

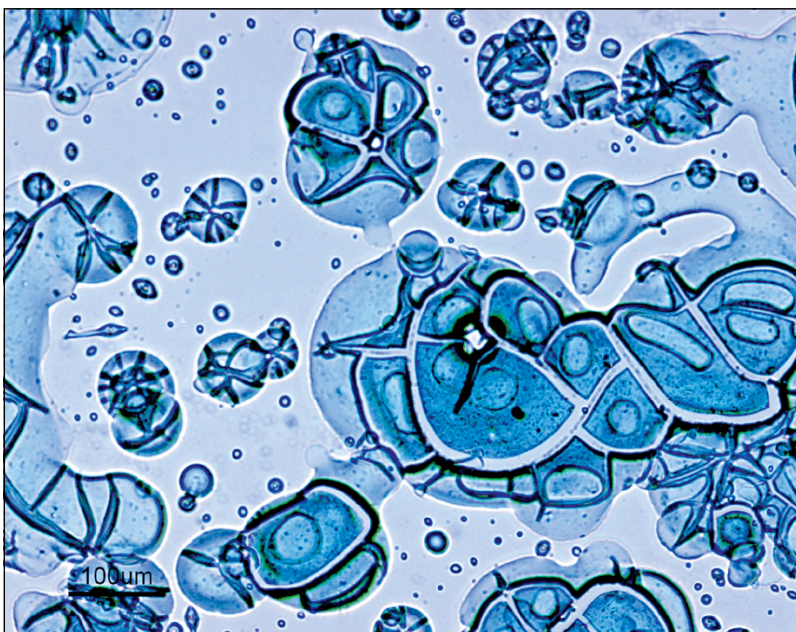
# INTER/MICRO 2013

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## A Microscopy Symposium

July 15 – 19, 2013

McCrone Research Institute, Chicago



*Sponsored and hosted by*

**McCrone Research Institute**

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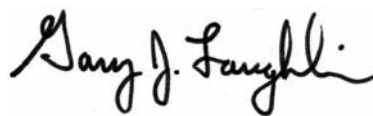
# INTER/MICRO 2013

## ***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 of these symposia came from having the world’s best microscopists visit Chicago to further his education. Thank you for supporting Inter/Micro — so that we can all continue to further our education.



Gary J. Laughlin  
Chairman, Inter/Micro

## **Cover image by Martin Kocanda, Northern Illinois University**

This image shows water-based paint droplets on glass, viewed with light microscopy under crossed polars at 300x magnification. The image was voted Best Overall Winner of the 2012 Inter/Micro Photomicrography Competition, sponsored by pH2, LLC.

**MONDAY, JULY 15**  
**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: Ethan Groves, Microtrace, LLC*

**Raman Analysis of Carbon Materials**

Ken Smith — Thermo Fisher Scientific

**Increasing Depth of Field in Photomicrography by Using Focal Stacking Techniques**

Julian C. Gray — Tellus Science Museum

**Mixed Greens — A Closer Look at Currency Using Photomicrography**

Martin Kocanda — Northern Illinois University, Department of Electrical Engineering

**An Examination of The Fiber Composition of Mid-20th Century Papers**

Walter Rantanen — IPS Testing Experts

**The Identification of Bicomponent Acrylic Fibers Using Dispersion Staining**

Jennifer DePirro — McCrone Research Institute

**Can Raman Spectroscopy Save the Polarized Light Microscope?**

Andrew Bowen — U.S. Postal Inspection Service

**Between the Fringes: Overlooked Topics in Microspectrophotometry**

Christopher Palenik and Jason Beckert — Microtrace, LLC

**Colorant Basics: Chemical Organization of a Dye and Pigment Database**

Ethan Groves and Christopher Palenik — Microtrace, LLC

*12:00 – 1:30 p.m. Lunch Break, McRI Garden*

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**Exposing the Myth of Creationism**

Brian J. Ford — Caius College, University of Cambridge

**World's Worst Microscopy — The New Normal?**

Andrew A. Havics — pH2, LLC

**Use of High Dynamic Range (HDR) Techniques to Increase  
Exposure Range in Photomicrography**

Julian C. Gray — Tellus Science Museum

**Low Vacuum Scanning Electron Microscopy (SEM) of Polymers**

Rich Brown — MVA Scientific Consultants

**Seeing Food Differently: X-ray Tomography**

Robert Myers — Wm. Wrigley Jr. Co.

**X-ray Tomography: Segmentation and Image Visualization**

Philip W. Urnezis — Wm. Wrigley Jr. Co.

**Instrumental and Microscopical Analysis of Artificial  
Sweeteners**

Andrew M. Bowen, Vincent J. Desiderio, Robert B. Moberley —  
U.S. Postal Inspection Service

**ATR Imaging with a FTIR Microscope**

Ken Smith — Thermo Fisher Scientific

**MONDAY, JULY 15**  
**AN EVENING WITH BRIAN:**  
**“MOVIE STAR MICROBES”**

*5:30 – 7:00 p.m.*

***Dinner, McRI Garden***

*Cost: \$25 (See the front desk to pay for the dinner if you did not pre-register.)*

*7:00 – 8:00 p.m.*

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

*Free*

This year Brian looks at microbes in the movies. Living cells and microorganisms have been featured in films, occasionally in starring roles and in main title sequences. This evening we will encounter some remarkable examples. Films rarely do justice to the microbe world, but the representations seen in the cinema and on television are revealing and lead to a crucial conclusion: Filmmakers are missing a major opportunity to capture the microscopic world. It is the single theme that Hollywood has yet to explore, and the possibilities are endless.

*Brian J. Ford is a leading authority on the microscope and a best-selling author, who has presented his work on television and radio. His research is widely quoted in publications, and he is a popular keynote speaker worldwide. He is the author of the Critical Focus column, published in The Microscope journal. Ford has given his Evening with Brian presentations at Inter/Micro for almost 30 years.*

**TUESDAY, JULY 16**  
**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: Wayne Moorehead, OC Crime Lab, Santa Ana, CA*

**The New Trend in Optical Microscopy, Digital Imaging and Measurement: The Difference Between Optical and Opto-Digital (Digital) Microscopes**

Christopher Vander Tuuk — Olympus America

**Trace Evidence in Industrial Problem Solving**

Kevin Brady — Tredegar Film Products

**Microscopy of Some Bitumen, Asphalt and Mastic-Based Building Materials**

Andrew A. Havics — pH2, LLC

**Fitting the Mold: An Exploration into Sourcing of Glass Fragments**

Brendan Nytes — Microtrace, LLC

**Charred Starch: Characterization by Microscopy, Staining and Spectroscopy**

Katie White and Skip Palenik — Microtrace, LLC

**Aquatic Dinosaur Update**

Brian J. Ford — Caius College, University of Cambridge

**A Study of Select Refractive Index Liquid Stability, Series A and Series AA**

Wayne Moorehead, Kaycee Fontes and Cassandra Hayes — OC Crime Lab, Santa Ana, CA

*12:00 – 1:30 p.m. Lunch Break, McRI Garden*

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

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

**Identification of Contaminant Particles and the Determination of Syringe Penetration Angles in Pharmaceutical Vials with Rubber Stoppers**

Jason Beckert, Christopher Palenik and Ethan Groves —  
Microtrace, LLC

**Smokestacks — Can't Live With Them, Can't Live Without Them!**

Wayne C. Isphording — Tulane University School of Continuing  
Studies

**The Microscopy of Asbestos Products: Case Studies of the Unique and Interesting**

Steven P. Compton — MVA Scientific Consultants, Inc.

**Dispersion Staining: What's the Objective?**

Peter M. Cooke — MICA

**Effects of Hydration on Erionite Refractive Index Measurement**

Lou Solebello and Frank Ehrenfeld — International Asbestos  
Testing Laboratories, Inc.

**Comparative Characterization of Erionite Specimens from Three Locations**

Lou Solebello and Frank Ehrenfeld — International Asbestos  
Testing Laboratories, Inc.

**What Causes Some Mineral Fibers to be Carcinogenic?**

Eric J. Chatfield — Chatfield Technical Consulting Limited

**WINE AND CHEESE RECEPTION WITH EXHIBITORS**

*5:00 – 6:00 p.m., McRI Exhibit Room*

*Free*

Meet representatives from Campbell Center, Foster+Freeman,  
Leica Microsystems, Olympus, Scientific Device Laboratory,  
Thermo Fisher Scientific and Tousimis.

**WEDNESDAY, JULY 17**  
**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: Patrick Buzzini, Forensic and Investigative Science Program, West Virginia University*

**The Use of Blue-Light Curing Resins in Forensic Sample Preparation**

Ethan Groves and Christopher Palenik — Microtrace, LLC

**SEM/EDX Microtrace Analysis in Forensic Casework**

P.D. Zoon, S.B.C.G. Chang and E.J. Vermeij — Netherlands Forensic Institute

**A New Technique for the Identification of Dyes Extracted from Single Fibers**

Katelyn Hargrave and Skip Palenik — Microtrace, LLC

**Techniques in Cross-Sectioning Fibers, Tapes and Paints**

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

**What is the Problem with Dichroic Fiber Evidence?**

John A. Reffner, Dale K. Purcell, Lai Sze Wan and Laura Pritchard — John Jay College, CUNY

**Development of a Systematic Approach to Forensic Dye Identification**

Skip Palenik and Christopher Palenik — Microtrace, LLC

**Examining the Effects of Environmental Degradation on the Optical Properties of Manufactured Fibers of Natural Origin: Year One Review**

Kelly Brinsko — McCrone Research Institute



**The Role of Light Microscopy Examinations in the  
Discrimination of Textile Fibers in a Forensic Setting**

Patrick Buzzini — Forensic and Investigative Science Program,  
West Virginia University

*12:00 – 1:30 p.m. Lunch Break, McRI Garden*

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

*Chair: Peter Diaczuk, John Jay College of Criminal Justice, CUNY*

**A Look at the Origins of Chemical Microscopy**

Andrew A. Havics — pH2, LLC

**The Development of a Microcrystal Test for the Detection of  
Clonazepam**

Danielle Silletti — McCrone Research Institute

**Development of a Modern Compendium of Microcrystal Tests  
for Illicit Drugs and Diverted Pharmaceuticals: Year Two  
Research**

Sebastian Sparenga — McCrone Research Institute

**Horse Racing and the Art of Microchemistry**

Meggan King — McCrone Research Institute

**Airborne Pollen Recoveries on the Big Island of Hawaii**

Michael Muilenberg — Aerobiology Instruction and Research

**Takayama Test vs. Modern Forensic DNA Analysis**

Arthur Young — Guardian Forensic Sciences

**The Development of an SEM-EDS Method for Forensic Mineral  
Identification**

Jack Hietpas and Jefferey M. Davis — National Institute of  
Standards and Technology

**Analysis of Shotgun Shell Buffers**

Peter Diaczuk — John Jay College of Criminal Justice, CUNY

**WEDNESDAY, JULY 17**  
**STATE MICROSCOPICAL SOCIETY OF ILLINOIS**  
**2013 AWARDS DINNER**

*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.*

Join McCrone Research Institute and the State Microscopical Society of Illinois as they honor **Thomas J. Hopen**, the recipient of the SMSI 2013 Émile M. Chamot Award.



The award will be presented at the SMSI Awards Dinner at Mercat a la Planxa, 638 S. Michigan Avenue, Chicago.

*Thom Hopen has more than 30 years of experience in forensic science. He currently specializes in trace evidence investigations for the Forensic Science Laboratory (Arson and Explosives Section) at the Bureau of Alcohol, Tobacco, Firearms and Explosives in Atlanta, Georgia. He has also served as Senior Research Microscopist, a teaching and research position at the McCrone Research Institute in Chicago. Mr. Hopen has presented papers at numerous professional meetings on more than 30 different topics and has authored over 20 technical publications. He contributed a chapter on "Light Microscopy" in the Wiley Encyclopedia of Forensic Science (2009) and a chapter on "The Value of Soil Evidence" in Trace Evidence Analysis: More Cases in Mute Witnesses (2004).*

**THURSDAY AND FRIDAY, JULY 18–19**  
**WORKSHOP: MODERN POLLEN IDENTIFICATION**  
**AND AEROBIOLOGY**

*9:00 a.m. – 5:00 p.m., McRI Classroom*

**Taught by Michael L. Muilenberg**, Senior Research Fellow in Public Health at the University of Massachusetts and an Instructor at Harvard School of Public Health in Boston.

This two-day Inter/Micro workshop will introduce the microscopist to modern pollen, particularly its morphology and aerobiology (i.e. sources, release, dispersal and effects). Modern pollen can appear quite different from fossil pollen (i.e. from quaternary or more recent sediments) in that the protoplasm and inner wall of the modern pollen grain often remain intact. Attendees will learn to recognize microscopical characteristics of a variety of common airborne pollen types through presentations and microscopy (using both prepared reference slides and air-sample slides).

In addition to identification, aerobiological aspects will also be discussed (floral biology and pollen release mechanisms, airborne concentrations, seasonality, approaches to sampling for pollen from air, etc.). Attendees will become familiar with the morphological structures necessary to identify pollen grains and learn to identify some of the more commonly encountered airborne pollen types. They will also learn the basic principles of airborne pollen sampling and become familiar with the advantages and disadvantages of different pollen sampler types.

*Michael L. Muilenberg is a partner in Aerobiology Instruction and Research, LLC (AIR), an educational and consulting company. He has extensive experience in the set-up, operation, evaluation, and theory of aerobiological sampling equipment, and has broad knowledge of both pollen and fungal spore identification.*

## **PRESENTATION ABSTRACTS**

**MONDAY, JULY 15**

### **TECHNIQUES AND INSTRUMENTATION**

#### **Raman Analysis of Carbon Materials**

Ken Smith — Thermo Fisher Scientific

Increasing Raman spectroscopy has been used for many years in the analysis of carbon materials such as diamond and graphite. Carbon materials have very strong Raman scattering properties, which makes Raman a preferred technique for the molecular characterization of the various phases and morphologies of carbon. With the significant current research efforts to make, modify and utilize carbon materials such as nanotubes and graphene in advanced composite materials and electronics, Raman microscopy has become an invaluable tool for probing carbon materials down to the single atomic layer. Applications of Raman microscopy in the characterization of these new materials will be presented.

#### **Increasing Depth of Field in Photomicrography by Using Focal Stacking Techniques**

Julian C. Gray — Tellus Science Museum

Photographers striving to produce crisp images of microscopic subjects are challenged by decreasing depth of field at high magnification. Several computer software packages currently available make it possible to create sharp images with extended virtual depth of field. In this technique, called focal stacking, multiple images are taken with different parallel planar slices of the subject in sharp focus. The software retains only the sharpest regions of each respective image to create a composite image with all areas of the subject in acceptable focus. To optimize this technique, consecutive focal slices should overlap by half the depth of field as determined empirically using trigonometry and

a stage micrometer at a known angle to the optic path. Some means of moving the subject in controlled and precise increments are employed (microscope focus knob, stepping motor or z-axis stage micrometer). This technique provides an efficient way to overcome the depth of field limitations inherent in single-shot photomicrography. The focal stacking technique can be used with great success with subjects viewed in reflected or transmitted light, and will therefore find applications in a wide array of scientific imaging fields.

### **Mixed Greens — A Closer Look at Currency Using Photomicrography**

Martin Kocanda — Northern Illinois University, Department of Electrical Engineering

Paper currency has been used extensively throughout Western civilization. Attempts have been made to duplicate currency using printing, scanning and photocopying. Schemes have been employed to mitigate fraudulent currency such as watermarks, embedded threads and micro printing. In this work, techniques that have been employed in domestic and foreign currency fabrication are presented using digital imaging.

### **An Examination of the Fiber Composition of Mid-20th Century Papers**

Walter Rantanen — IPS Testing Experts

This study examines the fiber composition for selected printing- and writing-grade papers of the mid-20th century. In that era, there were various fiber types used, which also reflect the economics and other factors that were developing at the time.

### **The Identification of Bicomponent Acrylic Fibers Using Dispersion Staining**

Jennifer DePirro — McCrone Research Institute

Bicomponent acrylic fibers can be hard to identify microscopically without performing infrared analysis. One technique that

may be useful in quickly identifying bicomponent fibers is dispersion staining. Dispersion staining utilizes the difference in refractive index between a sample and the liquid it is mounted in. This difference produces a color at the edges of the fiber. If the two polymers of a bicomponent fiber have different refractive indices, they will display different colors under dispersion staining. In this experiment, the dispersion staining colors of seven acrylic fibers were identified. Six of these fibers had dispersion staining color differences. Infrared analysis was then performed on the fibers. The same technique was then performed on four fibers from the Microtrace Reference Collection.

### **Can Raman Spectroscopy Save the Polarized Light Microscope?**

Andrew M. Bowen — U.S. Postal Inspection Service

Chemical microscopy is a field with few practitioners, which is surprising for those of us who work in the field and regularly witness the tremendous power of polarized light microscopy (PLM) in solving analytical problems. In my opinion, some of the most powerful applications of PLM exploit its ability to visually discriminate different sample components, including the differentiation of various hydration states, polymorphs, and particles of identical composition that were processed differently. This enables microscopists to visually “separate” components that are challenging to physically separate using traditional chemical methods. However, manual isolation of these particles is often required in order to obtain instrumental data for identification purposes. This is a daunting task to those who are inexperienced. The necessity of physically isolating extremely small particles from complex mixtures may limit the application of PLM. Raman microspectroscopy, on the other hand, makes the physical separation and isolation unnecessary. Particles can be analyzed directly through a cover slip without additional sample preparation. This means that the visual discrimination ability of the polarized light microscope can be coupled with a powerful spectroscopy technique, on a microscopic scale, without the need to even sharpen and use a tungsten needle, the traditional instru-

ment for handling small particles. For this reason, I believe that Raman microspectroscopy has the potential to revive the use of PLM and further develop the field of chemical microscopy.

### **Between the Fringes: Overlooked Topics in Microspectrophotometry**

Christopher Palenik and Jason Beckert — Microtrace, LLC

On the surface, the microspectrophotometer (MSP) is one of the more straightforward and commonly employed instruments in the forensic trace evidence lab — both conceptually and practically. The design, learning curve and maintenance is relatively straightforward compared to many modern analytical instruments. In its most basic arrangement, it consists of a light microscope coupled to a visible spectrometer. This arrangement is simple enough that we were able to build our own MSP system, which performed well enough in the visible region to apply to casework. After having built our own system as well as having used a variety of commercial systems over the years, we have encountered a variety of seemingly minor spectral features and results that have arisen (either during experimental setup and sample analysis or were noted during spectral interpretation) that can have a major impact on not only the quality of data but on its interpretation.

This talk will present a survey of overlooked and often dismissed subtleties that arise during micro-colorimetric analysis. Topics will include MSP instrument evolution, choice of illumination; sample alteration, spectral variations in the low and high energy regions of the spectrum, a discussion of the evidentiary significance of fluorescence imaging and microspectrofluorimetry data, and spectral comparison as it relates to forming conclusions. The talk will not provide final solutions but instead will raise these issues and possible ways one might address them.

## **Colorant Basics: Chemical Organization of a Dye and Pigment Database**

Ethan Groves and Christopher Palenik — Microtrace, LLC

Colorants are the materials that make the world around us vivid. They are added, in some form, to virtually every commercial product. These colorants can be separated into two major classes: pigments and dyes. The limitless array of available colors is often a result of the use of several colorants together to obtain a final product appearance. In fact, there are more than 500 commercial colorants in use today. Characterization and identification of a pigment or dye from the ever-growing population of colorants allows for the potential to more specifically associate or dissociate two samples during a forensic comparison. In some cases, identification of a colorant can provide information about the source, end-use or manufacturer of a product. Maintaining an authenticated and up-to-date reference collection is the best way to facilitate colorant classification and identification. The building of such a database requires many considerations, including colorant chemistry, source information and replicate samples, to name a few. This talk will provide an introduction to colorant chemistry and the imperfect systems of colorant categorization.

## **Exposing the Myth of Creationism**

Brian J. Ford — Caius College, University of Cambridge

The prevalence of blind religious faith in civilizations across the world remains an enigma. Surveys show that about half of Americans believe in creation — yet the notion that intelligent design lies behind living organisms cannot be sustained. Humans have poor sight (our retina is the wrong way round), we take air, food, and drink through the same orifice (which kills people in alarming numbers) and we are rich in superfluous trouble-causing features from the appendix to tonsils.

People are so badly designed that most suffer back pain simply because of maintaining an upright stature. There are similar



problems across the living world of animals and plants. All the world's religions imagine God as a wise and forgiving Father; yet if such a being had designed humanity (and other forms of life) they would have had to be both thoughtless and incompetent. However people regard their deity, none believes that God is an idiot — but He'd have to be, if we were to delve into intelligent design.

### **World's Worst Microscopy — The New Normal?**

Andrew A. Havics — pH2, LLC

Several years ago at Inter/Micro, Professor Brian Ford introduced the subject of poor microscopy and microscopical technique in mainstream science and media by coining the phrase, “world's worst microscopy.” I have gathered several examples of such work but seek to probe and explore a theory that these are not just chance cases of poor microscopy or microscopical training, but are the result of microscopy being perceived as a commonplace utility of the masses instead of an instrument of the professional. This has become the “new normal.”

### **Use of High Dynamic Range (HDR) Techniques to Increase Exposure Range in Photomicrography**

Julian C. Gray — Tellus Science Museum

Microscopic subjects occasionally have extremes of light and dark areas that fall outside the optimal range of exposure values that can be properly recorded by digital photography equipment. In these situations, photographers are forced to compromise and chose an intermediate exposure. Resulting images will have poorly resolved light areas, dark areas or both. It is now possible to overcome this high dynamic range (HDR) problem with computer software. To employ this technique, separate images are taken at various exposure values (EV) that bracket the lightest and darkest subject areas. The HDR software selects areas of each respective image with an optimal exposure and retains only that information to produce a final composite image with all ar-

eas of the image acceptably exposed. Experimentation on a given subject may be necessary to determine the correct EV step interval (one-half, one-third or whole steps) and range (+/- three to five EV) that will produce the best final result. This technique is rapid, easy and efficient, and solves an issue that commonly plagues photomicrography.

### **Low Vacuum Scanning Electron Microscopy (SEM) of Polymers**

Rich Brown, MVA Scientific Consultants

The ability to bleed air (or an inert gas) into the SEM sample chamber allows imaging of non-conductive materials without the application of a conductive coating such as carbon (evaporated) or gold (evaporated or sputtered). The immediate advantage is the speed at which a sample, even a sample on a filter membrane in a plastic holder can be popped into the SEM and examined. The use of a backscattered electron (BE) detector allows rapid discrimination between carbon-rich materials, inorganic materials and mixtures. Further discrimination is possible between different polymer blends using the atomic number contrast inherent to the BE detector. Energy dispersive X-ray spectrometry is also possible while operating in the low vacuum (high pressure) mode. Scanning electron microscopy-energy dispersive X-ray spectrometry (SEM-EDS in the low vacuum mode requires some experimentation as the apparent beam size that one grows accustomed to during high vacuum (normal) SEM-EDS operation becomes quite large resulting in EDS data from areas around the object/particle of interest requiring additional interpretation.

### **Seeing Food Differently: X-ray Tomography**

Robert Myers — Wm. Wrigley Jr. Co.

Advanced product development and innovative research can be greatly enhanced by understanding the internal structures of food products. X-ray tomography allows the researcher to visualize the internal structures with limited sample preparation. The sample can be “virtually sliced” in any desired direction. This

presentation will briefly describe the theory of X-ray tomography. It will also show virtual slices of various food products and discuss the information that can be gleaned from these virtual slices.

#### **X-ray Tomography: Segmentation and Image Visualization**

Philip W. Urnezis — Wm. Wrigley Jr. Co.

X-ray tomography is a non-destructive technique that allows the interior of an object to be imaged without having to slice it open. Multiple 2-D X-ray images are taken of the object and then computer reconstructed into a 3-D depiction. The ability to “see” the internal structure of an object depends on the ability to separate (segment) domains from each other. If the different domains cannot be segmented from each other, they cannot be visualized as different domains. This presentation will discuss data collection parameters that can affect segmentation and visualization.

#### **Instrumental and Microscopical Analysis of Artificial Sweeteners**

Andrew M. Bowen, Vincent J. Desiderio, Robert B. Moberley —  
U.S. Postal Inspection Service

After the 2001 Amerithrax investigation involving mailed letters with anthrax spores, it became relatively common for copycat individuals to include powders in threatening communications. The U.S. Postal Inspection Service is frequently involved in investigating these cases, and as a result their National Forensic Laboratory regularly receives submissions containing unknown powders. While some of these powders are hazardous, most are common household products. The majority of these are fairly straightforward to identify; however, some materials pose analytical challenges for the typical laboratory. Several of the commonly used artificial sweeteners are so sweet that they are present in very small quantities in the final consumer product. The vast majority of the powder in these products consists of fillers (commonly dextrose). The National Forensic Laboratory has re-

ceived artificial sweeteners as evidence from threatening letters in the past, prompting the laboratory to acquire and characterize standard artificial sweeteners using a wide variety of analytical techniques, including stereomicroscopy, polarized light microscopy, Fourier Transform-infrared spectroscopy, scanning electron microscopy, energy dispersive spectroscopy, X-ray diffraction, Raman spectroscopy, and liquid chromatography-mass spectrometry. Preliminary results from this research will be presented.

### **ATR Imaging with a FTIR Microscope**

Ken Smith — Thermo Fisher Scientific

FTIR microscopy is a tool for sample characterization that has been utilized in a wide range of industries and applications. However as materials become more complex and feature sizes get smaller, the spatial limit of the FTIR microscope for discrete analysis in a standard transmission or reflectance mode is being tested. ATR imaging offers a means of reaching to smaller domains through the use of high NA ATR crystals. Applications in the areas of polymer laminates and composites will be discussed to demonstrate the utility of ATR imaging.

### **An Evening with Brian: “Movie Star Microbes”**

Brian J. Ford — Caius College, University of Cambridge

This year Brian looks at microbes in the movies. Living cells and microorganisms have been featured in films, occasionally in starring roles and in main title sequences. This evening we will encounter some remarkable examples. Films rarely do justice to the microbe world, but the representations seen in the cinema and on television are revealing and lead to a crucial conclusion: Filmmakers are missing a major opportunity to capture the microscopic world. It is the single theme that Hollywood has yet to explore, and the possibilities are endless.

**TUESDAY, JULY 16**  
**ENVIRONMENTAL AND INDUSTRIAL**  
**MICROSCOPY**

**The New Trend in Optical Microscopy, Digital Imaging and Measurement: The Difference Between Optical and Opto-Digital (Digital) Microscopes**

Christopher Vander Tuuk — Olympus America

Modern industrial QA/QC, FA and R&D labs tend to prefer turn-key laboratory equipment that can provide accurate and repeatable results, often over multiple shifts and with multiple users, and can be certified against a known standard. As industry and manufacturing continues to trend towards the micro and nano levels, the need for high-magnification and high-resolution imaging and measurement is as important as ever. Optical microscope systems fill that need by providing a non-destructive, non-contact way to image and measure very fine detail for a wide variety of materials and applications. Traditional optical microscope and digital imaging systems are still very popular and meet the majority of the needs of the modern lab. However, visual and digital measurements through an optical microscope system are not certified for accuracy and repeatability, and it is often difficult to duplicate observation conditions from one user to the next making consistent images a challenge. The latest Opto-digital microscopes have been designed to address the limitations of traditional optical microscope systems by providing a turn-key digital imaging and measurement solution that is certified for accuracy and repeatability. This talk will compare and contrast the Optical and Opto-Digital microscope systems.

**Trace Evidence in Industrial Problem Solving**

Kevin Brady — Tredegar Film Products

Locard's exchange principle — "Every contact leaves a trace." — is fundamental to forensic science investigations. This principle is

not limited to crime-scene investigations — it also serves as a useful guide in solving industrial problems. A few examples of trace evidence collection and an analysis in an industrial manufacturing environment will be presented.

### **Microscopy of Some Bitumen, Asphalt and Mastic-Based Building Materials**

Andrew A. Havics — pH2, LLC

Over the years, the terms asphalt, bitumen, and mastic have been applied to a variety of components used in building materials. The formal terms of each will be explored, and the uses and investigative aspects of each, using specific applications, will be presented. Mastic (used in the general sense) has been used in floor tile adhesives, roof and building envelope flashings, waterproofing, and other applications. Many have contained asbestos as a filler and/or rheologic agent. The examination of these materials under these circumstances involves several separation techniques, including solvent separation, ashing, freeze fracturing, general heating, and Soxhlet extraction. Polarized light microscopy (PLM) is the instrument of choice in the follow-up examination, although scanning electron microscopy (SEM) and transmission electron microscopy (TEM) can be advantageous. Similar, but less difficult, preparation is used for asphalt-based materials such as pipe wrap, panel wrap, flooring, and roofing. Nevertheless, PLM is still the most appropriate instrument. Bitumen has been used in many roofing and waterproofing applications, both hot and cold applied. These are best compared with mastic in terms of basic examination, although they contain both asphaltenes and lower molecular weight oily maltenes. Outside of looking for asbestos or fillers and rheology agents, each of these products can also be evaluated for performance issues. Bitumen has been examined for phase analysis by micro-Fourier Transform Infrared ( $\mu$ -FTIR) spectroscopy and hot stage microscopy. Of these products, bitumens, in particular, have been modified with plasticizers for a greater range of temperature and better aging characteristics in a host of applications such as road

paving, hence the term “modified bitumen” or “mod-bit.”. Investigation of the chemical composition and interaction of these (especially changes from thermal response and aging) is well served by  $\mu$ -FTIR and hot stage microscopy. However, the morphology and matrix interaction is best examined using epifluorescence microscopy, although transmitted fluorescence of cold-microtomed thin-sections is possible. Sometimes,  $\mu$ -FTIR or fluorescence microscopy is combined with hot stage microscopy for a more accurate understanding of the material. Often times, other non-microscopical tests (softening point, gel permeation chromatography, etc.) are used to find the correlation between micro-structure and macro-response; but the micro-structure as revealed by microscopy can be the only true way of understanding these components.

### **Fitting the Mold: An Exploration into Sourcing of Glass Fragments**

Brendan Nytes — Microtrace, LLC

Glass is a commonly encountered type of trace evidence in forensic science. Typically, questioned glass particles are submitted with a known source, and analyses are comparative in nature. Such samples are commonly analyzed by X-ray fluorescence and/or high precision refractive index measurement to determine if they could originate from a common source. In many cases, particularly those involving consumer complaints in the food and pharmaceutical industries, these alleged contaminants are rarely accompanied by comparison samples. In such cases, the analyst must obtain as much information as possible from what may be a single, small fragment of glass. Ultimately, the goal is to provide information that addresses the related questions of glass type, shape of the original object, potential applications for such glass, and possible sources. Elemental composition and refractive index methods, typically used for comparisons, can in many cases provide general compositional classification, i.e. borosilicate vs. soda lime. Other characteristics such as color, surface shape and texture, thickness, and fluorescence can provide fur-

ther insights and constraints on possible sources. In some cases, the presence of exotic trace elements in the glass composition can further limit the list of potential sources. In other cases, more advanced analysis by transmission electron microscopy (TEM) can indicate phase separation within the glass or the presence of colloidal metal particles. Taken together, the complete characterization of an unknown glass particle can provide a great deal of investigative information about the possible history and source of a glass fragment.

### **Charred Starch: Characterization by Microscopy, Staining and Spectroscopy**

Katie White and Skip Palenik — Microtrace, LLC

Ungelatinized starch can be readily identified by polarized light microscopy (PLM). Other methods such as staining and spectroscopy can provide additional information or aid in the identification of gelatinized starches. But what happens when dry starch is exposed to increasing amounts of heat? In the absence of water, starch will char, eventually altering its carbohydrate chemistry, which leads to deterioration of its spherulitic structure. Ultimately, charring proceeds until the starch is converted into elemental carbon and is no longer identifiable. Because unwanted chars often arise in a variety of manufacturing processes, particularly those in the food, cosmetic and pharmaceutical industries, it is useful to know the limitations of these detection methods and at which point in the charring process they cease to be effective. In this study, dry starch grains were heated to varying degrees and then characterized by 1) microscopic morphology and optical properties, 2) iodine-potassium iodide staining and 3) infrared absorption spectroscopy. These three parameters were monitored for each of the charred samples. This presentation will present the results of these experiments and discuss their implications for the analysis and identification of charred starch.



### **Aquatic Dinosaur Update**

Brian J. Ford — Caius College, University of Cambridge

Studying the energy demands of single cells makes nonsense of giant dinosaurs holding their tails aloft. Yet the new “aquatic dinosaur” theory, which was expounded at last year’s Inter/Micro, attracted the universal condemnation of palaeontologists. Recent findings — including the fish scales in the digestive tract of spinosaurs and oxygen isotope ratios of dinosaur bones that are unique to aquatic creatures — have supported the theory. This year, additional evidence is being published, and some fossil trackways now confirm that dinosaurs were wading and swimming. We will review the latest research in this presentation.

### **A Study of Select Refractive Index Liquid Stability, Series A and Series AA**

Wayne Moorehead, Kaycee Fontes and Cassandra Hayes — OC Crime Lab, Santa Ana, CA

Refractive index liquids are used to identify unknown particles or limit possible identifications before they are confirmed by instrumental methods. After the refractive index liquids are opened and stored in a cool, dark place in the laboratory for months or years, questions arose about their sustainability during this time. The laboratory had several different sets of refractive index liquids (labeled as sold by Cargille Laboratories Inc.) covering a wide range of values.

Additional refractive index liquids were obtained, including sets reportedly manufactured in a geology lab at one of the local universities with a provided calibration-check performed in the early 1970s for each liquid, a set of Shillaber labeled liquids and a set of Ward’s labeled liquids. A Digital Leica Abbe refractometer with RS-232 output was used to perform the refractive index determinations. The instrument was permitted to equilibrate for at least one hour before use. The refractometer’s calibration was checked with 18 ohm deionized water with temperatures recorded before use. We found that the Cargille refractive index

liquids maintain stability for many years. Due to the upper limit of the refractometer, measurements above 1.700 were not possible.

### **Identification of Contaminant Particles and the Determination of Syringe Penetration Angles in Pharmaceutical Vials with Rubber Stoppers**

Jason Beckert, Christopher Palenik and Ethan Groves —  
Microtrace, LLC

The identification of contaminant particles in pharmaceutical vials is a task befitting well-trained chemical microscopists. It generally involves the careful isolation of microscopic material and the subsequent analysis by one or more applicable analytical techniques. Polarized light microscopy is typically the first method employed, and the information obtained here determines subsequent steps (if required): often chemical (e.g. FTIR and/or Raman) or elemental (e.g. SEM-EDS) analyses. A thorough analysis may be able to suggest, or in some circumstances, prove the origin of the contaminant particle(s). Occasionally, additional information is requested from the examining scientist. This talk will first briefly discuss a case in which the contaminant particles from several pharmaceutical samples were shown to originate from the rubber stoppers used to cap the vials. However, the majority of this presentation will focus on the client's wish to estimate the syringe penetration angles through the rubber stoppers. Additional experimentation was subsequently conducted to evaluate the accuracy of these estimates.

### **Smokestacks — Can't Live With Them, Can't Live Without Them!**

Wayne C. Isphording — Tulane University School of Continuing Studies

Modern industrial processes frequently employ high-temperature furnaces for a number of reasons. These include concentration of metals from ore (or scrap), the generation of electric power, production of building materials, and manufacturing of

paper products, just to name a few. A consequence of these processes, however, is the generation of waste particulate (and volatile) phases. While modern industrial operations remove the bulk of these particulates by means of electrostatic collectors and baghouse filtration systems, varying quantities escape trapping and are emitted from smoke stacks into the atmosphere. Ambient wind currents can then distribute these over wide areas and, in some instances, they may pose either health hazards or cause property damage. A major problem with the former is the fact that, regardless of the collection system employed, the particles are frequently of a size that the EPA considers “easily respirable” and potentially harmful (i.e.,  $<2.5\ \mu\text{m}$ ). This presentation will describe several examples of particulate trespass and the microscopic evidence that can be used to obtain relief and redress by property owners impacted by airborne industrial debris.

### **The Microscopy of Asbestos Products: Case Studies of the Unique and Interesting**

Steven P. Compton — MVA Scientific Consultants, Inc.

The use of the “miracle” product, asbestos, spawned some common and widespread uses that many people are familiar with such as insulation, cement and other building materials. There were some more imaginative and uncommon uses as well: fire suits, cigarette filters and backyard fencing. A collection of unique asbestos products and fiber release studies performed at the laboratories of MVA Scientific Consultants will be presented along with analytical results from polarized light microscopy, phase contrast microscopy, scanning electron microscopy and transmission electron microscopy.

### **Dispersion Staining: What’s the Objective?**

Peter M. Cooke — MICA

The majority of U.S. environmental laboratories offering bulk PLM asbestos analyses measure refractive indices using an optical focal-screening method widely called dispersion staining. Fifty years ago, W.C. McCrone and K.M. Brown published *Disper-*

*sion Staining: Part 1 — Theory, Method and Application.* McCrone popularized the method for routine bulk asbestos analysis. Cherkasov, Dodge, Crossman, Speight, Wilcox, Clarke and others have discussed dispersion staining (focal screening) or similar methods for various uses. All these methods are based on the manipulation of the Becke line colors known as the Christiansen effect. This presentation traces the evolution of commercially available dispersion staining objectives that have been offered in the last 50-plus years. The dispersion staining objectives are evaluated; included are the objectives used in the Japanese Industrial Standards 1481 and 3850-1.

#### **Effects of Hydration on Erionite Refractive Index Measurement**

Lou Solebello and Frank Ehrenfeld — International Asbestos Testing Laboratories, Inc.

Erionite is a fibrous zeolite classified as a Class I IARC carcinogen that can produce mesothelioma-like cancer and respiratory disease similar to asbestos. Awareness of erionite as a health hazard became evident from exposure studies performed in the 1970s in the Cappadocia region of Turkey. Unlike asbestos, erionite has not been used extensively in the manufacture of building and industrial materials. Erionite exposure risks can arise from mining of volcanic rock for aggregate and disturbance of soil/rock during roadway construction in populated areas. It can be difficult to identify and differentiate fibrous and elongated zeolites from each other due to similarities in chemistry, optical properties, and structure. Previous studies using a bench formulated 1.48 high-dispersion refractive index liquid indicate the potential for central-stop dispersion staining (CSDS) as a technique for the identification of erionite and other elongate zeolites. The identification and classification of zeolites is, in part, based on structural water content that is not affected by changes in relative humidity. Most zeolites also have pore spaces (zeolitic channel) that can accommodate moisture (relative humidity changes), which may affect refractive index measurement. This study was conducted to assess the potential affect of moisture

content on the refractive index measurement of erionite. A 1.47 bench-formulated high-dispersion refractive index liquid was used to measure the refractive indices of a traceable specimen of erionite from Durkee, Oregon after equilibration in an environmental chamber at controlled relative humidities. Results of the study performed for WK39550 “New Standard Polarized Light Microscopy Analysis of Erionite in Soils and Gravels” will be discussed.

### **Comparative Characterization of Erionite Specimens from Three Locations**

Lou Solebello and Frank Ehrenfeld — International Asbestos Testing Laboratories, Inc.

Awareness of the zeolite mineral erionite as a health hazard emerged as a result of exposure studies performed in the 1970s in the Cappadocia region of Turkey. The studies conducted then have indicated that erionite can cause mesothelioma-like cancers and respiratory disorders similar to asbestos. As a result, erionite was classified as a Class I IARC carcinogen (together with asbestos) but is currently not regulated as an asbestos mineral. Current research (NIOSH roadmap, EPA/North Dakota investigation) may lead to future changes in the regulation of erionite as a carcinogen. Erionite (unlike asbestos) has not been used extensively in the manufacture of building and industrial materials. Potential erionite exposure risks arise mainly from mining of volcanic aggregate used in construction and soil disturbance during engineering activities that contain zeolites in populated areas. Identification of erionite can be difficult due to similarities in chemistry, optical properties, and structure as compared to other fibrous and elongated zeolites. Traceable specimens of erionite from Rome and Durkee, Oregon in the United States and from Karain Cave in Turkey were characterized using TEM, SEM, XRD, and PLM techniques for ASTM WK39550 “New Standard Polarized Light Microscopy Analysis of Erionite in Soils and Gravels.” Results of the comparative characterization will be discussed.

### **What Causes Some Mineral Fibers to be Carcinogenic?**

Eric J. Chatfield — Chatfield Technical Consulting Limited

Ever since the recognition in the middle of the last century that mesothelioma was associated with asbestos exposure, significant research has been made to determine the mechanisms involved in causation of this disease. These research efforts have included epidemiology, in vitro experimentation, implantation in animals and inhalation in animals. Although at first it was thought that mesothelioma always implied exposure to asbestos, epidemiology and animal studies have shown otherwise. In 1974, it was discovered that some villages in a region of Turkey had a very high incidence of mesothelioma among inhabitants, but that there appeared to be no asbestos exposure involved. Investigations showed that a fibrous zeolite mineral, erionite, was responsible. More recently, it has been determined that a cluster of mesothelioma cases in the Biancaville area of Sicily are associated with exposure to a fibrous fluoroedenite. Animal experiments conducted by Stanton in the 1970s also showed that mesothelioma could be produced by a whole range of fibrous minerals, synthetic fibers and glass fibers, provided that the dimensions of the fibers were within specific ranges. Therefore, it appears that a carcinogenic response can be obtained, regardless of the chemical compositions of fibers or whether fibers are crystalline or glassy, provided that the fibers are sufficiently durable to remain in tissues for an extended period.

A significant issue for the mining and crushed stone industries is the presence of non-asbestiform amphibole in some deposits. Although amphibole asbestos is justifiably regulated because of its known carcinogenicity, the Occupational Safety and Health Administration concluded that there was insufficient scientific evidence to warrant regulation of non-asbestiform amphiboles with the same chemical compositions. Given that a carcinogenic response has been obtained from such a wide variety of fibers, both crystalline and glassy, the observed difference in carcinogenic response between amphibole asbestos fibers and elon-

gated fragments of the non-asbestiform analog is difficult to explain other than on the basis of differences in the fiber size distributions.

## **WEDNESDAY, JULY 17**

### **CHEMICAL AND FORENSIC MICROSCOPY**

#### **The Use of Blue-Light Curing Resins in Forensic Sample Preparation**

Ethan Groves and Christopher Palenik – Microtrace, LLC

In the evaluation of trace evidence, the internal structure of a sample can provide key information for a variety of evidence types (e.g. paint, polymers, paper, wood). Fixing media (i.e. resins) are used to hold a specimen in a particular orientation, which upon sectioning allow for the structure to be visualized. This will examine the properties of an array of readily available embedding media that were considered for the purpose of mounting and sectioning of paint specimens. In addition to the more common two-part epoxy systems, we have evaluated a blue-light curing acrylic resin (Tuffleye), which has several appealing features for embedding and sectioning and has shown to be useful for an array of sample types.

#### **SEM/EDX Microtrace Analysis in Forensic Casework**

P.D. Zoon, S.B.C.G. Chang and E.J. Vermeij — Netherlands Forensic Institute

A multidisciplinary team at the Netherlands Forensic Institute (NFI), consisting of forensic pathologists, forensic anthropologists, toolmark examiners and microtrace experts, work together to determine the manner of death in murder cases. The results obtained by each of the different disciplines are statistically independent of each other and can be readily combined. Combining the results of the four separate disciplines leads to an increase in evidentiary value or a better description of occurred events. Cases in which this integrated multidisciplinary approach is used include victims who have died as a result of sharp and/or blunt force trauma. During the forensic autopsy, potentially damaged bone is excised for subsequent examinations. The clean bone is



studied with optical light microscopy for the presence of foreign material prior to being studied with SEM/EDX in low vacuum mode. Obviously, microtrace analysis with SEM/EDX at the NFI is also employed in other types of cases as well. Forensic cases in which SEM/EDX microtrace analysis yielded interesting results will be presented. Examples include microtraces in bone and weapons as well as bullets and materials analysis.

### **A New Technique for the Identification of Dyes Extracted from Single Fibers**

Katelyn Hargrave and Skip Palenik — Microtrace, LLC

Fibers are commonly submitted as a form of forensic trace evidence. Color analysis of the dye on the fiber is essential for finding similarities or differences between samples. To increase information gained from extracted dye analysis, a new technique has been created to analyze dyes in solution taken from a thin layer chromatography plate. This talk will present this new technique to analyze extracted dyes from single fibers using a flat capillary, thin layer chromatography (TLC), microspectrophotometry (MSP), and microspectrofluorimetry (MSF). It will explain the new technique as well as the approach taken to improve the procedure. Comparisons of the same dye using UV-Vis spectra, solution MSP and MSF spectra; fiber MSP and MSP spectra; and extracted dye solutions from single fibers extracted from a TLC plate will be explored. Procedures for each method and how trace analysts can utilize the new technique will also be discussed.

### **Techniques in Cross-Sectioning Fibers, Tapes and Paints**

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

There are probably more methods than one could imagine regarding techniques on how to get "good" cross-sections of materials quickly. Yet, every time the topic is brought up at a meeting, there seems to be a flurry of discussion by the participants.

Questions arise about whether to embed or not to embed the material, which embedding medium is best and hand sections versus microtome sections. A light overview of common cross-sectioning methods will be presented, as well as video demonstrations of techniques the authors have found useful. Hopefully, this presentation will generate more discussion on the topic!

### **What is the Problem with Dichroic Fiber Evidence?**

John A. Reffner, Dale K. Purcell, Lai Sze Wan and Laura Pritchard – John Jay College, CUNY

Recent studies have shown that most dyed textile fibers are or appear to be dichroic. This is a property that is useful when questioned and known fiber evidence is compared to determine if they could possibly have come from the same source. However, it is uncommon for the fiber examiner to measure the dichroic spectra when making these comparisons. So, what is the problem? Well, there are several issues with measuring the spectra of dyed fibers. Most of the problems have been reported or alluded to, but many are ignored. When recorded properly, dichroic spectra provide strong additional information for fiber comparisons. When ignored, dichroic properties can lead to serious errors. The factors leading to errors in spectral analysis of dichroic fibers will be illustrated and discussed.

### **Development of a Systematic Approach to Forensic Dye Identification**

Skip Palenik and Christopher Palenik — Microtrace, LLC

Color is an obvious characteristic and an important comparison property of many types of trace evidence. For example, two, otherwise identical fibers could not have originated from the same source, if they exhibit different colors. Microtrace research into pigments has resulted in the development of a systematic method for pigment identification in trace evidence such as paints, plastics and inks. We have now turned our attention to developing a method to identify dyestuffs on fibers, which,

unlike pigments, are chemically bound to the substrate they color. The colors of evidential fibers are presently compared using comparison microscopy, UV-Vis microspectrophotometry (MSP) and, in exceptional cases, thin layer chromatography (TLC). The first two methods show that the colors of the known and questioned fibers are the same. TLC of extracted dyes can show that the colors were obtained using the same dyes. Our current research is aimed at increasing the evidential value of such examinations by going beyond mere comparison of the dyes (by color and Rf value) to identifying them by microchemical analysis. The key to achieving this is our in-house reference collection of dyestuffs consisting of approximately 5,800 physical specimens of dye powders or pastes, which we have assembled over many years. We have also collected shade card books produced by dye manufacturers, which contain physical samples of different fibers dyed with their dyestuffs, often using different concentrations and sometimes in combinations of two or more of their dyes. These collections contain approximately three times the number of dyed fiber specimens as in our dye powder collection. Using a variety of newly developed methods and microtechniques we have started to characterize these dyes both on the fiber and after extraction, isolation and concentration. This presentation and several others by Microtrace staff at this meeting will report on the status of this research at the present time.

**Examining the Effects of Environmental Degradation on the Optical Properties of Manufactured Fibers of Natural Origin: Year One Review**

Kelly Brinsko — McCrone Research Institute

Beginning in 2012, McCrone Research Institute was awarded federal monies to conduct a three-year research study investigating the effects of environmental degradation on the optical properties of selected fibers: polylactic acid, azlon and rayon. These fibers are produced from naturally occurring polymers (proteins, sugars or cellulose), and little is known about the changes occur-

ring in their optical and physical properties as an effect of moisture, sunlight exposure and exposure to various temperatures. For the past year, fabric swatches representing each fiber type have been exposed to freshwater, saltwater, heat, cold, ultraviolet light and compost conditions. Every eight weeks the swatches were analyzed with polarized light microscopy, and various observations were made, including morphology, pleochroism, refractive index, birefringence, extinction characteristics and sign of elongation. Infrared spectra were also collected as part of this analysis. In addition, solubility and melting point behavior were assessed every six months. This presentation will summarize observations to date, as well as expected trends for the next year.

### **The Role of Light Microscopy Examinations in the Discrimination of Textile Fibers in a Forensic Setting**

Patrick Buzzini — Forensic and Investigative Science Program,  
West Virginia University

Forensic examinations based on the applications of light microscopy have been used for decades to characterize unknown textile specimens. After the initial and imperative step of fiber characterization, the forensic scientist typically conducts comparative examinations between questioned specimens and reference samples, classically using a comparison microscope. Therefore, one of the most important criteria to the selection of a forensic technique is its ability to distinguish features that vary in a given population. In this presentation, the discriminating abilities of light microscopy will be presented following two approaches. The first is concerned with the discrimination of different fiber type/color combinations among the most commonly encountered in casework, including blue, black, and red acrylics, cottons, and wools. The discriminating role of microscopical examinations is discussed in the context of an analytical sequence that includes UV-Vis microspectrophotometry, thin layer chromatography, and Raman spectroscopy. The second part consists of detecting, characterizing and comparing purple fibers from movie

theater seats to show how unlikely it is to recover a large number of matching fibers by chance on a given public surface. The results combat the defense argument that “the recovered fibers are common and ubiquitous; therefore this recovery is valueless to this case.”

### **A Look at the Origins of Chemical Microscopy**

Andrew A. Havics — pH2, LLC

Although microscopy was solidly on its way by 1800, microscopists (and particularly chemists) had not embraced the microscope as a tool for solving chemical problems and answering chemical questions. Warren de la Rue was perhaps the first to combine microscopy and chemistry, but his work was less in insight and intrigue than Edward Craig, who published his *Remarks on Microscopical Chemistry* in 1836. Craig was apparently unaware of Tobias Lowitz’s investigations into the characteristic forms of the chlorides of the elements as well as the researches of the “microchemist” Raspail. There was also Andrew Pritchard, a well known optician and microscope maker, who was certainly a promoter of the combination of microscope and chemicals. However, it was F.V. Raspail who effectively coined the term “chemical microscopy” with his *Essai de Chimie Microscopique* in 1830 and drove the philosophy of the microscope as a tool to investigate and solve chemical inquiries and issues. Raspail did this despite a microscope of poor utility. At that time, there was also Charles Chevalier who had supplied numerous French scientists, including Dumas the chemist, with his Universal microscope. These microscopes could be used inverted and could also be specially fitted with electrical apparatus for observing electrochemical reactions. The French Raspail and Chevalier microscopes, however, were made obsolete for chemical studies by the introduction in 1851 of the inverted chemical microscope designed by the American J. L. Smith. It was first made and exhibited at the London Exhibition of 1851 by the Parisian optician Nachet. Following on the heels of Craig and Raspail, it was Warrington who studied the adulteration of China tea from a chemi-

cal perspective. Warrington showed that tea leaves achieved their color from a microscopic coating compound of white, orange, and blue particles. Separation under a microscope followed by chemical tests revealed the true nature of these adulterants to him. Along a similar vein was Jonathan Pereira, the pharmacist, who advocated use of the microscope for controlling the quality of starting materials for the preparation of pharmaceuticals and also for identifying particles such as pollen and starch. This was evidenced by his work, *The Elements of Materia Medica and Therapeutics* in 1849. Each contributed to the early development of chemical microscopy — and set the stage for the likes of Chamot, Mason and McCrone.

### **The Development of a Microcrystal Test for the Detection of Clonazepam**

Danielle Silletti, McCrone Research Institute

Microcrystal tests are chemical microscopy techniques that date back to the 1800s. They have been used as an analytical technique for the identification of certain drugs or classes of drugs in the forensic sciences. Microcrystal tests are relatively inexpensive, fairly simple to conduct and use very little drug sample. However, there are disadvantages to this technique, including the need for comparison standards and a relatively “pure” sample (some adulterants may cause interferences). While some drugs, such as cocaine and heroin, have well-validated microcrystal tests, other classes of drugs, i.e. the benzodiazepines, have not been extensively studied for this purpose. Clonazepam is a benzodiazepine drug is sold under the trademark name Klonopin. It is known for its sedative and hypnotic properties and has therapeutic uses as an anticonvulsant and anxiolytic. Benzodiazepines have been abused for these sedative effects, but are more popularly known for their use in drug-facilitated sexual assaults. In the past decade, emergency department visits involving the abuse and misuse of benzodiazepines have increased, indicating the benefit of validated microcrystal tests for their detection. The goal of this project was to optically characterize

and develop a microcrystal test that could be used to detect clonazepam.

**Development of a Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals: Year Two Research**

Sebastian Sparenga — McCrone Research Institute

This talk will give an update of the grant that McCrone Research Institute received through the National Institute of Justice (NIJ-2011-2805 SL# 000944). Research during this second year has focused on performing microcrystal tests on the drugs to record how sensitive the tests were, as well as how quickly crystal formed. These tests are also being performed on various delivery methods such as tablets, liquids and gels. Highlights of these tests will be presented along with some of the difficulties encountered throughout testing. A plan for the remainder of the year, including work on the optical properties of the test precipitants, will also be discussed.

**Horse Racing and the Art of Microchemistry**

Meggan King — McCrone Research Institute

The search for a microcrystal test for methylphenidate (Ritalin) lead back to a 1950s-era horse racing laboratory run by Dr. Elsie S. Bellows. Dr. Bellows was a director of the Florida State Racing Commission Laboratory and a president of the Association of Official Racing Chemists (AORC). Part of a current research grant involves determining, locating and compiling analytical data and literature from Dr. Bellow's work and numerous other sources, which after many decades are out of print or difficult to locate. This task provided an opportunity to learn about Dr. Bellows and refocus her work under the microscope.

### **Airborne Pollen Recoveries on the Big Island of Hawaii**

Michael Muilenberg, Aerobiology Instruction and Research

To explain pollen prevalence and patterns of potential exposure, as part of a larger study of exposure to airborne agents and human respiratory health on the Big Island of Hawaii, pilot airborne pollen data was collected during three summer months in 2010 in Pahala (Ka'u district) using a Burkard recording sampler on a one-story rooftop. Pollen and wind direction were analyzed at two-hour intervals each day. A total of 18 pollen morphological types were identified with a total pollen geometric mean (GM) 24-hour concentration of 25 grains/m<sup>3</sup> (min.–max.; 1–136/m<sup>3</sup>). Predominant types included: *Cecropia*, *Trema*, *Broussonetia*-like, *Poaceae*, *Myrica faya* and *Myrtaceae* (including *Eucalyptus*). Meteorological influences included land breezes (NW direction) and sea breezes (E and SE direction), which predominated in Pahala. Precipitation seemed to play a relatively minor role in pollen concentrations although, on average, more pollen was recovered on dry than on rainy days. Also of interest is that all of the predominant pollen types recovered were from naturalized plant types not native to Hawaii.

### **Takayama Test vs. Modern Forensic DNA Analysis**

Arthur Young — Guardian Forensic Sciences

Experiments were performed to determine which was more sensitive, the classic Takayama microcrystal test or modern forensic DNA technology. In serial dilutions, the Takayama microcrystal test could produce positive results whereas no DNA results could be obtained at the same volume and dilution. When using microscopic flakes of known blood, both the Takayama microcrystal test and forensic DNA analysis were successful, however, the DNA analysis was only successful after the test substance was purified using the organic extraction method. Furthermore, when working without quantity limitations, it was determined that samples could be taken directly off of the microscope slide used for the Takayama microcrystal test and placed into forensic



DNA analysis with successful results when using the organic extraction method. When working with quantity limitations, the Takayama microcrystal test was still successful, whereas only a partial DNA profile was obtained.

### **The Development of an SEM-EDS Method for Forensic Mineral Identification**

Jack Hietpas and Jefferey M. Davis – National Institute of Standards and Technology

When properly collected and examined, soil ranks extremely high in evidential value primarily because earth materials vary widely from one location to the next. Despite the well-known utility of forensic geology, soil evidence is significantly under-utilized. The reason for this discrepancy is believed to stem largely from the lack of sufficient background of the forensic analyst to identify detrital minerals. This talk will present a method that couples polarized light microscopy (PLM) observations and Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS) microanalysis for high-throughput, accurate, detrital mineral identification. EDS spectra were modeled (using DTSA-II, Ritchie 2011) for ideal formula minerals, published mineral compositions, and microprobe mineral standards. The published analyses were treated as “unknowns” and the idealized compositions were treated as a reference database. The “unknowns” were used to query the database using Pearson Correlation. Preliminary results demonstrate that the database spectrum with the highest correlation score (top hit) correctly identifies the “unknown” mineral 64% of the time, with the correct database entry in the top five hits approximately 80% of the time. Subsequently, approximately 70 detrital mineral spectra were measured from sand collected in the Adirondacks, NY. These spectra were searched against the database and resulted in a 95% correct identification for the “top hit” when compared to PLM observations.

### **Analysis of Shotgun Shell Buffers**

Peter Diaczuk — John Jay College of Criminal Justice, CUNY

Buffers are not just for DNA analysts. In firearms, buffers are small polymer granules that are often added to shotgun shells to cushion larger sizes of buckshot pellets. The cushioning effect is important because maintaining the uniformity of the pellets during their sudden acceleration and subsequent travel down the barrel will allow them to maintain a stable, predictable flight to the target. When one of these buffered shotgun shells is fired, the polymer granules are also discharged with the pellets. This objective of this research was to determine whether different ammunition manufacturers use different buffers and, if so, can they be differentiated from each other? Being able to associate a sample of shotgun shell buffer to a specific manufacturer could offer useful information to shooting scene reconstructions. Several brands of ammunition were obtained, representative shotgun shells were dissected, and the buffers were examined microscopically. This study includes analysis of pre- and post-discharge material.

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