THIRD INTERNATIONAL INFRARED USERS' GROUP MEETING

IRUG3

Program and Abstracts

Winterthur Museum, Garden and Library Winterthur, Delaware 19735 May 28,29,30,1998 IRUG 3 at the Winterthur Museum, Garden and Library May 28 - 30, 1998

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PROGRAM

THIRD INTERNATIONAL INFRARED USERS' GROUP MEETING

Winterthur Museum, Garden and Library Winterthur, Delaware May 28 - 30, 1998

<u>THURSDAY – MAY 29, 1998</u>

- 7:45 am Bus departs the Wilmington Hilton Hotel
- 8:00-8:30 Registration in the Winterthur Pavilion
- 8:30 8:45 Welcome in the Winterthur Rotunda: **Dwight P. Lanmon**, Director, Winterthur Museum

Session I Moderator: Janice Carlson, Winterthur Museum

8:45 - 9:30	Data Processing Pitfalls: Possible Problems with Spectral Data Processing and Sample Preparation	Jim de Haseth , Univ. of Georgia
9:30 - 10:15	Infrared Spectral Search Algorithms	Mike Fuller, Nicolet
10:15 - 10:45	Coffee Break	
10:45 -11:10	Infrared Analysis at the Canadian Conservation Institute: Infrared Spectroscopy, Microspectroscopy and Non-destructive Reflectance Spectroscopy	Kate Helwig, CCI
11:10-11:55	Non-Destructive Mid-IR Fiber Optic Reflectance Spectroscopy Applied to the Study of Paintings	Marcello Picollo , Institute di Ricerca Sulle Onde Elettromagnetiche "Nello Carrara"
11:55-2:00	Lunch - Analytical laboratory will be open for instru	ment demonstrations

Moderator: Herant Khanjian. Getty Conservation Institute Session II

2:00 - 2:30	Rapid Imaging of Paint Cross-Sections in the Mol	art Project Ron M. A. Heeren , FOM Institute for Atomic & Molecular Physics
2:30 - 3:00	Mid-IR Spectroscopic Imaging: Technique and Applications	Richard Crocombe Norman A. Wright , Bio-Rad
3:00 - 3:30	Micro-ATR Application to Cultural Materials	John Reffner, Spectra-Tech
3:30-4:00	Coffee Break	
4:00-4:30	Micro-ATR of Challenging Samples	Milan Milosevic, Harrick Scientific

Use of Raman Spectroscopy in Art Conservation John Hellgeth, SRN Corp. Reception and Poster Session, Winterthur Gallery Reception Area 5:30-7:00

FRIDAY - MAY 29. 1998

4:30-5:15

7:45 am Bus departs the Wilmington Hilton

Session III Moderator: Kate Duffy, Winterthur Museum

8:00-8:30	Coffee Break	
8:30 - 9:00	IR Spectroscopy of Glass: Identification of Art I Iridescent Glass Artifacts by FTIR	Nouveau Manfred Schreiner Institute of Chemistry, Academy of Fine Arts, Vienna
9:00 - 9:45	Infrared Analysis of Minerals and Inorganic Pig	ments Richard Newman Museum of Fine Arts, Boston
9:45-10:15	Coffee Break	
10:15-10:45	History of Use of Resins in Furniture Finishes	Gregory Landrey, Winterthur Museum
10:45 -11:15	Infrared Spectroscopy of Urushi Resins	Masanori Sato, Nara National Cultural Properties, Research Institute
11:15-11:45	FTIR Analysis of Organic Materials from the Haifa Faisal Collection	Gretchen Shearer McCrone Associates

11:45 - 1:45 Lunch - Analytical laboratory will be open for instrument demonstrations

Session IV Moderator: Beth Price. Philadelphia Museum of Art

1:45 - 2:45	Infrared Spectroscopy at the National Gallery, Lond	don Raymond White National Gallery, London
2:45 - 3:00	Fatty Acid Efflorescence on Works of Art	Eugena Ordonez Museum of Modem Art
3:00 - 3:20	Organic Coatings on Metals: Late 19th C. to the Pr	esent Andrew Lins Philadelphia Museum of Art
3:20-3:50	Coffee Break	
3:50-4:10	J-CAMP.DX Joint Committee o	Robert McDonald n Atomic and Molecular Physical Data
4:10- 4:40	Interpretation of Spectra: Synthetic Resins	Jerry Lillquist 3M Corp.
4:40 -5:30	Closing remarks Spectral Interpretation Workshop	Janice Carlson, Winterthur Museum Jerry Lillquist, 3M Corp.

SATURDAY - MAY 30.1998

8:00 am Bus departs the Wilmington Hilton

Session V IRUG Data Base Workshop and Business Meeting

- 8:45-9:00 *Coffee Break*
- 9:00 10:00 IRUG Business Meeting Moderator: Boris Pretzel, Victoria and Albert Museum
- 10:00-10:30 Coffee Break
- 10:30 12:00 Data Base Workshop in the Analytical Laboratory Moderators: **Beth Price**, Philadelphia Museum of Art, **Michael Boruta**, Bio-Rad, Sadtler Div.
- 12:00 Lunch

Afternoon: Optional Museum Tours

1. Data Processing Pitfalls: Possible Problems with Spectral Data Processing and Sample Preparation

Jim de Haseth, University of Georgia

The infrared spectroscopist is faced with many obstacles in the analysis of complex samples. Sometimes we have to be concerned about how the data are processed, or how we prepare our samples. The current state-of-the-art of infrared instrumentation is excellent, and to the point that experimentalist errors often are greater than the instrumental ones. The spectroscopist must be sure that instrumentation is operating in an optimum manner, and that samples are prepared correctly and presented to the spectrometer appropriately. Some care can lead to a substantial improvement in the reliability and reproducibility of spectrometric results.

Author

Jim de Haste is Professor of Chemistry at the University of Georgia, Athens, and a well-known expert on FTIR spectrometry. Professor de Haste received his BS in Chemistry from the University of Illinois, and his PhD in Analytical Chemistry from the University of North Carolina, Chapel Hill, under the directorship of Thomas Eisenhower. He was a post-doctoral research associate with Professor Glebe Mamantov at the University of Tennessee, Knoxville. He taught at the University of Alabama prior to joining the faculty at the University of Georgia. Dr. de Haseth is the author or co-author of more than 80 scientific publications and has received five patents. With Peter Griffiths, he co-authored "Fourier Transform Infrared Spectrometry" which is considered the leading text on FTIR spectrometry.

2. Infrared Spectral Search Algorithms

Mike Fuller, Nicolet Instrument Corporation

The quality of results obtained when searching an unknown spectrum against a spectral library depends on a number of factors. The quality of the library being searched is critical to producing the correct results. When creating a spectral library many factors such as sample preparation methodology, signal-to-noise requirements, resolution, artifacts due to water and carbon dioxide, ordinate/abscissa accuracy and precision must all be carefully considered. There are also a number of search algorithms to choose from when performing a library search and each has unique advantages and disadvantages. This presentation will discuss these issues and present suggestions for optimizing the quality of results in spectral library searching.

Author

Michael F. Fuller received a BS in chemistry and a PhD in analytical chemistry from Ohio University. For five years he was a research chemist and group leader for Phillips Petroleum Company. Since 1985 he has worked for the Nicolet Instrument Corporation in a number of positions including Applications Chemist, Product Manager, Applications Manager, Technology Manager, and Director of Software Development. He is currently the Director of Marketing.

3. Infrared Spectroscopy, Microspectroscopy and Non-destructive Reflectance Spectroscopy

Kate Helwig, Elisabeth Moffatt, Scott Williams and Gregory Young, Canadian Conservation Institute.

A wide range of analyses is undertaken at the Canadian Conservation Institute using infrared Spectroscopy. Three main systems are employed: a Bomem MB 100 infrared spectrometer with a microbeam sample compartment; a Spectra-Tech IR plan research microscope interfaced to a Bomem MB 120 spectrometer; and a portable, non-destructive system composed of a MIDAC infrared spectrometer with a REMSPEC mid infrared fiber-optic probe for reflectance. Examples of analytical projects showing interesting applications of the three systems will be presented. These include: characterization of the iron oxide pigments; the use of infrared linear dichroism to study the deterioration of collagen fibers from native skin and semi-tanned leather artifacts; and the scientific examination of the 1670 Royal Charter of the Hudson's Bay Company.

Author

Kate Helwig received her BSc in chemistry from the University of Toronto, MSc in physical chemistry from Stanford University and Master's degree in art conservation from Queen's University. Since 1993 she has been working as a conservation scientist in the analytical research laboratory of the Canadian Conservation Institute where she undertakes scientific examinations and analyses of museum objects.

4. Non-destructive Mid-IR Fiber Optic Reflectance Spectroscopy Applied to the Study of Paintings

Marcello Picollo, Institute di Ricerca sulle Onde Elettromagnetiche "Nello Carrara", Firenze (I)

Nowadays IR reflectance Spectroscopy is quite widely applied to the study of works of art. Usually reflectance Spectroscopy needs additional physical quantities and more experimental parameters when compared to transmittance. Nevertheless, there is an increasing interest in using new reflectance techniques to such objects.

Among these techniques, Mid-IR Fiber Optic is the newest available technique which allows us to bring the "sampling accessory" to the object under investigation and to work without the need of any sampling operations. For these reasons, Mid-IR Fiber Optic is one of the best solutions for the analysis of those samples which are at a remote location, and/or whose shape and size do not fit into the spectrometer's sample-compartment or on the microscope's sample-holder.

In the present communication, the first available spectral data collected with a Mid-IR Fiber Optic Reflectance Probe on several tempera samples made with organic and inorganic pigments will be discussed and compared with data obtained with more traditional IR techniques.

Author

Marcello Picollo is a researcher (geologist) who has been working on spectroscopic investigations on works of art at the Institute of Research on Electromagnetic Waves (IROE), CNR, Florence (Italy) since 1991. His main focus is on pigment characterization using totally non-destructive spectroscopic and X-ray techniques. He has worked closely together with the Opificio delle Pietre Dure conservators on the study of panel paintings.

5a. Rapid Imaging of Paint Cross-Sections in the Molart Project

Ron M.A. Heeren, FOM Institute for Atomic and Molecular Physics, Amsterdam

The start of the priority program MOLART of the Dutch science foundation NWO, marked the beginning of a new consolidated effort to develop new methodologies and strategies in paintings research. The MOLART project focuses on the molecular aspects of aging in painted art. These molecular processes occurring in the course of time underlie the chemical and physical changes in a painting. Especially the aging mechanisms in the organic constituents of an aged paint formulation are poorly understood. We have developed new spectroscopic methodologies to study the organic composition of individual paint layers. In this presentation, we will focus on infrared imaging of paint cross-sections. A detailed description of the microscopic analysis of an embedded paint cross section with a novel FTIR imaging technique will be provided. This technique allow the creation of functional group images of paint cross-sections of a 400 x 400 micrometer area with a diffraction limited resolution (~ 6 micrometer @ 2000 cm-1). A FTIR. image with 16 cm-1 spectral resolution can be acquired in 8 minutes. The advantages and disadvantages of this technique in painting research will be evaluated. Sample preparation turns out to be crucial for the success of the methods used and some of its practical aspects for organic surface analytical techniques will be discussed. Various examples of FTIR imaging analysis carried out with this novel technique will be presented. These experiments demonstrate the feasibility of chemical imaging of the organic constituents of paint cross-sections. An outlook towards the usage of macroscopic FTIR imaging will be presented if time permits.

Author

Ron M.A. Heeren obtained a BSc in 1988 after which he proceeded to obtain a PhD degree in technical physics in 1992 at the University of Amsterdam on plasma-surface interaction in the framework of a pan-European nuclear fusion project. After two years of post-doctoral work on high-resolution mass spectrometry he joined the MOLART research team at the FOM-Institute for Atomic and Molecular Physics as a project leader, heading the instrumental developments in early 1995. In a collaborative effort with art historians and conservation scientists he pursued his main research interest in spatially resolved surface analytical techniques for organic compounds with the emphasis on FTIR and mass spectroscopic imaging techniques. Additional research topics included Infrared reflectography, the interaction of lasers with paintings, fundamental studies on the energetics of macromolecular systems and the development of new mass spectrometry.

5b. Mid-infrared Spectroscopic Imaging: Technique and Applications

Richard A. Crocombe and Norman A. Wright, Bio-Rad Spectroscopy Division

Last year we described the development and applications of an FT-IR spectroscopic imaging system using a stepscan FT-IR spectrometer, FT-IR microscope accessory and an MCT focal plane array detector. This detector has 64×64 pixels, and covers the range 3800 - 800 cm⁻¹. This technique can rapidly generate very high quality, chemically specific images from a wide variety of samples. Prior to that we described the use of an InSbFPA in this application, working in the near-IR, and as far as 2000 cm⁻¹ as the long wavelength limit.

Most of the examples in the literature have dealt with microscopic samples. In this paper we will also describe a macrosample spectroscopic imaging system, based on the same technology, for the analysis of samples up to 10 mm in diameter. This imaging macrosampler incorporates visual imaging of the sample, as well as infrared spectral imaging.

FT-IR microscopy, which has been used in these early applications, does not require exotic sample preparation. Samples can be viewed either in transmission or reflection and no staining or coating is required. In the case of a sample that is visibly transparent, because the refractive indexes of different components are so similar, the infrared image derived from spectral absorption gives intrinsic contrast enhancement. The technique is non-destructive and works well for solids and liquids. The contrast that is seen in these images arises from the inherent infrared absorptions from the sample, i.e., every chemical compound has its own characteristic pattern of absorptions which create an infrared signature.

This paper describes applications of this technique in many fields, concentrating on polymer analysis. For instance, many commercial packaging materials are polymer laminates, and this technique allows very rapid visualization and identification of all the layers of the sample, with no prior knowledge of its components. Typical samples will be shown, with spectra from each layer, and identification of the composition of the layers via spectral searching.

REFERENCE

RA. Crocombe, N.A. Wright, D.L. Drapcho, W.J. McCarthy, P. Bhandare, E.J. Jiang, "FT-IR Spectroscopic Imaging in the Infrared "Fingerprint" Region Using an MCT Array Detector", Microscopy and Microanalysis Proceedings 1997, Vol 3 Supp 2, p 863

Author

Richard A. Crocombe, BA Oxford University (England) 1973 (Chemistry); PhD Southampton University (England) 1977 (Spectroscopy/Inorganic Chemistry); Postdoctoral Fellowship University of Tennessee 1977-81 (Spectroscopy); Bio-Rad Digilab Division 1981 - present. Working in product development and product management, especially on items like step-scan FT-IR hardware and applications, imaging and array detectors, FT-IR software. Most recently headed the development team for Bio-Rad's latest FT-IR spectrometer, Excalibur.

Norman A. Wright, BS Chemistry, Cat. Polytechnic State Univ., MS Chemistry (Analytical), Univ. of Arizona, PhD, Analytical Chemistry, Univ. Of California, Riverside (Spectroscopy), 1987 to present: Instrumentation Engineering for the Bio-Rad Digilab Division. His work has included developing Raman Accessories, hyphenated techniques (GC-IR, TGA-IR) and most recently the IR array detector products.

6. Micro-ATR Application to Cultural Materials

John A. Reffner, PhD., Trace Consulting, Stamford, CT 06902

The chemical analysis of artistic and cultural materials challenges the analyst because of their complexity and restrictions that limit removal of material. Since its introduction into infrared analytical spectrometry in the mid-1950's, internal reflection has demonstrated unique advantages for the analysis of difficult, intractable samples. It is a standard method for the collection of infrared spectra of solids and liquid samples with unique properties for nondestructive analysis. The spectra recorded using internal reflection are called attenuated total reflection (ATR) spectra. With the invention of the ATR microscope objective in 1989, a new technology was created that has great value for the analysis of artistic and cultural materials. The fundamental principles, instrumentation and applications of micro-ATR will be presented.

Author

For the past fifteen years John A. Reffner has been involved in developing infrared microspectrometry instrumentation and applications. His long association with Spectra-Tech, Inc. ended in May 1998, with his retirement. He now operates Trace Consulting. Before joining Spectra-Tech, Dr. Reffner was a principal research scientist with American Cyanamid, Assistant Director of the Institute of Materials Science at the University of Connecticut, Storrs, CT, and Research Director at McCrone Associates, Chicago, IL. Dr. Reffner has taught many courses in microscopy. He established a course in polymer microscopy at U-Conn., instructed courses in Forensic microscopy at John Jay College, NY and the U. of New Haven, taught textile microscopy at The Fashion Institute, NY, and presented numerous short courses. In addition to over sixty publications, Dr. Reffner holds seven patents. His scientific interests are in microscopy and infrared microspectroscopy. He has applied these disciplines to polymer, materials and forensic sciences. As a visiting scientist at the National Synchrotron Light Source, Brookhaven National Laboratory, he and Gwyn Williams conducted the first experiments combining infrared microscopy and Synchrotron radiation. He is a special consultant to the Connecticut State Police Forensic Laboratory and serves on the editorial board of the Academy of Forensic Sciences.

7. Micro-ATR of Challenging Samples

Milan Milosevic and Susan Berets, Harrick Scientific Corporation

In 1991, the field of infrared internal reflectance micro-spectroscopy virtually exploded. Internal reflectance (ATR) micro-sampling offered an alternative to transmission beam condensers and microscopes. This new alternative was more convenient and offered higher sample throughput than transmission methods.

Internal reflectance (ATR) micro-sampling is a powerful tool for examining small samples and small areas of large samples. Because of the small sampling area and the ability to achieve high contact pressures, these micro-samplers can be used to examine a much broader range of samples with minimal sample preparation.

This presentation utilizes ATR micro-sampling to examine various samples that are of particular interest to art historians and conservators. These samples are analyzed using Harrick's SplitPea.

Author

Milan Milosevic is the Executive Vice President of Harrick Scientific Corporation. He has been designing FT-IR accessories for over ten years and is the author of numerous papers on FT-IR Spectroscopy. He studied physics at Brown University where he earned an MS degree.

8. Use of Raman Spectroscopy in Art Conservation

Dr. John W. Hellgeth, Principal Associate, The SRN Company, LLC.

Throughout the past decade, several advances in technology have revolutionized the practice of Raman Spectroscopy. As a Spectroscopy complementary to infrared Spectroscopy, Raman Spectroscopy is also an important tool for art conservation in that valuable molecular information may be gained with minimal sample prep and no material destruction. The current "state-of-the-technology" for Raman Spectroscopy affords much greater ease of use, selectivity and versatility in the analysis of materials at a molecular level. Its applicability to diverse material analysis has expanded greatly with the advent of better laser sources, detectors, and light filters. These devices have been employed effectively in both dispersive and Fourier transform spectrometers to yield utility on par with infrared Spectroscopy.

Author

Over the past 23 years, John Hellgeth has pursued interests in hyphenated techniques and vibrational micro Spectroscopy. He received degrees in Biochemistry (BS 1977) and in Analytical Chemistry (PhD 1986) from Virginia Polytechnic Institute and State University. As a research scientist at VPI & SU, he established the Digital Imaging and Vibrational Spectroscopy Lab for the NST Science and Technology Center on High Performance Polymeric Adhesives and Composites, one of thirteen National Centers of Excellence in Science. He joined Nicolet Instruments Corporation in 1992 as a scientist providing IR/Raman and Microspectroscopy applications development and training for their clients. In 1995, he joined Spectra-Tech, Inc. as the product manager for vibrational microspectroscopy instruments. Most recently, he has established the SRN Company, LLC, a company which provides scientific expertise and lab/research resources for chemical and materials analysis. He is actively involved with the ASTM El 3 Committee on Vibrational Spectroscopy, the Society for Applied Spectroscopy and The Coblentz Society.

9. IR Spectroscopy of Glass: Identification of Art Nouveau Iridescent Glass Artifacts by FTIR

Manfred Schreiner, Institute of Chemistry, Academy of Fine Arts, Vienna, Austria

Art Nouveau iridescent glass has been highly esteemed for its splendid rainbow colour gleam. Iridescence itself is an interference effect occurring whenever a reflecting material is coated by a very thin layer with a refractive index different from that of the bulk. L. C. Tiffany was the first one to be granted a patent for the manufacturing glass with iridescent surfaces in 1881 followed by several centers in Europe, where the highest quality was achieved by J. Loetz Wwe. from Klostermuehle in Bohemia. After the Loetz-Patent in 1898 an intensive production of iridescent glass artifacts with exceptional quality started. Although influenced by objects of Tiffany, Loetz artifacts were not the result of an imitation but rather of a parallel development due to alternative technological approaches. In a project of Austrian research institutions with the Museum of Applied Arts, Vienna, a "recognition" procedure based on nondestructive analytical techniques supplemented with efficient experimental data processing, which allows quick and reliable identification of Art Nouveau iridescent glass provenance, was developed. More than 400 glass fragments and samples provided with the kind agreement of the Museum of Applied Arts, Vienna, a "recognition" procedure based on non-destructive analytical techniques supplemented with efficient experimental data processing, which allows quick and reliable identification of Art Nouveau iridescent glass provenance, was developed. More than 400 glass fragments and samples provided with the kind agreement of the Museum of Applied Arts in Vienna, the New York Historical Society, the Passauer Glasmuseum in Germany as well as private collectors have been analyzed by FTIR (Fourier Transform Infrared Spectrometry) and XRF (X-ray Fluorescence Analysis) in a non-destructive way. Small glass splinters taken from special fragments could also be investigated by SEM/EDX (Scanning Electron Microscopy with Energy Dispersive X-ray Microanalysis) after embedding in resin. Although this method is not entirely non-destructive, its application enables a better understanding of the chemical composition of the bulk glass and the iridescent layer. The clusters obtained by the statistical evaluation of the FTIR spectra could be proved by XRF as well as by SEM/EDX. In general, the chemical composition of the bulk glass used by Tiffany differs profoundly from Loetz glass. Additionally, differences in the elements present in the iridescent surface layer could be detected due to the different technologies used for the production of the iridescent glass artifact by Tiffany and Loetz.

Author

Manfred R. Schreiner studied chemistry at the Vienna University of Technology. In 1975, he received an engineering degree in chemistry; in 1979, a PhD in material science (hard metals, cemented carbides). In 1980, he received a Fulbright Stipend to conduct research at UCSD (University of California San Diego) on hydrogen storage in metals and intermetallic compounds. He became an Assistant Professor at the Academy of Fine Arts, Vienna, in 1981. In 1988, he became an Associate Professor at the Institute of Chemistry of the Academy of Fine Arts, Vienna. His research projects include weathering of medieval stained glass, non-destructive analysis of artifacts by x-ray fluorescence analysis, authenticity of medieval silver coins.

10. FTIR Analysis of Pigments and Minerals

Richard Newman, Museum of Fine Arts, Boston, MA

This paper reviews the application of FTIR analysis to identification of inorganic compounds. The types of compounds that can and cannot be analyzed by a typical mid-IR range instrument are described, using as examples materials that could be encountered in a museum laboratory, including corrosion products, pigments, efflorescent salts, etc. and a number of common minerals. The following topics are discussed in detail: (1) analysis of inorganic compounds that contain polyatomic ions such as carbonates and sulfates; (2) analysis of silicate minerals; (3) the importance of hydroxyl absorption bands in identifying inorganic compounds. As much as possible, examples will be actual samples taken from artifacts.

Author

Richard Newman has a BA in art history, an MA in geology and completed a three-year apprenticeship in conservation science at the Center for Conservation and Technical Studies, Harvard University Art Museums. He has been Research Scientist at the Museum of Fine Arts in Boston since 1986. Among his specific interest are applications of FTIR and Raman microscopy to analysis of inorganic compounds.

11. A History of the Use of Resin in Furniture Finishes

Gregory J. Landrey, Director of Conservation/Senior Conservator, Winterthur Museum, Garden & Library.

This presentation will focus on the nature of traditional furniture finishes. While the emphasis will be on resins including colophony, copal, mastic, sandarac and shellac, oils and waxes will be discussed as well. Period recipes, both published and hand written accounts, will be presented. The types of materials historically used in the care and polishing of furniture will be included. The complexity of traditional coating materials and the subsequent challenges in the analysis of a surviving finish will be emphasized.

Author

Gregory Landrey worked as a cabinet shop assistant at the Comer Cupboard Antiques in Strafford, PA, from 1972-1979. In addition to the bench training, Gregory went to Gettysburg College, graduating with a BA in History in 1977. He started at the Winterthur Museum in 1979 as an assistant furniture conservator. He served as the Head of Winterthur's Furniture Conservation Laboratory from 1986-1995. He now is the Director of Conservation and Senior Conservator. Gregory is an adjunct associate professor for the Winterthur Museum/University of Delaware Program in Art Conservation. His most recent publications include: "Furniture with a Secret" in <u>American Woodworker</u> and "Deceit, Deception and Discovery" in <u>Today's Chemist at</u> <u>Work</u> co-authored with Janice H. Carlson.

12. Infrared Spectroscopy of Urushi Resins

Masanori Sato, Nara National Cultural Properties Research Institute

The urushi (Japanese lacquer) had been commonly used for various archaeological remains. This report describes the application of FT-IR microscopic Spectroscopy to the rapid and versatile identification of urushi excavated from several Japanese archaeological sites. The FT-IR spectrum of ancient urushi samples clarified that urushi molecules are rather stable, irrespective of preserved environmental conditions during the long term. As for urushi of late Edo period, several samples of urushi layer were obtained from excavated tools used for the refinement of urushi raw material. The distinction of other natural resins from urushi was also possible for FT-IR Spectroscopy. The characteristic absorption peaks of some natural resins were summarized.

Author

Masanori Sato received BSc (1954), MSc (1956) and PhD (1959) in chemistry from the Faculty of Science, Kyoto University (Japan). He was associate professor (1968-1978) and full professor (1978-1996) in analytical chemistry at the Kyoto Institute of Technology. Since 1996, he has been a guest conservation scientist at Nara National Cultural Properties Research Institute. His principal interest focuses on the instrumental analysis of excavated organic matter.

13. FTIR Analysis of Organic Materials from the Haifa Faisal Collection

Gretchen L. Shearer, McCrone Associates

The purpose of this paper is to illustrate the use of FTIR in the conservation and study of ethnographic collections. The Haifa Faisal collection represents an interesting and varied collection since the nomadic lifestyle of the Saudi peoples resulted in a wide variety of materials used in their traditional arts. These objects present many potential challenges to the conservator. Several illustrations will be presented where FTIR is used to identify organic materials used in composite objects in order to accurately catalogue the objects, determine the best choice of treatments and make better informed decisions about long term storage/display.

Author

Gretchen Shearer received a BA in chemistry from the College of Wooster in 1985 and a PhD in archaeology from the University of London in 1989. Her thesis work, ("An evaluation of Fourier transform infrared Spectroscopy for the characterization of organic compounds in art and archaeology"), focused on the analysis of both natural products and early synthetic polymers to form a reference library for archaeological samples. As part other L.W. Frohlick research fellowship from the Metropolitan Museum of Art, she conducted research in the area of alkoxysilane based stone consolidants and continued her study of early plastics. She has done postdoctoral work in biochemical research at the University of Iowa and the University of Chicago. Since 1993, she has been employed as a research chemist at McCrone Associates in Chicago. As a member of their chemistry group, she uses infrared micro Spectroscopy, gas and liquid chromatography and polarizing light microscopy to solve contamination problems for a diverse group of pharmaceutical and other industrial clients. Her continuing research interests are in the application of infrared Spectroscopy to problems in art and archaeological conservation.

14. Infrared Spectroscopy at the National Gallery, London Raymond White, Scientific Department of the National Gallery, London

The paper deals with the role played by FTIR-microscopy within the Organic Section of the Scientific Department at the National Gallery, London, The emphasis is on the importance of the interplay between different analytical techniques and how, by combining FTIR-microscopy with techniques such as gas chromatography- mass spectrometry or HPLC-electrospray mass spectrometry, a more satisfactory analytical outcome may result for Old Master paints. The strengths and shortcomings of the technique of FTIR-microscopy are discussed.

Author

Raymond White joined the Scientific Department of the National Gallery, London in 1969 as an organic chemist to specialize in the study of and chromatographic analysis applied to natural products encountered in easel paintings. As Principal Scientific Officer, he heads the organic research and analytical facility within the National Gallery and has published a number of papers on the chemistry and identification of organic materials in paintings. For many years he has specialized in the development and application of gas chromatography-mass spectrometry and FTIR-microscopy to Old Master medium analysis, as well as the application of high performance liquid chromatography and electrospray spectrometry to the constituents of lake paints. He regularly publishes upon this subject in the National Gallery Technical Bulletin and has lectured widely on natural product analysis and chemistry, as well as painting technique. He is joint author of "The Organic Chemistry of Museum Objects".

15. Fatty Acid Efflorescence on Works of Art

Eugena Ordonez, Museum of Modem Art

Although the presence of hazy, obscuring, whitish patches on works of art has been frequently encountered, the consequent conservation treatment procedure has usually not involved determining their causes. This research looked at twenty museum objects exhibiting this condition. In seventy-five percent of the cases, it was found that localized accumulations of fine particles on the surface of the work were responsible for scattering incident light thus causing the hazy patches. The identification of the surface particles as fatty acids (or related compounds) was done primarily with FT-IR microanalysis, a versatile, available method that produced considerable information from minute samples. The findings led to reference research into the sources of the fatty acids and the circumstances favoring their aggregation on the surface.

Author

Eugena Ordonez received a BA in Studio Art and a BA in Biological Sciences from the University of California at Santa Barbara. She graduated from the Winterthur Museum/University of Delaware Art Conservation Program specializing in the study and treatment of paintings. She has been at the Museum of Modem Art in New York since 1986 and is also presently a doctoral candidate in the University of Delaware Art Conservation Research Program. Her dissertation title is "An Investigation into the Faktura of Paintings."

16. Clear coatings on metals, 1870-1998

A Lins, B. Price, Philadelphia Museum of Art; and J. Carlson, Winterthur Museum

This paper begins with a brief review of the historical use of clear coatings on metal from 1870 to the present. Following is a brief discussion of some of sampling and contamination issues or coatings on metals, illustrated by examples from architectural and sculptural surfaces:

- 1. balustrade from the US Treasury, 1870, Washington, DC,
- 2. balustrade from Philadelphia City Hall, 1872,
- 3. ceiling of the Lincoln Memorial, 1918-22, Washington, DC,
- 4. Rodin sculptures, Rudier casts, 1920's.

Author

Andrew Lins is a graduate, BA, of the New York University conservation program and completed an MSc in electrochemistry and corrosion engineering. Since 1979, he has been head of the decorative arts & sculpture conservation laboratory at the Philadelphia Museum of Art; in 1997 he became Head of the Conservation Department. He has served as a technical consultant for the Department of Public Works and Historic Commission (City of Philadelphia) and the United States National Parks Service. In addition, he has consulted and lectured widely on metals and their corrosion. His architectural projects have included the Lincoln Memorial ceiling, the Liberty Bell, the Statue of Liberty, and William Penn atop Philadelphia's City Hall.

17. J-CAMP

Robert McDonald, Joint Committee on Atomic and Molecular Physical Data

JCAMP-DX is a standard file transfer protocol for exchange of spectra and related chemical information between spectrometer data systems of different manufacture, general purpose laboratory computer systems, and personal computers. JCAMP-DX is compatible with all media; telephone, the Internet, magnetic and optical, magnetic tape, and even the printed page (via optical reader).

All data are stored as labeled fields of variable length using printable ASCII characters. A JCAMP-DX file is a text file that can be viewed, corrected, and annotated via an ASCII text editor. JCAMP-DX can accommodate Raman, infrared, UV, NMR, mass, and other types of spectra, x-ray powder patterns, chromatograms, thermograms, and other data which require the capability of representing contours as well as peak position and intensity. JCAMP-DX provides for combining adequate information about sample and method of observation with the numerical output of instruments.

Author

Robert S. McDonald, BS in Engineering Physics, Univ. of Maine, and PhD in Physical Chemistry, MIT. At American Cyanamid Stamford Research Laboratories during WW2, he was part of the team that developed the prototype of the first Perkin-Elmer infrared spectrometer. Later, at General Electric R & D Center, he supervised infrared Spectroscopy and pursued research studies of surface-functional groups and point defects in semiconductors by IR. For a number of years, he authored the Biennial Review of Infrared Spectrometry for Analytical chemistry. More recently, he has been involved in the development of JCAMP-DX, a standard protocol for exchange of data between laboratory instruments, such as infrared, Raman, NMR, and mass spectrometers.

18. Interpretation of the Infrared Spectra of Coatings

Jerry Lillquist, 3M Company

The interpretation of infrared spectra is based both on experience and on spectra of known compounds, but even then, there are many situations where the exact identification of an unknown may never be determined by its infrared spectrum alone. This talk will be a short review of basic infrared spectral interpretation showing spectra of various commercial coatings and components as examples. Coatings are one example of the broad category of compounds called polymers which may have more than one function, i.e., rubbers, polyesters, epoxies, etc., that can be used both as adhesives and as coatings.

Author

Jerry Lillquist graduated from the University of Minnesota with an MS in organic chemistry but he had more fun figuring out what he had made than in making it. He joined 3M in 1965 in the Analytical Section of Corporate Research Labs where he interpreted infrared and NMR spectra. In 1995, he moved to the Analytical Section of Specialty Materials Division where interpreting fluorine NMR spectra is his major task.

DATABASE WORKSHOP

19. IRUG Database Workshop

Quality issues for generating IR Spectra for use in databases will be discussed. Also covered will be the organization and maintenance of databases.

Author

Michael Boruta is currently the Product Manager for Bio-Rad - Sadtler's IR product line. He graduated in 1976 with a BS in chemistry from Delaware Valley College of Sciences and Agriculture and employed in Sadtler's IR research laboratories since 1977.

POSTER

Characterization of Elastane Fibres Using Fourier Transform Infrared Spectroscopy

Silvia Valussi Royal College of Art/Victoria & Albert Museum Joint Course in Conservation Conservation Science Area: Twentieth Century Materials

Elastomeric fibres are used as the minor component in stretch garments to provide stretch with recovery. The main categories of elastomeric fibers are rubber and Olsten. Olsten fibers, also known as Spandex, by definition consist of at least 85 of segmented polyurethane. Segmented polyurethanes contain alternating "soft" and "hard"segments linked by urethane bonds. The soft segments can be polyethers or polyesters in which the hard segments usually consist of substituted polyurethanes and polyurea. It is this molecular structure that confers the fiber the capacity to be stretched up to 5-6 times the original length and completely recover.

Elastane fibers were created in the 1950's to improve the process and properties of rubber threads and became commercial around 1960. Since then elastanes have changed the whole face of fashion by bringing possibilities of fit, comfort and shape retention, that designers and manufacturers had previously only dreamed about.

Since elastomers are polyurethanes, they are likely to deteriorate significantly, even in the benign environment of the museum. Cases of degradation of polyurethanes in 20th C. Textiles in museum collections have been already reported. It is actually well-known that polyurethane based materials are sensitive to atmospheric conditions and require care in storage, display and cleaning if preservation of them is to be achieved.

This poster illustrates a preliminary study of different contemporary samples of base Lycra by FTIR Spectroscopy using the ATR-golden gate-diamond cell and SEM. Fibre thickness was in the range between 20 and 1800 dtex. Also a number of Lycra fibers of 10-15 dtex were extracted from combination yarns provided by an English manufacturer.

FTIR spectra indicate that all samples analyzed are made from polyether-polyurethanes while SEM photographs show the multi filament structure of each fiber. In particular, evidence of degradation on the surface of the fibers is observed in 10-year-old Lycra samples. The research is still in progress and other analytical techniques are expected to provide more information about the fiber composition and structure.

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