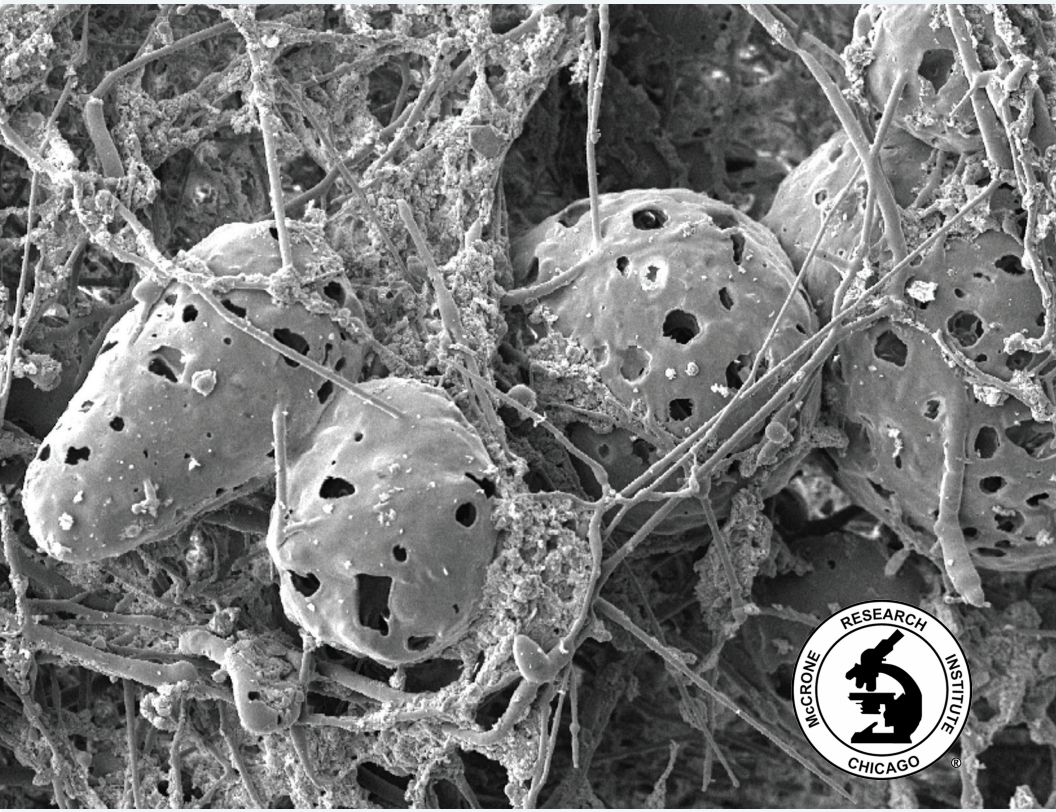
70th Anniversary

Inter/Micro 2018

An International Microscopy Conference
June 11–15, 2018 • Chicago



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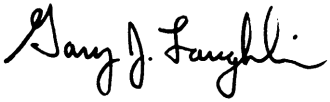
Welcome to Inter/Micro 2018

This year marks the 70th anniversary of the Inter/Micro conference, which was first introduced by Dr. Walter C. McCrone in 1948 and is now held annually at the McCrone Research Institute in Chicago. Inter/Micro gives us all the opportunity to learn about new instruments, new techniques, and new applications of microscopy and microanalysis, and includes the application of light and electron microscopes, microspectroscopes, microprobes, digital and image analysis systems, and many other highly specialized microanalytical methods.

Inter/Micro presentations from the world's leading professional and amateur microscopists will cover new research and techniques for improving contrast, increasing resolution, and obtaining and recording additional small particle characterization and identification data. Participants will learn from the experts how new and time-proven techniques and instruments are used to solve important problems encountered in laboratories today. Students participating in the annual two-day workshop will learn specialized skills and gain hands-on experience working with renowned microscopists.

We encourage all Inter/Micro presenters to submit their research papers for publication in *The Microscope*, the official, quarterly journal of this conference. Papers will be peer reviewed and published in the order in which they are received.

Thanks to all the past, present, and future attendees and participants — and welcome to Inter/Micro 2018.



Gary J. Laughlin
Chairman, Inter/Micro

ON THE COVER: A scanning electron micrograph of ceiling tile core after a fire test, showing expanded perlite, mineral wool, and clay fused together. Micrograph courtesy of Arthur Struss, USG Corp. (retired).

Monday, June 11

Techniques and Instrumentation

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

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

Chair: Martin Kocanda — Rapid City Police Department, South Dakota

Image Acquisition and Analysis of Soybean Stem Sections
Aryeh Weiss — Faculty of Engineering, Bar Ilan University, Ramat Gan, Israel

Eight Is Enough: Fabric Physical Matches in an International Drug Smuggling Case
Jason C. Beckert — Microtrace LLC

Discriminating Glass Fragments Using Micro-XRF Spectrometry with Polycapillary Optics
Bruce Scruggs — EDAX, Inc.

Morning Break

Measurement of Nanoparticle Size Using Transmission Electron Microscopy
Jacob M. Spry — MVA Scientific Consultants

Hacking a Dinosaur: Upgrading a Vintage Microscope Using the Raspberry Pi
Martin Kocanda — Rapid City Police Department, South Dakota

12:00–2:00 p.m. Lunch, McCrone Garden

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

Chair: Charles Mazel — NIGHTSEA

Problems in Raman and FTIR Spectral Library Searches
Lawrence Wayne — Forensic Analytical Laboratories, Inc.

Evaluation of the Criteria for the Discrimination of Inkjet Printer Inks Using Micro Raman Spectroscopy
Patrick Buzzini — Sam Houston State University

Development of Modern Microcrystal Tests for Controlled Drugs, Diverted Pharmaceuticals, and Bath Salts: Project Update
Sebastian Sparenga — McCrone Research Institute

Afternoon Break

Taking a Closer Look at Some Photochromic Materials
Joseph Insana — Microtrace LLC

Microscopy and Poetry: A Union of Art and Artifice
Andrew A. Havics — pH2, LLC

Further Adventures in Fluorescence
Charles Mazel — NIGHTSEA

See Monday presentation abstracts on page 12.

Photomicrography Competition

We invite you to submit photomicrographs taken with any microscopy technique — darkfield, brightfield, fluorescence, interference contrast, polarized light, SEM, etc.

The deadline for in-person submissions at Inter/Micro 2018 is 5 p.m. Tuesday, June 12.

Awards for Best Overall Image, Most Unique Image, and Best SEM Image will be announced at the SMSI Awards Dinner, Wednesday, June 13. The competition is sponsored by pH2, LLC.

Monday, June 11

An Evening with Brian
"The Robot and the Single Cell"

5:30–7:00 p.m. Cuban cuisine dinner, McCrone Garden

7:00–8:00 p.m. An Evening with Brian presentation with ***Brian J. Ford****, McCrone Lecture Room*

The news media are filled with reports of artificial intelligence (AI) and robots taking over the responsibilities of humans. Some have even said humans are shortly going to be made redundant. Yet consider the living cell: although we never hear spokespeople who say so, its capacity surpasses anything that robotics can achieve. Tonight's talk takes us through the contorted history of AI and compares it with what we now know single cells can do. The view of robots to the cell is simply expressed: very artificial, not very intelligent.

Professor Ford is a leading authority on the microscope and a best-selling author, whose research is widely quoted in journals and encyclopedias. He is the author of the Critical Focus column, which has been published quarterly in *The Microscope* journal since 2010. When he is not speaking at Inter/Micro, Ford travels the world as a speaker and presents his work on television and radio. In 2017, Ford was named an Honorary Fellow of the Royal Microscopical Society. He is a fellow of the Cardiff University; former tutor at Madingley Hall, Cambridge University; and president-emeritus of the Cambridge Society for the Application of Research. He has also served as a fellow of the Open University and visiting professor at Leicester University. Ford has given his Evening with Brian presentations at Inter/Micro for more than 30 years.

Tuesday, June 12

Environmental and Industrial Microscopy

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

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

Chair: Kelly Brinsko Beckert — Microtrace LLC

The "Sinkhole" Conundrum: Legal Challenges to XRD-SEM Evidence for Liability for Substructure Soil Collapse
Wayne C. Isphording — University of South Alabama and Tulane University

Creating an Aqueous Precipitation and Crystal Table for Microcrystal Tests of Common Illicit Drugs
Casey Turney — University of Illinois at Chicago Forensic Science Program; and McCrone Research Institute

Surface Samples for Indoor Environmental Quality: What Can They Tell Us?
Andrew A. Havics — pH2, LLC

Morning Break

The Curious Case of Asbestos Dental Tape
James R. Millette — Millette Technical Consulting

Elongate Mineral Particles: What Sizes Matter?
Eric J. Chatfield — Chatfield Technical Consulting Limited

Pigments in Olefin Fibers
Kelly Brinsko Beckert — Microtrace LLC

12:00–2:00 p.m. Lunch, McCrone Garden

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

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

A Survey of Elements Detected in Multi-layered Automotive Paint Samples by SEM-EDS
Ethan Groves — Microtrace LLC

A Survey of Analytical Considerations for the Elemental Analysis and Forensic Comparison of Automotive Paints by SEM-EDS
Christopher S. Palenik — Microtrace LLC

Solving Problems with Chemical Microscopy
John A. Reffner — John Jay College, CUNY

Afternoon Break

Antibiotic Anarchy
Brian J. Ford — Cardiff University

The "Green" Movement and Paper Fiber Usage
Walter J. Rantanen — SGS-IPS Testing

A Case Example Demonstrating the Value of Raman Microspectroscopy in Forensic Rope Examinations
Andrew M. Bowen — U.S. Postal Inspection Service

See Tuesday presentation abstracts on page 19.

Tuesday, June 12

Exhibitor Booths

9:00 a.m.–5:00 p.m. Tuesday, June 12 and Wednesday, June 13, McCrone Classroom

Learn about the latest microscopy innovations and products from our exhibitors, NIGHTSEA and EDAX, Inc.

SMSI Silent Auction

12:00–5:00 p.m. Tuesday, June 12
9:00 a.m.–3:40 p.m. Wednesday, June 13, McCrone Classroom

Bid on microscopy equipment and other related items of interest at the annual silent auction benefitting the State Microscopical Society of Illinois (SMSI). Winners will be announced Wednesday afternoon after the speaker presentations.

Reggie's Rooftop Dinner

5:30–8:30 p.m. Reggie's Rock Club Rooftop Deck, 2109 S. State Street

Unwind on a pleasant summer evening with fellow Inter/Micro attendees and sponsors for refreshments and dinner on Reggie's spacious rooftop patio, located a few blocks away from McCrone institute. A trivia contest will follow dinner. Transportation to Reggie's from McCrone will be provided by Reggie's colorful bus. The rooftop dinner is sponsored by Cargille Labs and the American Society of Trace Evidence Examiners (ASTEE).

Wednesday, June 13

Chemical and Forensic Microscopy

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

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

Chair: Patrick Buzzini — Sam Houston State University

***The Technician*: A Short-Lived 1940s Forensic Science Journal**
William A. Randle — Missouri State Highway Patrol Crime Lab

The Burning Rock
Skip Palenik — Microtrace LLC

The Curious Case of the Yellowing Labels: An Investigation into the Discoloration of Package Labels
Brendan Nytes — Microtrace LLC

Morning Break

Product Discoloration: Analysis of an Unknown Red Colorant
Katie M. White — Microtrace LLC

The Effect of Ultraviolet Radiation on the Microspectrophotometry (MSP) of Dyed Fibers, Part 1: Photobleaching Effect
Meggan B. King — McCrone Research Institute
Patrick Buzzini — Sam Houston State University

The Effect of Ultraviolet Radiation on the Microspectrophotometry (MSP) of Dyed Fibers, Part 2: Spectral Differences as a Function of Time
Patrick Buzzini — Sam Houston State University
Meggan B. King — McCrone Research Institute

12:00–2:00 p.m. Lunch, McCrone Garden

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

Chair: Casey Brown — The Pennsylvania State University

Aquatic Dinosaur Update II
Brian J. Ford — Cardiff University

Contributions of Polarized Light Microscopy and Microanalysis to a Recent Mail Bomb Case
Andrew M. Bowen — U.S. Postal Inspection Service

Afternoon Break

Screenshot: Analysis of Bullet Hole Impact Dynamics
Peter Diaczuk — The Pennsylvania State University, Eberly College of Science, Forensic Science Program

Semi-Automated Micromorphometry of Small Arms Propellants for Brand Identification
Casey Brown — The Pennsylvania State University

See Wednesday presentation abstracts on page 27.

Wednesday, June 13

State Microscopical Society of Illinois 2018 Awards Dinner and Live Auction

Eric J. Chatfield • 2018 SMSI August Köhler Award Recipient

Presented at Harry Caray's Italian Steakhouse, 33 W. Kinzie Street, Chicago

6:30–7:30 p.m. Social hour, and live auction hosted by Brian J. Ford

7:30–8:30 p.m. Dinner

8:30–9:30 p.m. Award announcement and presentation

Join Inter/Micro and the State Microscopical Society of Illinois (SMSI) as they honor Eric J. Chatfield with the 2018 August Köhler Award. The SMSI will also be celebrating its 150th year (1868–2018).

Dr. Chatfield received his B.A., M.A., and Ph.D. from Cambridge University in the United Kingdom. From 1958 to 1968, he was with the United Kingdom Atomic Weapons Research Establishment at Aldermaston. His work there involved generation and characterization of plutonium aerosols that could be released as a consequence of accidents involving nuclear weapons or plutonium-fuelled nuclear reactors. For these studies, a range of techniques for air sampling methods were developed using electron microscopy and electron diffraction to characterize the particulate materials. In 1968, Dr. Chatfield moved to Canada, where he was head of the Electron Optical Laboratory at the Ontario Research Foundation (ORF) until 1986. Light microscopy, electron microscopy, and electron microprobe analysis were applied to research on behalf of industrial and government clients. In the early 1970s, asbestos research and analysis became a large part of the laboratory's work. At ORF, he established the first commercial PLM and TEM asbestos analysis service in Canada. In 1986, Dr. Chatfield founded Chatfield Technical Consulting Limited, where he is president and principal analyst. He has developed a number of analytical methods for the determination of asbestos and continues to conduct research in the area of asbestos characterization. He is convenor of the International Organization for Standardization (ISO) working group that has produced a number of international standards for determination of asbestos in air samples and bulk materials. Dr. Chatfield is a founding member of the Microscopical Society of Canada, a fellow of the Chemical Institute of Canada, and has more than 60 publications relating specifically to asbestos measurement.

Thursday–Friday, June 14–15

Workshop: Wood Structure and Identification

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

Taught by Regis B. Miller, Ph.D.

This two-day beginner's workshop will concentrate on the wood structure and identification of common hardwoods and softwoods. One full day will be devoted to softwoods and the other day to hardwoods. Participants will examine blocks of wood with the naked eye, exploring such features as color, grain, and density. They will examine the cross (transverse) surface of the blocks with a hand lens, noting features such as vessel size and arrangement, ray sizes, and axial parenchyma patterns. Finally, participants will learn to make thin sections of the radial and tangential surfaces for use with the light microscope.

This workshop will be taught by wood identification and information specialist **Regis B. Miller, Ph.D.**, retired wood anatomist with the Forest Products Laboratory. Tuition includes lunch and refreshments for both days.

Monday, June 11

Techniques and Instrumentation

Image Acquisition and Analysis of Soybean Stem Sections

Aryeh Weiss — Faculty of Engineering, Bar Ilan University, Ramat Gan, Israel
Anne Alerding — Department of Biology, Virginia Military Institute, Lexington

Soybeans are among the major agricultural crops planted in the United States, with a total production of 4.39 billion bushels in 2017. Over the last decade, production research efforts have been unable to create soybean cultivars that consistently surpass 40 bushels per acre in Virginia fields. Improving yields requires new directions in production research. As part of this work — and in order to understand the influence of plant tissue morphology on new branch growth (cell and cell wall synthesis) and seed abortions — we are studying the growth of tissues in the stem using brightfield and fluorescence imaging. In these studies, tiled, large-area transmitted light and fluorescence images of histoslides of stem cross-sections were acquired, stitched, and analyzed using the Fiji version of ImageJ.

In this talk, acquisition and subsequent image processing workflows will be presented, and the following topics will be discussed:

1. Segmentation of brightfield color images, using color deconvolution.

2. Acquisition of large area fluorescence, and the advantages of fluorescence over brightfield imaging.

3. Segmentation of the fluorescence images, using spectral unmixing.

4. A tool that can be used to determine optimal combinations of objective magnification and numerical aperture, relay optics magnification, and camera properties.

Eight Is Enough: Fabric Physical Matches in an International Drug Smuggling Case

Jason C. Beckert — Microtrace LLC

Physical matches represent the highest level of associations that can be made using trace evidence. When a physical match is demonstrated, the examiner can conclude that two separate objects were once joined together as a single object. However, some substrates (e.g., fabric) can deform during the breaking process, and the significance of the conclusion is often reduced as a result.

This presentation will focus on a case study involving the physical matching of fabric straps to a bag containing drugs in a smuggling case. The condition of the evidence was a key factor and multiple fracture matches were observed in the slightly distorted fabric. While most of the individual fracture matches would be compelling enough on their own, the totality of the evidence clearly indicated that the objects were originally joined together.

Discriminating Glass Fragments Using Micro-XRF Spectrometry with Polycapillary Optics

Bruce Scruggs — EDAX, Inc.

Glass fragment analysis is a common problem found in forensic and industrial laboratories. The typical issue is to compare glass fragments from a known source to an unknown source and show that the unknown fragments are either elementally consistent or inconsistent with the known fragments. There are various characterization tests available, including optical methods, physical methods, and elemental analysis. Micro-XRF spectrometry has been widely used for elemental analysis because it is non-destructive, it has acceptable detection limits, and data acquisition can be run unattended.

XRF intensity ratios are commonly used in U.S. forensic labs according to ASTM E2926-13 for window glass fragment comparisons. The method is commonly implemented using one set of spectral acquisition conditions to collect XRF spectra. This presentation demonstrates the use of multiple acquisition conditions to optimize data collection over different energy ranges. The analysis is facilitated by

using polycapillary X-ray optics with spot sizes <100 μm FWHM, while generating high signal intensities for improved detection limits. The goal for this collection methodology is to eliminate spectral artifacts that may complicate comparisons of desired trace elements while maintaining good detection limits for the most important elements involved in the comparisons.

Measurement of Nanoparticle Size Using Transmission Electron Microscopy

Jacob M. Spry and Richard S. Brown — MVA Scientific Consultants

This presentation addresses challenging tasks associated with achieving quality measurements of various nanoparticles using transmission electron microscopy (TEM). Critical to the imaging and sizing process is the preparation of the sample prior to transfer to a TEM grid. Preparing nanomaterials in such a way that will minimize agglomeration and allow for reliable sizing of individual nanoparticles is critical. Various methods will be discussed, such as the media and physical separation methods utilized for nanoparticle dispersion, additional agents that can be added to suspensions to further improve dispersion quality, and methods used to transfer nanoparticle suspensions to TEM grids. TEM imaging methods will also be explained, including selection of quality regions for imaging, identifying a suitable magnification for sizing, and maximizing image quality for accurate particle sizing. Finally, image sizing will be discussed, including software utilized for nanoparticle sizing, criteria considered when sizing nanoparticles, and discerning particles compared to potential particle agglomerates during the sizing process. Examples of various nanoparticle TEM preparations will be shown, to illustrate the various challenges.

Hacking a Dinosaur: Upgrading a Vintage Microscope Using the Raspberry Pi

Martin Kocanda — Rapid City Police Department, South Dakota

Vintage microscopes have occasionally surfaced on the surplus market. With some effort, the scopes can be rebuilt, but retrofitting

them for photomicrography with commercial hardware can be challenging. With the availability of low-cost, single-board computers, camera peripherals, PVC fittings, and some programming experience, an older compound microscope can be upgraded to capture quality video and images. Using a Raspberry Pi 3, an open-source hardware and software platform, a digital imaging system is easily retrofitted for under $75.

Problems in Raman and FTIR Spectral Library Searches
Lawrence Wayne — Forensic Analytical Laboratories, Inc.

Raman spectroscopy and Fourier-transform infrared spectroscopy (FTIR) frequently rely on search libraries to identify an unknown substance. While analysts are usually familiar with the inherent limitations of the use of spectral libraries, their use is based on the assumption that the libraries have been prepared, examined, and labeled properly. Examination of many available libraries, private and commercial, has revealed library spectra that are either poorly collected or mislabeled entirely. This may result in misattribution or misidentification of an unknown substance. Examples of poor quality and incorrectly labeled spectra will be presented, together with suggested methods to reduce the possibility of misidentification due to poor library quality.

Evaluation of the Criteria for the Discrimination of Inkjet Printer Inks Using Micro Raman Spectroscopy
Patrick Buzzini, Carrie Polston, Madison Schackmuth — Sam Houston State University

Inkjet printers are ubiquitous devices in our society. Therefore, it is not surprising that they are used to commit different types of illicit activities such as threats or extortions by means of anonymous letters, frauds in the context of disputed contracts, alterations to or counterfeit of ID documents, and counterfeit of currency banknotes. Inkjet printed documents produce microscopic colored dots that can be detected using a microscopical approach in conjunction with Raman spectroscopy. In the phase of this study, the criteria necessary

to achieve discrimination between inkjet printer inks from different manufacturers, same manufacturers, and even same manufacturer models have been evaluated on a set of 11 samples. Although Raman spectroscopy is already a relatively well-established method for the characterization of colorants (both dyes and pigments), this study demonstrates that the contribution of minor peaks within Raman spectra improves the discriminating capabilities of the technique. The joint consideration of the three colored components was also effective in differentiating inkjet printer ink samples, especially those from the same manufacturer, and in some instances, from the same model as well.

Development of Modern Microcrystal Tests for Controlled Drugs, Diverted Pharmaceuticals, and Bath Salts: Project Update
Sebastian B. Sparenga — McCrone Research Institute

Beginning in 2016, McCrone Research Institute began a research project, awarded by the National Institute of Justice (2015-NIJ-CX-K010), to discover and develop microcrystal tests for a number of emerging drugs and pharmaceuticals that were seeing a marked increase in abuse. After two years of research, successful microcrystal tests for 10 additional drugs have been developed. Each new microcrystal test includes recommended protocols and morphologies of crystals including photomicrographs, infrared spectra of microcrystal precipitates, potential interferences, and optical crystallographic properties of the resultant microcrystals and will be included in *A Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals*, originally published by McCrone in 2015. This presentation will discuss the current progress and examine what is still needed for project completion.

Taking a Closer Look at Some Photochromic Materials
Joseph Insana — Microtrace LLC

Photochromism is the phenomena by which a particular substance has a composition that allows it to transition or transform into a different chemical state by exposure to electromagnetic radia-

tion. One of the more common consumer examples of a photochromic material are transition glasses. The lenses in the frames of these glasses are clear under artificial lighting conditions; however, when exposed to UV radiation generated by the sun, they become increasingly more opaque and block out a portion of the sunlight. This effect is created by the addition of crystalline halide inclusions within the glass that react to the UV radiation, which in turn, creates the opaque finish. This same principle can even be used to create colorful motifs on various consumer goods. This presentation will discuss the results of examinations of multiple photochromic materials and how microscopy, along with chemical analysis, can be used to understand the basic principles of photochromism.

Microscopy and Poetry: A Union of Art and Artifice
Andrew A. Havics — pH2, LLC

Microscopy allows one to peer into a visual world unseen by the naked eye, evoking thoughts and sometimes emotions; whereas poetry inspires one to create visions, emotions, and thought patterns using only words. The two worlds have been destined to intersect in both predictable and unexpected ways. The words alone have less meaning without first having viewed the microscopical images, and yet, the images evoke a desire to put words to paper. It is a marriage of art and artifice that often inspires microscopists to continue writing outside the lines of science. Thus, one begins an exploration of verse and rhyme: sonnets, odes, epigrams, limericks, poetry masquerading as quotes, and so on.

Further Adventures in Fluorescence
Charles Mazel — NIGHTSEA

As a developer of equipment for viewing and documenting fluorescence, NIGHTSEA encounters diverse opportunities and challenges in applying the technique. In the past year, we have worked with subjects including microplastics, planaria, cement, fingerprints, and bathroom towels. We have added fluorescence to platforms ranging from underwater vehicles to high-end digital microscopes

at magnifications up to 2,000×. This talk will review some of these experiences, what we have learned, and what we have developed.

The "Sinkhole" Conundrum: Legal Challenges to XRD-SEM Evidence for Liability for Substructure Soil Collapse

Wayne C. Isphording — University of South Alabama and Tulane University

A ongoing confrontation exists when claims for coverage for property damage are challenged by companies who defend wording in insurance policies, even when that wording is shown to be ambiguous. This problem is especially common in areas where subsidence has taken place due to dissolution of the substrate by the action of groundwater.

Unfamiliarity with the nuances of geochemical systems, for example, results in attorneys believing that all subsidence resulting from groundwater solution takes place on either limestone or gypsiferous-rich bedrock. The idea that other types of rock, such as sandstones, argillaceous sediments, granites, or even peridotites can undergo incongruent dissolution and give rise to topographic features typical of "karst terranes" is totally foreign to them and, understandably, beyond their normal "expertise." Therefore, they react negatively when insurance policies that restrict subsidence coverage to structures located on "limestone, or similar rock" are challenged by plaintiffs, whose subsidence damage has taken place beneath structures constructed on non-carbonate rocks. Ambiguity in a legal document, however, must *always* be interpreted in favor of the "non-drafter of the contract." Such was the case for a home in Mobile, AL built on argillaceous sediments belonging to the Plio-Pleistocene Citronelle Formation, a Gulf Coastal Plain unit that extends from Mississippi, eastward through Alabama, Florida, and Georgia. X-ray diffraction (XRD) and scanning electron microscopy (SEM) analyses reveal that the sediments of this unit are composed chiefly of quartz and kaolinite, but also contain abundant gibbsite. The origin of the gibbsite has been identified as resulting from the incongruent dissolution of kaolinite by the reaction shown below [next page]:

$Al_2Si_2O_5(OH)_4 + 5H_2O \rightarrow 2Al(OH)_3 + 2H_4SiO_4(aq)$

Kaolinite	Gibbsite
S.G. 2.6 (258 amu)	S.G. 2.4 (78 amu)

The irreversible mass transfer associated with the above reaction, and the concurrent volume change of solids, amounts to 34.6%. As such, it is wholly sufficient to explain the settlement phenomenon that has and continues to take place beneath the home. Therefore, it accurately substantiates that the settlement and subsoil collapse resulted from "subterranean voids created by the action of water *on limestone or similar rock formations*" [author's italics] and, therefore, should be covered by the policy purchased by the homeowner. The jury accepted the arguments proffered by the plaintiff's expert witness.

Creating an Aqueous Precipitation and Crystal Table for Microcrystal Tests of Common Illicit Drugs

Casey Turney — University of Illinois at Chicago Forensic Science Program; and McCrone Research Institute

Microcrystal tests are chemical precipitation tests using a variety of aqueous reagents to identify various drugs using a polarized light microscope. Thirty-four microcrystal test methods were checked for unique microcrystal formation with 21 different drugs. These tests were selected from *A Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals*, published by McCrone Research Institute. The results were tabulated for easy access and for use as an identification tool in forensic science laboratory settings. Of the 21 drugs in the table, two of the microcrystal test results using picric acid and dilituric acid reagents for methylphenidate and pseudoephedrine, respectively, are difficult to interpret and additional microscopical characterization or alternate microcrystal tests may be required for their identification.

Surface Samples for Indoor Environmental Quality: What Can They Tell Us?

Andrew A. Havics — pH2, LLC

Indoor environmental quality (IEQ) studies often include sampling surfaces. These may be tape-lift samples, bulk dust, micro-

vacuum samples, RODAC plates, or settling plates. For mold cases, samples help evaluate presence, distribution, and growth or mold while the identification can help assist with conditions leading to their presence. For irritation and allergy concerns, the identification of particles can aid in narrowing or eliminating sources and origins. For soot particles, the identification and surface distribution can provide information on sources and the likelihood of them needing cleaning. Other particle types fill in gaps for understanding the environment, the inhabitants, the buildings they inhabit in addition to the particle sources and sinks.

The Curious Case of Asbestos Dental Tape

James R. Millette — Millette Technical Consulting

In 1976, after a review of death certificates, Menck and Henderson concluded in an article published in the *Journal of Occupational Medicine* that "Occupational groups found to be at excess risk (of asbestos disease) who have not been previously implicated included roofers, dental technicians…." Asbestos had been used in dental lining tape from the 1930s until the 1980s. The inner surface of a crucible or ring used in the "lost wax method" of casting dental prostheses was lined with the asbestos tape prior to casting at a high temperature. In 1977, dentists at the Army Institute of Dental Research in Washington used scanning electron microscopy (SEM) to find that asbestos fibers are released when ring liner was torn from a roll of asbestos by each of two laboratory workers. Our lab tests using polarized light microscopy (PLM), phase contrast microscopy (PCM), SEM, and transmission electron microscopy (TEM) showed chrysotile contents ranging from 40% to 95% in two brands of dental tape. Glove box testing of tearing the tape showed levels from 0.5 to 4 F/cc (fibers per cubic centimeter). Full chamber tests were 0.66–4.6 F/cc. Tests showed that more than 60,000 asbestos fibers (>5 μm long) were released during a single tear.

Elongate Mineral Particles: What Sizes Matter?

Eric J. Chatfield — Chatfield Technical Consulting Limited

The length distributions of single fibrils of Coalinga chrysotile,

UICC-B chrysotile, and wet dispersed chrysotile were measured by transmission electron microscopy (TEM). It was found that the length distributions significantly diverged only above approximately 10 μm in length. These results correspond to differences in published results of animal experiments. This observation suggests that chrysotile fibrils shorter than approximately 10 μm are not correlated with the tumor incidences observed in prior animal experiments in which these sources of chrysotile were tested. This result is in contrast to published data in which counting of an insufficient number of fibers resulted in an erroneous conclusion that the length distribution of Coalinga chrysotile fibrils was indistinguishable from those of other sources of chrysotile.

The size distributions of the respirable particle size fractions from acknowledged tremolite asbestos samples were found to be dominated by elongate particles longer than 5 μm, which are within the dimensional range of non-asbestiform amphiboles. Prior studies have shown that these elongate particles obscure a correlation between a specific size range of particles and results of animal implantation studies using tremolite of various morphologies. In the prior studies, a reference protocol was developed from TEM measurements of particles from four crushed non-asbestiform amphiboles to differentiate the size range of amphibole particles that correlates with the mesothelioma frequencies observed in the animal studies. In the work reported here, this correlation was tested using TEM analyses of amphiboles from Libby, MT; Sparta, NJ; and Homestake Mine, Lead, SD, which represent known environmental/occupational situations.

Further TEM analyses of the tremolite samples used in the original animal implantation studies have shown that the numbers of elongate tremolite particles with lengths ≤5 μm implanted into the animals are not correlated with the observed mesothelioma frequencies. The results suggest that elongate tremolite particles with lengths ≤5 μm do not contribute to the carcinogenic response observed in the animal experiments.

Pigments in Olefin Fibers

Kelly Brinsko Beckert — Microtrace LLC

Due to their lack of binding sites, olefins such as polypropylene and polyethylene are difficult to dye after the fiber has been formed. Therefore, nearly all olefins are colored with pigments while the polymer is still molten prior to extrusion. Using pigments instead of dyes results in lightfast fibers, and this feature, together with other desirable properties such as stain and chemical resistance, give olefins application across many industries, including apparel, upholstery, carpeting, and building materials. These pigments are often difficult to see by light microscopy due to their small size, but there are several microscopical techniques which may be helpful, including phase contrast and polarized light. The pigments may also be analyzed using instrumental methods such as Raman microspectrometry, microspectrophotometry (MSP), or micro-FTIR.

A Survey of Elements Detected in Multi-layered Automotive Paint Samples by SEM-EDS

Ethan Groves, Lina T. Michely, and Christopher S. Palenik — Microtrace LLC

This talk presents the results of a survey of 300 automotive paints, comprised of over 1,200 layers, which have been characterized by energy-dispersive X-ray spectrometry (EDS) in a scanning electron microscope (SEM) to identify detectable elements and their frequency of occurrence as a function of layer type (clear coat, color coat, and primer). This seemingly trivial exercise is subject to numerous challenges, which illustrate the impact that data collection, data interpretation, and selection criteria can have on the ultimate list of detected elements. For example, selection criteria based upon auto-identification, a fixed element list, or evaluation by standardless quantification (e.g., >3 standard deviations, counting error) can lead to different results. Because this data is collected from "street" samples where the ground truth is not known, it is not possible to establish absolute selection criteria. However, despite this challenge, the resulting list provides a context for developing more formalized

approaches to the interpretation of elemental analysis data for forensic paint comparisons.

A Survey of Analytical Considerations for the Elemental Analysis and Forensic Comparison of Automotive Paints by SEM-EDS
Christopher S. Palenik, Ethan Groves, and Lina T. Michely
— Microtrace LLC

There is no doubt that the forensic comparison of paint based upon elemental analysis is probative, but the present ASTM guide on the forensic comparison of paint samples by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS) offers little guidance on the impact that sample preparation, analytical parameters, and data evaluation have on the results. While both the theoretical underpinnings for the elemental analysis of materials by SEM-EDS and a practical approach to the comparison of paints have been well established, the theoretical and practical have never been married. For example, paint samples are often prepared with little attention to how sample preparation may impact analytical results; the analysis of a given paint layer is conducted with limited considerations for the impact of analytical parameters; and the criteria for associating or excluding samples is based on a subjective comparison of data with little understanding of significance. This talk will discuss various theoretical factors that should be considered in addition to the practical limitations that exist. Data from experiments will be presented to illustrate the impact such considerations may have on the interpretation and conclusions formed from the EDS analysis of paints.

Solving Problems with Chemical Microscopy
John A. Reffner — John Jay College, CUNY

Happiness lies in the joy of achievement and the thrill of creative effort. Microscopy provides true happiness by providing the means to observe the micro world, creating beautiful images, and for solving problems. I'll share a few examples of the joy I have achieved and the thrill of creative effort that I have experienced. For instance, I

became my daughter's hero when, aided by a magnifying glass and needle-nose tweezers, I skillfully removed a small glass fragment from her foot. In another example, I was introduced by Dr. Roy P. Allen to his method for differentiating carbon blacks by their color. Carbon black rubber samples were made as reference standards. These were used to analyze other black rubber samples. Small fragments of each rubber and a small drop of mineral oil was placed on a microscope slide. A second slide placed on top. Pressure forced the two pieces of black rubber to come together. For rubbers containing the same carbon black, the colors were indistinguishable. I was happy when I applied this technique to solve a problem involving a conducting carbon black. I hope sharing my experiences will stimulate open discussion of favorite stories from the audience of chemical microscopists.

Antibiotic Anarchy
Brian J. Ford — Cardiff University

Future medicine is being threatened by the widespread development of antibiotic resistance in bacteria. This is not new. When Alexander Fleming accepted the Nobel Prize in 1945 for his discovery of penicillin, he warned of the dangers posed by resistance of bacteria to antibiotics. There has been flagrant misuse of these potent antibacterials, (in agriculture for instance), but currently our problems are caused not by overuse, but by a failure to target therapy more effectively. Surprisingly, it is the underuse of antibiotics that poses the greatest problems.

The "Green" Movement and Paper Fiber Usage
Walter J. Rantanen — SGS-IPS Testing

The "Go Green" movement is promoted and embraced by environmental activists and supporters. This concept had also been adopted by many companies to encourage customers to use digital contact or records to replace the paper hard copy. Within the paper industry there have been fiber-type changes and sustainability claims to accommodate the "Green" narrative. In certain paper products,

there are marketing labels claiming tree-free, non-wood plant fiber; natural fiber; cellulose fiber; recycled fiber; post-consumer waste fiber; sustainably sourced furnish; and other terms. Some claims specify fiber types that may be popular with many consumers such as bamboo, hemp, wheat straw, and cotton. Some of these claims may be suspect or partial truths. Light microscopy is usually the best method to confirm, refute, or understand the claimed contents in paper products.

A Case Example Demonstrating the Value of Raman Microspectroscopy in Forensic Rope Examinations

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

An outside agency case was submitted to the U.S. Postal Service Forensic Laboratory Services in 2017 requesting comparison of rope and sand samples to assist a major drug investigation. The results of the laboratory examination of these materials will be shared during this presentation. In particular, Raman microspectroscopy analysis was used to identify several pigments in two submitted ropes, and this information made the comparison of both rope samples considerably more probative. Polarized light microscopy was used for characterization and comparison of the submitted materials and to assist in the selection of pigment particles for Raman analysis.

Wednesday, June 13

Chemical and Forensic Microscopy

The Technician: A Short-Lived 1940s Forensic Science Journal

William A. Randle — Missouri State Highway Patrol Crime Lab

John E. Davis was a well-known forensic scientist and director at the Oakland Police Department Crime Laboratory. What is not so well known is that he started his career at the Missouri State Highway Patrol Lab in Jefferson City, MO, and at that time, he also edited, published, and contributed to an early forensic science journal called *The Technician*, published from May 1943 to April 1944. This journal was unique for its time because it was written specifically for laboratory technicians working as forensic scientists and criminalists. This talk will present the lost volumes of *The Technician* and also provide some information about Mr. Davis.

The Burning Rock

Skip Palenik — Microtrace LLC

On a cold Thursday in Chicago, January 2018, a construction crew working on an empty lot on which a school was being built in the South Loop, was surprised when the walls of a trench they had just dug began to smoke. Smoking sewers are not an unusual site in the city during winter. The crew assumed this was due to the condensation of steam, until a backhoe was digging in the same area later in the day, and a piece of gravel that fell off the shovel began to emit white smoke, which suddenly became incandescent and burst into flame. At this point, the site was closed and an environmental firm was brought in to evaluate the safety of the situation. They had a rush analysis performed on the item at a local laboratory, which issued a five-page report (three of which were their credentials and certifications), showing the presence of several dozen elements and their quantities (by ICP-OES), in addition to a two-sentence result of their microscopical examination on one of the pages indicating that the sample looked like "wet drywall." Because the report did not, understandably, solve the problem, we received a call to see what we

might be able to do on Friday afternoon. The item was delivered to our laboratory early Saturday morning by a messenger from the environmental firm, and we began our analysis of it.

This presentation will describe the course of the analysis that we undertook that weekend, which led to the surprising solution to the problem and permitted work to begin at the site again on Monday. It will also explain why the substance responsible for the incident did not appear among the list of elements (amounting to almost half of the periodic table) in the report of the first analysis. This also helped illustrate the speaker's warning that not all laboratories are the same.

The Curious Case of the Yellowing Labels: An Investigation into the Discoloration of Package Labels

Brendan Nytes — Microtrace LLC

As a microanalytical laboratory, we encounter many different types of samples, with discolorations being one of them. They are typically easier to observe using the unaided eye and are often barely visible, if at all, by light microscopy. A seasoned microscopist understands that discolorations are a challenging endeavor that is often not straightforward. In some cases, they are just simply fine iron oxide particles imparting an amber color to a material. In other cases, the discoloration is the result of a diffuse color that is not composed of discrete particles. The use of extractions, fluorescence, and microanalytical techniques become essential in the identification of theses discolorations.

In this case example, we will discuss the presence of a yellow discoloration that developed on the surface of labels in a warehouse. The curious part was that the yellow discoloration was not uniformly present, seemed to develop randomly, and only appeared on labels in one particular warehouse.

Product Discoloration: Analysis of an Unknown Red Colorant

Katie M. White and Christopher S. Palenik — Microtrace LLC

In the manufacturing industry, unwanted discoloration can result from many different mechanisms, including charring, chemical change, and transfer of a liquid or fine particles. Regardless, our

analytical approach typically begins with a microscopical examination of the sample. When discrete colored particles are observed and suspected to be pigment, Raman spectroscopy is an ideal analytical tool for characterization. In situ analysis can be particularly useful for pigments, but interferences with the sample matrix may require further isolation of the colorant for analysis.

Some discolorations are submitted to us as complete unknowns, and we rely on our spectral reference library to determine possible sources for the colorant. In other cases, clients come to us with a suspected source in mind, but before a definitive association can be made, the known colorants must be characterized and compared to the unknown sample.

This presentation will discuss our analysis of a discolored chemical product submitted to our laboratory for identification and comparison to a known source. It will also demonstrate the importance of subtle differences when comparing Raman spectra. Additional case examples involving pigment identification will be highlighted.

The Effect of Ultraviolet Radiation on the Microspectrophotometry (MSP) of Dyed Fibers, Part 1: Photobleaching Effect

Meggan B. King — McCrone Research Institute
Patrick Buzzini and Carrie Polston — Sam Houston State University

Single fibers from a collection of 53 textile samples were analyzed using microspectrophotometry (MSP) after undergoing controlled, instrument-induced photobleaching. The sample set consists of 20 nylon, 14 polyester, eight acrylic, six viscose rayon, and five acetate rayon fiber types dyed with a variety of colors (15 yellow, 14 red, 11 blue, four orange, four violet, three brown, one black, and one green) and dye-application classes (29 disperse, 10 basic, six acid, five direct, two mordant, and one reactive). The dyed fibers were each exposed to the xenon light source on a CRAIC FLEX UV-visible-NIR microspectrophotometer for times ranging from 0 to about 3,700 seconds, with spectra collected every 60 seconds. Spectral alterations were observed in all but one sample. Spectral alterations occurred at different times ranging from about 20 seconds and 1,730 seconds. Spectral changes occurred in various forms: band shifts in

the x-axis (wavenumber units) were commonly observed, and cases where spectral shapes were preserved but the overall intensity of all the bands was out of the range of the intra-source variation of spectra collected without further exposing the fibers to the excitation radiations. Changes in intensity values of one or few bands within a spectrum were also observed. Effects of band flattening, bump formation, bands fusion, slope change, and band disappearance were also noted. Combinations of the aforementioned spectral alteration modes were also recorded. In 28 instances, spectral alterations were observed in the visible range of the spectrum, while in 13 samples such changes were observed in the short UV range. In nine cases, spectral changes were observed in both the UV and visible ranges, where they did occur at different times. This research was funded by the National Institute of Justice (Award No. NIJ-2016-DN-BX-0145).

The Effect of Ultraviolet Radiation on the Microspectrophotometry (MSP) of Dyed Fibers, Part 2: Spectral Differences as a Function of Time

Patrick Buzzini and Carrie Polston — Sam Houston State University
Meggan B. King — McCrone Research Institute

Single fibers from a collection of 53 textile samples were analyzed using UV-Vis microspectrophotometry (MSP), at eight week intervals, after being exposed to outdoor sunlight and placed into a laboratory UV radiation box. Differences between MSP spectra collected from fibers prior to exposure (T0) and after exposures of 8, 16, and 24 weeks (T8, T16, and T24, respectively) will be discussed. These samples span a variety of fiber types, dye types, and colors including 20 nylon, 14 polyester, eight acrylic, six viscose rayon, and five acetate fiber types dyed with a variety of colors (15 yellow, 14 red, 11 blue, four orange, four violet, three brown, one black, and one green) and dye-application types (29 disperse, six acid, 10 basic, five direct, two mordant, and one reactive). After a 24-week exposure, 15 samples out of the 53 did not exhibit changes in the shape and intensity of the collected spectra. However, 32 samples displayed spectral differences after exposure to sunlight or laboratory UV radiation. In 20 cases, spectral alterations were observed in the visible

range only; changes in only the UV range were observed for two samples, while the remaining 10 samples displayed spectral differences in both the UV and the visible spectral ranges. The majority of samples (16) started showing spectral differences at T8, while seven samples showed spectral differences starting at T16. Finally, in six samples changes were observed only after T24. Observed types of alterations consisted of a decrease of band intensities, formation of new bands, or band shifts in wavelength values. Six samples displayed a large within-source variation, preventing the determination of whether spectral variations were due to alterations as a function of UV exposure. This research was funded by the National Institute of Justice (Award No. NIJ-2016-DN-BX-0145).

Aquatic Dinosaur Update II

Brian J. Ford — Cardiff University

What has happened since Brian J. Ford put the cells in a dinosaur's tail under the lens for Inter/Micro on July 17, 2012, wrote in *The Microscope*, 60:3, pp 123–131 in 2012, and presented his talk "Aquatic Dinosaur Update" at Inter/Micro on July 16, 2013? His views attracted worldwide hostility from paleontologists, yet May 2018 saw the publication in London of his 400-page book on these new theories. Today, we will learn what has happened since. This is a saga of rivalry and double-dealing, of vitriol and bitter animosity — with lessons about the way modern science is being conducted.

Contributions of Polarized Light Microscopy and Microanalysis to a Recent Mail Bomb Case

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

A recent explosives case involving two mail bombs that exploded when opened by customers demonstrates the value of polarized light microscopy (PLM) in explosives examinations. The combined use of PLM with Raman microspectroscopy, Fourier-transform infrared microspectroscopy, and energy-dispersive X-ray spectroscopy enabled a very small deposit of material on a match head to be examined and compared to matches recovered during the execution of

search warrant at a suspect's residence. The small quantity of available material necessitated a microscopical approach to its analysis. In addition, these same techniques were valuable in the analysis of a number of other materials submitted for examination in this case.

Screenshot: Analysis of Bullet Hole Impact Dynamics

Peter Diaczuk and Xiao Shan Law — The Pennsylvania State University, Eberly College of Science, Forensic Science Program

This research project originated from a shooting case involving a suspected bullet hole through a window screen. After passing through the window screen, the bullet then passed through a plastic bottle on the window sill and continued until it encountered an occupant of the premises. When submitted for examination, the window screen contained two holes, so it became important to determine whether one or both holes found in the screen were made by one or more bullets, and could an angle be approximated (or excluded) based on the shape of the holes. The pre-existing gaps in aluminum window screens, which are formed by the warp and weft pattern of aluminum wire (similar to fabrics) were examined. Likewise, the damage to the bottle had to be assessed and evaluated. To determine differences in the entry and exit hole deformation, we fastened pieces of window screen material to wooden frames to simulate the frame of a typical window and fired upon them with different caliber bullets at different velocities and angles. High-speed photography was able to clarify the mechanism of bullet impact dynamics both through the screen and through the bottle on the window sill.

Semi-Automated Micromorphometry of Small Arms Propellants for Brand Identification

Casey Brown, Jack Hietpas, and Wayne Moorehead — The Pennsylvania State University

According to a 2016 U.S. Bomb Data Center report, smokeless powders were one of the top explosive charges used in illicit detonation events. Smokeless powders are commonly used as the main charge in pipe bombs, the most common form of an impro-

vised explosive device, likely due to their ease of procurement and unrestricted purchase. The focus of this study is to investigate the strengths and limitations of quantitative size and shape descriptors for smokeless powder sample differentiation and brand identification. Digital images of smokeless powder samples were captured using a macro camera and processed using open-source image analysis software. For each powder particle ($n \approx 34{,}000$), eight shape and size parameters were measured. Using 80% of the data, linear discriminant analysis (LDA) was used to classify the 90 brands of smokeless powders. The remaining 20% of the data set was treated as unknowns and "matched" to the main data set samples using the shortest Mahalanobis distance. The algorithm correctly classified 83% of the unknown dataset. If the first two shortest distances are observed, the success rate increases to 97%. The results from this study are very encouraging but will require more samples to obtain a more robust assessment.

Many Thanks to Our Sponsors and Exhibitors!

Jan Burmeister **Millette Technical Consulting**

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