INTER/MICRO 2014

An International Microscopy Symposium
June 2 – 6, 2014
McCrone Research Institute, Chicago

Sponsored and hosted by

McCrone Research Institute
2820 S. Michigan Avenue
Chicago, IL 60616

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INTER/MICRO 2014

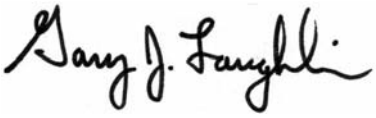
Welcome!

This year marks the 66th anniversary of the Inter/Micro conference, which was introduced in 1948 and is now held at McCrone Research Institute in Chicago. Inter/Micro gives you the opportunity to come up to date on new instruments, new techniques and new applications of microscopy.

Microscopy includes any instrument or technique that enables the microscopist to characterize, identify and study microscopic substances. This includes all light and electron microscopes, microspectroscopes, microprobes, automatic image analyzers and other microscopes based on X-rays, sound, protons, etc. Inter/Micro presentations from some of the world's leading microscopists will cover new techniques for improving contrast, increasing resolution, and obtaining and recording more characterization data. You will also learn how new techniques and new instruments are used in solving important problems.

Most papers presented at Inter/Micro will be considered for publication in *The Microscope*, the official journal for this conference. Papers are published in the order they are received during the year. Inter/Micro 2014 attendees are offered an introductory one-year subscription for $38.50 (regular rate is $75).

Thank you for attending Inter/Micro 2014.



Gary J. Laughlin
Chairman, Inter/Micro

Cover image by Julian C. Gray, Tellus Science Museum
The photomicrograph shows myristic acid, an organic acid named after *Myristica fragrans* and commonly known as nutmeg, viewed between crossed polars. The image was voted Best Overall Winner of the 2013 Inter/Micro Photomicrography Competition, sponsored by pH2, LLC.

MONDAY, JUNE 2

TECHNIQUES AND INSTRUMENTATION

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

9:00 a.m. – 12:15 p.m. Morning Session, McCrone Lecture Room

Chair: Donald J. Petka, Orange County Sheriff's Department

World's "Wurst" Microscopy: Déjà Vu
Andrew A. Havics — pH2, LLC

The Discriminating Power of High Performance Thin Layer Chromatography (HPTLC) for Commercial Textile Dyestuffs
Ethan Groves, Microtrace, LLC

Microscopy Training: Interns, TEM Class, Kid's Course
James R. Millette — MVA Scientific Consultants, Inc.

Circularly Polarized Light: A Forgotten Yet Invaluable Technique for Microscopists
Thomas J Hopen and Natasha Neel — Bureau of Alcohol, Tobacco, Firearms and Explosives, Forensic Science Laboratory

Three-Dimensional Visualization Using X-ray Tomography
Philip W. Urnezis and Robert G. Myers — Wm. Wrigley Jr. Company

Sénarmont Compensator Validation
Donald J. Petka — Orange County Sheriff's Department

12:15 – 1:45 p.m. Lunch Break, McCrone Garden

1:45 – 5:00 p.m. Afternoon Session, McCrone Lecture Room

Chair: Richard Brown, MVA Scientific Consultants, Inc.

Is Microscopy Subjective?
Andrew M. Bowen — U.S. Postal Inspection Service

The Comparison of Similarly Colored Fabrics and Yarns Using Comparison Microscopy and Microspectrophotometry
Katelyn Hargrave — Microtrace, LLC

Surprising Future for the Simple Microscope
Brian J. Ford — Caius College, University of Cambridge

Rapid Chemical Imaging with a Raman Microscope
Kenneth J. Smith — Thermo Fisher Scientific

The Gospel of Judas Ink Analysis: Technical Literature Comes to the Rescue
Joseph G. Barabe — Barabe & Associates, LLC

Examination of Contaminated Medical Devices
Richard Brown — MVA Scientific Consultants, Inc.

MONDAY, JUNE 2

AN EVENING WITH BRIAN: "THERE IS LIFE AFTER DEATH"

5:30 – 7:00 p.m. Dinner, McCrone Garden $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, McCrone Lecture Room (free)

Brian examines death and the arguments over the end of life — its causes, triggers and the experiences it brings — which have raged for thousands of years. Experiments began in Soviet Russia, Nazi Germany and America between the two World Wars, and the subject has modern repercussions in how terminally ill patients are managed. But what happens at the moment of death and afterwards? This illustrated talk presents some of the most ghoulish experiments in the history of medicine and demonstrates the role of cell biology when we reflect on metabolic and cognitive processes as life comes to an end.

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, JUNE 3

ENVIRONMENTAL AND INDUSTRIAL MICROSCOPY

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

9:00 a.m. – 12:15 p.m. Morning Session, McCrone Lecture Room

Chair: Meggan B. King, McCrone Research Institute

You Found WHAT in Your Pizza?
Brendan Nytes, Christopher S. Palenik and Katie White — Microtrace, LLC

Seeing Color: Practical Methods in Pigment Microscopy
Christopher S. Palenik and Skip Palenik — Microtrace, LLC

Cell Intelligence Through History
Brian J. Ford — Caius College, University of Cambridge

Air Quality and Microscopy: Is It a Convergence or a Divergence?
Andrew A. Havics — pH2, LLC

Examination of Wood Fiber "Contamination" in the Paper and Allied Industries
Walter J. Rantanen — Integrated Paper Services

Evaluating Different Methods of Comparison for Fibers with Subtle Variations in Dye Concentration
Katie M. White, Christopher S. Palenik, Jason C. Beckert and Katelyn Hargrave — Microtrace, LLC

Cocaine: Microscopy of the Devil's Dandruff
Meggan B. King — McCrone Research Institute

12:15 – 1:45 p.m. Lunch Break, McCrone Garden

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Chair: Martin Kocanda, Northern Illinois University

PLM: Still Alive and Kicking
Lawrence Wayne — Forensic Analytical Laboratories, Inc.

Acceptability of Method Short Cuts in TEM Asbestos Analysis
R.J. Lee, M. Sanchez, D. Van Orden and Long Li — R.J. Lee Group, Inc.

Asbestos vs. Look-Alike Contamination in Thermal Insulation: A Case Study
Randy Boltin — MVA Scientific Consultants, Inc.

Asbestos Analysis and Conformance with Regulatory Control Limits
Eric J. Chatfield — Chatfield Technical Consulting Limited

A Review of Some ASTM Standards for the Microscopist
Andrew A. Havics — pH2, LLC

The Dilemma in Providing Evidential Data in Cases Involving Industrial Particulate Trespass
Wayne C. Isphording — Tulane University School of Continuing Studies

Not Just Blowing Bubbles: A Microscopical Perspective of Polyurethane Foams
Martin Kocanda — Northern Illinois University, Department of Electrical Engineering

TUESDAY, JUNE 3

WINE AND CHEESE RECEPTION WITH EXHIBITORS

5:00 – 6:00 p.m. McCrone Exhibit Room (Free)
Meet representatives from Boeckler Instruments Inc., Campbell Center, Leica Microsystems and Thermo Fisher Scientific.

REGGIE'S ROCK CLUB ROOFTOP DINNER

6:30 p.m. Reggie's Rock Club, 2105 S. State Street, Chicago
$25
Kick back on a pleasant evening with fellow Inter/Micro attendees, exhibitors and sponsors for refreshments and dinner on Reggie's rooftop, located just a few blocks away from McCrone Research Institute. Transportation to Reggie's will be provided by the Reggie's bus!

WEDNESDAY, JUNE 4
CHEMICAL AND FORENSIC MICROSCOPY

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

9:00 a.m. – 12:15 p.m. Morning Session, McCrone Lecture Room

Chair: Douglas Ridolfi, Buffalo State College

Microscopy Training in a Forensic Chemistry Program
Douglas A. Ridolfi — Buffalo State College

A Novel Microcrystal Test for Detecting Clonazepam
Danielle Silletti — University of Illinois-Chicago

Microcrystal Analysis of the Tropane Alkaloids of Datura
James Dunlop — Kalamazoo County Sheriff's Office

When Color Isn't Enough: Practical Lessons Learned From an Accident Reconstruction Involving Suspected Paint Transfer
Jason Beckert — Microtrace, LLC

A Study of Select Refractive Index Liquid Stability: Part 3, Series B (n_D = 1.642–1.700) and Part 4, Series E and HD (n_D = 1.500–1.640)
Aletha Basconcillo — Los Angeles Police Department
Wayne Moorehead, Kaycee Fontes and Cassandra Hayes — OC Crime Lab

The Detective Handbook: A Neglected Aspect of Forensic Science Literature
Douglas A. Ridolfi — Buffalo State College

12:15 – 1:45 p.m. Lunch Break, McCrone Garden

1:45 – 5:00 p.m. Afternoon Session, McCrone Lecture Room

Chair: Sebastian B. Sparenga, McCrone Research Institute

Particle Investigations: Addressing Product Recalls and Quarantines
Mary A. Miller and Ming Z. Zhou — MVA Scientific Consultants, Inc.

Drugs and Microscopy in the Last 30 Years
Karl Larsen — University of Illinois-Chicago

The Strange Case of Timmothy Pitzen, and From Genoa to Montreal
Skip Palenik — Microtrace, LLC

PTFE or Wax?
Bill Neuberg — Shamrock Technologies

Examining the Effects of Environmental Degradation on the Optical Properties of Manufactured Fibers of Natural Origin: Year 2 Review
Kelly Brinsko — McCrone Research Institute

Development of a Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals: Year 3
Sebastian B. Sparenga — McCrone Research Institute

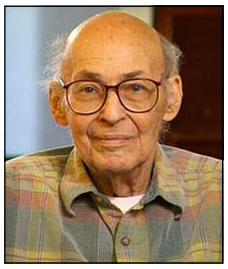
WEDNESDAY, JUNE 4

STATE MICROSCOPICAL SOCIETY OF ILLINOIS
2014 AWARDS DINNER

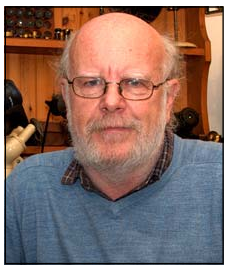
Presented at Mercat a la Planxa, 638 S. Michigan Avenue, Chicago. $65

6:30 – 8:30 p.m. Social hour and dinner
8:30 – 9:30 p.m. Award announcements and presentations
9:30 – 10:00 p.m. Silent auction

Join Inter/Micro and the State Microscopical Society of Illinois as they honor Marvin Minsky, recipient of the SMSI 2014 Émile M. Chamot Award and Steve Gill, recipient of the SMSI 2014 August Köhler Award.



Marvin Minsky is the inventor of the confocal scanning microscope, which he patented in 1957, and a founding father of the artificial intelligence field. He is also the author of key theoretical and practical contributions in cognitive science, robotics, mathematics and philosophy. A co-founder of the prestigious Artificial Intelligence Laboratory at the Massachusetts Institute of Technology, Minsky was also instrumental in establishing the MIT Media Lab.



Steve Gill is a research analyst for a software communications company. His contributions to microscopy include research on the lives of eminent microscopists of the past, a subject on which he has published widely. His expertise in this field of research has been utilized by a host of authors as an adjunct to their own work. Gill has also produced many diatom-related publications, including the Web-based Klaus Kemp's Diatom Database, which is used in many universities worldwide.

THURSDAY AND FRIDAY, JUNE 5–6

WORKSHOP: PHARMACEUTICAL FOREIGN PARTICULATE — EXAMINATION, ISOLATION AND ANALYSIS

9:00 a.m. – 5:00 p.m., McCrone Classroom

Taught by Richard Brown, Mary Miller and Ming Zhou
MVA Scientific Consultants, Inc.

This two-day Inter/Micro workshop provides a framework for the analytical approach used to investigate pharmaceutical product contamination and will focus on foreign particulate examination, isolation and analysis. It also aims to help students understand what foreign particulate is and why it is important to identify its composition and potential sources.

Techniques covered are sample preparation, polarized light microscopy, infrared microspectroscopy and scanning electron microscopy with energy dispersive X-ray spectrometry. After having taken this hands-on course, students will have developed particle isolation skills and an understanding of the methods used in identifying particulate material.

Richard Brown *is an analytical microscopist with more than 25 years experience specializing in material and particle characterization. Mr. Brown's area of expertise focuses on solving issues in coatings, polymers, surface profiling, contaminants, and product failures for medical device companies.*

Mary Miller *is an analytical chemist with more than 20 years experience serving the pharmaceutical and medical device industries. Ms. Miller's expertise focuses on foreign particulate isolation and analysis, particle sizing, and helping manufacturers find the source of contaminants.*

PRESENTATION ABSTRACTS

MONDAY, JUNE 2
TECHNIQUES AND INSTRUMENTATION

World's "Wurst" Microscopy: Déjà Vu
Andrew A. Havics — pH2, LLC

Another installment of microscopical woes catalyzed by Professor Brian Ford's "World's Worst Microscopy" presentations at Inter/Micro several years ago. One asks, "Where do I begin?" The answer is that it doesn't take a microscope to find a microscopical mishap; but apparently it does take a microscopist, and I suspect there are fewer of them around compared to the microscope users. Or perhaps, it's just heightened awareness by the presenter.

The Discriminating Power of High Performance Thin Layer Chromatography (HPTLC) for Commercial Textile Dyestuffs
Ethan Groves — Microtrace, LLC

Thin layer chromatography (TLC) is a well-developed routine analytical tool that, when performed correctly, can provide a fingerprint identification of complex mixtures. The advent of high performance thin layer chromatography (HPTLC) plates has brought greater resolution with smaller sample requirements, allowing for greater discriminating power from minute quantities of material. From a forensic casework viewpoint this allows for smaller segments of colored fibers to be analyzed. Fiber evidence is arguably the most diverse evidence type encountered in forensic casework and its analysis, specifically the colorants they contains, can be very important in comparing and associating textiles.

HPTLC has historically been limited to a comparison technique, meaning that known samples had to be analyzed in tandem.

However, when a series of analysis conditions are maintained HPTLC has been found to be extremely discriminating. We will demonstrate that controlled conditions yield highly reproducible HPTLC analysis. This allows for reference libraries to be constructed for comparison and the potential presumptive identification of dyestuffs. This presentation will discuss the analysis of 300 commercially common and important dyestuffs by HPTLC with emphasis on analysis conditions including tank saturation, developing distance and eluent stability as they relate to retardation factor (R_f) reproducibility. Under a controlled system, a developed dyestuff's characteristics (e.g. color, retention, fluorescence, band shape and number of bands) can be accurately assessed in determining the discriminating power of HPTLC.

Microscopy Training: Interns, TEM Class, Kids Course
James R. Millette — MVA Scientific Consultants, Inc.

MVA Scientific Consultants is an independent testing and consulting laboratory with a staff of 15 part-time and full-time employees. In the past 23 years of existence we have had over 40 paid interns and a number of unpaid interns who received college credit for their time working with us. Many of the interns have received training and become operators of light and electron microscopes, as well as users of the infrared and Raman instruments. One intern won a scientific microscope photography contest with a submission of a 3-D cenosphere fly ash particle that he took with a scanning electron microscope during an environmental forensic project. Another intern won the annual company holiday card contest with an image of a talc particle that she colorized to look like a Christmas stocking. A collaboration between MVA Scientific Consultants and Gwinnett Technical College's Bio-technology program has provided a number of students with the opportunity for on-the-job microscopy training and commercial business hours they needed to complete their technical degree. Since 1988, I have been teaching a course concerning the use of transmission electron microscopy for the analysis of asbestos. Limited to only 6 students, the participants

get hands-on training in the preparation of samples and the analysis involving selected area electron diffraction and energy dispersive X-ray analysis. MVA Scientific Consultants has contributed to microscopy education by hosting the Georgia Microscopical Society Young People's course on Microscopy for over 20 years. Staff members of MVA join other members of the Atlanta area microscopy community in teaching this free course for middle-school students and teachers on seven Saturday mornings in the winter. In addition to basic instruction on how to use a microscope, the course includes sessions on criminal forensic and environmental forensic microscopy as well as geology, biology and photography through the microscope.

Circularly Polarized Light: A Forgotten Yet Invaluable Technique for Microscopists

Thomas J Hopen and Natasha Neel — Bureau of Alcohol, Tobacco, Firearms and Explosives, Forensic Science Laboratory

Circularly polarized light is a simple yet rarely used microscopical technique. The photomicrographs for *The Particles Atlas, Volume 5* were taken using this technique. Circularly polarized light is accomplished using an anisotropic plate whose thickness is such that the retardation is a quarter of a wavelength for a particular color. This color is usually in the yellow/green region (~550 nm) using white light resulting in a pale gray background color with crossed polars when the privileged directions of the anisotropic plate are 45 degrees to that of the polarizer. The resulting quarter-wave retardation results in a spiral wave motion resembling a cork screw. If viewed looking down the direction the wave is traveling it would appear to be vibrating in a circular motion; hence is said to be circularly polarized light.

When the quarter-wave plate is placed below the sample, the light traveling through anisotropic particles will be doubly refracted and travel along the two privileged directions of the crystal. Because the light is traveling in a circular motion, the sample anisotropic crystals will not show the characteristic extinction

normally seen when viewed with crossed polars. To obtain a black field and the characteristic retardation colors for the sample particles, a second quarter-wave plate can be inserted between the sample and the analyzer with the slow ray and fast ray perpendicular to the first quarter-wave plate. This technique is particularly useful when searching for isotropic particles such as glass in a mix of soil particles, when doing soil mineral grain counting, and, as demonstrated by the Particle Atlas, when taking photomicrographs of samples with crossed polars. Also, it is interesting to note circularly polarized light was used to make gun sights for fighter aircraft during World War II which will be demonstrated during the presentation.

Three-Dimensional Visualization Using X-ray Tomography
Philip W. Urnezis and Robert G. Myers — Wm. Wrigley Jr. Co.

X-ray tomography is a technique that allows the interiors of object to be imaged without having to slice open the object. Multiple 2-D X-ray images are taken of the object and then are computer reconstructed into a 3-D depiction. The power of tomography is the ability to view the sample in 3-D and to see how the different domains of the interior interact. This presentation will discuss segmenting samples and showing 3-D videos of the different domains.

Sénarmont Compensator Validation
Donald J. Petka — Orange County Sheriff's Department

The Sénarmont compensator is used to accurately determine retardation values within a few nanometers. Compared to other compensators, it is relatively inexpensive, but it is limited to retardation values less than 546 nm. In this work, an overview of the technique will be presented and a detailed explanation of the theory will be provided. Additionally, a simple validation method will be submitted. This will demonstrate that the application has acceptable accuracy, exceptional linearity and excellent precision.

Is Microscopy Subjective?

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

I have often heard students in microscopy training courses comment about the subjective nature of the observations they are making. I have also heard reference to the subjectivity of microscopy in presentations at professional meetings. The field of forensic science is moving toward greater standardization, a process that is driven by guidelines published by scientific working groups (soon to be scientific area committees), accreditation of laboratories, and certification of individuals. Many scientists who are not microscopists are helping to shape the new guidelines and standards that most of us are likely to be beholden to in the future. The view that microscopy is by its very nature subjective, a view held by some non-microscopists, may impact how microscopy is incorporated into new guidelines being produced. It is my belief that, while some applications of microscopy are fairly described as subjective, other applications are as objective as most other methods of instrumental analysis. It is critical that microscopists are proactive in educating their colleagues to avoid misconceptions about microscopy. When working groups (or area committees) make new guidelines available for public comment, we must take the time to read them and ensure that microscopy is treated in an appropriate manner in these documents.

The Comparison of Similarly Colored Fabrics and Yarns Using Comparison Microscopy and Microspectrophotometry

Katelyn Hargrave, Jason Beckert, Christopher S. Palenik and Katie White — Microtrace, LLC
Dr. Michael Sigman — University of Central Florida

Fibers are a common form of trace evidence; however, one outstanding (and frequently pondered) question is whether similarly colored fibers can be discriminated (and by which methods). Here, we present the comparison results from two particularly interesting sets of similar samples. The first set consists of three fabric pairs, each pair is dyed with a chemically similar (though

slightly different) dye. The second sample set consists of six yarns purchased for their similarities in macroscopic color.

Fibers were compared on the basis of color by comparison microscopy and microspectrophotometry. The resulting spectra were analyzed using transmission, absorption, and first derivative spectral overlays. The results showed that the sample pairs dyed with chemically similar dyes could always be discriminated. The similarly colored yarns (with unknown dye compositions) could be discriminated in 13 of 15 comparisons. The two pairs that could not be discriminated turned out to be dyed with indistinguishable colorant packages (based on high performance thin layer chromatography and Raman microspectroscopy of the separated dye components), with the sole potential difference being the concentration of dye utilized.

Within the set of samples studied in this research, fibers dyed with different dyes can all be discriminated; however, those dyed with the same dyes (possibly in different concentrations) are more difficult to confidently discriminate without more quantitative approaches.

Surprising Future for the Simple Microscope

Brian J. Ford — Caius College, University of Cambridge

For decades the author has been speaking of the potential importance of single-lens microscopes. Designed by Robert Hooke in 1660s London, and then used by Leeuwenhoek until his death in 1723, single-lens microscopes underpinned early microscopy. During the 1990s, a microscope was designed by the author for the European Space Agency based on the principle and he has often shown that they could have present-day applications in microscopy. The interest now being shown in smartphones has encouraged other design ideas to be announced, and he will discuss them today. He will also update the fate of the existing Leeuwenhoek microscopes.

Rapid Chemical Imaging with a Raman Microscope
Kenneth J. Smith — Thermo Fisher Scientific

By improving the sensitivity of a CCD detector in a Raman microscope it is possible to move from a spectroscopy centric approach to Raman analysis of materials to an imaging centric approach. This shift allows a focus on the microscopy of a sample with the Raman data aiding in characterization and differentiation of material phases and types. This approach also requires new data handling methods as well as improved sample location and scanning. Applications of this approach using the Thermo Scientific DXRxi Raman microscope will be presented in the areas of graphene, pharmaceuticals, semiconductors and minerals.

The Gospel of Judas Ink Analysis: Technical Literature Comes to the Rescue
Joseph G. Barabe — Barabe & Associates LLC

Dr. Walter C. McCrone once said that an analytical report should answer these basic issues:
1. What we received
2. What we did
3. What we found
4. What it means
In analyses conducted to determine the authenticity of art and historical objects, the analysis itself may be the easy part; certainly, the client generally could care less what we find, as long as it addresses the issue of authenticity. In the analysis of the ink in the Gospel of Judas, conducted for the National Geographic Society in 2006, the authenticity of the document was the only concern. In that our analysis generated unexpected data, we were initially unable to express any opinion to our client. In the end, a diligent literature search provided an answer which suggested that the material was in all likelihood appropriate for circa 250 CE. This paper will summarize the analyses conducted and the data collected, and will focus on the ultimately successful effort to answer that burning question: What it means.

Examination of Contaminated Medical Devices

Richard Brown — MVA Scientific Consultants, Inc.

A practical approach to documenting, sampling and analyzing contamination on medical devices involves techniques that require three hands; especially when the devices have been used. Documentation involves confirmation that the device has been decontaminated (if previously used) and that permission to sample destructively has been granted. Photographic documentation involves a photographer and a “handler” to "pose" the sample for imaging and to sub-sample for analysis by microscopy. The logical sequence for microscopy involves sub-sampling for low vacuum SEM-EDS using a beryllium substrate, Raman microscopy, FT-IR microscopy and PLM. Since a medical device can be anything from a glove to a micro-surgery tool, this type of investigation is guided by the types of materials found, the type of failure and the circumstances surrounding the failure or customer complaint. The usual micro-techniques for particle analysis, liquids and separation are applied to aid in the investigation.

TUESDAY, JUNE 3
ENVIRONMENTAL AND INDUSTRIAL MICROSCOPY

You Found WHAT in Your Pizza?
Brendan Nytes, Chris Palenik and Katie M. White — Microtrace, LLC

While many of the industrial samples that we receive are examined solely for the purpose of identification, sometimes our clients ask us to determine if complaint materials were introduced at their facility, restaurant, etc. These complaint materials can range from metal to wood to plastic and even mice. Depending on the substance itself and the circumstances of the case, the question of when it was introduced into the product may be relatively easy or very difficult to answer. This question may often be answered by not only identifying the complaint material and any residues adhering to it, but through questioning our client about their processes and the detailed specifics of the consumer's complaint. In this talk, we will discuss different approaches that may help to determine when a complaint material was introduced to a product. The presentation will conclude with an example of how these principles were applied to the solution of a peculiar case in which we were asked to determine if a condom was baked in a pizza.

Seeing Color: Practical Methods in Pigment Microscopy
Christopher S. Palenik and Skip Palenik — Microtrace, LLC

This presentation will demonstrate the methods by which the microscopic particles responsible for color in paints, inks, polymers, rubbers and cosmetics can be directly and readily observed. The presentation will utilize examples of pigments in paint, fibers, and cosmetics to show sample preparation and imaging methods that permit these pigments to be visualized and utilized as evidence in forensic and industrial examinations.

Preparation methods that range from the efficient (smears) to the traditional (cross sections) to the state of the art (ion polished cross sections) and imaging methods that span scales of millimeters to nanometers (including polarized and oil immersion light microscopy as well as scanning and transmission electron microscopy) will be illustrated. While the focus of this presentation is on forensic paint and fiber applications, the same approaches would be relevant to ink chemists, art authentication efforts, and the coatings and pigment industries. For not only does such analytical information provide the opportunity to make observations not possible by indirect methods (such as infrared spectroscopy comparisons) or the casual application of magnification that is common in most forensic laboratories (e.g. paint chip layer determination by stereo microscopy), but it opens the possibility to find true differences in samples at the micrometer and nanometer scale that may be suggestive of a specific manufacturer, batch difference, or quality issue. Finally, the resulting images provide a simple and visually compelling means by which to convey such similarities or differences to a lay audience or jury.

Cell Intelligence Through History

Brian J. Ford — Caius College, University of Cambridge

The microscope reveals that living cells can manifest remarkable properties, some of which can be argued as revealing the roots of intelligence. The importance of these remarkable abilities is poorly understood, though several workers have tried to tackle the problem in the past. The coinage of the term “cell intelligence” dates back a century, and this illustrated presentation reveals the importance of the term and discusses whether present-day stem cell research fits into the concept.

Air Quality and Microscopy: Is It a Convergence or a Divergence?

Andrew A. Havics — pH2, LLC

There are a number air quality agents that have been detailed at Inter/Micro over the years, and many are straight-forward characterizations — a client or customer wants to know either what something is/is not, or they know what it is and want that something quantified, or sometimes both. There are times, however, when a client wants to know — what does that something mean in terms of their investigation. Is that agent relevant? Can the presence or relative amount tell me something about the building environment or the occupants' habits? Can you explain why it is there, or why it is there at the concentrations found? These are aspects that quickly turn into pseudo-forensic consulting (if that's really a term), which is what the clients are supposed to be getting paid for, not the lab. So it's time to charge them consulting rates, and then answer the questions. Several case examples will be used to demonstrate (read that as justify the consulting charges).

Examination of Wood Fiber "Contamination" in the Paper and Allied Industries

Walter J. Rantanen — Integrated Paper Services

Various fiber based products can have other fibrous materials present which are not intended to be part of the furnish. These "contaminants" may cause unsightly appearance problems, difficulty in processing and production, or negative perceptions with customers and/or the general public. A few examples will be presented along with some techniques used to determine the nature and likely source of these undesirables.

Evaluating Different Methods of Comparison for Fibers with Subtle Variations in Dye Concentration

Katie M. White, Christopher S. Palenik, Jason C. Beckert and Katelyn Hargrave — Microtrace, LLC

From garments to upholstery, textile fibers are routinely recovered as trace evidence at crime scenes. Through analysis and proper interpretation, these microscopic clues can establish facts and provide valuable investigative leads. Some fibers can be readily distinguished based on fiber type, but it is often color that holds the greatest potential for discrimination. Comparison microscopy and microspectrophotometry (MSP) are two of the most common methods utilized by trace evidence analysts today for comparing dyed textile fibers. Both techniques are used to characterize colors, with one method relying on the visual perception of color and the other measuring molecular absorptions of the dye molecule(s) present.

As differences in dye composition become more subtle, discrimination between samples becomes more difficult. In this research project, 14 swatches of fabric colored using slightly varied dyestuff concentrations of the same known dyes, producing highly similar shades, were examined.

Fibers from each of the 14 swatches were evaluated in 91 pairwise comparisons to determine whether samples could be discriminated by MSP and/or comparison microscopy. This work will present results of this research, offering a comparison of the analytical techniques and a discussion of their limitations under these circumstances.

Cocaine: Microscopy of the Devil's Dandruff

Meggan B. King — McCrone Research Institute

For more than two years, McCrone Research Institute researchers have been working on an NIJ-funded research grant (2011-DN-BX-K528) that involves evaluating different microcrystal tests for some of the most common illicit drugs and diverted pharmaceuticals. This presentation will highlight our efforts with the schedule II stimulant, cocaine. Of the microcrystal tests evaluated thus far, we have found that gold and platinum chloride reagents are the most reliable, sensitive and discriminating. The

process of locating, evaluating and selecting the microcrystal tests will be discussed as well as any interferences that we have noted.

PLM: Still Alive and Kicking
Lawrence Wayne — Forensic Analytical Laboratories, Inc.

Polarized light microscopy (PLM) has been waning in use for the last several years. Trace evidence sections are closing in law enforcement labs, older scientists are retiring and industry is increasingly reliant on instrumentation run by technicians. Some laboratories, however, still rely on the use of PLM as a basic tool of analysis. New instrumentation can be successfully added to supplement it, but at the core is still an inquisitive scientist trying to observe something too small to be seen with the naked eye.

Acceptability of Method Short Cuts in TEM Asbestos Analysis
R.J. Lee, M. Sanchez, D. Van Orden and Long Li — R.J. Lee Group, Inc.

Amphibole asbestos identification by transmission electron microscopy (TEM) has been a challenging technical problem for more than 30 years. The Yamate method first recognized the need for quantitative zone axis analyses to reliably identify amphiboles. All standard methods developed since Yamate have had similar requirements. However, as the process has become routine, laboratories have increasingly taken shortcuts as a cost saving measure. Recently, there have been suggestions that this level of rigor is not required, and that use of the “aperture method” publish by Webber provide an equally reliable result. This paper will review the issue of amphibole identification and provide examples of the issues encountered when shortcuts are taken, particularly when attempting to determine asbestos content of products such as cosmetic talc or measuring asbestos exposures in naturally occurring environments, where interferences are likely.

Asbestos vs. Look-Alike Contamination in Thermal Insulation: A Case Study

Randy Boltin — MVA Scientific Consultants, Inc.

Insulating material from steam and gas turbines at a power generating station were tested for asbestos by polarized light microscopy. Approximately 30% of the samples were reported to contain measurable concentrations of chrysotile asbestos. The installer of the turbines had further analytical work performed on the samples and the results indicated the fibrous material in question to be a non-regulated asbestos look-alike mineral.
Our lab was asked to assist in resolving the discrepancy. The issue was resolved through a combination of microscopical methods and X-ray diffraction coupled with a consideration of the effects of thermal alteration on the physical and chemical properties of the questioned fibers.

Asbestos Analysis and Conformance with Regulatory Control Limits

Eric J. Chatfield — Chatfield Technical Consulting Limited

When regulations regarding asbestos-containing materials were first promulgated in the U.S. and Canada, the materials being analyzed were primarily fireproofing and pipe insulation. These materials contain either 15% to 100% asbestos, or none at all. At that time, many jurisdictions defined an asbestos-containing material as any material containing more than 1% asbestos. For fireproofing or pipe insulation, conformance with a 1% regulatory control limit presented no problems, because the PLM analyst could readily determine that the concentration of asbestos was obviously much greater than 1% or that no asbestos fibers could be detected.

Regulatory interest eventually shifted to materials such as plasters, texture coats, wall joint compounds and historical materials containing vermiculite. If asbestos is present in these materials, the concentration may be 0.1% up to 5%. In addition, various jurisdictions have introduced regulatory control limits of “any

asbestos," 0.1% or 0.5%. Bureaucrats rarely consider statistics or the limitations of PLM analysis, and consequently these regulations are established with little or no regard to the feasibility of conformance determination.

The inaccuracy and lack of precision of PLM analysis on an untreated sample of asbestos-containing materials has been extensively documented, and it shows that determination of asbestos concentrations below 5% is largely guesswork. It is hoped that two new International Standards will establish a rational approach to conformance determination for materials containing low concentrations of asbestos. ISO 22262-1 (Bulk materials — Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials) was published in 2012. ISO 22262-2 (Bulk materials — Part 2: Quantitative determination of asbestos by gravimetric and microscopical methods) will be published in 2014.

A Review of Some ASTM Standards for the Microscopist
Andrew A. Havics — pH2, LLC

In addition to sponsoring symposia and producing several special technical publications on the subject of microscopy, ASTM has served as a de facto proponent of the microscope. There are a number of ASTM standards that consider the microscope as a tool of choice. The range is far, including analysis of concrete, fibers, water deposits, microbial agents in various media, paint, glass, metals, inclusions in metals and polymers, carbon black, soot, whiskers, asbestos, gunshot residue (GSR), drugs, paper, coal and so on. They also include providing specific characterization such as hardness by indentation, fracture morphology, particle size, grain size or orientation, point counting, corrosion pit shapes, residual stress determination, type of fungal spore in the air, live-dead bacteria in water, etc.

The Dilemma in Providing Evidential Data in Cases Involving Industrial Particulate Trespass

Wayne C. Isphording — Tulane University School of Continuing Studies

Cases involving trespass of particulate dust/debris derived from industrial and manufacturing sources depend largely on presenting to a jury evidence that, unequivocally, ties foreign materials on private properties to a specific anthropogenic source (or sources). Mineralogical information, either from petrographic, XRD or SEM analysis obtained from soil samples on plaintiff's properties, is generally successful especially if deposition of the invasive materials can be demonstrated to have been carried to the site as "water-borne detritus" (e.g. surficial or stream run-off from an up-gradient industrial source). Trespass of particulates derived by airborne transport, however, is more challenging. This arises from the fact that, while acceptable methods for collection of soil samples and "wet wipe" samples for chemical analysis are well documented by EPA and ASTM protocols (as well as by various State guidance manuals), there are no such protocols for collection of "dry wipe" samples that are subsequently used for mineral identification by XRD, SEM or petrographic analysis. As such, this provides an immediate avenue for the defense to prevent introduction of data produced by the plaintiff's expert witness or to demand the information be excluded by use of a legal procedure known as a Daubert Challenge. The inclusion of such critical data can be successfully accomplished, however, by arguing that, although no written protocol yet exists, the collection of "dry wipe" samples stands the test of objective data gathering by a methodology that has "reasonable scientific certainty."

Not Just Blowing Bubbles: A Microscopal Perspective of Polyurethane Foams

Martin Kocanda — Northern Illinois University, Department of Electrical Engineering

Foams are defined as a broad class of colloids consisting of a dis-

persed gas phase contained in a dispersing medium and further subdivided into two classes where the dispersed gas phase is contained in a liquid medium or a solid medium. The challenge has been to capture images of solid foams because the dispersed phase pore diameters are too large to be resolved using scanning electron microscopy but too small to be resolved using conventional low magnification light microscopy. In this work, polyurethane foams are examined using an alternative method of low magnification digital imaging to examine the dispersed phase from a forensic perspective.

WEDNESDAY, JUNE 4
CHEMICAL AND FORENSIC MICROSCOPY

Microscopy Training in a Forensic Chemistry Program
Douglas A. Ridolfi — Buffalo State College

Forensic Science 416 Optical Microscopy is a newly required course for students in an undergraduate forensic chemistry program at Buffalo State College. The challenge of teaching this course is that it is a two unit class. With two hours of instruction per week over the course of a semester, what is the appropriate mix of lecture, demonstration and practical experience that would be most suitable for building some degree of proficiency and appreciation for the application of the polarizing light microscope in the examination of trace evidence? Some suggestions for useful demonstrations, topics and lab exercises will be presented.

A Novel Microcrystal Test for Detecting Clonazepam
Danielle Silletti —University of Illinois-Chicago

Recently, microcrystal tests have been used as an analytical technique for distinguishing certain drugs in the forensic sciences. While some drugs, such as cocaine and heroin, have well validated microcrystal tests, others, i.e. benzodiazepines have not been extensively studied for this purpose. 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.

Since that time, a microcrystal test was developed that is able to detect clonazepam in its pharmaceutical preparations and in all of its therapeutic dosages. Previously published microcrystal tests for this drug were unable to be replicated. Additionally numerous popular microcrystal test reagents and methods were experimented with, but none provided successful results. Since that time, a microcrystal test was developed that is able to de-

tect clonazepam in its pharmaceutical preparations at all of its therapeutic dosages. The test involves the addition of acetone and a 10% platinum chloride solution to microgram quantities of clonazepam. The presence of clonazepam is indicated by the formation of rosettes made of colorless, blunt-ended rods. No other drug is known to exhibit the same reaction with these reagents making the test a good candidate for use in forensic lab settings. In addition, it follows a simple procedure, uses minute quantities of the drug sample, and involves reagents that are common and involved in other known drug microcrystal tests.

Microcrystal Analysis of the Tropane Alkaloids of Datura
James Dunlop — Kalamazoo County Sheriff's Office

Tropane alkaloids are nitrogenous compounds naturally found in a variety of plants. The most notorious and perhaps the most widely recognized is cocaine found in erythroxylum coca, indigenous to South America. However, additional tropane alkaloids are present in numerous species of the Solanaceae (nightshade) family and are commonly encountered throughout the U.S. Among them are the nine species of the Datura genus known by such common names as moonflower, jimson weed and angel's trumpet. As is the case with cocaine, the alkaloids of the datura plant have beneficial pharmaceutical uses but also can be abused by illicit drug users seeking a cheap local high, often with catastrophic and tragic results. The lecture will include a brief history of the datura plant as well as an explanation of the extraction of its three chief alkaloids (Atropine, Scopolamine and Hyoscyamine). Additionally, the reagents used in the microcrystal analysis of each of the listed alkaloids will be discussed and supported with photomicrographs.

When Color Isn't Enough: Practical Lessons Learned from an Accident Reconstruction Involving Suspected Paint Transfer
Jason Beckert — Microtrace, LLC

Color is an incredibly discriminating property for a variety of

types of forensic evidence including paint. It is often the most important feature focused on by forensic scientists when they are examining objects for the presence of trace evidence. However, it is always important to remember that color is not everything. This presentation will focus on an accident reconstruction involving the suspected transfer of paint from one vehicle to another. While seemingly scientifically simple, it serves as a good case study touching on evidence collection, forensic paint analysis techniques, the "expertise" of scientific experts, and how the legal system places value on these elements in the broader context of the real world where financial considerations often reign supreme.

A Study of Select Refractive Index Liquid Stability: Part 3, Series B (n_D = 1.642–1.700) and Part 4, Series E and HD (n_D = 1.500–1.640)

Aletha Basconcillo — Los Angeles Police Department, CA
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 confirming their identity by instrumental methods. Whether being opened and used regularly for casework or factory sealed and sitting in a cool, dark place within the laboratory for years, questions arose about the sustainability of laboratory refractive index liquids over many years. With accreditation requiring calibration checks, certificates of analysis, and periodic checks of solvents, chemicals, drugs, and reagents used analysis, examining stability of refractive index liquids was considered.

Several sets of different series (AA, A, B and E) of refractive index liquids (labeled as sold by Cargille Laboratories Inc., Cedar Grove, NJ) with a wide range of values (n_D = 1.320–1.700) were used for the study. Additional refractive index liquids were obtained including sets reportedly manufactured in the early 1970s in a geology lab at one of the local universities with a calibration-

check performed for each liquid at the time of manufacture as well as a set of Ward's labeled refractive index liquids.

A Digital Leica Abbe refractometer having a five-digit display with temperature measurement and a refractive index resolution of 0.0001 was used to perform the refractive index determinations. The instrument was permitted to equilibrate for at least one hour before use. The refractometer was calibration checked each day before use with 18 Mohm deionized water, the temperature recorded, and calculations made. Due to the upper limit of the refractometer, measurements above n_D = 1.700 were not possible.

Due to the large amount of data the study is broken into several parts. Parts 1 and 2 were presented at Inter/Micro 2013. In Part 3 at Inter/Micro 2014, refractive index liquids in the set termed "Series B" from n_D = 1.642 to 1.700; in Part 4, the set termed "Series E & HD" from n_D = 1.500 to 1.640 will be explored. The Cargille refractive index liquids in these series have good stability for many years over the entire range.

The Detective Handbook: A Neglected Aspect of Forensic Science Literature

Douglas A. Ridolfi — Buffalo State College

This presentation will highlight some of the early and more recent literature in forensic science and microscopy that has been directed to a little appreciated audience — the scientifically trained or at least scientifically aware detective. Over the years, handbooks have been produced that have provided scientific guidance to the evaluation of evidence to the detective and other non-laboratory members of the law enforcement profession and others who may have the need to do field evaluation of evidence or conduct some examination without the benefit of extensive equipment. Some of these ventures into the forensic field have included relatively sophisticated microscopic and microchemical examinations of a variety of potential evidentiary materials.

Particle Investigations: Addressing Product Recalls and Quarantines

Mary A. Miller and Ming Z. Zhou — MVA Scientific Consultants, Inc.

Pharmaceutical recalls involving foreign particles have been widely reported in the past several years, creating anxiety among patients and consumers, and trouble for the manufacturing companies. Whether voluntary or issued by the FDA, the cost of pharmaceutical product recalls are expensive: in monetary terms, human resources devoted to the investigation, and the company's reputation. Ensuring that products with foreign particles are not administered to patients is critical. Finding the source of particles is an essential part of the corrective action to reduce or prevent future contamination. This presentation describes two case studies of investigations involved in a recall situation and a product containing particles quarantined pending the results.

The recall investigation involved particles in several lots of product. Analyses of the particles determined they were a specialty glass, and the consideration of several items as potential sources. Comparative analysis of the suspect glass confirmed the source of the particles in the product. The quarantined product investigation required the identification of particles in an injectable drug prepared at a compounding pharmacy. The particles were a result of stopper coring, and it was determined that the type of rubber used was incompatible with the product.

Drugs and Microscopy in the Last 30 Years

Karl Larsen — University of Illinois-Chicago

During the last several decades, the use of the microscope in the drug chemistry sections in many forensic science laboratory settings has changed. Crystal tests have gone from a rapid means of identification in the drug chemist's arsenal to a means of testing many newer drugs that chemists are not even exposed to in training. We will travel through the years to see why, in the pre-

senter's opinion things have changed and why this technique should be re-visited now and in the future.

The Strange Case of Timmothy Pitzen, and From Genoa to Montreal

Skip Palenik — Microtrace LLC

This presentation describes our investigation into two recent cases involving the application of our expertise and experience in geosourcing.

Case 1. On 11 May 2012, 6-year-old Timmothy Pitzen was checked out of his school by his mother. After having a repair performed on her vehicle, she travelled about northern Illinois and southern Wisconsin with Timmothy, staying over at a water park. She was last seen, without her son, on closed circuit television at about 1:30 on Friday afternoon shortly before it began to rain for the first time in over a week. She was found dead in her hotel room Saturday afternoon by police who were asked to enter her room when she failed to check out. She left a note saying Timmy was in a safe place but that no one would ever find him. We were asked by the Aurora Police to attempt to find the site from which the mud on the running board and undercarriage originated.

Case 2. A shipment of cargo bound for Canada was shipped by freighter from Genoa to Montreal. From there it was transported by truck to Toronto. When delivered to the consignee the container and the seals were intact, however, on opening it was found that the merchandise had be removed and substituted with soil. We were retained by the marine insurance company to determine if the sand had originated from the port of Genoa or from Canada. Our analysis of the soil components and interpretation of the results permitted us to answer this question.

PTFE or Wax?
Bill Neuberg — Shamrock Technologies

That polytetrafluoroethylene (PTFE) and wax behave as a solid lubricant better in combination than either one alone was demonstrated in the early 1970s. Microscopy became the tool to investigate the mechanism. It was concluded that the wax melted under pressure and the PTFE sheared along crystal planes. The current investigation shows an unexpected phenomena. Don't miss the exciting outcome!

Examining the Effects of Environmental Degradation on the Optical Properties of Manufactured Fibers of Natural Origin: Year 2 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 occurring in their optical and physical properties as an effect of moisture, sunlight exposure and exposure to various temperatures. For the past two years, fabric swatches representing each fiber type have been exposed to freshwater, saltwater, heat, cold, ultraviolet light and composter conditions. Every eight weeks the swatches were analyzed via 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.

Development of a Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals: Year 3

Sebastian B. Sparenga — McCrone Research Institute

This talk will give an update of the grant that McCrone Research Institute was awarded through the National Institute of Justice (NIJ-2011-2805 SL# 000944). Research during this third year has focused on performing infrared analysis as well as optical characterization on the resultant crystals. The discussion will also cover some of the difficulties encountered throughout testing, along with a timeline for completion of the project.

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