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Department of Archaeology, Boston University

Center for Nanoscale Systems, Harvard University

FTIR Spectroscopy for the Study of Material Culture

Saturday May 6, 2017	Center for Nanoscale Systems, Harvard University Laboratory for Integrated Science and Engineering (LISE), Room 303 11 Oxford St. Cambridge, MA 02138
Sunday May 7, 2017	Department of Archaeology, Boston University 675 Commonwealth Ave., Room 251 Boston, MA 02215

Register online at: goo.gl/iRLr9s

Organizing committee:

Ilaria Patania
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Matthew Chastain
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Saturday, May 6
Harvard University Center for Nanoscale Systems

9:00am	Welcome	
9:15	Arthur McClelland (*Harvard*)	Introduction to FTIR Spectroscopy
10:15	Coffee break	
10:30	Matthew Chastain (*MIT*)	Estimating Ceramic Firing Temperature Using FTIR: Bronze-Casting Molds from Ancient China
10:50	Arthur McClelland (*Harvard*)	Using Specular Reflection FTIR to Analyze Surface Coatings on Historic Photographs
11:10	Daniel Fallu (*Boston University*)	Soil Formation, Taphonomy, and Pastoral Models at Iron Age Nichoria, Greece
11:30	Ilaria Patania (*Boston University*)	Palaeolithic Pyrotechnology: Understanding Firing Temperatures and Cooking Practices at Yuchanyan Cave Site, China
12:00pm	Lunch (*sponsored by Thermo Fisher*)	
1:00	Tour the Center for Nanoscale Systems	
1:45	Lab activity session 1	
3:45	Lab activity session 2	
5:45	Drinks on the patio	

Sunday, May 7
Boston University Department of Archaeology

10:00am	Xiao Ma (*National Gallery of Art*)	The Application of FTIR on Ancient Building Materials: A Case Study on Multi-Layered Wall Earthen Plasters from Yuzhen Palace of Ancient Building Complex of Wudang Mountains in China
10:20	Richard Newman (*Museum of Fine Arts, Boston*)	Mopa Mopa: An Unusual South American Resin Used by the Inka
10:40	Coffee break	
11:00	Georgina Rayner (*Harvard Art Museums*)	What am I? Using FTIR in the Art Museum to Solve the Puzzle
11:20	David Schiering (*Czitek*)	The Application of a New FTIR Microspectroscopy Accessory for Far-IR Analysis of Artists' Pigments
12:00pm	Lunch (*sponsored by Agilent*)	
1:00	Agilent Technologies presentation	
1:45	Thermo Fisher Scientific presentation	
2:30	Coffee break	
3:00	Vendor hands-on demonstrations	
5:00	Closing	

Laboratory activities (Saturday, May 6)

(A)

Building a Specular Reflection Library and Using it to Identify Coatings on Historic Photographs

with Arthur McClelland

(B)

Firing Temperature Estimation: FTIR Bench Analysis of Powdered Ceramic Samples in KBr Pellets

with Matthew Chastain

(C)

µFTIR Analysis of Clays and Bone to Understand Palaeolithic Pyrotechnology

with Ilaria Patania

Each lab activity will be offered twice, once at 1:45pm and again at 3:45pm. Workshop attendees may take part in up to two activities.

Space may be limited. Anyone not participating in a lab activity during either session is encouraged to visit one of the nearby museums on Harvard's campus

Vendor demonstrations (Sunday, May 7)

Agilent Technologies, Inc.

Instruments:	**4300**	handheld FTIR with interchangeable sampling interfaces (diamond and Ge ATR, diffuse reflectance, external reflectance, specular reflectance)
	Cary 620	Focal Plane Array (FPA) chemical imaging FTIR microscope

Representative: Dr. Yanqia Wang, *Application Engineer, Molecular Spectroscopy*

Dr. Yanqia Wang received his Ph.D. in analytical chemistry from Duke University in Durham, North Carolina. His research work involved the method development of dynamic infrared spectroscopy to combine dynamic mechanical analysis with FTIR spectroscopy. Extensive working experiences on vibrational spectroscopy including MIR, Near-IR, Raman Spectroscopy and IR microscopy. He joined Agilent in 2013 as an application engineer to help application development and pre- and post-sale support on FTIR mobile and microscopic products.

Thermo Fisher Scientific

Instruments:	**Nicolet iS5**	field portable FTIR spectrometer configurable for microsampling, bulk-sampling ATR, and diffuse reflectance
	Gemini	handheld combination FTIR (diamond ATR) and Raman spectrometer
	iXR	field portable Raman spectrometer with multiple excitation lasers

Representative: Dr. Ron Rubinovitz, *Senior Applications Scientist*

Ron Rubinovitz is a senior applications scientist at Thermo Fisher Scientific. He is experienced with a number of optical spectroscopic methods and currently his chief areas of interest are FT-IR, and IR microscopy. Other fields of activity are FT-Raman, Near-IR and chemometrics. He earned his Ph.D. in Physical Chemistry at the University of Pennsylvania and has gone on to work with a variety of qualitative as well as quantitative vibrational spectroscopic applications related to the pharmaceutical, industrial and food industries.

Workshop talks: abstracts and speaker biographies

Matthew Chastain
MIT

Estimating Ceramic Firing Temperature Using FTIR: Bronze-Casting Molds from Ancient China

The elaborate forms, precisely rendered details, and large sizes of cast bronze ritual vessels from China's Shang and Zhou periods (c. 1600 – 221 BCE) make these objects a triumph of both art and technology. Their production required a mastery of ceramic materials in the bronze foundry. In particular, casting molds were formed from an unusual ceramic paste that was soft, porous, silica-rich, and altogether different from pottery clays.

In order to better understand the nature of this special material, a collection of casting molds excavated at three foundry sites (c. 1100 – 771 BCE) in the Zhouyuan area of Shaanxi province were analyzed with FTIR. Results were compared with FTIR spectra of lab-fired soil samples. The firing temperatures of the archaeological samples were estimated based on changes in clay Si-O absorption peaks resulting from structural alteration of the clays during firing. It was found that the casting molds had been subjected to low-temperature firing, in the range of 400 to 700 ºC. Comparison among the three foundry sites indicates distinct strategies for material processing and firing.

Matthew Chastain is a PhD candidate in the Archaeological Materials program at MIT's Department of Materials Science and Engineering. His dissertation research seeks to understand an unusual ceramic material used to make the casting molds that were critical to ancient China's extraordinary bronze-making tradition. Previous projects have involved early Chinese pottery, Native American copper working, and historic French paints. Matthew first learned to use FTIR in the science laboratory at the Art Institute of Chicago, where he was a summer intern while completing a BS in engineering at Northwestern University. He has been a visiting researcher at the Chinese University of Hong Kong and at Peking University in Beijing.

Daniel Fallu
Boston University

Soil Formation, Taphonomy, and Pastoral Models at Iron Age Nichoria, Greece

The assumed increase of cattle in Dark Age Nichoria has been a key piece of evidence for the "cattle-ranching" model of the Dark Age Greek economy. New zooarchaeological analysis, however, demonstrates a distribution of skeletal elements which may have resulted from preservation bias, rather than an over-reliance on cattle. Geoarchaeological analysis of "archival" soils retrieved from uncleaned bones provides some confirmation: the abundance of cattle bones at Nichoria is likely the result of taphonomic, rather than economic, processes.

Initial micromorphology revealed that the Dark Age assemblages likely represent a Bt soil horizon, while the Bronze Age remains appear to come from a calcareous C horizon. FTIR analysis confirms that $CaCO_3$, in particular calcite, is largely absent in the Dark Age soils. Further comparison of spectra shows that apatite, likely resulting from the degradation of bone, is present within the soil matrix. This matrix apatite is present in similar proportions in both the Bronze Age and Dark Age soils, suggesting some uniformity in process. While yet inconclusive, these results call into question the validity of a long-held economic model for the so-called "Dark Ages," and show the potential for geoarchaeological research even on long-excavated materials.

Daniel Fallu is a PhD Candidate at Boston University. His research in Greece focuses on micromorphology and site formation at sites from multiple periods with a focus on the end of the Bronze Age. His dissertation, entitled "Bronze Age Landscape Degradation in the Northern Argolid," investigates the changes in the landscape surrounding the citadels of Tiryns and Mycenae at the end of the Bronze Age and their potential impact on settlement. Daniel was recently awarded a Research Associateship at the Malcolm H. Wiener Laboratory for Archaeological Science of the American School of Classical Studies at Athens, for his project "Geoarchaeology of Resilience at Lechaio," focusing on the impact of tsunamis on the late antique Harbor Town of Lechaio near Corinth, Greece.

Xiao Ma
National Gallery of Art

The Application of FTIR on Ancient Building Materials: A Case Study on Multi-Layered Wall Earthen Plasters from Yuzhen Palace of Ancient Building Complex of Wudang Mountains in China

Vibrational spectroscopies such as FTIR have been commonly used in the analysis of ancient building materials. A case study will be performed on the multi-layered wall earthen plasters from Yuzhen Palace of Ancient Building Complex of Wudang Mountains in China, a UNESCO World Heritage Site. The characterizations of the raw materials is of primary importance for understanding the ancient raw materials and building technology, as well as providing guidance for future maintenance and conservation of the buildings. The FTIR analysis, in combination with some common characterization techniques was performed on several different layers of the plasters and fiber additives. The analytical results, advantages and limits of using the FTIR technique in this case study will be discussed.

Xiao Ma obtained his B.S. from Huazhong University of Science and Technology and Master of Science in materials engineering from Purdue University. He then joined Prof. Ioanna Kakoulli's group in the Department of Materials Science and Engineering at UCLA and obtained his Ph.D in January 2017. His doctoral research focused on the in-situ synthesis and characterizations of hydroxyapatite-based consolidant for calcium-rich matrices (sculptures, wall paintings, mortars, etc.) and archaeometric studies of ancient materials including mortars/plasters and Asian lacquers. In 2015-2016, he worked in Museum Conservation Institute of Smithsonian Institution and the science department of Getty Conservation Institute as a graduate intern. Currently, he is Charles E. Culpeper Fellow working with Dr. Barbara Berrie in the science department of the conservation division at the National Gallery of Art. He has co-authored over 10 articles and is recipient of several awards including Ralph C. Altman Award, R. E. Taylor Best Poster Award, Martin J. Aitken Best Poster Award, UCLA Dissertation Year Fellowship, etc.

Arthur McClelland
Harvard University

Using Specular Reflection FTIR to Analyze Surface Coatings on Historic Photographs

Characterization of photographic coatings is important for dating and identifying photographs, contributing new scholarship to our understanding of the history of image reproduction technology, and in the making of preservation decisions. Specular reflection FTIR can be used as a non-contact, non-sampling chemical analysis technique.

William Henry Fox Talbot introduced the salted paper print technique in 1839. The Weissman Preservation Center at Harvard Library has undertaken a multiyear project to enhance the understanding of salted paper prints and to ensure their long-term preservation. The majority of the estimated 8,000 salted paper photographs at the Harvard University Archives reside in 75 historic class albums dating from 1852 to 1864. The Harvard class albums containing the salted paper prints were produced annually over a period of 13 years by three prominent Boston photographers: John Adams Whipple, James Wallace Black, and George Kendall Warren. These class albums provide a glimpse into the technical evolution of the salt print process as practiced by some of Boston's pioneering photographers. A specular reflection FTIR spectral reference library was created for this project using modern samples of the possible salted paper print coatings. This new spectral reference library and the specular reflection FTIR technique allowed for the positive identification of the coatings.

Dr. Arthur McClelland is a principal scientist at the Center for Nanoscale Systems at Harvard University, where he manages the optical spectroscopy and optical microscopy instruments. He joined the technical staff at CNS in 2011. His main expertise lies in optical techniques for materials characterization. Arthur earned his BS from the University of Pittsburgh in engineering physics in 2003, his MS in electrical engineering from the University of Michigan in 2006, and his PhD in applied physics from the University of Michigan in 2009 under Dr. Zhan Chen. Prior to joining CNS, Arthur did a biophysics postdoc in the lab of Dr. Paul Champion at Northeastern University.

Richard Newman
Museum of Fine Arts, Boston

Mopa Mopa: An Unusual South American Resin Used by the Inka

Mopa mopa is the common name for an unusual resin from trees of the genus *Elaeagia* native to the Western Andean region of South America. The resin was used in two different regions. In southern Colombia, it was known well before the colonial period, but was repurposed for the elaborate decoration of small wooden objects by the Spanish; the resin continues to be used in an active craft industry in Pasto, Colombia. Further south, the Inka utilized mopa mopa for decorating ceremonial wooden drinking vessels known as qeros. The resin was apparently first used for this purpose around the beginning of the colonial period. This presentation will discuss the important role of FTIR microspectroscopy in identifying pigments and characterizing the binder in mopa mopa decorations on qeros. In an attempt to identify the species of *Elaeagia* from which the Inka obtained their resin, chemometric analysis on samples from Inka artifacts was carried out utilizing a database developed from botanical samples.

Richard Newman is Head of Scientific Research at the Museum of Fine Arts in Boston, where he has been a research scientist since 1986. His wide-ranging research on the materials and techniques of artists has included studies of Hindu and Buddhist stone sculpture of India, paintings of Diego Velazquez, and Japanese colored woodblock prints of the Edo era.

Ilaria Patania
Boston University

Palaeolithic Pyrotechnology: Understanding Firing Temperatures and Cooking Practices at Yuchanyan Cave Site, China

The cave site of Yuchanyan is known alongside Xianrendong for having produced the earliest pottery sherds yet discovered, respectively 18,600 cal BP and 20,000 cal BP. Through micromorphology, I identified clay-lined fire features and ash lenses at Yuchanyan, revealing technological behaviour concerning pyrotechnology and the manipulation of clays in the Chinese Upper Palaeolithic. Using µFTIR directly on the micromorphological slides, I was able to extrapolate heating temperatures of clays and bones in the sediments. Here I discuss pyrotechnology at the site and reflect upon use and firing techniques of the earliest pottery. This is the first step to more comprehensive investigations of production techniques, cooking practices, and and human behavior related the earliest pottery.

Ilaria Patania is a PhD candidate at the Boston University Department of Archaeology. Her current research is focused on the application of geoarchaeological techniques at Xianrendong and Yuchanyan, the two late Pleistocene karst caves in South China where the earliest known pottery has been found. Her investigation is centered on the micromorphological observation of sediments to reconstruct site formation processes connected to both human activities and natural inputs. To better understand anthropogenic use of fire at XRD and YCY, she is integrating micromorphological observations with µFTIR analysis directly on the slides to quantify heat treatment of bones and sediments. Ilaria is also collaborating as micromorphologist in the excavations at the Cane Notch Site, a Protohistoric Town on the Nolichucky River in Upper East Tennessee, and at Mortar Creek Rock Shelter, a multilayered Palaeoindian site in the Upper Cumberland Plateau of Tennessee.

Georgina Rayner
Harvard Art Museums

What am I? Using FTIR in the Art Museum to Solve the Puzzle

Fourier-transform infrared spectroscopy (FTIR) is one of the most important tools used in the Analytical Lab in the Straus Center for Conservation and Technical Studies at the Harvard Art Museums. It is generally the first point of call for the analysis of any microscopic sample, often smaller than a grain of sand, taken from an object. More often than not, little information is known about the composition of a work of art. FTIR is a simple and efficient method that allows us to begin to identify the types of material present which can include polymers, natural resins, drying oils, gums, waxes, proteins as well as organic (carbon-containing) and inorganic (metal-containing) pigments. This information is vital in helping to determine the next steps for analysis, treatment, and preservation.

This presentation will discuss some of the work being done by scientists at the Harvard Art Museums using FTIR in an ongoing survey to identify plastics in the collection.

Plastics were adopted by artists as soon as they became available. As a result, many museum collections contain a surprising amount of plastic, often in varying states of decay due to the inherent instability of many plastics. Plastics are often mis-identified or mis-labelled, sometimes referred to in passing as mixed media. This presents a large problem in our ability to care for the collection. The survey being undertaken at the Harvard Art Museums was designed to improve our records and our understanding of plastic objects in the collection so we can ensure they are receiving the best care possible. This will in turn increase their life expectancy. FTIR has played a vital role in the survey, which has thus far included some 250 samples. FTIR has allowed for quick identification of the most common plastics and additives present in the samples, helping to refine the appropriate method for further analysis

Georgina Rayner is the Andrew W. Mellon Postdoctoral Fellow in Conservation Science at the Straus Center for Conservation and Technical Studies at Harvard Art Museums. Georgina received her PhD in Chemistry, with a focus on polymer chemistry, from the University of Warwick (UK) in 2012. Before joining the Straus Center at the start of 2013, Georgina worked as a research technician within the Chemistry Department at the University of Warwick and spent time volunteering in the Conservation Science departments at both the Victoria and Albert Museum and Tate Britain.

Dave Schiering
Czitek

The Application of a New FTIR Microspectroscopy Accessory for Far-IR Analysis of Artists' Pigments *

** co-authors: Ron Rubinovitz (Thermo Fisher Scientific), Anthony W. Didomenico (Thermo Fisher Scientific), Beth Price (Philadelphia Museum of Art), and Kate Duffy (Philadelphia Museum of Art)*

The analysis of artists' materials by infrared (IR) spectroscopy is well-known. Analyses are broadly performed in the mid-IR region of 4000-400 cm^{-1}. The 400 cm^{-1} low frequency cutoff is due to KBr optics typically employed in the Fourier transform infrared (FT-IR) spectrometer. The mercury-cadmium-telluride (MCT) detectors employed in FT-IR microscopes further limit the spectral low frequency cutoff to 700-500 cm^{-1}. Many inorganic materials, such as pigments found in artworks, have diagnostic spectral absorption bands in the far-IR, while others yield bands exclusively in that region. Opening the far-IR to pigment identification in microscopic samples should provide a wealth of new chemical information for museum scientists and conservators, as well as academics. Advances in optical materials and design have opened the far-IR region, typically defined as 600-10 cm^{-1}, for application in materials characterization. This presentation will concern the use of a new FT-IR microspectroscopy accessory in the far-IR analysis of artists' pigments. This accessory allows the collection of spectra in transmission, reflection, and attenuated total reflection (ATR) modes and interfaces to the instrument-mounted detector. There are no optics in the accessory that limit the spectral range. The utility of this new optical accessory will be demonstrated in the far-IR analysis of artists' pigments. Reference spectra of pigments will be measured in the far-IR and these spectra will used to identify pigments in artworks. Data collected in transmission and ATR modes will be compared and discussed.

Dr. David W. Schiering is a founder and principal of Czitek, a small company dedicated to the development and marketing of vibrational spectroscopy products. He has more than thirty (30) years of experience in the business of instrumentation for chemical measurements. Prior to Czitek, Dr. Schiering has held numerous roles in management, science and technology, product development, and product management at Smiths Detection, SensIR Technologies, Thermo Electron Corp., and Perkin Elmer. Dr. Schiering, who has authored more than 25 publications on various aspects of vibrational spectroscopy, holds a PhD in analytical chemistry from Miami University, where he is also an adjunct Assistant Professor of Chemistry. Dr. Schiering has served the Coblentz Society as a member of the Board of Managers and as secretary from 1991 to 2010. In 2011, Dr. Schiering was made an Honorary Member of the Coblentz Society.