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GEOLOGICAL SURVEY

Notes on Cathodoluminescence Microscopy using the Technosyn Stage,  
and a Bibliography of Applied Cathodoluminescence

by

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# NOTES ON CATHODOLUMINESCENCE MICROSCOPY USING THE TECHNOSYN STAGE AND A BIBLIOGRAPHY OF APPLIED CATHODOLUMINESCENCE

by Charles E. Barker and Teresa Wood

## INTRODUCTION

Cathodoluminescence (CL) microscopy has become an essential tool in the petrographic description of sedimentary rocks. CL also has important applications in igneous-metamorphic petrography, ore deposits, and mineralogy. In this brief treatment, the primary goal is to summarize our techniques in CL microscopy because of requests from other users. This report also summarizes our experience in the operation of the Technosyn<sup>1</sup> CL stage. The Technosyn stage is used here as an example of a CL system for descriptive purposes only. The other commercial CL stage we are aware of is made by Nuclide Corporation.

## Cathodoluminescence in Minerals

In the vacuum chamber of the CL stage, the electron beam is absorbed by the surface it hits. Much of the energy of the incident electron beam is absorbed by the specimen molecules causing an increase in the energy levels of absorbing atoms. Normally the excited atoms (also termed cathodoluminescent centers) return to the ground state by transfer of the excess energy to adjacent atoms by inelastic collisions. Under certain circumstances, the absorbed energy is re-emitted as light energy in the visible range before these collisions can take place. The conditions for luminescence often occur in impure crystalline substances where the impurities act as the luminescent centers. The intensity of light emitting from any particular point will be proportional primarily to the surface density of luminescent centers. The electron energy is readily absorbed in the sample, and little luminescence is emitted from below the surface. Transition metals and the rare earth elements are particularly susceptible to electron beam excitation. For instance, in transition metals, the 3d electron shells are available for excited electrons to enter these levels.

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<sup>1</sup> The use of trade names in this report is for descriptive purposes only and does not constitute endorsement by the U.S. Geological Survey, or S and M Microscopes, Incorporated. The complete address of the manufacturers cited in this report are listed in appendix 1.

Electrons in this excited state are unstable, and they descend to lower energy shells by radiating visible light. Manganese is a common activator in minerals and rocks. Iron is a common quencher. This is largely due to the abundance of these trace elements in minerals (Sommers, 1972). Luminescent centers are also caused by radiation damage, and sometimes still unidentified sources. We refer the interested reader to Nickel (1979) and Amieux (1982) for a detailed discussion of CL phenomena, petrography, and a list of CL colors in minerals.

### X-ray radiation

CL stages use an electron beam to produce cathodoluminescence in susceptible materials. The Technosyn stage generates an electron beam rated at 25 kV at 250 microamps. This electron beam is capable of producing X-rays from any material it strikes. The stage is designed to absorb this radiation, but periodic inspections are important to assure leaks have not developed. Nuclide Corporation makes a compact radiation detector suitable for testing the CL stage while mounted on the microscope.

Tests of X-ray production from the Technosyn CL stage indicate that it is safe. This test involved placing a nickel target under an electron beam in the stage that optimizes the production of X-rays. The stage was operated between 20-30 kV and 400-100 microamps respectively. X-ray production from silicate, carbonate, or glass targets would be less under the same stage operating conditions. X-ray production was measured using a Philips monitor type PW 4517 placed directly above the top window. The stage windows were the only place X-rays were detectable during these tests. With the stage mounted on a microscope, a maximum X-ray level of 0.1 mRad/hour was measured 5 cm above the top window.

Safety regulations specify that anyone without special training or regular medical checks should not be exposed to more than 0.5 Rad/year above background radiation level (normally about 0.5 Rad/year). This radiation level divided into 2000 working hours indicates the maximum level is 0.25 mRad/hour. None of the measured values exceed this value, and the unit is safe under these standards.

Any alteration of the CL stage can potentially alter its X-ray production levels. After modification of the stage, it should be checked for excessive radiation levels.

### SAMPLE PREPARATION

Thin sections, rock slabs, and loose grains can all be examined in the CL stage. Fine grains should be cemented to a glass slide so they will not enter the vacuum system. The Technosyn stage will accommodate thin samples of maximum dimension 70 x 80 x 1 mm. Thick samples are restricted to 50 x 70 x 17 mm. The view area in both cases is 50 x 70 mm.

Optimal details under CL are produced by using high quality polished surfaces. Although polishing is not absolutely necessary, we recommend it for most microscopy, especially if photomicrography is anticipated. Methods of polishing rock specimens are listed in the bibliography.

Thin sections for CL microscopy should use a cement that is stable at the moderate temperatures generated in the sample. Heating and outgassing from the cement can coat the stage window and delay vacuum stabilization and extend pumpdown time. Epoxide or epoxy resins (Buehler, Ltd. or Hillquist) are satisfactory. Cover glass should not be used because the electron beam will be absorbed by it before reaching the sample. A conductive coat on the sample is not necessary.

### Cleaning the Sample

After sample preparation, lubricants, water, fingerprint oils, and other volatiles must be removed from the sample. These volatiles will vaporize in the chamber during pumpdown or CL observation, extending pumpdown time to operational vacuum levels, and also potentially depositing material on the chamber window. An effective cleaner and dehydrator for samples is a freon solvent (Miller-Stephenson brand MS-180).

### REQUIREMENTS FOR THE MICROSCOPE

Light intensity transmitted to the operator during luminescence observations is proportional in part to the degree of magnification. The lower power objectives transmit a brighter image. The lowest power objective possible should be used as long as the feature of interest is seen in sufficient detail. The point is that the fewer optical elements in the microscope light path, the brighter the image. This is one case where a more complex, optically sophisticated microscope may degrade the CL image rather than improve it.

### Objective Lens

The objective lens must have a minimum free working distance of 9 mm. Selected objective lenses with the required working distance are listed in Table 1. Other objective lenses with appropriate free working distances are listed in Dally and Wills (1985). The objective lens should be cover slip corrected in those above 10X power--otherwise optical aberrations can occur. The electron beam forms a 4 mm by 7 mm ellipse of luminescence on the sample surface, and a very low power objective lens may view a spot greater than that illuminated by CL.

A simple objective lens set for CL observations would be a 5x and a 10x. Higher power lenses may be useful in specific cases but generally have poor CL characteristics and transmit inferior images.

Table 1

Objective Lenses for Cathodoluminescence Microscopy  
(As used by the U.S. Geological Survey)

<u>Source</u>	<u>Lens</u> <sup>1</sup>	<u>Power(n.a.<sup>2</sup>) fwd</u> <sup>3</sup>	<u>Notes</u>
Lietz	EF4	4X(0.12) 24.0mm	Inexpensive
Lietz	L25	25x(0.35) 13.5mm	Long fwd lens
Nikon	CF 4	4x(0.10) 20.0mm	Inexpensive
Nikon	M Plan 5	5X(0.10) 20.0mm	210mm tube length <sup>4</sup>
Nikon	M Plan 10	10X(0.25) 9.0mm	Long fwd lens
Ziess	UD16	10X(0.11) 13.5mm	Universal-stage lens <sup>5</sup>

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Notes: (1) Manufacturers identification code engraved on lens. (2) n.a. = numerical aperture. (3) fwd = free working distance. (4) All Nikon Mplan lens are designed for microscopes with a mechanical tube length of 210mm and their use on 160 mm tube length microscopes reduces the rated magnification power and n.a.. (5) A universal-stage lens, designed for use with glass hemispheres. These lenses have a poor image, lower than rated magnification and n.a., when used without the spheres (effective magnification and n.a. shown). Any low power universal stage objective lens usually can be used but most lack the high n.a. necessary for good CL observation.

## Oculars

The same limitations on image brightness apply to oculars as discussed in the objective section. The optimal oculars are those with the lowest power that allow adequate resolution of details in the sample. For instance, 8X Nikon oculars are 56 percent brighter than the Nikon 10X. Remember that the effective resolution of the system is determined by the numerical aperture of the objective lens, and trying to increase the magnification beyond this value provides a larger image but poor resolution. Low power objective lens tend to have small numerical apertures and low effective magnification levels, such that using high power oculars is not recommended.

## Substage Condenser

A long working distance substage condenser is essential to provide an adequate image quality in the CL stage during observation and photomicrography in transmitted light. The substage condenser will not improve the CL image or photographs because the image is formed by light emitted from the upper surface of the sample. The free working distance of the condenser should be sufficient to focus a condensed beam of light on the upper plane of the sample. The longer working distance required by the Technosyn stage, only allows the closed condenser diaphragm image in the plane of the sample to a near-sharp focus. This image should be sharply focused for Kohler illumination.

## Polarizer and Analyzer

The analyzer (or any accessory plate) absorbs light and reduces the image brightness. It must be removed for CL observations. The polarizer, as it is placed below the sample, will not effect the CL image.

## Stage Clearance

The minimum clearance necessary to fit the stage between the objectives and microscope is 5.0 cm. Microscopes that have sufficient clearance are: Nikon Labophot or Optophot; Zeiss Universal; Zeiss WL, and some Lietz Labrolux. The Leitz Labrolux models 11, 12, and D require modification to allow the stage to be mounted. Microscopes without the necessary clearance is the Ziess model 16 and model 18.

It is also possible to put the CL stage on stereo microscopes, but these must be used in a completely darkened room. Stereo microscope objectives have a very long free working distance, and a low numerical aperture, allowing room-light to enter the system which degrades the image quality.

## Microscope Room

The area in which the microscope is used must be enclosed such that all light can be eliminated. Cathodoluminescence in certain samples is extremely dim and a darkened room allows the observer's eyes to adjust to lower light levels. A small light should be provided to allow ease of movement and allow writing notes.

A CL control box, cables, and the microscope will fit on a standard office desk or table. The vacuum pump should be set on the floor so that vibrations transmitted to the microscope are minimized.

CL systems require 10 amp, 110 volt outlet (in the United States). If the system is operated near equipment that draws a lot of electricity, the resultant power surges when this equipment is operated may damage samples during CL observations. A line voltage stabilizer may be required to correct this problem.

### Photomicrography

Photomicrography is an essential but difficult aspect of CL microscopy. Experience with a particular sample suite is perhaps the best guide. A good starting point is the discussion in Nickel (1979) or Mariano (1983). Since those publications, new films have been introduced that offer higher speed without loss of resolution. We have found these films to be useful:

- 1) Kodak Ektachrome P800/1600, EES 135 (for color slides)
- 2) Kodak Kodacolor 400 (for color prints)
- 3) Kodak 5294 or equivalent (for both color negatives and slides)

Color processing can distort the true color as perceived by the microscopist. We have found it useful to send to the processing laboratory a photograph that has the color qualities that are close to the actual CL view. This photograph allows the color analyzer to be set properly and realistic color reproduction.

### OPERATING THE TECHNOSYN STAGE

System pressure and power supply for the electron gun are set from the front of the Control Unit. The POWER switch controls electrical power to the control unit. VAC PUMP switch controls vacuum pump operation if the pump power line has been wired into the back of the control unit (otherwise it can be manually switched at the pump). INTERLOCK light is only lit when the interlock circuit is complete (jumper wire between pins 3 and 5 of EXT socket on back of control unit). VAC READY light lights up when system pressure is lower than 0.2 Torr. Both the INTERLOCK and VAC READY lights must be on for the control unit to allow the electron gun to turn on. kV ON (or EHT ON for some machines) switch turns on the electron gun. The LED on the kV ON switch is lit when the electron beam is on. GUN CURRENT meter indicates the gun current in microamps. When the gun is not switched on the gun current meter should be within  $\pm 9$  microamps of zero. The VAC CONTROL is a vacuum bleed valve that controls the amount of air leaking into the system, and therefore the pressure inside the stage chamber. The VAC CONTROL should be gently turned fully to the right (clockwise) before each pumpdown. ADJUST kV controls the level of drive to the extra high tension supply. The actual level of high voltage in the electron gun, indicated by the kV METER, is determined by the vacuum present: (1) low vacuum (high pressure) produces a low kV METER reading; and (2) high vacuum (low pressure) produces a high kV METER reading. The level indicated by the kV METER should not be left above 30 kV as this shortens the life of both the electron gun and gun power supply cable. MONITOR (test) meter indicates the vacuum level at the control unit in Torr (or in test mode, it is a voltmeter). The stage chamber vacuum slightly lower than the meter reading because it is



further down the pumping line. Normal operation in air is achieved at meter readings of 0.07 to 0.04 Torr. Vacuum levels higher than 0.04 Torr will cause the electron gun beam to extinguish. Vacuum levels lower than 0.07 Torr enable the system only to operate at low levels around 3-5 kV. The MONITOR meter also acts as a voltmeter to check the control unit electronics in conjunction with the SELECTOR switch located near the lower left hand corner of the front of the control unit (see troubleshooting section).

### Striking an Electron Beam and Controlling the System

- (0) The sample should be observed through the microscope. The analyzer and any compensators, shutters, etc., should not interfere with the optical path. Center a well polished portion of the sample which optimally should contain brightly luminescent material, such as calcite. This aids the initial observation of CL and further adjustment of the system.
- (1) After plugging the control unit in, turn the switch on the front of Unit marked "POWER" on (red light on). INTERLOCK LED lights up with power on. Turn the VAC PUMP switch on (red light on). Rotary vacuum pump comes on. Sometimes it is necessary to hold the stage cover in place and press the stage drawer closed when the vacuum pump is turned on.
- (2) When vacuum in chamber is sufficiently low for the electron beam to strike and hold, the green "VAC READY" light will come on. This occurs at a chamber pressure of 0.08 and 0.05 Torr (see MONITOR meter). The first daily pumpdown time varies in the range of 5 to 10 minutes. After this time if the VAC READY light does not come on proceed to troubleshooting section.
- (3) At this chamber pressure the kV ON (or EHT ON on some machines) switch is turned on. An additional 5 minutes or so of pumpdown is necessary to allow the specimen to complete outgassing from heating by the electron beam. Soon the vacuum will improve to the point that the electron beam will strike the sample and hold (kV ON switch LED comes on). This process is optimized by holding the beam on the sample surface rather than the glass slide. The beam tends to stabilize quicker when impinging on the sample. If the electron gun does not come on proceed to the troubleshooting section.
- (4) USE OF THE "VAC CONTROL" knob: At any operating voltage the gun current can be adjusted by using the vacuum bleed control valve (labeled VAC CONTROL) to vary the pressure in the stage. There is a minimum current below which the gun is unstable, generally 150 microamps. The vacuum in the chamber must be controlled to stabilize the beam current. This is done by turning the VAC CONTROL knob counterclockwise until the beam strikes. Under these conditions, rotation of the VAC CONTROL knob in either direction produces a change in the gun current. Counterclockwise rotation produces an increase in GUN CURRENT meter (the digital microamp meter) and a decrease in beam voltage (kV METER). Conversely, clockwise rotation will produce a decrease in gun current and an increase in gun voltage. The system is

quite sensitive at this VAC CONTROL setting. Further adjustment should be done carefully in small increments. The gun current should not exceed 600 microamps to avoid damage to resistors in the control unit. Never overtighten the VAC CONTROL -- finger tight is always sufficient.

- (5) USE OF THE "ADJUST kV" CONTROL: Initially the ADJUST kV control should be turned clockwise to the far right. Under normal operating conditions, the ADJUST kV control remains in that position.

In some cases, further adjustment of the ADJUST kV control may be desirable. After the electron beam strikes and holds, the kV METER will show the beam voltage. The electron gun voltage and current are interdependent on the operating vacuum. Once the beam has stabilized the gun current is controlled by adjusting the VAC CONTROL knob. The current is reduced by turning the VAC CONTROL knob counterclockwise. The kV METER indicates the accelerating potential in the electron gun. The ADJUST kV knob may be turned counterclockwise until the kV METER responds. In this way the uppermost kV limit may be set. However, the kV is not controlled directly by the ADJUST kV knob -- only indirectly through VAC CONTROL adjustments. We have found this adjustment to be more useful at lower settings in the 10-12 kV range rather than at higher kV levels.

#### Centering the CL Image in Optical Path

The area on the sample struck by the electron beam will appear as an elliptical spot of light approximately at the center of the window. For proper microscope observations this light spot should be centered until it fills the field of view in the oculars. This adjustment becomes critical at 5 power objectives or lower. This stage is physically centered using three 3 mm allen-head screws on the underside of the vacuum chamber. These screws should be loosened such that the chamber may be moved about by hand into the correct position. All three screws are then lightly tightened without moving the stage, and finally retightened with the allen wrench. These adjustments must be done with the beam on. The field of view will be completely filled when using a 5X objective. Lower power objectives will have a field of view that is not completely filled by the electron beam.

#### OPERATION SUMMARY

##### Initial Pumpdown

- (1) Place specimen or prepared microscope slide in stage using top cover or drawer. VAC CONTROL and ADJUST kV controls are turned fully to the right.
- (2) Close both the air admit valve on chamber and VAC CONTROL (a controlled leak valve) on Control Unit.
- (3) POWER switch on -- red power light on, INTERLOCK LED on.
- (4) VAC PUMP switch on -- red vac pump light on, rotary pump on.  
When vacuum is low enough -- green LED "VAC READY" light on.
- (5) kV ON switch on -- red LED on.
- (6) Adjust the gun current by turning the VAC CONTROL knob -- kV METER

indicates kV.

### Changing Samples

- (1) Turn POWER switch off.
- (2) Turn VAC PUMP switch off.
- (3) Open air admit valve on chamber, let it bleed to atmospheric pressure.
- (4) Open chamber using top cover or drawer, exchange specimens.
- (5) Repeat SAMPLE RUN instructions.

### System Shut-Down

- (1) Follow CHANGING SAMPLES instructions, take specimen out, omit step five.

## TROUBLE SHOOTING

### Vacuum Leaks

Vacuum leak detection can be difficult because even a small leak can reduce system vacuum below operating levels. Vacuum leaks are usually indicated by dim CL or the electron beam cannot be struck and stabilized.

First order leak elimination procedure. Tighten all hose clamps until firm. Wipe the O-rings clean -- make sure dust and oils removed. Coat O-rings lightly with Apiezon-L (Biddle Instruments) vacuum grease. System may respond and pump down to useful range. The majority of vacuum leak problems will be solved with this method.

Second order leak elimination procedure. This level of leak detection involves isolation of system components and checking the system response to identify the leaking subsystem. For instance, chamber leaks can be detected by removing the system to chamber vacuum hose, plugging it, and checking to see if this improves the system vacuum to a usable range.

Third order leak elimination procedure. If helium gas is available it can be used to test for small leaks in the vacuum system. Helium under low pressure from a lecture-type bottle is projected towards potential leak locations through a rubber hose with a Pasteur pipette inserted at the tip. Any vacuum leaks will be noted by a rapid decrease in system pressure when helium is directed to the location on the stage or vacuum hoses.

Electron Gun Leaks. If the beam voltage does not increase above 10 kv after 5-10 minutes, this may indicate a leak in the electron gun. Unscrew the electron gun being careful not to scratch the barrel. We wrap the gun in cloth or tape to prevent marring by the pliers. Considerable force may be necessary to start unscrewing the gun counterclockwise. When unscrewing the gun be sure not to loose the metal nipple (this is the anode) located at the base of the metal tube. The O-ring on the glass tube should be cleaned and greased with a generous amount of Apiezon-L (for moderate temperatures) or Apiezon-T (for high temperatures). A generous amount of grease because these tubes are often loosely fit into the gun. During reassembly, the position of the O-ring on the

glass tube is not critical, however, the narrow end of the metal nipple should be pointed towards the glass tube. Reassemble the electron gun. This procedure may not correct the vacuum problem in systems that have had extensive use. In this case, the gun may need to be replaced.

### High Voltage Problems

No High Voltage. First, check interlock LED. If not on, is shorting plug inserted in EXT socket on back panel of control unit? Second, check kV ON switch, it must be on. Third, check the MONITOR meter, the vacuum must be at least 0.07 Torr for the electron gun to operate, and it may be necessary to find and repair an air leak.

If there is still no electron beam, turn off the control unit with the POWER switch. Remove the high voltage cable from the back of the control unit. Turn POWER switch on, and pumpdown the vacuum system to operating levels (0.04-0.07 Torr) and switch on kV ON. Turn the ADJUST kV knob from fully right to fully left. The MONITOR meter should move continuously from 30 kV to about 5 kV. If this does not occur, test the control unit electronics, as described below.

Unstable high voltage. Unstable high voltage in the electron gun are usually caused by a dirty electron gun, chamber or small vacuum leaks in the area around the gun. Cleaning the chamber is discussed in the maintenance section. Check for vacuum leaks using the procedures outlined above. A dirty electron gun is cleaned by disassembling the gun as described above. Remove the glass tube from the brass housing that forms the electron gun. The inside of the brass tube can be cleaned using a fine-grit silicon-carbide sandpaper or a diamond polishing compound. The gun normally has coating from being burned in at the factory. It is not necessary or desirable to remove all of this coating. In any case, the gun should be only lightly cleaned.

### Control Unit Electronics Test

The SELECTOR switch changes the function of the MONITOR meter to a voltmeter. In normal operation the selector switch is turned fully to the right and the meter reads the system vacuum. The top LED next to VAC is then illuminated indicating vacuum gauge power supply is present. When a system check is required, use a small screwdriver to rotate the SELECTOR switch to the left through each position. The illuminated LED indicates which test is selected. If any of these readings are not normal (Table 2), call the Technosyn dealer for advice.

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Table 2

Control Unit Electronics Test

<u>POSITION</u>	<u>TEST</u>	<u>NORMAL RESULT ON MONITOR METER</u>
VAC	Normal operation	Vacuum level reading <sup>1</sup>
A+	Analogue + supply	12 volts +/- 1 volt
A-	Analogue - supply	12 volts +/- 1 volt
D	Digital supply	5 volts +/- 0.5 volt
EHT	Gun supply	21 volts +/- 2 volts

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<sup>1</sup> read vacuum level with vacuum pump on, VAC CONTROL valve fully closed to the right, and stage chamber closed.  
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Stage Maintenance

Maintaining a CL system routinely requires: (1) the gaskets be kept clean and lightly lubricated with vacuum grease; (2) volatile substances be removed from the chamber surface (fingerprints, vaporized plastics, etc.); (3) chamber window cleaned.

All cleaning that uses volatile substances should be done the night before to allow evaporation from the stage surfaces. After cleaning the CL stage will probably take longer than normal to stabilize. We usually run the stage for 5 minutes without a sample inside to allow the volatiles to vaporize.

Stage maintenance also requires that the stage be run daily to keep it in top operating condition. The daily routine is to operate the stage for five minutes with a sample in it. After a long period of inactivity, it is best to put in a sample and turn on the unit the day before actual microscopy, to minimize initial pumpdown time or indicate if the stage needs cleaning to pumpdown adequately.

Cleaning O-rings and Chamber. Remove O-rings from chamber cover. Clean the gaskets and mounts with a dry cloth and (or) cotton swab. Lightly lubricate the O-ring with Apiezon-L vacuum grease (Biddle Instruments). Routinely clean the top cover of the chamber and gun port with metal polish to remove condensed material. Rinse with freon solvent (Miller-Stephenson brand MS-180). Replace O-ring on chamber cover.

Freon-solvent-impregnated lint-free pads (Miller-Stephenson) are also useful for degreasing chamber surfaces.

Cleaning the Chamber Window. After each day's use, the chamber window should be cleaned of the material that condenses on its surface with ethyl alcohol. Alumina oxide polishing compound 0.3 micron (Buehler) in a water slurry, applied with a cloth and rubbed vigorously but lightly, does an adequate job of cleaning very dirty windows. A chrome polishing paste is also satisfactory.

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## APPENDIX 1

### Manufacturers Addresses

- Biddle Instruments, 510 Township Line Rd., Blue Bell, PA 19422. (215) 646-9200
- Buehler Limited, 41 N. Waukegan Rd., Lake Bluff, IL, 60044. (312) 295-6500
- Hillquist, 1545 northwest 49th St., Seattle, WA 98107. (206) 784-1960
- Eastman Kodak Company, Rochester, NY 14650. (800) 225-5352
- E. Lietz, Incorporated, Rockleigh, NJ 07647. (201) 767-1100
- Nikon, Instrument Division, 623 Stewart Avenue, Garden City, NY 11530  
(516) 222-0200
- Nuclide Corporation, AGV Division, 916 Main St., Acton, MA 01720. (617) 263-2936
- Miller-Stephenson Chemical Company, Incorporated, George Washington Highway,  
Danbury, CO 06810. (203) 743-4447
- Technosyn Limited, Coldhams, Cambridge CB1-3EW, England
- Carl Zeiss, Incorporated, One Zeiss Dr., Thornwood, NY 10594. (914) 747-1800

## PART II

### BIBLIOGRAPHY OF APPLIED CATHODOLUMINESCENCE

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