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Users' Group for **Mass Spectrometry and Chromatography**

MaSC 2015 Meeting Program

Art Institute of Chicago
Chicago, IL, USA

21-22 May 2015

Preface

The Committee of the Users' Group for Mass Spectrometry and Chromatography (MaSC) welcomes you to the seventh MaSC Meeting at the Art Institute of Chicago.

Since its foundation in 2003, MaSC has established itself as a vital international forum for interaction and discussion among scientists using chromatographic and mass spectrometric techniques for the study of art and historical artefacts. The Group currently has over 100 members, representing 85 institutions – primarily cultural and academic organisations – in 26 countries.

The 2015 Meeting was preceded by a three-day workshop on the topic of inorganic and isotope ratio MS techniques applied to the study of art and archaeology, hosted by Northwestern University, the Field Museum and Agilent Technologies. We are very grateful to the workshop instructors for sharing their expertise and discussing new research: Patrick Degryse, Andrew Shortland, Marc Walton, Monica Ganio, Laure Dussubieux, Mark Golitko and Jon Talbott. Several of the workshop instructors will also speak in the first session of the Meeting dealing with inorganic and isotope MS methods: while well established in the field of archaeology these techniques have not been applied so widely to the study of artists' materials and we hope that the Meeting will be an opportunity for scientists working in different fields to discuss new ideas and avenues for research in this area.

As usual, the 2015 Meeting has a broad programme, with the diverse specialities and research activities of MaSC members represented by further sessions that highlight challenges and strategies for the characterisation using chromatographic and MS techniques of proteins, plant gums, Asian and European lacquers, paint media and modern materials, to address questions including provenance, artists' techniques, and degradation of materials.

We would like to thank the staff of the Art Institute of Chicago for their support and assistance in organising and hosting this event – in particular Francesca Casadio, Frank Zuccari, Jann-Nicole Trujillo, Celine Daher, Kim Muir, and the staff of Special Events, AV and Information Services.

The Meeting was made possible with generous support from the Northwestern University/Art Institute of Chicago Center for Scientific Studies in the Arts (NU-ACCESS).

We hope you enjoy the Meeting, and your visit to Chicago!

The MaSC Committee:

Ken Sutherland
Christopher Maines
Klaas Jan van den Berg
Ester Ferreira
Catherine Higgitt
David Peggie

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Meeting schedule

MaSC 2015 Meeting Schedule
Art Institute of Chicago, Millennium Park Room

Thursday May 21st

9.15 Welcome, registration

9.30 Opening remarks
Ken Sutherland

Session 1: inorganic and isotope MS methods, chair **Laure Dussubieux**

9.40 Workshop review
Marc Walton, Klaas Jan van den Berg

10.10 Provenancing the raw materials of Greco-Roman glass making
Patrick Degryse

10.35 Scientific insights to the Berthouville Treasure: understanding the connectivity between silver objects in a Gallo-Roman shrine
Lynn Lee, Patrick Degryse, Marc Walton

11.00 Coffee

11.30 Making the most of ancient biomolecules through their isotope compositions
Richard Evershed

12.00 Radiocarbon and the Ancient Egyptian Chronologies
Andrew Shortland

12.25 Carbon and oxygen isotopes as indicators of provenance in cultural artefacts: a case study of Thomas Jefferson's tombstone plaque
Christine A.M. France, Carol A. Grissom

12.50 Lunch break/poster session

Session 2: degradation markers in modern materials, chair **Klaas Jan van den Berg**

2.20 The use of VOC analysis in the care of modern materials
Katherine Curran, Mark Underhill, Lorraine Gibson, Matija Strlic

2.45 GCMS method for calculating percent acetyl content in cellulose acetate
Joy Mazurek, Miriam Truffa Giacheta, Suzanna Etyemez, Michael Schilling

3.10 Short poster presentations I

3.40 Coffee

Session 3: MS strategies for the characterisation of proteins and gums, chair **Ester Ferreira**

4.10 Identification of plant gums in artworks: a promising strategy based on enzymatic digestion and MALDI-TOF MS
Clara Granzotto, Julie Arslanoglu, Caroline Tokarski

4.35 Unlocking protein-binder/pigment interactions by coupling ELISA with MS techniques
Natalya Atlasevich, Caroline Tokarski, Julie Arslanoglu

5.00 You can’t tell a book by its cover: analytical adventures in anthropodermic bibliopegy
Daniel P. Kirby, William S. Lane

5.25 Closing remarks
Contemporary collections and exhibitions at AIC: an introduction by James Rondeau, Dittmer Chair and Curator for the Department of Contemporary Art

6.00 Drinks reception at AIC

Friday May 22nd

9.15 Opening remarks

Session 4: composition studies of traditional lacquers and paint media, chair **David Peggie**

9.30 AMDIS & Excel: a powerful combination for evaluating Py-GCMS results from Asian and European lacquers
Michael Schilling, Henk van Keulen, Arlen Heginbotham, Mike Szelewski

9.55 Natural resins in European lacquerware: a systematic Py-GCMS quality control
Louise Decq, Michael Schilling, Frederic Lynen, Wim Fremout, Vincent Cattersel, Delphine Steyaert, Charles Indekeu, Emile Van Binnebeke, Steven Saverwyns

10.20 Binding medium analysis of Florentine paintings of the 14th-16th centuries: on the transition from tempera to oil technique
Ursula Baumer, Patrick Dietemann, Christoph Steuer, Daniela Karl

10.45 Coffee

Session 5: nineteenth-twentieth century paint media, chair **Christopher Maines**

11.15 Interpreting analyses of paint samples containing both aqueous and oleoresinous binders
Patrick Dietemann, Wibke Neugebauer, Cedric Beil, Irene Fiedler, Ursula Baumer

11.40 Novel application of LCMS for the characterisation of lipid binders in art: elucidation of the composition of paint materials used by Edvard Munch
Jacopo La Nasa, Marco Zanaboni, Daniele Uldanck, Ilaria Degano, Francesca Modugno, Hartmut Kutzke, Biljana Topalova-Casadiego, Eva S. Tveit, Maria Perla Colombini

12.05 Lunch break/poster session

1.35 The influence of oil paint constituents and time on 20th century oil paints as studied by ESI-MS
Klaas Jan van den Berg, André van der Doel, Eliane van Dam, Birgit van Driel, Art Ness Proano Gabor, Aviva Burnstock

2.00 Short poster presentations II

2.30 Coffee

3.00 Business meeting/closing remarks

4.00 CLOSE

MaSC 2015 Poster presentations

Microscale radiocarbon dating of paintings
Laura Hendriks, Irka Hajdas, Markus Küffner, Cameron McIntyre, Nadim C. Scherrer, Ester S.B. Ferreira

Characterization of the archaeological mortars of the ancient Al-Saraya Al-Hamra Castle in Libya
Ozden Ormanci, Ozge Hanyali, Burcu Kirmizi, Seda Tuncel, Veysel Boz, Ugur Alanyurt, Sedat Kurugol, Meric Bakiler

John Constable's drawing fixative: identification of a proteinaceous surface coating by peptide mass fingerprinting
Daniel P. Kirby, John Slavin

Not on your tintype? Py-GCMS analysis of materials present on historic tintype plates
Corina Rogge

Evidence of lignin oxidation in archaeological waterlogged wood by MS based techniques: the case of the Neolithic village of "La Marmotta", Italy
Diego Tamburini, Jeannette Jacqueline Łucejko, Erika Ribechini, Maria Perla Colombini

Py-GCMS with in situ silylation for the analysis of treated composite archaeological wood artefacts: the ArCo Project
Diego Tamburini, Jeannette Jacqueline Łucejko, Francesca Modugno, Maria Perla Colombini, Hartmut Kutzke, Susan Braovac, Martin Mortensen, Gilles Chaumat, Francesca Gambineri

Tibetan wall paintings in Nako: behind the scenes of Buddhist artists' craftsmanship
Tatjana Bayerová, Václav Pitthard, Wim Fremout

Study of painting materials of polychrome and monochrome colored pottery of the Eneolithic agrarian European cultures
Kamilla B. Kalinina, Elena G. Starkova

Comparative study of paint materials in three outdoor Keith Haring works: Pisa, Melbourne and Paris murals
Jacopo La Nasa, Sibilla Orsini, Francesca Di Girolamo, Ilaria Degano, Francesca Modugno, Maria Perla Colombini

Characterization of natural dyestuffs in plant extracts and historical textiles by GCMS
Chiara Riedo, Monica Gulmini, Oscar Chiantore, Laura Degani

Identification of red dyes by DART-MS: examples from North and South American archaeological contexts
Ruth Ann Armitage, Kathryn Jakes

A multi-disciplinary approach to analysis of historical red lake pigment samples
Diane Kunzelman, Ezio Buzzegoli, Mattia Patti, Claudia Marchese, Daniele Uldank, Ilaria Degano, Francesca Modugno, Ilaria Bonaduce, Margherita d'Ayala Valva, Francesca Rosi, Francesca Gabrieli, Sara Bellei, Austin Nevin

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Abstracts

Provenancing the raw materials of Greco-Roman glass making

Patrick Degryse
Department of Earth and Environmental Sciences, University of Leuven, Belgium

Using newly developed isotopic analysis methods, the primary provenance of Greco-Roman glass can be investigated. It is clear from geological prospecting that suitable sands for natron glass making are rare. A limited number of glass factories in the eastern Mediterranean and North Africa were melting sand to glass in the Hellenistic period. In imperial – early Roman times, the origin of primary natron glass lies in the western as well as the eastern Mediterranean and possibly in North Africa. Apparently, investments were made in several glassmaking units all over the Empire. Several North African flux sources are likely to have supplied flux for primary glass making. In late Roman – early Byzantine times, natron glass making falls back on glass making sites in the eastern Mediterranean, such as the factories known from excavations in Syro-Palestine. The discovery by scientific analysis of this phasing in glass making in the Hellenistic-Roman world adjusts our knowledge of the history of glass, and our interpretation of the archaeological record. This aspect of the Roman economy can now be integrated in further studies of the Roman world.

Scientific insights to the Berthouville Treasure: understanding the connectivity between silver objects in a Gallo-Roman shrine

Lynn Lee[1], Patrick Degryse[2], Marc Walton[3]
1. Getty Conservation Institute, Los Angeles, CA, USA
2. Department of Earth and Environmental Sciences, University of Leuven, Belgium
3. NU-ACCESS, Northwestern University, Evanston, IL, USA

In 1830, an ancient treasure of Gallo-Roman silver objects was discovered in the town of Berthouville in Normandy, France. The Berthouville Treasure, belonging now to the Bibliothèque nationale de France, made a journey to the J. Paul Getty Museum for a four-year programme of conservation treatment and research. Scientific analysis was carried out on approximately 90 samples using solution-based inductively coupled plasma mass spectrometry, in conjunction with lead isotope analysis, to provide more understanding of the larger context of silver production and distribution in the Roman Empire. In addition, because the majority of the objects were found in detached pieces – such as the foot ring, handle and body of a *skyphos* – the isotopic and trace elemental signatures were useful to elucidate the pairings of the separated parts of the same object and provide a contextualized understanding of the relationship between objects of similar groupings. The results of this study provide an understanding of metal production, trade, and the highly skilled craftsmanship of ancient Roman artisans.

Making the most of ancient biomolecules through their isotope compositions

Richard P. Evershed
Organic Geochemistry Unit, School of Chemistry, University of Bristol, UK

It is a remarkable fact that organic molecules that rapidly degrade in hours or days in extant environments will survive for many millennia in certain protected archaeological contexts. Amongst the wide range of classes of biomolecules now known to persist in the archaeological record lipids (in archaeological pottery) and the structural protein collagen (in skeletal remains) appear to be the most abundant. While confirming the chemical integrity of such biomolecules is vital to confirm their survival, the real key to exploiting their potential to answer questions relating to human activity in prehistory rests on unlocking their sub-molecular isotopic compositions. This presentation will explore the analytical approaches used in their determinations and present applications of the use of fatty acid and amino acid compound-specific isotopic compositions to explore a range of aspects of human diet and agriculture in the past.

Radiocarbon and the Ancient Egyptian Chronologies

Andrew Shortland
Cranfield Forensic Institute, Cranfield University, UK

The chronology of ancient Egypt forms the backbone of the chronology of the old world from at least 3000 BC until the middle of the first millennium BC. It is constructed from archaeological and historical evidence, particularly king lists, and pinned to absolute time by rare recordings of solar, lunar and astral phenomena. The chronology is neither complete nor uncontroversial and it is especially problematic for the early periods. Several projects run by a collaborative group of universities, including Oxford, Cranfield, and University College London, have attempted to apply independent scientific dates to the Egyptian chronology. This involved the radiocarbon dating of short-lived material from well-known contexts tightly dated to a single king’s reign or even a year. These dates were then combined using new Bayesian techniques to incorporate regnal order and sometimes length. This allowed the derived radiocarbon probabilities for the accession dates of kings to be determined with much higher precision, down to a decade in the case of the Egyptian New Kingdom. For the first time, this allows radiocarbon dating to have a say in the discussion about how the historical chronology should be reconstructed.

Carbon and oxygen isotopes as indicators of provenance in cultural artefacts: a case study of Thomas Jefferson's tombstone plaque

Christine A.M. France, Carol A. Grissom
Smithsonian Museum Conservation Institute, Suitland, MD, USA

Stable isotopes are a useful indicator of provenance in archaeological remains and cultural artefacts. In the case of stone, carbon and oxygen isotopes can indicate the quarry of origin. This presentation will focus on a case study examining the marble tombstone plaque (1833) from the grave of Thomas Jefferson, third president of the United States. Given the marble's grain size, the hypothesized quarry of origin for the plaque was either Carrara, Italy, or the northeastern United States. At the time marble was already imported to the US from the ancient quarries at Carrara, and marble quarries had been active in Vermont and Massachusetts since the late eighteenth century. Carbon and oxygen isotopes from the marble of Jefferson's plaque were compared to reference isotope datasets from Carrara and quarries in the US. Given the distinct carbon and oxygen isotope values observed in marble from these two regions, we are able to assign the most likely quarry of origin for this important historical object.

The use of VOC analysis in the care of modern materials

Katherine Curran[1], Mark Underhill[1], Lorraine Gibson[2], Matija Strlic[1]
1. Institute for Sustainable Heritage, University College London, UK
2. Department of Pure and Applied Chemistry, University of Strathclyde, UK

The analysis of volatile organic compounds (VOCs) as a diagnostic technique within medicine is a rich area of current research [1]. This work aims to assess the value of a similar approach within the conservation of modern materials and to explore the use of VOC emissions as a diagnostic tool for understanding the condition of modern heritage objects in collections [2, 3]. VOC emissions from over 200 plastic and rubber objects have been analysed using solid phase microextraction gas chromatography mass spectrometry (SPME-GCMS), including artificially and naturally aged objects. VOCs relevant to material degradation, e.g. volatile products of oxidation and hydrolysis, have been detected from many objects and the way in which detected levels of degradation products change following artificial degradation is shown. These results are compared to the levels of volatile degradation products detected from naturally aged objects and related to visible evidence of deterioration. Multivariate statistical analysis has been used to explore how combinations of VOCs can be used to distinguish between objects in different conditions.

1. Di Natale, C., et al., *Analytica Chimica Acta* 824 (2014) 1–17
2. Mitchell, G., Higgitt, C., Gibson, L.T., *Polymer Degradation and Stability* 107 (2014) 328–340
3. Curran, K., Strlic, M., *Studies in Conservation* 60 (2014) 1-14

GCMS method for calculating percent acetyl content in cellulose acetate

Joy Mazurek, Miriam Truffa Giacheta, Suzanna Etyemez, Michael Schilling
Getty Conservation Institute, Los Angeles, CA, USA

Cellulose diacetate (CDA) and cellulose triacetate (CTA) cels from animated feature films, dating from 1937 to 2003, in the collection of the Walt Disney Animation Research Library, were analysed by gas chromatography mass spectrometry to look for evidence of hydrolysis. Hydrolysis occurs in the cels with liberation of acetic acid, and a decline in acetyl values can be used as an indicator for degradation of the polymer. The cels contain plasticizers that must be removed prior to analysis. A method was developed to extract the plasticizer and isolate the polymer of the cels. Optimum results were obtained using a 1:1 mixture of hexane and absolute ethanol, since both CDA and CTA are insoluble in these solvents. The method developed to determine % acetyl uses pyrrolidine for aminolysis, converting the acetyl groups to acetylpyrrolidine. The acetyl content was calculated with reference to a linear calibration curve, forced through zero, derived from standard solutions of acetylpyrrolidine ranging from 1000 to 10,000 ppm in pyrrolidine. Data from the Disney animation cels show acetyl content values clustering in two groups: the first group is the CDA range (between 35 and 41%) and the second is the CTA range (between 43 and 45%). Results from other artworks are presented for discussion.

Identification of plant gums in artworks: a promising strategy based on enzymatic digestion and MALDI-TOF-MS

Clara Granzotto[1,2], Julie Arslanoglu[3], Caroline Tokarski[1]
1. Miniaturisation pour la Synthèse, l'Analyse et la Protéomique, Lille 1 University, France
2. Department of Molecular Sciences and Nanosystems, Ca' Foscari University, Venice, Italy
3. The Metropolitan Museum of Art, New York, USA

In the cultural heritage field, characterization of plant gums is usually based on the monosaccharide composition, as determined by gas chromatography mass spectrometry after complete acid hydrolysis. However, this technique might lead to misinterpretations. Therefore our research focused on the development of a strategy for plant gums identification involving partial enzymatic digestion of the gum polysaccharide component, followed by analysis of the released oligosaccharides by matrix-assisted laser desorption ionization mass spectrometry (MALDI-MS). Due to the different polysaccharide structures of the gums, the obtained MALDI mass spectra represent their unique profiles or fingerprints. The protocol was first optimized using standard polysaccharides which reproduce the potential gum structure (e.g. larch arabinogalactan has a 1,3-linked galactose backbone branched at C6 as gum arabic) and then applied to raw gum samples. The developed strategy allows the discrimination of gum arabic, tragacanth and cherry gums from guar and locust bean gums. Mass profile reproducibility was verified, and a unique enzyme cocktail, suitable for the digestion of all of the most common plant gums, was developed. Analysis of a watercolor sample from 1870, provided by The Metropolitan Museum of Art, revealed the mass profile of gum arabic, thus demonstrating the promising applicability of the method to aged materials.

Unlocking protein-binder/pigment interactions by coupling ELISA with MS techniques

Natalya Atlasevich[1], Caroline Tokarski[2], Julie Arslanoglu[3]
1. Previously at The Metropolitan Museum of Art, New York, USA
2. Miniaturisation pour la Synthèse, l'Analyse et la Protéomique, Lille 1 University, France
3. The Metropolitan Museum of Art, New York, USA

The recent application of immunological methods for the detection of specific proteins in egg, milk, and animal tissue-based binders has shown much promise as a complementary technique to more traditional (FTIR, GCMS) or sophisticated (MALDI, LC/MS/MS) approaches for paint analysis. Interactions between cations and proteins are well documented and the impact of pigment/ binder interactions and ageing have already been described in relation to common protein analysis techniques in cultural heritage. These phenomena would be expected to impact detection of proteins by enzyme-linked immunosorbent assay (ELISA) which depends on linear or conformational epitopes' antigen recognition. Thus, it is important to understand the influence of protein/pigment interactions on the molecular scale. Our research focuses on the use of both ELISA and mass spectrometric techniques to explore the influence of specific pigments on proteins. Our results suggest that specific pigments, when mixed with specific proteins, impact protein detection by ELISA. For example, our preliminary MS results suggest that casein peptides from verdigris paint show more distinct changes compared to casein from unpigmented samples in the peptide coverage. In contrast, casein digests from chalk show very few changes in the peptide coverage. Furthermore, we will present our first data using native protein analysis with/without pigments.

You can’t tell a book by its cover: analytical adventures in anthropodermic bibliopegy

Daniel P. Kirby[1], William S. Lane[2]
1. Peabody Museum of Archaeology and Ethnology, Harvard University, MA, USA
2. Microchemistry & Proteomics Analysis Facility, Harvard University, MA, USA

The practice of binding books in human skin, known as anthropodermic bibliopegy, is not in vogue so much nowadays, but was au courant well into the 19th century. A February 2006 article in The Harvard Crimson reported that no fewer than three such volumes were on the shelves of the Harvard University Library System. However, provenance supporting claims of human skin binding was sketchy and difficult to authenticate. For example, an inscription in one of the books declared: “the bynding of this booke is all that remains of my dear friende Jonas Wright, who was flayed alive by the Wavuma on the Fourth Day of August, 1632. King Mbesa did give me the book, it being one of poore Jonas chiefe possessions, together with ample of his skin to bynd it. Requiescat in pace.” Other volumes were under similar clouds of uncertainty. Except for inconclusive visual examination and unsuccessful DNA testing, no analyses had been done on Harvard’s or any of the supposed “human skin-bound” books, but the itch to learn the truth of the matter, one way or another, remained alive. This presentation will describe how the application of complementary analytical techniques – liquid chromatography tandem mass spectrometry, Fourier transform infrared spectroscopy, and peptide mass fingerprinting – finally set the record straight, with surprising results.

AMDIS & Excel: a powerful combination for evaluating Py-GCMS results from Asian and European lacquers

Michael Schilling[1], Henk van Keulen[2], Arlen Heginbotham[3], Mike Szelewski[4]
1. Getty Conservation Institute, Los Angeles, CA, USA
2. Netherlands Cultural Heritage Agency (RCE), Amsterdam, The Netherlands
3. J. Paul Getty Museum, Los Angeles, CA, USA
4. Winterthur Museum, Wilmington, DE, USA

Pyrolysis gas chromatography mass spectrometry using tetramethylammonium hydroxide for thermally-assisted hydrolysis and methylation (THM-Py-GCMS) is presently the most effective analytical technique for comprehensive characterization of organic materials in Asian and European lacquers. A daunting challenge, though, is systematically sorting through the vast number of chromatographic peaks in order to accurately locate marker compounds that are characteristic of lacquer raw materials. AMDIS (Automated Mass spectral Deconvolution and Identification System) software, developed by NIST, is capable of extracting and locating library marker compounds in highly complex GCMS data files. An AMDIS marker compound library containing over 450 compounds, typical for Asian and European lacquer, has been compiled from mass spectral data collected by the Netherlands Cultural Heritage Agency (European Lacquers) and the Getty Conservation Institute (Asian Lacquers). The resulting AMDIS search report is used in conjunction with a customized Excel workbook to semi-automatically analyse and interpret the Py-GCMS marker compound data. Using this system, it is possible to identify a wide range of Anacard tree saps, drying oils, fats, carbohydrates, proteins, resins, resinous diluents, preservatives and other materials present in samples from individual lacquer layers.

Natural resins in European lacquerware: a systematic Py-GCMS quality control

Louise Decq[1,2], Michael Schilling[3], Frederic Lynen[2], Wim Fremout[1], Vincent Cattersel[4], Delphine Steyaert[5], Charles Indekeu[4], Emile Van Binnebeke[5], Steven Saverwyns[1]

1. Royal Institute for Cultural Heritage (KIK-IRPA), Brussels, Belgium
2. Department of Organic Chemistry, Ghent University, Belgium
3. Getty Conservation Institute, Los Angeles, CA, USA
4. Faculty of Design Sciences, University of Antwerp, Belgium
5. Royal Museums of Art and History, Brussels, Belgium

The “European Lacquer in Context” (ELinC) project studies the technological history of European lacquers. These lacquers are mainly complex, layered combinations of various natural resins, including mastic, sandarac, shellac, elemi and copals. The project focuses on a number of japanned objects from Western Europe in the collections of the Royal Museums of Art and History in Belgium, aiming to improve the understanding of the materials used and the way they were applied. An important part of this study is the reproduction of lacquers following historical recipes. To assess the purity and labelling correctness of the commercially available natural resins to be used in this process, these resins were compared with trusted reference samples using pyrolysis gas chromatography mass spectrometry with thermally-assisted hydrolysis and methylation. The results showed that a significant portion of the commercial resins were impure or incorrectly labelled. To improve the efficiency of the quality-control process, a number of data processing techniques were used to avoid the need for manual identification of all individual compounds. The initial results of this ongoing project will be presented, with an emphasis on the quality control of the commercially available natural resins.

Binding media analysis of Florentine paintings of the 14th to 16th centuries: on the transition from tempera to oil technique

Ursula Baumer, Patrick Dietemann, Christoph Steuer, Daniela Karl
Doerner Institute, Munich, Germany

In an interdisciplinary research project, the materials and techniques of fourteenth to sixteenth century Florentine paintings of the Bavarian State painting collections are being studied. More than 80 works will be systematically investigated, including paintings by Bernardo Daddi, Fra Filippo Lippi, Sandro Botticelli, Andrea del Sarto and Leonardo da Vinci. An interesting case is the former high altar piece of Santa Maria Novella in Florence by Domenico Ghirlandaio, which was created 1491-94: one side panel and most parts of the centre panel were painted in tempera, but other areas in the centre and one side panel were executed in oil. However, the discrimination of tempera and oil by visual appearance was not reflected in the analyses of the binders. In all areas both egg protein and fatty acids from egg yolk and/or oil were found. In most oil paint samples, typical fatty acid profiles of drying oils were not as abundant as expected. On the other hand, egg tempera samples revealed surprisingly large amounts of fatty acids after solvent extraction. The talk will discuss the interpretation of these results and compare them with data from other paintings typical for tempera painting (Daddi, Lippi), or oil (Leonardo, del Sarto).

Interpreting analyses of paint samples containing both aqueous and oleoresinous binders

Patrick Dietemann, Wibke Neugebauer, Cedric Beil, Irene Fiedler, Ursula Baumer
Doerner Institute, Munich, Germany

German tempera paintings from around 1900 can exhibit complex build-ups of layers combining paints, glazes and intermediate varnishes in various ways. Because tempera paints may be absorbent, the binders of successive layers may penetrate into initial paints. For the identification of the initial binders of each layer, a constructive sampling is crucial. This is demonstrated by a case study of a painting by Arnold Böcklin, *Venus Genitrix* (1891-95, Kunsthaus Zürich). Some areas of the triptych were shown to have been painted with “cherry gum tempera” (“Kirschgummitempera”), probably containing egg as well as a mixture of oil and colophony, while other areas were painted in egg tempera with oil and colophony. Intermediate layers consisted of oil and colophony or of shellac with oil and colophony. A second case study of a copy after Giorgione by Franz von Lenbach (*The Concert*, 1865) discusses different ways of analysing egg-oil mixtures. Results of gas chromatography mass spectrometry and amino acid analysis are compared with Fourier transform infrared spectroscopic imaging and staining with SYPRO Ruby. As a result it was interpreted that Lenbach prepared his copy by applying alternating layers of tempera and oil paints. However, each analytical method gave differing results in different cases and only the combination of information from all methods was conclusive.

Novel application of LCMS for the characterization of lipid binders in art: elucidation of the composition of original paint materials used by Edvard Munch (1863-1944)

Jacopo La Nasa[1], Marco Zanaboni[1], Daniele Uldanck[1], Ilaria Degano[1], Francesca Modugno[1], Hartmut Kutzke[2], Biljana Topalova-Casadiego[3], Eva S. Tveit[3], Maria Perla Colombini[1,4]

1. Department of Chemistry and Industrial Chemistry, University of Pisa, Italy
2. Museum of Cultural History, University of Oslo, Norway
3. The Munch Museum, Oslo, Norway
4. Institute for the Conservation and Promotion of Cultural Heritage (CNR-ICVBC), Sesto Fiorentino, Italy

A multi-analytical approach based on mass spectrometry was used for the study of original paint materials from Munch's atelier, preserved at the Munch Museum in Oslo. The results obtained from the analysis of paint tubes were compared with a paint sample collected from one of the artist's sketches for the decoration of the Festival Hall of the University of Oslo (1909-1916). Gas chromatography mass spectrometry after hydrolysis and derivatization allowed us to determine the fatty acid profile of the paint materials, and to evaluate the molecular changes associated with curing and ageing. We characterized the triglycerides profile in the paint samples by liquid chromatography mass spectrometry (HPLC-ESI-Q-ToF) and identified oxidized species deriving from the drying processes. The combination of data obtained by these complementary techniques highlighted the differences between the binding media produced by different manufacturers, allowing us to identify the natural sources used to manufacture the paints, and giving us new insights on conservation issues. The obtained data will be exploited to better understand Munch's painting technique and will also help to address dating, authenticity and conservation issues by comparison with the results of the analysis of paint samples. The acquired information will be discussed in the frame of a wider project on modern artists' materials.

The influence of oil paint constituents and time on 20th century oil paints as studied by ESI-MS

Klaas Jan van den Berg[1], André van der Doel[1], Eliane van Dam[1], Birgit van Driel[1], Art Ness Proano Gabor[1], Aviva Burnstock[2].

1. Netherlands Cultural Heritage Agency (RCE), Amsterdam, The Netherlands
2. Department of Conservation & Technology, Courtauld Institute of Art, London, UK

Modern artists' oil paints are known to show degradation problems such as efflorescence and solvent sensitivity, along with other signs of ageing that relate to specific lipidic media as well as organic additives, extenders, driers and pigments. Electrospray ionisation mass spectrometry (ESI-MS) can give a good indication of the chemistry underlying these problems, by measuring the degrees of oxidation, hydrolysis and soap formation in the paints. This paper presents ESI-MS results from a set of naturally aged paints from Winsor & Newton, dating from 1947-1992, currently stored at Tate [1], and a set of recent paints produced in-house. Analyses were performed on paint extracts, showing the influence of driers, pigments and additives to the degree of hydrolysis and oxidation both on the surface and below. Interpretation of the results was greatly enhanced by combining positive and negative ion mass spectra (in combination with x-ray fluorescence data) using an ASCA chemometrics protocol which enabled assignment of measured differences between the paints to factors of age, composition and layer. This revealed essential information that would otherwise have been left unnoted.

1. http://www.tate.org.uk/about/projects/modern-oils-research-consortium

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Poster abstracts

Microscale radiocarbon dating of paintings

Laura Hendriks[1], Irka Hajdas[1], Markus Küffner[2], Cameron McIntyre[1], Nadim C. Scherrer[2,3], Ester S.B. Ferreira[2]

1. Laboratory of Ion Beam Physics, ETH Zürich, Switzerland
2. Swiss Institute for Art Research (SIK-ISEA), Zürich, Switzerland
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Clarification of the authenticity of artworks based on materials analysis, in the absence of a good knowledge of the artist's materials and technique, relies on the identification of so-called "dating materials". Unless there is a material anachronism, however, not much can be said of a painting's authenticity. Radiocarbon dating of materials in paintings provides a series of possible radiocarbon ages which, in combination with materials analysis, can be a very important piece of the puzzle of artwork authentication. Nowadays radiocarbon analysis typically requires a relatively large amount of canvas fibres (20 mg), thus its application as a routine dating method for artworks is rather limited. Nevertheless ^{14}C studies can provide reliable radiocarbon ages and have proved to be decisive arguments in establishing forgeries [1]. In this paper we investigate canvas sample size reduction for radiocarbon dating. Initial results showed that the required amount of carbon could be reduced to 100 µg using the conventional graphitization technique, and even smaller carbon sample sizes (in the order of tens of micrograms) were achieved by gas measurement on the gas ion source of the MICADAS (mini radiocarbon dating system [2]) at ETH Zürich. Additionally, a new approach was explored: the feasibility of dating the natural organic paint binder.

1. Caforio, L., et al., *European Physical Journal Plus* 129 (2014) article number 6.
2. Wacker, L., et al., *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* 294 (2013) 315-319.

Characterization of the archaeological mortars of the ancient Al-Saraya Al-Hamra Castle in Libya

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Al-Saraya al-Hamra, also known as Tripoli Castle, is situated in the city of Tripoli in the northwest of Libya. It was one of the great citadels of the Eastern Mediterranean and its earliest foundation dates back to the 2nd century AD. The aim of the present study is to characterize the mortar samples which were taken from different parts of the Al-Saraya al-Hamra Castle in order to understand the production technology of the mortars used. For this purpose, the physical properties such as density/unit weight, porosity, and water absorption by mass were determined. The content and ratio of the binder/aggregate parts, characteristic minerals, and general textural properties of the samples were investigated by loss on ignition, acid treatment, and petrographic analyses using stereo- and optical microscopy. The crystalline phases were identified by x-ray diffraction, the microstructure of the mortars were observed by scanning electron microscopy along with elemental analysis by energy dispersive spectroscopy, and the trace element composition was determined by inductively coupled plasma mass spectrometry. The results from this study are also being used to inform the restoration of the castle.

John Constable's drawing fixative: identification of a proteinaceous surface coating by peptide mass fingerprinting

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Although best known for his oil paintings, 19th century artist John Constable also created an extensive catalog of drawings from nature. Of these, a large number were fixed by the artist. What is exceptional is the unique appearance of the fixative—a clear, non-uniform coating with an iridescent sparkle. Attempts to identify the fixative by attenuated total reflection infrared spectroscopy were inconclusive but indicated the presence of proteinaceous material. This study applied a method of protein identification relatively new to conservation – peptide mass fingerprinting (PMF) – to identify Constable's fixative. PMF uses enzymatic digestion to cleave proteins, producing a mixture of peptides. The mixture is analyzed by matrix-assisted laser desorption ionization time-of-flight mass spectrometry, resulting in a spectrum of characteristic marker ions – the "peptide mass fingerprint". Markers are compared with those from reference materials for protein identification. Samples consisting of small surface fibers from four Constable drawings were analyzed and compared with data from reference materials. Constable's fixative was identified as fish-based, and other sources, such as gelatin, casein and ovalbumin, were ruled out. Correlating a specific protein source with the visual characteristics of a surface coating leads to a more sympathetic and informed conservation treatment. The effort Constable made to finish and preserve his drawings is indicative of the value he placed on them.

Not on your tintype? Py-GCMS analysis of materials present on historic tintype plates

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The tintype, a wet collodion photograph on a japanned metal support, was the most popular photographic technique in mid-19th century America. At least ten manufacturers produced tintype plates, consisting of metal sheets with the japanning layer on the recto and a varnish on the verso. Analysis by pyrolysis gas chromatography mass spectrometry of a study collection of over 220 tintypes indicates that drying oil is present in all of the japanning layers and backing varnishes, and that Pinaceae resin is present in 65% of the japanning layers and 99% of the backing varnishes. Shellac and sandarac are the only other natural resins detected, although camphor, a plasticizer, and asphaltum, a colorant, were found in a subset of layers. The co-occurrence of drying oil and Pinaceae allows the use of azeleic/palmitic acid ratios and index for the degree of oxidation (IDOX) values as probes of relative levels of oxidation of the japanning and backing varnish layers and previously analyzed artist-applied varnishes. While a subset of the collection displayed a linear correlation between IDOX values of the artist-applied varnish and the japanning layer, there were no clear correlations between the oxidation states of the japanning and backing varnish, or between the backing varnish and artist-applied varnish.

Evidence of lignin oxidation in archaeological waterlogged wood by MS-based techniques: the case of the Neolithic village of "La Marmotta", Italy

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Direct exposure mass spectrometry (DE-MS) and evolved gas analysis mass spectrometry (EGA-MS) were used to study 26 archaeological waterlogged oak wood samples, in order to investigate degradation patterns from a chemical point of view. The samples were taken from five piles from palafitte found in the Neolithic "La Marmotta" village (Bracciano lake, Italy). The sampling was done from the external to the internal part of the piles, following the annual growth rings five by five. Due to the complexity of DE-MS and EGA-MS results, principal component analysis was applied to the mass spectral data, highlighting differences among the samples. Our investigations were not only useful to study wood degradation in terms of differences between carbohydrates and lignin content, but also to show that lignin oxidation was the most important parameter to differentiate among the samples. In particular, aldehyde and ketone functionalities increased from the internal to the external part of all the piles. In addition, EGA-MS allowed us to obtain information on depolymerization of lignin. The combination of these two techniques proved to be a powerful tool to assess archaeological wood degradation with particular attention to lignin oxidation, allowing the time efficient analysis of a high number of samples.

Py-GCMS with in situ silylation for the analysis of treated composite archaeological wood artefacts: the ArCo Project

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The ArCo project Ageing Study of Treated Composite Archaeological Waterlogged Artefacts (Joint Programming Initiative on Cultural Heritage – Joint Heritage European Programme, en.natmus.dk/historical-knowledge/research/research-projects/arco) intends to develop and apply a characterization protocol to understand the decay of archaeological wood containing unstable salts and to investigate the interactions of applied consolidant/bulking agents with these salts. The purpose is to provide recommendations for the preventive conservation of treated wood artefacts. Samples from different archaeological finds contaminated by unstable salts were investigated using pyrolysis gas chromatography mass spectrometry with hexamethyldisilazane: alum-treated wood from the Oseberg collection (Norway); the Lyon (2nd century) and Saint Malo ships (France), post-treated with a solution of polyethylene glycol (PEG) and sodium sebacate; the Skuldelev and the Nydam ships (Denmark), treated with PEG. Particular degradation products were identified and their formation was attributed to different levels of acidity, metal ion content, and specific applied treatments. Chemical changes in the applied consolidating agents were noticed in some cases. Complementary techniques used in the project included FTIR and synchrotron radiation μFTIR mapping, SEM-EDX, elemental analysis by XRF and ICP-OES, and XRD. The results highlight different degradation pathways, influenced by the presence of alum and salts, in particular iron compounds for the Lyon and Saint Malo ships, and sulfates for the Oseberg collection. Further investigations will be performed after artificial ageing.

Tibetan wall paintings in Nako: behind the scenes of Buddhist artists' craftsmanship

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The goal of this project was a systematic documentation of wall paintings in four earthen Buddhist temples at the Nako monastery, located in the Indian part of the Western Himalayas. The research was not only meant to perform an assessment of the materials used in Tibetan art since the 12th century, but also aimed to facilitate the work of art historians and to guide treatments by conservators/restorers. The technical study was conducted by non-invasive methods in situ followed by non-destructive and destructive elemental, molecular and crystallographic analyses on micro-samples. The coupling of all methods and the results obtained delivered a relatively comprehensive picture of the painting techniques and materials of both the original and secondary wall paintings. In particular, this presentation will focus on results of gas chromatography mass spectrometry and liquid chromatography tandem mass spectrometry, which revealed the use of a proteinaceous binding material and indicated a possible animal source of the protein – tempera based on bovine (or similar animal) glue – used in original murals. In addition, the results indicated a plant gum, possibly in addition to a glucose-containing material. However, it is necessary to study more samples to provide more precise details, since Tibetans have been versatile masters in the use of paintings materials.

Study of painting materials of polychrome and monochrome colored pottery of the Eneolithic agrarian European cultures

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Study of the settlements of the agrarian Trypollian and Lublin-Volhynian cultures provides important insights into the early history of Europe. The Trypollian culture, which existed in the present-day southeastern European territories of Moldova, Romania, and Ukraine during the Eneolithic and Copper Ages, from approximately 5500 to 2750 BC, left behind hundreds of settlement ruins with archaeological artifacts. Pottery covered with polychrome and monochrome painting was one of the marks of this culture. Eneolithic Lublin-Volhynian culture, which occupied a historical region on northwestern Ukraine and southeastern Poland, coexisted with Trypollian culture. Ceramics covered with white monochrome ornament was characteristic of this culture. Depending on the neighborhood, the results of archaeological excavations have revealed that processes of production of the pottery were rather different. Kilns were used for the firing of pottery in Trypollya, whereas the roasting of ceramics in Lublin-Volhynya was carried out in special holes. Investigations by means of optical microscopy, scanning electron microscopy with energy dispersive spectroscopy, and gas chromatography mass spectrometry provided evidence that colors were applied after firing, allowed identification of both stratigraphy and composition of the studied paints, showed a presence of *engobe* on parts of the ceramics of Trypollya, and revealed differences in the composition of inorganic and organic paint components used in the Trypollian settlements Krinichki, Nemirov, Polivanov Yar, Vladimirovka, Nezvisko and in the Lublin-Volhynian settlement Vonvolnica.

Comparative study of paint materials in three outdoor Keith Haring works: Pisa, Melbourne and Paris murals

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Characterization of the paint materials in three of Keith Haring's outdoor murals, the Melbourne mural (1984), the Necker Hospital mural in Paris (1987) and Tuttomondo in Pisa (1989), was undertaken using pyrolysis gas chromatography mass spectrometry. Paint samples were collected during restoration campaigns and analyzed in order to identify the materials used by the artist, to evaluate the conservation state of the murals, and to provide conservators and restorers with information to support the choice of cleaning and protection procedures. The possibility to study the three murals allowed us to compare the painting techniques and to relate them to the state of conservation. The survey of the Melbourne mural highlighted a combination of paint materials deriving from different restoration interventions, including alkyds, vinyl and acrylic resins. In the case of the Paris mural, pyrolysis profiles of paint samples are characterized by the presence of benzene and acetic acid, together with small amounts of toluene, styrene, indene and other aromatic compounds. These results are indicative of a vinyl resin, and the occurrence of vinyl esters of different Versatic acid isomers was attributed to the commercial product VeoVa™. A styrene/n-butyl acrylate copolymer was identified as the paint binder for Tuttomondo.

Characterization of natural dyestuffs in plant extracts and historical textiles by GCMS

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Gas chromatography mass spectrometry (GCMS) applied to cultural heritage samples is particularly attractive due to the high separation efficiency, the MS detection sensitivity, the reproducibility of results and the possibility to identify in one step a wide range of analytes. In this work GCMS analysis was successfully applied to the characterization of anthraquinones, flavonoids, tannins and indigoids from plant extracts and historical samples. The analytical procedure is based on the silylating agent N,O-bis(trimethylsilyl)trifluoroacetamide with 1 % trimethylchlorosilane (BSTFA-TMCS) applied to pure chromophores and to plant extracts. The main marker molecules in each of the dyestuffs, as well as additional compounds, were successfully separated and identified by means of electron ionization mass spectra, thus demonstrating that a single GCMS procedure can be conveniently applied to the detection of the natural dyes. Other constituents of plant extracts, such as organic acids, oils and sugars, that hydrolyse during the extraction process, were also detected and recognized in the same chromatographic run. The GCMS method was tested on woollen references dyed according to traditional recipes and on samples collected from historical artefacts. The main dyestuffs used for colouring could be traced even with the low amounts usually available in archaeological samples. The proposed GCMS method is an efficient and fast analytical tool for the identification of natural dyestuff in plants and textile artefacts, providing results complementary to those from high performance liquid chromatography.

Identification of red dyes by DART-MS: examples from North and South American archaeological contexts

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To investigate the materials used as sources of red dye colorants in ancient objects, we applied direct analysis in real time time-of-flight mass spectrometry (DART-MS) to both reference materials and archaeological fibers. This new methodology has both advantages – analyses are rapid and require little or no sample preparation – and disadvantages (e.g. an inability to identify intact carminic acid) compared to existing ones used in the analysis of dye colorants. DART mass spectra were collected on chemical standards, botanical materials and comparative reference samples prepared with *Relbunium* and *Galium* species*, Sanguinaria canadensis* roots and *Dactylopius coccus* cochineal insects. We have applied DART-MS to three fragments shed from red and yellow textiles recovered from Seip Mound in southern Ohio (100 BC- 400 AD), and to red fibers obtained from several different textiles within two different funerary bundles recovered from the Paracas Necropolis in Peru (400 BC – 400 AD).The presence of anthraquinones consistent with the use of plant dyes in these textiles, and the absence of the bloodroot alkaloids and the anthraquinones found in cochineal insect dyes, supports previous studies suggesting the prehistoric use of *Galium* and *Relbunium* species as dye plants in Eastern North America and South America.

A multi-disciplinary approach to analysis of historical red lake pigment samples

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Interdisciplinary investigation of a group of historical red lakes and colorants from the Lefranc & Bourgeois Archive in Le Mans, France, has produced interesting results regarding their nature and significance in terms of new formulations for commercial organic dyes and pigments at the beginning of the 20th century. The materials were sampled for study as part of the Italian research project FUTURAHMA (www.futurahma.it), which unites art historians, conservators and scientists. We characterised a group of lake pigments found in 20 original jars labelled with inscriptions and dates from 1890 to 1921. The results of historical investigations were complemented by non-invasive multispectral imaging of the samples at various wavelengths. Invasive chemical and physical analysis followed: identification of colouring materials was carried out using high performance liquid chromatography (HPLC-DAD) and liquid chromatography mass spectrometry (HPLC-ESI-Q-ToF), Raman spectroscopy, time-resolved fluorescence spectroscopy and fluorescence lifetime imaging. Fourier transform infrared spectroscopy and x-ray fluorescence were also applied to assess the presence of inorganic salts used for lake production. Focus has been placed on discriminating among dyestuffs and lake pigments, and among their naturally-derived and synthetic versions. The most interesting results dealing with the interpretation of mass spectrometric data will be presented, along with relevant information acquired by complementary techniques.

MaSC

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