# INTER/MICRO 2019

### **An International Microscopy Conference**

June 10-14, 2019 • Chicago



Sponsored and hosted by

### **McCrone Research Institute**

2820 S. Michigan Avenue Chicago, IL 60616-3230 (312) 842-7100 www.mccroneinstitute.org



## Welcome to Inter/Micro 2019

This year marks the 71st anniversary of the Inter/Micro conference that 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 amateur and professional microscopists the opportunity to learn about new instrumentation, novel techniques, and modern applications of microscopy and microanalysis. Each year attendees can learn the practical and applied use of photon and electron microscopes, spectrometers, microprobes, digital and image enhancement and analysis systems, and other highly specialized microanalytical methods.

Inter/Micro presentations from the world's foremost microscopists 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 taking part in the intensive two-day workshop will learn specialized skills and gain hands-on experience working with leaders in their field of microscopy.

The Microscope, now in its 82nd year, is the official quarterly journal of this conference, and we highly encourage all Inter/Micro speakers to submit their work for publication. Submitted papers will be peer reviewed and published in the order in which they are received.

I would like to thank all of our past, present, and future attendees, workshop instructors, speakers, and participants — and hope you enjoy Inter/Micro 2019.

Gary J. Laughlin Chairman, Inter/Micro

Dany J. Laughli

Cover image by Martin Kocanda, Rapid City (South Dakota) Police Department This photomacrograph of a house fly in darkfield illumination was voted best overall winner in the Inter/Micro 2018 photomicrography competition, sponsored by pH2, LLC.

### Monday, June 10

## **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: Patrick Buzzini — Department of Forensic Science, Sam Houston State University

## Investigation into the Mechanism of Corrosion of a Pharmaceutical Glass Container

Richard S. Brown — MVA Scientific Consultants

# Artists' Materials Reference Collections: How to Create and Use Them, and Why They're Essential in Analysis

Joseph G. Barabe — Barabe & Associates LLC

### Pigment Packages of Modern Australian Tattoo Inks

Ethan Groves — Microtrace LLC

Morning Break

### Form Birefringence: Variable Birefringence?

Andrew A. "Tony" Havics — pH2, LLC

## **Evaluation of the Canon Rebel T7i for Photomicrography** Sebastian Sparenga — McCrone Research Institute

### The Classification of Raman Patterns of Inkjet Printer Inks: Comparing Visual Inspection and Different Variants of Linear Discriminant Analysis Methods

Patrick Buzzini — Department of Forensic Science, Sam Houston State University

12:00-2:00 p.m. Lunch, McCrone Garden

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

Chair: John R. Reffner — Dow Chemical Company

### **Force Feeding Physics**

Brian J. Ford — Cardiff University

### **Continuing Adventures in Fluorescence**

Charles Mazel — NIGHTSEA

#### **Unwanted Connections** — From Whiskers to Nanotubes

James R. Millette — Millette Technical Consulting

Afternoon Break

### FTIR Analytical Method for the Identification of Cellulose Fibers Anders Juul Lawaetz — Novo Nordisk Pharmatech, Køge, Denmark

## FTIR Microscopy Simultaneous with Visible Image Viewing Andy Bean — PIKE Technologies

# Improved Quantitative Assessment of Carbon Black Dispersion in Polyolefins by Light Microscopy

John R. Reffner — Dow Chemical Company

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 2019 is 5 p.m. Tuesday, June 11.

Awards for Best Overall Image, Most Unique Image, and Best SEM Image will be announced at the SMSI Awards Dinner, Wednesday, June 12. The competition is sponsored by pH2, LLC.

### Monday, June 10

# An Evening with Brian "Poo Bare"

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

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

Excrement is something we rarely discuss. Yet its production is vital for life, its composition of interest to microscopists, and this evening we discover how it has played a vital role



in human history. The ancient Chinese and ancient Egyptians thought excrement would provide a clue to immortality. Wars were waged over feces, which also became a valued item of commerce, and were even used to produce award-winning works of fine art. This extraordinary saga has never been told, and here we have a lecture that truly is complete crap, from start to finish.

**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 lecturer 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 11

## **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: John A. Reffner — John Jay College, CUNY

## Clouseau: "Does Your Dog Bite?" Asbestos Science in the Courtroom

Sean Fitzgerald — Scientific Analytical Institute

# Caught in the Act! Multispectral LANDSAT Imagery and SEM Identification of Potentially Toxic Fly Ash at Steam Power Plant Waste Disposal Sites

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

## Tremolite Asbestos from the "Saltworks" Mine, Inyo County, California

Eric J. Chatfield — Chatfield Technical Consulting Limited

Morning Break

## **Techniques for Amphibole Asbestos Determination in Sheet Silicates**

Sean Fitzgerald — Scientific Analytical Institute

### Three Char and Soot Fire Cases

Andrew A. "Tony" Havics — pH2, LLC

### Fiber Survey of Selected Postage Stamps

Walter J. Rantanen — SGS-IPS Testing

# The Ever-Expanding World of Microscopy, Imaging, and Microanalysis

John A. Reffner — John Jay College, CUNY

12:15-2:00 p.m. Lunch, McCrone Garden

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

Chair: Joseph Insana — Microtrace LLC

### "The Particle Was Identified as a Cellulose Fiber"

Skip Palenik — Microtrace LLC

## Conceptualization of Assessing the Evidential Value of Vehicular Transmission Fluids, Brake Fluids, and Lubricating Greases

Andra Lewis — Department of Forensic Science, Sam Houston State University

### Polymerography: Chemical Etching of Polymers

Andrew A. "Tony" Havics — pH2, LLC

Afternoon Break

### Nanoparticles as Trace Evidence

Kelly Brinsko Beckert — Microtrace LLC

### Plastics in a Refreshing New Light

Brian J. Ford — Cardiff University

## Benchtop Micromanipulator for Precise Sampling in the Forensic Sciences

Steven M. Barnett — Barnett Technical Services

### Locating and Analyzing Microtraces of Paint

Joseph Insana — Microtrace LLC

See Tuesday presentation abstracts on page 19.

### Tuesday, June 11

### **Exhibitor Booths**

9:00 a.m.-5:00 p.m. Tuesday, June 11 and Wednesday, June 12, McCrone Classroom

L earn about the latest microscopy innovations and products from the exhibitors, NIGHTSEA and EDAX, Inc.

### **SMSI Silent Auction**

12:00-5:00 p.m. Tuesday, June 11 9:00 a.m.-3:45 p.m. Wednesday, June 12, McCrone Classroom

**B**id 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 roof-



top 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 12

## **Chemical and Forensic 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: Katie M. White — Microtrace LLC

### Dating Fungi — A Non-Romantic Approach

Payam Fallah — IDEHL Laboratory

### Black Belt: The Art of Balance and Impact Concentration

Peter Diaczuk — John Jay College of Criminal Justice, Department of Sciences

## Sources of GSR Particles: One that Shouldn't and One that Wasn't

Martin Janssen — Netherlands Forensic Institute

Morning Break

## A Look at How Visual Aspects of Fiber Appearance Affect MSP Spectra

Meggan King — McCrone Research Institute

### An Update on the Effect of Ultraviolet Radiation on the Degradation of Dyed Fibers as a Function of Time Using UV-Vis Microspectrophotometry

Patrick Buzzini — Department of Forensic Science, Sam Houston State University

### Homemade Explosives: Not for Every Do-It-Yourselfer

William A. Randle — Missouri State Highway Patrol Crime Lab

### A Bone to Pick: Examination and Analysis of Bone Tissue

Katie M. White — Microtrace LLC

12:15-2:00 p.m. Lunch, McCrone Garden

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

Chair: Jack Hietpas — The Pennsylvania State University, Forensic Science Program

### The Mystery of Leeuwenhoek's Canoe

Brian J. Ford — Cardiff University

# Glass Bottle Thickness Variation: Measurements for Source and Comparison Determination

Brendan Nytes — Microtrace LLC

Andreas Sigismund Marggraf and the First-Time-In-History Use of a Microscope to Prove the Identity of a Chemical Substance Jan Burmeister

Afternoon Break

# How Low Can You Go? Determining the Smallest Steel Fragment for Quantitative Analysis

Peter D. Zoon — Netherlands Forensic Institute

# Medical Malpractice or BBQ Gone Wrong: Identification and Sourcing of a Metal Wire Removed from a Patient

Jason Beckert — Microtrace LLC

## Assessing the Utility of Smokeless Powder Micromorphometry for Brand Identification

Jack Hietpas — The Pennsylvania State University, Forensic Science Program

See Wednesday presentation abstracts on page 28.

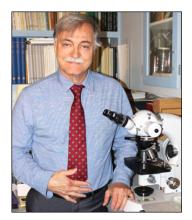
### Wednesday, June 12

## **State Microscopical Society of Illinois 2019 Awards Dinner and Live Auction**

### James Solliday • 2019 SMSI August Köhler Award Recipient

Presented at The Chicago Firehouse Restaurant, 1401 S. Michigan Avenue, 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 James Solliday with the 2019 August Köhler Award.

James Solliday is recognized for his numerous contributions to light microscopy and active participation in microscopical societies. He was a principal founder of the Microscopical Society of Southern California (MSSC) in 1996 and is currently the MSSC president, a chair he has held every year since 2002. He is also a long-time contributor to the Los Angeles Microscopical Society, and a member of the American Circuit of the Postal Microscopical Society, Quekett Microscopical Club, and the International Society for Diatom Research. Mr. Solliday has taught workshops on diatoms, illumination methods, staining, photomicrography, microscope maintenance, and the history and identification of antique microscopes. He has published dozens of microscopy articles, including contributions to the MSSC, San Francisco Microscopical Society, Los Angeles Microscopical Society, and The Quekett Journal of Microscopy. For 35 years, Mr. Solliday has operated his own business, Educational Photo Lab, which specializes in photomicrography and has produced photomicrographs for Hollywood movies, documentaries, litigation exhibits, contamination analysis, authors, Ph.D. students, and publishers, with more than 450 microscope images published in textbooks. He was instrumental in obtaining and donating surplus microscopes to area schools. Beyond the micro world, he served for 31 years as a professional firefighter and medical technician for the city of Costa Mesa, CA.

### Thursday–Friday, June 13–14

# **Workshop: Fungal Spore Identification** from the Air

9:00 a.m.-5:00 p.m., McCrone Classroom and Laboratory Taught by Payam Fallah, Ph.D.

This two-day basic workshop will introduce students to the science of mycology and fungal spore identification from the air. The three major phyla — ascomycetes, basidiomycetes, and



zygomycetes — will be discussed. Some mycological techniques will also be covered. Students will look at air samples taken from outdoors/indoors and learn how to identify spores at genus level. A brief discussion on basic fungal biology, ecology, and taxonomy will be presented. It is the goal of this workshop that participants be able to identify common allergy-causing fungal spore types from the air such as *Cladosporium*, *Alternaria*, ascospores, and basidiospores, to name a few.

Payam Fallah, Ph.D., has worked in the field of indoor air quality (IAQ) since 2000 and currently manages an IAQ lab, IDEHL Laboratory, in Redmond, WA. He earned his graduate degrees from the University of Illinois at Urbana-Champaign: a doctorate in mycology in 1998 and an M.S. in plant pathology in 1994, working with fungal pathogens and their dispersal in the air. He also has a B.S. in agronomy from the University of Delaware (1990). In 1999, Dr. Fallah was a post-doctoral fellow at the Systematic Botany and Mycology Laboratory, U.S. Department of Agriculture in Beltsville, MD. He has published more than 20 articles related to mycology, plant pathology, and indoor air quality, and has served as a mold expert in several legal cases.

#### PRESENTATION ABSTRACTS

### Monday, June 10

### **Techniques and Instrumentation**

### Investigation into the Mechanism of Corrosion of a Pharmaceutical Glass Container

Richard S. Brown and Jake Spry —MVA Scientific Consultants

A glass container with surface corrosion on its internal diameter (ID) was examined using a combination of differential interference contrast (DIC) microscopy, scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS), and analytical transmission electron microscopy coupled with EDS (ATEM-EDS). After mapping potential corrosion sites, the containers were cut with a diamond saw to expose the glass container's ID surface for additional direct examination by DIC and SEM-EDS. Selected corrosion pits were thin sectioned using a dual beam focused ion beam secondary electron microscope (FIB). The thin section was subsequently analyzed by analytical electron microscopy to analyze the corrosion surface in cross-section. Sub-surface modifications of the glass were apparent by imaging the thin-section by ATEM-EDS and extended well below the surface corrosion observed by DIC and SEM-EDS.

### Artists' Materials Reference Collections: How to Create and Use Them, and Why They Are Essential in Analysis

Joseph G. Barabe — Barabe & Associates LLC

The accurate identification of materials in a work of art or of historical importance may be vital in planning its treatment, or even in assessing its authenticity. The correct identification of such materials often depends on comparison with samples of known composition. The sample amounts necessary for a wide range of analyses are remarkably small, typically at the limits of human visibility for virtually all analytical methods such as PLM, SEM-EDS, FTIR, and others.

Mounted microscope slides are useful for polarized light microscopical analysis, and dry, unmounted material allows for the generation of reference spectra with instrumental analysis. While reference books and atlases are useful, even essential, they are no substitute for the comparison regarding size, shape, color, and optical characteristics of the actual material. The reference materials should meet these criteria: they must be typical, with the most common characteristics, however, outliers are also useful; be sufficient in quantity and quality; be uncontaminated whenever possible; and, if multiple forms are common, all should be included.

Reference samples can be acquired by gift, trade, or purchases. The author's collections include pigments, both traditional and modern, fibers, minerals, photographs, and printing process exemplars.

### Pigment Packages of Modern Australian Tattoo Inks

Ethan Groves — Microtrace LLC

Forty-nine tattoo inks, available in the Australian market, were analyzed to identify the range and types of pigments they contained. Raman microspectroscopy was used to identify the pigments in situ; however, microscopical examination of the raw inks proved pivotal for surveying the number of pigments in terms of the colors employed and for assessing their relative concentrations (major, minor, trace) within the ink. The combination of microscopical evaluation and confocal Raman microspectroscopy allowed for the identification of 89 pigments in the 49 samples analyzed. The resulting data reveals trends both in the pigments used within certain colors of tattoo inks at the time, as well as the suite of pigments used by the different manufacturers. Pigment identifications were also compared to the listed ingredients on 27 of the surveyed samples, revealing a positive correlation between listed and identified components for all but three of these samples.

### Form Birefringence: Variable Birefringence?

Andrew A. "Tony" Havics — pH2, LLC

ASTM E2228 defines birefringence as "the numerical difference in refractive indices for a fiber, given by the equation:  $n \parallel - n \perp$ ." For

crystals, it could be defined as the differences of refractive indices (RIs) of  $\varepsilon - \omega$  for uniaxial crystals or  $\gamma - \alpha$  for biaxial crystals. This is perhaps a simplification, as practical measurement of birefringence truly relates to the sum of four types of birefringence: intrinsic, form, strain, and circular. We will ignore circular birefringence for the time being and focus on linear birefringence. Most are aware of intrinsic birefringence and recognize it as being due to anisotropic periodicities in crystalline chain lattice that affect the velocity of linear polarized light. Many are also aware of strain birefringence, wherein stress modifies the polarizability of the molecules leading to a change of birefringence, typically from zero birefringence (isotropic) to some value of birefringence greater than zero. The concept of form birefringence has been limited in its description in texts and teaching materials for microscopy. Form birefringence could be described as the birefringence derived from a system of two periodically arranged components with anisometric forms with different RIs, where the size of the components must be small compared to the wavelength of light (< 500 nm). It comes in rodlet birefrigence and either layer or platelet birefringence. Form birefringence theory and practical examples help explain the phenomena and its observation in polarized light microscopy.

# **Evaluation of the Canon Rebel T7i for Photomicrography** Sebastian Sparenga — McCrone Research Institute

Photographic documentation is a must for any microscopy lab. There are numerous microscope-dedicated camera options currently on the market, but many of the higher performance models that are touted as being better suited for difficult-to-photograph techniques like fluorescence, come at a hefty cost. Plus, they only function on a microscope and cannot be used for any other regular photography needs that the laboratory may have. This talk will discuss a consumer DSLR option for all types of photomicrography, including brightfield, darkfield, fluorescence, and polarized light microscopy techniques.

### The Classification of Raman Patterns of Inkjet Printer Inks: Comparing Visual Inspection and Different Variants of Linear Discriminant Analysis Methods

Patrick Buzzini<sup>1</sup>, James Curran<sup>2</sup>, and Carrie Polston<sup>1</sup> — <sup>1</sup>Department of Forensic Science, Sam Houston State University; <sup>2</sup>Department of Statistics, University of Auckland, New Zealand

In the context of investigating counterfeit currency, a variety of information is gathered from seized specimens to develop investigative leads about printer source candidates. Raman microspectroscopy is proposed as a non-destructive and fast screening tool to identify unknown inkjet printers utilized to produce counterfeit banknotes. Inkjet printers generate microscopic dots that can be detected individually using a microscope coupled to the Raman spectrometer. In the present phase of this project, 231 Raman spectra were collected from the cyan, magenta, and yellow dot components of 11 inkjet printer ink samples using a near-infrared (NIR) laser wavelength at 785 nm. Spectra were first compared visually, and groupings were formed for each individual color and for the three colors considered jointly. Visual inspections of spectra are impractical and tedious for the intended investigative purpose; therefore, a sensible statistical classifier is sought. Three variants of linear discriminant analysis (LDA) were applied: 1) principal component analysis (PCA) followed by LDA, 2) partial least square discriminant analysis (PLSDA), and 3) "sparse" LDA. Although spectral comparisons by means of visual inspections are still superior to differentiate Raman spectra on the basis of minor peaks, "sparse" LDA provided the highest classification potential, i.e., highest accuracy.

This project was supported by Award No. NIJ-2016-DN-BX-0164, awarded by the National Institute of Justice, Office of Justice Programs, U.S. Department of Justice.

### **Force Feeding Physics**

Brian J. Ford — Cardiff University

The current fashion for physics has encouraged the view that functioning of living microorganisms can be reduced to elementary

constructs. However, many of the explanations under this Cartesian tradition are unduly simplistic, and their proponents fail to grasp the intricacies of living systems. In this presentation, we will contrast some current examples with the realities observed by microbiologists, proposing that greater effort should be taken to promulgate a more realistic interpretation of life under the microscope.

### Continuing Adventures in Fluorescence

Charles Mazel — NIGHTSEA

NIGHTSEA primarily develops equipment for viewing and documenting fluorescence, both off-the-shelf and custom, and in so doing we encounter diverse opportunities and challenges. Some of these do not even involve fluorescence. This talk will review some of our experiences in the past year: what we have learned, what we are working on, and even how Inter/Micro itself proved invaluable in one project.

### **Unwanted Connections** — From Whiskers to Nanotubes

James R. Millette — Millette Technical Consulting

Electrically conductive particles, especially those with elongated morphologies, are an important concern in areas housing electronic equipment such as data centers. A number of catastrophic computer system failures have been attributed to zinc whiskers reported from zinc-plated floor materials. Metal turnings and wear debris from carbon fiber products can also provide the opportunity for unwanted connections (short circuits) between electrical components. Most recently, ultra-microscopic "fiber" nanotubes have caused concern about unwanted electrical connections on the smallest scale. Microscopy is the best tool to find these very small electrical connectors among the dust particles that inhabit electronic information storage systems. Elemental analysis from scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS) is very useful for zinc and other elongated metal particles. Transmission electron microscopy coupled with EDS (TEM-EDS) is necessary for the investigation of nanotubes.

## FTIR Analytical Method for the Identification of Cellulose Fibers

Jonas Hoeg Thygesen and Anders Juul Lawaetz — Novo Nordisk Pharmatech, Køge, Denmark

Regulatory agencies call for the identification characterization of any intrinsic, inherent, or extrinsic particles present in pharmaceuticals. Among the many tools for particulate and foreign material identification, Fourier-transform infrared (FTIR) microscopy has developed into one of the industry-standard workhorses. The common approach during FTIR microscopy includes measurement of the unknown fiber and comparison of the spectrum with a set of known reference spectra. This comparison is commonly based on correlation between the unknown and reference spectra. However, in the case of cellulose fibers, this approach does not allow distinction between, e.g., paper and cotton. Hence, the identification may stop once a particle has been identified as cellulose, thereby limiting the root cause analysis. This issue has been addressed at Novo Nordisk Pharmatech. Employing multivariate statistics, we have developed a method that allows us to discriminate between different cellulose fibers and to classify them into one of four groups: cotton, viscose/rayon, paper, and other cellulose fibers. This presentation will describe the knowledge gained during the development work and show how tools such as multivariate data analysis can be used to gain more insight from data already gathered.

## **FTIR Microscopy Simultaneous with Visible Image Viewing** Andy Bean and Jenni Briggs — PIKE Technologies

The combination of FTIR spectroscopy and microscopical sampling has been a powerful tool for many years. Typically, the sample is identified in the visible, the microscope is switched to the infrared path, and the spectrum is collected. Although a complex design, an optical layout has been achieved that allows infrared light to return to the spectrometer simultaneous to the visible light being imaged onto a camera. Such a sampling accessory has been useful for identifying a field of view not only by the visible image but also

by its spectral fingerprint. Time resolved spectroscopy is presented to show the visible microscopical change in the sample in synchrony with its changing IR spectrum.

# Improved Quantitative Assessment of Carbon Black Dispersion in Polyolefins by Light Microscopy

John R. Reffner and Day-Chyuan Lee — Dow Chemical Company

Carbon black dispersion in polyolefin-based compounds is often assessed using visible light microscopy, and standard test methods such as ASTM D5596-03, ISO 18533, and ISO 11346 have long been established for the quantification. These methods are based on visual assessments of a very limited sample area. In this talk, I will discuss some limitations of these test methods and present a procedure using stage automation/image collection and image analysis. Stage automation allows a large number of images to be collected without operator bias. Image analysis provides quantitative information on both sample thickness and the size of undispersed carbon black domains. The combination provides a rapid and more statistically significant assessment with significantly more sensitivity to low levels of poor dispersion.

### Tuesday, June 11

## **Environmental and Industrial Microscopy**

## Clouseau: "Does Your Dog Bite?" Asbestos Science in the Courtroom

Sean Fitzgerald — Scientific Analytical Institute

In asbestos litigation, I am often reminded of the "3-dog defense," which goes like this:

- 1. My dog did not bite you, because I don't own a dog.
- 2. Okay, I do own a dog, but it is not the one that bit you.
- 3. Well, my dog may have bitten you, but I had no idea that he would!

Like the dog, the bite, and the onus, the definition of asbestos, the types that cause diseases, and the awareness of the health risk of asbestos in products by the manufacturer, present torturous labyrinths of doubt and shroud the ever-elusive facts necessary to convince juries or courts. In the sage words of Dr. James Millette, we are asking questions in the courtroom that should have been answered in the laboratory. He was talking about asbestos in consumer talcum powders as a cause of plural or even possibly peritoneal mesothelioma. Those who keep up with asbestos in the news know that talc was recently implicated in ovarian cancers. And now, the asbestos content of talc has been implicated, to the tune of billions and billions awarded to ovarian plaintiffs — enough to make Carl Sagan blush. But where is the science?

# Caught in the Act! Multispectral LANDSAT Imagery and SEM Identification of Potentially Toxic Fly Ash at Steam Power Plant Waste Disposal Sites

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

The Alabama Department of Environmental Management (ADEM) and the U.S. Environmental Protection Agency (EPA) strictly regulate disposal of industrially generated toxic waste and requires

its removal from sites where generation takes place. Permanent safe storage of such materials, similarly, is mandated and must be at locations deemed safe for projected long-term storage. One major source of such wastes are coal-fired, steam power plants used for generation of electricity. Historically, these have long been associated with power stations using coal, which produces a deleterious waste by-product in the form of fly ash. The cost of removing the large quantities of ash generated at power plants is sizeable, and to avoid this, ash has often been placed in on-site storage areas. Leaching of these waste sites by rainfall events has, in many cases, impacted adjacent water bodies that are used as sources of drinking water by nearby cities or towns and food sources derived from the resident aquatic fauna that are consumed by the local populace. As a consequence, most states now strictly prohibit on-site storage of fly ash.

Because of the large size of fly ash disposal sites, concealment is impossible, and they can be easily identified by their distinctive physical, mineralogical, and chemical properties. Even at an elevation of 700 miles above the Earth's surface, their distinctive spectral signal is apparent on photos taken by the orbiting LANDSAT system. For example, samples collected from the Barry Steam Plant located 30 miles north of Mobile, AL possess abundant alumino-silicate and iron oxide spherules that are universally identified with fusion reactions associated with the high temperature burning of coal. These are easily visible in SEM photos of the fly ash and their distinctive chemical composition is further confirmed by EDS analysis. The ash at the Barry Plant also possesses a marked heavy metal signature that has produced a "mercury anomaly" not only in the discharge canal associated with the power plant, but also in the adjacent wetland area. The EPA has posted signs warning that consumption of fish from this area is harmful to health and should be avoided. Problems associated with a number of environmental restrictions (ash storage, chemical and thermal discharge exceedances, etc.) at six of the power stations resulted in the assessment of \$1.25 million in fines against Alabama Power by the State in 2018. The company has acknowledged that planned conversion of their power plants to gas-fired systems will largely eliminate these problems.

## Tremolite Asbestos from the "Saltworks" Mine, Inyo County, California

Eric J. Chatfield — Chatfield Technical Consulting Limited

Five samples of tremolite asbestos were received over a 39-year period. The available evidence leads to a belief that all five of these tremolite asbestos samples originated from the same mine in Inyo County, CA. The morphological properties of these five samples have been compared by polarized light microscopy (PLM) and transmission electron microscopy (TEM).

Fiber width is known to be a particularly important factor that is associated with carcinogenic activity. However, at the magnification of approximately  $10,000\times$ , used for counting of fibers longer than 5  $\mu$ m, the 1 mm increments on the fluorescent screen of a TEM correspond to width increments of 0.1  $\mu$ m. For fiber widths lower than 0.5  $\mu$ m, 0.1  $\mu$ m increments of width do not provide sufficiently accurate measurements. Accordingly, a new fiber counting protocol has been implemented in this work, in which fiber widths below approximately 0.5  $\mu$ m are measured at a magnification of approximately 60,000×.

The fiber widths of two historical samples were found to be significantly thicker than those in samples collected more recently. The fiber size distributions of the three recently collected samples were found to be closely similar and with fiber widths close to those of the Korean tremolite asbestos used in the animal experiments reported in 1991 by Davis, Addison, McIntosh, Miller, and Niven.

## **Techniques for Amphibole Asbestos Determination in Sheet Silicates**

Sean Fitzgerald — Scientific Analytical Institute

Some of the toughest questions for the asbestos laboratory are whether amphibole asbestos is detectable in sheet silicates, like the phyllosilicates talc, serpentinite (e.g., chrysotile), and biotite (e.g., vermiculite as the industrial term for expanded hydrobiotite), or products manufactured with those minerals. As we are currently working to perfect the science of detecting and quantifying amphibole asbestos or their fibrous structures, threads are emerging that

are bringing together proven preparation and analytical techniques along with streamlined procedures. Together with more familiar air, dust, water, and bulk methods, this presentation will discuss more arcane techniques such as Addison-Davies reduction and Blount liquid separation and their effectiveness to accurately determine amphibole occurrence in these sheet silicate mineral resources.

Unfortunately, when entities like the Food and Drug Administration, U.S. Pharmacopeia, or Consumer Product Safety Commission ask us (the analytical thought leaders) how we can guarantee that a given mineral product can be dubbed "asbestos-free" and that no particles released into the air from use of those minerals could ever possibly be counted as asbestos consistent with airborne analytical protocols, we have no answer. Current asbestos-in-talc protocols and those in development will be compared, contrasted, and explained to illustrate what works and what does not.

#### Three Char and Soot Fire Cases

Andrew A. "Tony" Havics — pH2, LLC

Over the past two decades, restoration of fire- and smoke-impacted buildings has increased dramatically. In response to these impacts, inspection protocols and microscopical analysis methods have been devised by the Restoration Industry Association, in cooperation with the Indoor Environmental Standards Organization. The analysis can be supplemented by the ASTM method on soot, if so desired. Other techniques can also be used to help identify or supplement identification of sources of fire impact and the level of impact. A set of three fire cases involving the analysis of char and soot are used to illustrate the methods and techniques available in these cases. Two of these were from two-story residential properties and the third is from a multi-story commercial property.

### Fiber Survey of Selected Postage Stamps

Walter J. Rantanen — SGS-IPS Testing

Paper postage stamps have been used for well over a century. The fiber content of stamps has fluctuated depending on where they originated and as different fiber sources became available. This survey

will examine the fiber content from the early 1900s to the present day. Stamps for examination were selected from letters, postcards, and purchased sets, along with some from a prized collection.

# The Ever-Expanding World of Microscopy, Imaging, and Microanalysis

John A. Reffner — John Jay College, CUNY

Today, microscopy is being stimulated by the barrier of the diffraction limit of resolution being breached. The ability of scanning probes to reach nano-spatial resolution and the integration of microscopy with spectroscopy is a new technology. Confocal fluorescence microscopy and near-field optical scanning microscopy are capable of resolving molecular structure. Photo-thermal infrared spectroscopy has demonstrated sub-micrometer spatial resolution and high quality infrared absorbance spectra. Combining scanning probe with tunable laser technology or synchrotron radiation makes molecular vibrational spectra with nanometer scale spatial resolution possible.

The tentacles of microscopy extend to many disciplines and levels of complexity. The circus of life seen with a microscope in a drop of pond water has inspired many to pursue scientific careers. The hand lens gave us the means to explore the minutia of a fingerprint or discover the many minerals in rocks and sand. The pathologist's diagnoses of diseases, by studying the microstructure of tissue sections, play a vital role in public health. Metallurgists and material scientists use microscopy in many ways to improve materials or to determine why materials fail. Where will the new technologies take us? How will we become educated about these technologies and their application to real-world problems?

### "The Particle Was Identified as a Cellulose Fiber"

Skip Palenik — Microtrace LLC

In our laboratory, we are frequently asked to identify fibrous particles. New microscopists at Microtrace learn early in their careers that merely identifying an unknown particle as a cellulosic fiber (not a cellulose fiber) usually results in a gross under estimation of the information that such a particle can provide about itself, its history,

and its origin. This presentation will explain and demonstrate some of the characteristics that cellulosic particles can be coaxed into revealing about themselves by the well-prepared observer who has been trained to appreciate the telltale features and attributes, many of which are not obvious, even under the microscope, by someone trained to look for them and possessing that most important knowledge of what to look for.

# Conceptualization of Assessing the Evidential Value of Vehicular Transmission Fluids, Brake Fluids, and Lubricating Greases

Andra Lewis, Justin Day, and Patrick Buzzini — Department of Forensic Science, Sam Houston State University

Vehicular fluids bear the potential to be recovered at road accident scenes, crime scenes, or on objects belonging to individuals (e.g., garments). Questions of forensic interest pertaining to the identification of unknowns, sourcing, or the assessment of the degree of associations may need to be addressed in cases involving these materials. The current literature lacks of detailed guidance on the characterization and differentiation of substances such as transmission fluids, brake fluids, and lubricating greases.

An exploratory study of these types of fluids representative of the U.S. market was carried out using microscopical examinations, Fourier-transform infrared spectroscopy (FTIR), gas chromatography-mass spectrometry (GC-MS), and inductively coupled plasma-optical emission spectrometry (ICP-OES). The proposed conceptualization for the most suitable analytical approach focuses on the distinction between manufacturing and acquired characteristics detected using the adopted analytical methods. This study, which focuses on manufacturing features, shows complementarities between the various methods. For example, GC-MS proved promising to differentiate break fluids, while ICP-OES proved effective for differentiating transmission fluids. Preliminary microscopical examinations showed the presence of different microscopic particles dispersed in vehicular greases, but not in break fluids or transmission fluids. These particles differed considerably between different grease samples and offer a high potential for differentiation.

### Polymerography: Chemical Etching of Polymers

Andrew A. "Tony" Havics — pH2, LLC

There are many polymers for which etching techniques have been published. These include:

- polyolefins: polyethylene (LDPE, HDPE) and polypropylene (PPE)
- polycarbonates (PC)
- polylactide (PLA)
- natural rubber (NR)
- butadiene rubber (BR)
- nitrile rubber (NR)
- polyvinyls: polymethylmethacrylate (PMMA), polystyrene (PS), polyvinyl chloride (PVC), and polyacrylonitrile (PAN)
- styrene/acrylonitrile (SAN)
- acrylonitrile butadiene styrene (ABS)
- fluorocarbon polymers: polytetrafluoroethylene (PTFE), polyvinyl fluoride (PVF), and polyvinylidene fluoride (PVDF)
- polyethers
- polyesters: polyethylene terephthalate (PET)
- poly(aryl ether sulfone)s (PAESs)
- aliphatic polyamides (nylon)
- aromatic polyamides: aramids (Kevlar, Nomex)
- cellulose polymers (CELL)

This presentation will cover the theory behind polymer etching, followed by examples of what etching can reveal with light microscopy.

### Nanoparticles as Trace Evidence

Kelly Brinsko Beckert, Skip Palenik, and Christopher S. Palenik — Microtrace LLC

Sub-micron and nanoparticles comprise a new subcategory of trace evidence that is often easily overlooked and thus underutilized in forensic science. Despite the fact that these sub-microscopic particles are nearly ubiquitous in the environment and are found in a number of widely available and commonly used consumer products, from cosmetics to paint and food, they are rarely exploited in casework.

This may be due in part to a general lack of awareness regarding the existence of these particles, as well as the fact that methods for their detection, isolation, and analysis are rarely published in the context of forensic science. A protocol based on a published soil separation procedure has been developed, which allows forensic laboratories to utilize familiar equipment and instrumentation for the isolation and analysis of nanoparticles as trace evidence. Special attention is given to background contamination and its larger implications on the interpretation of results. This research demonstrates the efficacy of this technique and shows how nanoparticles or collections of nanoparticles may be used to help characterize soil, dust, or other unknown residues for identifications, comparisons, or the development of investigative leads.

A recent case study illustrates the value of such evidence, examples of some of the techniques that can be used to analyze them, the necessity for caution in interpretation, and the precautions that must be taken and considered in any such investigation.

### Plastics in a Refreshing New Light

Brian J. Ford — Cardiff University

The news is dominated by reports about the evils of plastic, and there is a rash of current books promoting the idea of a plastic-free future. Curiously, many plastics are biodegradable and can be metabolized by fungi, including *Aspergillus* and *Penicillium*, while expanded polystyrene has recently been shown to be a suitable foodstuff for insect larvae. Microbial polymers can offer us plastics for the future that are easily biodegradable, and even the massive drifts of plastic waste (like the Great Pacific Garbage Patch) could be usefully harnessed.

## Benchtop Micromanipulator for Precise Sampling in the Forensic Sciences

Steven M. Barnett — Barnett Technical Services

In criminalistics, as well as in a range of other fields, handheld sample manipulation is challenging because over time, particles,

fibers, and films of interest continue to decrease in size. A benchtop micromanipulator provides a simple way to perform many of these sample manipulations. A range of examples will be shown, including:

- isolation of short fibers from an adhesive surface
- isolation of glass particles from clothing
- separation of individual film layers from a multilayer paint structure
- scraping of oxide films from a knife
- isolation of sperm samples from clothing

### Locating and Analyzing Microtraces of Paint

Joseph Insana, Ethan Groves, Christopher S. Palenik, and Skip Palenik — Microtrace LLC

Forensic paint comparisons are generally conducted on samples that are, while small relative to their source, still visible to the unaided eye and are thus located and analyzed without great difficulty. This presentation will demonstrate that a more detailed examination of possible surface transfers can capture materials (e.g., questioned samples) even when such traces are invisible to the unaided eye. While certain analytical details, such as layer sequence or a pure FTIR spectrum, may not be obtainable from such minute traces due to their size and condition, a detailed analysis of other sample characteristics are still possible and may still provide sufficient analytical data to arrive at a probative result.

This particular case study serves as an example of the application of this approach and the analytical methods that can be used in such paint transfer incidents that may involve particles of paint as small as 40  $\mu$ m in size. Using a combination of microanalytical techniques, all of which were performed on a single, sub-sample of the original minute particle, it was possible to characterize the particle and relate these points of comparison back to a potential source.

It is especially noteworthy that even though most of the original evidence of transfer had been lost by carelessness, it was still possible to prove the presence of a two-way transfer.

### Wednesday, June 12

## **Chemical and Forensic Microscopy**

### Dating Fungi — A Non-Romantic Approach

Payam Fallah — IDEHL Laboratory

As more and more fungi find their way into our living spaces and start growing, insurance companies and homeowners struggle to put a timeline together for a specific fungal/mold growth. Some insurance companies have specific policies related to loss due to fungal damages, for example, no coverage for loss greater than 2 weeks. This has put tremendous pressure on both sides of the argument. Fungi, like many other organisms, have life cycles. In fact, many fungi have two or more life cycles. Understanding the life cycles of certain common species will help us provide not an exact growth date for a given fungal growth but an approximate timeline that can help answer the approximate time taken for a species to grow on building materials. We will explore common species within the kingdom fungi that can shed light on this important and costly issue. Fungal biology and ecology will be discussed so we can begin understanding these beautiful organisms.

### Black Belt: The Art of Balance and Impact Concentration

Peter Diaczuk — John Jay College of Criminal Justice, Department of Sciences

The Remington Arms Company has introduced a new ammunition in its Golden Saber® line called Black Belt. Currently available in three calibers 9 mm Luger, 40 S&W, and 45 Auto, it boasts a novel bullet design that is supposed to remain intact and retain its weight upon expansion. To accomplish this impressive feat, the bulletsmiths at Remington have added a black belt of reinforcing metal on the bullet circumference creating an hourglass cross-sectional profile. This presentation examines the microscopy and metallurgy of the new three-part bullet construction and its performance in some commonly encountered substrates.

## Sources of GSR(-like) Particles: One That Shouldn't and One That Wasn't

Martin Janssen — Netherlands Forensic Institute

Gunshot residue (GSR) can play an important role in shooting-incident investigations. For GSR-evidence to be used properly in court, information about the potential sources of the observed particles is critical. In this presentation, two sources are discussed. First a procedure that was in place at the Netherlands Forensic Institute to minimize the transfer of GSR and GSR-like particles from one object to another is shown to be a potential source of GSR-like particles. Secondly, a case in which the defendant successfully claimed a coffee shop to be source of GSR-particles is discussed.

One widely used procedure to prevent GSR-contamination in the lab is to cover the lab bench with fresh and unused paper to create a "clean" surface for a piece of evidence. In this way, the lab benches are not in direct contact with the GSR containing pieces of evidence and potential transfer of particles from pieces of evidence is minimized. In addition to earlier work that reported on the presence of indicative particles on brown recycled paper, it was found that "clean" unused paper can contain a large amount of particles containing the elements Pb, Ba, Ca, Si, Al, and P with various peak intensities. According to standard operating procedures in line with the ASTM standard, such an elemental compositions should be classified as indicative for gunshot residue. The presence of these kinds of particles can therefore have large implications in GSR investigations. Due to the large number of particles present in the paper, the particles are being transferred in large amounts contaminating the evidence itself and thus resulting in potential false positives.

During a shooting in a confined space, at least three shots were fired with a revolver, and the victim was shot through a door. As the suspect denied any contact with a firearm, the presence of GSR on the pieces of evidence (his trousers and a sampling of his car) were of prime importance in this case. The case was brought to trial by the public prosecutor after GSR particles were detected on the samples from the car. During the trial, the defense attorney successfully questioned the source of the GSR particles found in the

car. The attorney argued that the GSR particles could also originate from a coffee shop as a colleague of the defendant used the car to visit one before the car was sampled. As a result, the court concluded that this activity could offer an alternative explanation for the source of the GSR particles and that they should be omitted as evidence. Following the appeal of the prosecutor, additional investigations were performed to study the prevalence and persistence of GSR particles in cars and coffee shops in order to determine the likelihood of this alternative source.

## A Look at How Visual Aspects of Fiber Appearance Affect MSP Spectra

Meggan King — McCrone Research Institute

Photomicrographs and microspectrophotometry (MSP) spectra have been collected from man-made fibers that have been exposed to natural and artificial sources of ultraviolet radiation. As part of investigating the effect of ultraviolet radiation on the MSP of dyed fibers, spectra are collected every eight weeks over a period of 80 weeks. Over time, the fibers have faded in color and some have become very physically degraded. This physical degradation, seen visually through the microscope, has had an effect on the MSP spectra. This talk will highlight these visual and spectral changes that have occurred on some fibers observed in as few as eight weeks of ultraviolet radiation exposure.

This project was supported by Award No. 2016-DN-BX-0145, awarded by the National Institute of Justice, Office of Justice Programs, U.S. Department of Justice.

# An Update on the Effect of Ultraviolet Radiation on the Degradation of Dyed Fibers as a Function of Time Using UV-Vis Microspectrophotometry

Patrick Buzzini — Department of Forensic Science, Sam Houston State University

Single fibers from a collection of 53 textile samples were analyzed using UV-Vis Microspectrophotometry (MSP) after being exposed

to outdoor sunlight and placed into a laboratory UV radiation box at intervals of 8 weeks. Differences between MSP spectra collected from fibers prior to exposure (T0) and after exposures of 8, 16, 24, 32, and 40 weeks (T8 through T40, respectively) are discussed. The sample set consists of 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). Spectral changes were typically observed following alteration modes previously defined as decreases of band intensities, formation of new bands, or band shifts in wave number values. The complementarities of the two visualization and data dimension reduction techniques of Principal Component Analysis (PCA) and t-Stochastic Node Embedding (t-SNE) proved useful for identifying overlapping spectral curves and clusters. The Hotelling T<sup>2</sup> hypothesis tests for mean differences of two multidimensional groups were carried out using the principal components and the two t-SNE dimensions.

This project was supported by Award No. 2016-DN-BX-0145, awarded by the National Institute of Justice, Office of Justice Programs, U.S. Department of Justice.

### Homemade Explosives: Not for Every Do-It-Yourselfer

William A. Randle — Missouri State Highway Patrol Crime Lab

Explosives cases submitted to the crime laboratory consist of many different manufactured explosive mixtures, as well as homemade explosive mixtures. However, not all homemade mixtures intended to be explosive are explosive. A review of a few cases from the Missouri State Highway Patrol Crime Lab highlight some homemade "explosive" mixtures that were real duds.

### A Bone to Pick: Examination and Analysis of Bone Tissue

Katie M. White and Skip Palenik — Microtrace LLC

When a cadaver decomposes, bones are often all that remains from the body. They persist due to their hardness and durability, a result of their composite nature. When found as whole bones, their morphology can be very distinctive to pathologists or anthropologists. However, in our work as microanalysts, we often encounter them as fragments of an unknown material to be identified.

The cellular structure of bone results in a unique morphology. Microscopical examination of bone fragments permits recognition of these diagnostic structures. In addition to morphology, microchemical and microprobe analyses can provide information about the composition of suspected bone particles. The characteristics that are available for study may vary depending on the source and condition of the fragments themselves.

In addition to their significance in criminal forensic casework, identification of bone often plays an important role in other types of investigations. Bone is present in animal feeds and cremated remains and may be a component of decorative or historical artifacts. In food manufacturing, bone fragments may also appear in processed meat products, sometimes occurring as an undesirable by-product.

This presentation will demonstrate the properties and microscopical features that are most valuable for the identification of bone tissue in the laboratory, and the preparation methods used in these analyses. Case examples involving bone fragments will also be presented.

### The Mystery of Leeuwenhoek's Canoe

Brian J. Ford — Cardiff University

Recent publications in London have shone an unexpected light on the work of the pioneering microscopist Antony van Leeuwenhoek. It seems that he produced wooden paddles; while the unendingly fascinating investigation of his original microscopical specimens, discovered after more than three centuries by the speaker, has now been recast in a very different light. Illustrated with the latest video reports, this presentation will attempt to rectify some current misapprehensions.

# Glass Bottle Thickness Variation: Measurements for Source and Comparison Determination

Brendan Nytes, Ethan Groves, Rachel Rutter, Skip Palenik, and Christopher S. Palenik — Microtrace LLC

Forensic glass analyses are typically conducted as comparisons; however, the particles can also be analyzed for the purposes of placing constraints on a possible source, e.g., did a glass particle originate from a container or from a particular type of container? Regardless of the source, thickness (of full thickness glass fragments) represents a useful property for comparison.

For sheet glass, a goal of production is consistency in thickness. For container glass, thickness is a more complicated variable that is more difficult to exploit. Consider, for example, a wine bottle. A causal observer would note that the neck is made of thinner glass than the upper rim (where the cork is inserted). The variation exists in most container glass. In addition, it is anticipated that the permitted tolerances in glass variation, at a given location on a particular container, are larger than in sheet glass. Knowledge of the variation in container glass for a particular class of container, e.g., wine bottle, beer bottle, etc., has benefit for both sourcing and comparison. For sourcing purposes, the benefit is in knowing if a particular fragment is consistent with a particular type of container. For comparison purposes, knowledge of the variation in the thickness of a given source is important when attempting to compare a questioned fragment to a potential source.

Because the thickness of questioned fragments can be measured relatively easily by a variety of methods, e.g., calibers, under a microscope, using automated image analysis, the evaluation of thickness ranges in a container by this approach would be impractical. To that end, a novel (to forensic science) method of thickness measurement using an ultrasonic probe has been evaluated. This research provides the results of our method validation. The method was then used to produce detailed thickness maps of several different container types. These maps were used to determine the thickest and thinnest parts of each container type.

# Andreas Sigismund Marggraf and the First-Time-In-History Use of a Microscope to Prove the Identity of a Chemical Substance Jan Burmeister

The life of Andreas Sigismund Marggraf, an 18th century Berlin chemist, will be portrayed in light of his most notable and economically important discovery (published in 1749), that *Beta vulgaris* contains sugar, which is identical to "ordinary" sugar from sugar cane of tropical origin. The use of the microscope to prove the identity of the two compounds will be outlined as the first time in history that such an attempt was successfully made.

# How Low Can You Go? Determining the Smallest Steel Fragment for Quantitative Analysis

Peter D. Zoon — Netherlands Forensic Institute

At the Netherlands Forensic Institute (NFI) microtrace analysis of metal fragments embedded in bone is a routine part of examinations. These examinations are typically performed with scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS) analysis to determine the elemental composition of the fragments. Most of the encountered traces are stainless steel fragments. As previously discussed, obtaining reliable quantitative elemental compositions with SEM-EDS is not trivial under best of circumstances. To increase the evidentiary value of SEM-EDS analyses of small metal fragments embedded in bone, the quantitative elemental composition of small metal fragments was determined with laser ablation inductively coupled mass spectrometry (LA-ICPMS).

Small samples cut from knife blades showed promising results. These samples are however an order of magnitude larger than the traces typically encountered in bone. A method for generating small fragments without contamination was devised, and the small fragments were analyzed and the obtained compositions have been compared to the composition of the blades.

The smallest fragments that could be reliably analyzed are approximately  $50 \times 50$  µm in diameter. The thickness of the fragments

was not determined, but it seems reasonable that this should be approximately the same. This lower limit is not governed by the analytical measurement technique, but rather by the nature of the samples themselves.

To conclude this presentation, a case example will be presented in which the above-mentioned analysis was used to determine if metal fragment in the skull bone of a victim could originate from a knife handle.

# Medical Malpractice or BBQ Gone Wrong: Identification and Sourcing of a Metal Wire Removed from a Patient

Jason Beckert — Microtrace LLC

This presentation will focus on a case study in which a woman complaining of abdominal pain had an unknown foreign object removed from her abdomen. Believing this object was mistakenly left inside of her during a previous medical procedure, she sued the attending doctors and hospital for medical malpractice. Before the trial, the defense submitted the unknown material to our laboratory, where it was subsequently identified as a steel wire. When this wire did not correspond with any of the materials used during her surgery, a wire bristle from a BBQ grill cleaning brush was proposed as an alternative source. To test this hypothesis, a small-scale research project was conducted in which numerous commercially available grill brush bristles were analyzed and compared to the foreign object. This presentation will conclude with discussions of those results and the prevalence of injuries resulting from the inadvertent ingestion of BBQ grill brush bristles.

## Assessing the Utility of Smokeless Powder Micromorphometry for Brand Identification

Jack Hietpas<sup>1</sup>, Samantha Deibel<sup>1</sup>, Devin Kress<sup>1</sup>, Casey Brown<sup>2</sup>, and Wayne Moorehead<sup>3</sup> — <sup>1</sup>The Pennsylvania State University, Forensic Science Program; <sup>2</sup>MVA Scientific Consultants; and <sup>3</sup>forensicTRACE

Smalls arms propellants (SAP) are readily accessible and costeffective materials that firearms enthusiasts can acquire for the legitimate assembly of ammunition. Unfortunately the ease of procurement and the low cost of these materials is advantageous for their utilization in the construction of improvised explosive devices (IEDs). Typically, the SAP charge is loaded into a metal pipe (commonly steel) and sealed with screw-fit end caps. These devices are termed "pipe bombs" and are the most common IEDs in the U.S. Two recent high profile domestic terrorist attacks using IEDs (Boston Marathon bombing and NY/NJ attempted bombing) further demonstrate their continued usage. This presentation will provide an update on the utility of high-throughput automated image analysis of SAPs for potential brand identification and sample discrimination. Here we investigate three-dimensional micromorphometry of SAP by coupling data from planar images and thickness measurements made on manual cross-sections for flattened ball and flake-type powders. The results of this study show that samples are primarily using size-dependent parameters, with differentiated parameters providing limited separation. In addition, granule crosssections provided additional discriminatory information for flake and ball powders.

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