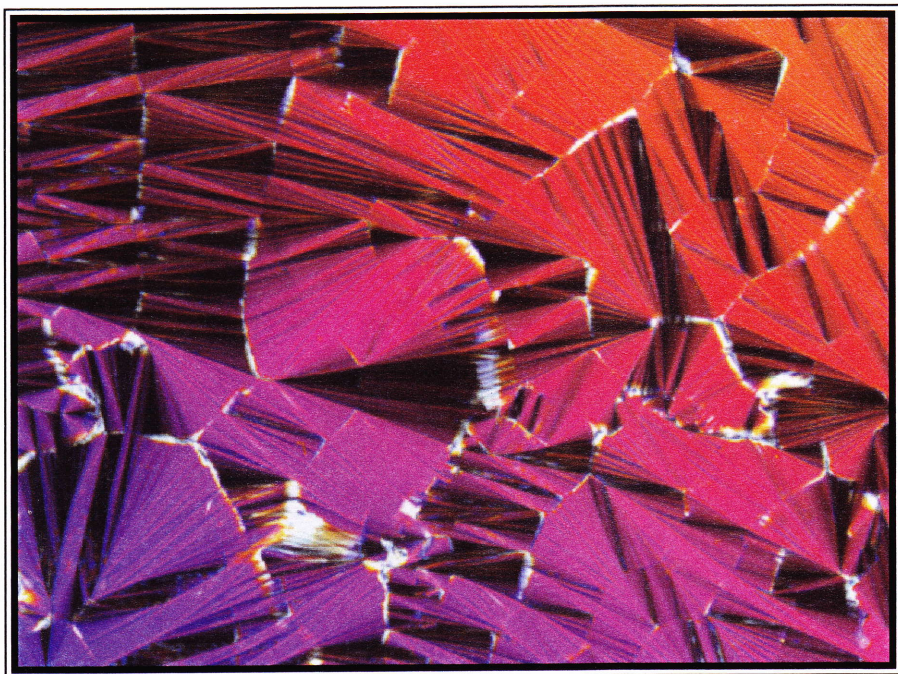


# INTER/MICRO 2012

A Microscopy Symposium  
July 9 - July 13



*Sponsored and hosted by*  
McCrone Research Institute  
2820 S. Michigan Avenue  
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# INTER/MICRO 2012

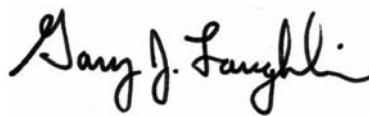
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## ***Welcome!***

Since its beginning in 1948, Inter/Micro, the premier International/Microscopy conference, has grown to attract Microscopists from all areas of light and electron microscopy. This meeting is now held every year in Chicago and continues to be sponsored and hosted by McCrone Research Institute.

The first Microscopy Symposium on Electron and Light Microscopy was developed by Walter C. McCrone (light microscopist in chemistry) and Charles Tufts (electron microscopist in physics) and was held June 10–12, 1948, at the Stevens Hotel, now the Hilton Chicago. The Inter/Micro symposia are believed to be the very first meetings to gather top people in light and electron microscopy together to discuss very small particles including the range of ultrafine particles that are commonly referred to today as “nanoparticles.”

Dr. McCrone’s personal satisfaction to these symposia came from having the world’s best microscopists come to Chicago to further his education! Thank you for your support of Inter/Micro so that we can all continue to further our education.



Gary J. Laughlin  
Chairman, Inter/Micro

## **Cover image by Thomas Hopen**

“Intal Liquid Crystalline Phase” shows a preparation of Intal (cromolyn sodium oral inhalation) in a droplet of water with a cover slip, viewed under crossed polars at 100X magnification. Voted Best Overall Winner of the 2011 Inter/Micro Photomicrography Competition.

## **MONDAY, JULY 9**

### **TECHNIQUES AND INSTRUMENTATION**

*8:00 a.m. – 5:00 p.m. Registration and packet pickup, McRI Front Desk*

*9:00 a.m. – 12:00 p.m. Morning Session, McRI Lecture Room  
Chair: Rich Brown, MVA Scientific Consultants*

#### **Microscopy: Art, Literature, Culture and Science**

Tony Havics, pH2, LLC

#### **Teaching the Teachers: Why is the Microscope Not Utilized in Your Science Class?**

Rich Brown, MVA Scientific Consultants

#### **Rectangular Field Diaphragm**

Thomas J. Hopen, Bureau of Alcohol, Tobacco, Firearms, and Explosives

#### **Mapping Elemental and Refractive Index Variation in Container Glass**

Stephanie Sliwa, Microtrace LLC

#### **An Assessment of GC-MS Ion Preparation Techniques as a Means of Increasing Sensitivity in the Detection of Ignitable Liquids Collected on Charcoal Strips Used in Arson Investigations**

Mark E. Palenik, Microtrace LLC

#### **As Much Fun as Watching Paint Dry: A Microscopical Perspective of Airbrushed Paint Droplets**

Martin Kocanda, Electrical Engineering, Northern Illinois University

#### **Photoconversion of Fluorescent Dyes Under Commonly Used Conditions Can Cause Serious Experimental Error**

A. Weiss, Faculty of Engineering, Bar Ilan University

**Stars, Cubes, Dog Bones and Rods: Morphology Control and Characterization of Iron Oxide and Iron-Manganese Oxide Nanoparticles**

Cristina H. Hartman, Rice University (currently an ORISE Postdoctoral Fellow at the FBI)

*12:00 – 1:30 p.m. Lunch Break*

*Complimentary lunch will be served McRI garden*

*2:00 – 5:00 p.m. Afternoon Session, McRI Lecture Room*

*Chair: Pauline Leary, Smiths Detection and the Graduate Center, CUNY*

**Characterization of Multilayer Foil Laminate Barrier Packaging Using a Combination of Scanning Electron Microscopy and Confocal Raman Microscopy**

Rich Brown, MVA Scientific Consultants

**Microscopical Dichroism of Small Particles**

Dale K. Purcell, The Graduate Center and John Jay College of CUNY

**Device and Kit for Pressing, Rubbing, and Smearing While Observing Microscopic Preparations**

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**DuoScan: The Answer to Sample Burning**

Michael Oweimrin, HORIBA Instruments Incorporated

**3-D Images with the Scanning Electron Microscope**

James R. Millette, MVA Scientific Consultants

**“Four Score and Seven Years Ago” or Was It?: Authenticating President Abraham Lincoln’s Signature**

Jennifer Herb, Microtrace LLC

**The Watercolors of Charles M. Russell: An Examination of the Artist’s Materials and Techniques on the Montana Frontier**

Jodie Utter, Amon Carter Museum of American Art

**Now in 3-D: Adding Another Dimension to Chemical  
Microscopy**

Kevin Brady, Tredegar Film Products

**Microscopy in the Environmental Laboratory**

Dickey Huntamer, Machester Environmental Laboratory

**Microscopy and Microspectroscopy in the Mobile Analytical  
Laboratory**

Pauline Leary, Smiths Detection and the Graduate Center,  
CUNY

**EVENING WITH BRIAN  
“When Living Cells Make Humans Explode”**

*5:30 – 7:00 p.m.*

***Dinner, McRI Garden***

*Cost: \$28. (See the front desk to pay for the dinner if you  
did not pre-register)*

*7:00 – 8:00 p.m.*

***Evening with Brian, McRI Lecture Room***

*Free*

There have been many theories put forward to explain this dramatic phenomenon. The only one to be widely accepted is the “wick effect,” in which fat melts into clothing and burns like wax in a candle. The continuous burning liquefies more body fat, setting up a continuous combustion. This theory fits few of the facts, and it would take at least twelve hours to work — spontaneous combustion consumes a person in a matter of minutes. This evening, we will discover a revolutionary new theory based on the metabolism of cells within the body. Spontaneous human combustion can be seen as a fleetingly rare, extremely dangerous, but potentially avoidable, condition.

**TUESDAY, JULY 10**  
**ENVIRONMENTAL AND INDUSTRIAL**  
**MICROSCOPY**

*8:00 a.m.—5:00 p.m. Registration and packet pickup, McRI Front Desk*

*9:00 a.m.—12:00 p.m. Morning Session, McRI Lecture Room*  
*Chair: Andrew Bowen, Stoney Forensic, Inc.*

**Pigmented Corrosion**

Brendan Nytes, Microtrace LLC

**Three Cases of Pipe Failure with Substantial Corrosion**

Tony Havics, pH2, LLC

**Characterization of Nanoparticles in Soil and Road Dust**  
**Using Scanning Electron Microscopy**

Jani Tuoriniemi, Chemistry and Molecular Biology, University of Gothenburg

**Paper Product Recycled-Fiber Claims and the Comparisons with a Fiber Analysis**

Walter J. Rantanen, Integrated Paper Services, Inc.

**Wood Without Annular Growth Rings**

John A. Reffner, John Jay College of Criminal Justice, CUNY

**Mold Damages: Microscopy Analysis by Tape Lift Versus Determination of Enzymatic Reactions and Evaluation of Analysis Methods**

Marianne Berdal, Mycoteam AS

**An Overview of the ASTM Method for Quantification of Air-borne Fungal Structures In and Inertial Impaction Sample by Optical Microscopy**

Tony Havics, pH2, LLC

**Thermite in the World Trade Center Dust?**

James R. Millette, MVA Scientific Consultants

*12:00 – 1:30 p.m. Lunch Break*

*Complimentary lunch, McRI Garden*

*2:00 – 5:00 p.m. Afternoon Session, McRI Lecture Room*

*Chair: James R. Millette, MVA Scientific Consultants*

**Scientific Working Group on Geological Materials (SWGGE0)**

Chris E. Taylor, U.S. Army Criminal Investigation Laboratory

**What the Cells in a Dinosaur's Tail Tell Us About Dinosaur's Evolution**

Brian J. Ford, Caius College, University of Cambridge

**Microscopical Analysis of Dust: Information Taken Out of Thin Air**

Marianne Berdal, Mycoteam AS

**Trace Mineral Solutions to a Sediment Trespass Controversy**

Wayne C. Isphording, Tulane University

**Calculated Average Ranges of Refractive Indices for the Amphiboles Found in the Rainy Creek Igneous Complex Near Libby, Montana, USA**

Matthew S. Sanchez, RJ Lee Group

**Limit of Detection Issues in Measurement of Low Concentrations of Tremolite in Mineral Products**

Eric J. Chatfield, Chatfield Technical Consulting Limited

**Mineral Identification Using Electron Backscatter Diffraction from Unpolished Specimens: Nothing is Easy; Some Things are Just Less Difficult**

Bryan Bandli, Geological Sciences, University of Minnesota

**Polarized Light Microscopy (PLM) Analysis of Cosmetic Talc for Asbestos Content**

Lou Solebello, International Asbestos Testing Laboratories, Inc.

**Compositional Analysis and Morphological Relationships of  
Minerals Found in Talc Tailings and Products from Near  
Talcville, New York, USA**

Brittani D. McNamee, Geological Sciences, University of Idaho

**WINE AND CHEESE RECEPTION WITH EXHIBITORS**

*5:00 – 6:00 p.m.*

*McRI Exhibit Room*

*Free*

Meet representatives from Leica Microsystems, Foster and Freeman USA, Olympus America, Inc., Campbell Center, State Microscopical Society of Illinois, and Microscope Publications.



**WEDNESDAY, JULY 11**  
**CHEMICAL AND FORENSIC MICROSCOPY**

*8:00 a.m. – 5:00 p.m. Registration and packet pick up, McRI Front Desk*

*9:00 a.m. – 12:00 p.m. Morning Session, McRI Lecture Room  
Chair: Arthur Young, Guardian Forensic Sciences*

**Developing a Microcrystal Test for a “Legal High” Drug**

Samantha Huntsman, McCrone Research Institute

**The Forensic Analysis of Paint Evidence Using Micro-Raman Spectroscopy Part I: Development of Libraries and Application Methods**

Christopher S. Palenik, Microtrace LLC

**The Forensic Analysis of Paint Evidence Using Micro-Raman Spectroscopy Part II: Case Examples**

Patrick Buzzini, Forensic & Investigative Science Program, West Virginia University

**Benefits of Using Cross-Sectioning in Forensic Analysis of Automotive Paints**

Ethan Groves, Microtrace LLC

**American Society of Trace Evidence Examiners (ASTEE)**

Chris E. Taylor, ASTEE

**Environmental Effects on Fired Cartridge Cases: Primer Shear and Breech Face Marks**

Peter Diaczuk, John Jay College of Criminal Justice, CUNY

**Role of Edmond Locard in the History of Forensic Science: “Every Contact Leaves a Trace” Dissected**

Patrick Buzzini, Forensic & Investigative Science Program, West Virginia University

**Microspectrophotometry (MSP) of Blood**

Larry Peterson, U.S. Army Criminal Investigation Laboratory

*12:00 p.m. – 1:30 p.m. Lunch Break*

*Complimentary lunch, McRI Garden*

*2:00 p.m. – 5:00 p.m. Afternoon Session, McRI Lecture Room*

*Chair: Jason Beckert, Microtrace LLC*

**Antibody-Based Forensic Body Fluid Identification**

Karl Reich, Independent Forensics

**Analysis Paralysis**

Brian J. Ford, Caius College, University of Cambridge

**Is it Blood?: Recovery and Microchemical Identification of Blood in Forensic Specimens**

Katie White, Microtrace LLC

**One Century Later: The Takayama Hemochromogen Microcrystal Test**

Arthur Young, Guardian Forensic Sciences

**The Alchemist's Tale**

Skip Palenik, Microtrace LLC

**Trace Evidence Analysis at the U.S. Postal Inspection Service National Forensic Laboratory**

Andrew Bowen, U.S. Postal Inspection Service

**Development of a Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals**

Sebastian Sparenga, McCrone Research Institute

**Examining the Effects of Environmental Degradation on the Optical Properties of Manufactured Fibers of Natural Origin**

Kelly M. Brinsko, McCrone Research Institute

**Whose Hair is it Anyway?: Hair and Fiber Microscopy Uncovers Fakes, Frauds, and the Occasional Genuine Article**

Jason Beckert, Microtrace LLC

**WEDNESDAY, JULY 11**  
**STATE MICROSCOPICAL SOCIETY OF ILLINOIS**  
**2012 AWARD DINNER**

**Èmile Chamot Award Recipient: Mickey E. Gunter**

Join McCrone Research Institute and the State Microscopical Society of Illinois on Wednesday, July 11, 2012, as they honor Mickey E. Gunter, the 2012 recipient of the Èmile Chamot award. Gunter is professor of mineralogy and chair of the Department of Geological Sciences at the University of Idaho in Moscow, Idaho.



*The Berghoff, 17 W. Adams Street, Chicago*

*Social hour: 6:30 – 7:30 p.m.*

*Dinner: 7:30 – 8:30 p.m.*

*Award announcement and speeches: 8:30 – 9:45 p.m.*

## **THURSDAY AND FRIDAY, JULY 12 AND 13**

### **WORKSHOP: MINERALOGY FOR MICROSCOPISTS**

*9:00 a.m. – 5:00 p.m. both days*

Taught by Mickey E. Gunter, Department of Geological Sciences,  
University of Idaho

In this two-day workshop, participants will learn about these areas by using specific mineralogical examples based on subtle similarities and differences between mineral structures and compositions as observed with different microscopical methods. Examples include 1) why framework silicates (quartz, feldspars, zeolites, etc.), as seen with polarized light microscopy, exhibit low retardation, while sheet silicates exhibit low retardation in one plane; 2) the differences and similarities of calculated selected area electron diffraction patterns for common silicate minerals; and 3) why it can be difficult to identify particles of similar composition with EDS spectra.

The workshop will also compare and contrast the nomenclature schemes for amphiboles and zeolites, the former being based on precise compositional data, while the later are based first on structural data and less so on compositional data.

*Mickey E. Gunter is a professor of mineralogy and chair of the Department of Geological Sciences at the University of Idaho in Moscow, Idaho. He is also a James Marsh Professor-at-Large at the University of Vermont in Burlington, Vermont. His research interests are in optical mineralogy, use of zeolites in remediation of nuclear waste and health effects of inhaled mineral dusts. Gunter has published more than 90 scientific papers, made presentations at over 50 professional meetings, given more than 70 public seminars, and received over 50 research grants. He is the co-author (with M. Darby Dyar) of Mineralogy and Optical Mineralogy, a new college-level mineralogy and optical mineralogy textbook with an interactive DVD. He earned a B.S. in geology with a minor in mathematics from Southern Illinois University, and an M.S. and Ph.D. in geological sciences from Virginia Tech in Blacksburg, Virginia. Gunter will receive the 2012 Émile Chamot Award from the State Microscopical Society of Illinois at Inter/Micro 2012.*

**PRESENTATION ABSTRACTS**  
**MONDAY, JULY 9**  
**TECHNIQUES AND INSTRUMENTATION**

**Microscopy: Art, Literature, Culture and Science**

Tony Havics, pH2, LLC

A number of photomicrography contests have developed over the years. These have been focused on either the technical or artistic aspects of microscopy. But beyond this simplistic, yet revered, attribute of microscopy, one sometimes ponders the connections and correlations of art, literature, science, and microscopy. In this vein of thought, I wish to explore 1) the inventions of “Art” of Robert Hooke, 2) the painting technique in the 17<sup>th</sup> century and the possible use of the camera obscura as a tool, 3) how Gulliver becomes a metaphorical microscopist in Lilliput, 4) the great debate over the microscope that took place in the Paris Academy of Medicine in 1854–55, which could be better termed the controversy over cancer and the microscope, 5) epistemological advancement, 6) the use of the microscope in science and culture via education, 7) the symbolism of the microscope for science, 8) the re-analysis of culture via the microscope, and 9) how microscopy still drives analytical instrument changes. Not a “micro-topic” and yet a micron of time available.

**Teaching the Teachers: Why is the Microscope Not Utilized in Your Science Class?**

Rich Brown, MVA Scientific Consultants

The Georgia Microscopical Society (GMS) has been teaching students for many years and the course is always overbooked, thanks to the dedicated GMS member volunteers that freely give their time (9 Saturday mornings, several evenings of preparation), expertise, and energy. I have taken a slightly different tack in holding one-day workshops that introduce the

light microscope to high school science teachers. The teachers are actually paid a small stipend to attend and the workshops are held at Gwinnett Tech's new Bio Science building. The day is spent discussing what types of problems are solved by microscopy, setting up microscope alignment for proper illumination, and examining slides prepared by GMS. Each attendee keeps their slide kit and, hopefully, remains enthusiastic about microscopy all the way into their classroom!

### **Rectangular Field Diaphragm**

Thomas J. Hopen, Bureau of Alcohol, Tobacco, Firearms, and Explosives

As I have stated in the past, the greatest aspect of INTER/MICRO to me, as compared to other professional meetings I attend, is the wealth of information that the technical presentations provide and the conversations one has with other attendees. One of the many tidbits of information that I picked up a few years back was a comment by Jan Hinsch during his presentation. Jan said that a rectangular field diaphragm is useful when looking at non-equant samples that appear to be opaque, such as heavily pigmented fibers. So when I got back from the meeting, I searched around and found an old rectangular aperture that belonged to a retired Nic-Plan IR microscope. I examined a dark Mongoloid head hair using the variable rectangular aperture by placing it on top of the microscope base and bringing the edges into focus with the condenser. It worked great! The pigmented area of the hair cortex was now visible and I could discern ovoid bodies, pigment size, pigment clumping, and the medulla. I was so impressed that I wrote a quick "Tricks of the Trade" article for *The Microscope* journal, in an effort to spread the word. This presentation will expand on that short "Tricks of the Trade" article and demonstrate the value of looking at pigmented fibers and hairs as well as other unusual samples such as cross-sections of pigmented paints and tapes.

### **Mapping Elemental and Refractive Index Variation in Container Glass**

Stephanie Sliwa, Microtrace LLC  
Ethan Groves, Microtrace LLC  
Mark E. Palenik, Microtrace LLC  
Christopher S. Palenik, Microtrace LLC

While it is known that refractive index within a glass container can show more variation than a similar quantity of sheet glass, this variation has never been studied in relationship to trace element variation. Here we study half of a wine bottle that has been cut into 96 pieces. Each piece has been characterized in 3 areas by micro-X-Ray Fluorescence ( $\mu$ -XRF) and by refractive index, which was measured to  $\pm 1 \times 10^{-5}$ . The resulting analytical data were plotted over the surface of the wine bottle to create high-resolution maps of trace element and refractive index variation. The refractive index has been shown to vary by as much as  $2 \times 10^{-4}$  over the wine bottle half studied. The trace element data shows similarly large variations relative to the variations typical of sheet glass. The results have implications for conducting comparisons between questioned and known glasses, particularly for sampling and characterization of known container glass samples.

### **An Assessment of GC-MS Ion Preparation Techniques as a Means of Increasing Sensitivity in the Detection of Ignitable Liquids Collected on Charcoal Strips Used in Arson Investigations**

Mark E. Palenik, Microtrace LLC

Solid phase microextraction (SPME) and activated charcoal strips (ACS) are the most sensitive passive means of sampling for, and detecting, ignitable liquids when used in conjunction with analysis by gas chromatography with mass spectrometry (GC-MS) in arson investigations. SPME does, however, have an advantage over ACS in terms of sensitivity. While the total adsorptive area of a SPME fiber is significantly less than that of

an ACS, limiting the total amount of sample collected on the fiber, desorption of the analytes takes place in the injector of the GC, eliminating the need for a solvent. Sensitivity reducing solvent effects are eliminated and the low mass of the fiber allows the analytes to desorb quickly, eliminating tailing of early peaks. As a SPME fiber becomes saturated, it can replace lower molecular weight analytes with higher molecular weight in the sample. ACS cannot be desorbed directly into the GC, and a solvent, usually carbon disulfide, is needed. Dilution of the analytes with a solvent reduces sensitivity, and solvent background can obscure low levels of early eluting analytes such as benzene and isooctane as well as others. ACS do, however, have a large adsorption area, and lighter compounds will not be replaced by those of high molecular weight. Ion preparation techniques like tandem mass spectrometry (ms/ms) and selected ion storage (SIS) can eliminate interfering ions that add to background and reduced sensitivity. These techniques are evaluated for the purpose of increasing the sensitivity for ignitable liquids collected on ACS.

### **As Much Fun as Watching Paint Dry: A Microscopical Perspective of Airbrushed Paint Droplets**

Martin Kocanda, Electrical Engineering, Northern Illinois University

Airbrushing has long been employed as a technique to apply a thin film of liquid media or colorants to a plethora of solid surfaces. Airbrush techniques have also been employed to enhance or adulterate photographic images. Facial blemishes and disfigurements of model subjects have been easily masked by carefully selecting the desired shade and consistency of paints prior to submitting artwork for publication. Brushed oil paint on canvas have been known to be adulterated in several instances thus suggesting that airbrush techniques could easily render undetectable modifications to many paintings of historical value.



The motivation of this work was to examine droplet size and the physical properties of paint thin-films applied using typical airbrush techniques. Physical properties, such as viscosity, surface tension, pigment particle size, and pigment charge, present unique constraints. These parameters affect the paint droplets during time of flight, during adsorption on the substrate surface, and during the drying process. In this work, the effects of several parameters are examined using light microscopy and SEM imaging where paint droplets are adsorbed on glass slides.

### **Photoconversion of Fluorescent Dyes Under Commonly Used Conditions Can Cause Serious Experimental Error**

A. Weiss, Faculty of Engineering, Bar Ilan University

Marina Piterburg, Mina and Everard Goodman Faculty of Life Sciences, Bar Ilan University

Hana Panet, Faculty of Engineering, Bar Ilan University

Alik Belitzky, The Institute of Chemistry, The Hebrew University of Jerusalem

Uri Raviv, The Institute of Chemistry, The Hebrew University of Jerusalem

One of the important features of fluorescence microscopy is its ability to separate multiple labels based on their spectral characteristics. It is usually assumed that the spectral characteristics of the dyes remain constant during observation. Here, we present two examples involving commonly used dyes where this assumption does not hold. We also discuss the imaging and analysis methods that enable quantitation of this phenomenon.

DAPI (4'-6-Diamidino-2-phenylindole) is a fluorescent dye commonly used to visualize DNA or cell nuclei in fixed cell preparations. DAPI is often used together with fluorescein or GFP, which can be excited without exciting DAPI. We found that following observation of DAPI using UV or violet excitation, it became sensitive to the blue/cyan excitation used in fluorescein/GFP filter cubes. This has serious implications for the use of

DAPI together with widely used green fluorophores.

Tetramethylrhodamine (TMR) is commonly used as a FRET acceptor together with fluorescein as donor. Increased fluorescein emission upon photobleaching of TMR can be used to detect FRET. We found that the excitation/emission spectra of TMR shift to higher energy upon photobleaching. This will produce a false positive for FRET when using the acceptor photobleaching method for detection of FRET in fluorescein/TMR FRET pairs.

### **Stars, Cubes, Dog Bones and Rods: Morphology Control and Characterization of Iron Oxide and Iron-Manganese Oxide Nanoparticles**

Cristina H. Hartman, Rice University (currently an ORISE Postdoctoral Fellow at the FBI)

Kenton H. Whitmire, Rice University

New nanoparticle shapes of  $\text{Fe}_x\text{O}$  (where  $0.8 < x < 1$ ; Wüstite) and  $\text{Fe}_{1-y}\text{Mn}_y\text{O}$  (where  $0 < y < 1$ ) were synthesized by decomposition of the corresponding metal formates in tri-n-octylamine/oleic acid mixtures, at elevated temperatures (ca. 370 °C), under an inert atmosphere. The morphology of the nanoparticles was modified as a function of the reaction parameters. Solid solutions of iron-manganese oxide nanoparticles were obtained by mixing the metal formate precursors, and nanoparticle shape was tuned by adjusting the iron to manganese ratio. Iron-manganese oxide nanoparticles with ca. 1:1 metal ratios in the crystal lattice displayed a morphology that can be considered a shape in between that of iron oxide nanoparticles and the shapes previously reported for manganese oxide (Manganosite). Crystalline phases were determined by using Single Area Electron Diffraction (SAED) and X-ray Diffraction (XRD). Nanoparticle shape and crystalline growth were determined using Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), and Atomic Force Microscopy (AFM). Details on the characterization

of the nanoparticles will be discussed in detail.

### **Characterization of Multilayer Foil Laminate Barrier Packaging Using a Combination of Scanning Electron Microscopy and Confocal Raman Microscopy**

Rich Brown, MVA Scientific Consultants

Multilayer foil laminate packaging was examined using a combination of FTIR, SEM-EDS and confocal Raman microscopy. The larger layers of polymer were easily characterized by FTIR microscopy after cross-sectioning the laminate by hand with a razor. The thinner bonding layers could not be observed until a sample of the laminate was cross-sectioned using an ion beam cross-section polisher. Imaging of the entire layer structure was then possible by reflected darkfield light microscopy and scanning electron microscopy. The analysis of the thin bonding layers was performed using confocal Raman microscopy. Chemical maps of the layer structure and sequence were also possible by automated confocal Raman microscopy.

### **Microscopical Dichroism of Small Particles**

Dale K. Purcell, The Graduate Center and John Jay College of Criminal Justice, CUNY

John A. Reffner, John Jay College of Criminal Justice, CUNY

Thomas A. Kubic, John Jay College of Criminal Justice, CUNY

Microscope photometry is recognized internationally by various technical and scientific working groups as a valuable instrumental analysis technique to spectroscopically compare color through a quantitative and objective method. Dichroism is the selective absorption of plane-polarized light between two orthogonal directions by an anisotropic material. Ordinarily the absorption of light in a dyed fiber, for example, is dependent upon a sample's thickness and concentration, in contrast, the ratio of absorbencies along the two principle directions is a function of orientation only and is independent of sample

thickness and concentration. The current research demonstrates the significance of how dichroism analysis may increase the classification and discrimination power of color analysis using microphotometry.

### **Device and Kit for Pressing, Rubbing, and Smearing While Observing Microscopic Preparations**

Bill Neuberg, Shamrock Technologies, Inc.

The best device for handling particles under the microscope may still be the hand-held needle. Some applications require a micromanipulator. Walter C. McCrone presented a sleeve, clamped onto an objective. The diamond anvil cell has been useful for geologists and others working with very high pressures. I have developed a simple device that is smaller than a hot stage. A threaded cylinder fits around an objective and holds the tip of a ball-point pen, with ball removed. I look thru this while pressing. Modifications using optical windows with appropriate holders will be discussed. I will show how these tools were developed and how they can be used without long working distance objectives.

### **DuoScan: The Answer to Sample Burning**

Michael Oweimrin, HORIBA Instruments Incorporated

HORIBA Scientific introduced the DuoScan in 2006 as a simple answer for both micro and macro mapping as well as the simple answer to sample burning. Using a patented method of rastering mirrors over the field of view of the objective, the DuoScan illuminates an area as large as the field of view of the objective, while at the same time distributing the laser power safely and effectively across the entire area without burning. This has proven quite effective with biological, explosive, toxic, thin films, and many other types of samples. You will learn about this new technology and how it has transformed the way Raman imaging is being done today!

### **3-D Images with the Scanning Electron Microscope**

James R. Millette, MVA Scientific Consultants

Laren Cyr, MVA Scientific Consultants

Vern Robertson, JEOL USA, Inc.

Because Scanning Electron Microscope (SEM) images have considerable depth of field, they may appear 3 dimensional (3-D), however, they are still just 2-dimensional representations of an object. 'Anaglyph 3-D' is the term used for the stereoscopic 3-D effect achieved by means of displaying each eye's image using filters of different (usually chromatically opposite) colors to create a stereo pair, an image is taken twice, at slightly different orientations, and then viewed simultaneously through special glasses that allow each eye to see the differences in the overlaid images. In this presentation, we will use images and glasses that are red/cyan (cyan being the absence of red) to illustrate several aspects of 3-D. We tilted the samples from angles ranging from 2° to 12° (more tilt for features with less relief, less tilt for features with more relief). For those who wish to make their own 3-D images, it is important that you can see all of the features of interest from both vantage points. For qualitative images, it is correct to refocus with the objective lens control. If you want to make quantitative measurements, you will need to refocus with the mechanical Z adjustment only.

### **"Four Score and Seven Years Ago" or Was It? : Authenticating President Abraham Lincoln's Signature**

Jennifer Herb, Microtrace LLC

Christopher S. Palenik, Microtrace LLC

Skip Palenik, Microtrace LLC

Given the value of historical documents and memorabilia, forgeries are a common occurrence. As more fraudulent documents of extremely high quality are produced, the physical and chemical characterization of materials (i.e., paper and writing ink) is becoming increasingly relevant for authentication

This talk explores the authentication of a letter purportedly signed by President Abraham Lincoln. The examination focused on the comparison of ink in the signature to ink in the body of the letter (wherein lies the value of the document). An initial examination of the “Lincoln Letter”, viewed under different wavelengths of light using a Visual Spectral Comparator (VSC), revealed no significant differences between the signature and body. Additional analyses by energy dispersive X-ray spectroscopy (EDS), micro-X-ray fluorescence ( $\mu$ -XRF), Fourier transform infrared micro-spectroscopy (micro-FTIR), and Raman microspectroscopy (micro-Raman) were completed to characterize the inks. The analyses determined that the inks on the letter are composed of iron gall. The letter inks were then compared to various iron gall ink formulations found in literature references that were synthesized in our lab. This study of multiple iron gall inks shows the variation between different inks formulations and the possibility of using their formulation as a method for authentication.

### **The Watercolors of Charles M. Russell: An Examination of the Artist’s Materials and Techniques on the Montana Frontier**

Jodie Utter, Amon Carter Museum of American Art

Analysis of Charles M. Russell’s (active ca. 1880–1926) watercolor materials and techniques were undertaken using magnification, polarized light microscopy (PLM), X-ray fluorescence (Tracer III XRF), infrared photography (IR), and ultraviolet (UV) radiation. Russell’s pigments, used over the course of his career, were identified as were shifts in his technique. Pigment samples were collected from Russell’s studio materials housed at the C. M. Russell Museum, the Britzman collection at the Gilcrease Museum, and the National Cowboy & Western Heritage Museum. Russell’s technique was studied after looking at 26 of his watercolor paintings. The paintings were chosen to represent all phases of the artist’s

career and ability. Traditional and unconventional techniques were noted, as well as shifts in the utilization of underdrawing. In addition, focus was given to the wide variety of high-quality artists' materials available on the Montana frontier in the late nineteenth and early twentieth centuries.

### **Now in 3-D: Adding Another Dimension to Chemical Microscopy**

Kevin Brady, Tredegar Film Products

Recent advances in surface roughness characterization provide new opportunities and challenges to observation of crystal formations. Using interferometry, a microscopist can gain new insight into microscopic structures. In this presentation, interesting 3-D images of various microstructures will be explored, including some crystals produced using the Chamot and Mason, Handbook of Chemical Microscopy.

### **Particulate Identification in the Environmental Laboratory**

Dickey D, Huntamer, Manchester Environmental Laboratory

Normally the environmental laboratory analyzes toxic chemicals in water and sediment samples using a variety of modern analytical techniques. This works fine in most situations but, occasionally, a sample arrives which is not amenable to the conventional EPA methods and other means of analysis are required to characterize the sample. The techniques employed come from forensic trace analysis and include Polarized Light Microscopy (PLM) and microchemical testing. Usually the questions being asked are what is it, and where did it come from?

Several cases will be discussed that involve fugitive dust sourcing, well contamination in drinking water, unusually high particulate loads on PM10 air filters, and unusual events in water treatment plants.

### **Microscopy and Microspectroscopy in the Mobile Analytical Laboratory**

Pauline Leary, Smiths Detection and the Graduate Center,  
CUNY

Keith Casserly, New York CST

John A. Reffner, John Jay College of Criminal Justice, CUNY

Analysis of samples in the field is a significant challenge for many military operations. Testing performed in these settings typically use field-portable instruments designed for use by non-scientists. Although these instruments may provide quick, reliable results, their sensitivity is usually compromised when compared with instruments based on the same technologies, but designed for use within the laboratory. The mission of many military operations is evolving, though, and requires that some specialized teams take the laboratory to the field. These teams are extending capabilities for field analysis not only by using a larger variety of technologies and capabilities, but also by using lab-grade equipment with improved analytical capabilities when compared with typical field-portable instruments. The US National Guard Bureau's Civil Support Team-Weapons of Mass Destruction (CST-WMD) units are one such group. Each of these teams supports a fully functioning mobile laboratory. These mobile laboratories are complex, but have been a successful part of the mission of the CST-WMD teams for more than a decade. While the on-board technologies continue to evolve, including more and more complex systems such as PCR and GC-MS, the use of microscopy and microspectroscopy techniques continue to remain as an integral part of the work flow of these mobile laboratories. The challenges to using these techniques in the field, as well as the successful integration of microspectroscopy in the CST-WMD mission, will be discussed.



## **Evening with Brian: When Living Cells Make Humans Explode**

Brian J. Ford, Caius College, Cambridge University

Spontaneous human combustion has been recorded for centuries. There was a new case reported recently from Ireland. A person simply bursts into flame, and all that remains is a pile of ash with largely undamaged arms and legs protruding from the remains. Accounts of witnesses are astonishing. They speak of a door handle being too hot to touch, of a smoldering pile being found in the middle of an otherwise undamaged room. Is this real? Are the cases imaginary? Have people been exaggerating?

There have been many theories put forward to explain this dramatic phenomenon. The only one to be widely accepted is the 'wick effect', in which fat melts into clothing and burns like wax in a candle. The continuous burning liquefies more body fat, setting up a continuous combustion. This theory fits few of the facts, and it would take at least twelve hours to work — spontaneous combustion consumes a person in a matter of minutes. This evening, we will discover a revolutionary new theory based on the metabolism of cells within the body. Spontaneous human combustion can be seen as a fleetingly rare, extremely dangerous, but potentially avoidable, condition.

**TUESDAY, JULY 10**  
**ENVIRONMENTAL AND INDUSTRIAL**  
**MICROSCOPY**

**Pigmented Corrosion**

Brendan Nytes, Microtrace LLC

Skip Palenik, Microtrace LLC

Mark E. Palenik, Microtrace LLC

While contamination problems plague all manufacturing companies, the food and pharmaceutical industries face high levels of scrutiny as humans ingest their products. Contaminants may be present in raw materials but also, often, arise from degradation of the machinery used in the manufacturing process. Although identifying the contaminant is the first step, determining the original source is generally the ultimate goal of the investigation. In this case study, we present the analysis of an unusual blue, black, and brown contaminant found at trace levels in a food product. The contaminant was characterized by polarized light microscopy, transmission electron microscopy, energy dispersive X-ray spectroscopy, and Fourier transform infrared microspectroscopy. Appropriate literature and reference samples were then consulted to confirm the identity of the foreign material and the mechanisms for its formation. With subsequent input from the manufacturer, we were able to determine the source of the contaminant particles.

**Three Cases of Pipe Failure with Substantial Corrosion**

Tony Havics, pH2, LLC

Pipes fail for a number of reasons, but often it is corrosion. It corrodes for a variety of reasons including: dissimilar metals, microbially induced corrosion (MIC), product incompatibility, erosion corrosion, chemical treatment (or lack thereof), and temperature induced diffusion or recrystallization, to name a few. This presentation examines two water-based systems and

the most likely causes of the corrosion and their relationship to failure. Examinations typically start with macro-observations using stereo or dissecting microscopes, followed by sub-sample material preparation, then light microscopy including reflected light with polarized light, metallographic etching, hardness testing, and scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS). In two of these cases, filtered water from the affected and non-affected areas were also examined for biological and non-biological components using microscopical techniques, such as epi-fluorescence, microchemistry and SEM-EDS, that can help point to a cause, or lack of one.

### **Characterization of Nanoparticles in Soil and Road Dust Using Scanning Electron Microscopy**

Jani Tuoriniemi, Chemistry and Molecular Biology, University of Gothenburg

Geert Cornelis, Chemistry and Molecular Biology, University of Gothenburg

Stefan Gustafson, Applied Physics, Chalmers University of Technology

Martin Hassellöv, Applied Physics, Chalmers University of Technology

The investigation of the hazards of nanotechnology demand new methods for the detection and quantification of nanoparticles in various matrices. The measurement of aerosols is already a mature field. For nanoparticles in liquid dispersions, there are promising new techniques such as single particle ICP-MS, and nanoparticle tracking analysis. However, there is a lack of methods capable of quantitative determination of nanoparticles in solid samples such as food and soil.

Scanning Electron Microscopy (SEM) with backscattered electron (BSE) imaging and energy dispersive spectroscopy (EDS) is widely used for characterization of particles. It is, in principle, also

as particles per mass, or volume of sample. However, the use of SEM for particle concentration measurement needs more development. Our current research is focused on the possibility to measure number concentrations of Ag nanoparticles spiked into soil. Characterization of tungsten carbide particles emitted from winter tire studs in road dust and storm water will also be presented.

### **Paper Product Recycled-Fiber Claims and the Comparisons with a Fiber Analysis**

Walter J. Rantanen, Integrated Paper Services, Inc.

There are many different types of paper products (including writing paper, envelopes, newsprint, tissue, toweling, bag paper, boxboard, and other packaging) that claim to include recycled fibers. Within the paper industry, and on many paper products, there are many different claims of recycled-fiber content addressing concerns for the environment. These claims are targeted at the consumers of the products, including the commercial distributor, and are also there to satisfy government requirements. Some only specify a “recycled” amount while others list a “post-consumer” fiber level. A microscopical fiber analysis can determine if a paper product appears to contain typical recycled fiber or more closely resembles all virgin fiber. The analysis may also help render an opinion, if the percentage claims on the paper product are consistent or not.

### **Wood Without Annular Growth Rings**

John A. Reffner, John Jay College of Criminal Justice, CUNY

Can a tree be a tree without growth rings? It can, if it is a palm tree. The microstructure of this unique wood is the subject of this paper. While visiting Hawaii, I acquired samples of several tropical hardwoods. They were all dark colored and most had intricate grain patterns, which makes them popular materials for carving bowls, statues and other ornamental art. The sample of

palm wood caught my attention because it lacked annular growth rings. When I tried to cut radial cross-sections, I had another surprise; I produced sawdust. The wood was too hard. A little water will solve the problem, I thought. Not so! Perhaps I should soak the block of wood in water overnight. When I dropped the block of wood into the water, it sank. Measuring the density of the palm wood, I found it to be 1.120 g /cc. Success in sectioning this wood came by using a slow speed diamond saw. The microstructure was revealed at last.

### **Mold Damages: Microscopy Analysis by Tape Lift Versus Determination of Enzymatic Reactions and Evaluation of Analysis Methods**

Marianne Berdal, Mycoteam AS

Catherine M. Whist, Mycoteam AS

Johan Mattsson, Mycoteam AS

In many cases of building surveys, focus on identifying possible mold damage is highly desired. Microscopical analysis of tape lift cannot give an exact identification of all the occurring species, but it is often possible to identify the genus and the species of the most commonly found mold fungi. By determining the species and using the knowledge of the ecological requirements the various species have, it is possible to determine the causes of the damage. Furthermore, analysis in a light microscope gives an exact description of the distribution between established mold growth and accumulated spores.

Often it is possible to estimate the relative age of the mold damage, based on the observation of microscopic details.

Measuring enzyme activity in occurring mold fungi is a newly developed method that is based on quantifying the activity of an enzyme that can be found in all mold fungi and the enzyme activity is correlated with fungal biomass. The method does not give any information about the occurring fungi. This leads to a restricted possibility for reading important information from

risk of misleading information regarding the interpretation of possible damages.

**An Overview of the ASTM Method for Quantification of Airborne Fungal Structures in an Inertial Impaction Sample by Optical Microscopy**

Tony Havics, pH2, LLC

Michael Breu, Fiberquant Analytical Services

Much has been made about the lack of reproducibility, from laboratory to laboratory, when analyzing commercially available spore trap samples for fungi. A good portion of that lack of reproducibility has been attributed to the absence of published methods describing how to test these common sample matrices. The American Society of Testing Materials (ASTM) formed a subcommittee under D22-Air Quality in 2005. That subcommittee, D22.08, was tasked with developing both sampling and analytical standards for mold. The pressing need for standardization of spore trap analyses meant that a counting method was made one of its top priorities. The Method, D7391-09 was their first standard published in June, 2009 and is called the Standard Test Method for Categorization and Quantification of Airborne Fungal Structures in an Inertial Impaction Sample by Optical Microscopy. It is a 12-page document that standardizes the minimum requirements for analyzing both slit-impactors as circular impactors. Examples of the many requirements include the resolution ability of the optical microscopes, the percentage of the sample that must be analyzed, reporting parameters for background debris loading, and specifying which fungal structures must always be reported when seen in a sample.

**Thermite in the World Trade Center Dust?**

James R. Millette, MVA Scientific Consultants

In 2009, Harrit et al., published a paper in the peer-reviewed literature which concluded that thermitic material was present

(WTC) buildings on September 11, 2001. Harrit described finding small red/gray chips that they claimed were nano-thermite. These chips were attracted by a magnet and showed an elemental composition primarily of aluminum, silicon and iron as determined by scanning electron microscopy and x-ray energy dispersive spectroscopy (SEM-EDS). Using samples of WTC dust from 4 locations, red/gray particles that matched the criteria of Harrit were subjected to analytical tests in accordance with the Recommended Guidelines for Forensic Identification of Explosives and the ASTM Standard Guide for Forensic Paint Analysis and Comparison. These tests included FTIR; SEM-EDS of cross-sections; low temperature ashing and residue analysis by TEM-SAED-EDS; muffle furnace ashing and residue analysis by PLM and TEM-SAED-EDS; and ultra-microtome sectioning and analysis by TEM-SAED-EDS. Our studies concluded that the red/gray chips were consistent with steel with an epoxy resin coating containing iron oxide and kaolin clay. There was no evidence of individual elemental aluminum particles of any size, therefore the red layer of the red/gray chips is not nano-thermite.

#### **Scientific Working Group on Geological Materials (SWGGE0)**

Chris E. Taylor, U.S. Army Criminal Investigation Laboratory

Currently, there are approximately 45 forensic science practitioners conducting soil analysis, and close to 25% of forensic science service providers within the United States that analyze soil evidence in trace evidence casework. Although the practitioner base is small in comparison to other areas of trace evidence, forensic science analysts practicing soil analysis are in desperate need of standards development, education/training and guidance, and uniform terminology for methods, reports, and conclusions. The SWGGE0 would represent a worldwide effort for soil examination and standardization and, ultimately, would improve and promote forensic science soil testing programs in laboratories and its use in criminal cases. The

SWGGE0 would leverage and formalize the efforts from previous ad hoc working group meetings to support short-term and long-term goals and anticipated deliverables. This presentation will outline the efforts, goals and way forward of this scientific working group.

### **What the Cells in a Dinosaur's Tail Tell Us About Dinosaur's Evolution**

Brian J. Ford, Caius College, University of Cambridge

Dinosaurs have been popular objects of mystery since Victorian times. Yet how could such colossal creatures have existed? We accept recreated images from movies like Jurassic Park (1993) and Dinosaur (2000) — in addition to innumerable television documentaries — without pondering the biological feasibility of maintaining a large body mass in a terrestrial environment.

When we consider the role of the body cells, and in particular the constraints they exert upon evolutionary development, a radically new interpretation becomes possible. Contemplating the cell community can allow us to postulate that dinosaurs and their lifestyle were actually very different from what the current orthodoxies dictate.

### **Microscopical Analysis of Dust: Information Taken Out of Thin Air**

Marianne Berdal, Mycoteam AS

Catherine M. Whist, Mycoteam AS

Johan Mattsson, Mycoteam AS

Indoor Air Quality (IAQ) surveys are often focused on single factors, in order to identify and eliminate any health complaints. This can lead to a risk of not finding the reason for the most relevant cause for problems.

Dust occurring in buildings represents an exceptionally good information source for understanding the IAQ. Whether it is air-



can give both a quantitative and qualitative description of the actual dust particles. This can be a great advantage when identifying the amount and the sources of dust that have impact on the IAQ. This knowledge improves the possibility for focusing on important aspects at IAQ surveys.

Our result is that a microscopical analysis provides both a qualitative and quantitative description of the dust, whether it is air-borne or settled on surfaces. Furthermore, it is possible to categorize the particles into four main groups, based on the origin:

- Natural/outdoor air
- Building materials
- Use and users
- Mold damages

This ensures that the evaluation of IAQ is based on sufficiently accurate facts, while measurements can be tailor-made and cost-effective.

### **Trace Mineral Solutions to a Sediment Trespass Controversy**

Wayne C. Isphording, Tulane University

“Trespass” of sediment runoff, airborne effluent from nearby industrial sources, etc. is, today, an all too commonplace phenomenon. Down slope movement of sediment is a natural process but it can be exacerbated in cases where “best management practices” were not followed. Damage from the movement of objectionable quantities of sediment may result in the in-filling of ponds, lakes, or streams or in alteration of the pre-impact topography. Proving in a court of law that trespass has taken place, however, may be a challenge for the engineer or geoscientist. A case history is described where developers of a large shopping center failed to prevent excessive sediment runoff from a site with the result that a stream was heavily impacted by sediment influx, preventing property owners from launching boats, or otherwise enjoying the stream. Identifying the quantity

the stream was made possible by detailed physical and microscopical examination of core samples. Differences in particle size characteristics, when compared with those from pre-construction bottom sediments, was apparent but most telling were the heavy minerals in the eroded material when they were compared with indigenous bottom sediments from the stream. Though present in quantities of less than one percent (1%) in samples, the ratio of Zircon+Tourmaline ÷ Rutile easily allowed identification of the precise impact boundary, even where particle sizes of the units in question were similar.

**Calculated Average Ranges of Refractive Indices for the Amphiboles Found in the Rainy Creek Igneous Complex Near Libby, Montana, USA**

Matthew S. Sanchez, RJ Lee Group

Mickey E. Gunter, Geological Sciences, University of Idaho

The amphiboles occurring in the Rainy Creek Igneous Complex (RCC) range in composition with the following amphibole species being identified: tremolite, winchite, richterite, magnesioriebeckite, and magnesioarfvedsonite. Combined compositional, structural, and optical data for only winchite and richterite have been determined on the same crystals from RCC. In an attempt to characterize the mean refractive index specifically for tremolite, magnesioriebeckite, and magnesioarfvedsonite species derivations of Gladstone-Dale constants specific for the RCC were attempted based on the multiple linear regression work by Bloss et al. (1983) using the compositional, structural, and optical data obtained by Bandli et al. (2003) on six single crystals from the RCC. Based on these data, it was not possible to obtain specific Gladstone-Dale constants for the RCC amphiboles. Therefore the Gladstone-Dale constants derived by Jaffe (1987) for the alkali amphiboles were applied to the published EPMA data from Meeker et al. (2003) specific to the tremolite, magnesioriebeckite, and magnesioriebeckite, as well as the data from

Sanchez et al. (2008) for magnesioarfvedsonite. The range of average refractive indices for the tremolite, magnesioriebeckite, and magnesioarfvedsonite are calculated as  $n=1.610$  to  $1.629$ ,  $n=1.646$  to  $1.665$ , and  $n=1.641$  to  $1.652$ , respectively. This range of refractive indices for the amphiboles occurring within the RCC is much smaller than the published ranges for magnesioarfvedsonite and magnesioriebeckite because the samples from RCC do not cover the entire solid solution range for these species.

#### **Limit of Detection Issues in Measurement of Low Concentrations of Tremolite in Mineral Products**

Eric J. Chatfield, Chatfield Technical Consulting Limited

Low concentrations of tremolite have been observed in a number of sources of industrial mineral products such as chrysotile, talc, vermiculite, sepiolite, dolomite and wollastonite. Detection of asbestos-forming amphibole in a mineral product can come as a surprise to both the producer and the user, and this takes on particular significance if any of the amphibole can be defined as asbestos.

The U.S. legal community has taken a keen interest in the presence or absence of tremolite contamination, specifically in chrysotile. Some studies have indicated that exposure to chrysotile alone does not cause mesothelioma in humans, whereas exposure to amphibole asbestos such as tremolite is known to cause this disease. Accordingly, plaintiff's attorneys will argue that even if exposure to chrysotile was not the cause of their client's mesothelioma, tremolite contamination present in the chrysotile was responsible. The defence will generally argue that chrysotile does not cause mesothelioma, and that tremolite, if present in the chrysotile, was only at trace levels, and therefore the plaintiff must have received a more significant exposure to amphibole asbestos and that this exposure was not the responsibility of their client. Sometimes, electron microscope examination of lung tissue obtained at autopsy can resolve the

question.

When no other amphibole exposure can be identified, the legal arguments in such cases are dependent on whether the product in question contains tremolite, and if so, what its concentration is and whether any of it is asbestiform.

Several examples will be discussed in which questionable measurements of tremolite in mineral products have resulted in either significant financial expenditures or subsequent publication of misleading papers in the peer-reviewed literature.

### **Mineral Identification Using Electron Backscatter Diffraction from Unpolished Specimens: Nothing is Easy; Some Things are Just Less Difficult**

Bryan Bandli, Geological Sciences, University of Minnesota

In order to identify a mineral, both the chemical composition and crystal structure need to be determined. Electron backscatter diffraction (EBSD) is a crystallographic technique that, when used in conjunction with scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS), can allow for the rapid identification of a crystalline phase using both chemical composition and crystal structure. SEM/EDS alone is limited as a tool for phase identification by the inability to collect useful crystallographic information from an unknown specimen. Most EBSD work is performed on carefully prepared polished bulk specimens. By selecting individual crystals or crushing and grinding a sample to achieve a particle size in the range of 5-100  $\mu\text{m}$  it is possible in a matter of minutes to produce a sample capable of providing high quality electron diffraction patterns and compositional data suitable for phase identification. When compared to the effort required to polish a bulk specimen, the methodology presented provides several advantages. The main advantage of the technique is speed, but also the ability to precisely select phases of interest from a hand sample or thin section.

### **Polarized Light Microscopy (PLM) Analysis of Cosmetic Talc for Asbestos Content**

Lou Solebello, International Asbestos Testing Laboratories, Inc.

The Cosmetic Toiletry and Perfumery Association (CTPA) defines cosmetic talc as a fine, white, odorless powder used for cosmetic purposes. CTPA specifies a minimum of 90% “hydrated” (hydroxylated) magnesium silicate (talc) with the remainder being composed of “naturally occurring associative minerals” (accessory minerals kaolin, chlorite, calcite, dolomite, magnesite) and “no detectable fibrous asbestos minerals”. Methods used for CTPA and FDA asbestos analysis of cosmetic talc are outdated, do not specify detection limits for asbestos, and do not provide definitions for fibrous amphibole asbestos. The purpose of WK00324 is to: develop a PLM method for asbestos analysis of cosmetic talc that is consistent with CTPA, FDA and other regulatory requirements; incorporate established techniques; and establish reportable detection limits and provision of fibrous amphibole asbestos definitions. The current method draft, and changes, will be discussed.

### **Compositional Analysis and Morphological Relationships of Minerals Found in Talc Tailings and Products from Near Talcville, New York, USA**

Brittani D. McNamee, Geological Sciences, University of Idaho

Mickey E. Gunter, Geological Sciences, University of Idaho

André E. Lalonde, Earth Sciences, University of Ottawa

Michael C. Rowe, School of the Environment, Washington State University

Compositional analyses were performed by WDS, via electron microprobe, on a thin section of an amphibole talc schist collected in the tailings and a commercial product from a former talc mine near Talcville, New York. The backscatter electron detector (BSE) was used to aid in mineral identification and to

better image the morphology. Within the rocks, tremolite exists as unaltered crystals. Anthophyllite occurs in an acicular habit with a series of fractures perpendicular to its elongation. Talc occurs as fine-grained masses filling in the anthophyllite fractures, between acicular anthophyllite partings, as pseudomorphs after anthophyllite, and as platy talc.

A polished grain mount of commercial product produced from the mine (i.e., mouldene) yielded similar results. Tremolite occurs as unaltered grains. Talc occurs in a platy habit, as long fibrous particles, and as an alteration product along the rims of some minerals. Anthophyllite grains are acicular and separated into smaller pieces along the ladder fractures with edges altering to talc. One grain of non-asbestiform manganoan-cummingtonite was found with rims altering to asbestiform talc. Also, a few grains of diopside were found altering to asbestiform talc. Thus, in the ore and product we found asbestiform talc as an alteration product of nonasbestiform minerals.

## **WEDNESDAY, JULY 11**

### **CHEMICAL and FORENSIC MICROSCOPY**

#### **Developing a Microcrystal Test for a “Legal High” Drug**

Samantha Huntsman, McCrone Research Institute

This research focuses on the development of a microcrystal test to detect the presence of 1-[3-(trifluoromethyl)phenyl]piperazine (TFMPP). TFMPP, a synthetic piperazine, has no approved medical use and is commonly marketed as a legal alternative to 3,4-methylenedioxy-N-methamphetamine (MDMA). Originally developed as an industrial chemical, TFMPP is currently unscheduled in the United States, but was temporarily designated as a schedule I hallucinogen in 2002 until, after investigations, the schedule was removed. Unfortunately, so-called “legal highs”, such as TFMPP, don’t often have sophisticated testing schemes developed due to their short lives as abused substances. Using platinic bromide reagent, dendritic crystals were obtained which converted to blade-like crystals with progression of the reaction. Studies were also done to verify the uniqueness of the crystals and to determine the limits of detection of the test.

#### **The Forensic Analysis of Paint Evidence Using Micro-Raman Spectroscopy Part I: Development of Libraries and Application Methods**

Christopher S. Palenik, Microtrace LLC

Patrick Buzzini, Forensic & Investigative Science Program

Jennifer Herb, Microtrace LLC

Ethan Groves, Microtrace LLC

Although the benefits and advantages of Raman spectroscopy have long been recognized and utilized in the academic and industrial fields, this method has yet to make a significant impact in the forensic laboratory. Raman spectroscopy is new to forensic laboratories but it has sparked a recent interest in the last few

important advantages such as its non-destructive nature, its fast analysis time, and especially the opportunity of performing microscopical in situ analyses. These advantages make this method suitable for the forensic examination of paint evidence. During this two-part presentation, we will discuss the ability of micro-Raman spectroscopy to selectively detect and identify organic and inorganic pigments. Part I will focus on the development of a Raman micro-spectroscopy pigment reference collection and its application to a collection 300 recently collected vintage automotive paint samples.

#### **The Forensic Analysis of Paint Evidence Using Micro-Raman Spectroscopy Part II: Case Examples**

Patrick Buzzini, Forensic & Investigative Science Program, West Virginia University

Christopher S. Palenik, Microtrace LLC

Genevieve Massonnet, Universite de Lausanne

Part II will focus on the application of Raman spectroscopy to casework for both identification and individualization purposes. See Part I, above.

#### **Benefits of Using Cross-Sectioning in Forensic Analysis of Automotive Paints**

Ethan Groves, Microtrace LLC

Jennifer Herb, Microtrace LLC

Christopher S. Palenik, Microtrace LLC

A common type of trace evidence is paint. Paint can be viewed many different ways. A survey of 300 automotive paints has shown that layer structure can be very distinguishing and complex. As the properties of “high-performance” pigments, binders, and polymers evolve the thickness of layers can be decreased for cost and environmental benefits. Decrease in layer thickness makes cross-sectioning the best, if not only,



technique to view a complete layer structure. Unlike other traditional methods for analyzing automotive paints, such as bevel cutting, grinding/smearing or collecting thin peels, thin-sectioning allows for information from each layer to be retained and collected individually. While bevel cutting allows for visualization of the layer structure, layer thickness cannot be measured or quantitatively compared in this way. In the examination of 300 paint samples studied, a variety of interesting layer structures, layer application methods, and considerations for forensic sampling will be discussed as well as their implications for accurate forensic comparison and interpretation.

### **American Society of Trace Evidence Examiners (ASTEE)**

Chris E. Taylor, ASTEE

The American Society of Trace Evidence Examiners is proud to partner with Inter/Micro by offering a Tuesday night social event. This interaction can promote idea sharing and create new professional and personal friendships. This presentation will tell you how ASTEE has successfully made it through its early years and its future plans. The membership has surpassed over 300 in just two short years and corporate support and sponsors have helped to establish a good financial foundation. ASTEE has its own peer-reviewed journal and with the support of its members and partners, new articles and valuable literature continues to be published. ASTEE pledges to mature by continuing to provide and increase resources to its members. Additions to the website [www.asteetrace.org](http://www.asteetrace.org) will afford each trace evidence practitioner and those interested in trace evidence a resource for all that is trace. ASTEE's long-term goals to improve member benefits will be presented and discussed. With the accomplishments of ASTEE (and most importantly its membership) the significance of trace evidence will continue to be valued and will grow in importance. "Be active, be passionate, and remember every contact you make leaves a trace on you and the forensic science community."

### **Environmental Effects on Fired Cartridge Cases: Primer Shear and Breech Face Marks**

Peter Diaczuk, John Jay College of Criminal Justice, CUNY

Stephanie Pollut, John Jay College of Criminal Justice, CUNY

There is, unfortunately, not much information about the comparison of discharged cartridge cases after being left in the environment. This situation can cause an undesirable change to the case, which could adversely affect the impressions left by the firearm. The purpose of this project was to determine the quality of a comparison after cartridge cases were placed in the environment. Ammunition consisting of four different case metals (brass, steel, aluminum, and nickel) was fired and the discharged cases were collected. After initial examination with a comparison microscope to confirm that the firearm used was imparting reproducible marks onto the primers, the cases were purposely placed onto open soil. At predetermined time periods, the cases were removed from the environment for examination. A representative of each group was set aside without exposure to act as a control. After collection, initial cleaning of the samples was done with a soft paintbrush and only minimal pressure to allow microscopical observation, however the need for more aggressive cleaning for some samples was soon apparent. Once loosely adhering soil and corrosion was removed, the samples were examined with a comparison microscope to determine whether environmental degradation would compromise the examination.

### **Role of Edmond Locard in the History of Forensic Science: "Every Contact Leaves a Trace" Dissected**

Patrick Buzzini, Forensic & Investigative Science Program, West Virginia University

"Every contact leaves a trace." This quote, universally known as the Locard's exchange principle, is only the tip of the iceberg of all contributions and development that Dr. Locard made in the

The path followed by Locard to become one of the most important pioneers in forensic science will be explored, including his educational and cultural background, his mentors, and all the figures that played an important role in his career. In this historical journey, the scientific methodology of physical evidence and the philosophy that Locard formalized during his activities (as director of one of the first forensic laboratories in the world) will be described. The author will also expose the different types of evidence that Locard processed, starting from his research on poroscopy to the more than 11,000 cases that he examined in the field of questioned documents. This will be undertaken to show the 'extreme variety of signs' as well as the 'flexibility and polymorphism of the method,' as he emphasized in his book of 1920, *L'enquête Criminelle et les Méthodes Scientifiques*. The origin of Locard's exchange principle, his original statement, and its role for recognizing evidence at the crime scene will be described.

### **Microspectrophotometry (MSP) of Blood**

Larry Peterson, U.S. Army Criminal Investigation Laboratory

The identification of blood during forensic investigations is a routine matter handled by DNA or serology units within the crime laboratory. However, trace evidence examinations can disclose microscopic-sized particles of suspected blood on hair, fibers, or other recovered trace evidence examinations can disclose microscopic-sized particles of suspected blood on hair, fibers, or other recovered trace evidence. These bloodstains may be probative to an investigation. The ability to identify a substance as blood by spectroscopy is a long-standing method and can now be performed in the modern laboratory using a microspectrophotometer. This non-destructive method allows a particle to be characterized as blood, by combining the particle's microscopical appearance and its UV-VIS spectrum.

### **Antibody-Based Forensic Body Fluid Identification**

Karl Reich, Independent Forensics

Jennifer Old, P.W. Boonlayangoor

The forensic identification of body fluids is well established as an essential laboratory tool for criminalistic investigations and is based on bio-marker recognition. Identify the marker associated with the body fluid and conclude that the body fluid is indeed present. The choice of bio-marker and the method of its detection are therefore crucial to the accuracy and reliability of this method. With the large-scale implementation of DNA-based forensic testing, source attribution of observed stain(s) from crime scenes or from recovered evidence has become particularly important.

Current forensic practice recognizes four body fluids and two cell types; blood, semen, saliva and urine, and epithelial and sperm cells. Historically, chemical and enzymatic methods, sometimes coupled with microscopy, were used to identify blood, saliva, semen, and sperm. More recently, antibody based methods have been developed that identify new bio-markers from blood, semen, urine and sperm, and provide more specific identification of saliva.

The development of the antibody-based RSID™ (Rapid Stain IDentification) series of lateral flow immunochromatographic strip tests and of the immunofluorescent SPERM HY-LITER™ stain for sperm will be described and the advantages and limitations of these tests in forensic practice (and in comparison with older methods) will be outlined.

### **Analysis Paralysis**

Brian J. Ford, Caius College, University of Cambridge

Studies of live blood using dark-field microscopy in the 1960s gave rise to a useful laboratory technique for the dynamic observation of hemostatic mechanisms. The idea was taken up by a system of "Live Blood Analysis" which claims to reveal a

range of hidden secrets about the body — your state of health, undiagnosed allergies, the presence of cancer — which are promoted without scientific knowledge to support the procedure.

More recently the idea has been seriously promoted on television and today we will see how organizations are seeking to promote the idea among people who do not understand the science.

### **Is it Blood?: Recovery and Microchemical Identification of Blood in Forensic Specimens**

Katie White, Microtrace LLC

Skip Palenik, Microtrace LLC

Potential bloodstains are commonly submitted as evidence in criminal investigations and can also be encountered in food complaints and civil cases. Beyond the advantage of possible DNA associations, confirmation of the presence of blood may also provide investigators with contextual clues, such as evidence of an injury or corroboration of a statement. Today, several presumptive and confirmatory tests for blood are available, including the modern immunoassay tests popular in many laboratories. This session presents three microscopical methods for the identification of blood: the Takayama and Teichman tests, both of which are confirmatory microcrystalline tests based on the formation of hemoglobin derivative crystals, and the porphyrin test, which examines potential blood for characteristic red fluorescence under ultraviolet excitation. In addition to a brief overview of blood and its chemical structure, the chemistry behind each test will be discussed. Procedures for each method will then be presented with applications demonstrated for known samples of blood. Additionally, these methods will be compared and contrasted with other techniques. Though testing for blood is often routine, this presentation will also explore some unique examples from forensic casework, specifically with regard to sampling potential blood from unusual sample matrices.

## **One Century Later: The Takayama Hemochromogen Microcrystal Test**

Arthur Young, Guardian Forensic Sciences

In 1912, Dr. Masao Takayama published “A Method for Identifying Blood by Hemochromogen Crystallization”. In the century that has passed, man has been through two world wars, harnessed the power of the atom, conquered space and explored the solar system, invented and evolved the computer, and unraveled the secrets of DNA. The Takayama method for identifying blood has all but vanished into the mists of history, having been perceived as insensitive, slow, and cumbersome. A closer examination of his method, including subsequent improvements and a modern understanding of crystallography, can propel this method to a sensitivity beyond that of even modern forensic DNA analysis. As with any microscopy technique, success is determined more by the skill and the technique of the analyst and less by the quality of the equipment that is used. As man’s technology continues to improve, the need to conserve more and more of a limited sample for additional analyses grows. Therefore, the confirmation of blood — the fundamental step upon which the significance of all other analyses rests — shall always remain relevant, into the this century and beyond.

## **The Alchemist’s Tale**

Skip Palenik, Microtrace LLC

In 2011, the opportunity arose to purchase a painting of an alchemist, attributed to the “circle of Teniers.” Teniers was a prolific seventeenth century artist who is believed to have produced several hundred alchemical paintings in his lifetime. This painting intrigued the senior author who became interested in adding it to the fledgling Microtrace collection of alchemical

art. The price, while not inexpensive, was less than one would expect to pay for an authentic Teniers and was reputed to have last been purchased from a highly reputable auction house. The painting was purchased and subjected to a scientific examination in our laboratory.

This presentation will describe and illustrate the microanalytical approach we took to learn the secrets of this small painting on wood. It will also describe some of the physical attributes that likely led the art community to make the attribution they did.

#### **Trace Evidence Analysis at the U.S. Postal Inspection Service National Forensic Laboratory**

Andrew Bowen, U.S. Postal Inspection Service

This talk will introduce the audience to the National Forensic Laboratory of the United States Postal Inspection Service. The mission and jurisdiction of the Inspection Service, its laboratory facilities, and typical casework will be discussed. The trace evidence submissions consist largely of identifying unknown substances (often white powders) sent through the mail, recovering trace evidence from threatening letters, supporting investigations into burglary of postal facilities, and analysis of improvised explosive devices. Several case examples illustrating the nature of the cases submitted to the National Forensic Laboratory will be presented.

#### **Development of a Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals**

Sebastian Sparenga, McCrone Research Institute

This talk will discuss the cooperative agreement that McCrone Research Institute was recently awarded through the National Institute of Justice (NIJ-2011-2805 SL# 000944). The purpose of this project is to compile a comprehensive compendium of microcrystal tests, which have previously been developed for

locating, and compiling analytical data and literature material from the numerous sources (many of which are out of print or difficult to locate) spanning past decades. Techniques will also be developed, where necessary, for drugs within non-traditional delivery mechanisms. All procedures will be vetted and appraised by McCrone Research Institute research microscopists, together with practicing forensic scientists in other collaborative laboratories. The resulting electronic compendium will include recommended protocols and morphologies of crystals (including photomicrographs), infrared spectra of microcrystals, and potential interferences. But most importantly, the compendium will also include optical and crystallographic properties of the resultant microcrystals. This optical data is absent in many references, which is unfortunate because microcrystals of a given substance are unique if optical properties, and not only morphology, are considered. Included with this optical data will be a refinement of the application of many microcrystal tests in order to strengthen their use within the criminal justice system. Furthermore, this Microcrystal Compendium will be available to all forensic scientists for free access from selected websites.

### **Examining the Effects of Environmental Degradation on the Optical Properties of Manufactured Fibers of Natural Origin**

Kelly M. Brinsko, McCrone Research Institute

With the production of manufactured fibers of natural origin increasing in recent years, products such as azlon and polylactic acid fibers are likely to become more common in regular case work in the forensic science laboratory. However, little is known about the changes occurring in their optical and physical properties as an effect of moisture, sunlight exposure, and exposure to various temperatures. A federally funded research study is underway to investigate the effects of such degradation on the optical properties of selected fibers (polylactic acid, azlon, and rayon). These fibers, which are often proclaimed by



manufacturers as being biodegradable (because they are made from naturally occurring proteins, sugars, or cellulose) are expected to show the most change compared to synthetic fibers such as polyester or nylon. Environmental conditions such as exposure to water (saltwater and freshwater), UV light, and hot and cold temperatures will be explored while documenting any change in optical properties. Polarized light microscopy observations, including morphology, pleochroism, refractive index, birefringence, extinction characteristics, sign of elongation, solubility, and thermal behavior will be monitored throughout two years of exposure to these conditions. Infrared spectra will also be collected at various time intervals to complement light microscopy data. Noticeable changes in optical properties of these types of fibers could prove to be important in a forensic setting, notably in fiber comparison and identification. An outline and strategy for this research project will be presented, along with preliminary data and an assessment of the occurrence of such fabrics in consumer textiles.

### **Whose Hair is it Anyway?: Hair and Fiber Microscopy Uncovers Fakes, Frauds, and the Occasional Genuine Article**

Jason Beckert, Microtrace LLC

Microscopy is a powerful analytical technique in the examination of hairs and fibers. Often overlooked or underappreciated, it is usually the first means of critically examining these materials, and in some cases, it is the only technique employed. Microscopy can exploit variations in morphology, color, and optical properties when performing identifications or comparisons between samples. This presentation will demonstrate the utility of microscopy as it relates to answering questions concerning authenticity and truth in advertising. While this presentation will focus on animal hairs, other materials including vegetable fibers, synthetic fibers, and human hair will be discussed. Diverse case examples will span the range from modern garments and wigs to purported ancient artifacts and mythological creatures.

### **Workshop: Mineralogy for Microscopists**

Mickey E. Gunter, Geological Sciences, University of Idaho

With an in-depth knowledge of mineralogy, mineral identification through various microscopical techniques becomes considerably less difficult. Many microscopists, however, lack formal training in mineralogy, which often hinders their ability to identify minerals and acquire an appreciation for mineral associations, alterations, nomenclature and, most importantly, the fact that the physical properties of minerals are directly related to their crystal structures.

In this two-day workshop, participants will learn about these areas by using specific mineralogical examples based on subtle similarities and differences between mineral structures and compositions as observed with different microscopical methods. Examples include 1) why framework silicates (quartz, feldspars, zeolites, etc.), as seen with polarized light microscopy, exhibit low retardation, while sheet silicates exhibit low retardation in one plane; 2) the differences and similarities of calculated selected area electron diffraction patterns for common silicate minerals; and 3) why it can be difficult to identify particles of similar composition with EDS spectra.

The workshop will also compare and contrast the nomenclature schemes for amphiboles and zeolites, the former being based on precise compositional data, while the later are based first on structural data and less so on compositional data.

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