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### EDITORIAL

My father taught me as a young boy how to skip stones on Lake Michigan while we walked along the shore. This eventually led to collecting pretty stones and sand abraded colored glass fragments washed onto the beach. A few years later on one Christmas Eve during the 1950's, a Porter Mineralogy Set appeared under the Christmas tree. Included in the blue wooden box were mineral specimens, a blowpipe, charcoal blocks, and a platinum wire loop for flame and borax bead tests. In less than a year, I realized that collecting 92 chemical elements would be far easier than a couple thousand minerals. A basement lab at home and an egg house lab on my aunt and uncle's farm in Michigan served me well.

My beginning interest in chemistry led to dyes, drugs, perfumes, polymers, an initial graduate thesis project in molecular beams, and then radiation induced defects in crystals. Again rock bottom!

A college chemistry teaching career followed which has permitted me to indoctrinate my students with my own concept of *Ethics of the Dust* (by John Ruskin). Then one day I invited Dr. McCrone to speak at one of our college's science lectures: Slides of the Vinland Map, the Shroud, and photomicrographs of fibers and mineral fragments between crossed polars blazed on the screen, followed by an empty feeling that I had been somehow cheated in my education.

One letter (returned critiqued!) and a phone call enrolled me in the beginning PLM course at McRI.

Once again a student, I am now caught in an ecstatic wave which pushes me gently to new shores of knowledge.

The contributors to this issue of  $\mu$ -Notes 2000 have walked along these shores. They have been taught how to skip stones and teach others to do so. In several articles you will discover more about the man who made this possible for so many: Dr. Walter C. McCrone, microscopist, mentor, teacher, and SMSI's Émile M. Chamot Award recipient for 1998.

Bill C. Mikuska

# $\mu \bullet DOTES 2000$

Volume 2, No. 2, August 1998

Bill Mikuska Editor

Dorothy Mikuska Managing Editor

 $\mu \bullet$  NOTES 2000 is a State Microscopical Society of Illinois publication. Its purpose is to provide a form of communication between amateur and professional microscopists, to share ideas and techniques, to ask questions, to obtain answers, to express opinions, and to publish results of experiments and research. It will also provide space for members to print wanted and for-sale notices of microscopical equipment.

All opinions expressed by contributing authors of  $\mu$ • NOTES 2000 are the responsibility of the author(s) and do not necessarily reflect the opinion of the State Microscopical Society of Illinois or that of the editor.

Contributions, typed and/or on computer disk, should be addressed to EDITOR,  $\mu \bullet \text{NOTES } 2000, 2820 \text{ S.}$ Michigan Ave., Chicago, IL. 60616. Telephone: 312.842.7100.



**Cover**: *Microscopical Discoveries: Crystallizations*–A portion of a hand colored page from an 18th century encyclopedia from the editor's collection.

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Volume 2, Number 2August 1998

### **INTER / MICRO-48**

### Walter C. McCrone\*

In August, 1944, I finally left Cornell after 10 very important years learning microscopy from Chamot and Mason. I was lucky to be able to leave with a Ph.D. even though I ignored my major Professor Jack Johnson's assigned thesis topic (synthesis of 20 derivatives of cis-endomethylene-3,6- $\Delta$ tetrahydrophthalic acid anhydride) and shocked him by handing him a rough copy of my thesis entitled *Application of the Polarized Light Microscope in Organic Chemistry*. As Professor of Organic Chemistry, he became my major professor because Professor Mason had told me a Ph.D. in microscopy was not possible since there was not any more research to be done in microscopy.

It is barely possible I got my Ph.D. because the Chemistry Department wanted to get rid of me. I had formed a union to address the needs of chemistry teaching assistants and managed to get the annual stipend increased 40%, effective the semester after I left. I also had a tutoring school where I lectured to chemistry students in evening lectures just before midterms and finals telling them what questions to expect. I was right on 2/3 of each of the exams because I had a file of past exams in most of the courses and the professors simply repeated their exams every 2-3 years; I often had the professor's complete class at my lectures at 10¢ a head. I also managed to procure a master key to all the doors in Baker Laboratory. I hasten to add I honestly never used it nefariously; but it was very useful to have access to the roof with its fantastic view of Cayuga Lake, Beebe Lake, the gorges, and the campus. We, the hard-working grad students, had lunches, bull sessions, and even beer parties up there. I could also get in and out of the building from the Beebe Lake side for my daily swims. The other grad students were glad to be able to borrow the key because I was there evenings and weekends. I hated to leave Cornell.

When I did, it was to start work on September 1, 1944, at IIT's contract research arm, Armour Research Foundation, ARF, now IITRI. I arrived at 35 West 33rd Street, the long-since demolished Chemistry building, at 8:00 A.M. and found the door locked. I knocked and a janitor finally opened the door and asked what I wanted. I said, "I'm supposed to start work here today." He said, "It's Labor Day and there's no one here." That's the first time I ever realized that some people don't work on holidays. I always have and still do. I got a perverse delight out of heading my research notebook pages with January 1, 19xx, December 25, 19xx, etc. For example, I started my study of the Shroud tapes on December 25, 1978, and in a few hours decided to my surprise that it had to be a painting.

Once at ARF, I apparently wasted no time. In December 1945, I had a paper in the ARF publication The Frontier (Vol. 8, No. 4) entitled "Recent Trends in Analytical Chemistry" and my job title shown there was Supervisor of the Analytical Section. Actually, I had walked into a vacuum. The chemistry, physics, metals, and fine particles departments existed; and each had analytical facilities and personnel but with almost no communication between them. There was no one coordinating their activities so I simply furnished that function informally. I called together a group of physicists and chemists from those departments and talked them into forming an analytical section. I promised to get projects to support "us" and did. I also talked some of them out of microscopes they had but didn't know how to use. I was then able to set up a good microscopy lab. One dark night I enschlused a beautiful Leitz Panphot from Physics (they didn't even miss it) and we were off and running. I spent a lot of time selling projects,

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Figure 1. Organization of the Armour Research Foundation's analytical section with pertinent fields of study.

mostly to the U.S. / D.O.D. and the Analytical Section prospered (Figure 1). By 1947, I had a nice group of problem solvers and researchers guided by microscopy, and I was Assistant Chairman of the Chemistry Department with about 20 people in my Analytical Section. Most of them were still officially in the different departments but all working on *my* projects.

Then Charles Tufts, an electron microscopist in physics, and I started talking about a symposium on electron and light microscopy. We found the microscopy community was ready and enthusiastic. We had no trouble developing a program with the top people in light and electron microscopy. Therefore, the first INTER / MICRO was he<sup>1</sup>d Thursday, Friday, and Saturday, June 10-12, 1948, at the Stevens Hotel, now the Hilton Hotel. Looking back, I'm surprised at the program (pp.3-6) and some of the speakers: a Nobel prizewinner, Fritz Zernike; Clyde Mason from Cornell; Charles Saylor from the National Bureau of Standards (now NIST); Ernie Fullman then at the General Electric Co.; Alexander Winchell; Ted Rochow, who introduced us to Resinography at this meeting; and Edwin E. Jelley from the Eastman Kodak Co. Nearly 300 microscopists attended and the vote in the question of an "I / M-49" was unanimously positive. You will be interested, I'm sure, that the registration fee for "I / M-49" was \$5.00 and a double room at the Stevens was \$8.00.

My personal reaction to I / M symposia was one of extreme satisfaction to have the country's best microscopists come to Chicago to further my education.

The full program for the Symposium on Electron and Light Microscopy follows.

### PROGRAM

Unless otherwise noted meetings are in the North Ball Room.

### Thursday Morning, June 10

8:00	Complete registration, corridor outside North Ball Room, 3rd fl. Presiding: PAUL L. COPELAND, Physics Department, Illinois Institute of Technology
9:00	Welcome WILLIAM A. LEWIS, Dean of the Graduate School. Illinois Institute of Technology HALDON A. LEEDY, Acting Director, Armour Research
	Foundation
9:15	Collation and Collocation of Light and Electron Microscopy WILLIAM G. KINSINGER, Hercules Powder Company
9:50	The Role of the Microscopist Viewed at High and Low Power CLYDE W. MASON, <i>Cornell University</i>
10:25	Advances in Light Microscopy $\implies$ Advances in Electron Microscopy ROBLEY C. WILLIAMS, University of Michigan
11:00-11:30	Light and Electron Microscopy: Some Correlations and Analogies CECIL E. HALL, Massachusetts Institute of Technology
12:00- 1:30	Lunch recess
	Thursday Afternoon, June 10
	Presiding: ALFRED L. ELLIS, International Harvester Company
1:30- 1:50	Electron Metallography ROBERT D. HEIDENREICH, Bell Telephone Laboratories, Inc.
2:00-2:25	Electron Microscopical Study of Organic Pigments FRANK A. HAMM, General Aniline & Film Corporation
2:40- 2:55	Electron Diffraction—An Instrument and Some Applications J. G. HUTTON, <i>General Electric Company</i>
3:10- 3:25	The Principles of Phase-Microscopy F. ZERNIKE, Johns Hopkins University
3:30- 3:50	Special Experiments in Phase-Microscopy CHARLES P. SAYLOR, National Bureau of Standards
4:00- 4:15	Instrumentation in Phase-Microscopy HELEN JUPNIK, American Optical Company
4:30-4:50	Light and Electron Microscopical Studies of Cellulose Fibers CHARLES W. HOCK, Hercules Powder Company
5.00 8.00	Dinner recess

### Thursday Evening, June 10

Presiding: WILLIAM G. KINSINGER, Hercules Powder Company

8:00- 8:30	Resinography THEODORE G. ROCHOW, American Cyanamid Company
8:40- 8:55	Electron Microscope Goniometry ALAN F. KIRKPATRICK, American Cyanamid Company
9:00- 9:20	Application of Microscopy to Polymorphism of Tristearin-Type Fats OSCAR T. QUIMBY, The Procter and Gamble Company

9:30-9:45 Crystal Optics on Microscopic Views WILLIAM A. O'BRIEN, Celanese Corporation of America; J. D. H. DONNAY, Johns Hopkins University

### Friday Morning, June 11

Presiding: CHARLES P. SAYLOR, National Bureau of Standards

9:00- 9:20	The Statistical Analysis of Electron Microscope Particle Size Determinations PAUL L. COPELAND, Illinois Institute of Technology
9:25- 9:45	Electron Microscopy of Particle Aggregation in Carbon Black JOHN H. L. WATSON, Medical Research Institute, Henry Ford Hospital
9:50-10:05	Effect of Particle Size on the Diffraction Image in Microscopy HAROLD OSTERBERG, American Optical Company
10:15-10:35	The Application of Microscopy to the Pigment Industry CHARLES MARESH, American Cyanamid Company
10:45-11:15	Results of Electron Microscope Studies of Bacteriophage Action THOMAS F. ANDERSON, Johnson Research Foundation, University of Pennsylvania
11:30-12:00	Ultraviolet, Visible, and Infrared Microscopy K. J. HEINICKE, Bausch & Lomb Optical Company
12:00-1:30	Lunch recess

### Friday Afternoon, June 11

Presiding: ROBLEY C. WILLIAMS, University of Michigan

1:30-1	:50	The Microscope Objective and Its Function LEON V. FOSTER, Bausch & Lomb Optical Company
2:00-2	2:20	The Study of Crystals, Oriented Aggregates and Lyotropic Mesomorphs of Strongly Absorbing Substances with the Polar- izing Microscope EDWIN E. JELLEY, Eastman Kodak Company
2:30-2	2:50	The Properties of Evaporated Gold P.G. WILKINSON and L.S. BIRKS, Naval Research Laboratory

	Friday Afternoon (Continued)	
3:00- 3:15	High Vacuum Metallizing GEORGE H. BANCROFT, Distillation Products, Inc.	
3:30- 4:00	Techniques of High Speed Microtomy ERNEST F. FULLAM, General Electric Company	
4:15- 4:45	Electron Microscopy of the Tubercle Bacillus (BCG), by Metal Shadow Casting Technique C. I. REED, S. R. ROSENTHAL, and B. P. REED, University of Illinois	
5:00-7:00	Recess	
Friday Evening, June 11		
7:00- 8:00	Reception, West Ball Room; 3rd floor, Stevens	
8:00-10:00	Banquet, South Ball Room; 3rd floor, Stevens	
	Guests: H. J. C. IRETON, University of Toronto	
	A. N. WINCHELL, Consulting Geologist and	
	C W M C Ultim	
	C. W. MASON, Cornell University	

mann's talk.

Saturday Morning, June 12

The morning is devoted to two concurrent panel sessions. Each should furnish opportunities to discuss instrumentation and techniques in detail. Semi-formal discussions will be presented by the persons listed below. Informal discussion by all is invited.

9:00-12:00 I. Instrumentation for Electron Microscopy, South Ball Room;

B. B. DAYTON, Distillation Products, Inc.

Presiding: ALBERT F. PREBUS. Bell Telephone Laboratories, Inc.

PERRY C. SMITH. Radio Corporation of America PHILIP NOLAN, Farrand Optical Company, Inc. L. S. BIRKS, Naval Research Laboratory

STERLING NEWBERRY, General Electric Company

3rd floor, Stevens

RODOLFOH. CORZO, Director, School of Biological

NICOLAS AGUILERA, School of Biological Sciences, Mexico City C. E. BARTHEL, Armour Research Foundation L. KOENIC, Armour Research Foundation Speaker: HENRY N. BAUMANN, Carborundum Company Research in Abrasives and Refractories

Mr. Baumann will describe his technique for

observing crystallization, fusion, phase changes, etc., at temperatures of the electric furnace. Several reels of film illustrating these phenomena will be shown.

This talk is heartily recommended by the Symposium Committee. If you can't attend the dinner plan to come later and hear the speaker; 9:15 is the scheduled time for Mr. Bau-

Sciences, Mexico City

F. A. HAMM, General Aniline & Film Corporation C. S. FOSTER, Eastman Kodak Company J. H. L. WATSON, Henry Ford Hospital

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### Saturday Morning (Continued)

9:00-12:00 II. Preparation of Metal Surfaces for Microscopic Examination. West Ball Room, 3rd floor, Stevens

Presiding: A. L. ELLIS, International Harvester Company R. D. HEIDENREICH, Bell Telephone Laboratories, Inc.

J. R. VILELLA, U. S. Steel Corporation of Delaware, Research Laboratory, Kearny, N. J.

M. BAEYERTZ, Armour Research Foundation

A special talk has been scheduled during the above panel session. Dr. N. P. Allen and Mr. B. E. Hopkins of the National Physical Laboratory, Teddington, England are visiting in this country and have accepted an invitation to present a summary of their work. The talk titled "The Electron Microscope and the Phase Microscope in Metallurgy" will be presented by Dr. Allen at 10:00 A.M.

12:00-1:30 I

Lunch recess

### Saturday Afternoon, June 12

1:30-4:00 III. Problems in High Speed Microtomy, South Ball Room, 3rd floor. Stevens

Presiding: H. C. O'BRIEN, JR., University of Pittsburgh

J. J. KELSCH. Interchemical Corporation

R. R. ALLEN, Custom Scientific Instruments, Inc.

W. FULLAM, Hemsdale International, Inc.

E. F. FULLAM, General Electric Company

C. E. GREY, Interchemical Corporation

### INSTRUMENT DISPLAY

An instrument display containing equipment that is of interest to workers in microscopy will be an additional feature of the Symposium. The following organizations have instrument or other displays. These can be studied in the North Assembly Room on Thursday and Friday until 5 P.M. Some of the displays will be exhibited in the South Assembly Room on Friday evening and Saturday.

American Optical Company Bausch & Lomb Optical Company Custom Scientific Instruments, Inc.

DISTILLATION PRODUCTS, INC. GENERAL ANILINE & FILM CORPORATION GENERAL ELECTRIC COMPANY

Optical Film Engineering Company

### PHOTOGRAPHIC EXHIBIT

Electron and Light micrographs submitted for exhibit have been made a part of the Instrument Display and can be seen as noted above.

# A REMINISCENCE OF INTER / MICRO AT CAMBRIDGE, ENGLAND - 1973

Anna S, Teetsov\*

have attended many scientific conferences, but the one that made the biggest impression on me was INTER / MICRO '73 that I attended 25 years ago at King's College in Cambridge, England. In those days INTER/MICRO meetings alternated between England and Chicago. Since this was the first meeting at King's College, microscopists from the United States had the unprecedented opportunity to participate in a conference in an academic environment that is unmatched anywhere else in the world.

Most of the INTER/MICRO attendees arrived from London by train early on July 16, 1973. Most people traveling from the States had flown into Heathrow a few days earlier to spend some time sightseeing in London. Some of the attendees stayed at King's College and some at neighboring Queen's College, directly to the southwest. One of the few students still at the college at that time escorted us to our rooms.

Our accommodations at Queen's College consisted of a large living room comfortably furnished and a small bedroom with two beds and a sink. The rooms were not heated and were quite cold, even in the summer. Windows looked out directly onto the River Cam from which the town, Cambridge, takes its name. Displayed on one wall was a handsomely painted oar with the Queen's College coat-of-arms and the names of the students who had won second place in that spring's rowing event.

Cambridge University is made up of 31 colleges. Founded in 1448, Queen's College sits astride the River Cam, the two halves joined across the river by the famous Mathematical Bridge, more correctly called the Wooden Bridge. King's College, the first of the Royal Foundations in Cambridge, was founded in 1441 by Henry VI. Although it is neither the largest nor the most beautiful, it does possess the most famous chapel, which was founded by Henry VI and finally completed by Henry VIII in 1551.

The Hall at King's College, still magnificent after more than 400 years, was the site for lunches and dinners. The high, plaster ceilings were delicately gilded, and the beautifully carved oak walls were decorated with portraits. We all ate at the long, heavy oak tables. For dessert the first day we had gooseberry pie, served in a large bowl with fresh sweet cream. In England, dessert is called a sweet, to be eaten with a large spoon and a fork.

Dr. Walter McCrone, who planned the meeting, opened the proceedings with a short after-lunch speech about the purpose of the conference. The rest of the afternoon was free for sight-seeing. After supper, the evening session would begin and last from 7-10 PM. When that first session ended, many of the attendees gathered at the bar adjacent to The Hall for a drink. These friendly, informative gatherings took place on each subsequent evening of the conference.

After a busy first day, filled with marvelous impressions of a historic town and topped off by some interesting papers, we picked our way back along the dimly lit passages leading to the Queen's College main gate. We spotted a small sign that read, "Gates will be closed between 12 and 5 O'clock." We were just in time; it was nearly midnight.

The next morning, July 17th, was sunny and cool. Looking out our windows we could see the tall, wrought iron fence with its sharp spikes surrounding the neatly kept grounds. Less than one hundred yards away, a herd of black and white cattle peacefully grazed on lush grass (Figure 1). Although a large

<sup>\*</sup>McCrone Associates, 850 Pasquinelli Drive, Westmont, IL 60559



Cows grazing on the banks of the river Cam as seen from the window of Queens' college dormitory.

Figure 1

city, Cambridge had expanded around the perimeter, leaving large, open fields within the city center and colleges. These fields were still being used as common pasture lands as they had been for centuries.

A self-serve breakfast awaited us in the strikingly decorated Queen's College dining hall. The lower half of the walls were painted black with colorful red, white and gold designs; the ceiling was white with black beams and similar in design to the walls. A 17th century Flemish artist had redecorated the hall in those colors and the Dons liked it so much that it has been preserved this way ever since. Heavy oak tables with long benches stretched along the length of the room. Facing the rows of tables was another long table covered with a white tablecloth where the Dons were having their substantial breakfast.

Presentations started at eight, followed by lunch and a tour of the colleges given by a very proper English lady with an umbrella. That summer was a very wet one and it rained sporadically every day of the meeting.

Our first stop was King's College Chapel, a structure that took almost a century to build. The interior is noted for its wonderful fan-vaulted roof stretching 80 feet above the floor and its enormous stained glass windows. These 16th century windows were the work of both Flemish and English glaziers and are the most complete set of church windows surviving from the time of Henry VIII. The carved wooden screen and choir stalls are in the Early Renaissance style. Behind the altar is the great painting by Rubens, *The Adoration of the Magi*, which had been installed just 5 years earlier.

Clair College, founded by Lady Elizabeth de Clair is the most attractive of the colleges. The Clair Bridge over the Cam River and a stroll through the lovely Clair Gardens were highlights. The tour ended with a visit to Trinity College where we were shown the E entry on the first floor where Isaac Newton had lived for 30 years. We also learned about some of Trinity's famous graduates like Newton, Ernest Rutherford and Lord Byron.

The INTER/MICRO Banquet took place in the festively decorated Hall where dark shadows were cast on the walls by candles burning brightly in rows on the long tables. Bowls of sweet peas accompanied the candles and fresh bouquets of flowers topped the tall pedestals lining the walls on both sides (Figure 2).

The excellent dinner was served in seven courses with three wines. Following the meal, Dr. Walter McCrone gave the customary toast to the Queen, and Dr. John Bunion reciprocated with a toast to U.S. President Nixon. Dr. John H.L. Watson, accompanied by his wife, gave the after-dinner speech, which was really more like a recital (Figure 3). The microscopists' "Victor Borge", Dr. Watson entertained all with his fine voice and songs specifically about microscopy and science. What a memorable evening!

Of course, we stopped at the bar once again for a pint or half-pint of beer before returning to our rooms. It was now very close to midnight and the rain was still coming down. To save time, Dr. Watson



Figure 3: Dr. McCrone introduces Dr. Watson as Jan and Anna Teetsov look on.



Returning to Queens' college after the banquet Figure 4

suggested using the back gate to return to Queen's College. As we reached it, we realized it had been locked at 10:45 PM. We had to run down dark passages and across the now empty courtyard to reach the front gate. Dr. Watson, leading with an umbrella held high over his head, was the first to reach the front gate (Figure 4). In a flat tone he announced, "Closed." For a moment we saw ourselves climbing over the wall, but he was only kidding and we entered through a small side gate that was still open. As we walked through the gate, we saw the porter coming slowly towards us with his keys. What a strange feeling to be locked in for the night! We walked down the narrow passages, crossed the Wooden Bridge over the River Cam and groped our way up the narrow wooden stairs to our room. A noisy commotion outside drew us to the window, and leaning out we could see a group of our friends outside the fence. They had walked all the way around the buildings looking for a way to get in, but found all gates closed. One of the more agile men tried to scale the tall, spike topped fence and, with encouragement from friends on both sides of the fence, he succeeded.. The others, however, could not make it over the fence, even with lots of humorous advice from Dr. Watson; eventually they left. We learned the next morning that somehow they had made it inside.

A bright, sunny morning welcomed us as we

spied from our window a double row of flat-bottomed boats, called punts, tied along the shore.' The punts, navigated with a long pole like a gondola, were a source of amusement for many members who wanted to try their hand at punting. After the morning presentations and lunch, some of the Conference attendees were taken on a tour of the Cavendish laboratories, followed by workshops and supper. Presentations resumed promptly at 7 PM. That evening we finished our pints of beer by 11:45 PM and made it safely back to our rooms before midnight. The hearty breakfast tasted delicious on the next cold, rainy morning. Geoff Woodard chaired a section of excellent papers. After lunch we toured more colleges -Emmanuel, Christ, Sussex, and St. John's - each with beautiful courtyards and gardens.

At 5:30 PM we were escorted to reserved seats at the King's Chapel for Evensong. The choir, composed of small boys and students from King's College, is one of the finest in England. As we sat directly facing the choir, the magnificent sounds of the organ and the angelic voices of the boys surrounded us (Figure 5).

Supper was already being served when we arrived at the Hall. Since this was the final meeting day, Dr. McCrone gave a farewell speech and invited everyone to the 1974 INTER/MICRO in Chicago. Most of us stopped, again, for one final pint of beer at the bar. Many of the students from the choir were in the bar. One started to play his accordion, another his guitar. As they sang and played Russian, Jewish, American and English folk songs, from time to time everyone would join in. Some of the songs were lively, some sentimental; everyone felt so at home. How sad that midnight was approaching. We said our good-byes, exchanged addresses with newly made friends and left the bright hall, walking briskly to our dormitories through the cold, rainy night. The next day, with the cows quietly grazing across the river, we packed our bags and said farewell to the great historic and academic environment of Cambridge. What a privilege to have been a part of it all!

Special thanks to Dr. McCrone, who planned the meeting in Cambridge and encouraged us all to attend, and to Sara Mark of McCrone Scientific Ltd., London, for the many weeks of planning and arranging the wonderful INTER / MICROs held in England.



Kings College choir singing at Evensong in King's college Chapel

Figure 5

### Case History #M-368 Failure of Black Paint Coating

consumer products А manufacturer experienced premature coating а failure, and suspected that the wrong paint carrier system was used during the finishing operation. Using micro pyrolysis techniques with FTIR microscopy, Microspec Analytical was able to match the coating from the failed part to one of several possibly used at the facility.



### Case History #M-519 White Corrosion on Hinges

An office furniture manufacturer noticed white deposits developing on hinges inside one of their products. Optical microscopy combined with microchemical analyses showed the deposit to be zinc oxide. The project was completed in a manner of minutes.



3352 128th Avenue Holland, MI 49424-9263 **Phone: 616-399-7400** Fax: 616-399-6185 E-mail: *info@mspec.com* Internet: *www.mspec.com* 

### Case History #M-699 Industrial Fire Investigation

Optical microscopic examination of debris from the source of a fire at a manufacturer furniture showed pigmentary iron Micro extraction oxide. revealed a combustible oil. Sparks from a welding torch ignited apparently а container of dried wood stain that still had enough oil content to start a fire.



### Case History #M-947 Particles in Powder Coatings

metal finisher А experienced occasional fiber contamination on finished parts. Though a powder coating was suspected as the source, no visible contaminants were obvious. Microspec Analytical was able to isolate and identify several types of fibers by FTIR optical and microscopy.

 $\mu \bullet \text{NOTES}$  AUGUST 1998

### The Polam 213 Polarized Light Microscope

David J. Roy\*

he polam microscope, manufactured by LOMO<sup>TM</sup> (Leningrad Optical-Mech Organization), is a rather remarkable microscope in its own way. First, let me say something about LOMO (1), which has an interesting history but is completely unknown to most Americans. LOMO's predecessor, a joint stock company called Russian Optical and Mechanical Products, was founded in 1914 as a producer of artillery sights. The company went through a number of changes before uniting with several other optical ventures in 1962 as LOMO. In Soviet times the enterprise produced optical equipment for the military, engaged in scientific research, and developed optical devices for medical applications - most of Russia's eye clinics now use LOMO equipment. The company has undergone western style restructuring which resulted in a reduction of staff from 20,000 in 1992 to 9,000 in 1997 and the introduction of business units with individual accounting responsibilities. Ultimately, because of the unreliability of state contracts and payment, LOMO has been pushed into the international market, and, as a result, exports have increased from \$80,000 in 1992 to \$16,000,000 in 1996. LOMO is a capable competitor in the international market of inexpensive optical devices, manufacturing such items as microscopes, telescopes (including night vision), binoculars, medical optics, cameras, and even electronic voting machines. Lomo is also noted for having made a six meter mirror for the observatory telescope in the Crimea (Ukraine).

Many of these items are being sold in the United States and it has been estimated that, by the year 2000, 70% of LOMO's production will be exported.

It is interesting to note that Austrian students discovered the inexpensive, all-manual, compact cam-

era manufactured by LOMO and made it into an underground art phenomenon.

How about the Polam microscope? The Polam microscope, currently marketed in the US by LOMO America, Inc., Prospect Heights, IL, has an amazing number of capabilities considering its current price of \$5,000. It is a trinocular microscope equipped for polarized light microscopy, photomicro-



Figure 1. Polam 213 microscope with its monochromator located below the condenser.

<sup>\*</sup>Retired, Kraft Foods Technology Center, Glenview, IL

graphy, dark field, phase-contrast, and dispersion staining. There is a ball bearing stage, Bertrand lens, monochromator, and diaphragms in the head and photo tubes (Figure 1).

A more detailed description of the Polam microscope would include the following mechanical and optical features:

**Mechanical**: A tripod frame provides coaxial focusing and attachment of the light source, stage, condenser, analyzer, and head.

**Illumination**: Critical Köhler illumination is available and allows for vertical, horizontal, and focusing adjustments of the 9 volt 70 watt lamp.

**Condensers**: Two condensers are supplied. One has a 0.85 N.A. and provides diaphragms for dark-field, phase contrast with a 40X objective, dispersion staining (central and annular), The other condenser has a 1.25 N.A., (Figure 2) for use with high power objectives.

**Eyepieces**: Three 6.3X eyepieces are provided, one general purpose, one with cross hairs, and another accepting a grid or scale reticle. The 10X eye pieces include one general purpose, one keyed with cross-hairs, and a third wide-field. A keyed, extra wide-field, 16X eyepiece with cross-hairs is also included



Figure 2. 1.25 N.A. condenser with mount.

for use with the monocular head.

**Objectives**: The objectives are all strain-free, infinity corrected achromats or planachromats. All together the seven include: 2.5X 0.05 N.A., 10X 0.20 N.A. with an iris diaphragm, 25X 50 N.A. with an iris diaphragm, 40X 0.65 N.A. phase contrast, 40X 0.65 N.A. bright-field, 60X 0.85 N.A., and 100X 1.25 N.A. (Figure 3).

Analyzer: The analyzer, located in the adapter tube, has a vernier scale readable to 0.1 degree. A centerable and focusable Bertrand lens is also located in the adapter tube.

### Monochromator: The monochromator consists of



Figure 3. Nose piece of Polam scope accommodates five objectives.

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Figure 4. Tilting monochromator with six interference filters and tilting wheel.

six interference filters mounted in a disc below the condenser. The disc may be tilted at various angles to change the wavelength of the filters in a range from about 400 nm to 700 nm (Figure 4).

Accessories: Various other accessories included are two 1/4 wavelength mica plates, a first order quartz plate, a three order quartz wedge, various reticles, and a Zenit 122 autoexposure single lens reflex camera for 35 mm photography.

A microscope, costing \$5,000, with the features and accessories that the Polam has seems almost too good to be true; but there is, as you might expect, a down side. Although the quality of the optical and mechanical components is acceptable for all but a few critical applications, it is not up to the same standards as the more expensive, better known microscopes. There are, in my opinion, a need for a few changes in some of the design features. An encouraging note here is that LOMO has been consulting with the McCrone Research Institute (3) in an effort to improve the Polam microscope and, according to Mark Levitin, Vice-President of Sales and Marketing for Lomo America, Inc., the general features of the microscope will remain the same; but significant changes will be made to improve the quality. He also indicated that the price would be in the same general range as the current model. An improved Polam microscope would be an excellent alternative for some of us with not-so-deep pockets. The new model is expected to be on the market after the beginning of next year.

I should also like to mention that the Polam microscope has some very attractive design features such as the 0.85 N.A. condenser with a revolving disc which contains six diaphragms. This is a convenient arrangement allowing for dark-field, phase contrast, and dispersion staining with 10X and 25X objectives. I believe that the dispersion staining works very well on the Polam microscope, and that there is an advantage having the diaphragms built into the condenser. It is possible to adjust the annular diaphragm so that it is off center, and to achieve what Cherkasov (4) terms unilateral screening. This is another form of dispersion staining which is referred to as "focal screening" by Cherkasov. Good unilateral and annular dispersion staining colors may also be obtained by removing the condenser, positioning the field aperture, and adjusting the size of the iris diaphragm in the objective. Other important features are diaphragms in the head and in the phototubes which greatly improve conoscopic observation and photography of interference figures. There is a double race ball bearing stage which I think is a big advantage over a greased stage. I have a Polam 213 microscope, which I purchased used, and have found it to be capable of performing a number of tasks satisfactorily.

My own experience with the Polam microscope indicates that some improvement could be made in the following areas:

• The present turret design allows objective set screws to protrude which makes gripping and turning the turret inconvenient. I believe that it may be possible under some circumstances, that if pressure is applied to the set screws when the turret is turned, the objective may become uncentered due to movement of the set screws.

Removing or replacing the top lens of the 0.85 N.A. condenser requires raking the condenser down and then back up and refocusing the field aperture. A lens that could be easily inserted or removed would be very desirable. Another suggestion would be to replace the 0.85 N.A. Abbé type condenser with an achromatic condenser of the same or slightly higher numerical aperture. The 1.25 N.A. condenser and the 100X objective could be eliminated or sold as accessories in order to cover the cost of an achromatic condenser. Although there may be instances where the 100X objective may be desirable, I do not believe that this objective is used too often in polarized light microscopy. The monocular head could also be eliminated or the microscope could be supplied as a monocular scope for those who are not interested in a binocular scope. The modular design of the Polam microscope makes a number of options possible.

• A small inexpensive voltmeter on the lamp to indicate color temperature would be very useful.

• The detent for the disc in the 0.85 N.A. condenser does not position the diaphragms as precisely as it might. • The auxiliary ground glass condenser lens, required for use with the 2.5X objective, is inconveniently located. With the present setup the lens may be installed below the condenser so that it rotates either to the right or to the left for removal from the condenser system. In either of these positions there are times when it tends to interfere with the operation of the microscope.

I might also mention that placing a large heavy rubber band over the turret rim has helped solved the problem of gripping the rim without disturbing set screws. A plastic or metal ring that would fit over the turret rim would be useful. Set screws which do not remain in place are a common problem on many instruments and may be treated using Loctite Threadlocker 222MS (5). Be sure to check to see you are getting the correct material for the application so that the set screw will remain adjustable.

It will certainly be interesting to see what changes have actually been made when the new Polam microscope finally comes out. I believe the Polam microscope has the potential to fill an important existing need for a moderately priced polarized light microscope with extended capabilities.

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# THE CAP GUN AND THE MICROSCOPE: FIRST STEPS IN A MICROSCOPICAL ODYSSEY

John Gustav Delly\*

We did you get started in the field of microscopy? This question frequently comes up whenever small groups of microscopists find themselves together at meetings etc., engaged in informal shoptalk. The answers are always both varied and interesting. This is my story of how and when I got started on this fascinating odyssey into the microscopic realm.

My journey started in 1939, and although I do not have personal recollection of the events, I have it on the highest authority, *my mother*, that it started this way: My mother had been lying down on the couch, resting from work in the grocery and meat market that my parents owned and operated. I no-

ticed her hair turning gray, and I asked her *why* it was turning gray. She answered that that is just what happens to mothers when they work hard and get older. I declared that when I grow up I would discover some way to keep mothers from turning gray (the cosmetics companies beat me to it!). Accordingly, the next year, when I was seven years old, I asked for and received for Christmas a large Gilbert Chemistry Outfit, similar to the one shown in Figure 1—no doubt at considerable sacrifice on the part of my parents. This Gilbert Chemistry Outfit would later be supplemented with a large Chemcraft Chemistry Set (Porter Chemical Company, Hagerstown, Maryland). Within a few months, I realized that the chemical



Figure 1. Gilbert Chemistry Outfit.

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approach to the study of graying hair was not enough; I needed a microscope.

December 1941 was a memorable month for me on two counts: First, America entered World War II, following the December 7th attack on Pearl Harbor; Second, I got my microscope! Here is how it happened: the father of a school chum of mine was Manager of the toy department at Goldblatts, a large department store in Chicago. After Christmas, the father would allow his son and one friend, me, to go into the stockroom where returned and unsold merchandise was kept, to pick out toys prior to sales and inventories. We were both quite excited at the prospect. Meandering around the stockroom, we both spotted a Gilbert Microscope Set at about the same time, and we both made a dash for it. He got to it first and claimed it his. I then spied a beautiful Pirate Cap Pistol with "ivory" grips, double barrels, double hammers, and a double-release trigger, and declared it mine.

When we got home, I tied a bandanna on my head the way I had seen the romantic pirate swashbucklers do in the movies. A pair of black gloves, a homemade eye-patch, and the loaded pistol tucked into the front of my belt at a rakish angle completed the picture. I still remember the astonishment and desire in my boyhood friend's eyes as I drew my beautiful pistol and let loose both barrels! A quick reload and another two shots from the exquisite piece, and my school chum announced a strong desire for the pistol. We struck up a trade: he got the pistol; I got the Gilbert Microscope Set. Over the years, I have often wondered whether his life was influenced as much by our trade as mine was!

That all took place over 50 years ago. Recently. I was in an antique mall, and in one of the dealer's cases I saw a pirate cap pistol (Figure 2) exactly like the one I had had as a kid! The asking price was an unbelievable \$60. The sight of this pistol, however, sent me into an instant time warp; the memories came flooding back, and the prospect of having both the pistol and the microscope at long last was too much to resist. I bought the pistol, which I had forgotten actually said "Pirate" on the escutcheon of the extended scales. I disassembled it, cleaned and oiled it, and discovered a patent number inside. This led me to research the pistol. There is a Schiffer book for antique collectors called Cap Guns; With Values by James L. Dundas (1996) in which I found an illustration of my pistol with the following legend (p. 114):



Figure 2. "Pirate" cap gun, made by Hubley in 1940.



Figure 3. Gilbert Microscope Set.

"Pirate. 9 3/8"-long automatic made of nickel-plated die-cast in 1940 by Hubley. \$80-\$100." Also, I obtained a copy of the patent (U.S. Patent 132,661) and found that it was issued in the year 1872 to cover an improvement in shutter- fasteners! The designer of the pistol had incorporated the patented mechanism into the double-hammer double-trigger release. This pistol is now mounted on a burgundy-colored velvet backing board in a custom-made shadowbox frame, along with some caps, which I later found in another antique mall.

The Gilbert Microscope Set is illustrated in Figure 3. It came in a wooden box painted blue, or green, or yellow, or covered with a textured paper; mine was painted green. The microscope lamp—the shiny metal box, with cord attached, second shelf up utilized a white Christmas tree /night-light bulb. The microscope, unfortunately, was of inferior quality [see my article "Microscopes for Kids," *The Microscope* **40**, 269-274 (1992)]. But the manual, written by Oscar Richards of Yale University—later of Spencer Lens Co. / American Optical Co., Editor of *The American Microscopical Society Journal*, etc.—was absolutely outstanding. I still have my copy—autographed for me by Oscar Richards in 1986. The set in Figure 3 is not my original—the contents of mine were used up, the microscope was replaced with my first, real, brass microscope, and the manual, now hard-bound, is in my rare-book room.

So, here I am, more than a half century after the original events, with both the Pirate Cap Gun (who knows...perhaps it is the *same* gun?) and the Gilbert Microscope Set—and, of course, a Chemistry Outfit; it is very satisfying to see them all together again. The memories they invoke every time I look at them are as vivid as if the events took place yesterday: I can still hear the click-click, click-click of the double hammers being cocked; I can still smell the pungent combustion products of the spent caps; on my forehead I can still feel the heat emanating from the microscope lamp as I lean over the instrument peering

into the eyepiece, exploring the world; I can smell the alcohol from the vials where the bee and the fly are preserved, the fragrance from the Oregon balsam, and all the wonderful sights, smells, sounds, and sheer magic associated with the chemistry sets—all in the serene peace of my very own personal laboratory.



Ah....Paradise!

### A QUICK AND EASY DRY MOUNT PREP

### James J. Benko\*

few years ago I detailed a method for making dry mounts by using one inch by one inch poster mounting squares (1). This method has worked quite well for dry powders, fibers, dust samples, sand, dirt, etc., and will even serve to replace well slides in the making of hanging drop preparations.

Basically, a small square is cut out of the interior of a poster square using a razor blade (or Exacto knife) to leave an outside frame of the poster square. The adhesive on both sides of the frame forms a seal between the microscope slide and a 22 mm square coverslip. While this method is quick, the method given below is even quicker and produces a mount that is more secure since there is more adhesive area contacting the slide and coverslip.

### I. ITEMS REQUIRED

- 1. Poster squares
  - a. 1" x 1" squares for a 22 mm coverslip
  - b. 3/4" x 3/4" squares for an 18 mm coverslip
- 2. Hand-held paper punch
- 3. Microscope slides / coverslips

### **II. METHOD**

- 1. Using the paper punch, punch one or more holes in the poster square as desired.
- 2. Peel away the adhesive strips, placing the poster square on the microscope slide.
- 3. Add specimen(s) to the well formed by the punch.
- 4. Add a square slipcover over the poster square frame to complete the mount.

This method works well with the 3/4" poster squares.

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Figure 1. Paper punch and poster squares for dry mounts.



Figure 2. Finished dry mount prep.

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### **CENTERING WRENCHES—ELUSIVE AND CAPTIVE**

### John Gustav Delly\*

hat is it about centering wrenches—those absolutely vital little accessories so necessary for the centering of phase-contrast annuli, and objectives and/or stages of polarizing microscopes-that you can never find them when you need them? They are as elusive as Baroness Orczy's Scarlet Pimpernel; indeed, centering wrenches are the elusive pimpernels of the microscopical world. The McCrone Research Institute (McRI) has over 100 polarizing microscopes and 30 phase-contrast microscopes because often two or three classes take place in the Chicago facility and one on-site. That translates to 260 centering wrenches. They are short and round, so they easily roll under a microscope or into a pile of papers or off the table top. Or, they will be placed, inadvertently, into the wrong hole in the instrument holder and irretrievably lost. McRI uses three different brands and models of microscopes, and, of course, the centering wrenches are not interchangeable. No matter how carefully the classrooms are set up, when it comes time to center the objectives, or the stage and objectives, there is always a student who says, "my wrenches don't fit," or "are we supposed to have two wrenches?" In any case, by the end of a week, there is a good chance that at least one centering wrench will have vanished.

Individuals suffer from this malady as well. Every month there is somebody who phones or emails that they bought a polarizing microscope, but it didn't come with centering wrenches; do I know where they can get a pair. Most recently, I heard from State Microscopical Society of Illinois fellow member Garth Ziemba, who bought an older Leitz polarizing microscope, but it lacked centering wrenches. Although he had contacted several Leitz dealers and Leitz headquarters, no one could supply him with replacement wrenches for his model. Fortunately, he persisted and, ultimately, located a Leitz distributor in Canada, who knew where to find a part number and actually located a pair of the wrenches for him.

At McRI, we periodically had to order a couple of dozen replacements, but eventually we are told that they are no longer available from the manufacturer, and we have to pay a machinist to make them for us. The costs vary, as sometimes the wrenches consist of cylinders with incorporated crossbars, for use with slotted-head centering screws; sometimes they are female hex-head openings; sometimes they are female square-head openings; the current Olympus microscopes require male hex-head wrenches.

The real impetus for writing about this subject now was a recent demonstration of the new Olympus B-Max series of polarizing microscopes, the B-Max 50 and the B-Max 60. During the demonstration, it was mentioned that the centering wrenches cost \$40 each. To look at them, you wouldn't think to pay \$80/pair. They are made from aluminum and have a short hex-head wrench protruding from the end. The blackened finish is amateurish. Figure 1, far left, shows the original centering wrenches supplied by Olympus-I hope in reproduction the poor finish will be as apparent as in the original photograph. The local distributor, realizing \$80 to be an outrageous cost for new wrenches, had a local machinist make them. These replacements, shown in Figure 1, as "new replacement" are also made from aluminum, with a hex-head wrench protruding from the end. These are not blackened, but do show a poor job of crosshatch knurling; they sell for \$17 each. The real surprise came when I went to use these replacements-they didn't fit the hex opening in the nosepiece!

I spent a few minutes with a rule and a micrometer and came up with the following: the original Olympus male hex head is 0.0590 inch, that is, 1.5 mm across the flats and protrudes 4 mm out of

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Figure 1: A variety of centering wrenches. See text.

the wrench where it is epoxied in a hole; the new replacement hex head is 0.0610 inch, that is, 1/16 inch (0.0625 inch) hex stock, which translates to 1.55 mm. Therefore, the 1.55 mm wrench could not fit the 1.5 mm holes. As for the protrusion of the hex wrench in the new replacement, one was 4 mm, as in the original, and one was 5 mm. There were other, noncritical dimensions, that made it clear the new wrenches were made on the English system (15/32 inch long knurled portion; 19/32 inch non-knurled portion), rather than made on the original's metric system (12 mm long knurled portion; 15 mm long non-knurled portion); same for the respective diameters.

I asked myself, if I have a B-Max 50 and lose the wrenches for some reason, what do I do? Pay the \$80 for a new pair or \$34 for a pair that does not fit? My answer is *neither*. I went to a hardware store and bought a 1.5 mm hex L-key wrench; cost, 20¢—see Figure 1, "1.5 mm HEX." This is the "short-arm Lkey"; it is 1-3/4 inch long. The "long-arm L-key" is 3 5/64 inch long and costs 22¢. They don't have the nice knurled handles, but they work, and you can buy a lot of them for \$80. Some local hardware stores may not carry metric hex wrenches; in which case, any industrial supplier, such as McMaster-Carr Supply Co. [Elmhurst, Illinois; (630) 833-0300] can supply them.

A handle would be nice, though; and so I thought about my Chapman "G" set, which consists of 19 bits that are accessories to my Chapman gunsmithing screwdriver set. Sure enough, one of the bits was a 1.5 mm HEX #40 (Figure 1). Now, this bit is designed to fit in the end of a number of different kinds of drivers, but note that the body itself is hexagonal, and it is knurled on the end, so that the bit itself acts as its own non-rolling handle. I'm sure that the single bit is available from Chapman, but the whole set of 19 different ones is available at *less* cost than *one* of the new replacement centering wrenches. [One source for the Chapman screwdriver set and the "G" set is the gunsmithing supplier, Brownells, in Montezuma, Iowa (515) 623-5401].

These two solutions, though cheap and elegant, do not prevent the replacements from being lost. The problem is that there is no place to store the centering wrenches on the microscope or to otherwise make them captive. Looking over the "Y" base of the Olympus B-Max, I noticed two polymer plugs on each side of the base; these plugs are concealing 1 cm diameter holes in the base. I do not know what the holes are for, but I would use them to bolt my microscope to my bench top. So, I thought, why not just drill a hole through these polymer caps and use them as places to store the centering wrenches? Figure 2 shows a centering wrench in just such a storage location. Now, at least, there is a place on the microscope to store its vital wrenches. It is ironic that there is just such a storage hole in the top of the body available for the handled wrench that is needed to assemble/ disassemble the instrument. The knurled handle of the centering wrench could also be painted with a base coat of white, then a blaze-orange top coat to increase its visibility.

Still, the wrenches are not captive. That this has always been a problem with centering wrenches, I cite the example of the photomicrographic bench built on a lathe bed—one of five made—that was at one time used by Roger Loveland at Eastman Kodak Company to study particle size and other characteristics of photographic film. The photomicrographic camera, which now resides in the museum of the McCrone Research Institute, was made so that every adjustment was graduated and could be recorded. A centerable stage was also fitted to it. Eventually, Roger had the same problem of elusive centering wrenches, and his solution was a model of simplicity: he tied a piece of string between the two wrenches—see Figure 3. Mind you, we are talking about the 1930's, certainly more than half a century ago.

What might we do for an update, in the way of captive centering wrenches: Figure 1, far right, "Captive Swivel Attachment" might be one solution. First of all, start with a good job of knurling the main body of the wrench; then drill a hole in the end for the hex wrench; and finally, apply a satin chrome finish. Second, epoxy a 1.5 mm hex wrench in the end hole so that it protrudes 4 mm. Third, install a miniature screw-eye in the end opposite the hex wrench, and attach a fisherman's Size 14 snap swivel (note the ball bearing before the chain). A Size O ballbearing snap swivel, or a Size 12 brass swivel could also be used as alternatives. Fourth, add a chain to the snap swivel; I used a #130 chain having 17 links



Figure 2. Centering wrench stored in hole drilled in polymer cap in base of Olympus B-Max microscope.



Figure 3. Centering wrenches belonging to Roger Loveland's microscope, tied together with a piece of string.

per inch. The other end of the chain is attached to the microscope anywhere that it is convenient; I went to the microscope base. The snap swivels can be bought cheaply anywhere fishermen buy their supplies; and the chain is available amongst the "findings" in any jewelers' supply catalog, or in a hobby shop where model ship builders buy their supplies.

I have sitting on my microscope bench right now one of the original AO Ortho-Illuminators. For the alignment of the light source to the microscope, a pinhole disc 1 inch in diameter, is supplied. I imagine the designers of this illuminator actually had to use it because they had the experience and/or foresight to realize that the little disc was a natural to become lost—it was just too small and seemingly insignificant. These wise designers saw fit to thread the pinhole disc beneath and to use a slightly longer screw protruding from one of the clamp-down rings, so that when not actually in use, the pinhole disc could be screwed down onto the protruding bolt. This gave me still another idea: the portion of the centering wrenches between the knurled head and the protruding hex wrench is of the right length and almost the right diameter (or could be made so) to thread, either 6-32 or 8-32, for example. That way, the microscope body or base could be drilled and tapped anywhere for the same thread so that when the centering wrenches are not in actual use, they could be screwed back into their storage position right on the microscope. I think I like this best of the non-tethered methods.

A few minutes thought will yield other ideas:

simply drill a hole through the knurled end of the wrench and tie on a piece of nylon fishing line, or place on a key ring, or....

Some enterprising manufacturer should come up with a contest or other means of eliciting creative solutions to the problem of elusive centering wrenches.



## Alteration When it Alteration Finds<sup>†</sup> A MICROSCOPICAL APPROACH TO WRITING SEQUENCE DETERMINATION

Joseph G. Barabe\*

t is a pleasure to contribute a short paper in honor of Dr. Walter C. McCrone on the event of the 50<sup>th</sup> Anniversary of Inter/Micro. Among countless others, I consider myself fortunate to have had the experience of learning the use of the microscope under the broad umbrella of his influence.

#### Introduction

Determining the order of sequence between two ball point pen writings, or ball point pen and pencil writings can be a difficult, even exasperating, experience. While numerous techniques have been suggested for their decipherment, the first approach (and often the last) should be a careful look at the writings with sufficient magnification to see microscopic details.

The approach proposed herein was discovered through a small research project, which in turn was inspired by a difficult case. After creating and studying numerous exemplars, I became aware of a number of general microscopic characteristics that indicate writing sequence. Some of these characteristics have been noted in the literature, but I am not aware of the more general approach advocated in this paper. Specific reference to these contributions will be made in a future, more detailed paper on this subject. For the sake of brevity, I will present my findings as a preliminary report from an on-going research project. As such, continuing research will surely augment and modify these conclusions.

The goal of the project was to explore those microscopic characteristics that may allow the document examiner to determine the sequence of writing and to gain some understanding as to the unique circumstances governing each set of characteristics.

The main factor determining decipherability was found to be the force and speed with which intersecting lines were made. Several other factors contribute to the decipherability of a writing sequence as well: The type of writing instruments, the angle of line intersection or crossover angle, the number of crossovers present, and the total area comprising each crossover are all important factors. Yet, the document examiner may not have enough information available to make a clear determination of sequence. Many crossovers exhibit the microscopic characteristics listed below either poorly or not at all. Therefore, it is vastly better to be honestly inconclusive than mistakenly certain.

Testing was done using two different writing instruments: ball point pens of various types and manufacturers and pencils, both # 2 and # 3. No dip or fountain pens, or any other wet ink pens such as "felt tip" pens were tested in this project.

### **Preliminary Considerations**

Paper starts out blank, flat and smooth. Then, it is altered by the first writing, which puts a groove into the paper and deposits ink or pencil particles. This first writing is characterized by a high degree of continuity; that is, the whole set of interactions between the writer, the writing instrument and the paper substrate is uniform. When the second writing, W2, encounters the first writing, W1, W2's previously unhindered course of movement can be altered in several characteristic ways.

<sup>&</sup>lt;sup>†</sup>Shakespeare, Sonnet 116

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#### **Approach: Forcefulness of the Lines**

A very useful way to approach sequencing decipherment is by the force employed in creating the lines W1 and W2. With each combination of light and heavy lines, different microscopical characteristics present themselves.

### **Microscopical Characteristics**

### Light W2 over Heavy W1

For the sake of clarity, we will distinguish between *light* lines, which are written with relatively less force, and *heavy* lines, which are written with greater force. A heavy W1 has a strong tendency to alter the path of a lighter W2. In fact, as a light to medium W2 line encounters W1, a characteristic sequence of events occurs.

#### 1. Hourglassing.

When W2 first enters W1, it enters a space opened at the sides, so that the W2 writing instrument strikes the concave trough constituting the W1 line. The W2 line is narrowest in width at the deepest portion of the W1 line, at the bottom of the trough. As the W2 instrument again encounters the far side of the W1 trough, the W2 line widens, thus the hourglass shape of the W2 line (Figure 1).

Hourglassing can exhibit many forms. When W2 is light, the hourglass has a narrow waist. In other cases, especially when the proximal edge of W1 is deep and steeply angled, and/or the W1 line is written with great speed, the W2 line skips over the proximal edge, leaving a short blank spot. We might then see a cone widening towards the far side of the W1 groove. A heavier W2 produces a wider waist, and when W2 is written as forcefully and deeply as W1, the hourglassing disappears. At that point, we lose hourglassing as a distinguishing characteristic.

### 2. Blobbing.

After the W2 instrument passes over the bottom of the W1 trough, the waist of the hourglass, it strikes the distal wall of the W1 trough. If the W1 wall is steep enough, the W2 instrument slaps against it with considerable force, depositing a noticeable blob of ink or pencil material on the distal wall (Figure 2).

There is considerable variation in the amount of blobbing that occurs, ranging from none at all to a thin line at the top of the trough to a large mass of ink or pencil particles.

### 3. Skipping.

Blobbing is frequently accompanied by skipping. As one follows W2 along its path, the high angle of W1's distal wall causes the W2 instrument to bounce upward briefly, leaving a small bare spot made even more noticeable by the blob of material just preceding. Shortly after the bare spot, the W2 instrument returns to the surface of the paper and continues without further incident (Figure 3).

### 4. Jogging.

The hourglass, blob, skip and return sequence is most frequently seen when the crossover occurs at more or less right angles but can occur at oblique angles. At more oblique crossover angles one frequently encounters a different alteration of the W2 path by the W1 trough. When the W2 instrument tip slaps against the W1 wall at an oblique angle, the side of the wall offers enough resistance to alter the course of the W2 line in several possible ways. It can alter the path of the line, such that a noticeable "jog" is seen in line W2. The thickness of the line may also be changed. Generally, the W2 line becomes narrower due to having been pushed upward by the steep wall of W1. Blobbing on a jogged line is often seen as an oblique smear (Figure 4).

If the sequence of hourglass, blob, skip and return is encountered, one can conclude with a high degree of certainty that the altered line is W2. If multiple crossovers are available for analysis, and they all present the same characteristics, one can be even more certain. Jogging is a reasonably reliable marker for sequence decipherment; however, it should be evaluated with caution as jogging can have several other possible causes.

### Heavy W2 over Light W1

As noted above, these characteristics are most often seen when W2 is written with less force than W1, and can often be seen to some extent when W2 approaches and surpasses W1 in force and depth.





Figure 1. Hourglassing.



Figure 2. Blobbing.



Figure 3. Skipping

Figure 4. Jogging.

However, when W2 is written with a force equal to or greater than W1, a different set of characteristics obtains:

1. A lack of the characteristics as described above: specifically, hourglassing, blobbing, skipping and jogging are all absent.

2. Continuity of *width* of W1. Remember: W1 was written on an unaltered substrate.

3. Continuity of *depth* of W2. Although W1 had altered the surface of the paper, the more force-fully written W2 overpowers it. Please note that there is a noticeable deepening of the grooves at the cross-over spot that may mask depth continuity.

Both of these principles deserve careful scrutiny and thoughtful evaluation. At this point in my research I prefer to consider these characteristics as pointers, strong indicators of sequence, rather than as conclusive evidence. These characteristics need considerably more study.

#### The Principle of Continuity

The principle of continuity is most frequently evoked when developers of new sequencing techniques present their findings. Although this principle can be helpful in some instances, the difficulty is in knowing when to call it forth. Continuity as a sequencing characteristic is a thorny issue.

Material drag from W1 along the W2 trough is an-

other determining characteristic available to the document examiner. One occasionally finds small amounts of material that have been dragged from the W1 line into the W2 trough. This can be a strong indicator of sequence, but it too should be evoked with thoughtful caution. There are other mechanisms for material transfer from one place to another. Pencil dust does not adhere as strongly to paper fibers as does ink, and one frequently finds particles scattered randomly about a document. Even small droplets of ink can be shaken from the pen tip by sharply tapping the tip of the pen against the paper.

Material drag is, however, a fairly rare phenomenon. The dragged material is usually very close to the W1 trough, is frequently seen as strings of material lined up in a row, and tends to occur on the high fibers "downstream" on W2.

#### Instrumentation

A good quality stereo microscope is generally adequate for sequence decipherment. For difficult cases, a compound microscope with a set of metallurgical objectives, corrected for optimal quality without the use of a cover glass, is recommended. A "high-dry" objective of 60-80 times magnification will prove especially useful in searching for evidence of material drag.

#### **Create Exemplars**

One helpful way to approach a difficult sequence of writing problem is to create exemplars matching the questioned document in as many ways as possible: Thus, the same paper, the same types of writing instruments, the same colors, the same hardness (of pencil), and, of course, the same relative writing pressure should be employed. Then study the results. Patterns will emerge that may very well provide the specific key to your particular decipherment.

#### **Other Factors**

There are also several other factors that affect decipherability. In general, the greater the *writing pressure*, the more information is present. In practice, one finds that the combination of a heavy line with a light line presents the least ambiguity. A corollary to this statement is important: Two lightly written lines interact minimally and are in most cases inconclusive. Two heavy lines may be able to be deciphered evoking the principle of continuity, that is, a continuous W2 over a discontinuous W1.

The *width* of the writings is also an important factor. Writings with normal (wide) points are easier to decipher than writings with fine (narrow) points, the fine point is W1.

Writings that differ in either *color* or *material* are easier to decipher than writings of similar materials.

#### **Future Work**

As mentioned previously, this paper is a preliminary report of ongoing research. It is especially important to better elucidate sequences where W1 is lighter than or equal to W2. Also, blind trials of various sequence combinations should be conducted and statistical results tabulated.

Acknowledgement: David A. Wiley, McCrone Associates for illustrations.

### **Historical Perspectives**

### **Richard Hoyt Lee\***

**M** icroscopy and history make a fascinating combination for me because the history of science and microscopy makes our current accomplishments more appreciated. Ultimately, we find that the earliest discoveries and microscopes are often our best tools even today.

I enjoy finding details about the whole person in history, their background and personality, especially scientists with humble beginnings who many times are ignored until they make a major discovery or after they have died. Many early scientists can teach us how to do research with simple equipment. Pasteur began by solving practical problems with the use of his microscope in very simple laboratory conditions. Without much funding and sophisticated equipment, his work led to outstanding accomplishments in food science and medicine, although he was not a doctor. Did anyone question his credentials or equipment then? Some even call him the father of microbiology. Today we have the luxury of hightech equipment, an immense body of scientific knowledge, and collaborative teams that were unknown to our scientific forefathers. What might Pasteur accomplish today in our community of microscopists!

Some of the research that I do occasionally involves discovering the history of a culture through the analysis of ancient objects such as Mexican murals painted on stone temples (Figure 1). I get a real thrill examining something that no one has seen for a thousand years and determining how it was made. Various microscopes reveal a great deal about the materials and pigments used, their preparation (technology), and the state of their preservation. The knowledge gained from this kind of research is every bit as



Figure 1. Mayan mural.

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rewarding as solving cutting-edge technological problems. Microscopy is much more than just a tool for examining artifacts because it enables us to formulate how the materials interact to make a durable composite. The right materials mixed with the appropriate binding agents make a real engineered material. Materials science has been around for a long long time, only we call it art technique. The selection of microanalytical techniques permits us to analyze almost any material we may find, but without the microscope, we may not understand the function of each component.

Because of the variety of technologies available for analyzing our samples, we have created a collaborative team of scientists from several areas. The polarized light microscopist compliments the electron microscopist to determine the distribution of pigments by their color and to identify many minerals by their optical properties. The electron microscopist identifies the material phases and subtle differences in composition. For example, the differences in composition can tell us what region of the country the silica came from, for example. The infrared microscopist identifies the organic materials. Our team at Argonne also included a nano-phase materials scientist, Richard Siegel, who determined how the materials formed a durable composite. Without that assistance we would have been only speculating. He gave us a real appreciation of the extensive work of the ancient artists who ground and blended the plaster substrate and pigment materials. We could see the extremely fine pigment materials interspersed with the plaster particles.

Our visiting art historian/preservationist, Diana Magaloni, oversaw of the project for the University of Mexico in Mexico City. She brought the materials to us and carefully catalogued all the data from several Mayan sites (Figure 2). Her expertise allowed us to determine the importance of what we were seeing. Since the facilities in Mexico City are always in great demand, Argonne's equipment was graciously made available for a few days. Our art historian learned that many areas of microscopy available to her provided exciting new information that allowed us to weave together a story of the site and its condition.

By encapsulating and sectioning small pieces of the murals, the microscope permitted us to see far



Figure 2. Mural sites

below the murals' surfaces without really damaging them. These murals are valuable works of art and, in many cases, the only record of the history of these vanished civilizations. Cross-sections were examined to determine the surface deterioration and weathering and the materials below the weathered surface. The samples reveal many things about the societies that made them: the materials that were available, which helps identify the location; the historical time period (evaluated by the degree of the processing); and the artists' adaptation to the environment by applying surface finishes. For example, Figures 3 and 4 show how microscopy can be used for approximating dating of the murals. Note the difference in grain size and shape in the two SEM micrographs. We expect many books to be published explaining the findings at the various sites, culminating in an overview that compares the sites and socieities.

It may be satisfying to work alone, as Pasteur did, but it is so much more satisfying to be part of a scientific team in which everyone makes a significant contribution. We microscopists are part of a world-wide "quality team"!

### RICHARD HOYTE LEE



Figure 3. SEM micrograph of primitive or early plaster-300-400 AD



Figure 4. SEM micrograph of advanced or late plaster-900 AD

### Can It Be Thirty Years Ago?

### Brian J. Ford \*

Least the spoken to Walter about a paper with a biological slant - I'd observed strange behaviour in leucocytes and we had obtained some images of these beautiful cells on 16 mm cine film. Walter explained that the INTER / MICRO meetings were normally confined to non-biological topics, but I said I wished to change that. It was time to bring in more biology. Walter thought for the merest moment before inviting me to make my presentation. That year, INTER/MICRO was in London (at Imperial College), and I spent a most enjoyable time with Walter and the other delegates. There was I, a tyro in his twenties; and there was Walter, already a formidable presence on the international scene - yet willing to embrace a new approach in the meetings with openness and immediacy. His example was one to which I immediately warmed.

My first INTER/MICRO visit to Chicago came in 1971, and I fell in love with this stunning city. The ebullient sense of culture and refinement was perfectly balanced with fine food and a love of living life. Since then I have visited every state (except Alaska and Kentucky, for some odd reason), many on numerous different occasions. I adore the West Coast, have revelled in the Keys, know New York better than many British cities, and have watched Pittsburgh and Philadelphia reborn. Yet Chicago has remained been my favourite in the US.

After one lecture, Walter simply remarked: "Well, nobody goes to sleep when Brian's on." In 1984 I was given that special invitation to be the banquet speaker. I remember it well; so do some of you, I think. Indeed I have given an evening lecture every year since then. After a few years, the "Evening with Brian J. Ford" became something shorter: "An evening with Brian." I remarked that this seemed a bit, well, informal; and I was told that being known for one's surname can become transmuted into first-name terms when one became established. What - like Galileo? Leonardo? "No, like Marilyn, or Elvis", I was told. My own approach to a lecture is simple: I listen to whatever advice Walter hands out as the hallmark of a good talk and then do the opposite. Once he said never to mix your media, so I made sure that the next year's 'Evening with …' had video, slides, and overheads all running at the same time. Recently Walter warned against showing too many transparencies, which is why I squeezed 150 slides into last year's half-hour presentation.

Much of my work has been influenced by Walter's clear insight, the breadth of his knowledge, his inspirational candor, and his wide-ranging interests. I rarely get through a paragraph of his without learning something new and mind-expanding. Walter has been acknowledged in several of my publications, and I owe him much. We also share an interest in underprivileged youngsters, and indeed I once gave a presentation as Walter's guest to the Chicago Social Services on my experiences in Britain as a foster-parent.

Above all stands his reputation as a fine scientist and an excellent microscopist. His role as *the* figurehead explains why the nation's leading optical microscopists gather here each year to celebrate our common interests. I have lectured around the world, from dusty lecture-theatres in India where the power fails every few minutes to large auditoria in the Far East crowded with beautiful, eager, scintillating students who hang on every word. I have chaired meetings at Oxford and Cambridge, hosted conferences from Australia to Scotland; but there is no meeting I am so anxious to attend as INTER / MICRO.

I salute Walter as a great figure of twentieth-century science. My own crude grasp of reality would be woefully inadequate without the annual infusion Walter administers. These INTER / MICRO symposia are like a high school reunion or an alumni meeting. They are aerobics for the microscopical mind. To attend is always a pleasure; to know Walter, and to interact with his fertile mind, is an honor and a pleasure.

Having been a small part of the action for thirty of those fifty years, I salute our friend and father-figure with warmth and admiration. I have learned far more than I could convey in all my lectures strung together.

Thanks, Walter.

<sup>\*</sup>Rothay House, Cambridgeshire, England

### **One Man's Odyssey Starting from Ithaca**

Walter C. McCrone\*

During the past 30-40 years light microscopy and, in particular, polarized light microscopy (PLM) as taught and applied by Émile M. Chamot and Clyde Mason in the States and by Norman Hartshorne and Alan Stuart in Britain has passed from zenith to nadir. Having lived through and survived this near-full eclipse of PLM, I may be able to record a close observer's account of the changes involved.

Professor Chamot began teaching courses in what he christened "Chemical Microscopy" at Cornell University in the 1890's. I was introduced to PLM by "Chammy" when I took his introductory course in 1936. I proceeded to absorb his enthusiasm for the subject and after a '38 B. Chem. degree, I managed to stay at Cornell through four years of graduate and two years of post-doctoral study concentrating on chemical microscopy. I was also fortunate to be a teaching assistant to Professor Mason during most of that period. I also spent several years on war-time research projects on high explosives and their problems. I worked especially on RDX and HMX, two "British" explosives. (I later learned that what I assumed was His Majesty's Explosive, HMX, was instead High Melting Explosive.) I was, however, able to use PLM to discover and characterize the four different crystal forms (polymorphs) of HMX. This project ended with the development of a plant production process used to produce millions of pounds of war-time RDX containing only the one safe crystal form of HMX. I also did most of the work on a 490-page report covering the microscopical characteristics of all known high explosives and their mixtures. This was used as a basis for simple microscopical procedures for the identification of enemy explosives (mines, bombs, and shells) collected as duds in the field.

All of this made me certain I wanted to spend my life applying PLM to production and processing problems in industry. I soon found that few company research directors know what a microscopist could do. One letter elicited the response "What is a 'chemical microscopist'?" I did find one sponsored research organization in Chicago, the Armour Research Foundation (now IITRI, Illinois Institute of Technology Research Institute) willing to take a chance on PLM and me. I stayed there 12 years, from 1944-1956 working on a wide variety of problems and products. I managed to do some research, some in connection with paid projects but most of it "extracurricular". I did publish 75 papers during these years and write the book Fusion Methods in Chemical Microscope. I also learned how to write proposals as well as develop and mange a group of 25 analytical chemists and physicists. Chemical microscopy was by then (1956) riding high in the sky. Many chemistry curricula in colleges and universities included required courses in chemical microscopy. Mary Willard at Penn State, Witt and Poe at the University of Colorado, Mason at Cornell, Norman Hartshorne at Leeds and many others were going strong and I felt secure enough to give up a regular paycheck to start McCrone Associates in Chicago.

There were signs about this time, however, that the future of PLM included competition with electron microscopy. The idea was voiced repeatedly that if the TEM could magnify objects 100,000X or more, why bother with a light microscope capable of 1,000X or a bit more? Mary Willard was not replaced when she retired at Penn State and this was repeated across the country as professors of chemical microscopy courses retired. Even at Cornell microscopy died in

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1980. Still, McCrone Associates was increasingly successful. True, they purchased a TEM, followed soon by a SEM, electron and ion microprobes, infrared spectrophotometers, X-ray diffractometers, etc. It is still highly successful (gross business in 1989 was in excess of \$11 million) and their approach to all problems and projects begins (and often ends) with the PLM.

In 1960, I organized McCrone Research Associates (MRA, Ltd) in London and McCrone Research Institute (McRI, Ltd. Inc.) in London and Chicago. The latter were designed principally for teaching microscopy courses. Both were successful although McRI, Ltd. became less needed as the RMS developed its excellent courses, and their teaching activities have now been absorbed by McCrone Scientific, Ltd.

The Chicago Institute now absorbs my full attention. Its nearly 100 intensive one-week courses this year require five full-time "professors" and other support staff. We have, since 1960, taught over 1,900 such courses to more than 20,000 students.

Other activities include an annual symposium we call INTER/MICRO, covering technical papers on new instrumentation, techniques and applications in light (and electron) microscopy. These meetings began in 1948 and, from 1963-1985, were held in odd years in England.

We see overall that light microscopy is still a healthy enterprise, at least with the RMS in Britain and with us in the United States. PLM is steadily proving its worth in solving problems. It has, for example, solved several highly visible problems in the face of failure by other so-called High-Tech methods and instruments. I refer, of course, to the Vinland map and the Turin Shroud. Both were shown, with certainty, to be fakes by PLM (in 1974 and 1979, respectively). No other tool or technique of those employed (PIXE, XRF, IR, etc.) has been able to come to any conclusion except that they don't know whether either one is authentic or not. The light microscopy work on the Vinland map and the Turin Shroud is noted but our conclusion that they are fakes is ignored. This is partly, at least, due to the fact the light microscope is no longer regarded as a 20th century analytical research tool. An entire generation of analytical chemists has not had the advantage of training in light microscopy. Few present-day research directors have taken academic courses in PLM and, therefore, cannot conceive that such training is essential or useful.

This is a serious situation for microscopy and microscopists. We must find a way to reverse the present attitude toward microscopy so that future generations of scientists will be able to learn light microscopy and gain acceptance of its findings.

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Walter C. McCrone