

Inter/Micro 2010

Gary J. Laughlin
McCrone Research Institute*

McCrone Research Institute celebrated its 50th anniversary by hosting and conducting the annual Inter/Micro conference on July 12-16 at its facility on Chicago's South Side. The 62nd annual conference drew more than 100 attendees and speakers from around the world. It marked the first time that McCRI held Inter/Micro in its building, using newly remodeled lecture rooms, laboratories and classrooms.

Attendees heard in-depth research presentations by leading microscopists, who covered advancements in instrumentation, techniques and applications in various fields of microscopy and microanalysis. Presentations focused on PLM, SEM, EDS, Raman, hot-stage and infrared microspectroscopy; microchemistry; forensic trace evidence; pharmaceutical sciences; materials analysis; art authentication; environmental health; food and air quality.

During the first three days of the conference, speakers gave research presentations on such diverse topics as "The Making of a Masterpiece: Picasso's 'First Steps'," "Use of PLM, SEM, TEM and Raman in the Characterization of Coal Fly Ash," "The Sugary Sweet Side of Microscopy," "Analysis of Chinese Drywall that Produced Irritant Health Effects and Corrosion in the Southeast United States," "Fungi as Forensic Tools,"



Rich Brown gives a talk at Inter/Micro 2010, held at the McCrone Research Institute in Chicago.

and "Microanalysis of Invasive Traumas." In his annual Evening with Brian talk, "Censoring the Cell, How the Microscope is Abused by the Media," Professor Brian J. Ford explained how today's television documentary programs avoid showing the microscope and microorganisms.

Inter/Micro participants enjoyed a Southern-style barbecue dinner in the McCRI garden and also participated in a silent auction held by the State

Microscopical Society of Illinois (SMSI). A wine and cheese reception featured products from conference exhibitors, including Leica, BioExpress, CRAIC Technologies and Microscope Publications.

This year's SMSI Awards Dinner was held at The Berghoff restaurant in Chicago's downtown Loop. The SMSI 2010 Émile Chamot Award went to Skip Palenik, a leading forensic microscopist and founder of Microtrace, LLC, in Elgin, Illinois (Chris Palenik accepted the award on his behalf). Daniel Kile, scientist emeritus with the U.S. Geological Survey (now retired), received the SMSI 2010 August Köhler Award and gave a talk on optical mineralogy and crystallography.

The winners of the Inter/Micro 2010 Photomicrography Competition were also announced at the dinner. Kelly Brinsko won Best Overall Photomicrograph, Sebastian Sparenga claimed the Best SEM Photomi-

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Inter/Micro 2010 participants take a break from presentations to chat with exhibitors and learn about the latest microscopy equipment on the market.

crograph and Ming Zhou had the Most Unique Photomicrograph (see winning images on page 114.)

A two-day workshop on animal hair identification in McCrone's classrooms highlighted the second half of Inter/Micro 2010. Jason Beckert, research microscopist at Microtrace, taught the hands-on course and guided participants through the techniques involved in animal hair identification with the light microscope. The workshop covered mammalian taxonomy, microscopic hair anatomy, identification criteria, and examination of hair cuticles, medullas and cross-sections.

Inter/Micro, the premier International/Microscopy conference, was initiated by McCrone Research Institute founder Walter C. McCrone and Charles Tufts, and was first held in Chicago in 1948. Each year, the conference attracts professional and amateur microscopists from all areas of light and electron microscopy.

McCrone Research Institute would like to thank all speakers, exhibitors, sponsors and participants for making Inter/Micro 2010 an overwhelming success.

PROGRAM

Monday, July 12: Techniques and Instrumentation

Morning Session – James R. Millette, Chair

Cyanoacrylate as an Embedding Medium for Fiber Cross-sections – Sebastian Sparenga, McCrone Research Institute

Hot Stage Microscopy Before 1900 – Tony Havics, pH2 LLC

Scanning Electron Microscopy Characterization of Epitaxially Grown Aluminum Oxide Employed as Sensor Substrates – Martin Kocanda and Bryn M. Wilke, Northern Illinois University

The Making of a Masterpiece: Picasso's "First Steps" (1943) – Patricia Garland, Yale University Art Gallery

Hot Stage Microscopy 2000-2009, Where Does it Stand? – Tony Havics, pH2 LLC, and John A. Smoliga, Boehringer Ingelheim

Use of PLM, SEM, TEM and Raman in the Characterization of Fly Ash – James R. Millette, MVA Scientific Consultants

Afternoon Session – Jan Hinsch, Chair

Sample Preparation Using an Ion Beam Cross-Section Polisher – Richard S. Brown, MVA Scientific Consultants

Nanoparticle Research in the 1960s – Eric J. Chatfield, Chatfield Technical Consulting Limited

Advantages of Silicon Drift Detectors (SEM/EDS) in Pharmaceutical Applications – R.A. Carlton, GlaxoSmithKline

Introducing Children to the Micro-Life of Fish Lake – Theodore M. Clarke, Retired Materials Engineer

Should Microscopists Take Note of the Micro-Four-Thirds Standard? – Jan Hinsch

Evening with Brian: "Censoring the Cell: How the Microscope is Abused by the Media" – Brian J. Ford, Gonville & Caius College, Cambridge University



Attendees enjoy scrumptious German-American cuisine at Chicago's historic Berghoff restaurant during the SMSI 2010 Awards Dinner.

Tuesday, July 13: Environmental and Industrial Microscopy

Morning Session – Richard S. Brown, Chair

The Sugary Sweet Side of Microscopy – Meggan King, McCrone Research Institute

Asbestos and Vintage Cosmetic Talc Products – Randy Boltin, MVA Scientific Consultants

Use of Malachite Green Stain as an Auxiliary Technique for Differentiating Sepiolite from Chrysotile – Lou Solebello, International Asbestos Testing Laboratories, Inc.

Using Aqueous Elutriation to Isolate Respirable Fractions of Asbestos Fibers from Talc – S.P. Compton, MVA Scientific Consultants

How Microscopic Particles Shut Down the Airways – Brian J. Ford, Gonville & Caius College, Cambridge University

Ettringite: Not Really a Very Nice Mineral!! – Wayne C. Isphording, University of South Alabama

Environmental Microscopy at 200 Feet – Richard S. Brown, MVA Scientific Consultants

Afternoon Session – Tom Kremer, Chair

Investigation of Polarized Light Microscopy Differentiation of Erionite from Other Fibrous Zeolites – Lou Solebello and Craig Liska, International Asbestos Testing Laboratories, Inc.; Gary Tomaino, Minerals Technologies Inc.

The Great Mouse Detective – Brendan Nytes, Microtrace, LLC

Cellulose Ester Filter Quality Issues – Eric J. Chatfield, Chatfield Technical Consulting Limited

Analysis of Chinese Drywall that Produced Irritant Health Effects and Corrosion in the Southeast United States – Arthur W. Struss, USG Research (retired)

Field Microscopy in Environmental, Safety and Health – Tony Havics, pH2 LLC

Methods and Benefits of Staining in the Microscopy of Consumer Products – Tom Kremer, Integrated Paper Services

Wednesday, July 14: Chemical and Forensic Microscopy

Morning Session – Jason Beckert, Chair

The Micro-Aquarium...Why Don't You Have One?! – Richard S. Brown, MVA Scientific Consultants

The Hammer is Back – Peter Diaczuk, John Jay College; Gerard Petillo

Fungi as Forensic Tools – Bernadette O'Reilly, Microtrace, LLC



Dan Kile, the SMSI 2010 August Köhler Award recipient, presents his research in optical mineralogy and crystallography during the awards dinner at The Berghoff restaurant.

Microanalysis of Invasive Traumas – P.D. Zoon, Netherlands Forensics Institute; S.B.C.G. Chang, I. Keereweer, R. Pieterman, M.C. de Boer, M.P.J. Pit and R.R.R. Gerretsen, E.J. Vermeij

A Method for Isolating Trace Evidence from Plastic Explosive Samples – Andrew Bowen, Stoney Forensics, Inc.

Microcrystal Tests for the Detection of Cathinone and Cathine in Khat – Kelly M. Brinsko, McCrone Research Institute

A Dog by Any Name: The Microscopical Identification of the Hair of the Japanese Raccoon Dog (*Nyctereutes procyonoides*) – Jason Beckert, Microtrace, LLC

Afternoon Session – R.A. Carlton, Chair

A Microchemical Approach to Meth Labs – Jim Dunlop, Kalamazoo County Sheriff's Department

What Was I Thinking? – Mark E. Palenik, Microtrace, LLC

The Microscope and the Royal Society – Brian J. Ford, Gonville & Caius College, Cambridge University



Instructor Jason Beckert (right) explains a laboratory technique to Pete Diazcuk during the two-day Inter/Micro 2010 workshop on animal hair identification.

A Compound Microscope by Harris of London, a Classic of the Pre-achromatic Era – Jan Hinsch
Forensic Science in the Pharmaceutical Industry – A Microscopy Perspective – Pauline Leary and John A. Reffner, John Jay College

Microscopical Analysis to Facilitate Problem Solving in the Paper Industry – Walter J. Rantanen, Integrated Paper Services, Inc.

Applications of Optical Microscopy in the Study of Polymorphism – R.A. Carlton, GlaxoSmithKline
McCrone Research Institute's 50th Anniversary – Gary J. Laughlin, McCrone Research Institute

State Microscopical Society of Illinois 2010 Awards Dinner

Émile Chamot Award recipient: **Skip Palenik**
 August Köhler Award recipient: **Daniel Kile**

Thursday, Friday, July 15, 16: Workshop

Animal Hair Identification

Jason Beckert, Microtrace, LLC

Lectures, demonstrations and laboratory sessions guide students through the techniques and practice of animal hair identification by microscopy:

- Mammalian taxonomy
- Necessity and assembly of a reference collection
- Microscopic hair anatomy
- Most useful books for hair identification
- Low magnification examination for color, shape, banding and dimensions
 - Examination of the cuticle, preparation of casts
 - Examination of the medulla, infiltration and staining

- Examination and preparation of cross-sections
- Criteria for identification

ABSTRACTS

Monday, July 12: Techniques and Instrumentation

Cyanoacrylate as an Embedding Medium for Fiber Cross-Sections

Sebastian Sparenga, McCrone Research Institute

Although there are several great methods for cross-sectioning fibers, another technique using cyanoacrylate (Krazy Glue®, Super Glue®, etc.) has been brought to our attention. This talk will discuss how to perform fiber cross-sections using cyanoacrylate as well as evaluate several different brands of cyanoacrylate-based glue. Comparison of this method to other cross-section techniques will also be explored.

Hot Stage Microscopy Before 1900

Tony Havics, pH2, LLC

Many seasoned microscopists are familiar with the work of Dr. Walter McCrone, the Kolflers and Maria Kuhnert-Brandstätter, covering the period from about 1930 to 2000. A few also are aware of the early work by Otto Lehmann entitled *Molekularphysik* in 1888, as well as his later work with liquid crystals. There were, however, well over two dozen hot stages constructed by different scientists prior to 1900. The idea of a warming stage dates back to at least 1838 with Raspail, while creation and publication didn't seriously start until the 1870s and continued through the turn of the century. The primary types of heating varied and can be classified into four categories: hot air, electricity, conduction through metal and water-based. A survey of these glorious instruments will be presented.

Scanning Electron Microscopy Characterization of Epitaxially Grown Aluminum Oxide Employed as Sensor Substrates

Martin Kocanda and Bryn M. Wilke, Northern Illinois University

Anodic Aluminum Oxide (AAO) films have been employed as protective coatings since the 1950s and more recently as a decorative metallic finish. The porosity, surface morphology and fabrication methods of these films have been studied extensively. Common to the commercial AAO films is the anodization process of aluminum fabricated in the (100) crystallographic plane using inorganic electrolytes. A more recent application of this anodic process using low solubility salts has been the fabrication of hexagonal tem-

plates to grow domains of nanowires and nanotubes.

The use of microelectronic fabrication methods to epitaxially grow (111) aluminum thin films and the subsequent anodization method has been recently employed to implement nanostructured AAO materials as commercial moisture sensors and as substrates to study the adsorption response of volatile organic compounds (VOCs) and biochemical compounds. These epitaxially grown films contain nanoporous structures having pore morphologies similar to the (100) film but appear to grow radially from the tetrahedral and hexagonal domains. In this work, the surface morphology of epitaxially grown porous nanostructures is elucidated using scanning electron microscopy (SEM) and atomic force microscopy (AFM).

The Making of a Masterpiece: Picasso's "First Steps" (1943)

Patricia Garland, Yale University Art Gallery

Technical examination of this important painting belonging to the Yale University Art Gallery was undertaken in advance of "Picasso and the Allure of Language," an exhibition on view at the Yale University Art Gallery from January 27 to May 24, 2009 that later traveled to the Nasher Museum at Duke University. "First Steps" was first exhibited at the Salon d'Automne in Paris in October 1944, the first exhibition of French art since the city's liberation from German occupation during World War II. The child is monumentally painted, a strong central image that presses toward the edges of the canvas. The mother hovers over the child but recedes into the background, appearing as a quiet, stabilizing figure.

The discovery of earlier compositional layers helps to clarify issues surrounding the date of completion of the painting. Technical examination of "First Steps" has shed light on the evolution of this important composition, positioning it more decisively within the artist's oeuvre. It has aided scholarship and furthered insight into Picasso's working methods. Picasso's facile ability to amend his composition to his liking, using earlier elements and discarding others, provides a fascinating window into the artist's genius.

This presentation will highlight art historical research completed in tandem with the technical study.

Use of PLM, SEM, TEM and Raman in the Characterization of Coal Fly Ash

James R. Millette, MVA Scientific Consultants

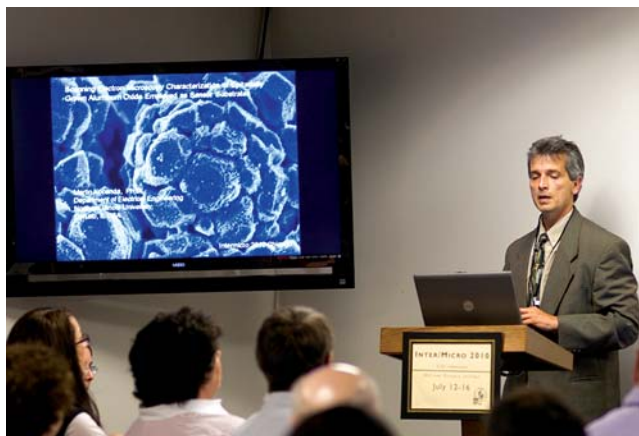
In December 2008, a dam of a landfill holding coal fly ash at the Tennessee Valley Authority Kingston power plant broke, releasing about 5.4 million cubic



Patricia Garland of the Yale University Art Gallery describes the techniques that artist Pablo Picasso used to create his 1943 masterpiece painting "First Steps."

yards of ash into the surrounding area. It is estimated that the spill will cost up to \$975 million to clean up. Questions were raised about how the coal ash particles might disperse into local waterways and adjacent properties. Reference samples of fly ash were obtained from various sources including the National Institute of Standards and Technology (NIST – SRM 2689). Fly ash particles were characterized by several light and electron microscopy procedures. The samples were first analyzed by stereomicroscopy and then by polarized light microscopy (PLM) augmented with top light illumination (reflected light). Samples were also analyzed by scanning electron microscopy (SEM) coupled with an X-ray energy dispersive spectrometry (EDS) system. Automated SEM analysis was performed to determine the size range of the fly ash particles and to obtain information about the range of elemental compositions. The fine fractions were analyzed by analytical electron microscopy (AEM) using a transmission electron microscope (TEM) equipped with an X-ray analysis system. Crystals found within some fly ash particles were analyzed using a WITec alpha 300R confocal Raman microscope equipped with a 532 nm excitation wavelength laser.

PLM was used to classify fly ash particles into a number of different morphologies including solid spheres, cenospheres (spheres with air bubbles), plerospheres (spheres packed with other spheres), and opaque magnetic spheres. SEM and TEM elemental analysis showed that fly ash particles could be characterized based on their elemental composition; primarily aluminum and silicon ceramic spheres with



Martin Kocanda of Northern Illinois University discusses how the surface morphology of epitaxially grown porous films is identified by scanning electron microscopy.

varying amounts of iron, potassium, titanium, calcium, sodium and/or magnesium.

Automated SEM analysis shows that for some samples of fly ash, over 60% of the particles are less than 2.5 micrometers in diameter, but over 70% of the mass is in the particles greater than 10 micrometers in diameter.

Sample Preparation Using an Ion Beam Cross-Section Polisher

Richard S. Brown, MVA Scientific Consultants

Sample preparation has been the key to successful problem solving in materials analysis. Relatively soft materials and hard materials can be cut and polished to examine in cross-section. Soft materials can also be prepared by microtomy. Materials that are a combination of soft and hard substances—either hard particles in a soft matrix or soft particles in a hard matrix—are difficult to polish and difficult to microtome. Ion polishing offers a solution to difficult sample preparation and can be used incrementally to approach inclusions at different depth for characterization by SEM-EDS.

Nanoparticle Research in the 1960s

Eric J. Chatfield, Chatfield Technical Consulting Limited

Although “nanoparticle” is a relatively recent term, measurements of particles significantly smaller than one micrometer were being made before 1940. In 1938, the first electron microscope in North America to achieve a resolution better than that of the optical (light) microscope was built in Toronto. This instrument was applied during World War II to determine the particle size distribution of carbon black, which

was produced in the Toronto area to be incorporated into rubber tires.

In the period from 1960 to 1968, there was concern that any serious incident involving a fast breeder nuclear reactor could result in the dispersal of very fine condensed radioactive particles with unknown properties. The fast reactor at Dounreay, Northern Scotland, contained a eutectic mixture of sodium and potassium metals as the primary coolant, with plutonium as the fissile material. Little was known about the interaction of these elements in a very high temperature and oxidizing environment. Accordingly, a series of experiments was conducted to investigate the type of particles generated when plutonium was explosively vaporized along with alkali metals and several other metals used in the reactor system. The particulate was collected and studied by transmission electron microscopy (TEM) and electron diffraction using a Philips EM 100B transmission electron microscope. Other techniques such as centrifugation, ultrafiltration, optical absorption spectroscopy and electrophoresis were also used to determine particle size and chemical species.

Stereo TEM micrographs showed that the particulate often consisted of three-dimensional aggregated networks of submicrometer particles. It was also found that condensation of a mixture of plutonium metal vapor and the vapor of each of the alkali metals in an oxidizing atmosphere yielded particles of alkali plutonates that were soluble in water. Although these studies were conducted over 40 years ago, they demonstrate that the electron microscopes and analytical methods of that period provided the ability to characterize particulate materials in the size range now referred to by the term “nanoparticles.”

Advantages of Silicon Drift Detectors (SEM/EDS) in Pharmaceutical Applications

R.A. Carlton, GlaxoSmithKline

Silicon Drift Detectors (SDD) have a number of clear advantages over Lithium Drifted Silicon (SiLi) detectors in energy dispersive spectrometry for non-beam sensitive materials. The primary advantage is the high X-ray count rates of SDD compared with SiLi detectors. The majority of the applications have been to non-beam sensitive, high atomic number materials such as metals, ceramics and minerals. Recent studies have demonstrated that SDDs can also be quite useful for beam sensitive materials such as pharmaceuticals. This talk will highlight the advantages of SDD detectors over SiLi detectors for elemental mapping using common, over-the-counter products such as a multi-

vitamin tablet, generic naproxen sodium and aspirin. While the SDD has clear advantages over the SiLi detector, one must be careful with interpretation of results because certain unexpected sum peaks will also be present. Prospects for future developments will also be presented.

Introducing Children to the Micro-Life of Fish Lake

Theodore M. Clarke, Retired Materials Engineer

My friend's family has enjoyed exploring the micro-life of Fish Lake in rural Indiana. We collected our water samples near the shore. The specimens were examined in micro-aquarium slides using a Meiji stereomicroscope with multimode transilluminator and the 2X and 4X paired objectives. The same specimens were then examined with a modified Biolam using the 4X through 40X objectives equipped with water immersion caps and multimode condenser. The girls were most fascinated watching live copepods with the stereomicroscope using transmitted darkfield illumination. Their father was interested in the color bands of birefringent crystals in the alkaline lake water viewed with crossed polarized light and the 4X objectives of the stereomicroscope. The Meiji stereomicroscope with a multimode transilluminator will be demonstrated.

Hot Stage Microscopy 2000-2009, Where Does it Stand?

Tony Havics, pH2, LLC; John A. Smoliga, Boehringer Ingelheim

It has been nearly a century and a half since the first illustration of a hot stage appeared in the scientific literature. Much has come and gone in the way of instrumentation and one wonders what roll the hot stage plays in science today. We will present a survey of the past decade's published literature on the construction and use of the hot stage, focusing on classical polarized light microscopy but also stepping into the next generation with hot stages for electron and atomic force microscopy. Representative stages and their application to liquid crystals, pharmaceuticals, food, polymers, polymorphs and other crystals will be discussed.

Should Microscopists Take Note of the Micro Four Thirds Standard?

Jan Hinsch

In regard to versatility, the digital incarnation of the single lens reflex camera (DSLR) has been without a rival until recently. There were digital cameras with an electronic viewfinder that, like DSLR cameras, displayed parallax-free, real-time images, but they lacked interchangeable optics. With the implementation of



Photo courtesy of Jan Hinsch

Jan Hinsch introduced cameras using the micro four thirds standard (m4/3) such as the Panasonic Lumix (above), and explained why they are becoming good photomicrography alternatives to DSLRs.

the micro four thirds standard (m4/3) in cameras from Olympus and Panasonic, this has changed and there is now an interesting alternative to the DSLR camera. The flange-to-image distance is 20 mm in m4/3 cameras and that makes possible the adaptation of just about any SLR lens, macro bellows and microscope camera attachments of any vintage. The Panasonic Lumix G1 was the first m4/3 camera available and I have owned one and used it extensively for micrographic purposes for nearly a year. This talk will present my experiences with this instrument and add a few general comments on the sensor's pixel count and microscopic resolution.

Evening with Brian: "Censoring the Cell: How the Microscope is Abused by the Media"

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

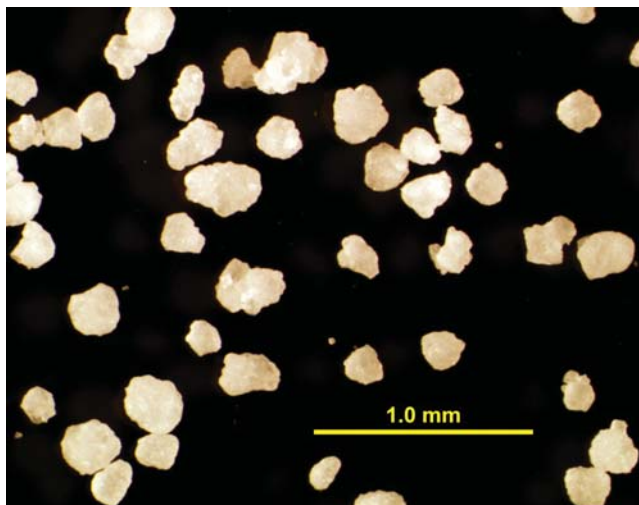


Photo courtesy of Meggan King

Meggan King of McCrone Research Institute revealed the optical crystallographic properties of the artificial sweetener sucralose, which she determined by polarized light microscopy.

In his annual Inter/Micro evening presentation, Brian discusses why television ignores the microscope. Programs cover all the varied life forms, from wildebeests in Africa and sloths in the Amazon to penguins and walruses at the poles — with one exception. Microorganisms are hardly ever seen. Documentary producers won't screen programs on the astonishing world of the microscope, and television commentators seem convinced that the subject will not work on TV. Brian will show us what the viewer is missing.

Tuesday, July 13: Environmental and Industrial Microscopy

The Sugary Sweet Side of Microscopy

Meggan King, McCrone Research Institute

Artificial sweeteners are widely used in today's food industry. Sucralose is but one of the newest forms and is best known for its claim to be made from natural sugar. However, it is manufactured by selective chlorination of sucrose. Because it is several hundred times sweeter it is typically added to foods in minute quantities. Most of the edible crystalline sugars (sucrose, lactose, dextrose, etc.) have already been characterized by polarized light microscopy but the optical crystallographic properties of artificial sucralose have not previously been determined or, at least, are not readily available. A United States Pharmacopeia (USP) reference sample of sucralose was obtained and its optical

crystallographic properties were determined by polarized light microscopy and the spindle stage.

Asbestos and Vintage Cosmetic Talc Products

Randy Boltin, MVA Scientific Consultants

Cosmetic grade talc is often assumed to be derived from a pure ore or a talc ore that has been processed to gain a certain prescribed level of purity. The tendency has been to draw a distinction between so-called pure cosmetic-grade talc and impure industrial grade talc, with the latter containing substantial amounts of impurities that render it unsuitable for use in the cosmetics industry. A survey of vintage cosmetic talc products indicates that, historically, there has been limited delineation based on the purity of materials that end up in the final product.

Regulated asbestos minerals found as natural contamination in talc ore bodies include chrysotile (serpentine asbestos), tremolite-actinolite series amphibole, and anthophyllite. Transitional anthophyllite-talc fibers exhibiting asbestiform habit may also be present. Further complicating the issue is the occasional occurrence of other calcic amphiboles with talc, such as the Libby-type amphibole fibers winchite or richterite.

Use of Malachite Green Stain as an Auxiliary Technique for Differentiating Sepiolite from Chrysotile

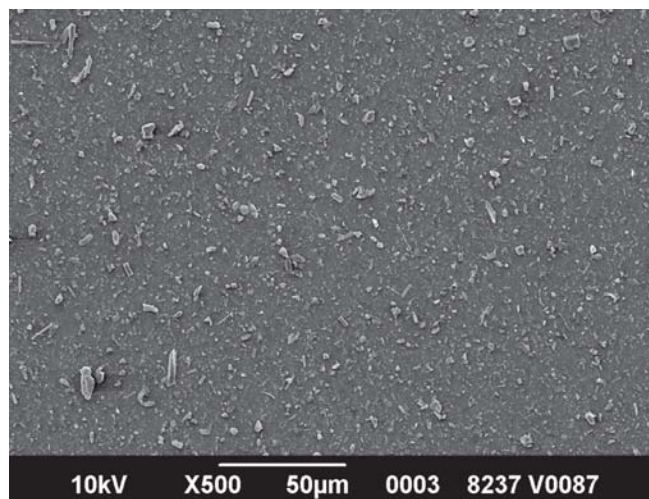
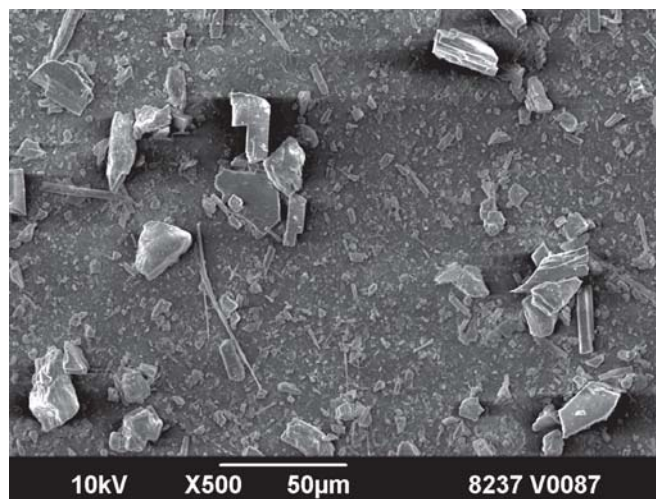
Lou Solebello, International Asbestos Testing Laboratories, Inc.

Staining techniques have been used by microscopists over the years with moderate success for differentiation of clay minerals. E.G. Faust (1940) developed a simple acid-base staining technique using malachite green for a rapid determination of swelling (smectite) versus non-swelling (kaolinite) clays in natural and beneficiated products. Palenik (1979) applied the principles of Faust to forensic microchemical investigations of unknowns. Historical aspects of staining techniques for clay and the application of Faust's procedure as an auxiliary technique for the differentiation of asbestiform sepiolite clay from chrysotile in gaskets will be discussed.

Using Aqueous Elutriation to Isolate Respirable Fractions of Asbestos Fibers from Talc

S.P. Compton, MVA Scientific Consultants

In 2008, a water elutriation method was reported by J. S. Webber et al. as a technique for isolating respirable amphibole fibers. In the report, fractions of the vermiculite contaminant from Libby, Montana were separated from larger fibrous and non-fibrous material. As with vermiculite, talc materials can be contaminated with amphibole fibers. The application of



Photos courtesy of Steve Compton, MVA Scientific Consultants

Steve Compton of MVA Scientific Consultants presented SEM images of a talc sample before (left) and after (right) it was filtered by the water elutriation technique, which he used to isolate asbestos fibers.

the elutriation method to asbestos-containing talc materials will be discussed.

How Microscopic Particles Shut Down the Airways

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

In April 2010 the whole of European air transportation shut down for a week. People near airports could hear birdsong again (above the noise of traffic) and the skies were clear of contrails. The cause was the threat of damage caused by microscopic abrasive particles from a volcano in Iceland. The cost was nearly \$2 billion. Was the scare justified? And what was in the dust?

Ettringite: Not Really a Very Nice Mineral!!

Wayne C. Isphording, University of South Alabama

Present-day utilization of the petrographic microscope in many forensic investigations is often considered less important as an analytical technique than some of the more modern procedures, such as fully automated, quantitative, scanning electron microscopical methods (e.g. QEMSCAN). While few would argue the value of information that can be generated from such systems, there are situations when less-sophisticated methods provide better, less expensive, and faster results. A case in point involves extensive heave-and-swell reactions in subsoils that continue to take place at a major shopping mall in Florida. Prior to construction, the subsoils were treated with a mixture of bed ash and fly ash to promote soil stability. Expansion of the soils, however, has caused cracking of walls, floors, and parking lot surfaces.

The cause of the problem has been traced to a reac-

tion between the soil and ash additive which has generated the mineral ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$). The formation of this mineral, by replacement of gypsum, results in a several-hundred-fold increase in volume. Its presence in the soil as euhedral, elongated, well-formed crystals precludes any possibility of it having been transported to the site as a detrital component. The matted and randomly oriented crystals are clearly visible. Light (optical) and SEM photomicrographs were used to convince the contractor that the mineral is a true, authigenic reaction product. Its easily identifiable form also allows rapid scan of core samples to determine the depth to which remediation measures must be carried out.

Environmental Microscopy at 200 Feet

Richard S. Brown, MVA Scientific Consultants

A unique sampling opportunity presented itself to me: help determine the cause of stained windows — 20 stories high. Sampling strategies, difficulties and challenges will be presented along with the microscopy (PLM, SEM, FTIR and TEM) used to characterize the particles causing the problem.

Investigation of Polarized Light Microscopy Differentiation of Erionite from Other Fibrous Zeolites

Lou Solebello and Craig Liska, International Asbestos Testing Laboratories, Inc.; Gary Tomaino, Minerals Technologies, Inc.

Erionite is a naturally occurring fibrous mineral belonging to the zeolite group. Zeolites are complex framework structure aluminosilicates with large cavities occupied by water molecules and exchangeable ions



Photo courtesy of Eric J. Chatfield

In his talk on "Cellulose Ester Filter Quality Issues," Eric Chatfield compared the three commercially available air sampling cassettes used to monitor worker exposure to asbestos.

lending themselves to physical properties of industrial importance. Erionite and other zeolites are typically found in hydrothermally altered volcanic ash deposits. Documented occurrences of erionite include North Dakota, Arizona, Nevada, Utah, Australia, Canada, France and the Central Anatolian Region of Turkey (CAR). Although currently not regulated, erionite induced malignant mesothelioma prevalence in the CAR has resulted in Group 1 Carcinogen classification of erionite by International Agency for Research on Cancer (IARC). There are more than 100 types of natural and synthetic zeolites. Less than a dozen natural zeolites occur in a fibrous habit, but possess similar optical, structural and chemical properties. A microscopy (PLM, SEM and TEM) comparison of erionite with mesolite, natrolite, thomsonite and mordenite will be discussed.

The Great Mouse Detective

Brendan Nytes, Microtrace, LLC

Customer complaints are a common problem for the food industry. A very common complaint is an alleged mouse or rodent contaminant in a food product. Even though the customer identifies the alleged foreign material as a mouse, it may not be what is received. Microscopy, microchemistry, and other micro-analytical techniques were used to identify the true composition of the material. This talk will explore several case examples of alleged contaminants in food products, including mice.

Cellulose Ester Filter Quality Issues

Eric J. Chatfield, Chatfield Technical Consulting Limited

NIOSH 7400 is the phase contrast microscopy (PCM) method specified for monitoring worker exposure to asbestos and, in some jurisdictions, for post-abatement clearance after asbestos removal. In the past year, it has been noticed that the quality of disposable air sampling cassettes has deteriorated to the point that worker exposure measurements and post-abatement clearances of work sites are being seriously compromised.

There appears to be only three commercial sources for these cassettes. The components of the cassettes from one manufacturer fit so poorly that the adhesive tape around the outside is the only thing that holds them together. These cassettes also have a cellulose support pad under the filter that distorts under routine flow-rate conditions, leading to a stretched filter with a saucer-like profile. Cassettes from the other two available sources are generally satisfactory, but the filter medium used in them is not. From one source, clearing in an acetone vaporizer produces a background with a network of fine fiber-like structures, leading to an unacceptably high blank fiber count. The filter medium used in cassettes from the other source sporadically exhibits the same problem, and often a non-uniform particulate deposit. The filter background problem, which is a consequence of incomplete mixing of polymers during filter manufacture, can be overcome by the use of alternate clearing methods. But this does not overcome the non-uniformity of the deposit.

NIOSH 7402 is specified for transmission electron microscopy (TEM) evaluation of filters, if an unacceptably high fiber concentration observed by PCM is thought to be a consequence of a mixture of asbestos fibers with fibers of unregulated materials. In the original issue of NIOSH 7402, the asbestos fiber concentration was calculated directly from the TEM data, using the number of asbestos fibers counted, the area of filter examined, and the volume of air passed through the filter. This method of calculation was changed in the 1994 revision. In the current method, the asbestos fiber concentration is calculated by multiplication of the PCM count by the ratio, determined by TEM, of the number of asbestos fibers to the total number of fibers. Since the TEM fiber count will not include the background fibers observed by PCM on currently available filters, the asbestos concentration obtained from use of NIOSH 7402 will not be correct.

Analysis of Chinese Drywall that Produced Irritant Health Effects and Corrosion in the Southeast United States

Arthur W. Struss, USG Research (retired)

Chinese drywall was imported in 2004 and 2005 for reconstruction in the Gulf Coast after hurricanes such as Katrina caused heavy destruction. The gypsum drywall exposed to humid weather conditions emitted hydrogen sulfide that produced irritant health effects and copper corrosion that adversely affected electrical appliances, even the loss of air conditioner refrigerant. As of March 2010, the U.S. Consumer Product Safety Commission (CPSC) had received 3,031 incident reports related to drywall; 90% of the reports were from the Southeastern United States. The purpose of this investigation is to determine the source of the sulfide that produced the hydrogen sulfide emissions. The complaint was that Chinese drywall contained higher concentrations of strontium and sulfur than drywall produced in North America. Another possible source of the hydrogen sulfide was sulfur reducing bacteria. The CPSC published a protocol to identify Chinese drywall in residences and has recommended the complete removal of the complaint drywall, corroded components and appliances. Thousands of lawsuits have been filed to recover costs associated with damages in homes ruined by the hydrogen sulfide emitting drywall made in China.

Field Microscopy in Environmental, Safety and Health

Tom Havics, pH2, LLC

Most microscopy is performed in a fixed lab. Despite this, Environmental, Safety and Health projects sometimes require installing a microscope or two on a short-term basis. These projects might involve sample collection and processing, in addition to actual microscopy. The agents of interest might be asbestos, mold, bacteria, suspicious white powders, soot or even unknown "wastes." The challenges of arranging and managing a field microscopy setup include equipment selection, site setting, set up, calibration, personnel training and so forth. An overview of these considerations will be presented with a good dose of real-life examples from projects.

Methods and Benefits of Staining in the Microscopy of Consumer Products

Tom Kremer, Integrated Paper Services

In the manufacture of disposable tissue and sanitary products, a wide variety of additives are used for a wide range of purposes. Additives may add strength, maintain strength when wet, lose strength when wet, provide strength while retaining softness, provide a moisturizer to the skin, hold dissimilar parts together, absorb moisture, or absorb odors, to name just a few.

Aesthetics is as important as performance in such



Tom Kremer of Integrated Paper Services answers questions about his talk on stains that are used to identify the physical properties of additives in consumer products.

products. Consequently, the aforementioned additives are typically not visible to the naked eye or, in many cases, even under the microscope. Therefore, it becomes helpful, even necessary, to render them visible either (light) optically or electron optically. Specific stains have been extremely instrumental in identifying the physical structures of these products in product development and problem solving.

Examples will be shown from a variety of consumer products, such as facial tissue, bath tissue, paper towels, shop towels, baby diapers and feminine products.

Wednesday, July 14: Chemical and Forensic Microscopy

The Micro-Aquarium...Why Don't You Have One?!

Richard S. Brown, MVA Scientific Consultants; Bobby Martin, Martin Microscope Company

Given the state of microscopy in school systems and in the public eye, the micro-aquarium offers all the benefits of a regular size fish aquarium with a mini-



Photos courtesy of Bernadette O'Reilly

In her talk on "Fungi as Forensic Tools," Bernadette O'Reilly explained how morphology is used to identify fungal spores and how these identifications are used to provide information about the environment from where samples are collected.

mum of care and at less cost than a basic salt water aquarium. An example will be assembled during the presentation with guidelines on how to build one for your own home, school or museum.

The Hammer is Back

Peter Diaczuk, John Jay College of Criminal Justice; Gerard Petillo

When the trigger is pulled on a loaded revolver, the firing pin strikes the primer of a cartridge to initiate a sequence of events that culminate with the discharge of a bullet. A phenomenon known as hammer bounce has been noted in the literature as a partial rearward movement of the hammer just after firing, followed by its forward return. Recently, photographs have appeared that show a revolver at the instant of discharge with the hammer at its fully rearward position. This seemingly improbable coincidence prompted this research to be undertaken. In addition to determining the amount of rearward movement of the hammer using high-speed photography, the indentation of the primer was examined with both stereomicroscopy and incident-light comparison microscopy. This was done to determine the ramifications of potential double strikes of the firing pin on the primer and its effect on the analysis of the resulting tool mark.

Fungi as Forensic Tools

Bernadette O'Reilly, Microtrace, LLC

Fungi, as a group, are nearly ubiquitous. They are found in temperate regions, rainforests, deserts, tundra and even Antarctica. Individual species of fungi, however, have more narrow ranges and habitats. Some are associated with specific plant hosts or forest types. Some are commonly found in buildings, while others are rarely found indoors. Most fungi produce spores — some copiously — that remain in the environment as a part of the soil, surface dust or air particulate

matter. These spores have a broad range of morphology that has long been used by mycologists as a feature for fungal identification. This talk discusses the implication of using morphology to identify spores in particulate matter and how these identifications can be used to provide additional information about the environment from where samples are collected.

Microanalysis of Invasive Traumas

P.D. Zoon, Netherlands Forensics Institute; S.B.C.G. Chang, I. Keereweer, R. Pieterman, M.C. de Boer, M. C. Pit, R.R.R. Gerretsen and E.J. Vermeij

Edmond Locard postulated that when two objects have contact, material is transferred. This principle also holds for blunt and sharp force traumas. In a multidisciplinary approach, forensic anthropology and micro-traces experts work together to link observed injuries with a weapon. After excision of the wounded area, the material is macerated and dried at 40 °C. The clean bone material is studied with optical light microscopy for the presence of foreign material, prior to being studied with scanning electron microscopy and EDS in low vacuum mode.

One might argue that most weapons — knives in particular — are made of stainless steel, and the observed micro traces thus are non-specific. However, quite often material present on the weapon, prior to crime, is transferred as well. In other cases the weapon has a coating or a more unique composition. A selection of interesting case studies, in which microanalysis allowed us to link observed micro traces with suspected weapons, will be presented.

A Method for Isolating Trace Evidence from Plastic Explosives Samples

Andrew Bowen, Stoney Forensics, Inc.

Plastic explosives samples often contain large numbers of fine particles adhering to their surfaces.

Analysis of these particles can be useful in forensic investigations involving plastic explosives. Data obtained from particle analysis can be used to develop investigative leads regarding the origin of an unknown explosive or to compare two or more samples to determine whether they share a common origin. A method for isolating fine particles from C4 plastic explosives will be described, and numerous examples of particles recovered by this method in actual casework will be provided.

Microcrystal Tests for the Detection of Cathinone and Cathine in Khat

Kelly M. Brinsko, McCrone Research Institute

The leaves of the khat plant (*Catha edulis*), grown in parts of Africa and the Middle East, are habitually chewed for their stimulating and euphoric effects. The two principal alkaloids in khat responsible for this high are cathinone and cathine, which are chemically similar to the amphetamines. These are controlled substances in the United States, and are classified by the Drug Enforcement Administration as Schedule I and Schedule IV drugs, respectively.

Cathinone (S-2-amino-1-phenyl-1-propanone) is the chief stimulant in the khat plant, but breaks down readily after harvesting into two compounds: cathine (1S, 2S-2-amino-1-phenyl-1-propanol, or (+)-norpseudoephedrine) and cathine's diastereomer, (-)-norephedrine (1R, 2S-2-amino-1-phenyl-1-propanol). When suspected khat plants are seized, cathinone and cathine may be identified via chromatographic methods such as HPLC, GC-MS or GC-IR. However, no microcrystal tests have yet been published for either cathinone or cathine, which is problematic for labs that lack instrumentation. The initial development of a microcrystal test will be presented here, along with extraction protocols, suitable reagents and the differentiation of stereoisomers.

A Dog by Any Name: The Microscopical Identification of the Hair of the Japanese Raccoon Dog (*Nyctereutes procyonoides*)

Jason Beckert, Microtrace, LLC

This presentation will focus on the microscopic hair morphology of the Japanese raccoon dog (*Nyctereutes procyonoides*). The identification of this animal's hair has become increasingly relevant, as its use in the fur trade has grown in recent years. Native to eastern Asia, the animal has been introduced to multiple areas of the former Soviet Union and Europe. The Japanese raccoon dog, a basal canid, is commonly bred in captivity for its fur and goes by many com-



Peter Zoon of the Netherlands Forensic Institute in The Hague presents case studies in which microanalysis was used to determine materials on suspected weapons in violent crimes.

mon names including tanuki, Chinese raccoon, Asiatic raccoon and Finnish raccoon. The acquisition of authentic Japanese raccoon dog hair samples and the ability to differentiate the hair of other species common to the fur trade will be also discussed.

A Microchemical Approach to Meth Labs

Jim Dunlop, Kalamazoo County Sheriff's Department

The rapid and persistent proliferation of clandestine methamphetamine laboratories has been documented across the nation. Analysis of the associated materials often poses vexing problems for analysts examining the precursors, reagents, solvents, acids and catalysts submitted for analysis.

Rather than being tethered to expensive, and in some cases temperamental, modern laboratory equipment, many microchemical tests for these materials can be culled from such texts as *Microchemical Methods* (Chamot and Mason), *Identification of Materials* (Benedetti-Pichler), *Modern Microcrystal Tests for Drugs* (Fulton), *Qualitative Organic Microanalysis* (Schneider), as well as numerous others. Employing these microchemical techniques provides the analyst a timely and economical method to identify nearly all of the components used by today's meth cook.

This presentation will include a brief history of clandestine laboratories with an emphasis on the household materials utilized in the synthesis. This will be followed with detailed information, including photomicrographs, regarding the microchemical reagents and proper application to identify the materials in use.



Robert Carlton of GlaxoSmithKline ponders a question from the audience during his presentation on the "Advantages of Silicon Drift Detectors (SEM/EDS) in Pharmaceutical Applications."

What Was I Thinking?

Mark E. Palenik, Microtrace, LLC

Most of us have stories of having done something that seemed to be a really good idea at the time only to realize, after having done it, that we were lucky to have escaped without injury or worse. We have only ourselves to blame for our actions but have hopefully learned from our mistakes. However, when tragedy does occur, it is often the job of the forensic scientist to examine clues or evidence from the scene. While it is not possible for the scientist to get into someone's head and determine what he was thinking, proper analysis of the evidence can help to determine and understand what did actually occur at the time. Several case studies are examined where actions resulted in what might be described as less than desirable results.

The Microscope and the Royal Society

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

The Royal Society of London may not be the oldest science academy in the world, but it does have the longest continuous history of them all. The microscope is one of the areas of research that the Society has helped to promote since its earliest years, and this talk will review the highlights from the past three and a half centuries.

A Compound Microscope by Harris of London, a Classic of the Pre-achromatic Era

Jan Hinsch

I am privileged to have access to a collection of microscopes that the New York Microscopical Society

has accumulated in the 133 years of its existence. Among these, a late brass Culpeper style instrument caught my particular interest because it represented the state of the art at the end of the era of one-lens objectives. I made it a leisurely project to see what is possible with such modest means and also to appreciate where the invention of the achromatic objective has taken us since.

Forensic Science in the Pharmaceutical Industry – A Microscopy Perspective

Pauline Leary and John A. Reffner, John Jay College

Forensic science is an evolving but critical discipline within the pharmaceutical industry. Since often only a small amount of an active pharmaceutical agent (API) is available for analysis, microscopy and microanalytical techniques are preferred for much of the testing to establish and protect intellectual property and maintain quality assurance throughout a drug product's life cycle within this highly regulated industry. In recent years, the infiltration of counterfeit pharmaceuticals into the legitimate supply chain created a new area of forensic science within the pharmaceutical industry.

The need to differentiate authentic from counterfeit goods in both the laboratory and remotely has become a significant focus for brand owners and generic companies alike. Development of methods to differentiate these items is a challenge to pharmaceutical microscopists. Counterfeit sourcing and detection, though, is only one of many disciplines within forensic science that are widely applied within the pharmaceutical industry. The job title "Forensic Scientist" is becoming more and more common within the pharmaceutical industry. Forensic scientists rely upon microscopy and microanalytical methods and are using these methods to solve and support complex industry problems including intellectual property protection, analysis of, and tracing the source of contaminants and other types of quality assurance testing.

Microscopical Analysis to Facilitate Problem Solving in the Paper Industry

Walter J. Rantanen, Integrated Paper Services, Inc.

Many times in the industrial process problems arise in production. Due to the wide range of variables encountered in the paper industry, it is necessary to isolate the likely cause so that corrective action can be implemented. Examination with the light microscope has been an effective tool to help to solve some of these production problems. Understanding the process and raw materials is also important for effective use of

these techniques. A few case studies from different areas of the paper industry will be discussed.

Applications of Optical Microscopy in the Study of Polymorphism

R.A. Carlton, GlaxoSmithKline

The use of the optical (light) microscope in the study of polymorphism in the pharmaceutical industry will be highlighted by some recent work with carbamazepine and caffeine. Each of these compounds have been studied intensively for many years, yet there is little in the literature on the optical crystallography of the different forms and hydrates. Some additional work in this area will be presented, along with some of the inherent difficulties in obtaining the crystallographic properties of these forms, owing to problems with making good crystals for analysis. An application of hot stage microscopy will be presented for the determination of the transition temperature of the enantiotropic forms of caffeine. This determi-

nation is complicated because each form sublimes near the transition temperature. A hot stage method for determining this temperature will be presented along with a more refined estimate of this temperature value.

McCrone Research Institute 50th Anniversary

Gary J. Laughlin, McCrone Research Institute

Since 1960, the McCrone Research Institute (McRI) has fulfilled its mission to increase the use of the microscope in various fields of science and technology. With its center of operation located in the same South Side neighborhood for more than 50 years (now called the New South Loop of Chicago), McRI is uniquely equipped to conduct all of its various educational, research and publishing activities. This includes hosting and sponsoring the Inter/Micro conference for more than 60 years, production of *The Microscope* journal (now in its 73rd year of continuous publication) and teaching more than 2,687 courses, here and abroad.

Inter/Micro 2011

Chicago, July 11-15

***An international microscopy
conference sponsored by
McCrone Research Institute***



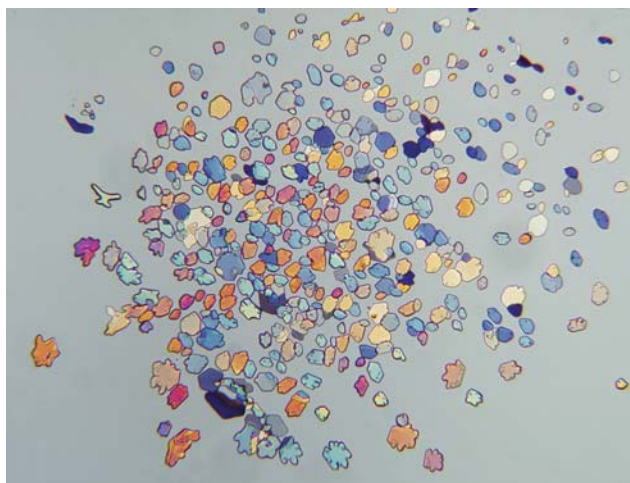
Visit **WWW.MCRI.ORG**
for more information.

Winners of the Inter/Micro 2010 Photomicrography Competition

Three winners were selected in the Inter/Micro 2010 Photomicrography Competition. Kelly Brinsko, Best Overall Photomicrograph; Sebastian Sparenga, Best SEM Photomicrograph; and Ming Zhou, Most Unique Photomicrograph. The winners were announced at the SMSI 2010 Awards Dinner, held at The Berghoff restaurant in Chicago.

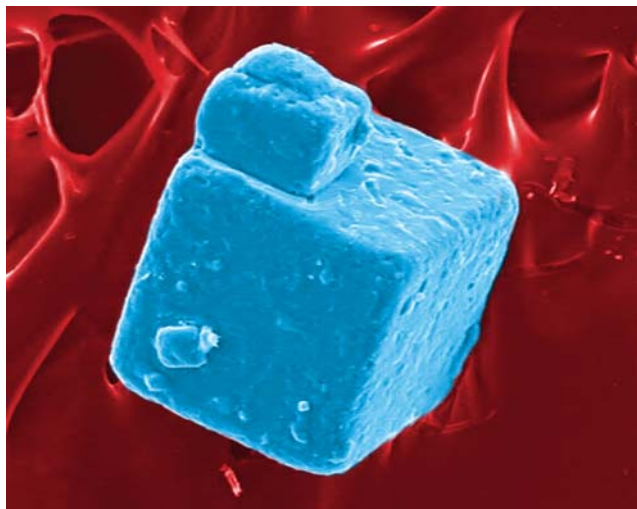
What makes up a winning photomicrograph? Entries were judged by a number of factors, including:

- Even, well-distributed illumination in the field of the image.
- Placement of specimen within the image (centered or artistically offset).
- Brilliance or representative color and contrast.
- Uniqueness or originality of the specimen.
- Information revealed.
- Beauty or impact of the image.
- Subject matter and composition.
- Depth and breadth of focus.



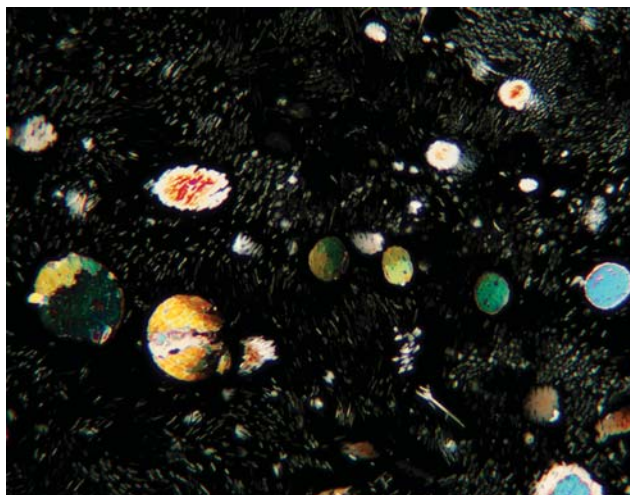
Kelly Brinsko: Best Overall Photomicrograph

A specimen of sublimed anthracene viewed between crossed polars and a quarter-wave plate. The width of the photomicrograph (longest side) is about 100 μm .



Sebastian Sparenga: Best SEM Photomicrograph

An SEM image of a sodium chloride (NaCl) crystal with coloring added in Adobe Photoshop.



Ming Zhou: Most Unique Photomicrograph

The image shows a fusion prep of adipic acid viewed between cross polars at 100X magnification. It was melted twice to achieve a colorful Solar System effect.