

MaSC 2019 Meeting Program

National Gallery of Canada
Ottawa, Ontario, Canada

6 –7 June, 2019

Preface

The Committee of the Users' Group for Mass Spectrometry and Chromatography (MaSC) welcomes you to the ninth MaSC Meeting at the National Gallery of Canada.

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 2019 Meeting, the first to be held in Canada, was preceded by a three-day workshop on the topic of sample preparation and derivatisation methods for several different GCMS applications. In the workshop, hosted by the Canadian Conservation Institute (CCI) and Parks Canada, pyrolysis of natural resins, dye analysis, and evolved gas analysis for the evaluation of museum exhibition and storage materials were performed using a Thermal Separation Probe (TSP), a novel and versatile sample introduction technique. We are immensely grateful to the workshop instructors, Jennifer Poulin (CCI) and Greg Smith (Indianapolis Museum of Art at Newfields), for sharing their expertise and discussing new research.

As in previous years, the 2019 Meeting has a diverse programme, with the specialities and research interests of MaSC members represented by sessions that highlight challenges and strategies for sample derivatisation and data interpretation in GCMS, and for the analysis of volatile organic compounds, among other topics. Applications will be presented of various chromatographic and MS techniques to inform conservation treatments and preservation strategies, and for the understanding of historical artists' techniques and industrial manufacturing processes, as illustrated by a wide range of objects and materials: from proteins to cellulosic materials, and from ancient Egyptian artefacts to modern paintings and plastics.

We would like to thank Jennifer Poulin and her colleagues at CCI and the National Gallery of Canada (NGC) – in particular Liliane Azzi (Program Coordinator, CCI), Stephanie Letemplier (Partnerships and Special Events Officer, NGC), Yves Lahaie (Special Events Coordinator, NGC), Stephen Gritt (Director of Conservation and Technical Research, NGC), and Michelle Robitaille (Chief, Partnerships and Community Engagement, NGC) – for their efforts in organising and hosting this event.

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

The MaSC Committee:

Ken Sutherland

Christopher Maines

Klaas Jan van den Berg

Ester Ferreira

David Pegg

Catherine Higgitt



**National
Gallery
of Canada**

**Musée
des beaux-arts
du Canada**



**Canadian
Conservation Institute**

**Institut canadien
de conservation**

Meeting schedule

MaSC 2019 Meeting Schedule

National Gallery of Canada

Thursday, June 6, 2019

9:00	Welcome, registration, coffee
9:30	Opening remarks
10:00	Workshop review
Session 1: Cellulosic materials , Chair Ester Ferreira	
10:30	<u>Na Yao</u> Characterization and identification of Chinese traditional handmade papers by using Py-GCMS
10:55	<u>Michael Schilling</u> Wood species identification using TD-GCMS
Poster lightning talks	
11:20	8 short poster presentations
12:00	lunch/poster break
Session 2: Volatiles & extractables , Chair Greg D. Smith	
1:30	<u>Anna Micheluz</u> Challenges in characterization of 3D cellulose nitrate objects by EGA-MS and TD/Py-GCMS
1:55	<u>Jacopo La Nasa</u> Selected ion flow tube-mass spectrometry (SIFT-MS) in heritage science: non-invasive VOCs profiling of natural and synthetic resins
2:20	<u>Jonas Veenhoven</u> GCMS identification of de-polymerized leachable molecules in organic solvent extracts from unaged and artificially light aged Asian lacquer films for evaluating cleaning procedures
2.45	coffee/poster break
Session 3: GCMS data interpretation strategies , Chair Christopher Maines	
3:35	<u>Louise Decq</u> European lacquer in context: strategies to find resin markers and application on historical objects
4:00	<u>Henk van Keulen</u> ESCAPE to reality: qualitative results quantified
4:25	<u>Václav Pitthard</u> Art of chromatography – chromatography in art
5:00 – 7:00	Reception at NGC

Friday, June 7, 2019

Session 4: GCMS derivatisation strategies, Chair Klaas Jan van den Berg

- 9:00 Eliise Tammekivi Quantitative analysis of binders in cultural heritage objects
- 9:25 Dario Durastanti Chilkat robe dye analysis by GCMS to identify historical dye sources
- 9:50 Kate Fulcher Approaches to the analysis of complex black liquids applied to ancient Egyptian coffins
- 10:15 coffee/poster break

Session 5: Protein analysis, Chair David Pegg

- 10:45 Tania Oudemans Blood or no blood? Identifying 2000-year-old protein remains on ancient ceramics using various MS techniques
- 11:10 Clara Granzotto Animal glue and beyond: palaeoproteomic analysis of paint binders and adhesives in ancient Egypt
- 11:35 Francesca Galluzzi Study of protein crosslinking in art paintings using high resolution mass spectrometry
- 12:00 lunch/poster break

Session 6: Oil and tempera paints, Chair Ken Sutherland

- 1:30 Patrick Dietemann Fluidising dripping oil paints on two paintings by Nikolaus Moser
- 1:55 Corina Rogge Go fish: the menhaden and 'Manhattan style' house paints
- 2:20 Klaas Jan van den Berg Mass spectrometric analysis of Royal Talens' oil and tempera paints 1920-1950
- 2:45 coffee

MaSC Business Meeting

- 3:15 ESCAPE follow up from Évora 2017 Workshop
- 3:30 MaSC business and discussion
- 4:30 CLOSE

MaSC 2019 poster presentations

“The Birdnester”: technological research on Peter Bruegel’s panel painting

Sabine Stanek, Václav Pitthard, Katharina Uhlir, Martina Griesser, Elke Oberthaler, Ingrid Hopfner and Georg Prast

Disclosing the complexity of triarylmethane formulations through the use of liquid chromatography and high resolution mass spectrometry

Francesca Sabatini, Ilaria Degano and Maria Perla Colombini

Comparative chemical investigations of alum treated archaeological wood from different museum collections

Jeannette Jacqueline Łucejko, Malin Sahlstedt, Caitlin M. A. McQueen, Francesca Modugno, Susan Braovac and Maria Perla Colombini

Analysis of archaeological pitch using GCMS and flow injection analysis-high resolution tandem mass spectrometry (FIA-ESI-Q-TOF)

Jacopo La Nasa, Federica Nardella, Marta Giani, Flora Silvano, Maria Perla Colombini and Erika Ribechini

Synthetic polymers in heritage objects: multi-pyrolysis based techniques to investigate polyurethane foams in 1960s sculptures

Jacopo La Nasa, Francesca Sabatini, Greta Biale, Ilaria Degano, Maria Perla Colombini and Francesca Modugno

Examination of archaeological Asian lacquered wooden objects by Py-GCMS

Kamilla B. Kalinina, Marina V. Michri, Marina V. Guruleva and Nikolay N. Nikolaev

Matrix-assisted laser desorption ionisation-mass spectrometry (MALDI-MS) for the characterization of plant gums in works of art: new developments and applications

Amra Aksamija, Clara Granzotto and Ken Sutherland

Coptic manuscripts at The Morgan Library & Museum: revealing the impact of conservation treatments by LCMS

Federica Pozzi, Elena Basso, Julie Arslanoglu, Francesca Galluzzi, Caroline Tokarski, Maria Fredericks and Frank Trujillo

Oral Presentation Abstracts

Characterization and identification of Chinese traditional handmade papers by using pyrolysis-gas chromatography/mass spectrometry

Na Yao^{1,2} and Shuya Wei^{1,2}

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² *University of Science and Technology Beijing, China*

Chinese traditional handmade papers were mainly made of plant fibers, including ramie, bark, bamboo, kozo and Langdu grass, etc. The common method for the identification of paper from different origins relies on the fiber morphology. This method requires the person who has extensive experience to achieve reliable results. In this paper, Py-GCMS was applied for the characterization of the Chinese papers. The results showed that this method could be used to differentiate ramie, bamboo, mulberry bark, kozo and Langdu paper from each other by their marker compounds. In addition, a great quantity of phytosterol compounds in Langdu paper revealed the reason why this paper possesses the property of preventing insects and mildews. Subsequently, the method was applied on the characterization of a book of ancient rubbing. The paper origin, ink and binding media used in the rubbing book could be determined, which provides scientific knowledge and support for the conservation of the book.

Wood species identification using thermal desorption-gas chromatography/mass spectrometry

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¹ *Getty Conservation Institute, Los Angeles, California, USA*

² *J. Paul Getty Museum, Los Angeles, California, USA*

In cultural heritage research, wood species identification provides information about materials and their trade routes, and guides conservators in the selection of suitable replacements for damaged pieces of wooden objects. However, not all species can be differentiated by wood anatomy, and it takes significant time and practice to develop the required expertise. An alternative to wood anatomy is chemotaxonomy, in which wood species are identified on the basis of compounds originating from secondary metabolites that are unique to each species.

A thermal desorption-GCMS procedure, employing F-Search for data interpretation, was developed for this purpose. The potential of the method was assessed by analyzing five wood samples taken from 17th – 19th century objects within the J. Paul Getty Museum collection and comparing the results to identifications made through wood anatomy. All of the samples were correctly identified through the combined use of the F-Search match quality and chromatogram comparisons, and marker compound identification. Through strategic partnerships with the Yale Institute for Cultural Heritage, the Winterthur Museum and the British Museum to analyze vouchered wood specimens, development of a shared F-Search wood library will benefit the cultural heritage and Convention on International Trade in Endangered Species (CITES) communities.

Challenges in characterization of 3D-cellulose nitrate objects by EGA-MS and TD/Py-GCMS

Anna Micheluz¹, Christina Elsässer^{1,2} and Marisa Pamplona¹

¹ *Conservation Science Department, Deutsches Museum, Munich, Germany*

² *Technical University of Munich, Munich, Germany*

In order to test the effectiveness or harmfulness of cold storage of 3-D cellulose nitrate (CN) objects, we conducted preliminary aging tests followed by EGA-MS and TD/Py-GCMS analyses to characterise the molecular degradation, with a particular focus on volatile components (i.e. plasticizers) of artificially and naturally aged specimens.

In the EGA-thermograms, the beginning and the duration of evaporation of the volatile compounds, mainly camphor and NO_x compounds, are specific for each condition state. In particular, an earlier release of volatile compounds and a second band originating from the pyrolyzed cellulose matrix were detected for the most aged CN specimens.

The CN components were identified by TD/Py-GCMS analysis, which highlighted an increase of fragments originating from camphor and cellulose for more aged specimens. Moreover, a semi-quantitative approach was applied for monitoring camphor. Its presence was detected in both TD and Py analyses and the comparison of its peak areas suggests a decreasing trend in the ratio from the fresh to severely aged specimens.

We obtained complementary results from the two analytical approaches, which confirmed their feasibility for the characterization of the degradation state of CN objects. Especially for the monitoring the loss of camphor, we plan to develop this promising approach further.

Selected ion flow tube-mass spectrometry (SIFT-MS) in heritage science: non-invasive VOCs profiling of natural and synthetic resins

Jacopo La Nasa, Francesca Modugno, Maria Perla Colombini and Ilaria Degano
SCIBEC, Department of Chemistry and Industrial Chemistry, University of Pisa, Italy

The identification of artistic materials and the study of their degradation pathways is crucial to define the best conservation strategies. At present, the identification at a molecular level of organic materials in heritage objects requires in most cases the collection of micro-samples followed by micro-destructive analysis, after specific sample pretreatments.

In the last years the analysis of volatile organic compounds (VOCs) released by organic materials in heritage objects has been investigated as an approach to achieve information on the composition and degradation of materials, and air quality in confined environments. Generally, solid phase micro extraction-GCMS has been used for the analysis of VOCs emitted from plastic materials and for studying the degradation of paper.

SIFT-MS is a direct mass spectrometric technique, recently introduced as a portable device, which achieves quantitative analysis of VOCs at trace levels in real time, by applying precisely controlled ultra-soft chemical ionization using eight different chemical ionization agents, and, importantly, avoiding any sampling step.

In this preliminary study, we explored the possibility to identify natural and synthetic resins used as paint varnishes by SIFT-MS on the basis of the VOCs released by the materials.

GCMS identification of de-polymerized leachable molecules in organic solvent extracts from unaged and artificially light aged Asian lacquer films for evaluating cleaning procedures

Jonas Veenhoven^{1,3,4}, Steven Saverwyns¹, Henk van Keulen², Maarten van Bommel³ and Frederic Lynen⁴

¹ *Royal Institute for Cultural Heritage (KIK/IRPA), Brussels, Belgium*

² *Cultural Heritage Agency of the Netherlands (RCE), Amsterdam, The Netherlands*

³ *Conservation and Restoration of Cultural Heritage Programme, University of Amsterdam, The Netherlands*

⁴ *Separation Science Group, Department of Organic and Macromolecular Chemistry, Ghent University, Belgium*

Durability, high gloss and extraordinary decorating techniques are, amongst others, unique qualities of Asian lacquers, which have since ancient times been applied on different substrates in Asian countries. This natural thermosetting polymer exudes as a liquid sap from trees within the Anacardiaceae family and consists of substituted catechols along with minor amounts of glycoproteins, polysaccharides and enzymes. Despite its exceptional qualities, Asian lacquer is one of the most light-sensitive coating materials, discoloring and becoming dull after long-term light irradiation. Its inertness towards any solvent, alkali or water drastically changes, caused by oxidation of the alkenyl sidechain of the substituted catechols, making the lacquer surface progressively more acidic and highly sensitive towards water and polar solvents, which complicates cleaning and other conservation related practices.

Our current research is focusing on de-polymerized leachable compounds, formed upon light aging of Asian lacquers, which can be extracted from the polymer matrix during solvent cleaning. This presentation proves the presence of these “free” de-polymerized solvent leachable molecules through carefully selected solvent extractions and dedicated sample preparation methods. Artificially light aged mock-up samples and organic solvent immersion procedures are used, combined with analysis using principally gas chromatographic separation and mass spectrometric identification.

European lacquer in context: strategies to find resin markers and application on historical objects

Louise Decq^{1,2}, Delphine Steyaert³, Wim Fremout¹, Vincent Cattersel⁴,
Emile Van Binnebeke³, Charles Indekeu⁴, Frederic Lynen² and Steven Saverwyns¹

¹ *Royal Institute for Cultural Heritage (KIK/IRPA), Brussels, Belgium*

² *Separation sciences group, Department of organic and macromolecular chemistry, Ghent University, Belgium*

³ *Royal Museums of Art and History, Brussels, Belgium*

⁴ *Conservation Studies, Faculty of Design Sciences, University of Antwerp, Belgium*

After four years, the “European Lacquer in Context” project (ELinC) has come to an end. The project gained chemical, art historical and technological insights on the production, the trade and the chemistry of lacquered objects from Western Europe. Central in the unravelling of the composition of European lacquers was Py-GCMS. Composed of mixtures of resins, oils, solvents and additives, chromatograms can feature a large variety of known and unknown compounds. Different strategies to find additional significant natural resin markers and to assist in a better interpretation of this large amount of Py-GCMS data will be discussed during this presentation.

In addition to the construction of a marker library generated by AMDIS, as presented in the previous MaSC meeting in Evora, statistical approaches were also investigated. A method normally used in metabolomics for generating biomarkers of interest for medicine and biotechnology was successfully tested to generate markers for copals.

The benefits of the multidisciplinary approach of the ELinC project will finally be illustrated with the discussion of the analysis of several lacquered objects.

ESCAPE to reality: qualitative results quantified

Henk van Keulen¹ and Michael Schilling²

¹ *Cultural Heritage Agency of the Netherlands (RCE), Amsterdam, The Netherlands*

² *Getty Conservation Institute, Los Angeles, California, USA*

ESCAPE, the Expert System for Characterization using AMDIS Plus Excel, combines an AMDIS identification with a customized Excel workbook. AMDIS (Automated Mass spectral Deconvolution and Identification System) software is proven to be capable of extracting and locating library marker compounds in highly complex (THM-Py-)GCMS data files. The ESCAPE AMDIS marker compound library has over 1400 entries representing artists' materials, such as resins, oils, proteins, carbohydrates, waxes, pigments and synthetic materials.

The customized Excel workbook, the ESCAPE report template, is developed to semi-automatically analyse and interpret the marker compound information in the AMDIS search report. ESCAPE only uses known and stable marker compounds for identification. As a result, the material content in the peak area reports is based on identified markers and not on the actual amount of a (aged) material. One approach to estimating the composition of samples before aging is the use of correction factors, calculation of fatty acid and resin ratios, compound subtraction in case of overlapping composition, correction of reduced THM-pyrolysis yield. The ESCAPE report template shows two pie charts; a 'peak area composition' and an 'estimated composition before aging'.

The applied peak area corrections are presented and explained.

Art of chromatography – chromatography in art

Václav Pittthard

Kunsthistorisches Museum Vienna, Conservation Science Department, Vienna, Austria

The prestige position of the Conservation Science Department in the Kunsthistorisches Museum Vienna has built up its reputation in supporting not only research on the museum's collections, but also world-wide. The department, with its research programs involving both organic and inorganic analyses of art objects, provides assistance with scientific information on e.g. the painting technique and alteration due to former restoration treatments, the development of new conservation treatments, or preventive conservation measures.

This lecture will specifically focus on the application of (pyrolysis)-GCMS for organic material analysis in the field of heritage science. A set of analytical procedures for the investigation of complex binding media compositions (lipids, resins, waxes, proteins, polysaccharides and Asian lacquers) has been optimised and applied on samples from numerous works of art.

In the frame of the lecture, examples of analyses of natural binding media mixtures from Old Master paintings, analyses of varnishes from Renaissance bronze sculptures or lacquer analyses from both European and Asian antique decorative objects will be highlighted. It will be pointed out that the ageing of these organic materials does play a crucial role and, therefore, the identification of such sensible complex natural polymers requires a search for markers, which are either stable during the ageing or are the products of the ageing processes.

Quantitative analysis of binders in cultural heritage objects

Eliise Tammekivi¹, Signe Vahur¹, Ott Kekisev¹, Inez D. van der Werf², Lauri Toom¹, Koit Herodes¹ and Ivo Leito¹

¹ *University of Tartu, Institute of Chemistry, Tartu, Estonia*

² *Cultural Heritage Agency of the Netherlands (RCE), Amsterdam, The Netherlands*

Quantitative analysis of binders used in cultural heritage objects is necessary to acquire information about the exact type of the used binder or to determine the different components of a binding mixture. It is complicated by the fact that the samples are small, they include many components and the analyte content may be very low. Therefore, the most effective procedures are required for this kind of analyses.

GCMS is a widely used method for the analysis of organic compounds in coating materials. In the case of oils, waxes and resins, this technique usually requires derivatization prior to the analysis. Various methods have been used for derivatization: however, there is almost no systematic comparison of these methods to each other. We present the comparison of four derivatization procedures (TMTFTH, acid-catalyzed methylation and two combined methods – NaOEt–BSTFA and KOH–BSTFA) applied to the analysis of oils. The comparison is based on the absolute quantification of the main five fatty acids in common oils and monitoring of the appearance of degradation products. Besides showing the advantages and drawbacks of the methods, the results indicate that TMTFTH is the least work-intensive and the most accurate derivatization procedure.

Chilkat robe dye analysis by GCMS to identify historical dye sources

Dario G. Durastanti¹, Ellen Carrlee² and Tami Lasseter Clare¹

¹ *Portland State University*

² *Alaska State Museum*

A partnership between the Alaska State Museum, Chilkat weavers, and a research lab at Portland State University has led to an investigation of the chemical identifiers of colourants used in Chilkat robes. These textiles, historically woven for ceremonial use by Northwest Coast indigenous cultures, have not been extensively analyzed regarding their dye materials and associated chemical composition. With the recent passing of two master weavers, the local community has proposed that we aid in accurately identifying and documenting the dye components used in the historical robes to aid in understanding old robes and inform the weavers' ongoing cultural practices. With samples from present-day weavers, our lab has analyzed several naturally dyed fibers with known origin to build a specific spectral database for the comparative analysis of historical Chilkat robes with unknown dye origins. Historically relevant commercial dyes are also included in this investigation. The analysis was done using GCMS for organic dyes extracted from sources such as wolf moss (yellow dyes) and hemlock bark (black dyes). X-ray fluorescence spectroscopy was utilized to detect metallic colourants such as those achieved using copper ammonia baths (turquoise dyes), and metallic mordants such as iron and copper.

Approaches to the analysis of complex black liquids applied to ancient Egyptian coffins

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¹ *British Museum, London, UK*

² *Institute of Archaeology, UCL*

Several 22nd Dynasty Egyptian coffins in the collections of the British Museum have been extensively painted with a black coating, and other examples show the application of a black liquid over cartonnage and coffins, presumably during funerary rituals. What are these black substances, where were they sourced from, and why were they applied? The ingredients of the liquids will have determined their material properties and contributed to their ritual significance.

The black substances have been analysed using GCMS in order to identify the components. Both the coating and the funerary liquid are a complex mixture of organic components including plant oil, resin, beeswax, and bitumen.

Samples that were large enough were treated using three preparation methods prior to GCMS: solvent extraction with DCM; de-asphalting samples and fractionating using solid phase separation; and pyrolysis with on-line silylation. The components that could be identified from the resulting chromatograms varied according to method, leading to questions as to how the sample preparation influences the results with implications for the analysis of these types of complex samples.

Blood or no blood? Identifying 2000-year-old protein remains on ancient ceramics using various MS techniques

Tania F.M. Oudemans¹ and Henk van Keulen²

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Archaeologists studying ceramics from the Netherlands and Belgium regularly see a specific thin, red-brown residue on the outside and inside of vessels from the Late Iron Age and Roman period. Many archaeologists have proposed these residues consist of blood. Combined analysis using Fourier transform infrared spectroscopy (ATR-FTIR) and direct temperature-resolved mass spectrometry (DTMS) has regularly shown the presence of a relatively well-preserved protein fraction. However, the identification of the exact protein origin is another matter. In this paper we present the results of a study using pyrolysis-GCMS with tetramethylammonium hydroxide (TMAH) for thermally assisted hydrolysis and methylation (THM-Py-GCMS) showing the presence of an albumin, possibly originating from blood.

Animal glue and beyond: palaeoproteomic analysis of paint binders and adhesives in ancient Egypt

Clara Granzotto¹, Rebecca Stacey², Neal Spencer², Mia Broné³, Sofia Häggman³, Diana Craig Patch⁴, Carl Heron² and Enrico Cappellini¹

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⁴ *The Metropolitan Museum of Art, New York, USA*

Protein-based materials have been mentioned in classical sources to be used as binding media and adhesives by ancient Egyptians, but a limited number of analytical studies have been conducted so far. Were certain proteins preferred for specific applications? Are there differences/similarities among similar objects from different periods and geographical areas? Can we discriminate original from modern proteins?

The aim of the research is to answer these questions by applying high-throughput tandem mass spectrometry-based protein sequencing to an extensive range of artifacts. This new and reliable approach, named palaeoproteomics, allows confident sequencing of ancient proteins and the study of their molecular damage, thus providing an analytical evidence of the proteinaceous materials used.

Micro-samples from ancient Egyptian painted coffins, cartonnages and mural paintings, dating 3000 BC – 600 AD, were investigated by palaeoproteomics. Animal glue from the genus *Bos* was identified in ground and paint layers, but more was discovered. Glue was prepared from both bones or soft tissues, it seems depending on the application, and other animal species were employed. In addition, specific plant seed proteins were unexpectedly identified in two completely unrelated objects. The results demonstrate how palaeoproteomics provides new evidence to advance understanding of the use of specific protein sources.

Study of protein crosslinking in paintings using high resolution mass spectrometry

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Proteinaceous substances implemented as binding media in tempera paintings are subject to various chemical modifications. Molecular alterations start immediately during the formulation and continue during the drying process and ageing.

The present study aims to investigate the mechanisms and products of protein crosslinking in egg tempera paintings using high-resolution mass spectrometry proteomics through a bottom-up approach. The impact of pigments (lead/zinc white) in the reticulation process during formulation, drying and ageing (natural and artificial) was examined. The methodology, developed on model paints formulated with lysozyme and lead white, succeeded in the identification of several intra- and/or inter-protein crosslinking such as Tyr-Tyr, His-Lys and Trp-Trp. Applied to very low sample amounts (few tens of μg), we have successfully identified crosslinkings in various historic paintings from the Metropolitan Museum of Art collection. For example, in the study of a tempera painting on wood by Lorenzo di Credi, Madonna Adoring the Child with the Infant Saint John the Baptist and an Angel (1490s), several crosslinkings were identified such as dityrosine (46NTDGSTDYGILQINSR61)₂ at m/z 1168.883 ($[\text{M}+3\text{H}]^{3+}$) and its characteristic fragment ions. Methodologies and results obtained will be discussed, pointing out the main differences in protein structures in function of the composition of paint formulations and their ageing.

Fluidising dripping oil paints on two paintings by Nikolaus Moser

Patrick Dietemann, Christoph Steuer and Ursula Baumer

Doerner Institut, Munich, Germany

Two paintings by Nikolaus Moser (b. 1956) were studied: “Feuer, Erde” (Fire, earth, 1992) and “Ohne Titel” (without title, 2000). Both paintings show areas with rather thick paints of various colours that seem to turn liquid again: the paints are soft and sticky, and liquid paint runs down the surface in many places. Three paints were studied, each was sampled twice for comparison: the soft and sticky but “solid” paint as well as the running, “liquid” paint drips.

GCMS analyses did not reveal fatty acids that are specific for non- or semi-drying oils (erucic or ricinoleic acids). However, a very large amount of oleic acid indicates such an oil nevertheless, presumably sunflower or safflower oil. P/S values varied considerably in many samples, thus they could not be used for identification of the oil. It is also unclear whether the degradation resulted in a lower or higher P/S values, because both phenomena were found. The same is true for the azelaic acid content, which also did not correlate with the degradation state of the samples. While in earlier models, a phase separation between the non-polar and the oxidised, polar fraction of the oil was proposed as the cause of the re-fluidising paints, this was not confirmed by the current study. Instead, a model that includes pigment-pigment interactions is proposed to explain the inconsistent data.

Go fish: the menhaden and ‘Manhattan style’ house paints

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² *The Getty Conservation Institute, Los Angeles, California, USA*

Throughout much of history, linseed, poppy and walnut were the oils commonly encountered in paint media. Industrialization of farming, exploitation of new crops and the invention of new media (i.e. alkyds) in the 20th century introduced more diverse vegetable oils into paints, including palm, castor and safflower. Fish oil was also industrially important in the United States, particularly that derived from menhaden (*Brevoortia* sp.). Once processed to remove offensive odors, this oil was recommended for use in linoleum, stoving paints, and inexpensive house paints. Although trade literature of the early and mid-20th century discusses the use of menhaden oils, paint manufacturers were understandably reluctant to advertise their use of this material, so its presence may be revealed only through chemical analysis. Recent GCMS studies on samples from paintings by Franz Kline (1910-1962) revealed unusual fatty acid profiles, including detectable levels of odd numbered fatty acids, indicative of an animal source, and high levels of myristic acid; comparison with authentic menhaden oil confirms these markers. These findings represent the first known identification of menhaden oil in artworks and introduce a new means of distinguishing between artist and industrial paints.

Mass spectrometric analysis of Royal Talens' oil and tempera paints 1920-1950

Klaas Jan van den Berg^{1,2}, Rika Pause¹, Brynn Sundberg², Inez van der Werf¹,
Art Ness Proano Gabor¹, Maarten van Bommel² and Alina Astefanei²

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Royal Talens is a prominent artists' paint manufacturer. Founded in 1899, Talens started producing oil paints almost immediately. The famous Rembrandt oil paints were introduced in 1904 and continue to be the best known range of paints. In addition, Talens produced many other types of paints including a variety of tempera paints.

Thanks to RCE's collaborative agreement with Royal Talens, new information has become available on formulations and applications. These formulations have been tested in historic paints from RCE's and Royal Talens' historical materials collections, using a range of techniques including GCMS, HPLC(MS) and surface acoustic wave nebulisation-MS (SAWN-MS).

In this paper, the mass spectrometric approaches used for the detection of the tempera mediums as well as synthetic organic pigments will be discussed.

Poster Abstracts

“The Birdnester”: technological research on Peter Bruegel’s panel painting

Sabine Stanek¹, Václav Pitthard¹, Katharina Uhler¹, Martina Griesser¹,
Elke Oberthaler², Ingrid Hopfner² and Georg Prast²

¹ *Kunsthistorisches Museum Vienna, Conservation Science Department, Vienna, Austria*

² *Kunsthistorisches Museum Vienna, Picture Gallery, Conservation Department, Vienna, Austria*

“The Birdnester” (59.5 x 68.3 cm, Inv. no. GG_1020) was painted on oak panel in 1568 and belongs to the three latest works of the twelve panel paintings by the Flemish master Peter Bruegel the Elder displayed at the Kunsthistorisches Museum Vienna (KHM). The analytical results shown in the poster are also representative for the other paintings investigated over last five years in the frame of the project dedicated to Bruegel. To gain some insights into Bruegel’s painting technique, different analytical methods were used in combination to answer the following questions: How did he prepare his grounds, and did he apply an imprimatura? How complex is the paint layer structure? What kind of pigments and binders did he use? The following methods were applied at the Conservation Science Department of the KHM: optical microscopy (OM) to characterise the paint layers by preparing cross-sections to get more details about the stratigraphy and by histochemical staining to localise binding media within the layer structure. Scanning electron microscopy (SEM) and X-ray fluorescence analysis (XRF) helped us to study the pigments, while the binding media and varnish compositions were identified by GCMS.

Disclosing the complexity of triarylmethane formulations through the use of liquid chromatography and high resolution mass spectrometry

Francesca Sabatini, Ilaria Degano and Maria Perla Colombini

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One of the earliest classes of synthetic dyes ever produced were the triarylmethanes (1859). Since then, they have become the most widely used synthetic dyes thanks to their versatility and their bright colors ranging from purple to blue/green hues.

The analysis of synthetic colorants is highly complex due to lack of reliable information on the formulations: different trade names, often fantastical and meaningless, were used to commercialize products constituted by the same compounds, and vice versa. Moreover, this class of colorants is amongst the poorest in terms of lightfastness. Another critical analytical issue is the complex composition of these formulations. Due to patent wars, several synthetic strategies were proposed for the production of analogue dyes with scarce attention to the purification steps.

This poster presents results obtained by the application of HPLC-DAD and HPLC-ESI-Q-ToF on reference triarylmethane formulations such as Fuchsine, Crystal/Methyl Violets, Methyl Blue and Diamond Green. Both ionization modes and different collision energies were tested to optimize the MS conditions for each dye. The detection of specific fragmentation patterns enabled distinction between different classes of compounds, while that of specific fragment ions distinguished isomeric species belonging to the same series.

The approach proved effective to separate and identify several miscellaneous components, major and minor, present in each formulation. The determination of the detailed composition of several fresh and artificially aged reference materials, dyed with triarylmethanes, was fundamental to identify the dye formulation and the conservation state of contemporary artworks.

Comparative chemical investigations of alum treated archaeological wood from different museum collections

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From the mid-1800s to the late 1950s, conservation by alum salts (potassium aluminium sulphate) was a popular method to prevent shrinkage and to impart strength to waterlogged wooden objects, mainly in Scandinavian and Baltic States. The original method consisted of immersing the wood fragments in a hot solution of alum. In 1911 George Rosenberg modified the formulation by including glycerol. In many cases the objects were coated after treatment with various types of oils, such as linseed oil, as well as melted beeswax and shellac or nitrocellulose varnishes. Today many of the objects treated with alum feature extreme deterioration and very low pH.

In the context of the “Saving Oseberg” project we investigated the extent of current chemical degradation in wooden objects conserved with alum salts. We compared samples taken from four different collections: the Dejbjerg collection (1883) at the National Museum of Denmark; the Oseberg collection (1905-13) at the Museum of Cultural History in Oslo; the Glimmingehus collection (1936) at the Swedish History Museum, and objects from the Colonial Williamsburg Foundation, Williamsburg, USA (1950s or 1960s).

The samples were treated using different recipes involving alum salts and other additives, such as linseed oil and/or glycerol. Analyses of lignocellulosic polymers and of inorganic compounds were undertaken. The investigations were performed using a multi analytical approach applying pH measurements, analytical pyrolysis (Py-GCMS), X-ray diffraction (XRD), scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS) and Fourier transform infrared spectroscopy (FTIR).

Analysis of archaeological pitch using gas chromatography/mass spectrometry (GCMS) and flow injection analysis-high resolution tandem mass spectrometry (FIA-ESI-Q-TOF)

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Archaeological excavations carried out by the Istituto Papirologico “Girolamo Vitelli” (Florence, Italy) between 2006 and 2007 in the northern necropolis of Antinoopolis (Egypt) brought to light a large number of glass and stone findings. Of particular interest were the monochrome and mosaic glass plaques and stone inlays dating to the 4th–5th century AD, which came from the parietal and floor decorations of the religious buildings and mortuary chapels of the northern necropolis.

A combined approach based on GCMS and flow injection-high resolution tandem mass spectrometry (FIA-ESI-Q-ToF) was used in the chemical characterization of the original adhesives used to fix monochrome and mosaic glass and stone plaques coming from archaeological site.

The GCMS analyses were performed after saponification, extraction and derivatization while the samples for the FIA analysis were extracted using a microwave-assisted approach in order to maximize the extraction yields. The use of this method allowed us to obtain a complete picture of the compositions of the adhesive, such as the presence of beeswax, drying oils, or animal fats.

Moreover, the use of FIA-ESI-Q-ToF experiments allowed us to identify the presence of several oligomers deriving from transformation process of the natural resin during the preparation of the pitch, and the presence of beeswax.

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Synthetic polymers in heritage objects: multi-pyrolysis based techniques to investigate polyurethane foams in 1960s sculptures

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Synthetic materials of new formulations have influenced contemporary artists since the beginning of the 20th century. Among the wide variety of synthetic polymers that can be encountered in 20th century art, the family of polyurethanes (PU) can be found in works of art as flexible and rigid foams, or in design furniture as upholstery foams. The limited amount of data about long-term stability of PU, along with the existence of a wide variety of possible types and compositions for these materials, make the preservation of historical PU foams in artworks a critical issue.

We evaluated the composition and the state of preservation of the PU foams constituting two Italian pop art sculptures both dated 1968: “Disgelo” by Piero Gilardi and “Contentitoreumano n.1” by Ico Parisi and Francesco Somaini. Evolved gas analysis/mass spectrometry (EGA-MS) and multi shot pyrolysis-GCMS were applied to define the chemical composition of the PU foams, paint binder and organic pigments. The results demonstrate the high potential of the adopted analytical approach in investigating plastic objects, achieving thermal and chemical information on different components in the same micro-sample.

Examination of archaeological Asian lacquered wooden objects by Py-GCMS

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Lacquered wooden archaeological objects found during excavations in Tuva and the foothills of the Altai were studied. Some of the excavated artifacts were classified as household items, while some were very damaged so their purpose was not clear. A stratigraphic investigation of cross-sections from the studied objects showed that a lacquer layer was laid on the ground, which was applied on a wooden base. Pigments and other inorganic materials of lacquer and ground layers were identified using a combination of polarized light microscopy and scanning electron microscopy. Py-GCMS was employed to identify the materials of the lacquered surface and ground by analyzing the individual layers. Markers of Urushi lacquer were detected in the black, red and ground layers indicated that the coating material was an Asian lacquer.

Matrix-assisted laser desorption ionisation-mass spectrometry (MALDI-MS) for the characterization of plant gums in works of art: new developments and applications

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Plant gums are widely encountered in cultural heritage materials (such as paint binders for manuscripts, watercolours). Their characterization is challenging: differentiation by chromatographic or spectroscopic techniques can be ambiguous, and generally only undersized, heterogeneous and complex samples are available for analysis.

Promising results have been obtained using a novel methodology based on partial enzymatic digestion of plant gums and analysis by MALDI-MS to generate characteristic oligosaccharide-mass profiles for the different gums [1]. The innovative method is applicable to small, complex samples from works of art, and has shown potential to discriminate gum sources at the species level, which opens new doors to addressing questions of geographic provenance and improving knowledge about ancient recipes and artist's materials [2].

This paper reports on the development and enhancement of the MALDI-MS strategy, including a systematic investigation of possible interference on gum identification by pigments and organic materials addition, and expansion of the reference database of MS profiles to include gums from various species of *Prunus* (fruit tree gums) and *Astragalus* (tragacanth), in addition to the more widely studied *Acacia* (gum arabic). The technique has been applied to historic reference materials and works of art in the collection of the Art Institute of Chicago.

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Coptic manuscripts at The Morgan Library & Museum: revealing the impact of conservation treatments by LCMS

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The collection of Coptic manuscripts at The Morgan Library & Museum consists of over fifty volumes that formed the library of the Monastery of St. Michael, whose existence was unknown until 1910, when the manuscripts were discovered in Egypt's Fayum Oasis. The volumes date from 822 to 914 A.D., are written on parchment, and include a range of religious texts. Shortly after being excavated at the bottom of a well, the manuscripts underwent extensive treatment at the Vatican Library. In particular, the presumed use of gelatin to repair the volume leaves has rendered them stiff and brittle, limiting their exhibition and use.

The present work constitutes the first in-depth technical study of the materials employed to create and repair this body of manuscripts. Analysis of the pigments, inks, and metallic elements by X-ray fluorescence and Raman spectroscopy was complemented by examination of the repair material by Fourier-transform infrared spectroscopy and LCMS. Results revealed that the latter was composed of collagen glue treated with formaldehyde. Notably, this is the first time that the chemical changes induced by formaldehyde treatment of collagen have been documented. These results demonstrate the power of mass spectrometry to address questions related to the impact of conservation treatments on artworks through the materials' molecular reactivity.

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