboratories have responded. A preliminary report, containing the results from seven laboratories, was circulated among the participating laboratories in December of 1993; a final report, with the results from 11 laboratories, was circulated in August, 1994.
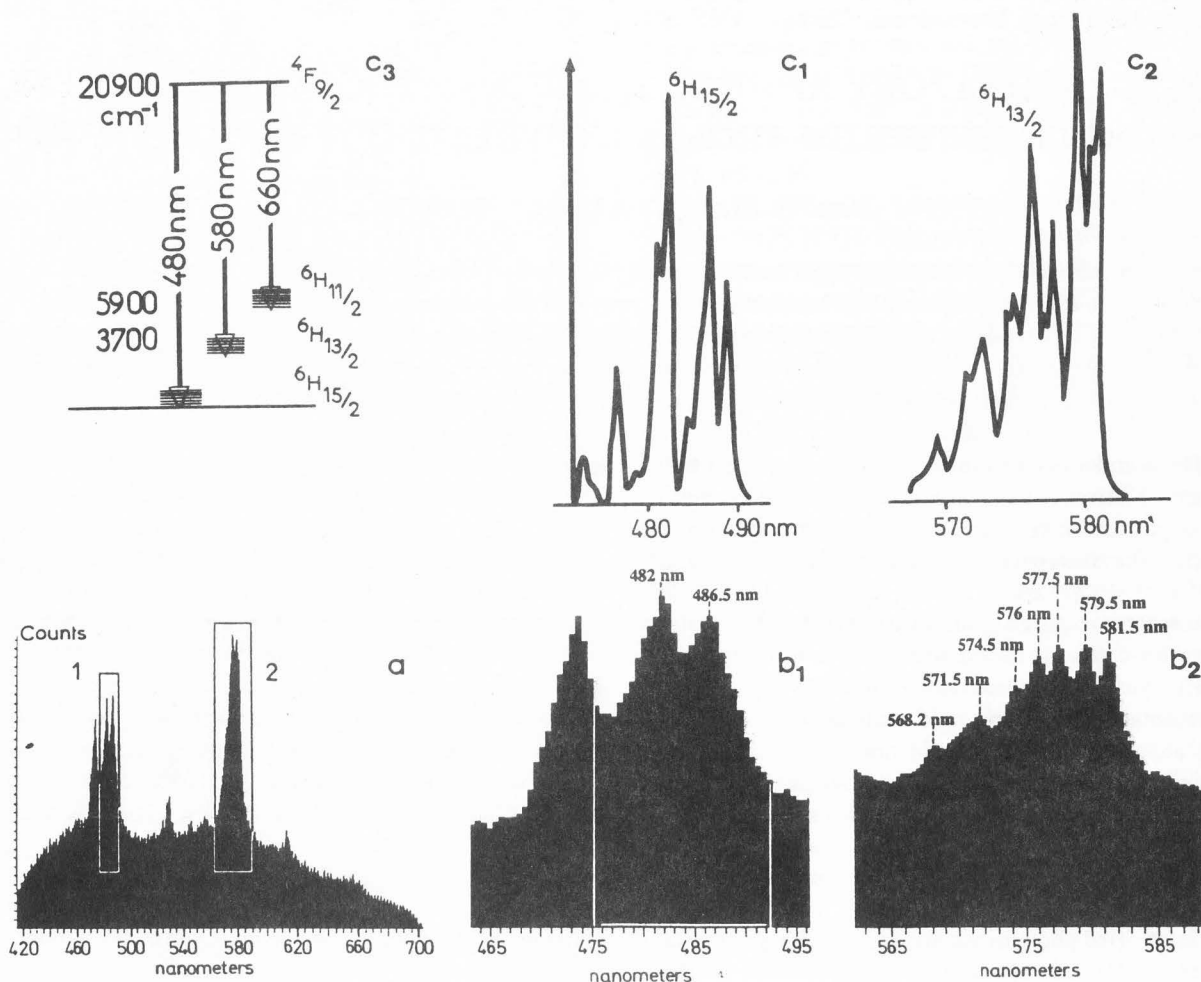
Figure 1 (top). Fine structure of CL peaks near 482 nm and near 575 nm for Dy^{3+}-doped zircon, as presented by Remond *et al.* (1992). Data recorded with multichannel spectrometer.

Figure 2 (bottom). Luminescence spectrum and energy level diagram for Dy^{3+} in zircon according to Taraschan (1978). Reproduced from copy presented by Remond *et al.* (1992).

The participating laboratories used a variety of CL instrumentation, including cold cathode optical microscope attachments (Marshall, 1993), hot cathode optical microscope attachments (Ramseyer *et al.*, 1989), and EMPA/SEM-based systems. Operating conditions were suggested but each laboratory made a final decision as to how they would normally observe such a sample. In particular, it was recommended to use an electron beam of 4.5 keV energy, but actual electron energies varied between 4.5 and 30 keV.

Previous Work

Dieke's compilation lists 101 lines in the visible region (400-700 nm) for the fluorescence spectra of $LaCl_3$ crystals, doped with Dy^{3+} (Dieke, 1968). Additional lines outside of the visible region are also listed.

Mariano (1988) states that the luminescence spectrum of Dy^{3+} in zircon is complex and consists of narrow bands (multiplets) in the 480-490 nm and 570-585 nm regions. These are superimposed on the intrinsic CL. Remond *et al.* (1992) presented the CL emission spectrum from a Dy-doped zircon which is reproduced in Figure 1. Marfunin (1979) presented the spectrum shown in Figure 2 which is actually photoluminescence data from Taraschan (1978).

We concluded that this complex spectrum would provide a useful comparison among laboratories with different spectral acquisition systems and different electron sources. Participants were requested to obtain the spectrum at 4.5 keV electron energy. All but two laboratories actually used higher electron energies, ranging from 6 to 30 keV.

Table 1. Summary of instrument parameters.

Lab	Focal Length[1] (mm)	f no.	Entr. slit (mm)	Exit slit[2] (mm)	Grating groove density (gr/mm)	keV/mA (power density) watts/cm^2	Detector	PMT volts (volts)	Coupling method[3]	Area sampled (diameter) (μm)	Acquisition times
A	200	f/4.2	0.25	0.25	1200	8/0.45 (18)	PMT	950	FO	312	2-3 min.
B	200	f/3	0.1	N/A	200	4.5/0.7 (44)	Array	N/A	FO	312	10 sec.
C	—	—	—	N/A	150 and 300	15/2 nA (4)	Array	N/A	FO	—	—
D	227	f/4	0.25	0.25	1200	4.5/0.7 (16)	PMT	750	FO	500	2-3 min.
E	300-500	f/3.2	0.5	0.5	—	15/40 nA (12)	PMT	790	FO	80 x 80	—
F	125	f/3.5	0.05	N/A	400	16.500.1 (23)	Array	N/A	DP	—	0.24 sec.
G	—	f/4	—	—	—	6/0.6 (0.11)	PMT	—	—	—	> 2 min.
H	155	f/4	0.025	N/A	1800	12/0.7 (11)	Array	N/A	DP	2000-3000	2-3 min.
I	200	—	2.5	2.5	1200	30 (0.76)	PMT	1200	FO	250	—
J	220	—	2.5	2.5	—	8/0.9 (12)	PMT	—	FO	50	250 sec.
K	175	f/3.4	4	1	1200	15/0.5 (106)	PMT	1000-1200	FO	100-200	200 sec.

[1]---: information was not furnished. [2]N/A: not applicable. [3]FO: Fiber optics; DP: direct projection.

Present Work

Standard samples

The purpose of this standard was to provide a means for different laboratories doing spectral analysis of CL emission to compare results on the same sample. Circulation of a single sample was not deemed satisfactory for a number of reasons, so we chose to use doped synthetic crystals from a batch of a large number of such crystals prepared at the same time under identical conditions.

Approximately 100 zircon standard crystals of approximately 1 mm largest dimension were grown, doped with Dy_2O_3. Dy^{3+} was selected for the zircon standard because of its importance in previous CL studies of zircons (e.g., Marfunin, 1979; Mariano, 1989).

Methods

The synthetic zircons were grown by John Hanchar from a flux consisting of 7.5 mol% Li_2MoO_4 and 86.5 mol% MoO_3, and stoichiometric proportions of ZrO_2 and Li_2SiO_3. The method used is a modification of the method reported in Chase and Osmer (1966). Reagent grade chemicals were used. The Dy_2O_3 used in the Dy-doped zircons was spectroscopic grade. The reagents were mechanically mixed and then placed in a 10 ml Pt crucible. The crucible was placed in a $MoSi_2$ furnace at 1250°C and allowed to "soak" for 4 hours, and then

cooled to 1000°C at a cooling rate of 2°C per hour.

The dysprosium oxide concentration is 1.4 wt. %. A group of crystals observed with CL appeared homogeneous in their CL color. However, we did not obtain the individual CL spectra of all of the samples on a single instrument to guarantee uniformity (but see later section on **Additional Observations**).

The dysprosium concentration of 1.4 wt. %, analyzed with EMPA, is the maximum amount that can be introduced without the simultaneous introduction of a charge compensating species (e.g., phosphorus) allowing a xenotime-type substitution:

$$Zr^{4+} + Si^{4+} \rightleftarrows REE^{3+} + P^{5+}$$

The concentration of Dy in natural zircons is much less, generally in the 100 to 1000 ppm range.

General considerations

The following aspects of the CL emission spectra generated with different systems are considered: (1) Resolution; (2) wavelength of observed peaks; (3) transmission variations with wavelength; (4) intensity and signal-to-noise ratio; and (5) stability of CL intensity with time.

The Society for Luminescence Microscopy and Spectroscopy does not presume to say what the correct

values are for the wavelengths of the various peaks observed or for any other aspects of the results. Our function is simply to report the data in a format which facilitates interlaboratory comparisons.

Instrumentation

The instrumentation and conditions used by the several participating laboratories are summarized in Table 1 (the identification of the laboratories and a slightly more expanded instrument description is provided in Appendix 1).

Those instruments with photomultiplier (PMT) detectors use scanning of the wavelength range to record data. This scan typically requires between 30 seconds and 2 minutes, depending on the scan parameters selected (the term "scanning" is used in the general sense, but actually stepping motor drives are used on most instruments and these step by discrete wavelength intervals).

Those instruments with array detectors do not use scanning and collect data at all wavelengths within a selected range simultaneously. Data collection times typically range from less than 1 second to a few seconds. If the CL exhibits decay characteristics, the shorter collection time realized with an array detector is an advantage.

With an array detector, the physical length of the detected region is fixed by the size of the detector, nominally 15 to 25 mm for presently available arrays, and the wavelength range this length represents varies with the size of the spectrograph and the grating selected. The user may collect the entire visible range in a single exposure, at reduced resolution, or may (depending on the instrument design) select other gratings which cover only a portion of the visible range but produce higher resolution. Several exposures at different grating orientations can then be used to acquire data for the entire visible range. The resolution improvement is achieved at the expense of longer data collection times and the inconvenience of switching or adjusting gratings. The grating adjustment is usually done under computer control and may involve the use of a program which will "splice or glue" spectra together at the overlap region.

Laboratory H used an array detector with an 1800 grooves/mm grating; with this system, it was possible to collect only 40 nm per exposure. The data was collected in 12 "steps," with a 20 nm overlap between steps, to cover the visible range between about 450 and 625 nm. This required about 2 minutes time, of which about 24 seconds was spent in actual data collection and 80 to 90 seconds was spent in stepping the grating rotation across the region of interest.

Data reduction

The results are presented as raw spectra with the ex-

ception of two laboratories which provided both raw spectra and spectra corrected for transmission variations with wavelength. No data massaging procedures such as smoothing were reported. Peak deconvolution was used by some laboratories. The spectra submitted are reproduced in Appendix A of Marshall (1994).

Results Observed for Certain Key Performance Parameters

Resolution

The resolution differences are shown by comparison of the raw spectra. The focal lengths of the spectroscopes ranged from 125 to 500 mm with all but two in the 175-220 mm range. There are significant differences in the resolution. One test of the resolution is the number of separate peaks observed in the multiplets. For the 485 region, this ranged from 1 to 8, with most investigators observing evidence of at least 3 peaks in the multiplet.

Resolution is important, but it is often traded off against sensitivity and other factors as mentioned earlier. Higher resolution, as evidenced by separation of peaks in multiplets, is important when other chemical elements are also present and their spectra must be resolved; it may also be useful in confirming the identification of, e.g., dysprosium. However, to our knowledge, no use has yet been made of the multiplet CL emission spectra in geological samples to establish information on site occupancy or other crystal field parameters.

Wavelengths of observed peaks

Each of the laboratories reported the wavelengths at the peak maxima in the 485 and 575 nm regions. For those laboratories which obtained a single unresolved peak in each of these regions, the peak wavelengths are shown in Table 2. The observed peak wavelengths for these five laboratories differ by 12 to 14 nm in the extreme cases. For those laboratories which observed multiplet structure (2 or more components in the 485 and 575 regions), the individual peak wavelengths generally fell within the above ranges. In some cases, the multiplets were partially resolved and the apparent maxima were straightforwardly determined. In other cases, deconvolution procedures were used to determine the components of a peak.

Some of the laboratories used a Hg lamp to establish the wavelength scale. Most laboratories did not report their method for wavelength calibration.

Transmission function

All systems have different responses at different wavelengths associated with the transmission of the various optical elements in the light path, the response of the detector at different wavelengths, the efficiency of the

Table 2. Observed wavelengths of 485, 575, 650, and 750 nm region peaks for those laboratories which did not observe the multiplet structure. The observed peak wavelengths for these five laboratories differ by 12 to 14 nm in the extreme cases.

| Laboratory | Wavelength region | | | |
	485	575	650	750
C*	483	581		
G**	489	584	671	761
I***	476	572	657	749
J	481	575	663	751
K	483	577	664	752

*Spectrum obtained with coarser grating.

**Corrected and uncorrected spectra gave same peak wavelengths.

***Corrected spectrum.

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grating, etc. Some laboratories use a standard light source to correct for transmission variations, but most laboratories are apparently not equipped to do this.

The transmission variations are qualitatively indicated by looking at the relative peak heights of the different regions. The relative heights of the 485, 575, and 650 region peaks are shown in Table 3 along with the 575/485 ratio for each of the submitted spectra. There is an appreciable variation among the participants, the ratio varying from 0.6 to 3.93.

Transmission variations are normally associated with the optical system/detector components, but it should be noted that Steele (personal communication, 1994) has stated that relative peak intensities can also be a strong function of current density and refers to unpublished data for olivine in which 3 peaks are seen at lower currents and only 2 at higher currents.

Intensity and signal-to-noise ratio

Absolute intensities are not directly comparable from the information supplied, but an indication of the signal-to-noise ratio can be obtained from the raw spectra. Usually, a poor signal-to-noise ratio is indicative of low signal intensities.

Stability of CL intensity with time

Laboratory C reported a rapid decrease in intensity within 100 milliseconds. The intensity then became stable. No other laboratories commented on this aspect of performance.

Other

In addition to the two major bands, all laboratories

Table 3. Relative intensities for 485, 575, and 660 nm regions.

Lab	Detector	485	575	660	575/485
A	PMT	1	0.9	0.02	0.9
B	ARRAY	1	0.6	0.01	0.6
C	ARRAY	0.7	1	0.04	1.4
D	PMT	0.9	1	0.04	1.1
E[1]	PMT	0.9	1	0.02	1.04
E[2]	PMT	0.8	1	0.04	1.25
F	ARRAY	1	0.9	0.03	0.9
G[1]	PMT	1	0.76	0.03	0.76
G[2]	PMT	0.66	1	0.04	1.5
H	ARRAY	0.25	1	*	3.93
I	PMT	0.89	1	0.05	1.12
J	PMT	0.72	1	0.04	1.38
K	PMT	0.72	1	0.03	1.38

*Please see text. This region was not recorded.
[1] raw data; [2] corrected data.

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except Laboratory H reported an additional small peak in the 650-670 nm range. Laboratory H did not report this peak but they did not collect data above 625 nm.

In many natural samples, Sm^{3+} would also be present and one of its peaks would obscure the smaller 650 nm peak from dysprosium. But Mariano (1988; Fig. 8.11) shows a spectrum of fluorite with dysprosium but without samarium, which exhibits a peak at 660 nm.

A few laboratories reported a small peak in the 540 nm region. The strongest peak for terbium is at about 545 nm (Mariano, 1988) and initially there was some concern that terbium must be suspected as an impurity, present in very small amounts. However, Hanchar subsequently demonstrated that this small peak is a peak in the Dy^{3+} spectrum.

Three laboratories reported a small peak in the 750-760 nm region.

The orientation of the zircon crystal with respect to the impinging electron beam and with respect to the axis along which the CL emission was collected was not specified. Cesbron *et al.* (1996) state that the CL emission of zircon shows orientation effects, so this parameter should be controlled in any future study. It is likely that those crystals which were loosely mounted (i.e., simply laid on the sample holder without epoxy) would tend to have their c-axis in the horizontal plane so that most of the participants may have had (inadvertently) similar geometries.
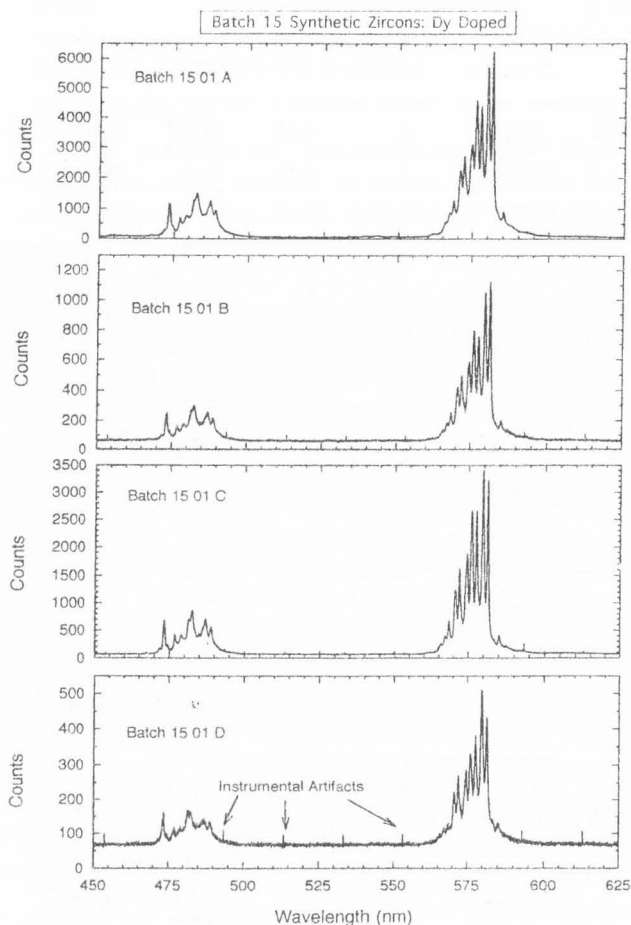
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Figure 3. Spectra recorded by Laboratory H on 4 different zircon crystals, demonstrating the close similarity of the spectra. Note that the actual intensities for A and D vary by a factor of 12. Operating conditions are as given in the spectra from Laboratory H.

Additional Observations

After the samples were distributed, there was an opportunity to obtain spectra from several crystals from the original batch under nearly identical conditions. The spectra from four randomly selected crystals were obtained on one CL system (Laboratory H) and are reproduced as Figures 3, 4, and 5. The agreement of peak wavelengths among these four crystals is excellent (within ± 0.04 nm for the 579.8 nm peak) even though the absolute intensities for these four crystals varied by more than a factor of 10. This strongly supports the assumption that the crystals that were circulated should have similar luminescence spectra (i.e., peak positions should be similar).
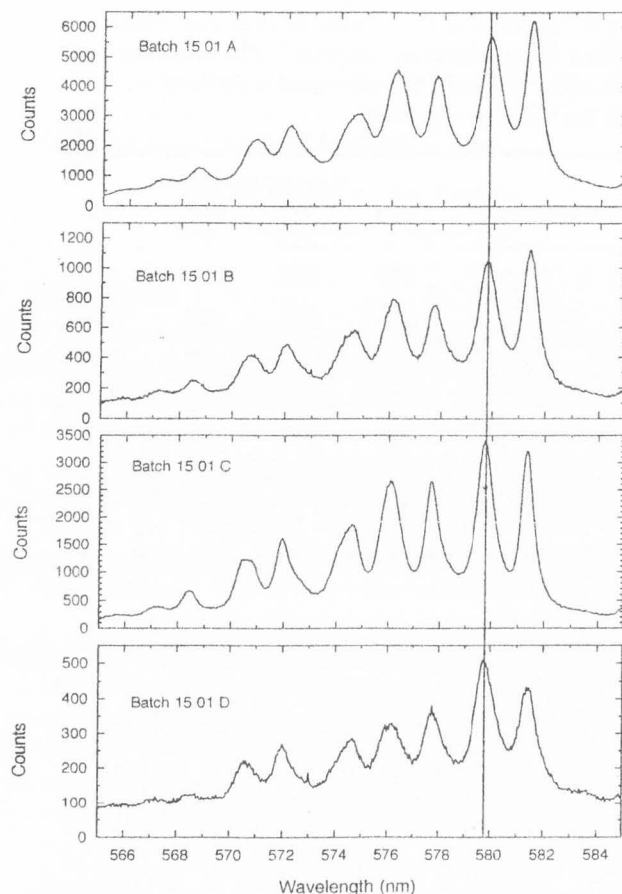

Figure 4. Horizontal expansion of the spectrum in the 580 nm region for the 4 samples shown in Figure 3. The position of the 579.8 nm peak is reproducible to ± 0.04 nm for these 4 samples.

Representative spectra from various laboratories have been published in Marshall (1994); a copy is available through the Society for Luminescence Microscopy and Spectroscopy (see Marshall, 1994 for address).

Recommendations For Future Studies

In future studies, participants should be asked to furnish details about the wavelength calibration procedure used and to furnish a spectrum of the light source to show both the wavelength calibration and also the peak shape which they obtain for a narrow source (such as the Hg lines in the usual light source represent).

All laboratories should be asked to provide both a spectrum at best operating conditions and also a spectrum at reduced resolving power, for comparison of peak location and relative peak intensities under low resolving power conditions.

Appendix 1. Participant Names, Laboratory Designations and System Descriptions

(A) Stefano Salvi, Department of Earth and Planetary Sciences, McGill University, 3450 University Street, Montreal, QC H3A 2A7, Canada.

CL Instrument: Model ELM-3R Luminoscope®
Microscope: Trinocular with 4X objective
Coupling method: Photo eyepiece with fiber optics cable
Spectral analyzer: Spectrometer
Detector: PMT (R928) at room temperature

(B) Andrew Locock, Department of Geology, 1-26 Earth Sciences, University of Alberta, Edmonton, AB T6G 2E3, Canada.

CL Instrument: ELM-3R Luminoscope®
Microscope: Trinocular
Coupling method: Photo eyepiece with fiber optics cable
Spectral analyzer: Spectrograph
Detector: Intensified diode array at +10°C

(C) Dr. Ian Steele, Department of Geophysics, University of Chicago, 5734 Ellis Avenue, Chicago, IL 60637 USA.

CL Instrument: Electron Microprobe
Microscope: Standard probe optics
Coupling method: Quartz fiber optics cable
Spectral analyzer:
Detector: Intensified diode array at -10°C.

(D) Dr. Anthony Mariano, Consultant, 48 Page Brook Road, Carlisle, MA 01741 USA

CL Instrument: ELM-3R/ELM-2E Luminoscope®
Microscope: Monocular with 2.9X objective, 0.25 NA
Coupling method: Photo eyepiece with fiber optics cable
Spectral analyzer: Spectrometer
Detector: PMT (R928) at room temperature

(E) Dr. Roger Mason, Department of Earth Sciences, Memorial University, St. Johns, NF A1B 3X5, Canada

CL Instrument: Electron microprobe
Microscope:
Coupling method: Fiber optics from microprobe
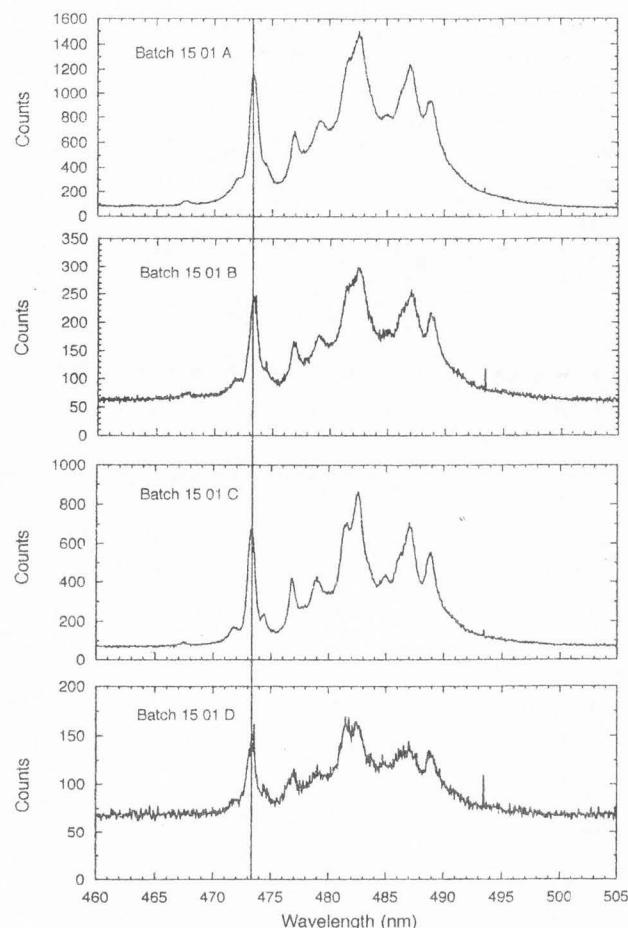Spectral analyzer: Spectrometer
Detector: PMT

Figure 5. Horizontal expansion of the spectrum in the 485 nm region for the 4 samples shown in Figure 3.

(F) Professor Michael Owen, Department of Geology, St. Lawrence University, Canton, NY 13617, USA

CL Instrument: ELM-2 Luminoscope®
Microscope: Binocular with 20X objective
Coupling method: Direct projection onto spectrograph input
Spectral analyzer: Spectrograph
Detector: Intensified diode array with thermoelectric cooling

(G) Professor P.D. Townsend, School of Mathematical and Physical Sciences, University of Sussex, Brighton BN1 9QH, United Kingdom.

CL Instrument: Please see Yang *et al.* (1992).
Microscope: none
Coupling method:
Spectral analyzer: Spectrometer
Detector: PMT

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(H) **Dr. John Hanchar**, Department of Earth and Environmental Sciences, Rensselaer Polytechnic Institute, Troy, NY 12180-3590, USA.

CL Instrument:	ELM-3R Luminoscope®
Microscope:	Olympus BH binocular with 10X objective
Coupling method:	Direct projection onto entrance slit.
Spectral analyzer:	Spectrograph
Detector:	Unintensified CCD at -36°C

(I) **Dr. Karl Ramseyer**, Geologisches Institut, University of Bern, Balzerstr. 1, CH-3012 Bern, Switzerland.

CL Instrument:	Hot cathode (Please see Ramseyer *et al.*, 1989)
Microscope:	Zeiss with 6.3X objective
Coupling method:	Fiber optic bundle
Spectral analyzer:	Spectrometer
Detector:	PMT (R928) at room temperature

(J) **Dr. Steve Kissin**, Department of Geology, Lakehead University, Thunder Bay, ON P7B 5E1, Canada

CL Instrument:	ELM-2 Luminoscope®
Microscope:	Leitz Laborlux® 12 POL with 25X objective
Coupling method:	Fiber optics bundle
Spectral analyzer:	Spectrometer
Detector:	PMT (R928) at room temperature

(K) **Dr. A. M van den Kerkhof**, Institut fur Geologie und Dynamik der Lithosphare, Goldschmidtstrasse 3, D-37077 Gottingen, Germany.

CL Instrument:	Hot Cathode, Zinkernagel design (Zinkernagel, 1978)
Microscope:	Leitz Ortholux® with 20X objective
Coupling method:	Fiber cable
Spectral analyzer:	Spectrometer
Detector:	PMT (S5) at room temperature

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