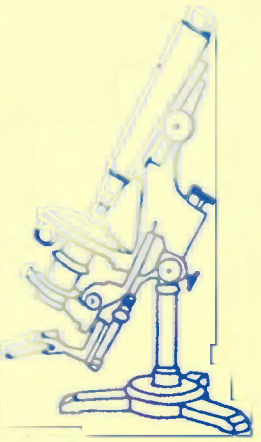
μ • NOTES 2000

Vol. 1 No. 1 February 1997



A Publication of the State Microscopical Society of Illinois

EDITORIAL

In the beginning there were four essences; air, earth, fire, and water: from these all matter was made—save for the heavenly bodies. For these, a fifth, quintessence, was required. Today, although we recognize many more essences (elements), some of us impassioned microscopists still only need five. Silicon and oxygen give us glass, and copper alloyed with tin or zinc give us brass. Glass and brass known to many cultures for centuries lack merely the fifth essence, the quintessence, the idea. An idea that changed the history of mankind.

A bit of glass, a bit of brass, and the idea can yield either the microscope or the telescope—two scientific instruments that are extensions of man's keenest sense, sight. So important were these instruments that specific scientific societies focused their very being on them. Both instruments, however, have evolved to capitalize on regions of the electromagnetic spectrum other than the visible. Even the notion of what a microscope is has evolved to the extent that sight is no longer directly involved; *viz.* AFM!

Today students of science are taught about modern, state of the art instruments. Are the optical forerunners "*passé*?" Perhaps, the enigmatic living organism provides an answer to this question. Of all types of microscopes only the visible light microscope enables us to study living matter non-destructively *in vivo*, living matter without a suit of golden armor. To be sure, all types of microscopes provide useful information about a system, not just the light microscope. But, only the light microscope allows us to look directly at what we are about, *in vivo*.

The light microscope and what it allows us to see first hand had also stimulated a group of gentlemen in 1868 to form the embryo of our society. They also had the foresight to include in the society's first journal, *The Lens*, that all items of scientific interest will be of import to members.

With this concept in mind, this reincarnation of *The Lens* in the form of μ • NOTES 2000 presents a cross-section of articles. Whether informing us about capturing the beauty of nature to let us study "her" in leisure (Benko), delighting us with the art of well-machined pieces of functional apparatus for the light microscope (Delly), proclaiming the frustrations of dealing with those who would deem to change the physical laws (McCrone), or explaining the nature of labradorescence (Ziemba), these articles and others will, I hope, stimulate a renewed interest in SMSI. As editor, I am essentially a microscopic part of a much larger living sample. If living things respond to stimuli, I urge you as microscopists to perform the experiment, take an active part to probe the sample, and see how it responds. This is, therefore, a call for papers with, perhaps, even letters to the editor. As with the forerunners of μ • NOTES 2000, SMSI members will have opportunities to exchange/swap equipment, books, samples, and, quintessentially, ideas.

Bill C. Mikuska

μ • NOTES 2000

Volume 1, No. 1, February 1997

Bill C. Mikuska
Editor

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

Contributions should be addressed to EDITOR, μ • NOTES 2000, 2820 S. Michigan Ave., Chicago, IL 60616. Telephone: (312) 842-7100

Founded In 1868

Cover: BISCO zone melted crystals, SEM 5000X by Richard H. Lee.

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Filter Holder and Specimen-Manipulation Apparatus Designed and Built by Charles H. Kruchten

by John Gustav Delly*

Sometime between October 1965, when I had just finished preparing a *Catalogue of Publications In The Library of The State Microscopical Society of Illinois*, and about 1970, The State Microscopical Society of Illinois (SMSI) was quite active in several areas: Leon Urbain, SMSI Honorary Member and Past President, was teaching the Young People's course on Saturday mornings at the Chicago Academy of Sciences, and the Society was holding well-attended, regular meetings, conducting workshops, and sponsoring an annual three-day symposium. During one of the Board of Governors' meetings, it was suggested that the Society should initiate an Adult Amateur Microscopy Group to complement the one for youngsters. I was chosen to head up this new group, which was to meet at the Chicago Academy of Sciences on Friday evenings. Everything was going fine until winter arrived. Most of the folks in the group were retirees, who simply couldn't or didn't want to travel to the Academy when it was cold, snowy, and icy, and, as a result, the group stopped meeting.

A year or so after the group disbanded, one of the former members, Charles H. Kruchten, who I believe may have been an SMSI member in years past, telephoned me to say he had a microscope to sell, and a couple of books to donate to the Society; I told him I was interested in buying the microscope—a Leitz binocular magnifier—and went to his home to conclude the transaction. As I was leaving, he told me to take two pieces of apparatus that he had designed and made for use with the microscope, because he would no longer have any use for them. One of the items was a device to hold either 2" x 2" square filters, or a 2" diameter filter beneath the substage condenser. The other item was a specimen holder/manipulator for use with a stereomicroscope or binocular magnifier. Both devices were beautifully executed. I had some machining experience, and immediately recognized the work of a professional. When I commented on the high quality of the work, Charles told me that before he retired, he had been a modelmaker at CENCO, the well-known scientific apparatus supply house in Chicago, where he designed and constructed the prototypes for much of the apparatus that CENCO sold.

He had made the two pieces of microscopical apparatus for his personal use, and, as far as I know, no others were made. I have used these two devices for over 25 years now in my own home laboratory, and they function as beautifully now as they did when he gave them to me. I would like to take this opportunity to describe for SMSI members some of the features of these ancillary microscopical devices, designed and built by Charles H. Kruchten.

MICROSCOPE FILTER HOLDER

Filters for use in visual microscopy and photomicrography should not be placed anywhere in or near the conjugate foci of the field (lamp) diaphragm. This dictum is often violated by users placing filters directly over the light exit ports in the base of the microscope, and by manufacturers who build filter holders immediately in front of the field diaphragm on detached illuminators.

The best place for a filter is in or near one of the conjugate foci of the aperture diaphragm. For image-quality purposes, the plane selected should be as near as possible to the aperture diaphragm itself. The old method of having a filter carrier built-in, or swing-in/swing-out immediately on the bottom of the substage condenser was excellent. Like many other good ideas, this has been dropped in modern manufacture. The only disadvantage to this location is that each

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manufacturer had a different diameter filter carrier, e.g., 32 mm, 33.5 mm, etc., and so, one was restricted to the blue, green, and frosted glasses usually supplied.

Charles H. Kruchten's Microscope Filter Holder answers all objections. It will be seen from the photograph (Figure 1) that this device will hold all 2" x 2", 2" diameter, and, through adapters, any smaller size filter immediately under the substage condenser. It is fully adjustable for height and distance to bottom of substage condenser. Further, the filter-carrying rod is fully rotatable about its axis so that filters may be introduced into any light path, regardless of angle. The filters are held in place by two spring clamps. All parts are made of aluminum, or nickel-plated brass.

For those interested in specifications, the base is 3" x 2-5/8" x 1/2"; the turned base for the upright rod is 3/4" high; the upright rod is 6" long and is made from 5/16" rod—notice the 3/32" diameter hole near the top of the upright for the insertion of a tommy-bar to secure the upright to the base; the beautifully-made split bracket has one knurled thumb screw to secure the bracket to the upright, and another, somewhat smaller, to secure the position of the filter-carrying rod. Looking at the base, you will see that all edges are gently relieved, so there are no sharp edges.

The filter carrier itself is milled from aluminum, and has been blackened. It is dimensioned to hold 2" x 2" square filters beneath spring clips. In the photo you may be able to see that there are two 3/16" wide ledges milled in to serve as edge-supports for the filter. A 1-3/4" circular opening has been provided in the filter carrier. The filter carrier is glued and screwed (3-screws) to the bracket at the end of the 5" long, 1/4" diameter filter-carrier rod. The entire device, without filter, weighs 371 grams.

SPECIMEN HOLDER/MANIPULATOR

Charles H. Kruchten's beautifully-designed and made specimen holder/manipulator for use with stereomicroscope or binocular magnifier is illustrated in Figure 2. Its function will be immediately apparent. Any specimen is impaled/glued, squeezed or otherwise secured to a needle-like holder made from a modified jeweler's screwdriver blade, and chucked into the end of the jeweler's screwdriver holder that protrudes from the device. It will be obvious from the photo, that a conventional jeweler's screwdriver has been modified so as to serve as an extension for a rotatable specimen-carrying arm.

The specimen is rotatable via the 13/16"

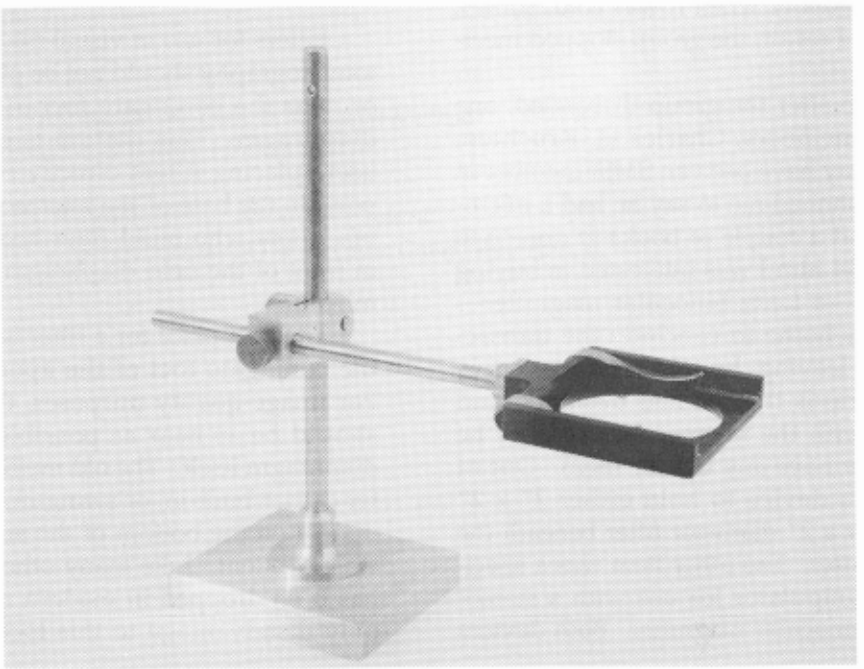


Figure 1. Microscope filter holder, designed and made by Charles H. Kruchten.

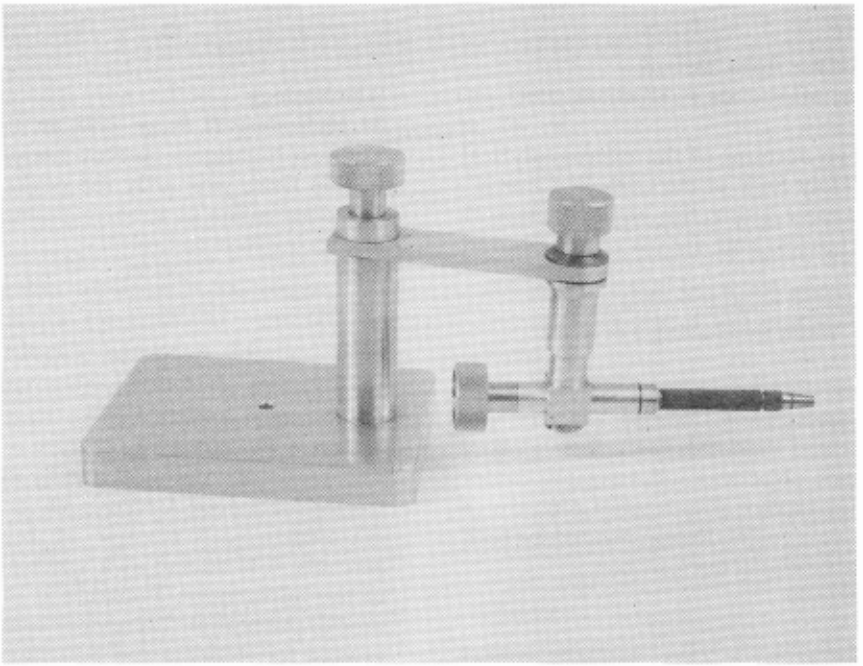


Figure 2. Microscope specimen-manipulation apparatus, designed and made by Charles H. Kruchten.

diameter x 3/8" knurled knob, against built-in phosphor bronze bearings. The rotating arm extension that the screwdriver clutch sticks out from is 2-1/8" long. The rotating specimen holder is held in its plane by a vertical extension, which is itself fully rotatable on bearings via a 3/4" diameter x 3/8" knurled knob at the top, which also acts to lock the azmuthal rotation of the specimen holder.

The vertical extension is, in turn, attached to one end of a 3-1/4" long x 3/16" bar, the other end of which is attached to a 2-1/8" x 3/4" diameter upright by still another knurled knob 7/8" diameter x 5/16". The upright is attached to a 4" x 3" x 1/2" base. The many degrees of freedom of adjustment are apparent from the photograph. The entire device weighs 447 grams, or just about a pound—heavy enough that the base does not need to be attached to a substrate, even though a hole in the base has been provided for that purpose. Again, the edges are all relieved, and all bearing surface operate very smoothly; all screws are countersunk and flush.

The only part on this device not made by Charles H. Kruchten is the jeweler's screwdriver extension. He used a brand I am not familiar with; it is not a Starrett or Lufkin.

Several specimen holders are provided, the most general one being a 0.025" jeweler's screwdriver blade, which has been ground down to a point.

I am very pleased to be the recipient of both of Charles H. Kruchten's "one-of-a-kind" ancillary apparatus for the microscope. I admire their beauty, precision, and exquisite craftsmanship, in addition to their utility.

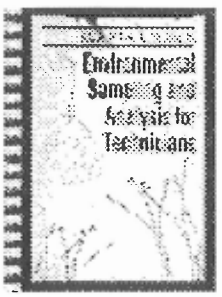
NEW BOOKS AT MAC

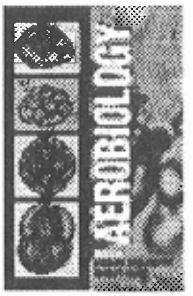
Judgement Day for the Turin Shroud
Walter C. McCrone, Microscope Publications, 1997, pp. 350.
Dr. McCrone's own account of the microanalytical research on this "Holy Relic of the Catholic Church". To be published in early spring, this work will have 11 color plates, 68 figures, and details of the Shroud research since 1969.
#537 $28.00 (Special prepublication price)
$24.00 ea. when purchasing 2 or more

The Practical Use of the Microscope
Charles C. Thomas, Springfield IL, 1958, pp. 493.
Available for a limited time, this book details many types of microscopes, objectives and accessory equipment, and provides explicit step-by-step procedures on how to use this equipment. Recommended for both the amateur and professional microscopist.
#534 $70.00

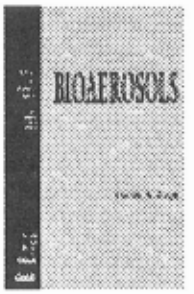
Manual of Mineralogy - 21st Edition

C. Klein, C.S. Hurlbut Jr., John Wiley & Sons, Inc. 1977, pp. 667.
Provides an up-to-date understanding of basic concepts and principles in crystallography, crystal chemistry, chemistry, physical aspects of minerals, and intro petrology concepts essential to understanding of genesis of minerals and rocks; and is a quick reference for unambiguous identification of the common minerals in the field and lab. This text is filled with diagrams, photomicrographs, tables and charts.
#536 $80.00

Environmental Sampling for Unknowns

Kathleen Hess, 1966, pp. 298
Modern approaches to indoor and outdoor environmental sampling with an emphasis on identifying unknowns is covered in this text. Also included is information for purchasing equipment, sampling strategies, sampling and analysis protocols, and several charts, tables, photographs, and drawings.
#538 $59.95

Aerobiology


M. Muilenberg, H. Burge, 1996, pp. 155.
The text highlights current interests in this field, primarily the ecology and distribution of airborne particles and their effects on health.
#541 $39.95

Bioaerosols


Harriet A. Burge, 1995, pp.318.
This work was commissioned by the Center for Indoor Air Research as a state-of-the-art review of bioaerosols. It is offered as a synthesis of the information available at the time of publication. It provides background for students and practitioners of air pollution research and should be a valuable resource in this field.
#542 $84.95

Bioaerosols Handbook


C.S. Cox, C.M. Wathes, 1995, pp. 621
The handbook provides up-to-date knowledge and practical advice from established authorities in aerosol science. It covers principles and practices of sampling, descriptions and comparisons of samplers, calibration methods, and assay techniques. It also offers critiques concerning handling samples to provide representative and meaningful assays for their viability, infectivity, and allergenicity.
#543 $79.95

Wastewater Organisms - A Color Atlas


S.G. Berk, J.H. Gunderson, 1993, pp.98.
Serves as a guide to the organisms commonly found in wastewater treatment facilities. It should aid in identifying organisms to the genus level, more detailed techniques in the literature cited.
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Some Hints for Snow Fun

by James Benko*

Over the years I have made many snowflake replicas on 3 x 1" slides using the Schaeffer replication technique. This technique of using a 1% Formvar resin solution in ethylene dichloride has been detailed previously in *The Microscope* [Vol. 43:4, pp. 195-197]. Basically a drop of resin solution is put onto a snowflake on the slide and allowed to evaporate in the cold. After about 30 minutes, the slide can be taken inside for further study and photography. The resin makes a perfect (relatively permanent) replica of the snowflake.

Some helpful hints I have found to expedite the replication process are given below.

1. STORAGE

To maximize the number of slides that are allowed to evaporate in the cold, I have found that a small metal filing cabinet used to horizontally store 3 x 5" cards in plastic holders works just fine. I believe it is called a CARDEX file. The cabinet has six drawers that are approximately 6 x 18" and each can hold around 25 slides. I found mine in a surplus office equipment store for around $10.

I can prepare a number of slides at one time and then examine them later (usually the next day). The cabinet is built such that snow can't enter the drawers. The metal acts as a cold sink while the slides are protected from the elements. The whole arrangement works quite nicely.

2. REPLICATING SOLUTION

Eventually the solvent evaporates from the Formvar resin, depending on the type of cap used in the storage bottle. One of the best vials, I have found is the 7 mL vial sold by Wheaton (and others) that comes with a plastic cap having a Teflon liner. I like to keep several small vials filled with Formvar outside, ready for use. If one spills, which sometimes happens, not much is lost. If it is snowing hard, not much snow can get into one vial between usage. One can always use another one if too much snow gets into one vial.

The right cap and liner can minimize evaporation. I have had some vials for several years with replicating solution in them without much evaporative loss.

Even if all of the solvent has evaporated, additional solvent can be added to the dried resin without much detriment to its replicating ability.

Chloroform can be used as a substitute for ethylene dichloride.

Formvar solutions can be colored with felt tipped markers, highlighters, etc., to provide some color to the replicas. Just dip the pen into the Formvar solution until the solution becomes colored. The replicas made with colored Formvar sometimes show internal detail better than "plain" Formvar.

3. TRANSFERRING DEVICES

A fine tipped plastic rod can work quite well in transferring flakes from a black cloth to the slide, instead of using a fine brush. Such a rod can be made from a disposable plastic hypodermic syringe or pipette tip. The tip can be drawn out after heated in the flame of an alcohol lamp. A small bead on the end of the tip wetted with replicating solution helps keep the flake attached during transfer. It seems like static electricity also helps. In cold, dry air the flake seems to leap onto the plastic tip and becomes attached for the transfer operation.

4. GLASS SLIDE SUBSTITUTES

It's fun to try the replicating resin on other substrates besides glass slides. One substrate

* Microspec Analytical, 3352 128th Avenue, Holland, MI 49423

that can be used is the heavy paper board used for packaging baked goods. Many times this packaging material has a metallic gold finish that provides a good colored, reflective background. After washing excess frosting, etc., off the board and drying, it can be cut into 1 x 3" strips and used in place of glass slides. Foil pans can be used in the same manner.

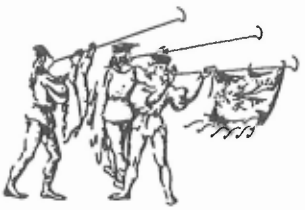
5. PICTURES ON COMPUTER DISCS

Film developing companies like Seattle Film Works (800-445-3348) will develop PRINT film into prints and slides, and also put the images on a floppy disk all for around $20. Photomicrographs of any subject can then be put into word processing documents, etc., with free software they provide that converts the images to other popular formats. Snowflakes, of course, make interesting images.

6. LATEX RUBBER GLOVES

I often work outside in the cold making replicas without gloves. A handwarmer in the pocket provides needed warmth but there is a limit to how long anyone can do this without going inside to warm up. Latex gloves also provide some protection, while still allowing the dexterity needed to handle slides and other items necessary for the replicating process. When I wear latex gloves, I can usually stay out 2-3 times longer than when working without gloves.

This winter looks to be quite promising to have SNOW FUN with the microscope. I plan to put on my earphone, earmuffs, with a classical music selection in my tape player, put on my insulated boots, and a warm coat and hat and try to look for that rare 18 pointer.



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Instruments, techniques and applications are covered at the 49th annual INTER/MICRO meeting sponsored by McCrone Research Institute. Though an emphasis on polarized light microscopy, other instruments such as electron, optical, x-ray, acoustic microscopes and probe microscopes for problem solving in the materials and biological sciences are well represented. These tools and supplementary techniques such as EDS and FTIR are referenced in the wide array of presentations. Sessions are offered in **Hi-Tech** microscopy, **General** interest, **Forensic, Environmental** and **Art** and **Archeological** topics.

Most papers fit the 15-20 minute timeframe including the question period. The MICROSCOPE Journal serves as "Proceedings" for the meeting and papers are published after peer-review in the order they are received. We invite attendance and a title from all interested industrial, academic or governmental microscopists. Submit your title to help accelerate the renewing interest in microscopes and receive a 65% reduction in meeting registration fee.

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Ferrihydrite—An Unusual Material

by Richard H. Lee*

In 1994, I had the occasion to work with a graduate student from the Environmental Assessment Division of Argonne National Laboratories who wanted to better characterize dried ferrihydrite powder. They mainly wanted to obtain some high magnification photomicrographs in order to estimate particle size and porosity.

This seemed like a straightforward project until we saw what the particles looked like. Porosity was difficult to estimate because there were two kinds of particles and one appeared to be unexpectedly smooth. As seen from Figure 1, we discovered the expected iron oxide flakes, but also a wormy looking phase (b). A little research on the properties of ferrihydrite indicated that, if pure, it should be hydrated hematite; $Fe_2O_3nH_2O$ with n values between 1 and 3, and rhombehedral in structure. The usual crystal morphology was reported to be spherical, and 5-10 nm in size.

Clearly, what we had here was something different! After our SEM photographs were taken, we set out to do some EDS X-ray microanalysis. Figure 2 shows the typical spectra with mostly iron and definitely some sodium, which was not supposed to be there. So, I sent a small sample off to JEOL's Applications Lab in Peabody MA for more photos. I was having difficulty resolving the fine surface structure of the particles, and I thought that a low-voltage Field Emission Gun SEM would do a better job.

Some additional background may be useful here. The goal was to estimate the surface area that controls the sorption in the soil environment. Ferrihydrite is a key material that controls soil contaminant sorption. Literature values of the surface area range from 200 to 840 m^2/g. The

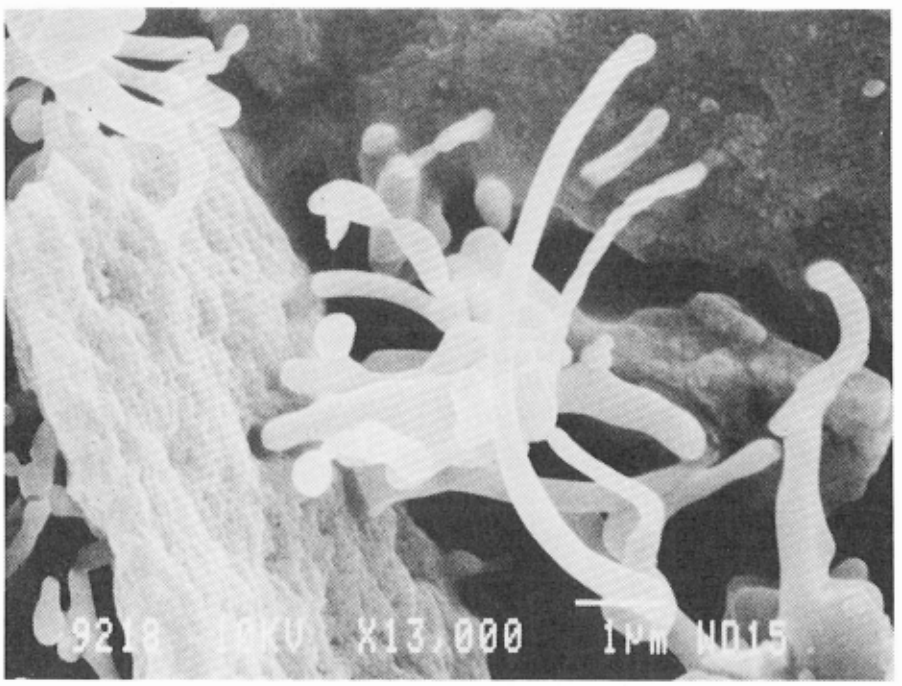

Figure 1

* Argonne National Laboratory, Argonne, IL 6043; E mail: Richard_lee@qmgate.anl.gov

flake-type phase is shown in Figure 1 and indicates a very fine and uniform porosity which could be computer analyzed with image analysis software to find the percent of the dark area. Figure 3 is a photo showing phase "A", the flake-type phase in the background, and "B", the smooth "wormy" phase.

Again, the "A" phase is iron oxide with sodium and some sulfur present. Another spot analysis of phase "B", indicates mostly sodium, oxygen and carbon, with very little iron and a small amount of sulfur. The SEM was used at 5 kV for EDS microanalysis since there was some charging due to high resistivity and very fine particle size.

It appears to me that somewhere in preparing the samples, such as slow drying, some new phase has been created which could be the result of contamination. In my opinion, the sodium came from volatile residue in the oven. Keeping ovens clean is not an easy task, as students learn not to assume they are.

Does anyone have experience with this material or fine particles of iron oxide in general? I'd like to resolve this question of identity, mainly out of curiosity. But it was a challenging material to work with and requires the best resolution possible.

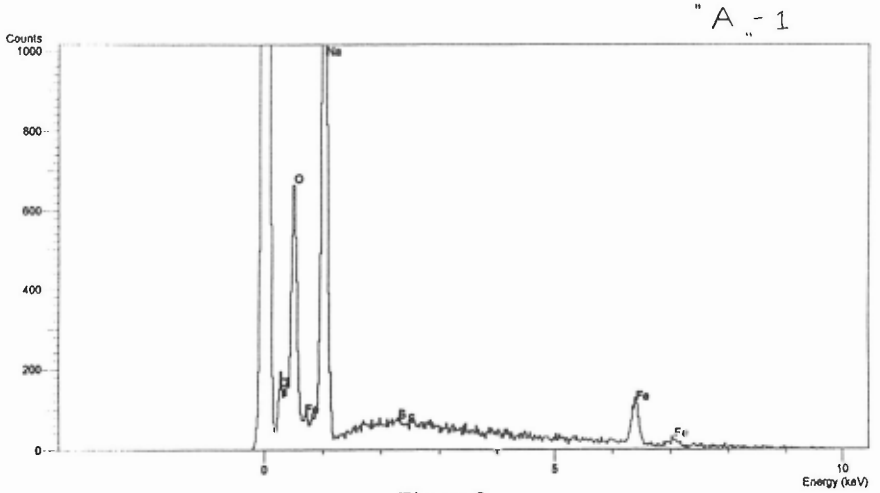

Figure 2

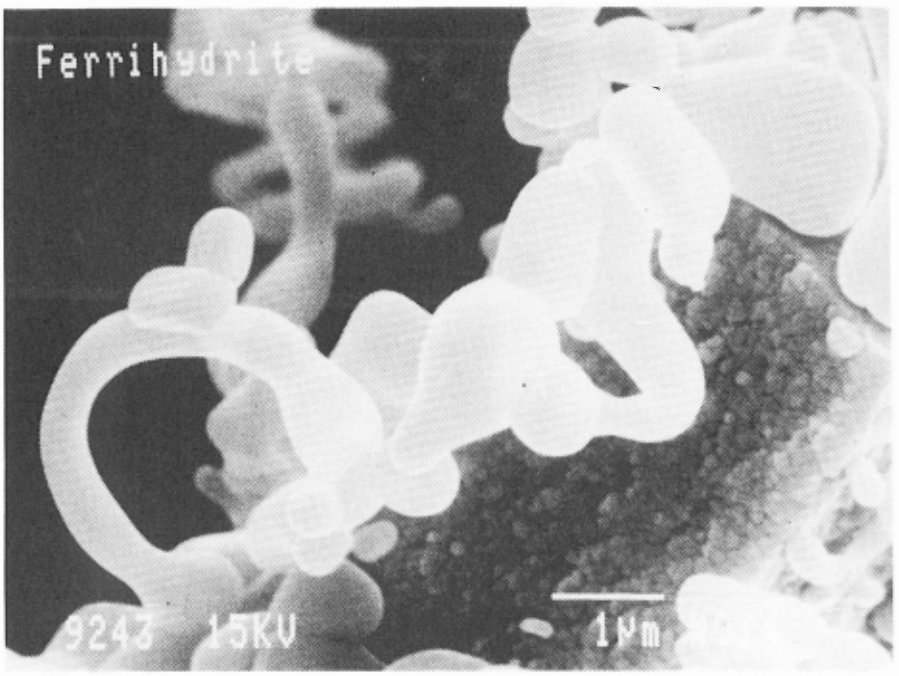

Figure 3

Preparation and Use of Needles for Manipulating Small Particles

by Anna Teetsov*

To successfully isolate samples from 1-100 μm for microscopic examination or analysis by other instruments, one needs a good set of microtools with the most essential one being the needle. This paper will describe how to make six commonly used needles and how each one is used.

Those most commonly used needles are fine, medium, curved coarse and flat tungsten needles, polyethylene needles and eyelash needles. These needles and their primary uses are summarized in Table I. The relative sizes of the six needle tips are shown in Figure 1.

TUNGSTEN NEEDLES

Twenty-four gauge, 520 μm, tungsten wire is cut into one inch lengths. Since tungsten wire is very brittle, care should be taken to avoid fracturing the ends.

A procedure for making tungsten needles has been carefully described in *The Particle Atlas*, Edition Two, Volume I, by McCrone and Delly. The procedure is as follows: the tip of the tungsten wire is heated until it is red hot; then it is quickly dipped into $NaNO_2$ powder and the

TABLE I

NEEDLES FOR MICRO SAMPLE PREPARATION

Needle Type	Primary Use
Very fine tungsten	Manipulating particles <20 μm
Medium tungsten	Manipulating >20 μm-100 μm
Coarse, curved tungsten	Manipulating drops of solvents
Flat tungsten	Scraping fine residues off
Polyethylene	Performing aqueous extractions
Eyelash	Manipulating samples on very fragile surfaces

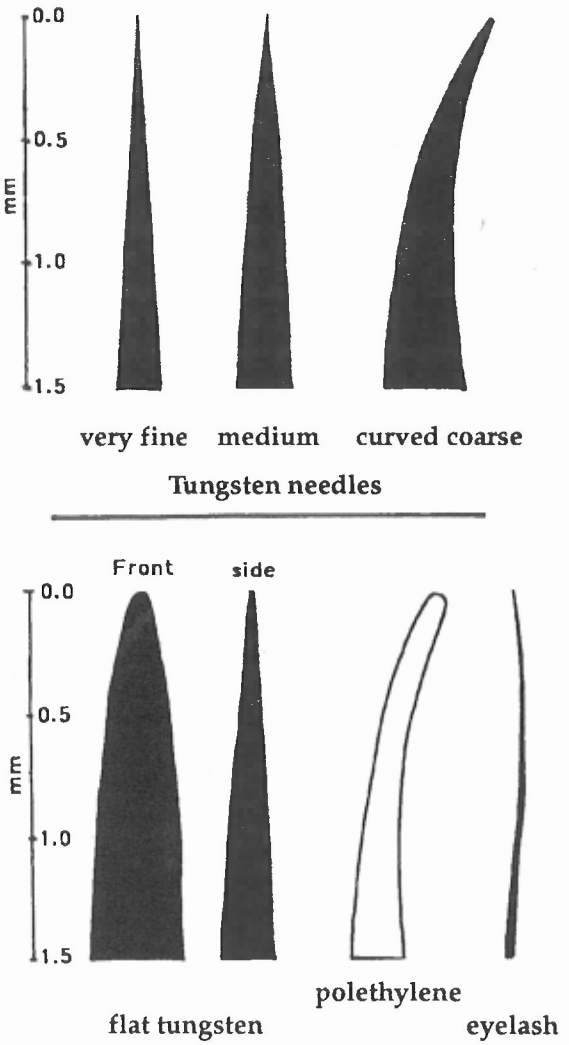

Figure 1. Relative size of the six needle tips.

* McCrone Associates, 850 Pasquinelli Drive, Westmont, IL 60559

ensuing exothermic reaction is allowed to proceed for 1-5 seconds. The tip of a freshly cut tungsten wire may require 5 seconds to etch and form a very fine needle. One second may be sufficient to resharpen a used needle. Over the past 20 years minor changes have been made in the procedure described in *The Particle Atlas*. The original procedure and changes are illustrated in Figure 2.

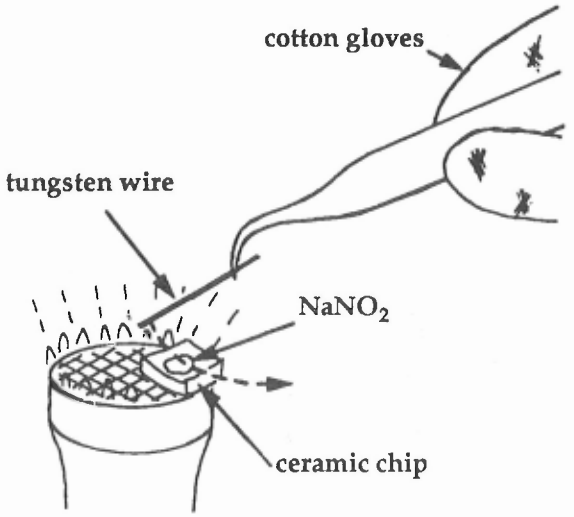

Figure 2. Sharpening tungsten wire with $NaNO_2$.

No special techniques are necessary for making fine, medium, coarse or flat needles. If a large quantity (>50) of needles is made at one time, one will obtain approximately equal amounts of each type plus rejects. Rejects are needles with round tips, double tips (resulting from fractured wire at the tip), and needles with uneven taper. These needles can be resharpened.

Freshly made needles are placed in a petri dish lined with paper and the excess $NaNO_2$ is removed by flowing a very fine stream of warm water into the petri dish for a few minutes. After decanting the water, the needles are sorted into five categories: fine, medium, coarse, flat and rejects. A large assortment of fine and medium needles should be on hand.

To make a curved, coarse tungsten needle, apply pressure with a tungsten carbide scribe just above the tip of a coarse needle as shown in Figure 3.

Since most micro sample preparations can be successfully accomplished if the proper needle is used, it is important to choose the correct needle from the beginning; therefore, I suggest storing

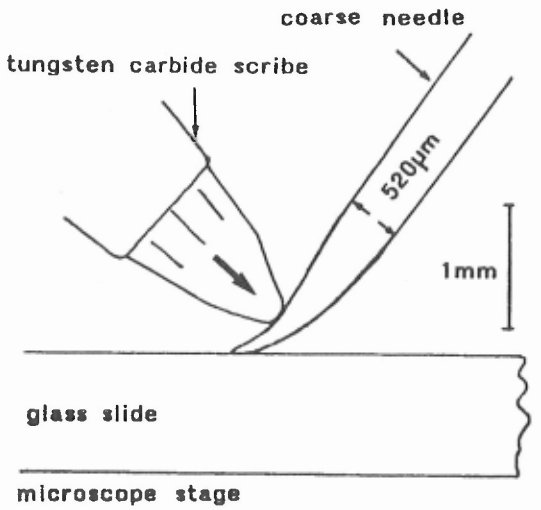

Figure 3. Making a curved needle for manipulating solvents.

large quantities of needles in clear, flat, plastic boxes (see Figure 4). The sharpened tips should be in a straight line allowing one to see a dozen or so tips at a time, thus making the proper selection of a needle easier.

Extra curved coarse and flat tungsten needles as well as polyethylene and eyelash needles are best stored separately in clear, flat plastic boxes. These needles are more durable and do not need to be replaced as often as the medium

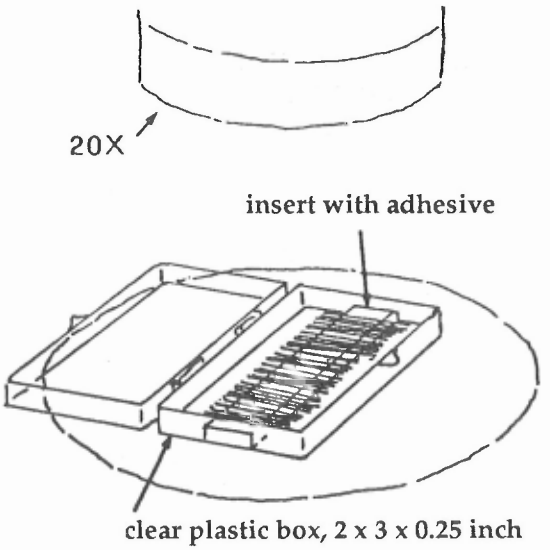

Figure 4. Choosing a proper needle, stereomicroscope, 20X.

or the fine tungsten needles.

POLYETHYLENE NEEDLES

Polyethylene needles can be made from high density polyethylene tubing. A 2-3 inch piece of tubing is rotated and heated over a very small flame and pulled out once it has softened sufficiently. The pulling may have to be done in two stages to get a very fine tipped fiber.

EYELASH NEEDLE

Highly curved eyelash needles are difficult to work with. A relatively straight needle can be made by cutting 3 mm off the tip of an eyelash and attaching it to a medium tungsten needle with a drop of epoxy. The eyelash needle can be cleaned by dipping the tip in ethanol and rotating it to remove any adhering particles.

NEEDLE HOLDERS

The 24 gauge tungsten wire will fit most needle holders; however, the aluminum needle holders shown in Figure 5 are preferred because they are light and long. In fact polyethylene needles do not require a needle holder since part of the tubing from which the needle is pulled can remain with the needle and serve as its holder.

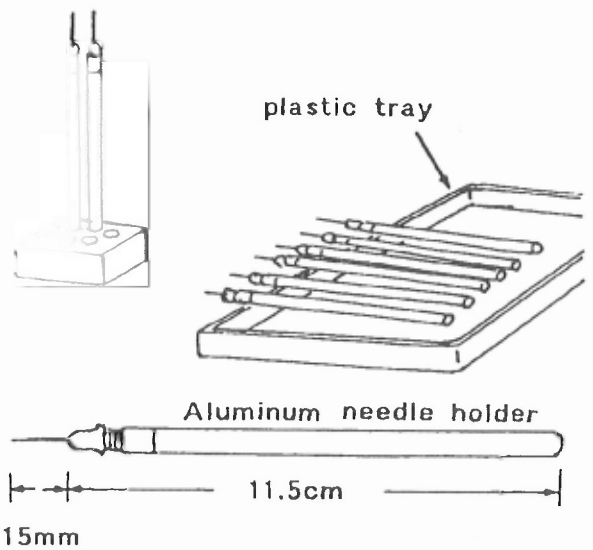

Figure 5. Needle holder, storage stray, and lucite block.

PROPER HANDLING OF NEEDLES

Before a fresh tungsten needle is used, it should be recleaned by passing it through soft, wet paper (see Figure 6). Dry paper may damage a fine tungsten needle. Collodion used in preparing small samples for analysis frequently contaminates the tips of tungsten needles. To remove the collodion, place a drop of amyl acetate on paper (see Figure 6), and pass the tip through the paper a sufficient number of times to remove the collodion.

To manipulate small particles and not damage the needle, the particle, or the substrate, the needle should be held at a <20° angle (see Figure 7). Small particles are best picked off a surface by placing the needle tip beneath or to the side of the particle. To keep the tip steady, the needle holder should be held lightly and as close to the tip as possible without getting ones fingers into the field of view of the stereo microscope. The tungsten needle should protrude no more than 3/4" from the needle holder (see Figure 7).

SOME COMMON USES FOR THE SIX TYPES OF NEEDLES

Very fine tungsten needles are used when it is necessary to manipulate particles that are <20 µm. By using a stereomicroscope at 20X-30X magnification, the fine 2-5 µm tip allows you to

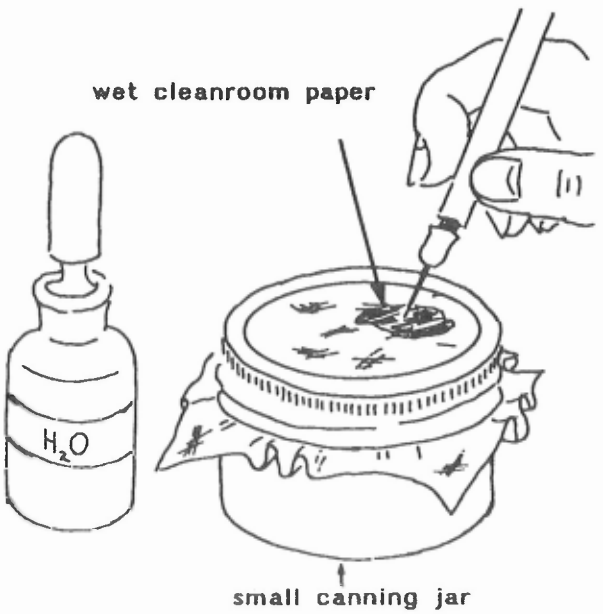

Figure 6. Cleaning tungsten needles.

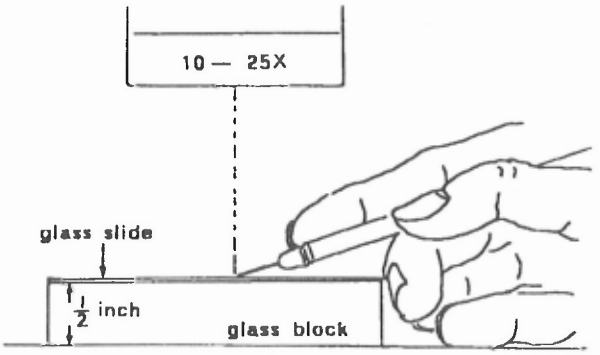

Figure 7. The glass block allows the hand to hold the needle comfortably at less than 20° angle.

see if the particle has been removed from, or successfully deposited on the desired surface. This type of needle is very fragile and, even though it is not used frequently, one should keep a large supply on hand.

Medium tungsten needles are used for handling samples >20 µm. Because of their greater strength, these needles are used most frequently. However, they quickly develop slight imperfections which are hard to observe at the low magnifications of 10-20X used for micro sample handling. Since these damaged needles may not release particles properly, they should be changed frequently even though they may look fine.

A curved coarse tungsten needle is primarily used to manipulate 1 mm diameter drops of solvent on substrates (see Figure 8), or to transfer embedding media for micro replication (see Figure 9). This needle can hold a large volume of solvent beneath it because of its large diameter and curved tip. These needles are used when small drops of solvent are necessary to dissolve mounting media (like collodion) from particles, or to determine the solubility of a micron size particle. They are also used for doing micro extraction on salt plates for IR.

A flat tungsten needle is ideal for removing fine precipitates from smooth, soft polycarbonate filters. Since the needle has no sharp tip and will not scratch a surface, it can be used like a spatula or a knife. It is very sturdy and can be reused many times (see Figure 10).

Polyethylene needles are used in place of

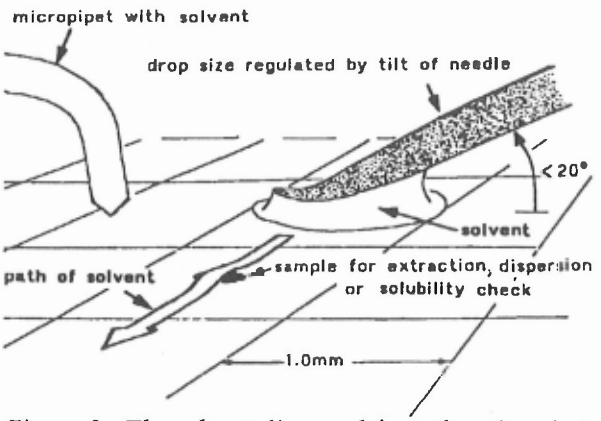

Figure 8. The solvent dispensed from the micropipet is manipulated by a curved tungsten needle on a polished beryllium surface.

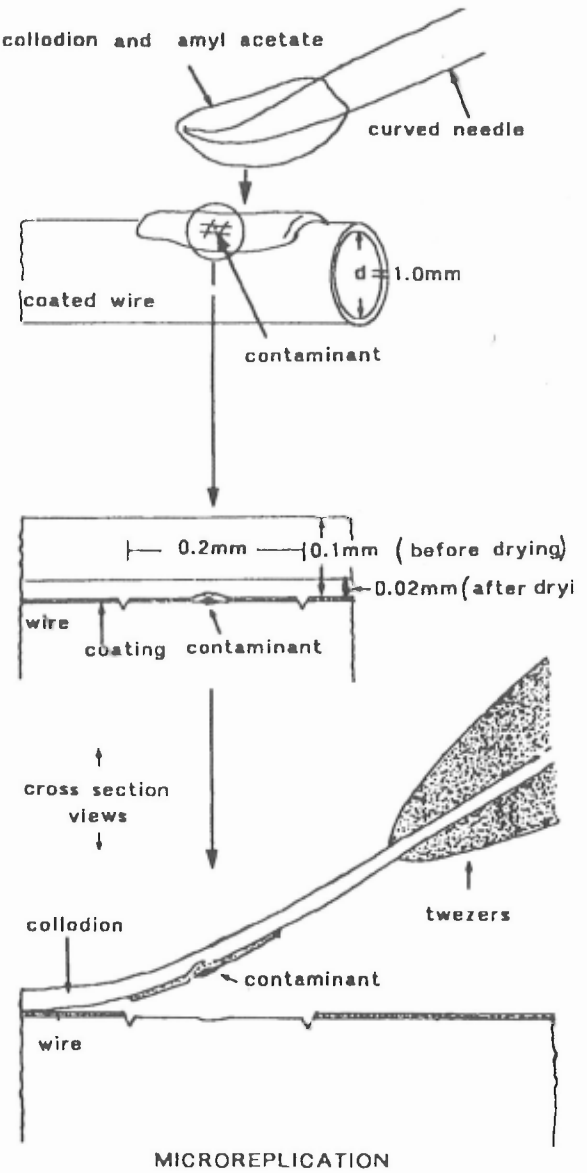

Figure 9. A small contaminant beneath a thin organic coating can be isolated with a drop of collodion on a curved tungsten needle.

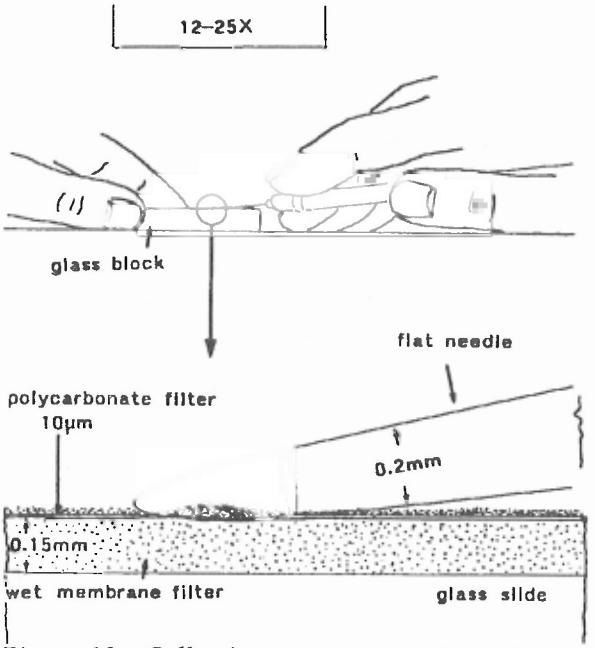

Figure 10. Collecting nanogram residues from a polycarbonate filter with a flat tungsten needle.

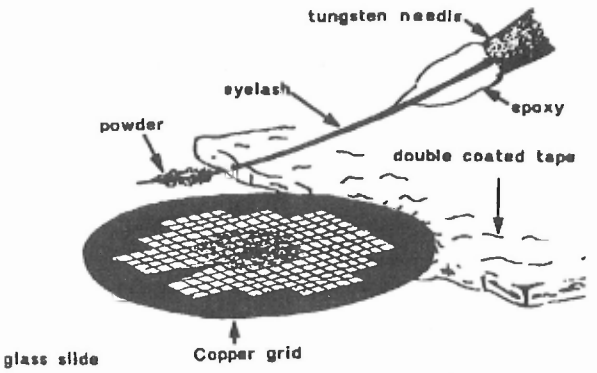

Figure 11. Dispersing a powder on a copper grid with an eyelash needle.

curved tungsten needles to handle small volumes of solvents that could react with the tungsten needle or to test for the presence of tungsten. Polyethylene needles are very inert and nearly indestructible.

The eyelash needle is used primarily to disperse, without using any solvents, fine powders on a carbon-coated copper grid for the Transmission Electron Microscope. It gives great dispersion of powders and requires little skill. The eyelash needle, unlike the tungsten needle, is not strong enough to break the thin carbon film and can be reused many times (see Figure 11).

Using the Photomicrography Record Form

by Joseph G. Barabe*

SYSTEM CALIBRATION WITHOUT A LIGHT METER

1. Place a typical specimen in the microscope, focus sharply, optimize illumination.
2. Photograph specimen using color transparency film, recording all conditions in your photomicrography record form and varying only shutter speed.
3. Have film processed at a good quality commercial photo lab.
4. Evaluate results. Note best exposure using that microscope, illumination system, that ocular and objective.
5. Record the best result in a separate photomicrography record form that you label Calibrations.
6. Run a series of tests for other oculars, objectives, illumination systems, filters, etc.
7. Record best exposures in Calibration Photomicrography Record form. These exposures should provide usable photomicrographs.

SYSTEM CALIBRATION WITH A LIGHT METER DETERMINING THE K FACTOR

1. Ideally, the meter should have a microscope tube attachment (can be improvised).
2. Set film speed (ASA or ISO) on meter.
3. Place a typical specimen in the microscope, focus sharply, optimize illumination.
4. Record all conditions in photomicrography record form.
5. Meter the subject. Record meter reading.
6. Photograph specimen using color transparency film, varying shutter speed in full stop increments, i.e., 1/250, 1/125, 1/60, etc.
7. Have film processed at a good quality commercial photo lab.
8. Evaluate results. Note best exposure.
9. The aperture setting (f/stop) opposite the best exposure is the K factor. The shutter speed coinciding with this aperture should give you a well exposed photograph.

Method Symbols for the Photomicrography Record form on p. 18.
A Photomacrographic Record form appears on p. 22.
Details on using this form will appear in a future issue of μ • Notes 2000.

BF = brightfield	PC = phase contrast	⊗ = crossed polars	λ = full wave plate
DF = darkfield	N = Nomarski	∅ = half crossed polars	λ/4 = 1/4 wave plate
T = transmitted	H = Hoffmann	O = uncrossed polars	
R = reflected	D = DIC		
O = oblique			

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Digital Imaging in the Materials Engineering Laboratory

by Theodore M. Clarke*

The recent completion of a company wide PC network system, supported by network servers intended to rapidly handle large data files from CAD systems, made implementation of digital imaging in the Corporate Materials Engineering laboratory of Case Corporation a wise investment. Laboratory reports in Microsoft Word with linked image files can be rapidly received through any of the networked PCís and are archived in the server system. The changeover from film imaging to digital imaging was very sudden. This change was accelerated by satisfied users, whose work was made easier and more productive, and satisfied customers within Case Corporation.

The change to digital imaging occurred with the nearly simultaneous startup of three digital imaging systems in the laboratory. These systems include a JEOL 5800 LV digital SEM with a Noran Voyager EDX system, a video camera connected to a frame grabber for color imaging, and an Eastman Kodak 1.6i AB digital camera for high resolution grayscale imaging.

The major investment was in the JEOL 5800 with the Noran Voyager EDX system. Digital images from the SEM are 960 x 1280 pixels and can be transferred easily to the Voyager system. The Sun work station on the Voyager is connected to the Token ring network to which the other systems are also connected. The image analysis software on the work station is also used to analyze images from the network connected video camera system and the Kodak system; therefore avoiding additional cost of a dedicated image analysis system for the light microscope. The Kodak Megaplus camera purchase was influenced by theoretical considerations indicating that it should be capable of nearly matching the resolution and field size of black and white 4 x 5 Polaroid prints, our standard for imaging in the materials laboratory (1).

About half of the images used in Case material evaluation reports are photomacrographs, showing details of machine components. Photomicrographs of microstructures from light microscopes are more frequently used than SEM images in our laboratory. The SEM images are usually fractographs. Nearly all of the laboratory imaging is grayscale. Sometimes color is important, typically when corrosion products with characteristic colors require documentation.

A Javelin video camera was salvaged from an obsolete Photophone system and connected to a ComputerEyes/1024 frame grabber installed in a network connected PC for images requiring color. The images are captured in 256 colors, because 600 x 800 resolution in 256 colors is the limit of the PC graphics cards and monitors on the company network. Hard copy is made on network connected 300 dpi color laser printers. VGA output from the camera is used for best image quality rather than composite video output. The ComputerEyes software makes it easy to adjust the hue of the captured image to match the object. The images are normally computer enhanced, which takes only seconds with the ComputerEyes software. The color matching and image resolution appear to be superior to the Polaroid MicroCam, so it is kept as a backup for the digital system. Polaroid 4 x 5 color film is available in the laboratory for those wanting higher color image. This digital system can also capture grayscale images.

The system using the Kodak MegaPlus camera, model 1.6i, is now the predominant imaging system used in the laboratory for grayscale imaging. The camera has an antiblooming sensor, which is an extra cost feature but absolutely necessary for imaging part surfaces with unavoidable specular reflections of the light sources. Image resolution is also slightly improved with the antiblooming sensor. The camera has a 9 x 13 mm CCD sensor with 1024 x 1534 pixel elements and the digital output of the camera is 10 bit

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grayscale. The Kodak camera is connected to a Digital Equipment Corporation 90 megahertz Pentium PC with 40 megabytes of RAM, a Coreco Oculus- f/64 image acquisition system and graphics card. The system has an NEC XP21 1200 x 1600 pixel monitor. The 1.2 gigabyte hard drive has Image Central software from Advanced Imaging Concepts so that an image database system can be built with reference images and associated data.

The cost of this system is less than that of a modern research metallograph, which would have no better resolution than the 1970's vintage Zeiss Universal Microscope on which the camera is mounted. This microscope has metallurgical objectives ranging from 2.5X (0.08 N.A.) to 100X (1.25 N.A.). A Diagnostics Instruments adapter, designed to be compatible with the flat field compensating eyepiece Zeiss system, images the same field size on the Kodak CCD sensor as would normally be recorded on 4 x 5 film. The Kodak camera has a Nikon "F" mount so that it can be used with 35 mm camera lenses on a Kaiser copy stand with the PC and monitor between the microscope and copy stand. Camera transfer from microscope use to copy stand use takes less than a minute. The Kodak camera has an electromagnetic actuated shutter which is controlled through a menu on the 1200 x 1600 pixel 21 inch monitor. Camera display is set at 1024 x 1280 pixels to avoid the use of dual monitors, a limitation which future systems should not have.

Maximum resolution is desired and exposure times are less than 1 second, so the gain and black level default values are set at zero. This is important for users who might forget to check these settings and obtain inferior image quality. Focusing of images from the microscope and setting the shutter speed are very easily accomplished while viewing the monitor. Monitor brightness is preset based upon getting a match in grayscale detail between the monitor and laser prints. The image acquisition software is the demonstration program provided with the f/64 system. The next revision of this software will have file overwrite protection along with pixel gray level histogram plotting. Computer enhancement (high pass filter) is normally used to sharpen all of the images, and is very quick with the f/64 system. All of the image files to be used in reports are coded with a job number obtained from Borland Paradox database system used for tracking all work in the laboratory. Image file code includes the consecutive image number and letters for the standardized image recording conditions. The images are filed on a network server and a laser print is immediately made on a 600 dpi network printer set so the 1280 pixel width is eight inches wide on the hardcopy. Laser print resolution is almost as good as the monitor image, which is normally scrolled in a 480 x 640 pixel window on monitors set for 600 x 800 resolution. The contrast range of the laser prints is much lower than the 256 gray levels of the monitor. This is a more important deficiency than the loss of resolution in the laser prints. Adobe Photoshop can be used to increase the contrast for printing, but this may cause an objectionable loss of highlight and shadow detail. Notation is added below the initial prints for future reference. Scale bars can be added by the materials engineer when he views and marks up an image for a final report, using Paintbrush or Adobe Photoshop. Images are stored as 1.31 megabyte "bmp" files rather than "tif" files, because all of the networked PC's have Windows Paintbrush for viewing the images. "Tif" file sizes and image quality are the same as the "bmp" files.

As previously noted, about half of all images used in the material evaluation report are photomacrographs. It is in this application that the Kodak Megaplus camera system has shown the greatest advantage over film imaging in our laboratory. The photomacrographs are typically of fracture surfaces and relevant features of a machine component. These are not flat surfaces and are often, in the case of gear tooth flanks or bearing components, spectrally reflecting. Establishing lighting conditions and the right camera exposure to show the desired details is much more difficult than photomicroscopy. Obtaining adequate depth of field is a further complicating factor. The digital system allows rapid establishment of illumination, focus, and depth of field while viewing the monitor. No time and film is wasted on test exposures. Time savings average at least 75%. The lenses used for photomacrography on the camera stand are a 28 mm f/2.8 Vivitar, 60 mm f/2 Nikon Micro Nikkor, a 100 mm f/6.3 Zeiss Luminar, and a 63 mm f/4.5 Zeiss Luminar. These lenses were designed to cover a 35 mm film format of 24 x 36 mm, but the Kodak cameraís CCD sensor records only a 9 x 13 mm portion of the image formed by

the lens. The digital images are equivalent to 4 x 5 Polaroid prints of the same fields with a maximum print resolution of about 5.6 lines/mm, requiring a lens resolution of about 60 lines/mm at the CCD sensor. The aperture setting to just achieve this resolution can be calculated from this equation:

$$f/n = \frac{24}{Mcamera + 1}$$

This calculation assumes the digital image is equivalent to a 10X enlargement of a portion of the image formed by the lens on the sensor to a 3.5 x 4.5 print size. A digital image resulting from the 60 mm Nikon lens focused at 1X camera magnification, with a lens aperture setting of f/11, is equivalent to a good quality 10X photomacrograph recorded on 4 x 5 Polaroid film. Zeiss Luminar lenses are used for higher magnifications. The 100 mm Luminar lens, optimized for 2X camera magnification, is used to cover field sizes equivalent to 4 x 5 Polaroids with magnifications between 10X and 25X. A 63 mm Luminar covers a field size range equivalent to 25X through 50X Polaroids. An extension tube was made in my home machine shop to connect the Zeiss lenses to the bayonet mount of an Olympus Auto Bellows. The Olympus Auto Bellows camera connection ring was modified to Nikon "F" mount to connect the Kodak MegaPlus camera to the bellows. This system provides an advantage of long working distance, 23 "for 50X and 4" for 25X. Since camera height adjustment on the copy stand is not sensitive enough for easy focusing at the higher magnifications, samples are placed on a fine focusing stage if their size and weight permit. A future improvement will be to modify the vertical illuminator for the 100 mm Luminar on the Ultraphot II so it can be used with the 60 mm Nikon lens and the 100 mm Luminar on the digital imaging system. Initial testing indicates that this illuminator will then cover a much larger field size than when used on the Ultraphot II. This will allow the digital camera to replace Polaroid film currently used for this application and the time spent digitizing these film images.

It is hoped this review of the introduction of high resolution digital imaging in the Case Corporation Materials Engineering Laboratory is helpful for other laboratories considering the changeover to digital imaging. However, the current cost of this technology makes 4 x 5 Polaroid and 35 mm film imaging the most cost effective means of imaging for smaller laboratories and the film images are superior to those from all but the most expensive digital cameras. This situation will rapidly change as high resolution CCD camera system costs drop. CCD arrays of 2000 x 3000 and then 4000 x 6000 will become affordable and eliminate the current advantage of film resolution. General user image acquisition systems should become available soon so that scale bars can be added at the time the image is acquired, perhaps automatically with computer links to the light microscope and macro imaging system. High resolution zoom lenses with brightfield illumination attachments will replace the bellows lenses. Aperture settings for the microscope illumination and the macro zoom lenses could be automatically set to values that provide an optimum combination of resolution and depth of field. Concurrent with these improvements will be improved ability to transmit, store, view, and print the digital images at full resolution in the sizes used for high quality photographs.

We will be happy to answer any questions raised by this article. An enhanced version of this article, with digital images and photographs of the system, will be submitted for publication in *The Microscope*.

ACKNOWLEDGEMENT

The successful transition to digital imaging in the Corporate Materials Engineering laboratory of Case Corporation was a team effort. Help from materials engineers Dave Dobbins and Ed Rahe with the computer system and the method for transmitting the reports, where image files are linked to a server rather than saved in the document, is gratefully acknowledged.

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PhotoMicrography Record

Client _____ Job # _____ Date _____ File # _____

Microscope _____ Camera _____ Format: 35 4x5 Pol _____

Film _____ ISO _____ Process _____ Remarks _____

#	Subject	Oc.	Obj.	Meter	Method	Filter	K	Exp.	Eval.
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An Introduction to Labradorescence

by Garth Ziemba*

The author has an interest in gem mineral labradorite and its unique play of colors, both from a mineralogical viewpoint and because it provides an excuse to be interested in Labrador--still one of the world's last areas to be explored in detail. Jacques Cartier in 1534 showed little interest in a land "...of stones and rocks, frightful and ill shaped...it is the land God gave to Cain." Captain James Cook found it so uninviting in the 1760's that even he avoided going ashore. The rockiness has been explained: "God made Labrador in six days and on the seventh He threw stones at it" (1). In reality, it is a place of desolate beauty and freedom that rewards those prepared for its idiosyncrasies and, of course, it has labradorite.

Labradorite of superior color may be found in Finland (2) whereas that found in Madagascar is of a lesser quality (3). Labrador is, however, the romantic source and the material from there was long used for ornamentaiton by the Inuit (Eskimos). In 1770, Mr. Wolfe, a Moravian missionary, brought this mineral to the attention of Europeans.

Today, only granular dimension stone containing small labradorite crystals is quarried in Labrador. Attempts to quarry Tabor Islands' richest deposit of gemstone labradorite by using explosives produced inferior quality material since two pronounced cleavages exist in labradorite. Mined material, therefore, developed a bad reputation for cracking during subsequent processing by lapidarists (3). An alternative mainland site to the Tabor Island source, is the Pearly Gates. This location is very difficult to access and produces somewhat inferior material which has criss-crossing non-labradorescent white lines. Tabor Island still has some debris remaining from earlier mining operatons and the local Inuit sometimes glean limited material. Tabor island and its minerals are strictly controlled by the Labrador Inuit Association.

ORIGIN OF LABRADORITE

Labradorite is a member of the plagioclase feldspar series–a group of six rock-forming silicates.

The feldspars are the most important group of rock-forming silicates, comprising about 60% of the outer 15 km of the earth's crust. Feldspars are best described as three-dimensional infinite frameworks comprised of similarly sized AlO_4^{-5} and SiO_4^{-4} tetrahedra linked together (see Figure 1). The larger oxygen atoms "surround" the smaller silicon and aluminum atoms. By sharing oxygen ions, these tetrahedra form an eight-membered ring (see Figure 2).

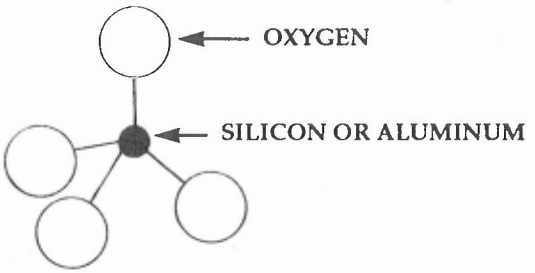


Figure 1

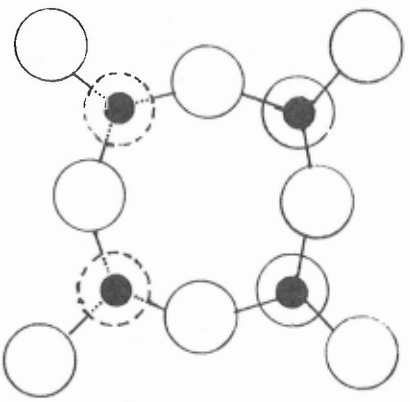


Figure 2

* Prairie Sun Consultants, 612 Staunton, Naperville, IL 60565-2607

Because Al has a positive three charge, its AlO_4 tetrahedron has an overall charge of -5 rather than the -4 of the SiO_4 tetrahedron. This excess negative charge is balanced by the entry into open areas of the lattice by one monovalent cation per AlO_4^{-5} tetrahedron or one divalent cation per two AlO_4^{-5} tetrahedra.

Typical cations are K^+, Na^+ and Ca^{+2} and give rise to compositions:

$KAlSi_3O_8$	Orthoclase or **Or**
$NaAlSi_3O_8$	Albite or **Ab**
$CaAl_2Si_2O_8$	Anorthite or **An**

Feldspars are classified into two groups:

A) The *Alkali feldspars* are composed of the potassic and sodic feldspars with their associated crystallographic variations and intergrowths.

B) The *Plagioclase series* is composed of albite/anorthite mixtures which are arbitrarily defined by the percentages of their end-members **Ab/An**.

	Ab	**An**
Albite	100 - 90	0 - 10
Oligoclase	90 - 70	10 - 30
Andesine	70 - 50	30 - 50
Labradorite	50 - 30	50 - 70
Bytownite	30 - 10	70 - 90
Anorthite	10 - 0	90 - 100

The plagioclase composition range responsible for labradorescence usually occurs in basic igneous rocks such as gabros and anorthosites (2, 5-7). Only in anorthosites do the mineral grains reach a large size; and it is only in rare circumstances that conditions favor the formation of labradorescent material.

The labradorescent play of colors was originally believed to be a "schiller" due to internal magnetite or ilmenite inclusions (6, 8). Schiller is a silver shimmer of light caused by platy inclusions or internal lamellae due to twinning or exsolution. However, electron microscopy revealed a sub-micron internal intergrowth structure which is inconsistent with schiller. Instead, labradorescence results from constructive interference of light reflected at interfaces between alternating lamellae which were initially believed to be the result of twinning. Subsequently, it was found that the structures are actually areas of different chemical compositions in the following ranges:

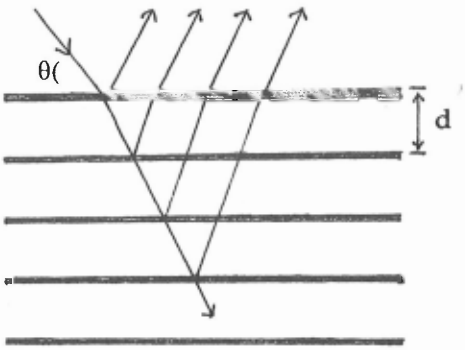
(A) [An 44 ± 4 Ab 56 ± 4] and

(B) [An 58 ± 6 Ab 42 ± 6] —

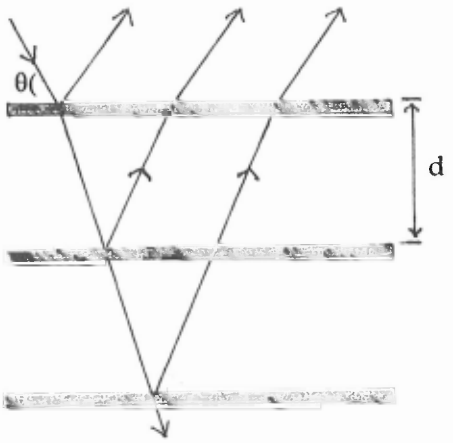
compositions corresponding to the arbitrary andesine and labradorite of the plagioclase series. These intergrowths have been named "Bøggild intergrowths" and are stacked like a pile of wrinkled leaves with spacings of hundreds of nanometers (9).

The different lamellae compositions have slightly different refractive indices; and when combined with the lamellae dimensions, the overall structure is simillar to a reflection diffraction grating. It is found that the color of the labradorescence varies with the lamella spacing. The wavelength of the color can be described by a modified Bragg equation: $\lambda = 2\,\eta\,d \sin\theta$ where η is the average refractive index of the bulk feldspar. A relationship between lamellae spacing, angle of incidence and blaze wavelength is illustrated below:

SHORTER WAVELENGTH BLAZE



LONGER WAVELENGTH BLAZE



The dimensions for d are somewhat similar to the wavelengths of visible light.

Labradorescence is considered to be most intense, or perhaps only present, on the (010) side pinacoid crystal face. It is possible that the (001) face is also involved. The author has seen two planes of unequal intensity in a single labradorite crystal.

Labradorescence is a complex subject and this paper represents only an introduction to the subject. Future papers will address the subject in greater depth.

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PhotoMacrography Record

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Format: 35 4x5 Pol _____ Film _____ ISO _____ Light _____

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Update on the Turin Shroud

by Walter C. McCrone*

It is now more than 20 years since I first became aware of the Shroud and began preparing a proposal to the Turin Center for Sindonology for a study of the Shroud image. My proposal in 1974 was sent to Father Peter Rinaldi, S.D.B., a champion of the idea that scientists should study the Shroud to learn more about the image that he and most of the world felt strongly had been formed through the use of the Shroud to cover Christ's body after the crucifixion. He and I expected that my proposed sticky tape samples from image areas would show body fluids including blood.

Thirty-two sticky tape samples were taken in October 1978 from body- and blood-image areas, as well as scorched areas, and control areas of the Shroud. I started work on them on December 25, 1978. Imagine my surprise to immediately find red ochre pigments. Over the next year (1979), I became convinced the Shroud was a painting and during 1980 I identified a paint medium, collagen tempera, and McCrone Associates electron optics group (Mark Andersen) reported vermilion in blood-image areas.

By the end of 1980, I was ready to publish the data that proved the Shroud to be an impressive medieval painting. Unfortunately, I had joined a group called STURP (Shroud of Turin Research Project) who had also submitted a proposal to Turin. It seemed reasonable at the time that I should join with them since we had the same goal. However, my first oral report to the group in March 1979 was greeted with disbelief and belligerence. We had all agreed that any publications by any of us would have to be approved by a STURP Publications Committee. My three papers in 1980 were reviewed with the following decision of the reviewers in a letter signed by Eric Jumper, dated April 10, 1980: "In short, your data is [sic] misrepresented, your observations are highly questionable, and your conclusions are pontifications rather than scientific logic; I cannot permit this paper to carry the Shroud of Turin Research Project's seal of approval." I soon learned the STURP members were convinced the Shroud is authentic. I got nowhere in my arguments to the contrary and soon resigned (June 1980). I then published my papers in *The Microscope* (Vol. 28, pp. 105, 115 and Vol. 29, p. 19).

STURP members published 35 papers all "proving" the Shroud is authentic; outnumbered 35:1 I didn't have a chance. The long-awaited carbon-dating didn't help a bit even though their 1325 date agreed well with my 1355 date. STURP simply said the resurrection had modified the cloth and its carbon-date. Soon after I had resigned from STURP, I gave up trying to argue with them. My conversations with them left me literally sick to my stomach. Instead, I turned my attention and my hopes to Father Rinaldi. At least, he was a gentleman. We exchanged frequent letters for several years. I, patiently and repeatedly, tried to explain what I had done, what I saw microscopically, and what it meant. He, patiently and repeatedly, kept giving reasons why he felt the Shroud was authentic.

This exchange of letters continued until again I gave up. It seemed too hopeless. At least a dozen books were published, none of them agreeing with me. Time passed and there seemed no hope I would ever be able to convince any one other than scientists who understood what I had done—or who were convinced by the carbon-dating.

I did renew my correspondence with Father Rinaldi after the carbon-dating but with little apparent change in his position. After a last flurry of letters between us, I decided my only hope was to write a book putting everything on the written record and depending on eventual vindication. I will have the book printed on archival paper so that it may last till vindication. After

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all, it took Galileo 400 years for his vindication.

During the writing of "Judgement Day for the Turin Shroud," I found myself rereading and rethinking my relationship with Father Rinaldi. It gradually became clear to me that he was being used by the Church to educate me and show me the error of my ways. He had performed the same service back in the 1970's when two members of an Italian Commission studied the Shroud and concluded it was a painting. Father Rinaldi told me how they had seen the error of their ways and recanted. He expected me to do the same.

Obviously, there was no way I would ever recant. I had much firmer evidence than the earlier 1973 Commission. Still, I must say that Father Rinaldi never gave up. At every opportunity for years he reiterated the same arguments intended to change my mind. Eventually, I did see some signs that everyone even in the Church did not agree regarding the Shroud. Father Rinaldi told me of several such occasions. For example, after the carbon-dating, Cardinal Archbishop Ballestrero in Turin agreed with the medieval origin but shortly I heard that he had resigned. Father Rinaldi then told me in a letter dated May 25, 1989 about a proposal to the new Archbishop Saldarini:

> What the outcome will be is yet to be seen. Two things are certain: 1) The "friends" of the Shroud will simply not let go of it. The Pope is on their side, and the Archbishop is under pressure to take some sort of action. 2) He will very likely take his time, after the debacle that exploded in the hands of his predecessor, Cardinal Ballestrero (now retired due to age) who was bitterly accused of accepting too quickly and simplistically the results of the carbon-14 test. Archbishop Saldarini will tend to be very cautious.

In July 1991, Father Rinaldi wrote to David Sox, a close Shroud friend of mine in England. A portion of that letter reads:

> Aside from such developments, it may surprise you to know that, since October 13, 1988, I have a completely new personal relationship with the Shroud. Almost unconsciously, I began to feel that I had given the Shroud far too much importance in my spiritual life. Suddenly I understood that what really mattered is the Lord of the Scriptures and of the Church and not the Lord of the Shroud. And, too, I saw clearly that falling back on science to bolster one's personal faith in and love for the Lord is only a mirage, a delusion.
>
> May I also add that the misunderstandings and the bitterness among Shroud groups have further convinced me that, when we deal with the Lord, "set your affection on things above, not on things on the earth" (Col. 3:2). Too many of us, in dealing with the Shroud, were "intent on things on the earth".
>
> Forgive me if this sounds like a homily, but it is the way I honestly feel at this point in my life (I am now eighty-one!) when the Master's final call cannot be far off.

And in a letter to me after the carbon-dating:

> Would you believe it, Walter? Maybe it is because I was subconsciously prepared for it, but the blow that came from the results of the carbon-14 test did not actually send me reeling...I admit to having penned the message (enclosed) to my Shroud friends a bit too hurriedly. It does sound less than honest on some points. I was overly concerned in trying to soften the blow on those good people. I know you will understand.

He refers here to a letter to his friends (10-5-88) in which he indicated in the margin the "less-than-honest" points. The italics are mine.

> Let me say, first of all, that not all the experts accept the results of the test. Some of them are actually calling for a new test on good scientific ground. I was intrigued by what one of them told me: *"Valid or not, the results of the carbon-14 test in no way solve the mystery of Christ's image on that cloth".*
>
> .....
>
> You can't be serious," I told him. *"Do you really think the Church will fall apart because the Shroud may not be what many of us supposed it to be?"* The Church has nothing to fear from the truth, provided, of course, it is backed by solid facts. For one thing, in the case of the Shroud, it was the Church that took the initiative to find out the truth.
>
> .....
>
> *Doubtless, that incomparable portrait, that no artist could have produced, will outlive all the scientists' tests, and will continue to touch the minds and hearts of countless people for ages to come.*

Near the end of the "Dear Friends" letter, he gives us the real reason for defending the Shroud.

> One thing does trouble me: the thought that the simple faith of many good people may be somewhat shaken by this turn of events. This could be due to an exaggerated notion they have of the importance of the Shroud in the scheme of our Christian faith. When lecturing on the Shroud, I often reminded my listeners that for us Christians, it is the Lord that matters, not the Shroud. If the Shroud does have a meaning, it is because it speaks to us of His sufferings as no other image does. But, at best, the Shroud is only a sign of our faith and hope in Christ. He and He alone is our greatest and dearest possession, the supreme gift of the love of the Father to us.

I do not think I am wrong here when I read between the lines to say Father Rinaldi is now accepting the Shroud's fall from grace. If I now step out from among the trees to look at the forest itself, the view is: Father Rinaldi felt constrained to defend the Shroud even as my efforts continued to reason and explain why the Shroud could not be Christ's Shroud. Even though Father Rinaldi's belief in the Shroud wavered and finally ended, he had maintained his defensive posture because he empathized with the many good people to whom the Shroud means so much and because many, if not most, of his Church colleagues still believe therein. Still in his 81st year "when the Master's call cannot be far off," he apologized for a lack of honesty in not acknowledging his changing awareness of the Shroud as only a poignant artistic rendering of the passion of Christ. This, then, is my final reward for patience and understanding as I continued for years to answer Father Rinaldi's verbal opposition.

Judgement Day for the Turin Shroud

The Shroud, a 14' long linen cloth, has faint sepia back- and front-view images of a man lying head to head and showing the marks of a beaten and crucified man. The marks of nail wounds in hands and feet, a spear wound in the chest, blood from the crown of thorns and from beatings with a whip all fit the description of Christ's crucifixion in the New Testament. Many millions of the earth's population of many religions believe to the point of absolute certainty that this cloth is the first century Shroud of Christ.

The Church encouraged this belief and convinced themselves that this revered image was indeed Christ's Shroud. In 1969 and 1973, they appointed a Commission of Italian Professors to study the Shroud. Two important members of this group decided the image was painted by an artist. The Church quickly convinced them "of the errors of their ways," and everyone continued in their belief that the Shroud was a result of Christ's crucifixion. The Church had enough confidence to allow a group of Shroud devotees identified as STURP (the Shroud of Turin Research Project) mostly from the United States to make a one-week "scientific" study of the Shroud in 1978.

Fortunately, one small part of this study included 32 2"-long sticky tapes from image and non-image areas of this Shroud that Walter McCrone studied microscopically to determine the composition and source of the image. "Fortunately," because most of this group proceeded only to try to prove the Shroud to be authentic—and, to their satisfaction, as well as the satisfaction of the Church and of millions of the world's population they succeeded. Evidence to the contrary, even 14th century carbon-dating results, were ignored or reviled and much evidence proving authenticity was invented (out of whole cloth). McCrone reported in three 1980-81 papers that the Shroud image was totally an artist's paint.

Thus began a two-front war. Opening salvo's from STURP:

"In short, your data is [sic] misrepresented, your observations are highly questionable, and your conclusions are pontifications rather than scientific logic...."

From the Church:

"...you are the one person to challenge the enduring mystery of the Shroud. Judging from first reactions at the Turin Center they seem to be eager to accept the challenge from you.

"....You might know that you have now become 'the adversary,'...."

From McCrone: The Shroud image is entirely red ochre and vermilion in a collagen tempera/paint medium. There is no blood on the "Shroud."

Scientists, in general, agree with McCrone: "You were the first to conclude the Turin Shroud is a fake. The carbon-datings of 1988 show how right you were..." [Dr. Ernst Martin, Crime Lab Director]; "All of us here agree with your 14th century date..." [Marigene Butler, Conservator, Philadelphia Museum of Art]; "Your evidence is conclusive..." [Prof. Mary Virginia Orna, College of New Rochelle]; "The objections to accepting the results of [McCrone's] scientific studies are just ludicrous..." [Linus Pauling].

Now, you can read the history of this war so far—it's far from over. "Judgement Day for the Turin Shroud," by Walter McCrone is a 340-page hardbound book with 68 figures, 50 of which are in color in 11 full-color plates. The author covers his light microscopy, McCrone Associates Hi-Tech confirmation, as well as STURP's misguided efforts and the Church's effort to "re-educate" him.

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A Computer Program for the Double Variation Method

by Thomas Polaski* and Jeffrey Hollifield†

The double variation method is a technique introduced by R.C. Emmons in 1929 as an aid to mineral identification (1). This method utilizes the fact that refractive indices are dependent on both wavelength and temperature. Some specimens have almost identical refractive indices when observed at room temperature and with white light, but the property of dispersion is exploited by the analyst with Emmons' method because the refractive indices may vary greatly when observed at a different temperature or with light sources of varying wavelength.

Although originally applied to the analysis of minerals, this technique can be used for many substances and is particularly useful for differentiating samples with very similar refractive indices, such as glass. Glass fragments are encountered frequently in the field of forensics, and many samples do not tend to vary widely with respect to refractive index. This decreases the confidence level in associating fragments with a common source; therefore, other methods for differentiating samples must be used. The double variation method is relatively simple, inexpensive and is a good method for measuring the dispersion of glass. Dispersion staining, density determination, and trace elemental composition are other valuable techniques commonly used in glass analysis (2-6).

At a given temperature, a specimen and the mounting medium in which it is immersed have a matching wavelength, a wavelength at which the specimen and mounting medium have the same refractive index. Emmons' method instructs that the sample be held at a constant temperature while the wavelength of the light source is varied continuously by means of a monochromator until the specimen disappears. This procedure is repeated at two additional temperatures. The matching wavelengths and corresponding temperatures can then be plotted on special graph paper, the Hartmann net (7).

The Hartmann net exhibits a plot of refractive index against a logarithmic wavelength scale. Dispersion curves for the chosen immersion liquid are then drawn for multiple temperature values. By using the logarithmic wavelength scale, these dispersion plots appear essentially as straight lines (7). With the dispersion curves for the liquid in place, the analyst is now ready to gather experimental data.

Upon plotting three experimental points for the unknown specimen, each at a different temperature and wavelength, a curve may be drawn through the points indicating the dispersion of the sample in question. In measuring and reporting refractive indices, there are three wavelengths that are generally taken as standard, these being the nD line (589 nm), the nC line (656 nm) and the nF line (486 nm) in the Fraunhofer series. These values can be of importance diagnostically and can be obtained directly from the plotted data.

When a particular immersion liquid is routinely used, as in forensic glass analysis, preparation of the Hartmann net with dispersion curves for the liquid, the plotting of experimental points and the drawing of dispersion curves for test samples can be eliminated through use of a computer program which has been written to perform the same function as the Hartmann net. An immersion liquid commonly used in glass analysis is #710 silicone oil and has thus been chosen as the basis for developing the computer program.

The computer program asks the user to input three pairs of experimental temperature-wavelength values. The wavelength values are converted to linear x values, and then the equations of the temperature lines on the graph for the immersion liquid are used to determine the y values for the experimental points plotted. Linear regression is then used to find the equation of the line that best fits the x and y values for

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the three data points. Finally, the intersection of this line with the nD line and the nF line are determined and the refractive index values corresponding to these points are displayed.

Simply stated, by typing in three experimental values for temperature and three for wavelength, the nD and nF values can immediately be determined by the computer. Temperature must be expressed in degrees celsius, and wavelength must be expressed in nanometers. The complete computer program is shown below.

```
/*  glass.c
*/
#include <stdio.h>
#include <math.h>
void main()
{    int t, w0, i, j, check1, check2;
     float r, v[23], w, x, y, sumx, sumy, sumxy,
     sumx2, b1, b0, d, f;
     char response;

     /* Initialize the scaling factors. */

     v[1] = 0.; v[2] =4.; v[3] = 9.;
     v[4] = 13.; v[5] =18.; v[6] = 23.;
     v[7] = 29.5.; v[8] =34.; v[9] = 40.;
     v[10] = 46.; v11] =52.5.; v[12] = 59.;
     v[13] = 66.; v[14] =74.; v[15] = 82.;
     v[16] = 90.; v[17] =99.; v[18] = 108.;
     v[19] = 118.5.; v[20] =129.; v[21] = 141.;
     v[22] = 153.; v[23] =166.;
     sumx = 0; sumy = 0; sumxy = 0; sumx2 = 0;

for (j=1; j<=3; j++)
{    t = 0; w = 0; w0 = 0; r = 0; i = 0; x = 0; y = 0;
     check1 = 1; check2 = 1;

/* Input the data. */
while (check1= =1)
{    check1 = 0;
     fprintf(stderr,
     "Input temperature %-1d\n", j);
     scanf("%d",&t);

     if (t<25 | |t>95 | | t % 5 !=0)
{    check1 = 1;
     printf("Inappropriate temperature\n");
}
     printf("\n");
}
     while (check2 = =1)
{    check2 = 0;
     fprintf(stderr,
     "Input wavelength %-1d\n", j);
     scanf("%f", &w>;
     if (w<470 | | w>690)
{    check2 = 1;
     printf("Inappropriate wavelength\n");
}
     printf("\n");
}
/*Change to linear x-scale: 0 at frequency 690,
measured in millimeters on graph. */

w0 = floor(w/10) * 10;
r = w - w0;
i = (700 - w0)/10;
x = v[i-1] + (10 - r) * (v[i] - v[i-1])/10;

/* Use the equations of lines on graph to find y-
values. */

if (t==25)
     y = 43. *x/332000. + 1.52625;
if (t==30)
     y = x/7840. + 1.5245;
if (t==35)
     y = 13. *x/100000. + 1.5227;
if (t==40)
     y = 107. *x/840000. + 1.52095;
if (t==45)
     y = 353 .*x/2860000. + 1.51935;
if (t==50)
     y = 197. *x/1550000. + 1.5173;
if (t==55)
     y = 143. *x/1140000. + 1.5157;
if (t==60)
     y = 161. *x/1285000. + 1.5139;
if (t==65)
     y = 9. *x/71750. + 1.512;
if (t==70)
     y = 391. *x/3150000. + 1.5104;
if (t==75)
     y = 19. *x/155000. + 1.5086;
if (t==80)
     y = 19. *x/155000. + 1.5068;
if (t==85)
     y = 113. *x/915000. + 1.505;
if (t==90)
     y = 219. *x/1720000. + 1.5027;
if (t==95)
     y = 31. *x/252500. + 1.50146;

sumx = sumx + x;
sumy = sumy + y;
```

```
sumxy = sumxy + (x * y);
sumx2 = sumx2 + (x * x);
}
/* Use linear regression to find equation of the
best line through the three data points. */

b1 = (3. * sumxy - sumx * sumy) /
     (3. * sumx2 - sumx * sumx);
b0 = (sumy / 3.) - b1 * (sumx / 3.);

/* Find the intersection of this line with the D-
line (x=53.15) and the F-line (x=145.8). */

d = b0 + b1 * 53.15;
f = b0 + b1 * 145.8;

print ("Intersection with D-line at %-5f\n",d);
printf("Intersection with F-line at %-5f\n",f);
}
```

Extrapolation of the experimental dispersion curve beyond a limited interval yields less linearity and thus less accurate results (7). The computer program, therefore, calculates indices for only two of the three standard wavelengths. The temperature values which are input can range from 25° to 95°, and the wavelength values can range between 470 nm and 690 nm. Values outside these ranges will not be accepted by the program and will result in an error message. The major limitation of the program is that no value for nC can be calculated. With the understanding that this computer program certainly has limitations as currently written, it does suggest that there are potential benefits in combining classical microscopical techniques and computer technology.

The program can be run on IBM compatible systems and may be obtained from Jeff Hollifield at McRI by those expressing an interest. A sample problem is shown below in graph form (A) and computer form (B).

A. Hartmann net: (see p. 30)

B. Input:

Temperature 1: 30	Wavelength 1: 620
Temperature 2: 40	Wavelength 2: 555
Temperature 3: 50	Wavelength 3: 490

Results:
nD = 1.529768
nF = 1.535353

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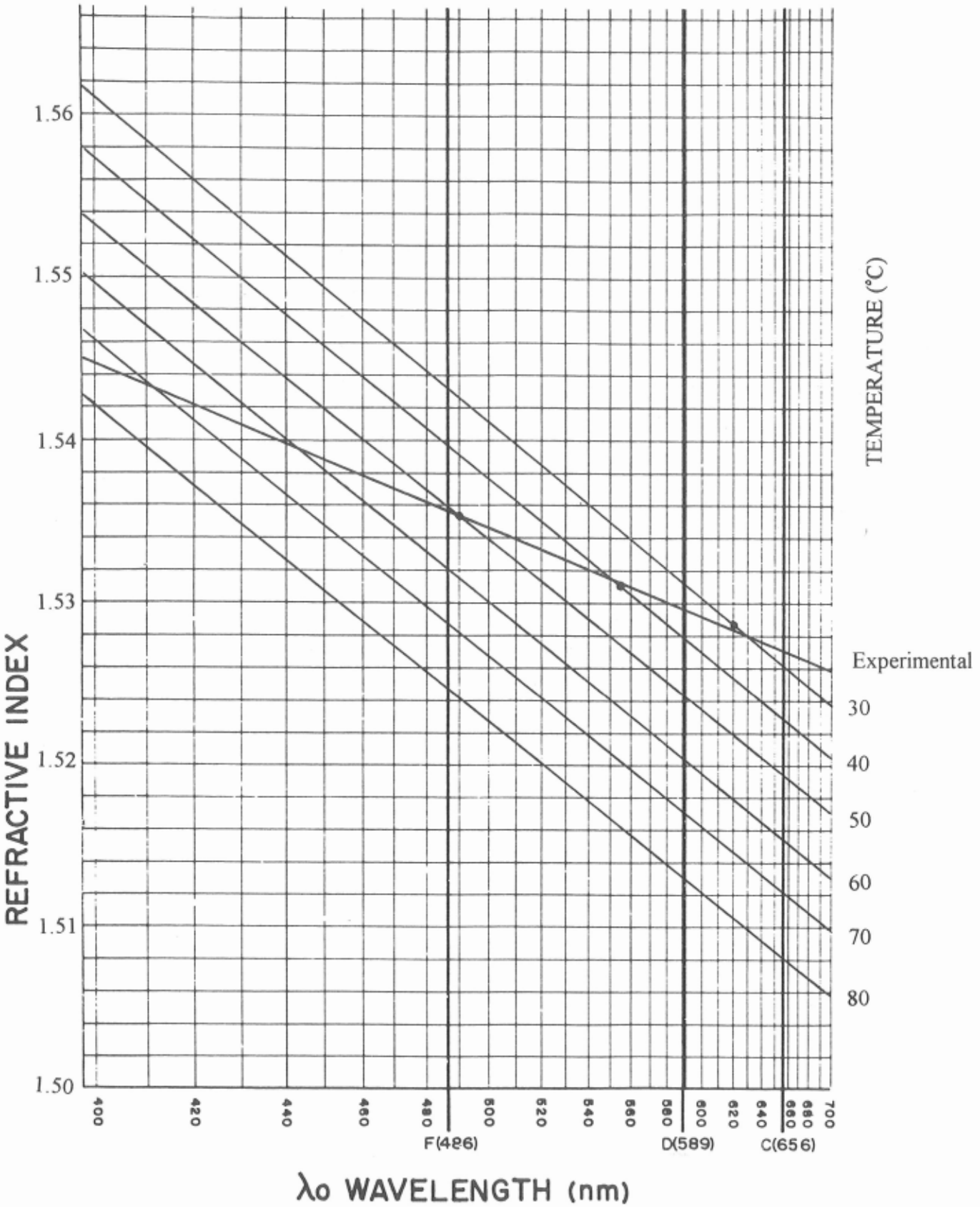
HARTMANN NET FOR #710 SILICONE OIL



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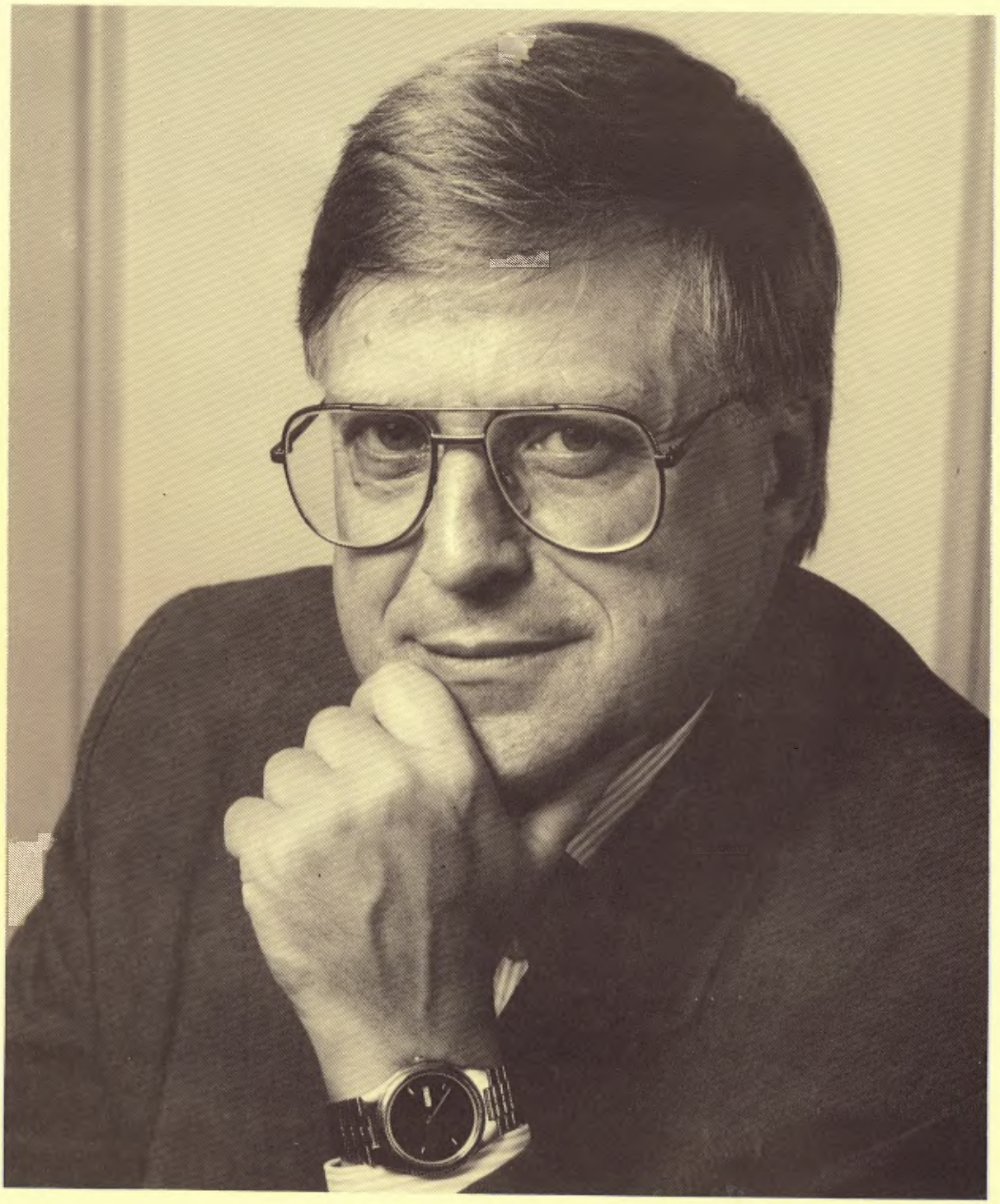

BRIAN J. FORD