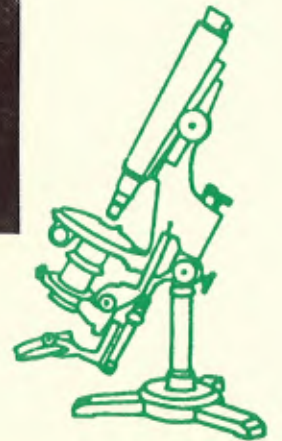
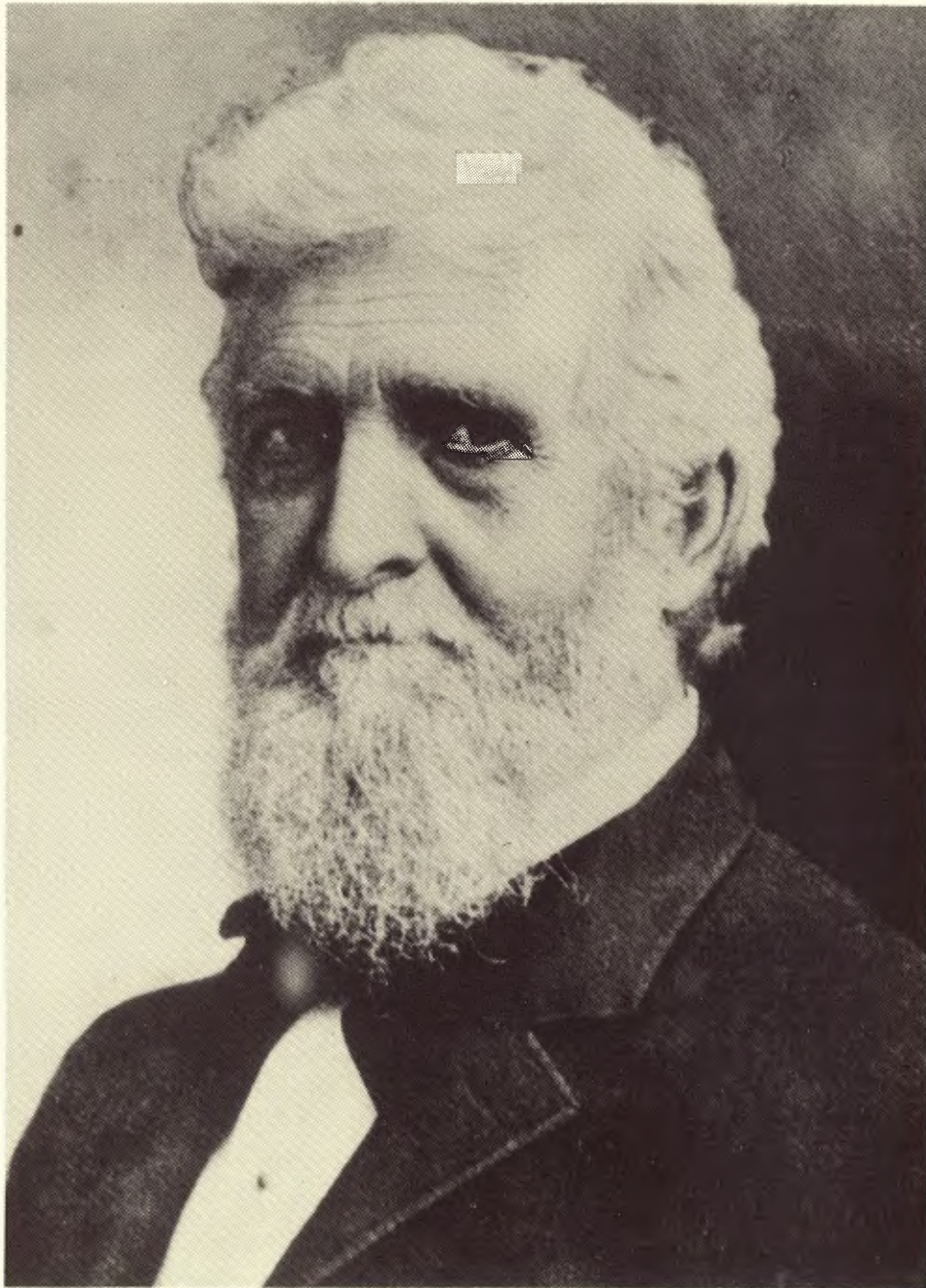


μ • NOTES 2000

Vol. 2 No. 1

March 1998



A Publication of the State Microscopical Society of Illinois

EDITORIAL

As a relatively new SMSI member, I began to ask questions about our society, its history, past officers, publications and, award recipients. I queried members and past officers, and although helpful, they could not begin to tell all of the myriad stories associated with the documenta and apparata in the SMSI room(s). There is a tremendous volume of HISTORY that awaits you or any member who enters our SMSI domain! *Frustrating, daunting, insurmountable, and intractable mess* are some of the words that come to mind upon first inspection. I prefer *ENTROPY*.

Major steps towards organizing the SMSI material began years ago; specifically, with the publication of the *SMSI Centennial Volume for 1969*! "Did I miss something?" some of you may ask. No, not at all. When this tome did not materialize, the SMSI Centennial Volume Publishing Committee circulated the following apology: "In addition to a world 'energy' shortage, we have been subjected to an energy shortage of our own! We plan to recover our normal publishing schedule in the Spring of 2069." I can assure you that due to major efforts by some of our members, the Centennial Volume is becoming a reality and will be published well before 200?.

Well, you may ask, why did it take me this much space, and in an editorial column, no less, to say this? Let me remind you that nearly 30 (THIRTY) additional years have passed since 1969! Our society is, as I mentioned to you in an editorial one year ago, a living entity; it does not stop growing or evolving! There is yet a new wealth of information to be organized, categorized, and yes, even purged after proper blessing. However, it must be done! Members must expend their free energy! It will not happen spontaneously. Otherwise, there will be a loss of information during the irreversible changes with time; hence, more entropy.

To remedy information loss, two articles in this issue of *μ•Notes 2000* are devoted to making some inroads to our past (Delly, Mikuska). Although the notion that a loss of information can be associated with frustration, the author of another article is frustrated by those who choose to ignore scientific information (McCrone), and yet another author is partly frustrated by too much information: a passion for music and strong interests in science (Maple).

One person to whom we are indebted for handling information related to microscopy is Arthur L.E. Barron, the founder and first editor of *The Microscope*. Mr. Barron is SMSI's Dr. August Köhler Award recipient for 1998.

Bill C. Mikuska

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All opinions expressed by contributing authors of μ•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.

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Founded In 1868

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Liquid Crystals

by

John Maple*

While basic science courses usually teach that there are three phases of matter, the solid, liquid, and gas phases, with some mention of the plasma phase, this conception is not completely accurate. Nature tempts man to explain phenomena by very simple rules, but so often provides exceptions and complications to these rules. The situation with phases of matter is a perfect example. Liquid crystalline phases are phases which generally have the outward appearance of fluids, but which contain greater ordering of the molecules than in a normal, isotropic liquid.

HISTORY

In the period from 1850-1888, European scientist Rudulf Virchow and German ophthalmologist C. Mettenheimer noticed that certain biological materials appeared to be fluids but produced unusual effects under polarized light normally associated with solid substances. In 1850 while studying fats, the chemist W. Heintz noticed that stearin turns cloudy at 52 °C, opaque at 58 °C, and clear at 62.5 °C, which he called a second melting point (4).

In 1888, Friederich Reinitzer, an Austrian botanist, studied the function of cholesterol in plants. He described cholesterol benzoate as having two melting points, one at 145.5 °C where the substance melts but remains cloudy, and the next at 178.5 °C where it turns clear (6). Some call Reinitzer the "discoverer" of liquid crystals, even though he mentioned Heintz to Otto Lehmann, another important figure in the study of liquid crystals (4). Others give credit to several additional researchers including L. Gattermann, D. Voländer, and R. Schenck. (9). However, Reinitzer is credited with bringing greater attention to liquid crystals and providing the impetus for further study (4).

The term "liquid crystal" is attributed to Otto Lehmann (6) who suggested the term based

on the fact that liquid crystals have the flow properties of liquids but many of the optical properties of solid crystals (4). The first literature citation of liquid crystals probably belongs to Edgar Allen Poe, however (9). Otto Lehmann began systematic studies of liquid crystals in 1899 (8). He was one of the early pioneers in the use of the polarized light microscope to which he incorporated the hot stage (4, 9). The polarized light microscope with a hot stage is one of most important tools for the study of liquid crystals (9).

Georges Friedel wrote a comprehensive paper on liquid crystals in 1922, and is the originator of the terminology for the three main phases of thermotropic liquid crystals, "nematic", "smectic", and "cholesteric" (6), which will be described shortly.

From 1922 until World War II, Carl Olseen of Sweden performed theoretical studies of liquid crystals and developed a "continuum theory" about the causes of liquid crystalline phases. In the late fifties, W. Maier and A. Saupe developed their own theory based on molecular statistics. The continuum theory is based on the conception of a liquid crystal as "an anisotropic medium with its own symmetry, viscosity, and elasticity parameters." The molecular statistisc approach is also called the swarm theory, where intermolecular forces are the basis of the theory (9).

The liquid crystal phase may be described as a phase where a substance has the freedom to flow like a liquid, but in which there is a certain degree of order in the arrangement of molecules within a sample. The liquid crystalline phase, also called a mesophase, may occur either within a certain temperature range for a given substance, or in a certain range of concentrations of a solute within a solvent. Substances exhibiting the former phenomenon are called thermotropic liquid crystals, while those exhibiting the latter are called lyotropic liquid crystals.

Within each of the two main types of liquid

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crystals there are certain common arrangements of molecules. The majority of substances exhibiting thermotropic mesophases consist of molecules which are elongated in shape. The remaining thermotropic liquid crystals have a disk-like shape, and are called discotic liquid crystals. Each of the two main types have their own forms of molecular arrangements in the mesophases.

PHASES OF ELONGATED THERMOTROPIC LIQUID CRYSTALS

If elongated molecules are arranged such that the long axes tend to point in the same direction, with no positional order, but are otherwise free to move and rotate, the nematic phase exists. The term nematic, which means thread (4), is based on the appearance of this phase when viewed between the crossed polars of a polarized light microscope (PLM), where long thin dark regions of extinction may be seen (9). The molecules are by no means all pointing in the same direction in this phase, however. The direction in which the long axes of these molecules are oriented is called the director. The director is actually a direction which describes the average orientation of the molecules at a given point in time. Or, it may be described as the average orientation of an individual molecule over a time period.

Various liquid crystalline substances or a single substance at different temperatures may exhibit differing degrees of uniformity of molecular orientation. Therefore, an order parameter, S , was devised to describe the degree of order. S equal to 0 describes an anisotropic liquid, and $S = 1$ describes a perfectly uniform arrangement. S may be calculated by this formula:

$$S = \frac{1}{2} \langle 3 \cos^2 \theta - 1 \rangle$$

where θ refers to the angle a molecule's long axis makes with the director, called n . The brackets indicate a statistical average (2). This average may be considered to be either the average of all the molecules in a sample, or the average orientations of a single molecule over a period of time. Experiments have never contradicted this assumption. Typical order parameters range from .3 to .9 (4).

The next major phase of the elongated type of liquid crystal is the smectic phase. In this

phase, the molecules are arranged in planes where the long axes of the molecules are oriented perpendicular to the plane. The arrangement is akin to a field of corn, where all the stalks are perpendicular to the surface of the earth, which corresponds to the plane (Figure 1). The molecules may either be arranged in rows, according to the corn field analogy, or at random, though still within the plane. The planes are free to slide over one another (6). In reality, this description is a little inadequate, because there are still many molecules between these layers, and some part-way within each layer. Chandrasekhar describes the phase as "an orientationally ordered fluid on which is superimposed a one-dimensional density wave" (2).

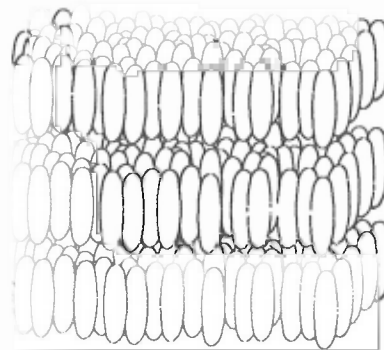


Figure 1.

The third major phase is the cholesteric, or chiral nematic phase. In this phase, the elongated molecules are arranged in planes as in the smectic phases, but lie parallel and not perpendicular to the planes, and in this regard they resemble the nematic phase. The molecules in a plane are oriented in the same direction, but in adjacent planes, the orientation shifts slightly. Over successive layers, the direction of orientation changes considerably, tracing a helical path (Figure 2) (6). This phase is called cholesteric simply because many of the earliest known substances exhibiting this phase were derivatives of cholesterol, though cholesterol itself does not form any mesophases. The term chiral nematic is more descriptive, since within each plane the molecules are arranged according to the nematic phase, but the molecules have a definite "handedness" to their shape, therefore, causing each successive layer to be shifted slightly. Typically, these molecules may be very flat but with side

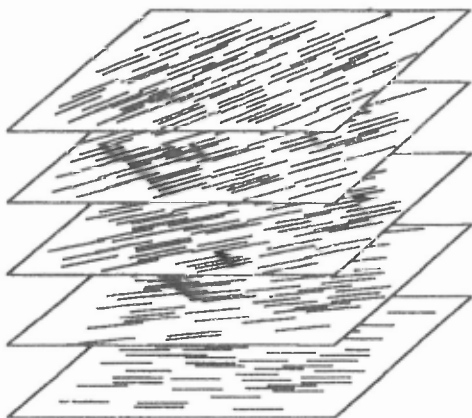


Figure 2.

chains of methyl groups (CH_3), for example, projecting upward, causing the axes of molecules in adjacent planes to shift slightly (6). There are many variations of smectic phases with a corresponding system of notation, where a letter ranging from A-K designates the phase. The letters apply to the various phases according to chronological order of discovery (9).

The smectic arrangement previously described was the first of the smectic phases to be discovered, and is called smectic A, or S_A .

While the smectic A phase has no positional order, smectic B, S_B , does. Here the analogy of the corn field with the stalks in rows is more appropriate, except that the molecules are in a hexagonal arrangement, with just enough space between the molecules to allow cooperative rotation. There is also a correlation between layers much as there is in a three-dimensional solid. Molecules in adjacent layers may either be above the interstitial holes or not, producing either an AAA form or an ABA form. If the molecules in adjacent layer are above interstitial holes, the layer below may cover either the same holes or the ones not covered, producing an ABC structure, just as is possible in a hexagonal close-packed crystalline solid (7). As in the smectic A phase, the molecules are perpendicular to the plane of the layer.

Smectic C applies to an arrangement similar to S_A , but with the molecules inclined with respect to the plane of the layer (2). There is no order within the layer as is the case in smectic A.

Smectic D is an arrangement which has not yet been determined, but which may have a type of micelle structure with about 10^3 molecules per

unit, and which should not be called a smectic phase (2). Some believe that there is some sort of cubic arrangement of the micelles. In any case, it is an optically isotropic phase and may be mistaken for an isotropic liquid, except that it is much more viscous (7).

The smectic E phase is a three-dimensional crystal structure with an "interlayer herringbone arrangement of the molecules" (2). This phase is very similar to the smectic B phase, except that the molecules are a bit closer together, and are not free to rotate 360° , but rather oscillate less than 180° . Contraction of the otherwise hexagonal lattice produces an orthorhombic lattice. Because the molecules may not be exactly cylindrical, they pack in a "herringbone" arrangement, where the molecules in each row are rotated from those in adjacent rows; the longest horizontal axes of the molecules' elliptical cross sections are rotated with respect to the axes of the rows. The molecules are still perpendicular to the layer, however. X-ray diffraction studies indicate that there is some sort of correlation of position between the layers (7).

In the smectic F phase, the molecules are arranged hexagonally within each layer, but the molecules are tilted with respect to the plane of the layer. To be precise, the lattice must be considered "C-centered monoclinic" when taking the tilt into account (2). The packing is in fact hexagonal close packed when considering the lattice from the plane perpendicular to the tilt direction. The tilt is toward the edge of the hexagonal lattice. Furthermore, while there is little positional correlation between the layers, the orientation of the axes of the hexagonal lattices within the various layers tends to remain the same from layer to layer. In other words, the layers are free to slide over one another, but not to rotate. A substance exhibiting the F phase will exhibit the C phase on heating, and therefore, the F phase is similar to the C phase but with less entropy (7).

The smectic G phase is similar to F in that it also has tilted molecules in the pseudo-hexagonal arrangement, but it differs in that the ends of molecules in each successive layer tend to line up with one another, the layers are correlated. Furthermore, the molecules are spaced closely enough that they oscillate cooperatively and are not free to rotate around their long axes independently of neighboring molecules. This means that over a local area the molecules have a

chevron arrangement at any given point in time (7). Here the tilt is toward the edge of the lattice (2).

The smectic H phase is the tilted version of smectic E. Again the tilt is toward an edge of the lattice (2). The pseudo-hexagonal lattice is even more distorted by the tilt in addition to distortion from contraction, and so the precise structure is monoclinic. There is positional order from layer to layer, and so the phase is considered to be of the "crystal type."

The smectic I phase has the same structure as smectic F, except that the tilt axis is toward a vertex of the hexagonal lattice. Again, distortion of the lattice from tilting requires the structure to be called monoclinic (7), and the layers are uncorrelated.

Smectic J is just like smectic G, except that the tilt is again toward the apex of the pseudo-hexagonal lattice, not the edge. It differs from smectic I in that the layers are correlated. The phase similar to the H phase, having the contracted pseudo-hexagonal lattice, but with the tilt toward the apex of the lattice is smectic K (2, 7).

Many of the smectic phases which incorporate tilted molecules with respect to the plane of the layers have chiral analogues, much like the chiral nematic phase. The tilt of the molecules causes a directional orientation relative to the axes of the layer planes, and this direction may shift slightly in successive layers, producing a helical twist with the twist axis perpendicular to the layer planes (Figure 3). The chiral smectic phases are designated by an asterisk, and are

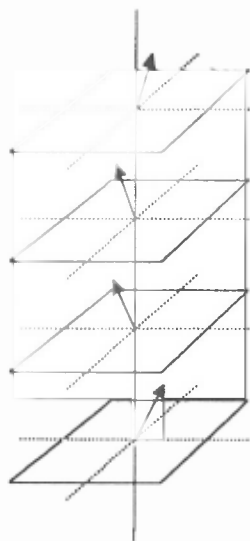


Figure 3.

found to include smectic C*, F*, H*, I*, J*, and K* (2). Single component G phases do not exhibit chirality (7), and some lists exclude G* (2).

The main method for distinguishing these phases has been miscibility. The assumption is that two phases will not mix unless they possess the same type of symmetry. X-ray studies have provided greater insight into these structures since the 1980's (2).

It is important to note that a single substance may exhibit more than one phase. A single substance may not exhibit both the nematic and cholesteric phases, but may exhibit either the nematic or cholesteric and a smectic phase (6). In fact, a substance may exhibit several of the smectic phases in addition to the nematic or cholesteric phases. An example is the mesogen TBBA, or terephthalidene-bis-(4-*n*-butylaniline). It is crystalline up to 113 °C, exhibits Smectic H (S_H) up to 144.5 °C, S_C up to 172.5 °C, S_A up to 199.6 °C, and exhibits the nematic phase up to 236.5 °C, after which it forms an isotropic liquid (9).

As the example of TBBA illustrates, certain smectic phases are characteristic of the lower temperatures, because these phases exhibit greater "short range order symmetry". The order of the smectic phases from lowest to highest order is: A<D<C<B<E<F, G, H...(9).

PHASES OF DISCOTIC THERMOTROPIC LIQUID CRYSTALS

The discotic liquid crystals were discovered much more recently than the more common elongated type, having been found in India in 1977. These substances are composed of molecules which have a flat, disk-like shape, and which form liquid crystal phases which are distinct from the phases exhibited by the more common elongated mesogens. These phases are called columnar, canonic, or discotic. The basic structure is one where the flat molecules are stacked "aperiodically" one on top of the other in columns (Figure 4). The molecules may also be stacked in a regular, or periodic fashion, or at an angle tilted from the perpendicular to the column axis. These columns may be arranged in a hexagonal or rectangular arrangement. There is also a nematic-like arrangement of discotic molecules where the planes of the molecules are parallel to each other, much like coins spilled randomly on a surface (1). Like the true nematic mesogens, there exists a twisted nematic phase

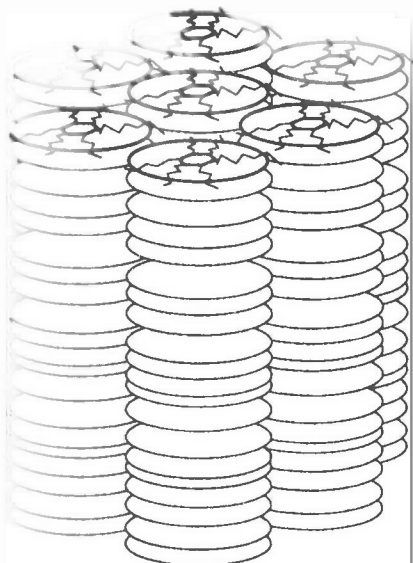


Figure 4.

Symbols for Discotic Phases (2)

D_{hd}	Columnar, hexagonal disordered	D_A
D_{rd}	Columnar, rectangular disordered	D_B
D_{ho}	Columnar, hexagonal ordered	---
---	-----	D_C
D_t	Columnar, tilted	D_L
N_D	Nematic discotic	D_F
N_D^*	Twisted nematic discotic	

for discotic liquid crystals, in which the molecules may be grouped into planes where each plane contains disk-like molecules with their own planes approximately perpendicular to the plane of the layer, and in which the orientation of the director of each layer is adjusted slightly in each successive layer, producing a helical pitch just as in the regular chiral nematic phase.

The following figure shows the arrangement

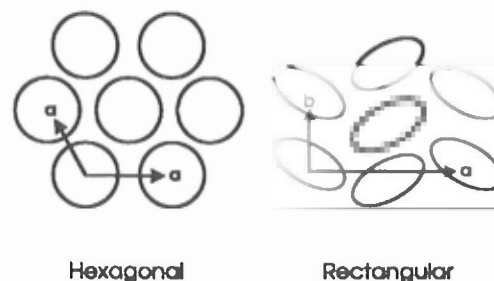


Figure 5.

of columns in discotic phases. The elliptical columns designate molecules tilted relative to the axes of the columns (1).

PHASES OF LYOTROPIC LIQUID CRYSTALS

While thermotropic liquid crystals constitute a very considerable number of liquid crystalline substances, there are also many lyotropic mesogens, having their own phases and textures, as do the discotic thermotropic mesogens. Lyotropic mesogens are always amphiphilic, where dissolution in either a polar or nonpolar solvent causes the molecules to orient with the polar head in contact with a polar solvent, and the nonpolar tails to orient away from a polar solvent, with the reverse situation in a nonpolar solvent.

The phases of lyotropic crystals can be generally separated into the micelle type phases, or middle phases, and the lamellar phases, or neat phases. Much of the terminology is borrowed from the traditional terms describing soaps, which are lyotropic mesogens.

The lamellar structure is similar to the smectic phase with the molecules arranged in layers, but are two molecular lengths thick, with either the polar heads or nonpolar tails all toward the center of the layer, depending on whether the solvent is polar or nonpolar (9). The D phase has relatively straight layers, and the B phase has a wavy structure, with adjacent layers bending in the same direction.

There are many micelle type phases, where the molecules are arranged in sphere-like structures or in columns or rods. Phase C contains rod-like micelles with square cross sections in a two-dimensional tetragonal arrangement, where the nonpolar tails fill the center of the micelles (Figure 6). Micelles having a nonpolar core are

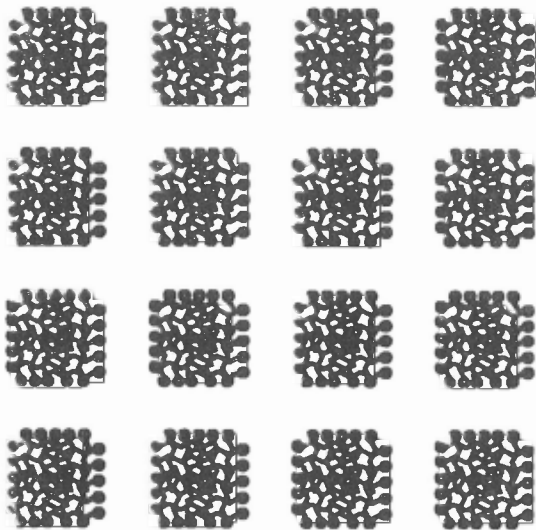


Figure 6.

called normal, while micelles having a water core are called reversed. Phase K is the opposite arrangement where the nonpolar tails create a hydrocarbon environment, with micelles having a center composed of water. Phase R consists of rectangular cross sectioned, rod-like micelles with the normal, hydrocarbon, or nonpolar cores. The micelle arrangement is orthorhombic.

Rod-like micelles with circular cross sections of the normal type and arranged in a hexagonal structure are called Phase E. The same arrangement with reversed micelles produces the F phase.

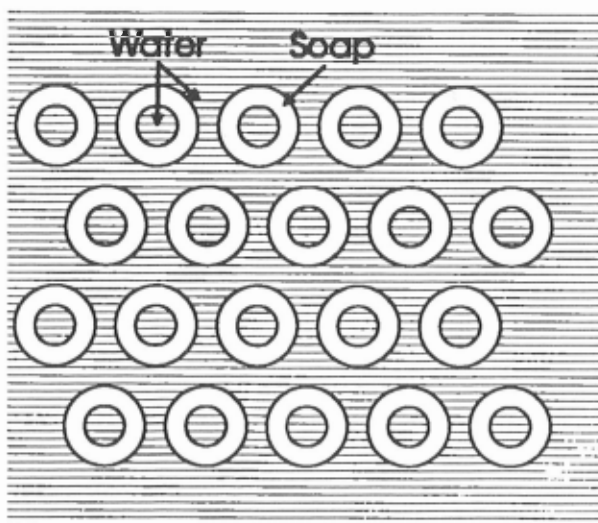


Figure 7.

Phase H_c consists of rod-like micelles with "hollow" centers called complex micelles (Figure 7). Complex micelles exist in a water environment with walls consisting of the amphiphile or soap, and having a water core. The micelles are in a two-dimensional array.

Spherical micelles of normal structure, arranged in a face-centered cubic lattice form the I_{f1} phase. Reversed spherical micelles produce the I_{f2} phase. Complex spherical micelles, that is, a water environment, "walls" made of the amphiphile, and a water core, arranged in a face-centered cubic lattice constitute phase I_{fc} . There is also a normal body-centered cubic lattice, I_{b1} , and a complex body-centered cubic lattice, Phase I_{bc} . This is not an exhaustive listing of the phases. There are still other phases borrowed from the description of soaps, such as waxy soap or curd fiber phase, each occurring at increasing concentrations of soap (9).

In addition to binary systems, liquid crystalline phases occur in ternary and multicomponent systems.

Lytotropic mesogens may be a soap, having a formula such as KC_{14} , NaC_{14} , KC_{18} , NaC_{18} , etc., or any amphiphilic compound. Especially important in biological systems are phospholipids which constitute cell membranes, which in themselves are actually lyotropic liquid crystals. The nature of liquid crystalline phases is essential to life, where a cell membrane must present some means of separating components of a cell from its surroundings, while allowing the transport of essential materials through the membrane when necessary. It is the delicate balance between a liquid crystal's order and fluidity that allows this situation (4).

TEXTURES

While two different samples of the same substance may exhibit the same phase, they may not necessarily look the same, especially when viewed by PLM. This is due to the fact that two samples may have differing textures, or long range pattern of the director. The polarized light microscope is especially useful in the study of liquid crystals in this regard, for certain textures produce certain characteristic patterns between crossed polars.

A homogenous sample, which only occurs in nematogens (9), is one in which the director is constant throughout the sample. This term gen-

erally refers to a sample where the director is parallel to the surface boundaries of the sample. A large sample of any liquid crystal will generally not be homogeneous by any means, but a small sample under the coverslip on a microscope slide, for example, may be induced toward a homogeneous condition by proper preparation of the boundary surface. One such method is to apply a thin coat of a long chain polymer to the surface of the glass, then to stroke the surface in one direction with a soft material (4). Tiny grooves created by rubbing the surface will tend to orient the long axes of the molecules of a nematogen in the same direction. Another method is to evaporate SiO_2 , MgF_2 , Bi_2O_3 or Au onto the glass surface at an oblique angle (5). The homogeneous texture is especially important in the production of display devices.

Related to the homogeneous texture is the homeotropic texture, in which the director of the molecules is perpendicular to the surface boundary of the sample (8). Such an orientation may be achieved by coating the coverslip of a slide with a thin film of lecithin or some other amphiphilic compound (9). If the nematogen has a polar structure, the amphiphile will induce the corresponding end of the permanent dipole of the molecules to orient toward or away from the surface. The homeotropic texture is also important in the production of display devices (4).

The textures which are characteristic to the various phases and which can be used to identify a liquid crystal phase using PLM are not homogenous or homeotropic, but rather contain disinclinations, or points or lines where the director of the sample changes direction abruptly. Such disinclinations result in distinctive patterns of extinction which are characteristic to the various phases. As already mentioned, the nematic phase is called such based on the observation of thin thread-like patterns of extinction.

In discussing these textures, it is important to recognize the basis for the observation of such phenomena as extinction. The elongated shape of the constituent molecules causes them to be anisotropic, and the degree of order present in a sample in a liquid crystalline phase allows these anisotropic properties to be observed. Liquid crystals may exhibit the same sort of anisotropic properties found in solid crystals, such as birefringence. While an isotropic liquid may also be composed of elongated molecules, the arbitrary arrangement cancels out the anisotropic proper-

ties of individual molecules. In a liquid crystalline phase, the general order of a sample allows such anisotropic properties of the individual molecules to affect the properties of the entire sample or region of a sample. Birefringence is, of course, the cause of observable extinction in the case of liquid crystals between crossed polars, where the consistent orientation of molecules in a given region may or may not allow the passage of polarized light through the analyzer. The orientation of the director may change near a disinclination producing a pattern of extinction characteristic to the particular mesogen.

The so-called Schlieren texture is characteristic of smectogens and nematogens (9), where extinction may be seen in "windmill" patterns converging onto points. The pattern results from the director orientation rotating continuously about the point disinclinations. Actually, these points are line disinclinations perpendicular to the plane of the sample on a microscope slide (2). A sample may have a rather "busy" appearance with many groups of "windmills" emanating from many points. The Schlieren texture may be seen by placing several small crystals of a mesogen on a slide which has not been treated in any way. The sample is then heated until it melts (9). If the sample is thick enough, it may exhibit long thin extinction lines connecting the point disinclinations, these are the nematic threads after which the phase is named. These nematic threads are then a form of the Schlieren texture.

The cholesteric phase also produces some interesting textures. One is the fingerprint texture, where the helical axis is along the plane of a microscope slide when viewed between crossed polars. Long thin figures may be observed which are nested against each other and which tend to form branches which may continue or stop. The lines coincide with individual half turns of the director. The overall effect resembles a fingerprint. If the helical axis is perpendicular to the plane of the slide, the Grandjean texture results, where an arbitrary web of interconnected line disinclinations forms against a predominantly clear background (4).

When the helical axis of a cholesteric liquid-crystal is perpendicular to the surface of a slide, a special technique may be employed to measure the pitch of the helix. By using a Cano wedge, a wedge-shaped piece of glass, in place of a coverslip (4), and by treatment of the surfaces to ensure that the molecules are oriented uniformly

and parallel to the surfaces, a series of lines will be observed when viewed between crossed polars. The distance between these lines permits the calculation of the helical pitch of the cholesteric sample based on the known angle of the wedge. Forcing the surface molecules to orient a given way causes a distortion in the total number of helical rotations that would otherwise occur over each given distance between the plates. At a thickness where too great a distortion occurs, that is, where the distortion is greater than 1/2 turn, the number of rotations changes abruptly producing a visible line. This number of rotations will then remain constant for a particular range of distances between the plates, corresponding to the distance observed between the lines of disinclination. This texture is called the Grandjean-Cano texture (2).

The smectic phase A may produce a focal-conic texture, where beautiful fan-like patterns result from cone-shaped disinclinations emanating from points.

There are still other unique textures, such as the nematic spherulite, in which nematic type molecules are arranged in a ball such that C_{∞} symmetry exists. These spherulites occur when a nematogen is cooled from the isotropic phase (9). The director may emanate from a point in the center of the sphere, or have a bipolar structure with the director parallel to the surface with point disinclinations at opposite poles of the sphere (2).

The various disinclinations or "singularities" of a sample are assigned a number, s , equal to 1/4 the number of brushes or "windmill" arms of extinction viewed between crossed polars. If rotating the polarizer causes the brushes to rotate in the same direction, s is positive. If the brushes rotate in the opposite direction, s is negative. Brushes of opposite sign and equal absolute value will tend to coalesce and extinguish each other, while brushes of opposite sign but differing absolute value will coalesce to create new disinclinations equal to the sum of the constituent singularities. Disinclinations of equal sign tend to repel each other (2).

The various defect structures of liquid crystals are one of the more interesting and unique aspects of the mesophases. The polarized light microscope is consequently a very important tool in the study of liquid crystals.

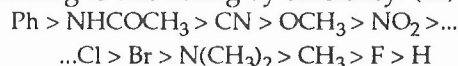
MOLECULAR STRUCTURE OF THERMOTROPIC LIQUID CRYSTALS

The structure of molecules of thermotropic liquid crystals requires either an elongated or disk-like shape. The elongated types generally have a rigid center, with more flexible ends. Typically they consist of one or more aromatic rings, either with or without a bridging group between the rings, and with some type of terminal group on opposite ends of the terminal carbon rings. The prevalence of carbon compounds as liquid crystals has partly to do with their thermal stability and relative ease of synthesis (11). Carbon rings help to provide a rigid framework for the molecule. If a molecule is too flexible it will not tend to form a mesophase. On the other hand, if just the ends of a molecule are somewhat flexible, a mesophase is more likely to form. A rigid middle region with flexible ends is then the ideal construction for an elongated thermotropic liquid crystal (4).

The search for nematogens suitable for display technology necessitated obtaining a nematogen with good thermal stability and a low isotropic to nematic transition temperature to ensure a liquid crystal state at room temperature. Experiments showed that different members of a homologous series had different transition temperatures, and allowed the discovery of compounds with the desired combination of attributes. Some of the first molecules found suitable for display technology had the structure of:



The R group consisted of alkyl groups (C_nH_{2n+1}) where n ranged from 1 to 12. The same structure used with alkoxy groups ($C_nH_{2n+1}O$) produced higher transition temperatures, and in general it was found that certain terminal groups produced lower transition temperatures than others according to this listing by efficiency: (11)



Terminal groups which increase the molecules'

overall length without increasing the width tend to promote better thermal stability and greater anisotropy of molecular polarizability.

Researchers found that as a homologous series is ascended, there is a tendency for the transition temperature to alternate up and down with each increase of n : this is called the odd even effect. It can be explained where the terminal chain produces a zig-zag pattern. In a terminal chain of carbon atoms, for example, even numbered carbon atoms may be along the major axis of the molecule, while the odd numbered carbons are not. Carbon atoms along the axis have the effect of enhancing the anisotropy of the molecules, while the other atoms decrease the anisotropy (2). According to the theory of Maier and Saupe, there is a link between the nematic to isotropic phase transition temperature and the anisotropy of molecular polarizability, $\Delta\alpha$ (11). The even carbons will cause relatively low transition temperatures, while the odds will cause high transition temperatures. As the terminal chains become longer, transition temperatures may decrease until a minimum is reached, after which the long chains begin to interfere with the ordering of the molecules. Furthermore, with increasing chain length, the difference between the odd and even transition temperatures tends to decrease as the flexibility of the end chains increases. See Figure 8 for the molecular structures of some *p*-azoxyanisoles, PAA's (2) and Figure 9 for a graph of the phase transition temperatures of a homologous series of PAA's (4).

The strong π bond found in the linkage group N_2O between the carbon rings in the above example is a characteristic feature of linkage groups of nematogens. The linkage groups must provide rigidity to preserve the linearity of the molecule, and lacking any associated hydrogen atoms, that is, unsaturated, it promotes conjugation between the two carbon rings. Good linkage groups for nematogens are listed below (11):

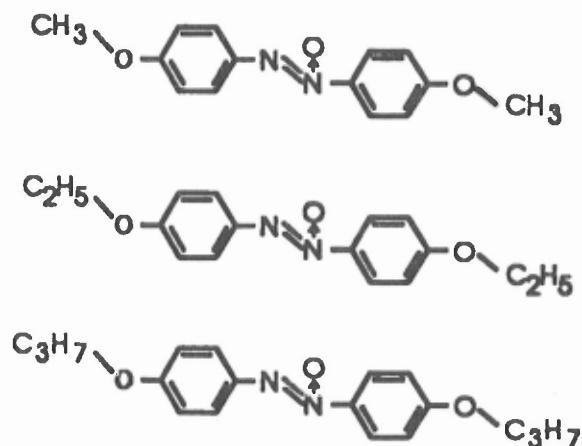
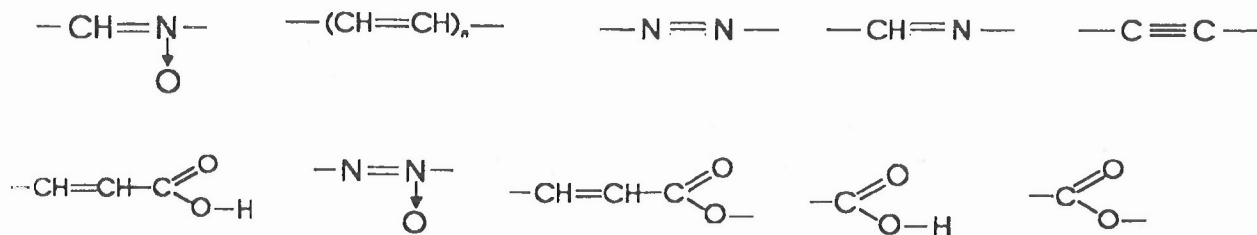


Figure 8. The first three members of the *p*-azoxyanisole (PAA) homologous series.

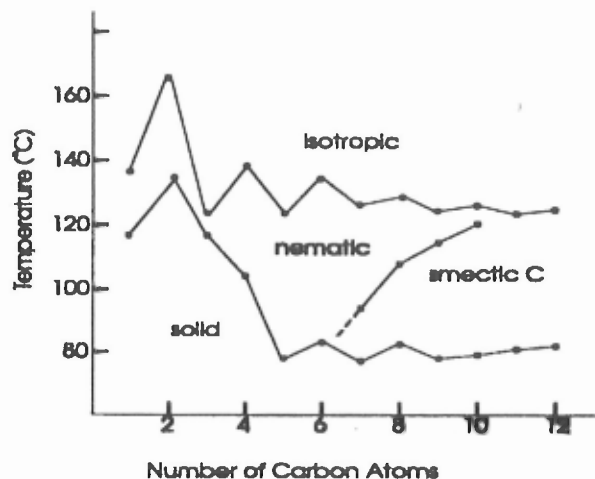
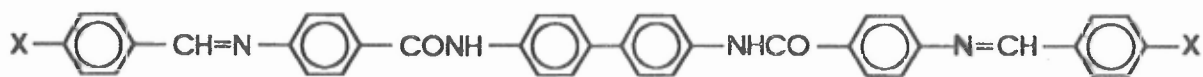


Figure 9. Graph of the phase transition temperatures of the first 12 members of the homologous series of PAA.



Structure of benzidine-bis-(*p*-benzylidenaminobenzoic amide)

Many linkage groups, however, have been found in general to be chemically unstable under high temperature, moisture or ultraviolet light conditions. The discovery of nematogens with carbon rings linked directly together paved the way for their use in display devices (4). The 4-*n*-alkyl- and 4-*n*-alkoxy-4'-cyanobiphenyls illustrated previously on page 8 are examples of such species (11). See structures A and B, respectively. These compounds have room temperature nematic ranges, chemical stability, high resistivity and a large dielectric anisotropy ($\Delta\epsilon$ ca +11) (11). Since the dielectric constant of a material is in effect a measure of its polarizability (10), having a large dielectric anisotropy means that the material may be influenced easily by the presence of an electric field in a display device, an important characteristic for a material being considered for display technology (11).

There are, of course, many other variations in the structures of the elongated mesogens, some with bridging groups, some without. Some have multiple bridging groups. Some have single carbon rings, others have multiple carbon rings. An example of a heteroaromatic liquid crystal with bridging groups is benzidine-bis-(*p*-benzylidenaminobenzoic amide), a carboxylic acid amide illustrated above (9).

An example of a discotic liquid crystal structure is that of the hexa-*n*-alkanoates of benzene, the first discotic liquid crystals to be discovered (1) (Figure 10). Evidence indicates that these particular discotic liquid crystals have a rigid core, much like the elongated, thermotropic mesogens, with flexible ends. The basis for this conclusion is the observation of circular domains of the liquid crystal using PLM, where the lines of extinction do not coincide with the polarizer-analyzer directions, but rather are inclined at a thirty degree angle. This implies that the molecules are arranged in a tilted manner relative to the column axis. If the whole molecule were a rigid disk, there would be a corresponding distortion of the hexagonal lattice under X-ray analysis. There is no such distortion, so the conclusion is that the "arms" are flexible, as they are in the

elongated mesogens (1).

APPLICATIONS

Liquid crystals have been employed in a great many applications, ranging from thermometers to gas vapor detectors to computer screens. While each application involves certain techniques, nearly all liquid crystal-based devices depend on the concept of a change in the order parameter of a liquid crystal sample, which in turn may be detected through the use of polarized light, sometimes in conjunction with dichroic dyes. The latent heat of transition from the solid phase to the liquid crystal phase is a large percentage of the latent heat of transition for the overall solid to isotropic liquid transition. For example, the latent heats of transition for cholesterol myristate are 65 cal/gram for the solid to liquid crystal transition, and 7 cal/gram for the liquid crystal to isotropic liquid transition. Relatively little energy is needed to effect a change in the phase or the order parameter. This fact is the basis for use in digital displays of

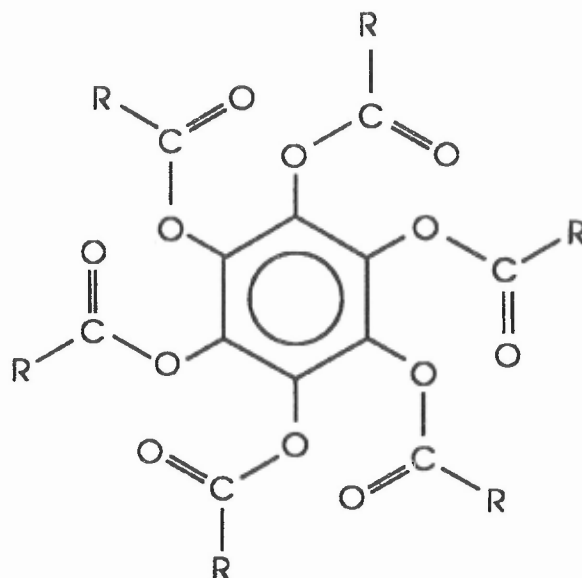


Figure 10. Hexa-*n*-alkanoates of Benzene ($R = n\text{-C}_n\text{H}_{2n+1}$)

watches and calculators, for example, where conservation of power is desired for long battery life. Liquid crystal thermometers may change color through the whole range of the visible spectrum for a change in temperature of one Celsius degree (4). Also, liquid crystal vapor detectors may register vapor amounts in the order of parts per million (6).

Substances exhibiting liquid crystalline phases are truly fascinating and useful. It seems ironic that such an important phase of matter, upon which life itself depends, has been known definitively for less than 150 years. It will be interesting to see what advances the study of these substances will bring to pure science as well as to applications.

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EDITORS NOTE:

John Maple wrote this paper during the spring of 1997 to fulfill a requirement for my second semester freshman chemistry class at Triton College.

Only a few weeks ago when I requested formal permission to publish his paper did I learn that John is and has been a member of the Nashville Symphony since 1984 and had merely taken a temporary leave of absence to pursue another of his interests, computer science!

McCrone Research Institute Flexi-Grad Program

The McCrone Research Institute (McRI) announces a Flexible Graduate Program in Light Microscopy. Few schools teach light microscopy and degree programs in microscopy are essentially non-existent. Biologists, mineralogists and metallographers still use light microscopes but with little training in microscopy. Biologists are presented with the microscope but, nearly always, with no instruction to help them achieve good images. Mineralogists do a little better because they do learn how to use the microscope for optical crystallographic identification of minerals. At McRI, we have always stressed Köhler illumination to obtain the best possible image followed by detailed study of each of the basic light microscopy techniques. Once these techniques are learned they can be successfully applied to simple problems of characterization, identification and comparison of subnanogram particles, quality control, failure or contamination analysis, trouble-shooting in general, as well as general process and product problems or development in industry, study of forensic trace evidence, pigment identification for art authentication, and correlation of performance with microscopical features (for polymers in any form, abrasives, pigments, ceramics, foods, cement, etc.).

McRI will offer Bachelors, Masters and Doctorates in microscopy. To see what we mean by a Flexi-Grad Program, please read the following.

The requirements for enrollment in this program irrespective of the desired degree include the following:

- 1.) You must already have at least 50 college credit hours in approved chemistry and physics courses. Other associated-discipline courses in geology, mineralogy, etc., may be substituted for up to 12 of the 50 hours. An additional 50 hours in other approved college courses are also required.
- 2.) You must be willing to complete at least a minimum number of McRI core courses (5 for the

Undergraduate Degree, 8 for the Master's Degree and 12 for the Ph.D. Degree) chosen from our course catalog. Each core course may include assignment of a small project (approximately 30 hours) to be completed back home for the 3-credit hours. An additional requirement will include one or more thesis courses (one for Bachelor's Degree, two for a Master's and three for a Ph.D). The numbers signify the increasing time and sophistication of the thesis program going from bachelors to Ph.D. In effect, the three thesis courses for the Ph.D. represent a single 9-credit hour course. These thesis research courses cover the costs of directing and administering your thesis work. The undergraduate candidate pays for his thesis course at the start; the Master's candidate pays at the start for one and at the end for the second; the Ph.D. candidate pays for his three thesis courses at the start, middle and end of his research.

- 3.) You must be employed in a position where microscopy will be useful and where you have the approval and support of your supervisor. You or your employer must have a polarized light microscope and accessories available to you for the supplementary course, projects and the thesis research.

- 4.) You must send in an application which will include your résumé, transcript of your college record, a statement of your present status microscopically, and your goal.

There is no residency requirement at McRI other than the one-week time for each course you take. The only costs are the course tuition fees now (1998) \$850-\$1100/course. The individual course projects and the thesis research subject will be mutually agreed by McRI, you (and your employer, if appropriate). We will usually be able to suggest topics of direct interest to both (all three?) parties and leading to one or more publications.

We invite further inquiries. Call or write Walter C. McCrone, Director: McCrone Research Institute, 2820 South Michigan Avenue, Chicago, IL 60616-3292, (312) 842-7100; FAX (312) 842-1078.

Making a Microscope Slide Ring Oven

James J. Benko*

I believe that the small book *MICRO-ANALYSIS BY THE RING OVEN TECHNIQUE* by Dr. Herbert Weisz is one of the most useful books in analytical chemistry. It details the use of the ring oven for separating many inorganic species, provides complete separation schemes for most common metals in qualitative analysis, and also gives many spot tests for identifying these metals. Directions for making several useful gadgets involved in microchemistry are also provided. The first edition was published by Pergamon Press in 1961 with a second edition printed in 1970. I have seen copies in used book stores every now and then. I have adapted many of the methods described in this book for microchemical methods performed on microscope slides, some of which are detailed below.

While a commercial ring oven, such as the Thomas Trace Oven at one time sold by A.H. Thomas (shown in Figure 1) will do everything a ring oven is designed to do, crude versions are so easy to make that it becomes difficult to sell commercial versions. This fact coupled with the trend away from microchemical methods to instrumental methods makes it impossible to buy a commercial version today. Yet, the ring oven is a valuable laboratory tool that can be used to great advantage and is a useful item to keep in your "bag of tricks." Basically, the ring oven serves as a separation tool utilizing the different solubilities of chemicals in different rinse reagents. A small circle of filter paper is placed on the oven and weighted down with the ring. A drop of sample from a capillary tube is placed in the center of the paper. The wash solution is then dropped slowly onto the center of this drop, thereby washing soluble ions from the center into a wash zone which radiates out from the center. The heated ring of the ring oven prevents the wash zone from reaching the outer edge of the filter paper and



Figure 1. Commercial Ring Oven

* Microspec Analytical, 3352 128th Ave., Holland, MI 49423

allows concentration of the soluble components into a thin ring. The filter paper circle after drying can be cut into many pie slices allowing for a large number of separate spot tests for compounds in both the soluble and insoluble ring zones. A whole gamut of separation possibilities is available if one substitutes ion exchange and thin layer media for filter paper. The temperature of the ring oven can be varied for other nonaqueous solvents.

A crude, but serviceable ring oven can be made from a large washer, some capillary tubes, filter paper circles, and a small hot plate. The washer is put over the paper and slide as shown in Figure 2. The hot plate is then set at the desired temperature to allow the wash solution to penetrate the center zone and wash soluble components from the center drop to the edge.

Rather than use a hot plate, I have devised a crude hot stage made from a block of aluminum with holes cut through the center and sides as shown in Figure 3. The center hole serves as a light source to view micro reactions when used as a hot stage. The holes in the side are for a thermometer and soldering iron heater. The iron will provide a temperature rise of 1-2° /minute when

plugged directly in a 110 volt outlet. This block can be used both as a low temperature hot stage and ring oven.

For a practice run that illustrates the use of the ring oven one can try separations of iron and copper ions. For this experiment prepare solutions containing 0.1% cupric chloride and 0.1% ferric chloride in deionized water. Place a circle of filter paper on a microscope slide. Then place the slide on a hot plate set at low heat. Place a metal washer over the filter paper circle on the slide. Allow the slide to come to thermal equilibrium on the hot plate. Place a small drop of each of the solutions in the center of the filter paper circle using micro capillary tubes. Slowly add drops of dilute aqueous ammonia solution to the center of the paper. The alkalinity of the aqueous ammonia immediately precipitates ferric ions to produced ferric hydroxide. The copper ions are solubilized into a blue copper ammonium complex and are washed from the center to outer edges of the filter paper. Eventually the copper will be concentrated into a ring zone like that in Figure 4.

In a similar manner other separations can be made with various precipitating and wash

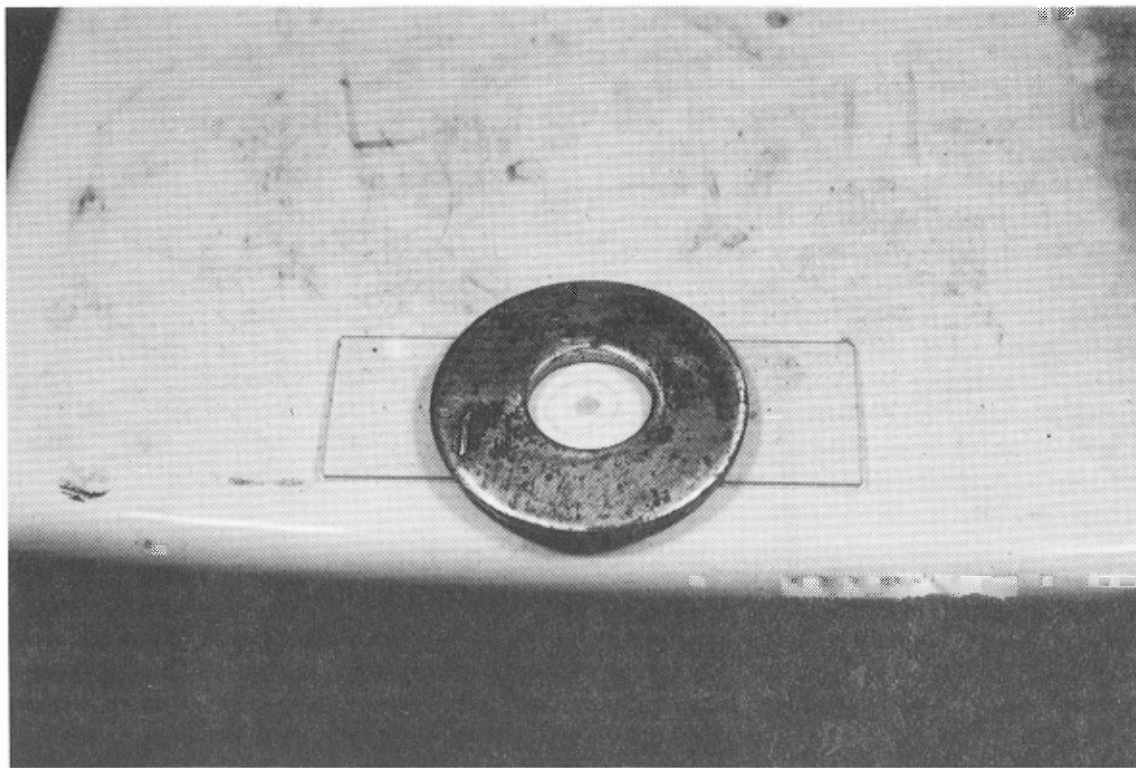


Figure 2. Hotplate Ring Oven

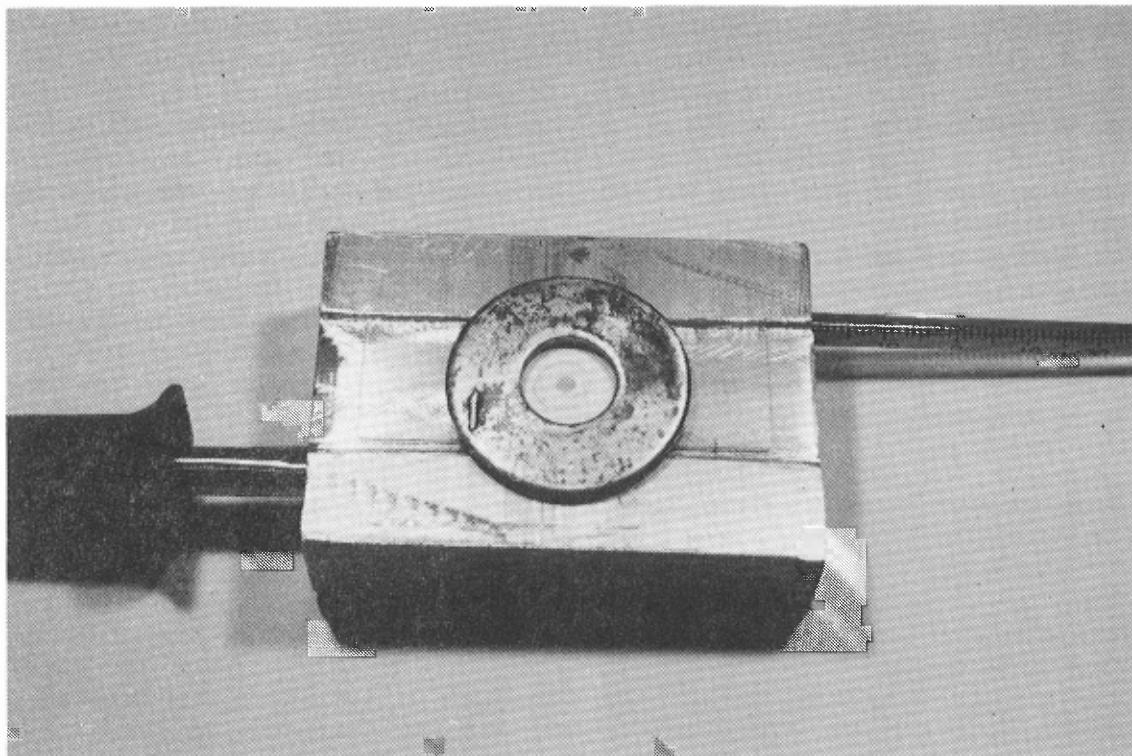


Figure 3. Hotstage Ring Oven

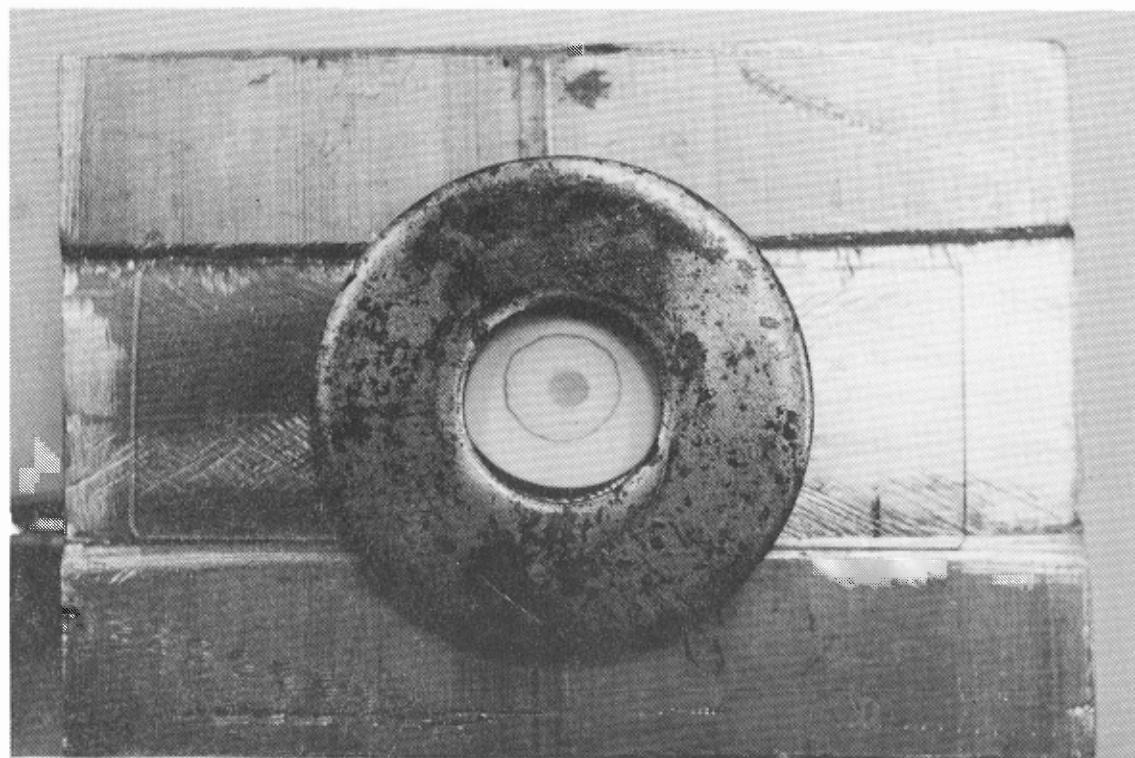


Figure 4. Hotstage Ring Oven with Cu^{+2} separated from Fe^{+3} ring zone.

reagents. For instance, moist test spots on the center of filter paper circles can be saturated with hydrogen sulfide to form precipitates of suspected metals at a certain pH and selectively solubilized using the ring oven according to standard qualitative analysis schemes.

A version of a hydrogen sulfide gas generator as shown on page 12 of Weisz's book can be fashioned from plastic labware commonly found in the lab. See Figures 5 and 6. A plastic disposable pipette is cut at the bulb end to form a tube funnel. This is put into one end of a two hole rubber stopper. The end of another pipette/ is cut and partially filled with cotton fiber and placed in the other hole of the stopper. This tube

is then connected to a plastic filter holder to accommodate the filter paper. The filter housing is attached to a hypodermic syringe to pull a slight vacuum. The apparatus is assembled with a solid sulfide (such as iron sulfide) in the bottom of the plastic bottle and dilute hydrochloric is added through the funnel as required to generate hydrogen sulfide. The gas then passes through the syringe filter. A slight pull from the syringe helps insure that the paper becomes saturated quickly.

With a little practice and ingenuity it is possible to use these tools to do a wide variety of separations at a very low cost.



Figure 5. Items for Hydrogen Sulfide Generator

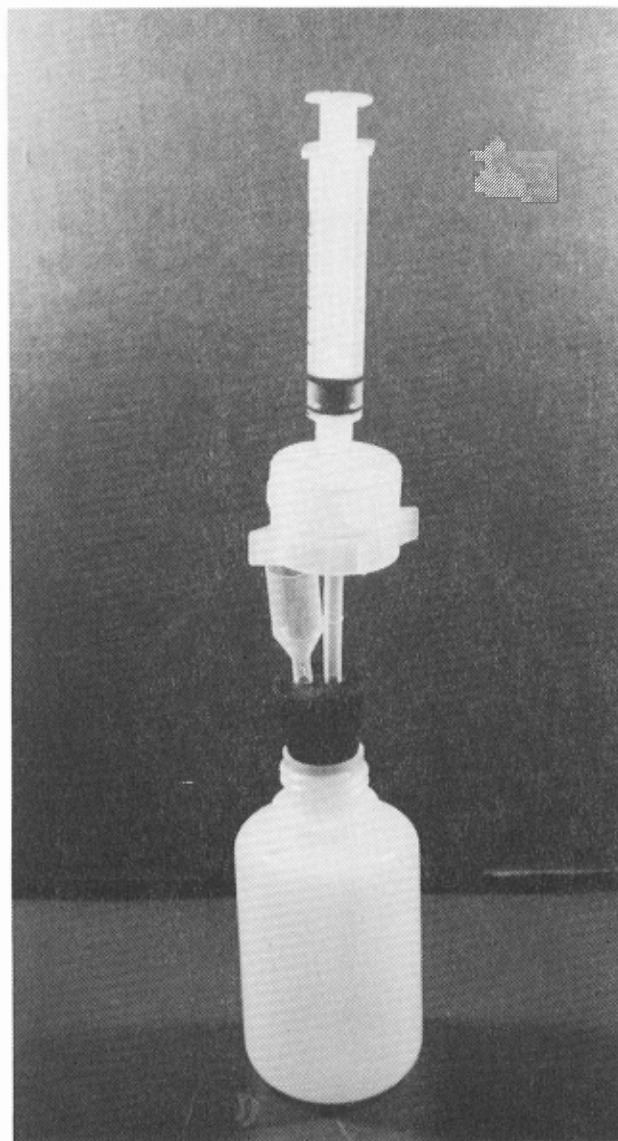


Figure 6. Assembled Hydrogen Sulfide Generator

New Use for an Old Grill

by
James J. Benko*

After a busy barbecue season I decided to clean out the grill and store it in the garage for the winter. During the cleaning process I noticed that the gas burner inside had corroded and needed replacing. I tore it out and was left only with the hard aluminum shell (which was still in pretty good shape). After a good, thorough cleaning I decided that next spring I would refurbish the inner workings for next year's barbecue season. Then a thought occurred to me: Why not use the grill to store slides and accessories needed for snowflake replication? The metal cover will protect slides and Formvar solution from the wind. The portability of the unit will allow movement anywhere on my patio or driveway.

The idea seemed to be the perfect solution for where to conveniently store everything needed, as well as to provide some working space. The inside ledge for the grill was fitted with a small board to give a working area to prepare and store slides. A few boxes of slides and all of

the necessary tools are now kept out in the cold in the bottom of the grill box under this shelf. The small wood shelves on each side of the grill provide a good surface to place a black cloth to catch and examine falling flakes. A small cookie sheet placed on the upper grill that moves with the cover is used to store slides in a protective cold environment while the replication process is taking place when the grill lid is closed.

So far, this arrangement has worked very well for me. I may decide to keep the grill shell just as it is for winter snowflake microscopy and forget about getting a new burner for it. This grill will now get more use in the winter than in the summer.

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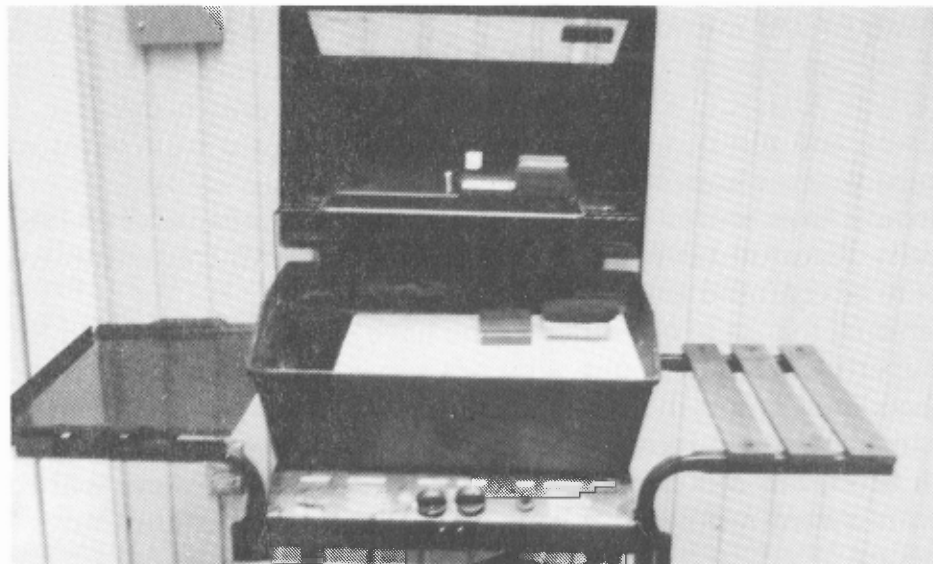


Figure 1. Grill fitted with wood shelf and supplies for snowflake replication.

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SMSI REPORT FOR 1997 BY RICHARD H. LEE

1996 — PRESIDENT — 1997



1997 was a good year on average but began in January with disaster. A snowstorm forced us to shorten our meeting and postpone updating the by-laws until Spring. My home was slightly flooded in February due to unusually heavy rain. We had good luck until May when disaster struck again. A storm knocked out power at McRI, forcing us to cancel our meeting on petrography. We plan to bring the Buehler people back next year to try again. That meeting had the best attendance I can remember.

Many thanks to Bill Mikuska for doing such a fine job on making our "μ-Notes 2000" a society publication again. Be careful about challenging him to do something. Dr. McCrone challenged him that he could not get Micronotes out before the *Judgement Day for the Turin Shroud* book and Bill, determined to surprise him, did. Bill also hosted the June picnic at his house when rain forced us indoors.

We completed updating the by-laws thanks again to Bill, Diane Richardson, Richard Lee, and Theodore Clarke. Mainly because of the publication costs for *μ-Notes 2000*, we decided it was time to raise the dues in 1998. Our newly modified by-laws permit more flexibility in selecting honorary award recipients. We also decided to name our annual awards, and present medals. We are looking into having the medals die struck, which would be less expensive per medal than engraving.

We also hope to make a new batch of society lapel pins in time for next year's 50th Annual Meeting at INTER/MICRO-98. I would like to see a special award for electron microscopy for two reasons: first, I would like to attract more electron microscopists to our meetings; and second, this year is now commemorating the discovery of the electron. As with the other awards, there are plenty of worthy candidates.

Our banquet and auction went very well and we expect next year to be really outstanding. We are grateful for generous donations of items for the auction, like the Electronic Particle Atlas CD ROM from the McCrone Research Institute, the Cargille index liquid sets, and plastic sample boxes. We also had a couple of microscopes for auction but next year we hope to have more.

We already have several excellent speakers for the remainder of 1997, including world authority Riccardo Levi-Setti of the University of Chicago who will speak on trilobites (not to be confused with tribolytes).

My personal thanks to our officers who have served with me: Bill Mikuska who has done a superb job; Susan Young who has served as Treasurer for the past several years; and Theodore Clarke who is also staying on in 1998. Ted did a fine job of getting our by-laws into their new form. Our annual awards committee, comprised of John Delly, Martin Scott, Bill Mikuska, Richard Lee, and Charles Allen, will continue the search for future award recipients. I have decided to try some new things in 1998 and do some traveling.

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Exposure Determination in Photomicrography Using Gossen Meters with the Microscope Attachment

Joseph G. Barabe*
Director of Scientific Imaging

INTRODUCTION

Determining exposure in photomicrography differs in several ways from the methods used in general photography. In conventional photography, an exposure reading might be taken by pointing at the subject with a conventional light meter, one with a fairly wide measuring angle. This approach assumes that the subject has an average brightness of 18%, about a middle gray if you disregard color as a factor, and this usually works fine. A refinement of this approach entails using a 1° spot meter to measure the brightness levels of each part of the scene, enabling the photographer to "place" the brightness levels of the scene into tonal "zones" in the finished photographs. In the studio, photographers find it more convenient to measure the light itself by means of an incident light meter.

As Delly points out, a single starch grain when photographed by cross-polarized light (with a resultant black background) requires exactly the same exposure as an entire photograph, filled with starch grains (2, 4). The microscopist, on the other hand, is often obliged to photograph subjects that vary greatly in manner of illumination and which do not lend themselves to straightforward exposure determination. In fact, illumination methods such as transmitted and reflected brightfield, transmitted and reflected darkfield, polarized light, dispersion staining among other illumination methods, offer serious challenges to the photomicrographer when the image field contains large amounts of black or white. The average brightness approach, which has often been suggested by many writers (1, 5), ultimately fails when conditions become extreme.

In practice, the "average brightness" approach does work well in many cases. A com-

mon application of this method is in biomedical transmitted brightfield photomicrography of stained histological specimens. Automatic exposure systems for photomicrography all work this way, and, especially when combined with spot metering, can provide acceptable exposures. Of course, even automatic systems must be calibrated through careful testing. With experience, the photomicrographer learns to compensate for deviations and variations in subject matter and adjusts the exposure accordingly.

But even in transmitted brightfield photomicrography, this approach can be misleading. The meter can be fooled by many factors, such as the percentage of the image field occupied by the subject, the thickness of the section, the depth of the stain and so forth. Loveland (3) described a method for brightfield photomicrography with many similarities to Ansel Adams' Zone System: meter your exposure for the white background, as that should always be represented as a very light grey. In zone system jargon, this would translate to "placing the white on zone VI," that is to say, one stop brighter than middle grey, which is zone V (zone VI may or may not be your choice for a good white). It then follows that the subject will naturally be recorded on the film at tonal levels appropriate to its attenuation of the transilluminating light. This is also akin to the studio photographer's use of an incident light meter in that the photomicrographer bases the exposure, not on the subject, but on the intensity of the illumination. In a well calibrated system, exposures are based on the amount of light striking the specimen and modified by the illumination method. Once calibrated for the method, the photomicrographer only need measure the total amount of light at the specimen plane.

Delly and McCrone later extended this insight into ways to determine exposure for any

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illumination method (2, 4). In situations such as transmitted or reflected brightfield, he suggests basing the exposure on the white background rather than the more inconsistent subject. For illumination methods that result in a black background such as polarized light or dispersion staining microscopy, he recommends modifying the illumination method to one that can be easily and unambiguously measured. We then calibrate the unambiguous measurement to the desired illumination method. For example, photomicrography with crossed polars is calibrated to a light measurement with the analyzer removed from the light path. This works because the brightness of the birefringent subject matter is proportional to the intensity of the illumination. This approach has great elegance, both conceptually and as a practical approach to exposure determination in photomicrography.

Delly described his calibrations in terms of the Science and Mechanics Meter, which subsequently became a favorite for photomicrographers for many years. As it has recently been discontinued, we are forced to look elsewhere for a suitable meter. There are many fine meters that can be adapted to photomicrography, but Gossen meters are the only commonly available instruments that have a microscope adapter.

GOSSEN PROFESSIONAL LIGHT METERS

Actually, my relationship with the Gossen Luna Pro series of light meters extends back to my days as a commercial catalog photographer. The Luna Pro was highly regarded among professionals for its accuracy and reliability. When I moved into scientific imaging, I came to appreciate its numerous accessories including attachments for narrow angle and spot metering, copy photography, a fiber optic probe, a color temperature meter, and, of course, microscope adapters. Besides providing a secure, light tight attachment to the microscope, they contain condenser optics that concentrate light into the meter window.

Today, Gossen makes several different models that accept the microscope accessory. The original Luna Pro uses a very sensitive cadmium sulfide photoconductive cell, as does the newer Luna-Pro S. The Luna Pro SBC employs a cadmium selenide cell which is even more sensitive and faster in response (this model, along with the microscope adapter, can be purchased through

McCrone Accessories). The Luna Pro F has, in addition to the SBC's features, the ability to measure electronic flash. This isn't an important feature for most microscopists, but it's very useful for many photographers. The totally digital Multi-Pro and Ultra-Pro meters utilize the selenium blue cell. The Ultra Pro incorporates many light measurement capabilities for photometry as well as photography. All the models listed accept the microscope adapter (Gossen product number 4102).

As conventional light meters, they are fast and simple to use. You set the film speed (ASA or ISO) along with any compensation, such as filter or magnification factors. All the Luna Pro models work by "nulling" the meter. After a light measurement is taken, a wheel is turned until the needle is brought to the fully vertical position (see Figure 1). The measurement is displayed as exposure times aligned with apertures (f/stops) and as exposure values (EV). The Multi-Pro and Ultra-Pro meters indicate exposure times on a liquid crystal display (LCD) in



Figure 1. The Gossen Luna Pro SBC.

1/10th stop increments.

The microscope attachment snaps over the meter window, and the light is measured by inserting the attachment into the meter port on the microscope (Figure 2). If your microscope does not have a metering port, a reading can be made through the eyepiece (Figure 3) or directly at the film plane of the camera. The film plane is a preferred measurement site for Polaroid and 4 x 5 cameras; for 35 mm photography, it is less convenient. For consistency's sake, the reading should be made at the same relative location (such as the center of the ground glass) every time. Ideally, the site chosen should incorporate all of the factors influencing exposure time, including photo eyepiece magnification and all filters.

The microscope does not have the familiar f/stop settings that camera lenses employ, so until we calibrate our meter with a series of practical tests, the readings given by the meter are meaningless. For use in photomicrography, the meter must be calibrated to the particular microscope, the method of illumination, and the actual metering method used. Each type of microscope is uniquely configured optically such that the calibrations for one type of microscope will not necessarily work for others (unless, of course, we take our light readings directly from the film

plane). Likewise, the microscopist must calibrate the meter for each different kind of illumination. The actual method of metering plays a role as well.

THE CALIBRATION PROCESS

We calibrate a particular microscope to a particular light meter, film, format and metering method. Our goal is to determine an "F" constant, a calibration constant that will provide us with a simple, convenient method to determine exposures for a variety of microscopical illumination methods. Writers on photomicrography are unanimous in their insistence that practical calibration tests be conducted and their results recorded and referenced in future work. All factors upon which successful photomicrography depend should be calibrated and recorded, including voltage settings, color balance filtration, reciprocity factors, etc.

The tests for this paper were all made with a Gossen UltraPro with an Olympus BH-2 microscope with the PM-10M photomicrographic camera using both Kodak Ektachrome 64 (EPY) 35 mm color transparency film balanced for tungsten illumination and Polaroid Type 55 4" x 5" black and white print film. The results of these tests are summarized in Table I.

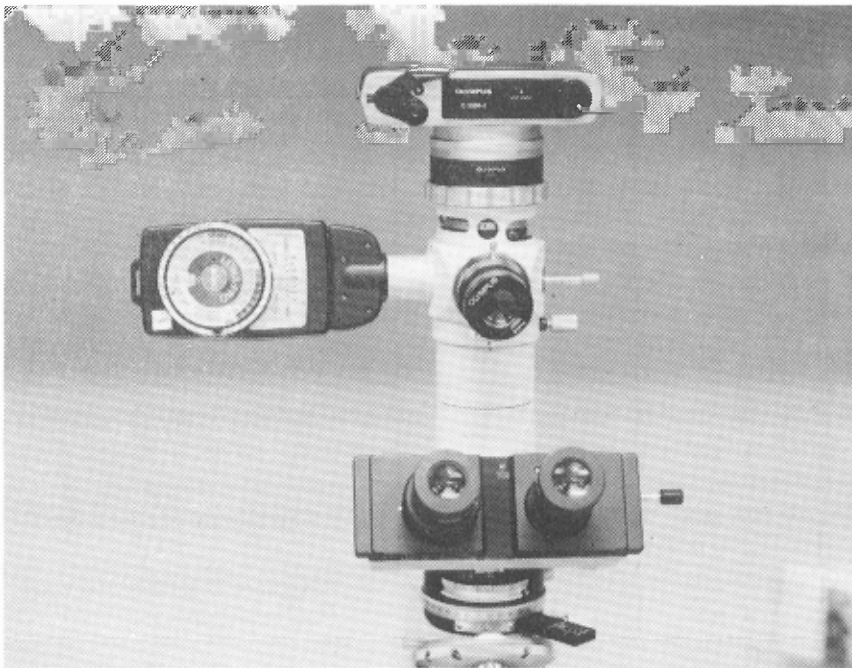


Figure 2. The Gossen Luna Pro SBC with the microscope attachment. It is inserted into the meter port of the Olympus PM-10 photomicrographic camera.

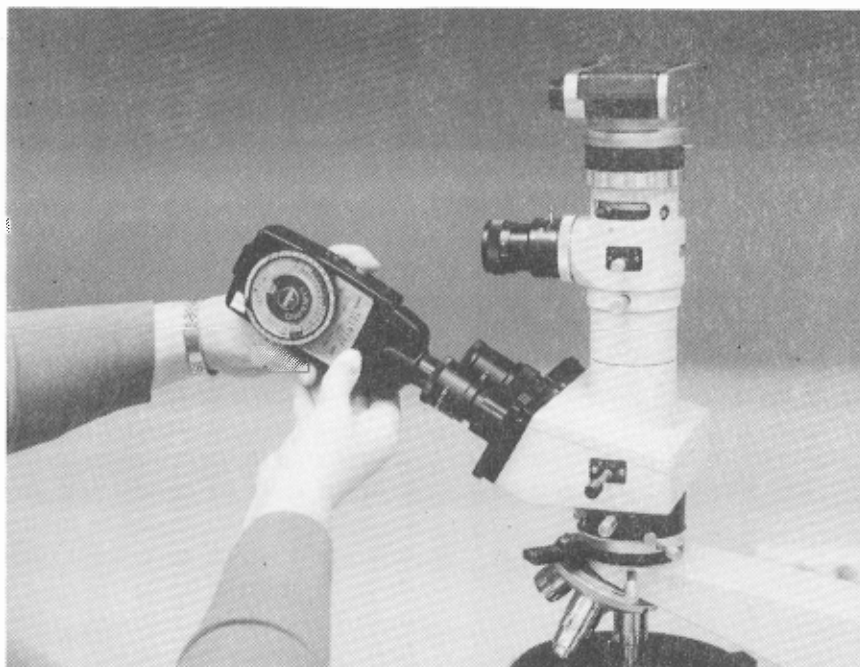


Figure 3. A light reading can be taken through the eyepiece of the microscope.

Table I.

METHOD	FORMAT	F CONSTANT	METERED REGION OR SUBSTITUTE
Middle Grey	35 mm	8 ⁵	Subject matter should fill field
	4" x 5"	32	
Transmitted Brightfield	35 mm	11 ⁵	Clear area of specimen slide
	4" x 5"	45	
Reflected Brightfield	35 mm	11 ⁵	White card replaces specimen
	4" x 5"	45	
Plane Polars	35 mm	11 ⁵	Clear area of specimen slide
	4" x 5"	45	
Crossed Polars	35	32	Remove analyzer from light path
	4" x 5"	90 ⁵	
Crossed Polars 1st Order Red	35	32 ²	Remove analyzer from light path
	4" x 5"	90 ⁶	
Dispersion Staining	35 mm	45 ³	Flip out top of condenser
	4" x 5"	128 ⁸	
	35 mm	32 ³	Replace D.S. objective with standard objective
	4" x 5"	90 ⁸	

F Constants for Kodak EPY (35 mm) and Polaroid Type 55 (4" x 5") using the Gossen UltraPro with the Microscope Adapter through the meter port of the PM-10M Photomicrographic System on an Olympus BH-2 microscope. The superscripts represent tenths of a stop.

The calibration process consists of the following steps:

1. *Selection* of an appropriate field capable of unambiguous measurement. This field may be the illuminated subject matter itself or it may be a substitute for the subject. It must be directly related to the intensity of the illumination.
2. *Measurement* of that field. You must choose a location from which to take the reading; that location might be the film plane or through one of the eyepieces, but most frequently today, one meters through the metering window of a trinocular head beam splitter. Successful calibration depends on being consistent in your location and manner of light measurement. The ideal location is above the photo ocular and all filters.
3. *Recording* that measurement onto a photomicrographic record form. The more data recorded, the easier it will be to achieve consistent results. The reader is invited to use the Photomicrographic and Calibration record forms included as addenda to this paper.
4. *Exposing* the film in a series appropriate to your subject matter. You can cover the entire range of exposures available to you with your camera, or expose in closer increments over a narrower range if you know your approximate F constant.
5. *Recording* the exact exposures in the series along with all changes made.
6. *Processing* the film. In baseline testing, this will usually be E-6 transparency processing at a commercial lab. Careful control of all variables such as time and temperature is most important. Again, consistency counts.
7. *Evaluating* the exposures and matching the best exposure to the exposure meter reading.

THE F CONSTANT

We match the best exposure to the f/stop recommended for that shutter speed. That f/stop then becomes the F constant. For example, say we find that a one second exposure gives us the best exposure with a particular microscope, metering method and illumination method. We

check the light meter reading recorded in our photomicrographic record form and find the f/stop aligned with one second exposure is f/8. Our F constant for that microscope, metering and illumination method is then 8. Changes in film speed do not change the F constant, but differences in magnification do (unless your method entails taking the reading from the film plane of the camera).

PRACTICAL TEST PROCEDURES

Our first test is actually two tests in one; we will calibrate our meter for both **brightfield transillumination** and an average middle grey (Figure 4). The **middle grey** calibration provides a useful approximation or point of departure in dealing with uncalibrated or mixed lighting situations.

1. Choose a specimen that fills the field with rich color. A biological specimen will do nicely if deeply stained and not too thin. For this I use a specimen of temporal bone.
2. Place the specimen on the microscope, establish Köhler illumination, and compose it so that it fills the field. For this test, the field should include some of the clear area next to the specimen. Make sure the voltage of the illuminator is at the recommended setting for proper color temperature.
3. Load your camera with color transparency film (negative films have too much exposure latitude to be useful for this test). Choose a film compatible with your light source. If it uses an incandescent tungsten lamp, use a film balanced for tungsten such as must be used with a light balancing filter such as the Wratten 80A or the Olympus LBD-2N.
4. Set the film speed (ISO) on the meter. Check the compensation setting: it should read 0.0 stops or 1X. If you have a standard calibration setting for general photography, leave that compensation factor in place—but make sure it's at that setting when doing photomicrography.
5. Move the specimen out of the viewing area. Take a light meter reading from a clear area outside the specimen field.

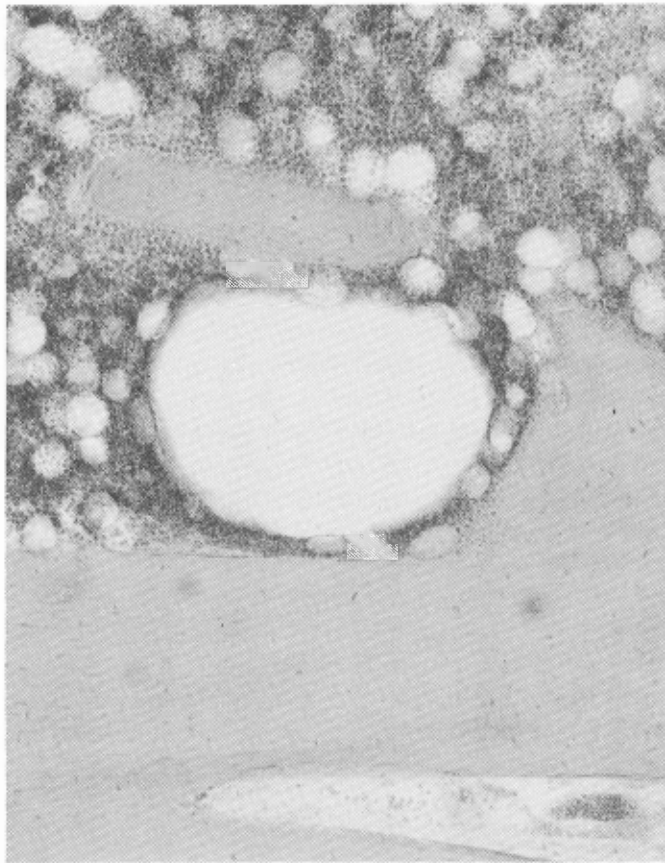


Figure 4. Temporal Bone, 40X, transmitted brightfield.

6. Read your exposure on the matching dials. It will indicate a series of shutter speed/aperture (f/stop) combinations. Record the indicated exposure reading on the Photomicrography Record form.

7. Move the specimen back into the field. Take another meter reading; this will be for your middle grey calibration. Record this measurement as well.

8. Make a series of exposures covering the full range of shutter speeds available from your system, say from one second to 1/250 of a second (other illumination methods may need more exposure).

For greatest accuracy, vary the exposures in 1/3 stops by using .10 neutral density (ND) filters. Part of your series should look like this: 1/60 sec + no filters, 1/60 sec. + .10 ND, 1/60 sec. + .20 ND, 1/125 sec. + .00 ND.... You can check shutter speed accuracy by including exposure

pairs such as 1/60 sec. + .30 ND and 1/125 sec. (.30 ND = 1 stop).

You can also bracket in half stops by combining different exposures without advancing the film. This is a good method for photomicrographic cameras with leaf shutters, not advised for SLR cameras without provision for multiple exposures. Part of your series should look like this: 1/60 sec., 1/60 + 1/125, 1/125 + 1/250, and so forth.

9. Process the film and evaluate the exposures. The f/stops that coincide with the best exposure from your exposure meter readings become your F constants. Record the F constants in your calibration record form. You should now have F constants for middle grey, based on the average brightness meter reading, and for transilluminated brightfield, based on a measurement of the white background.

A few additional thoughts on transmitted brightfield photomicrography: The calibration

just described may result in a transparency too low in contrast for your taste; in general photography, most photographers place a textured white on zone VII or even a little higher. One solution is to rate your film one to one and one half stops faster in film speed and have the processing lab increase development time accordingly. This results in a transparency with more contrast and cleaner whites. (You may have to adjust your color balance slightly as a result). A didymium filter greatly enhances color contrast and saturation for specimens stained with hematoxylin and eosin. Also, always use a green filter for black and white photomicrography, both for increasing contrast (by absorbing reds) and improved sharpness (by passing only the green portion of the spectrum).

Reflected light photomicrography can be metered in much the same way as transmitted brightfield, but instead of metering the clear area, insert a white card into the field and meter from that. Any repeatable reference standard will do. Delly suggests a 3" x 5" note card; I prefer the back of a business card, but a standard 18% grey card would work just as well. Don't

forget to move your specimen back into the field!

Polarized light microscopy (Figure 5) results in brightly colored subject matter against a black background. In this case, we must substitute the unmeasurable field with one that is easily measured. Simply remove the analyzer (the polarizing filter above the specimen) and take the meter reading with transmitted brightfield with only the polarizer in place. This will give us an F constant much larger than that used for middle grey, but one that will give consistent results. Happily, partially crossed polars receive the same exposure as fully crossed polars (Figure 6).

Repeat the series crossed polarized light with a 1st order red compensator (Figures 7, 8), and again with the 1/4 wave compensator.

Repeat the series once again for dispersion staining, if that is part of your instrumentation. Annular stop dispersion staining results in lightly colored particles on a light background. This method can be calibrated in the same way we calibrated for brightfield transillumination. Central stop dispersion staining (Figure 9), on the other hand, results in colored particles on a black background, so we need a substitute light-

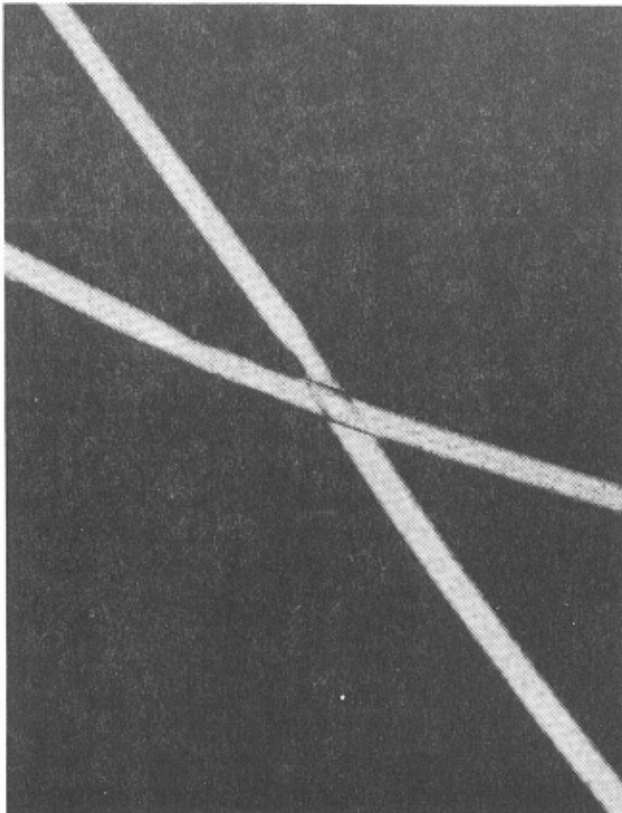


Figure 5. Nylon, crossed polars 100X. My F Constant is 90^5 .

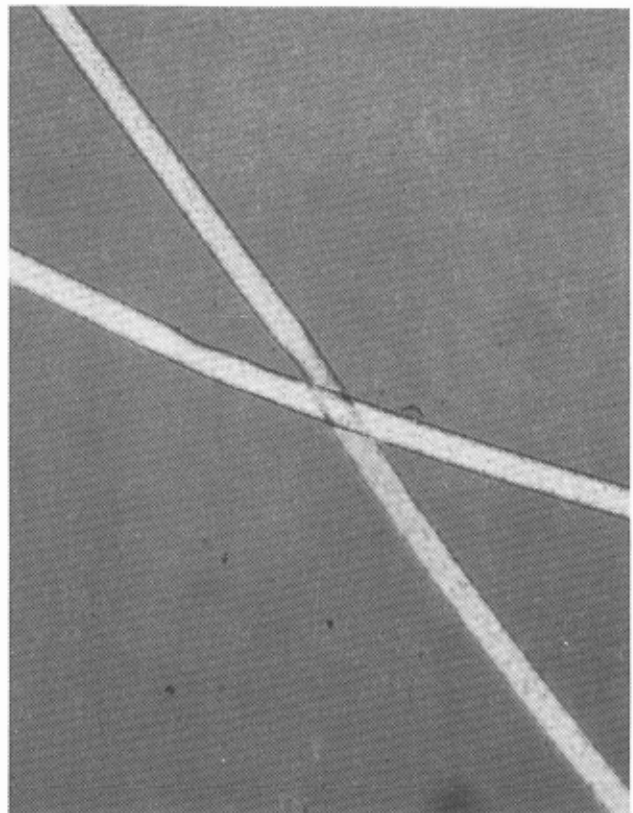


Figure 6. Nylon, partially crossed polars, 100X. Same F Constant as Figure 4.

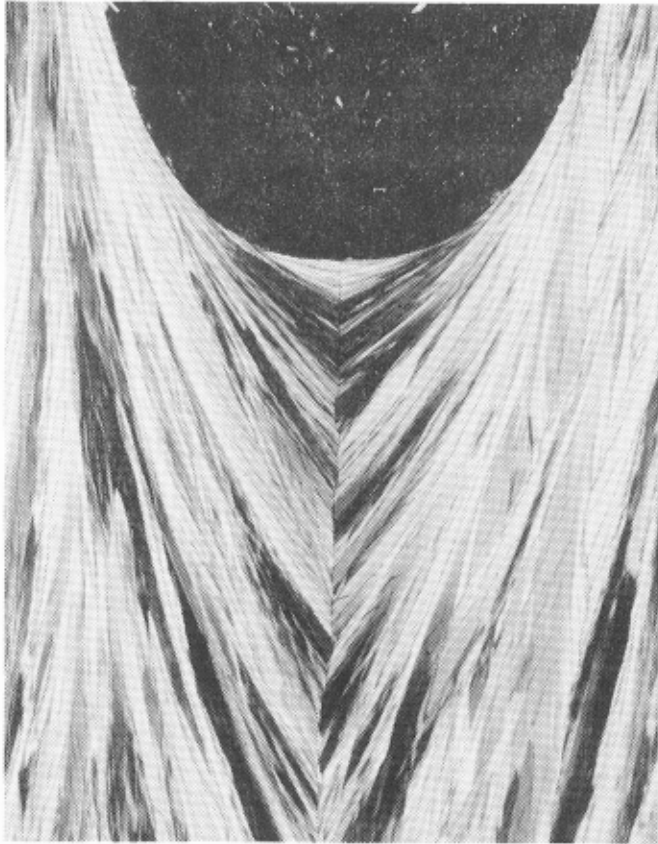


Figure 7. TNT, crossed polars, 100X.

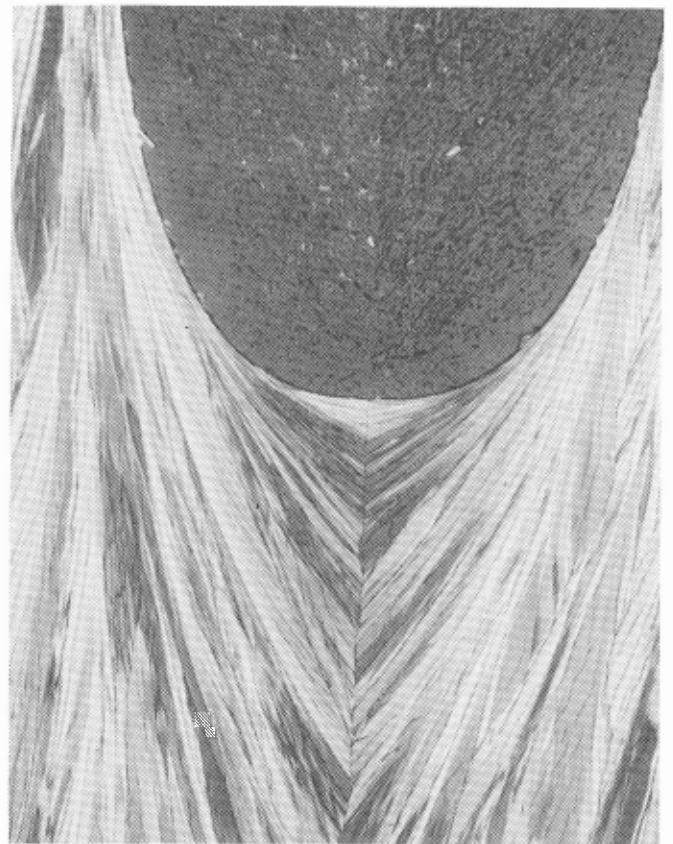


Figure 8. TNT, crossed polars with 1st order Red Compensator.

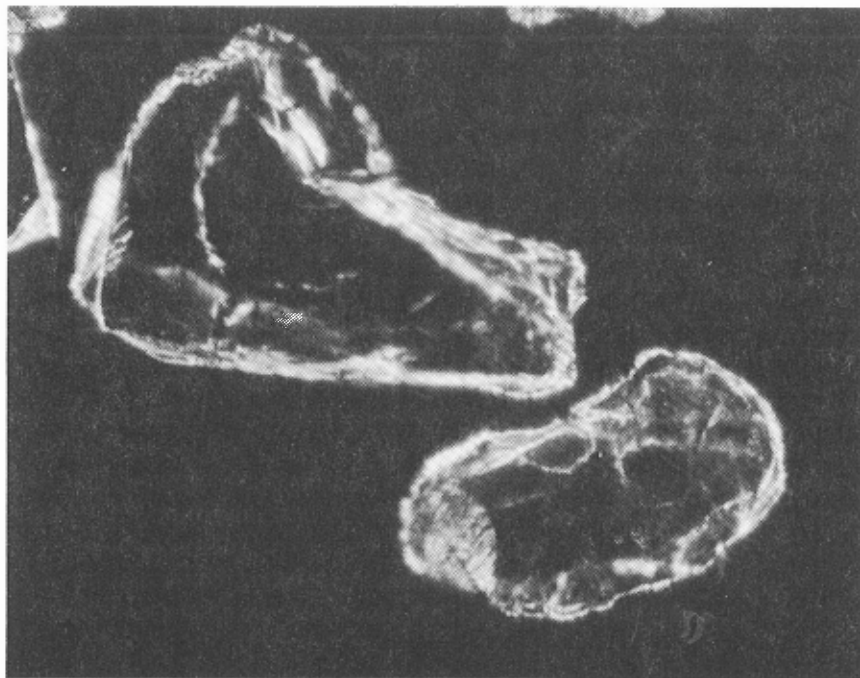


Figure 9. Sand grains, dispersion staining, central stop, 100X.

ing method. Actually, there are several practical possibilities. The 10X dispersion staining objective can be replaced with a conventional 10X objective, or the top condenser can be flipped into the light path for the light reading. If you own one of MAC's fine d.s. objectives incorporating an annular stop, a central stop and no stop, you can simply switch to the no stop position for both annular and central stop calibrations.

Darkfield microscopy represents a more difficult situation in that it's just not possible to easily substitute your lighting method; especially with improvised methods, you don't want to mess with your lighting situations. The solution is to substitute the specimen for a piece of ground glass or mat white diffusion material to determine your F factor (it may turn out to be an approximate factor that necessitates bracketing, but it should be pretty close).

A few final thoughts:

The references cited here are all quite short; the interested reader is highly encouraged to read them. The most important source is probably Delly's chapter on photomicrography in *Polarized Light Microscopy*. Do be aware that Delly's K constant and my F constant are related, but not quite the same...but they both work.

Finally, there are many illumination methods

not discussed in this short paper, but, armed with this approach, you can find the right F constants on your own, and your photomicrography will become more a creative undertaking rather than a frustrating technical challenge!

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3. Loveland, R.P. *Photomicrography, A Comprehensive Treatise*, New York, 1970.
4. McCrone, McCrone and Delly, *Polarized Light Microscopy*, Chicago, 1987.
5. Shillaber, C.P. *Photomicrography in Theory and Practice*, New York, 1944.

References 1-4 are available through McCrone Accessories & Components, Westmont, IL 630-887-7100 or 800-MAC-8122.

PhotoMicrography Record

Client _____ Job # _____ Date _____ File # _____
 Microscope _____ Camera _____ Format: 35 4x5 Pol _____
 Film _____ ISO _____ Process _____ Remarks _____

#	Subject	Oc.	Obj.	AD	V	Meter	Method	Filter	K	Exp.	Eval.
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Microscope Calibrations

Instrument	Film	Format	Illumination	Oc.	Obj.	Filtration	K	Exposure	Process

McCrone Research Institute

PROGRAM FOR CERTIFICATION IN APPLIED CHEMICAL MICROSCOPY

INTRODUCTION

For some time the McCrone Research Institute has offered a degree program based on (1) the candidate's prior academic qualifications and performance, (2) successful completion of McRI courses, and (3) completion of an approved thesis in an area of applied microscopy. The degree program has been small, but our experience with it has been very positive and it is continuing. It has become apparent, however, that a separate certification program would also be welcomed by our students and by their employers. Its purpose is to provide, outside of our research-oriented degree program, a formal way for students to be evaluated, monitored and recognized for their knowledge and practical abilities in chemical microscopy. Having this as a tangible training goal provides a positive structure to the student's training.

Certification in Applied Chemical Microscopy is based on (1) successful completion of six McRI courses, (2) passing a comprehensive written examination, and (3) passing a series of practical proficiency trials. Upon successful completion of the requirements, candidates will, on the nomination of the Director, be Certified by Board of Directors of the McCrone Research Institute that they have knowledge and ability in Applied Chemical Microscopy, as taught by McCrone Research Institute.

THE McRI COURSE EXPERIENCE

Our intensive one-week courses focus on the application of microscopy to the solution of chemical problems. The course material cannot reasonably be absorbed in one week and although we conduct quizzes in most of our courses to assess student progress, this serves primarily to help the instructor, not to evaluate students. As such, we do not employ a formal evaluation process during the course sessions.

Most students return to their laboratories after their initial (Primary) polarized light microscopy course equipped with a broad appreciation of the capabilities of chemical microscopy along with the theoretical and practical tools necessary to begin their development as chemical microscopists. Proficiency in the methods comes only with their continued practice.

After several months to a year of laboratory application, most students return to McRI for a Secondary course where the principles and techniques of the initial course are reviewed and expanded upon. The intervening experience provides an essential reference frame for the second course and even when portions of the second course duplicate the first, students are now in a position to acquire greater understanding and to refine their technique within the context of their practical work.

Subsequent courses provide uninterrupted time for intensive study in a specialized area and increasingly more sophisticated and focused instruction from the teaching staff.

THE ROLE OF CERTIFICATION

The Certification Program provides explicit evaluation of a student's knowledge and capabilities in Applied Chemical Microscopy. There are three general requirements: (1) successful completion of six McRI courses, (2) passing a comprehensive written examination following these courses, and (3) passing a series of practical proficiency trials.

COURSE REQUIREMENT

The McRI Course Experience is an important component of Certification by McCrone Research Institute. At least six McCrone Research Institute courses must be taken. Courses taken at McCrone Research Institute prior to enrollment in the Certification Program may be approved for credit

upon petition at the time of enrollment in the Program, however normally at least two courses will be taken after enrollment. Courses must satisfy the five breadth requirements: Primary, Secondary, Methods, Sample Preparation/Collateral Methods and Advanced Courses. Specific courses that satisfy these requirements are listed below. Substitutions will be considered upon petition. Additional courses are elective.

1. Primary Course Requirement

Filled by successful completion of any ONE of the following courses:

Photomicrography (#1101)
Applied Polarized Light Microscopy (#1201)
Identification of Small Particles (#1501A)
Pharmaceutical Microscopy (#1503)
Forensic Microscopy (#1504)
Microscopy for Art Conservators (#1506)

2. Secondary Course Requirement

Filled by successful completion of a second course, usually within 3 to 12 months of the initial course. During the intervening time students should have practiced the methods learned in the Primary Course and used the polarized light microscope in their area of application. The Secondary Course may be one of the four courses listed above, but cannot be a repeated course. Other courses meeting this requirement are:

Any non-repeated course listed under the Primary Course Requirement (1) above.

Polymer, Fiber and Film Microscopy (#1505)
Microscopical Identification of Asbestos (#1508A)
Comparative Microscopy of Soil (#1510)
Hair & Fiber Microscopy (#1519)
Microscopy of Explosives (#1522)
Mineral Grain Identification (#1523)
Microscopy of Botanical Traces (#1536A)

Any of the courses listed under the Advanced Course Requirement (5) below.

3. Methods Course Requirement

Filled by successful completion of any ONE of the following:

Photomicrography (#1101) [may not also be used to fill Requirement 1]
Hotstage Microscopy and Polymorphism (#1204)
Microchemical Methods (#1207A)
Biological Microscopy (#1235)

Crystal Morphology & Optics (#1301)
Asbestos Fiber Counting (NIOSH 582) (#1516)
Quantitative Asbestos Analysis (#1528)

4. Sample Preparation or Collateral Methods Course Requirement

Filled by successful completion of any ONE of the following courses:

Scanning Electron Microscopy (#1402)
TEM Asbestos Analysis (#1407B)
Advanced FTIR Microscopy (#1422)
Sample Preparation and Manipulation of Particles (#1501E)
Indoor Air Quality: Microscopy of Fungal Spores and Pollen (#1530)

5. Advanced Course Requirement

Filled by successful completion of any ONE of the following courses:

Advanced Microchemical Methods (#1207B)
Advanced Small Particle Identification (#1501B)
Microscopy and Microchemistry of Polymers (#1505B)
Advanced Asbestos Identification (#1508B)
Advanced Trace Evidence (#1514)
Microscopical Study of Paints and Extenders (#1520B)
Microscopy of Illicit Drugs and Excipients (#1526)
Forensic Examination of Building Materials (#1527B)
Advanced Asbestos QA/QC (#1529)
Advanced Pollen and Spore Identification (#1536B)

6. Elective Course Requirement

This requirement is filled by successful completion of additional course(s), making a total of at least six.

EXAMINATION REQUIREMENT

A comprehensive written examination must be passed after completion of the required courses. The 3-hour examinations may be taken at the end of the final course, during the annual Inter/Micro meeting, or by special arrangement. The content of the examination reflects that of the specific courses taken by the student. Application to take the comprehensive examination must be made at least 3 months prior to the examination. There is an application fee of \$200 (fees subject to change). Passing of the examination raises the student to Candidate status. If the applicant does not pass, the examination may be repeated once upon re-application. Failure to pass a second time is grounds for dismissal from the program.

PROFICIENCY TRIALS

Four sequential Proficiency Trials must be successfully completed by Candidates, after completion of the written examination. Each trial consists of the characterization of a sample using Polarized Light Microscopy, documentation by photomicrography and providing a written report of the sample characteristics. The content of the Proficiency Trials reflects that of the specific courses taken by the student. There is a \$200 fee for each Proficiency Trial (fees subject to change). Failure to pass the proficiency trials, or complete them in a timely manner, is grounds for dismissal from the program.

CONFERRING OF CERTIFICATION

After successful completion of the proficiency trials Candidates have satisfied the requirements for Certification and, upon nomination of the Director, the Board of Directors will confer Certification in Applied Chemical Microscopy.

APPLICATION

To receive an application for enrollment into the certification program send requests in writing to:

The Director
McCrone Research Institute
2820 S. Michigan Avenue
Chicago, IL 60616

SMSI Award Recipients - Part I: A Look Back

by

Bill C. Mikuska*

THE QUESTION AND THE QUEST

The editorial of this issue of *μ-Notes 2000* conveys the reason for this history of the SMSI Award Recipients. It was not my intention to write any article for this issue; however, as a relatively new SMSI member, I began to ask questions about award recipients. Instead of answers, I received the challenge: "If you *really* want to know more about SMSI's award recipients, why not look in the archives and then write an article about these awards since other members may also have these questions."

And so the quest to learn began: When did SMSI begin this practice, Who are these individuals, What did they do, and When were they honored? At first I encountered a mountain of information in the SMSI archives, then, only a mountain blocking my progress since the requisite information was not to be found in the archives! Therefore, this article represents only the first part of a longer trek.

IN THE BEGINNING

To the best of my knowledge, SMSI *systematically* began conferring awards/honors in 1967. Prior to this time SMSI records indicate that: a) Honorary Memberships were given to certain members[†]; b) a Chicago organization, The Chicago Technical Societies Council, CTSC, presented an award of merit for "His Outstanding Technical Achievement, Service to Science, His Fellow Scientists, and to the Community." The CTSC meetings and award ceremonies were held at The Furniture Club of America, 666 North Lake Shore Drive, Chicago, Illinois. The first award presented by CTSC was in 1946 followed

by a respite of six years. Thereafter, multiple annual awards were bestowed at least until 1963. The SMSI connection to CTSC is at least two-fold. First, SMSI was one of several member societies in the make-up of the Chicago Technical Society Council. Officers of CTSC were elected from the delegates of the member societies. For example, SMSI's Senior Vice-President, Robert A. Dallman, was the 1962-1963 delegate to CTSC. The second connection hardly needs an explanation. Vida Annette Latham and Leon F. Urbain, both past SMSI presidents, were honored by CTSC in 1957 and 1960 respectively. At the CTSC awards ceremony of November 22, 1960, SMSI Past President Dr. Walter McCrone introduced Mr. Urbain.

Shortly after the early 1960's, interest in SMSI waned. Between 1965 and October 1966 concerned members, spearheaded by Dr. McCrone, formed an SMSI Governing Board which had a plan to renew interest in the society. The initial plan consisted of five definite goals. Of the various committees formed to accomplish these goals, one was an Honorary Awards Committee to which Mr. Ed Lebryk was appointed chairperson.

"Worthy candidates for Honorary Membership should be members of the Society. These candidates will be considered for election by the Governing Board and would be announced at the Annual (June) Meeting" reads the October 10, 1966, Minutes of the Governing Board. Please note that the selection process has evolved along with the society's by-laws. Therefore, also pertinent to the discussion here is the introduction in 1970 of an annual award, the highest award conferred by SMSI to those individuals for "inestimable achievements advancing

[†] Membership lists in the archives indicate that there were various membership categories as there is now. Unfortunately, the Honorary Memberships and Life Memberships were, at that time, treated as *one category*, Category A, with no distinction made as to which member belonged to which sub category. Correspondence predating 1967 indicates only that Member X, for example, be deleted from category B and added to category A. Resolution of this issue may come from further research in the archives.

*Triton College, River Grove, IL 60171

the art and science of microscopy," and the Honorary Membership became the Honorary Award. Later SMSI awards will be described in Part II of this history since their relevance pertains to future recipients. Lets now take a brief glimpse at those who were honored in the beginning.

THE CHOSEN

1967 Honorary Memberships were awarded to:

Mr. Leon F. Urbain for his work as director of the course "Learning to Use the Microscope" (the Young People's Course). He has given over 1500 lectures and has donated all honoraria to the educational fund of the Society.

Dr. Walter C. McCrone for work in the field of microscopy and for enthusiastically endorsing the expansion of microscopy in many fields.

1969-1970 Annual Awards were presented to:

Dr. George Nomarski of the Institut d'Optique Theorique Et Appliquee, Paris, France, for his innovative method of Differential Interference Contrast Microscopy, a method which produces three-dimensional images with high contrast.

Dr. Crewe, a University of Chicago professor, who allowed man to "see" an atom for the first time with his single atom microscope, a very high voltage scanning electron microscope.

1970-1971 An Annual Award was presented to Irwin Müller for the development of the field-ion microscope.

Honorary Awards were presented to:

Roger P. Loveland for his comprehensive treatise on photomicrography and his work on the size and morphology of silver grains.

Mary Willard for her contributions to and teachings of forensic microscopy at Pennsylvania State University.

1971-1972 The Annual Award went to Prof. Marcel Françon of the Institut d'Optique de Paris, Paris, France for theoretical and prac-

tical contributions to the field of optical microscopy, and for publications on microscope optics with emphasis on interference phenomena.

Honorary Awards were presented to:

Dr. Kurt Michel of Carl Zeiss, Oberkochen (Württ.) West Germany for contributions to the theory and design of the optical microscope, and for publications in the fields of theoretical microscopy and photomicrography; viz. *Die Grundzüge der Theorie des Mikroskops* (1964) and *Die Mikrophotographie* (1967).

Dr. Manfred Nahmacher of E. Leitz, Inc., New York, NY for bringing information on theoretical and applied optical microscopy before the general public and microscopical societies and thereby promoting an understanding of the microscope's role in science and society.

1972-1973 Honorary Awards were presented to:

Dr. Ruth Patrick of the Limnology Department of the Academy of Natural Sciences of Philadelphia, Philadelphia, PA. for applications of the microscope to the systematic treatment of the diatoms of the United States, especially as expressed in publications on diatom structure and classification, diatom communities in polluted waters, and particularly for *The Diatoms of the United States*, Volume 1, (with Charles Reimer).

Dr. L.C. Martin, formerly Professor in the Technical Optics Department of Imperial College of Science and Technology, London, England, for books, *Practical Microscopy* (with B.K. Johnson) and papers on theoretical and applied optics, and especially for *The Theory of the Microscope* (1966) in which the optical theory of the microscope is updated to the recent work by theoretical physicists.

A Life Membership was awarded to Mr. George Maier of Maier's Aquarium, Chicago, IL. for unselfish service to the State Microscopical Society of Illinois, particularly for his willingness on every occasion to give his time and share his microscopical

experiences with the young people at the Saturday morning classes.

1973-1974 The Annual Award was presented to Dr. John McArthur of Cambridge, England, for the design and development of the McArthur Pocket Microscope

Honorary Awards were also conferred upon:

Dr. N.H. Hartshorne of East Sussex, England for the design and development of the Hartshorne Crystal Rotation Apparatus and the authorship of *Crystals and the Polarizing Microscope* and *Practical Optical Crystallography* (co-authored with A. Stuart).

Dr. Peter Gray Avinoff Professor of Biology, University of Pittsburgh, for the authorship of important papers and books in microscopy; in particular *The Microtometist's Formulary and Guide* and the *Encyclopedia of Microscopy and Microtechnique*.

1974-1975 An Annual Award was bestowed upon Dr. Charles Proffer Saylor of the Polymer Division of the National Bureau of Standards, Washington, D.C. for the development of significant improvements in the microscopical measurement of physical properties such as particle size and refractive indices. He is also recognized for his development of the "Freezing Staircase" purification procedure.

An Honorary Award was presented to Dr. Ralph Gander of Wild Heerbrugg, Ltd. Heerbrugg, Switzerland for his service to microscopy through articles and books in the field of light microscopy and through educational activities at Wild Heerbrugg.

1975-1976 An Annual Award was presented to Mitch Sieminski, Celanese Corporation, NJ, for his work in polymer/fiber microscopy.

Honorary Awards were presented to: Clyde W. Mason, Late Professor of Chemical Microscopy and Metallography at Cornell University at Ithaca, for coauthoring a fundamental text in chemical microscopy. The *Handbook of Chemical Microscopy* was jointly

authored with Émile M. Chamot.

Maria Kuhnert-Brandstätter, Professor of Pharmacognosy, and Director of the Institute of Pharmacognosy, University of Innsbruck, Austria for her work in fusion methods. As a student of the Koflers, she was well-known for the development of innovative hotstage techniques and her monograph, *Thermomicroscopy in the Analysis of Pharmaceuticals* (1971).

1976-1977 The Annual Award was presented to Oscar W. Richards, College of Optometry, Pacific University, Forest Grove, Oregon. As former chief biologist at the Research Laboratory of the American Optical Company, he was instrumental in developing AO's phase contrast and interference microscopy systems. In addition to being one of the authors of *Phase Microscopy* (John Wiley, 1951), he has written numerous articles pertaining to varied areas of microscopy such as vision and microscopy, and the history of American microscopy. He was also the former editor of the *Journal of the American Microscopical Society*.

An Honorary Award went to Mr. R.I. Firth of Sussex, England. As a diatomist of international renown, whose skills at mounting diatoms were known throughout the world, Mr. Firth also made to order mechanical fingers for diatom mounting.

1977-1978 The Annual Award was presented to W.M.D. Bryant of West Chester, PA, who was involved with microscopy at DuPont for many years. Bill Bryant has applied microscopy to archeological investigations and his home lab was replete with self designed and built equipment of simple, yet elegant design. His major interest was conoscopy.

Honorary Awards were presented to:

Dr. Dieter Krauter of the W. Keller & Company, Kosmos-Verlag, Stuttgart, Germany, for promoting the art and science of microscopy among amateurs. His book, *Mikroskopie in Alltag* (1954) was translated into English as *Experimenting with the Microscope* (1963) and is aimed at amateurs.

He is the publisher and editor of *Mikrokosmos*, a well-known journal slanted towards the amateur microscopist. He is also associated with the German Microbiological Society-Stuttgart and the microbiological societies of Hamburg, Mannheim, and Munich, the Micrographic Society of Vienna and the Microscopical Society of Zurich.

Dr. Max Frei of Zurich, Switzerland for outstanding contributions to the field of Forensic microscopy. He served as head of the Zurich technical police laboratory for over 20 years and his article, "Die Sicherung von Mikrospuren mit Klebband" published in 1951, introduced the use of adhesive tape to the collection of microclues from surfaces for subsequent examinations. As an authority in trace evidence examination, he was able in one case to trace a suspect to his doorstep from the crime scene from a clump of mud left at the site. He is the author of many articles dealing with the solution of crimes using microscopy.

1978-1979 The Annual awards went to:

F. Donald Bloss, Department of Geological Science, VPI and SU Blacksburg, Virginia. He has written numerous papers and books on chemical and optical crystallography and was honored for his contributions in theoretical and applied optical crystallography.

Mr. Charles C. Fulton of Venice, Florida for his lifelong efforts to promote the use and development of microcrystal tests. His numerous papers published in the field led to the publication of *Modern Microcrystal Tests for Drugs*. Currently retired, Mr. Fulton is working on an outline of the history of "microcrystalloscopy."

Honorary Awards were presented to:

Dr. Maksymilian Pluta of the Centraline Laboratorium Optyki, Warszawa, Poland. He was honored for his publications and also for his theoretical and applied work in the areas of stereoscopic phase-contrast

microscopy; amplitude-contrast microscopy using soot amplitude rings; highly sensitive phase-contrast microscopy; and shearing polarization interference microscopy.

Dr. Ernst P. Martin of Füllinsdorf, Switzerland, for his application of microscopical techniques to the forensics sciences. As director of the criminalistics laboratory of the city of Basel, he applied his knowledge in both his case work and research which resulted in numerous publications. After retiring in 1975, he continued to conduct research and served as a consultant to agencies in several countries.

THE VISION

This history of the individuals honored by SMSI reflects the vision of the Governing Board and the Awards Selection Committees over the years. Not only were honored individuals chosen in their own right for what they accomplished scientifically either using microscopy or advancing the art and science of microscopy, but they were also chosen for their macroscopic influence on the individuals they affected through their teachings. INTER/MICRO conferences afford attendees the opportunity to meet and converse with these people. They allow us, at least fleetingly, to be part of their special world.

For many of the seasoned SMSI members this first installment of the SMSI Award Recipients awakened past memories and for newer members it may serve as a part of SMSI's more recent history.

ACKNOWLEDGMENTS

This beginning history of the SMSI Award Recipients would have been impossible to write were it not for the meticulous early record keeping of Ed Lebryk and John Delly. Records supplied by Nancy Daerr, along with my conversations with Skip Palenik, Dr. McCrone, and Ed Lebryk, garnered more information for Part I of this history. It is to these individuals that I and all SMSI members are indebted.

Publications of the State Microscopical Society of Illinois

John Gustav Delly*

A complete list of publications issued by the State Microscopical Society of Illinois (SMSI) may not be possible, as the Society itself does not possess all of its own publications. I was Curator of the SMSI during the middle 1960's, and found the collection of books, records, instruments, and microscope slides in total disarray, housed in the rooms and a vault in the basement of the Chicago Academy of Sciences. I started organizing everything by systematically compiling a *Catalog of Publications in the Library of the State Microscopical Society of Illinois*, reading many of the Society's old records (correspondence, Secretary's reports, inventories, etc.), assembling the instruments and accessories, and organizing the microscope slides (first into animal, vegetable, and mineral; then into phyla, orders, etc.; I left the diatoms for last). This experience has placed me in what may be the best position to comment on the Society's publications. I list these publications to the extent that I know them, and perhaps somebody in the future can then add to my list if and when other publications are discovered.

HISTORICAL BACKGROUND

The SMSI's first publication, *The Lens*, was prepared within a year of the Society's Incorporation: in the autumn of 1868 several informal conferences were held by a number of gentlemen who were interested in the microscope at both amateur and professional levels. The Chicago Academy of Sciences issued a circular inviting them to meet at their rooms and organize as a section of the Academy. The founders, however, decided to form an independent society, but to hold their meetings at the Academy. Accordingly, the Chicago Microscopical Club was organized on 12 December 1868. Early members of the group included Walter H.

Bullock, the Chicago microscope maker, and later, Babcock, of the Babcock flask used in dairy science, and Ezekial H. Sargent, a local chemist, who founded what became the E.H. Sargent scientific supply house. By Spring of the following year, 31 March 1869, the Club had obtained a Charter from the State of Illinois to incorporate as the State Microscopical Society of Illinois. This makes SMSI the second oldest microscopical society in the world—the London Microscopical Club having obtained a Royal Charter as the Royal Microscopical Society in 1866.

By the close of the Society's first year there were 89 active members, 31 meetings were held, 10 papers were read, and a library and cabinet had begun. At the Second *Conversazione* on 19 May 1870, 65 microscopes and about 500 slides were exhibited. By 17 March 1871, for the Third *Conversazione*, the exhibit hall was divided into 22 sections to accommodate 120 microscopes and the 1,500 guests who attended.

It was in this climate that a publication was proposed. It was to be called *The Lens*. Articles were solicited, *The Lens* was published, but then tragedy struck—The Great Chicago Fire. On the 8th and 9th of October, 1871 the conflagration destroyed the city, including the Chicago Academy of Sciences, and the first number of *The Lens*, which had just been completed. The entire edition of the first number was burned, with the exception of a half-dozen copies which the editor happened to have at home. The spirit of the city was such that the residents immediately began to rebuild. And this spirit was no less true of the SMSI membership. The first meeting of the society after the fire was held on 8 December 1871. It was unanimously decided to reissue the first number of *The Lens* and get on with the meetings. The Chicago Academy of Sciences was also rebuilt. Both SMSI and the Academy lost everything in the way of books, instruments, and col-

* McCrone Research Institute, Inc., Chicago, Illinois 60616-3292

lections, and both were rebuilt by soliciting donations from around the world.

SMSI PUBLICATIONS

The SMSI's first publication was issued, or, rather, reissued, within a few months after the fire. The first number was followed by three more numbers, all issued in 1872, comprising Volume 1. 1873 saw the issue of four more numbers that comprised Volume 2:

The Lens, A Quarterly Journal of Microscopy and the Allied Natural Sciences, with the Transactions of the State Microscopical Society of Illinois

Volume I,	Number I	January, 1872
Volume I,	Number II	April, 1872
Volume I,	Number III	July, 1872
Volume I,	Number IV	November, 1872
Volume II,	Number I	January, 1873
Volume II,	Number II	April, 1873
Volume II,	Number III	August, 1873
Volume II,	Number IV	December, 1873

And then it ceased publication. There is nothing in the last issue of the last volume, nor in the records that I have seen to indicate why *The Lens* suddenly discontinued. There are some marvelous articles in these two volumes, including the first one in the first issue of the first volume, which is by Prof. H.L. Smith; it is a "Conspectus of the Families and Genera of the Diatomaceae," on which Wolle, and others, would base their diatom classifications. Babcock writes on "The Flora of Chicago and Vicinity." Briggs first describes "The Diatomaceae of Lake Michigan." And the famous military surgeon microscopist, Col. J.J. Woodward, contributes "Microscopical Memoranda, for the Use of Practitioners of Medicine." And this is all in the first issue only. Smith, Babcock, and Woodward continue their contributions in the second issue. The famous English microscopist M.C. Cooke contributes an article on the "Alternation of Generations in Fungi." At this time too, the Chicago River was having the direction of its flow reversed, and H.H. Babcock reports "On the Effect of the Reversal of the Current of the Chicago River on the Hydrant Water." Supplements to the second issue of Volume I are the illustrated catalog of Optical Instruments of James W. Queen & Co., and R.&J. Beck's illustrated catalog. The other

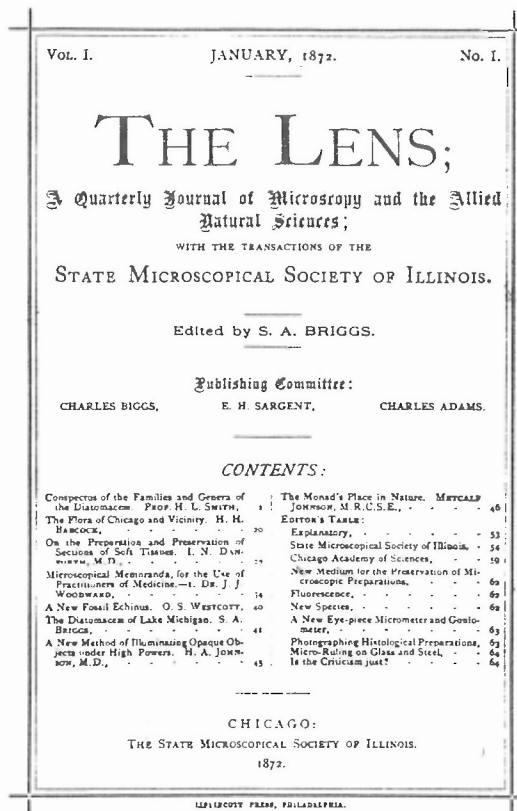


Figure 1. Front cover wrapper of the State Microscopical Society of Illinois' first publication, *The Lens*, Volume 1, Number 1, January 1872.

issues have equally interesting, useful, and historically important articles. The frontispieces of the first four issues consist of an engraving of monads by a Chicago engraving company, a Woodburytype of a diatom, a photomicrograph produced by the Albert-type process, and a tipped-in photomicrograph by Dr. J.J. Woodward of the diatom *Frustulia saxonica* magnified 1750 diameters.

Volume 2 of 1873 contains articles on the causes of influenza in horses (there were about 10,000 horses in Chicago at that time); a new mechanical finger (for micromanipulation and mounting); cancer cells; resolving and penetrating power of certain objectives; numerical aperture; the germ theory; hair in its microscopical and medico-legal aspects; the spectrum of chlorophyll; microwriting; and the best test for objectives.

A copy of these two volumes of *The Lens*, bound as one, are currently being offered by a dealer in rare books at \$650.00—and worth every penny, too!

My catalog of SMSI's publications has an entry referring to:

The Lens, (New Series), I (May, 1939), but I do not now remember anything about this publication, and cannot find it in the library's current state.

Following *The Lens* there are many ephemeral publications, such as brochures describing the Society and its history, and including a membership application; 10 to 20-page brochures made up as programs announcing Soirées, etc.

These ephemera can be quite interesting and informative. For example, the great Microscopical Soirée of 12 March 1913 was held at the Chicago Academy of Sciences, and included a demonstration by Harry Wells of "Direct Natural Colour Photography," in which he utilized the Autochrome, DuFay, and Ives methods of reproducing natural color. W.F. Willis demonstrated crystallization of various chemicals using a Balopticon projection microscope, with opaque attachment. Dr. Vida A. Latham, a very famous local physician and dentist who was also an SMSI member and officer for many years, showed many slides, and contributed a photomicrograph of a dental follicle from a 16th-week fetus. Dr. Ewell was there demonstrating his microrulings, and comparing them with H.J. Grayson's test plate. And, interestingly enough, one of the exhibits in the Main Hall was presented by Miss Mabel Smallwood, who was a biology teacher at Lane Technical High School. Her exhibit consisted of microslide specimens prepared by her high school zoology and botany students. This high school in Chicago has recently achieved Historic Building status, and soon appropriate restoration and preservation measures will be taken. I wonder how many of today's high school students there are preparing stained sections of earthworm, sponge, etc.?

There is, predictably, a noticeable absence of publications during the years of World War I, but the publications resume immediately after the war, with a *Bulletin*. In addition to meeting announcements and topics, these bulletins contained microscopical information, or hints and kinks, or methods, so that there is good reason to preserve them. I remember collecting these in a pile as I came across them, and the feeling of disappointment I had when I put them in numerical order and discovered that the run was incomplete, lacking, especially, the very first issues. I was working in the basement of the Chicago

Academy of Sciences at the time, and Dr. William Beecher, the Academy's Director came by and asked how I was doing. I told him of my problem, and then he told me to check the Academy's Library. He said that SMSI had placed the Academy on its mailing list as a professional courtesy, since the Academy was the Society's home from the very beginning. I checked, and, fortunately, they had the missing numbers. Dr. Beecher allowed me to make copies so that the Society could have a complete set.

Sadly, I have to report that the current Director has some new ideas, and much of the "old stuff" in the library has been sold off or donated to local book dealers and book sale organizations, and most of the old specimen collections dispersed. I am afraid that SMSI's old publications in the Academy library may have suffered the same fate. When I saw the Academy's copy of the Ultraviolet Edition of Gage's book on the microscope—now in private hands—marked, "Withdrawn," I can only imagine the extent of the disbursement.

Following is a list of the dates of issue of *The Bulletin*. The *Bulletin* started in March 1919, and was typewritten up to the middle of the fifth volume, and thereafter was typeset. It's history will be noted by the gaps in years, and its cessations.

Bulletin of the State Microscopical Society of Illinois

Volume I,	Number 1	March 1919
Volume I,	Number 2	n.d. [<April 15, 1919]
Volume I,	Number 3	n.d. [<June 28, 1919]
Volume II,	Number 4	January 1920
Volume II,	Number 5	February 1920
Volume II,	Number 6	March, 1920
Volume II,	Number 7	April, 1920
Volume II,	Number 8	May, 1920
Volume II,	Number 9	June, 1920
Volume III,	Number 10	January, 1921
Volume III,	Number 11	February, 1921
Volume III,	Number 12	March, 1921
Volume III,	Number 13	April, 1921
Volume III,	Number 14	May, 1921
Volume III,	Number 15	June, 1921
Volume III,	Number 16	July, 1921
Volume III,	Number 17	September, 1921
Volume III,	Number 18	October, 1921
Volume III,	Number 19	November, 1921
Volume III,	Number 20	December, 1921
Volume IV,	Number 21	January 1922
Volume IV,	Number 22	February 1922

Volume IV, Number 23	March 1922
Volume IV, Number 24	April, 1922
Volume IV, Number 25	May, 1922
Volume IV, Number 26	June, 1922
Volume IV, Number 27	September, 1922
Volume IV, Number 28	November, 1922
Volume IV, Number 29	December, 1922
Volume V, Number 30	January, 1923
Volume V, Number 31	February, 1923
Volume V, Number 32	March, 1923
Volume V, Number 33	April, 1923
Volume V, Number 34	May, 1923
Volume V, Number 35	June, 1923
Volume 5, Numbers 36-37	Sept.-Oct., 1924
Volume 5, Numbers 38-39	Nov.-Dec., 1924
Volume 6, Numbers 40-41	Feb.-April, 1925
Volume 6, Numbers 42-43	June-Aug., 1925

one issue:

Volume 6, Numbers 44-45	Oct.-Dec., 1925
Volume 7, Numbers 46-47	Feb.-April, 1926

[48 and 49 never issued]

Volume 8, Number 50	May, 1935
Volume 8, Number 51	June, 1935

To my knowledge, there were no publications following *The Bulletin*, until the years of World War II. This is somewhat surprising, because the runs of all of the journals of all countries involved in the war either slowed down considerably, or ceased altogether. In a sense, the history of the world can be seen at a glance by looking at shelves of complete runs of journals, and noting the relative thickness of the volumes and their years of publication.

From 1942 to 1945, SMSI members who were too old for the armed services, and even beyond the earning years, kept SMSI publications going in the form of *Diatom-Notes*, which consisted of one to three folded-over sheets of violet, spirit-duplicated text and drawings on some aspect of diatoms. These are now fragile, difficult to read, and extremely scarce. I never found all of them, but infer a number 1 and 3 from the finding of a number 2. Here is the best I can do:

Diatom Notes

Number 1	?
Number 2	June 12, 1942
Number 3	?

Number 4	July, 1942
Number 5	September 5, 1942
Number 6	November, 1942
Number 7	(no date)
Number 8	November, 1944
Number 9	January, 1945

Just before the end of the war, in April, 1945, a 92-page paperback booklet was issued:

Stump, Dan M. *An Adventure with Photo-micrography*. State Microscopical Society of Illinois (April, 1945).

This delightful—and scarce—booklet is subtitled, *A Detailed Description of How to Make a Good Photograph Showing Extremely Fine Periodic Structure*. And, of course, it has to do with making a photomicrograph of a diatom; in this case, the famous test diatom, *Amphipleura pellucida*. Dan Stump illustrates his apparatus, and describes how he uses polarizers and arranges

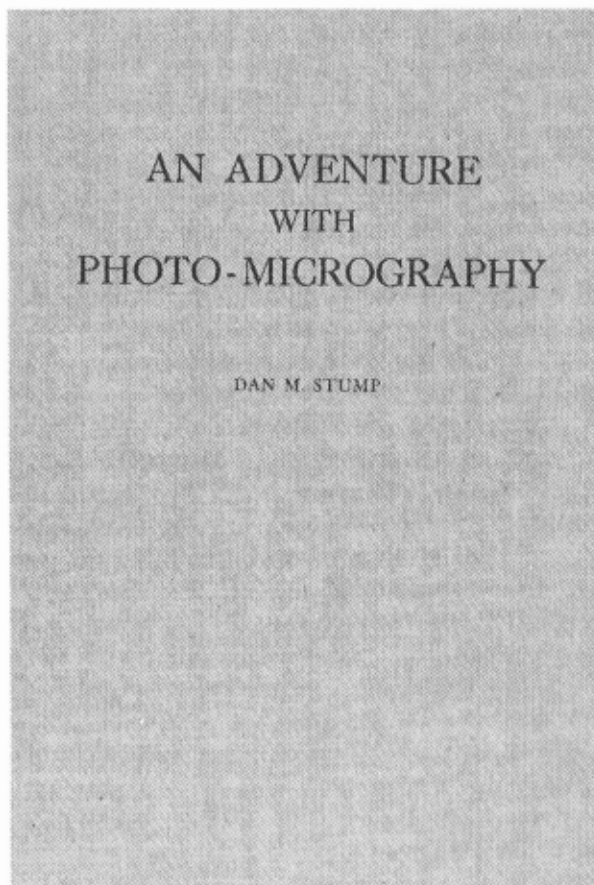


Figure 2. Wheat-colored paper front cover of Dan M. Stump's booklet *An Adventure with Photo-Micrography*, published by the SMSI, April, 1945.

his lighting; the spectacular results are illustrated. In 1945, this 92-page publication sold for 50 cents—"to defray the cost of printing". Dan Stump was from Oak Park, a suburb of Chicago—one wonders if his relatives know about the box full of his booklets that he must have stored somewhere in the attic or basement! How many of these could possibly be left?

Perhaps the publication of Dan Stump's booklet was the jump-start that SMSI needed, because the following month, May 1945, saw the introduction of a new publication, *Micro-Notes*. These were published from 1945 to 1950. The first volume, 1945-1946, was produced with the spirit duplicator, so they are rather modestly done in the typical violet ink. They become increasingly better produced, with the final copy being typeset. Here is the run:

Micro-Notes

Volume 1,	Number 1	May, 1945
Volume 1,	Number 2	August, 1945
Volume 1,	Number 3	November, 1945
Volume 1,	Number 4	February, 1946
Volume II,	Number 1	May, 1946
Volume II,	Number 2	August, 1946
Volume II,	Number 3	November, 1946
Volume II,	Number 4	March, 1947
Volume III,	Number 1	March, 1948
Volume III,	Number 2	June, 1948
Volume III,	Number 3	September, 1948
Volume III,	Number 4	December, 1948
Volume IV,	Number 1	Jan.-March, 1949
Volume IV,	Number 2	April-June, 1949
Volume IV,	Number 3	July-Sept., 1949
Volume IV,	Number 4	Sept.-Dec., 1949
Volume V,	Number 1	Jan.-March, 1950
Volume V,	Number 2	April-June, 1950

Jens Egede Nielsen was the editor of *Micro-Notes*, and the one who single-handedly started it with Volume 1. A limited number of copies of this spirit-duplicated issue were distributed gratis to members of the Society, as well as to selected others worldwide. Volumes II and III were supported by a small payment from the Society. During this time, due to illness in Mr. Nielsen's family, and later his own failing health, I.J. Coldevin, then the Curator of the Society, took on the duties of Editor. Volume IV was issued by Coldevin with the public announcement that *Micro-Notes* had been incorporated as

an independent identity, with the approval of the Society. In 1950, the subscription price to *Micro-Notes* was held to \$1.00 per year. Volume V, Number 2, however, was the last issue. This final issue is a memorial issue to Nielsen, who had died. Nielsen's black-bordered portrait is on the front cover.

SMSI was quite active during the 1950's; meetings were held weekly at times, and hands-on workshops were frequent. Wolfgang Zieler was active in the Society then. Many readers will know him from his two books in *The Microscope Series*—the first two volumes, in fact. I have come across many meeting announcements from this time, but no publications. Then the activities of the Society declined and became almost non-existent, until the mid-1960's when Dr. Walter C. McCrone revitalized the Society, mainly by appointing all of the officers from amongst the employees of McCrone Associates.

Since the Society's Centennial was coming up (1969), plans were made by the Governing Board for a suitable Centennial volume to commemorate SMSI's first 100 years. Accordingly, an Editor was chosen, manuscripts were solicited, and the book was set into type. Then, through a most unfortunate set of circumstances involving finances, misunderstandings, and much high emotion—the details of which are best left for another time—the Centennial volume never came about. However, by 1970, a year after the volume was to see the light of day, there was demand on the part of two contributors that, through some special arrangement, their two contributions to the Centennial volume were to be published as "reprints"—they were actually preprints. One of these preprints issued by the Society was by Wolfgang Zieler:

Zieler, H.W. *Low Power Photomicrography and Photomacrography*. 20-page booklet issued as preprint from (never-published) Centennial volume. State Microscopical Society of Illinois, Chicago, (1970).

The other preprint was by Marigene Butler, then at the Conservation Department of The Art Institute of Chicago, and SMSI's Curator during the Centennial volume time period:

Butler, Marigene. *Polarized Light Microscopy in the Conservation of Painting*. 17 pages; booklet issued as preprint from (never-published) Centennial

volume. State Microscopical Society of Illinois, Chicago (1970).

Both of these preprints were issued in blue wrapper papers. Not many of these were printed up, and I cannot now remember if they were issued to the entire membership or not. The respective authors were given a certain number of copies for distribution.

Five years later, in 1975, the need was felt for some kind of publication for SMSI members besides the meeting announcements. Since *Micro-Notes* was the kind of publication that was wanted, it was decided to call the new publication *Micro-Notes II*. These were edited, at first, by Skip Palenik, and, later, by Ross Brocato. It was here that as a "Contributing Editor" I started a regular column that I called "Diffraction Lines". These columns were later republished in *The Microscope*, and still later, when *Micro-Notes II* ceased, they became a regular feature of *The Microscope*. Here are the details for *Micro-Notes II*:

Micro-Notes II

Volume 1,	Number 1	Sept.-Oct., 1975
Volume 1,	Number 2	Nov.-Dec., 1975
Volume 2,	Number 1	Jan.-Feb., 1976
Volume II,	Number 2	March-April, 1976
Volume (2),	Number (3)	Spring-Summer 1976
Volume (2),	Number (4)	Fall-Winter 1976
Volume (3),	Number (1)	Spring-Summer 1977
Volume 3,	Number (2)	Fall-Winter 1977
Volume 4,	Number 1	January-April, 1978
Volume 5,	Number 1	April 1978-June 1979
Volume 6,	Number 1	June 1979-July 1980

After several years without a publication again, *Micro-Notes II* was revived under the co-editorship of Thom Hopen and Bob Kuksuk. It was issued at irregular intervals:

Volume 7,	Number 1	September 1985
Volume 8,	Number 1	January 1986
Volume 8,	Number 2	July 1986
Volume 9,	Number 1	January 1987
Volume 10,	Number 1	June 1988
Volume 11,	Number 1	July 1989
Volume 12,	Number 1	July 1990
Volume 12,	Number 1	December 1991
Volume 14,	Number 1	December 1992

Note: There are two Volume 12, Number 1's issued in different years; the second Volume 12 was regarded as a Volume 13, and that is why "13" is missing.

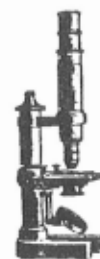
And once again publication ceased—which brings us to the present. Bill Mikuska found himself to be the current Editor of still another phoenix-like revival, but this time, so as to wipe the slate clean and start all over, Bill decided on a new name for SMSI's publication, *μ·Notes 2000*. The premier issue came out in February 1997, and the second issue was in time for INTER/MICRO-97:

μ·Notes 2000

Volume 1,	Number 1	February 1997
Volume 1,	Number 2	July 1997
Volume 2,	Number 1	March 1998

And this brings us to the present issue of *μ·Notes 2000*, and to the end of this narrative. It is hoped that readers who have knowledge of SMSI publications I have missed will bring them to the attention of the Editor for inclusion in a future issue.

From 1872 to 1997; 125 years of publications issued by the State Microscopical Society of Illinois. May the next 125 years of SMSI publications be as interesting and varied as the first 125 years.



A Study in Frustration

Walter C. McCrone*

When I left Cornell (this year will be my 60th class reunion) I had the assurance of my mentor, Prof. Émile Chamot, that there were no technical problems a chemical microscopist couldn't solve or help solve. Nothing since then has made me doubt that observation. What he failed to tell me, however, was that there were some technical problems best left unsolved. In fact, successful solution of good problems in some areas could be harmful to, at least, one's reputation. I refer, as you will have already guessed, especially, to religion or more broadly, to popular beliefs, such as astrology, UFO's, creationism, extrasensory perception, divining rods, fortune telling, poltergeists, etc. Not being forewarned, I naturally said YES! in 1974 when Father Rinaldi, on behalf of the Catholic Church, asked if I would be willing to study the Shroud of Turin.

My proposal for such a study was approved in Turin (1978) and eventually I received a group of two-inch tapes from 32 different areas of the Shroud. On my first day, my first notebook entry included the statement: "My objective is to find out what the image is. It is visible therefore it has atoms and we should be able to analyze those atoms. This will tell us what makes up the image and perhaps how it got there."

On the third day, my notebook says "We should prepare ourselves for a negative finding re: the Shroud. I had seen only colored inorganic particles in the image areas and they were absent in non-image areas. A few more observations of shape, size, color, and refractive index forced me to conclude they were red ochre ($\text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}$). They were 10 times smaller than red blood cells and their refractive indices were close to 3.0, far higher than the 1.55 index for most biological particles. At that point I made my second mistake (the first mistake was saying "YES!" in 1974). I decided to warn STURP that I was finding strong indications that the Shroud was a

painting and we should be careful in how we proceed. My "warning" was a "lead balloon" and quickly lead (no pun intended) to my ostracism from STURP.

I continued to work diligently to confirm or deny my early conclusion but within a year I had proof that the entire visible image was produced by an artist using essentially a very dilute water-color paint. During the second year my efforts crystallized as rock-hard conclusions. There was no blood on the Shroud and two paints had been used. One of red ochre in a gelatin vehicle for the entire image but a second paint had been applied to the "blood-image" areas. This paint was vermilion in a gelatin vehicle. McCrone Associates personnel then applied a variety of electron optical, spectroscopic and diffraction techniques to fibers and particles from the same tapes. They confirmed the presence of red ochre, found the vermilion I had missed, and identified hematite (Fe_2O_3) and vermilion (HgS) by electron microprobe and X-ray diffraction. Three papers covering two years of work were published in *The Microscope* 28, 105 and 115 (1980) and 29, 1981.

At this point (1980) I was happy and expecting recognition of the conclusion that the Shroud was painted at the time it first appeared in the historical record (1356). I soon found my hopes were premature and, since then, the time of prematurity has increased. In "Judgement Day for the Turin Shroud," I mentioned the year 2279 for my present prediction as the year for the Pope's first announcement that the Shroud is a painting.

So what is the present situation? In a nutshell, STURP and company first proceeded to "demolish my work and later, convinced this had been done, proceeded to assume what *should* be present on an authentic Shroud had to be there so they found it. The image was blood. McCrone was discredited and could be ignored¹.

Proceeding with McCrone out of the way

¹McCrone did not participate in the Turin exercise in 1978 but he was loaned some of the samples of Shroud surface debris.... After hurried superficial evaluation, he rushed into print.... In a scholarly and orderly manner, scientists, Adler and Heller and others proceeded to totally demolish the McCrone charges. The claim of McCrone—for the presence of an iron oxide pigment bound with an age-yellowed animal binder was disproved in every particular."

they could work on—how the image was formed. Fortunately(?) I am kept up-to-date by almost daily reports on “the situation” from the Holy Shroud Guild. One popular idea is that the resurrection created the image as the body *emitted* through the cloth. This theory had the advantage that it seemed reasonable to assume this event would, for their purposes, change the carbon date of the Shroud (to 1325 A.D.). Unfortunately, we have no standards from any such an occurrence so it is difficult to evaluate that possibility. Many others had competing ideas and one Shroud enthusiast remarked that he had no time to argue about religion or to debate with people who still think Joe Nickell or McCrone solved the mystery of the Shroud.

Some other bizarre suggestions include:

1. STURP’s final conclusion is that the image is due to oxidative dehydration of the linen.
2. Myrrh and frankincense on the body stained the cloth
3. Leonardo da Vinci (1452-1519) developed a photographic method and produced the Shroud in 1355!
4. SHC (spontaneous human combustion).
5. The carbon date is wrong because of biological contaminants on the Shroud. This ignores the fact that biological contaminants of double the weight on the Shroud itself would be required to change the date from the first century to the 14th century.
6. One well-known criminalist found 54 species of pollen on the Shroud most of them from trees and plants that grow in Palestine but not in France or Italy.
7. Not to be left out, a Russian physicist proposed that the Kirlian effect as the mechanism of image production.
8. Some STURP members said: “No, more likely the Volckringer effect caused the image.”
9. The fire in Chambery changed the carbon date ignoring the fact that all carbon-dating

samples are burned to carbon dioxide during preparation for dating. It is well known, however, that nuclear decay constants are temperature independent. Furthermore, the standard carbon-dating procedure involves burning the entire sample to CO₂.

10. A Swiss archaeologist thinks the carbon-dating samples came from a portion of the Shroud added during the Middle Ages.

11. Another criminalist found the blood on the Shroud to be type AB positive. Someone later said “NO, it’s AB negative.”

12. Several see imprinted images of coins on the eyes and others find many (28) species of flowers from Palestine, plus images of a nail, hammer, broom, ring of thorns, etc.

13. Finally, a book has now appeared covering an imprint “Jesus-Nazarene” they see on the Shroud. This, they say, is the sign-off by the Roman authorities on the crucifixion.

That’s not all by any means but it’s more than enough to spell out “frustration” for anyone who is convinced by the scientific evidence presented in “*Judgement Day for the Turin Shroud*”. I keep thinking about something another Cornell Professor (Hans Bethe) said:

“Science is always more unsolved problems but its great advantage is you can prove something is true or something is false. You can’t do that about human affairs—most human things can be right from one point of view and wrong from another. It is the most wonderful feeling when you come to a real answer. This is it and this is correct! In science, you know you know.”

How about these Shroud enthusiasts who find blood by all of the forensic tests when there is no blood on the Shroud? My suggestion is simply incompetence or deceit.

So, where does “Frustration” come in? I’m sorry to say it’s my present feeling about the Shroud. I know I know and my peers agree with me but a large cadre of non-scientific zealots continue to cause frustration for objective scientists.

**State Microscopical Society of Illinois
1997 Treasurer's Report**

CATEGORY	AMOUNT	BALANCE
A - Balance, January 1, 1997		\$17,899.90
 <u>INCOME</u>		
Dues	\$2,005.00	
Interest	\$393.56	
Auction (INTER/MICRO-97)	\$2,318.00	
Silent Auction (Amateur's Night)	\$383.50	
Workshop net (receipts/expenses)	\$20.00	
Meeting Receipts for Refreshments	\$795.50	
<i>Total Receipts</i>	<u>\$5,915.56</u>	
 <u>EXPENSES</u>		
Meeting Flyers, Postage	\$628.58	
Meeting Refreshments	\$1,114.70	
Meetings, Honoraria	\$700.00	
INTER/MICRO, Honoraria	\$300.00	
INTER/MICRO, Speaker's Expense	\$53.12	
INTER/MICRO, Award engraving	\$188.40	
Picnic	\$106.06	
Stationery, printing envelopes	\$85.00	
μ-Notes, preparation	\$930.00	
μ-Notes, printing	\$1,724.98	
μ-Notes, envelopes and postage	\$325.34	
Commemorative Microphotographs	\$987.50	
Microscope purchases	\$1,150.00	
Prize for Amateur Night	\$25.00	
Greeting Cards	\$4.01	
<i>Total Expenses</i>	<u>\$8,322.69</u>	
Ending Balance, December 31, 1976		\$15,492.77
 <u>Summary</u>		
Beginning Balance - Jan 1, 1996		\$17,899.90
Total Receipts		\$5,915.56
Total Expenses		\$8,322.69
Ending Balance - Dec 31, 1996		\$15,492.77

Arthur L.E. Barron

Arthur Lawrence Edward Barron (born Dunedin, New Zealand, 12.10.14) family returned to England 1916. My interest in microscopy began early when my father bought me my first microscope at age 13. My education at Watford Grammar School in Hertfordshire was clouded by ill-health and because I was excused sport, I was able to spend the weekly sports afternoon in the school physics laboratory experimenting with microscopy and photomicrography. It was there that I made my first photomicrographs using a projection microscope to record images on glass plates in the darkroom. Incidentally, one of these plates I still have. It was also fortunate that Watford Public Library had a very good microscopical section which included many of the classics such as Carpenter & Dallinger, Cross & Cole, and Spitta, as well as a good selection of natural history books: these I was able to borrow for home study. When I left school I joined my father in his book exporting business and entered the book publishing world.

It was in 1937 that I conceived the idea of publishing a journal for amateur microscopists but had I known what was to face Europe within just two short years, I doubt whether I should have had the temerity to do so. However it was launched with the aid of a staunch band of contributors, many of whom became good friends. Looking back now I marvel at the fact that we were able to keep it going (just) through all the difficulties and shortages of 1939-45.

During the war I worked for the Hawker Aircraft Company at Colnbrook just outside London, producers of the Battle of Britain Hurricane fighter, which, with the Spitfire, fought the battle of the skies. I started as an airframe inspector and later helped with the setting-up of the metallurgical laboratory in which I worked for the rest of the war.

After a few further years with my father I was offered a job with C. Baker of Holborn Limited, microscope manufacturers, preparing their catalogues and publicity material. It was during this spell that I made the acquaintance of Professor George Cunningham of the Royal College of Surgeons of England, who invited me to join his team as microscopist and photomicrographer at the College in Lincoln's Inn Fields. They were happy years there with a fine bunch of colleagues and interesting problems in both microscopy and photomicrography.

When Professor Cunningham retired he introduced me to Professor Thackray at the Middlesex Hospital in London and I joined the Cell Pathology Unit there, where I spent the rest of my working life as microscopist and photomicrographer, again with a fine group of people.

As for *THE MICROSCOPE*, in 1962 I felt I just was not able to carry on with it and as you will know, Walter McCrone took up the journal, producing it first in Britain and then transferring it to Chicago. When I receive my copy each time I marvel at the way things have changed but, after all, 60 years is a long time.

In the 1930s I joined the Postal Microscopical Society, the Photomicrographic Society (which merged with the Quekett Microscopical Club later) and the Quekett Microscopical Club, for whom for a spell I edited the *JOURNAL*. I was also for a time a Fellow of the Royal Microscopical Society.

I have few claims to fame in the world. The Photomicrographic Society presented me with the Martin Duncan Certificate for 1938/39 and the Barnard Medal for 1947/48. After the war I gained the Fellowship of The Royal Photographic Society with the photomicrographic work I did for Dr. Miriam Rothschild in illustrating two volumes of the *ILLUSTRATED CATALOGUE OF THE ROTHSCHILD COLLECTION OF FLEAS (SIPHONAPTERA) IN THE BRITISH MUSEUM* and which was published by the British Museum in 1953/56. At the request of Chapman & Hall I updated and rewrote their out of print book by C.W. Olliver: *INTELLIGENT USE OF THE MICROSCOPE* and which was published in 1965 as *USING THE MICROSCOPE*. The Quekett Microscopical Club, whose meetings I attended regularly until recent times, awarded me Honorary Membership in 1965.

I am not now an active microscopist, although I look forward to reading the literature still, and I hope you will forgive me for mentioning to a Society such as yours that I have now gone back to my much-loved early hobby of stamp-collecting, although even here the microscope has its uses.

May I end by thanking the State Microscopical Society of Illinois for the bestowal of the August Köhler Award for 1998 and to express my pleasure at receiving it.

**1998 State Microscopical Society of Illinois
August Köhler Award Recipient**



Arthur Lawrence Edward Barron