

## Inter/Micro 2009

Gary J. Laughlin  
McCrone Research Institute\*

**I**nter/Micro celebrated its 61st anniversary in 2009, reaffirming its reputation as the best and most enduring microscopy conference of its kind. Professional and amateur microscopists, working in all areas of light and electron microscopy, gathered early July at the historic Millennium Knickerbocker Hotel in downtown Chicago for three days of symposia. Presentations focused on microscopical applications of polarized light microscopy (PLM); computer, digital and video imaging; infrared and Raman methods; pharmaceutical sciences; indoor air quality; environmental and occupational health; forensic science; and art conservation and authentication. Attendees also learned about recent advancements in instrumentation, techniques and applications in the field of microscopy.

Inter/Micro 2009 symposia included presentations on such diverse subjects and titles as "Essential Photoshop for the Microscopist," "Optical Characterization of Sodium Lauryl Sulfate," "Microscopy and Lab Accreditation," "Interpretation of Gunshot Residue Patterns Without the Firearm," "The Next Step in Laser Confocal Technology," and "Indian Yellow: Historical Pigment and Modern Confusion." Prof. Brian J. Ford, meanwhile, probed the world of plagiarism in "The Cheat and the Microscope," the topic of his annual "Evening With Brian" talk.

The SMSI 2009 Émile M. Chamot Award went to Dr. Kenneth Libbrecht of the California Institute of Technology, who is known for his extensive research on snow crystals and snowflakes. He presented many of his beautiful snowflake micrographs at the Inter/Micro 2009 Banquet and SMSI Auction. Also at this

event, Mrs. Lucy B. McCrone of the McCrone Research Institute (McRI) received the 2009 Honorary Achievement Award from the Georgia Microscopical Society (see article on page 115). Winners of the Inter/Micro 2009 Photomicrography Competition were Sebastian Sparenga for Best Overall Photomicrograph, Brian Ford for Most Unique Photomicrograph, and Kelly Brinsko for an Honorable Mention.

The second half of Inter/Micro 2009 shifted gears to hands-on training in the laboratories of McCrone Research Institute (McRI), where Dr. John Haines led a two-day workshop on airborne fungal spores. Dr. Haines, scientist emeritus at New York State Museum and Science Services in Albany, NY, taught workshop participants how to explore the particulate content of air by using phase-contrast microscopes to examine air samples and building molds. They also collected and analyzed some of the fungi that are sources of airborne spores.

Inter/Micro was first held in 1948 by Walter C. McCrone and Charles Tufts in Chicago. It is sponsored annually by McRI.

### MONDAY, JULY 6: TECHNIQUES AND INSTRUMENTATION

#### Morning Session

**Chair:** Randy Boltin, MVA Scientific Consultants  
**Sparenga, Sebastian,** McCrone Research Institute  
– *Essential Photoshop for the Microscopist*

**Brown, Rich,** MVA Scientific Consultants – *An "In-expensive" Near-Infrared Microscope*

\* 2820 S. Michigan Avenue, Chicago IL 60616



Mark Palenik of Microtrace, LLC explains the detection of arsenic using gas chromatography with mass spectrometry.

**Millette, James**, MVA Scientific Consultants – *Soot*  
**Hopen, Thomas J. and David Babulski**, ATF Forensic Science Laboratory – *Creating Mineral Art*  
**Vander Tuuk, Christopher**, Olympus Industrial – *The Next Step in Laser Confocal Technology*  
**Havics, Tony**, pH2, LLC – *Unconventional Use of Compensators*  
**Boltin, Randy**, MVA Scientific Consultants – *Teaching Microscopical Analysis of Rocks and Minerals*

#### Afternoon Session

**Chair: Brian J. Ford**, Gonville & Caius College, Cambridge University  
**Clarke, Theodore M.** – *Dispersion Staining Using a 1.2-1.30 NA Cardioid Darkfield Condenser*  
**Palenik, Mark E.**, Microtrace, LLC – *Detection of Arsenic by Gas Chromatography with Mass Spectrometry (GC-MS)*  
**Brown, Rich**, MVA Scientific Consultants – *High-Speed Automated Particle Analysis by SEM-EDS*  
**Bales, Hazel**, Microtrace, LLC – *Mounting Particles on a TEM Grid For Analysis By Multiple Instrumental Methods*  
**Millette, James**, MVA Scientific Consultants – *Anti-Monkey Butt Powder and Other Non-Asbestos-Containing Products*  
**Ford, Brian J.**, Gonville & Caius College, Cambridge University – *A Unique Leeuwenhoek Microscope Sale in London*

#### 2009 Evening with Brian: “The Cheat and the Microscope”

Research findings are always likely to be stolen and re-used by others. In this year’s presentation, **Prof. Brian J. Ford** takes us to the world of misappropriated microscopy. For centuries, microscopists have had their work stolen and published by somebody else. In the modern era of computer cut-and-paste, plagiarism is so widespread that some people claim it is normal behavior. We will learn about some breathtaking examples, and even see how authors can plagiarize themselves!

#### **TUESDAY, JULY 7: ENVIRONMENTAL AND INDUSTRIAL MICROSCOPY**

##### Morning Session

**Chair: Kevin A. Brady**, Tredegar Film Products  
**Boltin, Randy**, MVA Scientific Consultants – *Microscopy and Laboratory Accreditation*  
**Sturm, Ron**, CTL Group – *Microscopical Characterization of Historic Cementing Material. Part 1: History*  
**Caffero, Ann**, CTL Group – *Microscopical Characterization of Historic Cementing Material. Part 2: Case Studies*  
**Lucas, Gabriel**, Buehler LTD – *Advances in Microstructural Analysis in the Use of Diamond Grinding Discs for Preparation of Thermally Sprayed Coatings*  
**Lucas, Gabriel**, Buehler LTD – *Pain-Free Metallic Specimen Preparation for EBSD Analysis*  
**Smoliga, John A.**, Boehringer Ingelheim Pharmaceuticals, Inc. – *Microscopical Characterization of Minerals Used in the Pharmaceutical Industry*



Inter/Micro 2009 attendees enjoy some camaraderie after an informative round of microscopy symposia.



SMSI President Sebastian Sparenga (left) congratulates Dr. Kenneth Libbrecht of the California Institute of Technology for winning the SMSI's 2009 Émile M. Chamot Award.

**Brady, Kevin A.**, Tredegar Film Products – *Microscopy of Fruit Snacks: Giving Microscopy Students Something to Chew On*

#### Afternoon Session

**Chair:** Eric J. Chatfield, Chatfield Technical Consulting Limited

**Ford, Brian J.**, Gonville & Caius College, Cambridge University – *How a Fashion Accessory Could Save Your Life*

**Havics, Tony**, pH2, LLC – *Blueberries on Earth and Mars: Correlations Between Concretions in Navajo Sandstone and Terra Meridiani on Mars*

**King, Meggan**, McCrone Research Institute – *Optical Characterization of Sodium Lauryl Sulfate*

**Isphording, Wayne C.**, Tierra Consulting Group – *Moon Rocks (or Bogus Rocks?): A Case History of Discovery and Litigation*

**Rantanen, Walter**, Integrated Paper Services, Inc. – *What They Claim Isn't Always So*

**Brinsko, Kelly**, McCrone Research Institute – *Microscopy of Bicomponent Fibers*

**Chatfield, Eric J.**, Chatfield Technical Consulting, Limited – *ISO 22262: The New Draft International Standard for Determination of Asbestos in Bulk Materials*

### WEDNESDAY, JULY 8: CHEMICAL AND FORENSIC MICROSCOPY

#### Morning Session

**Chair:** Chris Palenik, Microtrace, LLC

**Beckert, Jason**, Microtrace, LLC – *Indian Yellow: Historical Pigment and Modern Confusion*

**King, Meggan**, McCrone Research Institute – *Skeleton Crystals*

**Moorehead, Wayne** – *Myth Busting the Light Microscope*

**Nytes, Brendan**, Microtrace, LLC – *Characterization and Comparison of Ancient Roman Glass*

**Diaczuk, Peter**, John Jay College of Criminal Justice – *Interpretation of Gunshot Residue Patterns Without the Firearm*

**Wilke, Bryn and Palenik, Chris**, Microtrace, LLC – *Organic Pigments: Analytical Characterization and Classification by Raman Spectroscopy*

#### Afternoon Session

**Chair:** John A. Reffner, John Jay College

**Mikuska, Bill C.**, MicroChem Consulting, LLC – *Meteorites in Thin Section: A Petrographic Classification by PLM*

**Bowen, Andrew**, Stoney Forensic – *Foraminiferal Forensics*

**Hopen, Thom**, ATF Forensic Science Laboratory – *Cross-Sections of Duct Tape Backing*

**Ford, Brian J.**, Gonville & Caius College, Cambridge University – *What Linnaeus Missed*

**Palenik, Skip**, Microtrace, LLC – *Talking Rodents and What They Told Us*

**Reffner, John A.**, John Jay College of Criminal Justice – *Is Morphological Analysis Scientific?*

#### Inter/Micro 2009 Banquet and SMSI Auction

**SMSI 2009 Émile M. Chamot Award Recipient:** Dr. Kenneth Libbrecht, California Institute of Technology. Presentation on Snow Crystals.

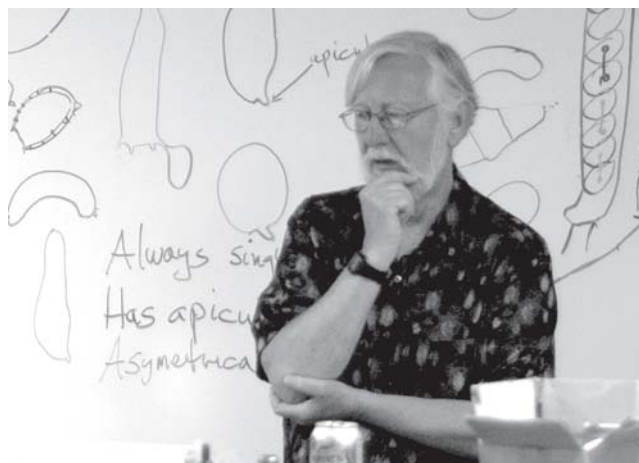
### THURSDAY, FRIDAY, JULY 9, 10, 2009: WORKSHOP

#### **Mysteries of the Ether: Airborne Fungal Spores and More**

*Dr. John Haines, Scientist Emeritus, New York State Museum and Science Services, Albany, NY*

Did you know that the most conspicuous biological particle in air is a fungus spore? Thousands of species of fungi produce airborne spores and in this two-day workshop we will tie the complex world of fungi to the dynamic world of the air. We will look at mushrooms, plant diseases, and the molds that are so much in the news when they grow in buildings. We will have





*Dr. John Haines, scientist emeritus at New York State Museum and Science Services in Albany, NY, leads his two-day workshop on airborne fungal spores.*

an opportunity to look at the particulate content of air in near real time. You will use phase-contrast microscopes to examine air samples and building molds from research studies and home inspections, and we will collect some of the fungi that are the sources of airborne spores.

We will discuss air sampling technology, the types of spores, the common airborne species, and use the technical resources for identifying hundreds more. We will have access to the mycology section of the McCrone Research Institute library and live video that displays through a digital projector. We will examine spores from different regions and investigate the effects of weather on air spora. Dr. Haines will discuss the history of the "mold scare," the effects of airborne spores on human health, and introduce the basics of how molds grow in buildings.

## ABSTRACTS

### **Monday, July 6: Techniques and Instrumentation** **Essential Photoshop for the Microscopist**

*Sebastian Sparenga, McCrone Research Institute*

Do you wish you could get rid of that speck of dust that, no matter how much you clean your scope, keeps showing up in your photomicrographs? Was that one image in your report a bit underexposed compared to the others? Although you take numerous shots at different focal depths to catch your sample at best focus, do you feel your image is not as sharp as it could be? Using Photoshop, considered by many to be the No. 1

image-processing software, I will show you how to correct the most common problems in photomicrographs that are otherwise taken properly with Köhler illumination, proper focus, exposure, and white balance. Are you not using Photoshop because it is too expensive? Photoshop Elements, available for only a fraction of the cost of the full program, will also be explored. Is this still too much to spend? Believe it or not, there is a bare-bones version of Photoshop online that is FREE!

### **Infrared Microscopy**

*Rich Brown, MVA Scientific Consultants*

One of the pleasures of using a variety of microscopes to solve problems on a daily basis is the sample that challenges the microscopist to extend his or her knowledge base to try something new, or at the very least, try a technique that is not routine.

Infrared microscopy is easy, especially if you have a night shot camera. Using a night shot camera as a source of infrared light and a second camera mounted on a transmitted-light microscope or a stereomicroscope allows the light microscopist to capture digital video and digital still images in the infrared, 700 nm-1200 nm. In this way, no IR pass filters are needed for imaging and it can be performed in total darkness to minimize the effects of stray visible light.

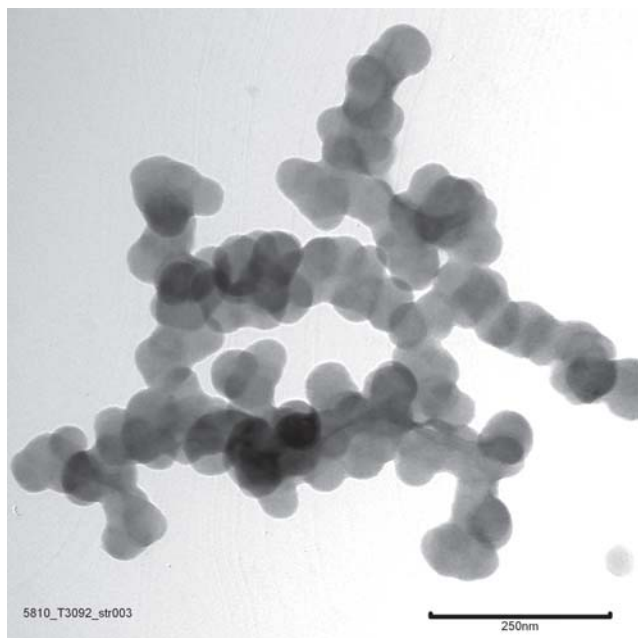
The details to duplicate the set-up and use of infrared lighting will be given. Images showing the potential for this old technique will be presented and revived through the use of digital imaging.

### **Soot**

*James R. Millette, MVA Scientific Consultants*

Soot is generated during the incomplete combustion of organic materials. Individual soot particles show different characteristics depending on the nature of the fuel and the parameters of combustion. The particle characteristics of the most common forms of soot (char soot, cenospheres and aciniform soot) can be used to distinguish between different sources. Commercial varieties of soot, like carbon black, lampblack and coke, have distinctive characteristics that often allow them to be distinguished from the non-commercial forms of soot. The ASTM Standard Practice D6602-03b provides a basis for investigations involving soot particles.

Using this Standard Practice, polarized light microscopy (PLM) can be used to differentiate between soot particles and other dark particles that may be present in a sample. PLM and scanning electron microscopy, equipped with energy dispersive X-ray spec-



*James R. Millette of MVA Scientific Consultants used this TEM image of aciniform carbon, or soot, to explain the types of microscopy for identifying such particles.*

troscopy (SEM-EDS) can be used to differentiate certain forms of coal and coke from other particles of char. Because aciniform soot particles are composed of aggregates of primary particles in the nanometer size range, transmission electron microscopy (TEM-EDS) is used to confirm its presence and provide diagnostic characterization. Aciniform soot particles can be classified according to their morphology (shape and appearance of the primary particles and aggregates), their elemental composition and primary particle size distribution.

High resolution TEM is also useful in looking at turbostratic layering, an attribute of the internal structure of some primary aciniform particles. This presentation will also include illustrations of microscopical characterizations of soot particles that were used in a number of investigations including those concerning California wildfires, industrial combustion sources, candles in residences and a site of a suspected arson.

### **Creating Mineral Art**

*Thomas J. Hopen, ATF Forensic Science Laboratory; David Babulski*

In the period from 1600 to 1900 A.D., as man began to explore the natural world, new discoveries (macro-

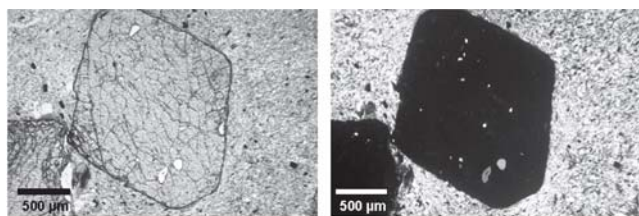
scopic and microscopic) were documented by the natural science artists of the times. This was a period in science B.P. (Before Photography). Many fine volumes of scientific watercolor illustrations from that period still survive. The procedure in this presentation to create a mineral painting utilizes a Motic K400 stereomicroscope with a custom gimbaled mechanical stage and an attached Camera Lucida drawing device in order to capture the rough proportions of the micro-mineral specimen. Once sketched, the small rough drawing of the micro-mineral is enlarged on a photocopier. The enlarged drawing is then overlaid with tracing paper and, using the microscope image as a reference, the drawing is refined. The drawing is then transferred to watercolor paper where it is rendered in color using the microscope image as a constant reference for accuracy of color and form. Once finished, the painting is signed and sent out to be framed. During the framing process, the micro-mineral used to render the painting is mounted in the frame. This makes the final framed illustration a truly unique piece of art because it is the only painting of the specimen.

This presentation chronicles the rise, decline and re-birth of natural science illustration from the microscope. Examples will be shown of the tools and techniques used by the natural science illustrator working with the microscope and micro-mineral specimens. The step-by-step creation of a watercolor painting from the microscope is shown much as it would have been done by an explorer/naturalist in the heyday of natural science illustration.

### **The Next Step in Laser Confocal Technology**

*Christopher Vander Tuuk, Olympus Industrial*

This year, Olympus is introducing the LEXT OLS4000 3D Laser Confocal Microscope (LEXT). The LEXT is a new submicron, 3D imaging and measurement system using 405 nm laser confocal technology. Of particular interest for users of LEXT are the new, noncontact surface-roughness profiling, measurement and analysis functions that are fully ISO compliant with many advantages over existing contact-roughness instruments. The LEXT is a perfect complement to laboratories currently using light and electron microscopes. It is nondestructive, does not require a dedicated operator, and requires scan times typically around 2 minutes. The X/Y resolution of this microscope exceeds 120 nm, with measurement reproducibility in Z of 20 nm; angles up to 85° can be imaged. To enhance the automation, Olympus now uses an ultrasonic stage system for accurate sample positioning.



PPL  
2X Plan Fluorite

XPL

*Tony Havics of pH2, LLC described how compensators can be used in unconventional ways to perform various functions, such as revealing the very low strain on isotropic substances.*

Combining this with application-specific functions makes this an easy-to-operate 3D imaging and measuring microscope for industrial applications and materials analysis. Details of the optical design, technology and applications will be discussed.

### Unconventional Use of Compensators

*Tony Havics, pH2, LLC*

The quarter-wave plate, Red I plate, Senarmont, and Berek compensators are customarily used to determine retardation, optic sign, or sign of elongation in a specimen. They can, however, be used in unconventional ways to perform other functions. These include: revealing very low strain in isotropic substances on the order of 3-5 nm, assisting in the extraction of phase object images, and evaluating birefringence distribution in fibers. Conventional use followed by these three unconventional applications will be demonstrated.

### Teaching Microscopical Analysis of Rocks and Minerals

*Randy Boltin, MVA Scientific Consultants*

"Mineralogy & Petrology" is a 3-hour session taught as part of the Georgia Microscopical Society's *Young People's Course: Learning To Use The Microscope*. The course consists of eight classes that concentrate on familiarizing middle-school-age students with the principles and applications of polarized light microscopy, low power stereomicroscope examinations and electron microscopy.

The focus of the Mineralogy & Petrology class is to combine macroscopic observations with the fine details ascertained through microscopical analysis. Physical properties commonly used for identification of some common rock-forming minerals are correlated with optical properties observed by microscopy. The importance of thin section analysis to study rock tex-

tures is emphasized and students learn how thin sections are prepared from rock samples. Students are introduced to mineral crystallization sequence recognition and the significance of relative grain size in interpreting the origin of igneous rocks. Textures associated with sedimentary rocks (grain sorting, rounding, mature vs. immature sediments), metamorphic rocks (plastic and brittle deformation, recrystallization), cross-cutting relationships (veins, fractures) and weathering are also presented.

### Dispersion Staining Using a 1.2-1.30 NA Cardioid Darkfield Condenser

*Theodore M. Clarke*

My Inter/Micro 2007 presentation demonstrated that the LOMO darkfield cardioid condenser gave good darkfield illumination with the 1.00 NA 60X LOMO apochromatic objective with the punctae of diatom *Frustulia rhomboides* resolved. This presentation will demonstrate dispersion staining with the same combination of condenser and objective. Comparable imaging can be obtained with the 90X 1.25 NA LOMO achromatic objective, when the reducing cone provided for this objective with the condenser has the bore diameter increased by 20%. More precise centering of the reduced diameter was needed and obtained with a home shop-made funnel insert with a closer fit of the guiding surfaces in the bore of the 90X objective.

### Detection of Arsenic by Gas Chromatography with Mass Spectrometry

*Mark E. Palenik, Microtrace, LLC*

Because of its lack of taste, odor and ease of access as a rodenticide (up to as recently as 1950), arsenic, usually in the form of arsenic trioxide, was often the choice of poisons for doing away with the woefully unsuspecting. The "Marsh test," a highly sensitive analytical method and refinement of earlier tests for arsenic, was developed to detect the presence of arsenic, particularly in stomach contents. The use of arsenic as a poison has given way to more exotic poisons, and the Marsh test been replaced by the newer instrumental methods. These instruments are expensive and impractical to purchase if not used on a regular basis. Still, at times, it may be necessary to determine the presence or absence of arsenic. A sensitive method, suitable for analyzing a variety of samples, using aspects of the Marsh test combined with GC-MS is discussed.

### High-Speed Automated Particle Analysis by SEM-EDS

*Rich Brown, MVA Scientific Consultants*





*This slide from Ted Clarke's presentation shows how his microscope setup is arranged for dispersion staining with a cardioid darkfield condenser.*

Scanning electron microscopy (SEM) is a complementary technique to polarized light microscopy (PLM) and transmission electron microscopy (TEM). Add an energy dispersive X-ray spectrometer (EDS) and you have an easy-to-use, rapid technique to image and collect elemental composition on a variety of materials or particles. All of the examinations are carried out by a microscopist who manually selects particles, captures an image and collects an EDS spectrum. This can be a tedious and time-consuming process.

With the commercial introduction of dry (no liquid nitrogen) Silicon Drift EDS Detectors (SDD), EDS collection has become very fast. Meeting attendees will gain an understanding of automated SEM-EDS systems and an appreciation of the time savings in automated and manual particle analysis using SDD detectors. A side-by-side comparison of an automated SEM-EDS using a liquid nitrogen cooled lithium drift silicon detector (SiLi) with an automated SEM-EDS using an SDD will be shown, with both instruments using the same software.

#### **Mounting Particles on a TEM Grid for Analysis by Multiple Instrumental Methods**

*Hazel Bales, Microtrace, LLC*

When dealing with true unknowns, identifying single particles often requires the use of multiple microscopical and other instrumental methods. Therefore, it is advantageous to minimize the amount of sample preparation required to prepare single particles for examination by multiple analytical techniques. The

ability to mount a particle once and then analyze it, as opposed to mounting it in a different manner for each analysis to be conducted, is efficient and reduces the chance that the particle may be lost while being transferred. Mounting particles on TEM grids is one method of approaching this ideal. This presentation will discuss the benefits and some of the drawbacks of employing this technique.

#### **Anti-Monkey Butt Powder and Other Non-Asbestos-Containing Products**

*James R. Millette, MVA Scientific Consultants*

Anti-Monkey Butt Powder is a talcum powder currently sold commercially to athletes for the prevention of chafing and irritation between the skin and athletic equipment. Analysis using the most sensitive polarized light microscopy (PLM) and transmission electron microscopy (TEM) methods did not detect asbestos fibers in the talc. Samples of South African vermiculite were analyzed using sensitive PLM and TEM techniques following special preparation techniques that increase the sensitivity of the analysis. This presentation will discuss the improved sensitivity of analytical methods for asbestos currently being developed and tested. These methods have the capability of detection limits well below 0.1%.

#### **A Unique Leeuwenhoek Microscope Sale in London**

*Brian J. Ford, Gonville & Caius College, Cambridge University*

Of the 500 or so microscopes that Leeuwenhoek made, only nine survive (and one of those is suspect). Just one remained in private hands, and it was sold at Christie's in London on April 8, 2009. This unique and tense occasion raised nearly half a million dollars! Brian shows us the microscope in close-up and explains the background. He also took a cell phone video of the auction sale – the first-ever screening of this remarkable event.

#### **Tuesday, July 7: Environmental and Industrial Microscopy**

##### **Microscopy and Laboratory Accreditation**

*Randy Boltin, MVA Scientific Consultants*

Recent years have seen increasing pressure on laboratories to participate in one or more accreditation programs in order to continue conducting business. The focus of programs such as AIHA, NVLAP and A2LA is to insure that participating labs have appropriate management programs and attending quality assurance policies and operating procedures in place to provide reproducible, defensible and accurate re-



*Ann Caffero of CTL Group awaits her turn to the podium for her presentation on the "Microscopical Characterization of Historic Cementing Material."*

sults to their customers. In 20 years of working as an assessor for the NVLAP program for bulk asbestos fiber analysis by polarized light microscopy, I have witnessed a number of changes, both in the breadth of the program and the attributes of accredited laboratories. The program has become more comprehensive as the emphasis has been placed on accrediting laboratories according to ISO/IEC 17025 requirements. Many requirements seem impractical when viewed from the perspective of small laboratory operations, particularly labs in which the microscope is the primary analytical tool. I believe one contributing factor to this point of view is the fact that training and calibration of the microscopist is at least as important as calibration of the instrument.

#### **Microscopical Characterization of Historic Cementing Material. Part 1: History**

*Ron Sturm, CTL Group*

Hydraulic cementitious materials have been used throughout most of recorded history to provide shelter and build infrastructure that progresses the development of civilization as we know it. Most of these hydraulic cements were made from locally available material, ground to a fine powder, and mixed with

water to produce a matrix that sets and hardened as plaster, mortar, or concrete.

Portland cement, the most recent major classification of hydraulic mineral-based cements, has been around for less than 200 years, and owes its origins to ongoing changes to ancient cements that have been going on for thousands of years. In this presentation, we will trace the major types of hydraulic mineral cements used through the last millennium or two, and describe existing microscopical methods for studying historic building materials.

#### **Microscopical Characterization of Historic Cementing Material. Part 2: Case Studies**

*Ann Caffero, CTL Group*

The second presentation on historic cementing material will provide discussion and illustration of several specific studies of historic structures where microscopical methods have provided great insight into the materials used in their construction. Cases studied will include: ancient Mayan plaster, early American masonry, World War II bunkers, the Berlin Wall, and modern, concrete high-rise structure.

#### **Advances in Microstructural Analysis in the Use of Diamond Grinding Discs for Preparation of Thermally Sprayed Coatings**

*Gabriel Lucas, Buehler LTD*

Microstructural Analysis has played, and continues to play, a key role in the development of thermally sprayed coatings. The advances in equipment and consumable technology for evaluating coatings have enabled laboratory personnel to accurately observe the coating characteristics in a cost effective manner and with great confidence.

Microstructural analysis has been important in the development of new, thermally sprayed coatings used for aerospace, automotive, electronics, medical devices, petroleum and other high and low-tech applications. The coatings that are being developed consist of a combination of materials that vary in hardness and general microstructural characteristics. Accurate microstructural analysis is more important than ever because these new, complex coatings cannot be produced successfully without control of the microstructure. Established metallographic techniques have not always been able to produce accurate reproducible results. A resin-bonded diamond surface was developed that reduces the damage created during grinding of a variety of coating types while maintaining the flatness and integrity of the overall coating microstructure.



## Pain-Free Metallic Specimen Preparation for EBSD Analysis

*Gabriel Lucas, Buehler LTD*

Proper specimen preparation is crucial for achieving the highest quality results from an Electron Back Scatter Diffraction (EBSD) analysis. Any type of damage or deformation on the specimen surface detracts from the pattern quality and makes reproducible results difficult. The first step in specimen preparation is sectioning. Using a metallographic abrasive cut-off saw or diamond precision saw minimizes thermal and mechanical damage and speeds up the grinding and polishing procedure. Mounting the specimen in a conductive mounting medium is recommended when mounting specimens for analysis. Proper sectioning allows grinding to start at the finest possible grit (320 grit or below), reducing the amount of deformation that must be removed during polishing. This presentation will discuss the choices for polishing surfaces (cloths), abrasive types and size, and the use of vibratory polishing to remove the final layer of deformation. The resulting patterns and an example of application for EBSD will be presented.

## Microscopical Characterization of Minerals Used in the Pharmaceutical Industry

*John A. Smoliga and Michelle Raikes, Boehringer Ingelheim Pharmaceuticals, Inc.*

The pharmaceutical industry utilizes approximately 30 minerals from both natural and synthetic sources for various purposes. These include active pharmaceutical ingredients (API's), excipients (non-active ingredients) and coloring agents, along with fillers used in polymeric packaging materials. Examples of API's include "Calkinsite" (La-carbonate tetra hydrate) used as a phosphate binder for dialysis patients and calcite (Ca-carbonate) used as an antacid. Excipient examples include brushite (dibasic Ca-phosphate dihydrate) and monetite (dibasic Ca-phosphate anhydrous) used as diluents. Minerals used as coloring agents include hematite ( $\text{Fe}_2\text{O}_3$ ) and goethite (Fe-oxide hydroxide). Finally, examples of packaging fillers include talc ( $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ ) and kaolinite (Hydrated Al-Silicate).

As with all pharmaceutical ingredients, the FDA requires that these minerals be controlled for quality and purity. Characterization and analyses of these minerals are conducted using polarized light microscopy, scanning electron microscopy, energy dispersive X-ray fluorescence spectroscopy, Fourier transform infrared microspectroscopy, Raman



*Kevin Brady of Tredegar Film Products gives his audience something to chew on during his "Microscopy of Fruit Snacks" presentation.*

microspectroscopy along with X-ray powder diffraction. A survey of minerals used in the pharmaceutical industry; their specific function, and the methods for their characterization and control will be presented.

## Microscopy of Fruit Snacks: Giving Microscopy Students Something They Can Chew On

*Kevin A. Brady, Tredegar Film Products*

Materials analyzed in light microscopy training classes are carefully selected as outstanding examples of the concepts under study. These subject materials usually also provide fascinating images that can capture the students attention and peak their curiosity. While looking for something new and different to observe, I subjected some candy fruit snacks to microscopical methods and was surprised at the number of techniques that could be applied to them. They might be useful for teaching microscopy or, at least, provide some sustenance while waiting for lunch to arrive. Images from a variety of microanalytical techniques will be presented.

## How a Fashion Accessory Could Save Your Life

*Brian J. Ford, Gonville & Caius College, Cambridge University*

Recent outbreaks of virus diseases are threatening major cruise lines, jeopardizing famous hotels, and closing down world-famous restaurants. Last year, headlines featured Norwalk virus (now renamed Noro



*Pete Diaczuk (left) and John Reffner (center) of John Jay College of Forensic Science share a few chuckles with Guy De Baere during a conference break.*

virus, which is easier for people to say). Presently it is influenza H1N1, the so-called swine flu. Much is known of these viruses, but less attention is paid to how they spread. There have been changes introduced on the advice of microbiologists, but there remain pitfalls for the unwary. I will reveal some unexpected places where the virus likes to hide and show how the re-introduction of an age-old fashion accessory could help prevent an epidemic-and save lives.

#### **Blueberries on Earth and Mars: Correlations Between Concretions in Navajo Sandstone and Tyerra Merdiani on Mars**

*A. Havics, pH2, LLC; W.C. Mahaney, York University (North York, Ontario); M.W. Milner, York University (North York, Ontario); D. Netoff, Sam Houston State University; James Dohm, University of Arizona; V. Kalm, Tartu University (Tartu, Estonia); D. Krinsley, University of Oregon; R.N.S. Sodhi, University of Toronto; R.C. Anderson, Jet Propulsion Laboratory; S. Boccia, University of Toronto; D. Malloch, New Brunswick Museum*

Concretionary Fe-Mn-rich nodular authigenic constituents of Jurassic Navajo sandstone (moki marbles) bear a certain relationship to similar concretionary forms ("blueberries") observed on Mars. Their origin on Earth is considered to invoke variable redox conditions with underground fluids penetrating porous quartz-rich sandstone leading to precipitation of hematite and goethite-rich material from solution, generally forming about a central nucleus of fine particles of quartz and orthoclase, recently verified by XRD and SEM-EDS analyses.

At the outer rim/inner nucleus boundary, bulbous lobes of fine-grained quartz often invade and fracture the outer rim armored matrix. The bulbous forms are interpreted to result from fluid expulsion from the inner concretionary mass, a response to pressure changes accompanying overburden loading. Moki marbles, harder than enclosing rock, often weather out of *in situ* sandstone outcrops and lie about on surfaces exposed to the subaerial atmosphere, developing a varnish-like crust. The marbles appear morphologically similar to "blueberries" identified on the martian surface in Terra Meridiani through the MER-1 Opportunity rover.

On Earth, redox fluids responsible for the genesis of marbles may have emanated from deep in the crust (often influenced by magmatic processes). These fluids, cooling to ambient temperatures, may have played a role in the genesis of the cemented outer rim of the concretions. The low frequency of fungi filaments in the marbles, contrasts with a high occurrence in Fe-encrusted sands of the Navajo formation, indicating that microbial content is of secondary importance in marble genesis relative to the fluctuating influx of ambient groundwater. Nevertheless, the presence of filaments in terrestrial concretions hints at the possibility of discovering fossil/extant life on Mars, and thus should be considered as prime targets for future reconnaissance missions to Mars. This presentation will look primarily at PLM and SEM-EDX analysis of thin sections and polished sections of some concretions.

#### **Optical Characterization of Sodium Lauryl Sulfate**

*Meggan King, McCrone Research Institute*

Sodium Lauryl Sulfate (SLS) is a common anionic surfactant found in cleaning and hygiene products. Some well formed crystals of SLS were recently encountered in the casework of a colleague and identified using instrumental analysis. A secondary confirmation using Polarized Light Microscopy (PLM) was not possible due to the fact that the optical properties of SLS had not been determined. Since SLS has such widespread use, it would be beneficial to have the optical properties readily available to microscopists. A standard of SLS was obtained and characterized using the PLM. The methods used and resulting data will be presented.

#### **Moon Rocks (or Bogus Rocks?): A Case History of Discovery and Litigation**

*Wayne C. Isphording, Tierra Consulting Group*

Thirty years ago on July 16, 1969, Apollo 11 left Launch Complex 39A at the Kennedy Space Center

bound for the moon. Four days later the Lunar module touched down at 4:18 p.m. EDT, July 20, 1969. Man's first step on the moon was taken by Neil Armstrong at 10:56 p.m. and, shortly thereafter, he was joined by his fellow astronaut, Edwin "Buz" Aldrin. The two spent a total of 2 hours, 31 minutes outside the lunar module where they collected 22 kg of lunar samples. Some 20 years later a consulting geologist was contacted and asked by an individual to confirm that a sample (55 grams) in his possession was actual "soil lunar material" from the Apollo 11 mission.

From its sale, the owner anticipated reaping a substantial profit. Lacking the necessary analytical equipment, the consultant contacted his former university mentor and requested his assistance. The sample was duly produced and subjected to both petrographic, X-ray, and geochemical analysis. To the owner's chagrin, all tests argued strongly against a "moon origin" for the sample. The owner, disappointed (and exasperated!) refused to compensate the consultant. Subsequently he was advised that all lunar samples from Apollo 11 were the property of NASA and even if the sample had been true lunar material, any attempted sale would be a Class A felony in violation of Federal law.

### What They Claim Isn't Always So

*Walter Rantanen, Integrated Paper Services, Inc.*

Certain parties may claim that only a certain wood was used or that a specific fiber type was added to the paper. We can determine how accurate these verbal and written claims may be, when microscopical examinations are performed. In a number of instances where we performed an analytical test, the professed type of wood that was used or the fiber type stated contradicts the scientific evidence. In some cases, the opposing side refutes these findings and insists on their original claim. Several examples of these contraventions will be presented.

### Microscopy of Bicomponent Fibers

*Kelly Brinsko, McCrone Research Institute*

Bicomponent fibers are man-made fibers that are comprised of two individual polymers, often in a side-by-side or sheath-core arrangement. Bicomponents have been manufactured for decades, but literature concerning their microscopical characteristics and identification is sparse. Furthermore, recognizing an unknown fiber as a bicomponent may sometimes be difficult. Several bicomponents of various fiber types were examined microscopically and include the analysis of longitudinal and cross-sectional shape,



*Dr. Eric Chatfield explains ISO No. 22262, a new draft international standard for determining the presence of asbestos in bulk materials.*

optical properties, and thermal (hotstage) methods. A brief history of bicomponents, including manufacturing processes, fiber end uses, and occurrences will be discussed.

### ISO 22262: The New Draft International Standard for Determination of Asbestos in Bulk Materials

*Eric J. Chatfield, Chatfield Technical Consulting Limited*

In 2004, Working Group ISO/TC 146/SC 3/WG1 of the International Organization for Standardization (ISO) began work to prepare a standard analytical method for determination of asbestos in bulk materials. Since then, there has been steady progress towards consensus on this analytical method, and the first part of the method, which specifies procedures for sampling and qualitative analysis, has been submitted for registration as a Draft International Standard.

The primary purpose of the method is to demonstrate whether the material being analyzed is subject to asbestos regulations. However, the definition of an asbestos-containing material varies between jurisdictions from "any asbestos," to concentrations of asbestos specified as "greater than 0.1%," "greater than 0.5%" or "greater than 1%," and each of these definitions must be accommodated by the method. Development of a single analytical method that addresses the needs of all of the participating ISO member countries presents considerable challenges because the different asbestos regulations in various countries impact upon both the limit of detection required and the analytical procedures that are appropriate.





*In his presentation on classroom microscopy, Wayne Moorehead set the record straight by disproving the 10 most common myths about the light microscope, which are typically learned in elementary and high school science courses.*

The primary fiber identification procedure in the method is based on polarized light microscopy and dispersion staining, which is the simplest procedure for identifying mineral fibers. It was therefore necessary to develop a dispersion staining chart that accurately represents the colors seen in the polarized light microscope. Alternatively, or as a supporting technique, fiber identification by either scanning electron microscopy or transmission electron microscopy with energy dispersive X-ray analysis is permitted. For the purpose of regulatory compliance, accurate quantification of asbestos is important only for those samples in which asbestos is present and the concentration is estimated to be lower than approximately 5%. Procedures for quantification of asbestos are described in a second part of the method, which is currently at an earlier stage of the ISO standardization process.

### **Wednesday, July 8: Chemical and Forensic Microscopy**

#### **Indian Yellow: Historical Pigment and Modern Confusion**

*Jason Beckert, Microtrace, LLC*

This presentation will briefly discuss Indian Yellow's history and its use as an artists' pigment. While relatively common centuries ago, especially in India, this pigment is not currently produced commercially. Its tentative identification in a recent case will lead to an examination of the modern pigments commonly used as substitutes, and the confusion surrounding the nomenclature currently in use.

### **Skeleton Crystals**

*Meggan King, McCrone Research Institute*

During the 2007 excavation season at the Byzantine city of Amorium located in central west Turkey, a burial place in the atrium of the lower city basilica was examined. It was noted by the archaeologists that the skeletal material of one of the occupants had developed tiny crystals on the surface. A small piece of the skull with crystals present and some loose bone fragments were collected and submitted to the McCrone Research Institute for analysis. Initial observations were made using a stereomicroscope. Single crystals were examined using the polarized light microscope (PLM).

Based on initial observations of the optical properties of these crystals, various microchemical tests were conducted. These crystals were found to contain calcium and phosphates. To determine the precise identity of the material, a single representative crystal was mounted on a spindle stage. By rotating the crystal around two axes we were able to determine the optic axial angle as orientations and magnitudes of the alpha, beta and gamma refractive indices and identify the crystal.

### **Myth Busting the Light Microscope**

*Wayne Moorehead*

The 10 most common myths involving the light microscope will be explored and the reality will be discussed. These myths are typically learned, but not taught, from elementary school through high school and occasionally continue in some college classes. Due

to the lack of proper texts for the student, ignorance of the proper use of the microscope, or for presumed ease of teaching by instructors, students learn these myths. These myths are reinforced each year the student progresses through school and has interaction with the most basic of scientific tools, the microscope.

Spectroscopists know their instruments must be properly setup and checked before each use. Chromatographers check the performance of their instruments before using them for industry or research. The microscope must be set up and used properly or the results will not be good. Describing the myths and the realities will better prepare the student to use the microscope properly and to obtain the most information from the available image.

### Characterization and Comparison of Ancient Roman Glass

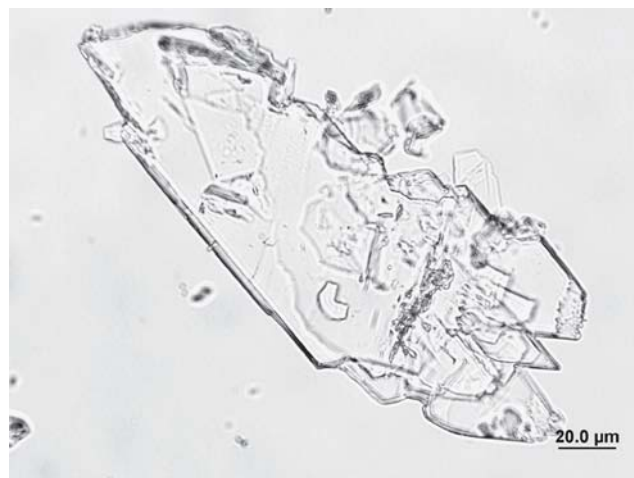
*Brendan Nytes, Microtrace, LLC*

Glass has been produced by humans for centuries. Over time, the process has been refined and the uses of glass have expanded. This talk will explore the characteristics and compositions of multiple pieces of ancient Roman glass. The pieces of glass were analyzed and compared to each other, and the characteristics and composition of the ancient Roman glasses were compared to the characteristics and composition of modern glasses.

### Interpretation of Gunshot Residue Patterns Without the Firearm

*Peter Diaczuk, John Jay College*

Gunshot residue (GSR) is often present at shooting scenes, especially at close range. The density of the GSR pattern and the approximate diameter of the pattern afford information about the muzzle-to-target distance, a factor often in dispute depending upon the circumstances. The standard method for pattern interpretation is to fire test shots at the appropriate material using the ammunition and firearm in question at known muzzle-to-target distances to create discharge patterns. These known patterns are then compared to the questioned pattern with the goal of bracketing the unknown distance as best as possible. However, if no firearm is recovered, GSR test patterns are usually not made. This research was undertaken to show that some useful information could nevertheless be derived from test shots without possessing the firearm but knowing the ammunition and limiting firearm variables to those in current manufacture. Pow-



*Meggan King of McRI observed that crystals found on a skeleton at an ancient burial site in Turkey were colorless, anisotropic and biaxial with symmetrical extinction.*

der particles embedded in the test fabric will be used as the criterion for drawing conclusions. Low-power microscopy will be used to demonstrate the presence and frequency of propellant particles on the fabric. Some of the variable powder morphologies used by the ammunition manufacturers will also be considered as a factor contributing to particle deposition.

### Organic Pigments: Analytical Characterization and Classification by Raman Spectroscopy

*Bryn Wilke and Chris Palenik, Microtrace, LLC*

While pigments are present in a wide variety of trace evidence that is commonly examined (e.g., plastics, fibers, paints, coatings, inks), they are not generally exploited for their evidentiary value. Despite this precedent, pigments offer a great deal of potential information as both a comparative and investigative tool due to the large variety of pigments that are currently produced and are in use. Through a variety of analytical methods, including polarized light microscopy, microchemistry, energy dispersive X-ray spectroscopy and especially Raman microspectroscopy, pigments can be characterized and identified.

While it is known that many organic pigments can be identified by Raman spectroscopy, little work has gone into systematically characterizing various classes of organic pigments. In particular, the extent to which pigments within a particular class can be analytically differentiated by Raman spectroscopy in a forensic context is not known. Here we present a sys-



*Bernadette O'Reilly (center) and fellow participants of the two-day workshop at McCrone Research Institute focus in on the complex world of airborne fungi.*

tematic study of several classes of organic pigments and the ways in which their Raman spectra vary as a function of chemical structure.

#### **Meteorites in Thin Section: A Petrographic Classification by PLM**

*Bill Mikuska, MicroChem Consulting, LLC*

Inspired by Ernst Florens Friedrich Chladni, father of meteoritics, Gustav Rose chemically analyzed, catalogued and classified the collection of meteorites in the University Museum of Berlin from 1821 until 1869. This pioneering work using wet chemical analytical techniques was augmented with transmitted-light microscopical observations of sturgeon glue replicas and thin sections of meteorites. Since that time, the number of known meteorites has grown from 230 to more than 32,000, as new falls or finds provide additional specimens, all needing classification. Although newer analytical tools such as SEM, XRD, mass spectroscopy and light spectroscopy are currently employed, optical properties are still relied upon to aid in classification. This paper presents current ideas of meteorite classification from information gathered by light microscopy.

#### **Foraminiferal Forensics**

*Andrew Bowen, Stoney Forensic*

Foraminifera are single-celled protozoa whose tests are found in great abundance on some beaches and as fossils in some marine limestones. A general introduction to foraminifera and their tests will be followed by a discussion of their potential applica-

tions in forensic science. Several case examples will be provided where foraminifera were of value to the speaker during forensic investigations.

#### **Cross-Sections of Duct Tape Backing**

*Thom Hopen, ATF Forensic Science Laboratory; Natasha Neel, ATF Forensic Science Laboratory; Jenny Smith, Missouri State Highway Patrol Crime Lab; Jennifer Snipe, ATF Forensic Science Laboratory*

Duct tape examinations and comparisons in the forensic laboratory involve looking at the backing, scrim and adhesive by a variety of different analytical techniques. In recent years, the duct tape backings have become more important in the scheme of things since manufactures have started producing multilayer backings as opposed to just single-layer backings. This presentation, which is part of a larger study, focuses on the examination and analysis of 17 recently manufactured duct tape backings by preparing cross-sections duct tapes and analyzing the layers by PLM, SEM-EDS and FTIR.

#### **What Linnaeus Missed**

*Brian J. Ford, Gonville & Caius College, Cambridge University*

The 18th century Swedish naturalist Carl Linnaeus laid down the present-day system of classification for living organisms. He established the binomial system of genus and species for the plant and animal world. Yet he had a blind spot for microorganisms, and his little-known booklet on microscopic life is inaccurate and lacks illustrations. His attempts at classifying these organisms were weak. He did have a microscope, which was designed by the English philosopher John Ellis. Today we will see how this microscope performed and look at the surviving drawings from Linnaeus's own hand that show how little he studied the use of it.

#### **Talking Rodents and What They Told Us**

*Skip Palenik, Microtrace, LLC*

In the last several years, we have noted a steady increase in food contamination complaints through our Food Forensics division. A surprising number of these concern dead rodents, most typically, mice. Because these problems involve *animals* and not *things* (which we are more used to), we initially approached these cases with some degree of apprehension. We were not certain that the problem-solving skills we had developed over the years would be useful in answering the questions that arise when a dead rodent is found in a food product. In this presentation, we will demonstrate that the skills of forensic chemical microscopy, coupled



with diagnostic veterinary medicine, can resolve these problems with a high degree of certainty in most cases. Three cases will be considered, which show how specific facts led to conclusions that answered the question of how and when the mouse got into the food.

### Is Morphological Analysis Scientific?

*John A. Reffner, John Jay College of Forensic Science*

The National Academy of Science report titled: "Strengthening Forensic Science in the United States: a Path Forward," released in February 2009, raises several issues that will impact forensic microscopy. In this report, DNA analysis is given high marks of scientific validity, while comparative analysis such as tool marks, firearms, fractures and fingerprints are graded as less scientific. Two issues addressed by the NAS committee that were listed in their executive summary are:

- "the fundamentals of the scientific method as applied to forensic practice—hypothesis generation and testing, falsifiability and replication, and peer review of scientific publications;"

- "the assessment of forensic methods and technologies—the collection and analysis of forensic data; accuracy and error rates of forensic analyses; sources of potential bias and human error in interpretation by forensic experts; and proficiency testing of forensic experts."

These issues challenge the way we approach morphological analysis. Human observation is the foundation for building a scientific analysis. An experienced and trained individual will see more than the novice.



*In his presentation on "Talking Rodents and What They Told Us," Skip Palenik explained how food contamination problems are solved with forensic chemical microscopy.*

Pathologist, geologists, biologists, fractographers, virologists, metallographers and forensic scientists rely on morphology to analyze matter, its properties and its history. Neither unanswered questions nor unquestioned answers are a satisfactory solution. We must question our methods and move forward to advance our science. Questions are raised, so let the debate begin.

Thank you to all the speakers, exhibitors and participants of Inter/Micro 2009!