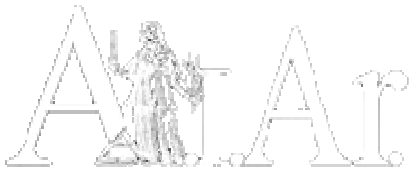
MaSC

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

MaSC 2013 Meeting Programme

Auditorium dell'Opera della
Primaziale Pisana,
Piazza Arcivescovado,
Pisa, Italy

5-6 June 2013



Preface

The MaSC Committee would like to welcome you to the sixth Meeting of the Users' Group for Mass Spectrometry and Chromatography (MaSC) at the University of Pisa.

After a lively and well-attended meeting at the Harvard museums in Cambridge, we are now back in Europe. The Workshop organized around this Meeting focuses on the analysis of organic colorants, lipids and proteins by LC and LCMS methods. We are very grateful to the instructors of the Workshop for sharing their expertise and recent developments with the participants: Ilaria Degano, Anna Lluveras Tenorio and Maarten van Bommel (analysis of natural organic pigments and dyestuffs), Francesca Modugno, Ilaria Degano and Jacopo La Nasa (analysis of lipid materials), and Leila Birolo and Eugenio Galano (analysis of proteins).

As always the Meeting has a diverse programme reflecting the wide range of research of MaSC members, covering a range of chromatographic and mass spectrometric techniques, classes of materials, and cultural objects. The sessions cover a range of topics including developments in analytical methodology and techniques, material characterisation, and material formulations and degradation studies. We trust there will be sufficient time for the exchange of ideas, during the poster sessions, at the end of every oral presentation and during breaks.

We would like to thank Agilent Technologies Italia for financial support, and the Italian Society of Archeometry A.I.Ar. for their support in the organisation of the Workshop and Meeting, and we would particularly like to thank our colleagues of the Group of Analytical Chemistry for the Conservation of Cultural Heritage (SCIBEC) for their support and assistance in organising and hosting these events.
We trust that you will enjoy the Meeting which takes place in the shadow of the *Torre*, leaning towards us and gently encouraging us to create a friendly and collegial atmosphere. In the meantime we hope you have some time to visit and enjoy Pisa and its cultural treasures.

The local organising Committee
Ilaria Bonaduce
Maria Perla Colombini

The MaSC Committee:
Klaas Jan van den Berg
Christopher Maines
Ken Sutherland
Ester Ferreira
Catherine Higgitt
David Peggie

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Meeting Schedule

Tuesday, 4 June

Please note – different location! *Department of Chemistry and Industrial Chemistry, University of Pisa, Pisa, via Risorgimento 35*

16.30 Early Registration

17.30 Nicola Cimino - Agilent Technologies Italia
Accurate Mass Platforms for Identification of Unknown Compounds: Data Mining and Structure Elucidation Strategies by LC and GC Q-TOF Systems

18.00 Close

Wednesday, 5 June

Auditorium of Opera della Primaziale Pisana, Piazza Arcivescovado

9.00 Registration

9.15 Klaas Jan van den Berg, Maria Perla Colombini, Ilaria Bonaduce
Welcome and Introduction

9.30 Maria Perla Colombini
New perspectives into chromatography mass spectrometry to study Cultural Heritage

Session 1: Analytical developments. Chair Ester Ferreira

10.05 Stepanka Kuckova, Irina Crina Anca Sandu, Michaela Crhova, Radovan Hynek, Igor Fogas, Vania Solange Muralha, Andrei Victor Sandu
Comparison of MALDI-TOF and nanoLC-ESI-Q-TOF mass spectrometry on identification of proteins in cross-sections of a medieval sculpture

10.30 Laura Cartechini, Silvia Castellini, Beatrice Moroni, Francesco Scardazza, Bartolomeo Sebastiani, Roberta Selvaggi,Roberto Pellegrino, Noemi Mancinelli, Brunetto Giovanni Brunetti, David Cappelletti.
Integrated analytical approach for indoor air quality monitoring of a museum environment by optical and chromatographic techniques

10.55 Coffee

Session 1 (cont'd): Analytical developments. Chair Ester Ferreira

11.25 Inez D. Van der Werf, Klaas Jan van den Berg, Cosima Damiana Calvano, Hannie Diependaal, Luigia Sabbatini
MALDI-TOF-MS and THM-GCMS analysis of a Karel Appel (1949) painting

11.50 Daniel P. Kirby, Ellen Promise
Species level identification of proteins in cultural heritage

12.15 **Poster pitch presentations (see page 8)**

12.45 Lunch and **Poster session part I**

Session 2: Material characterisation and degradation studies. Chair Ken Sutherland

14.30 Katherine Curran, Mark Underhill, Lorraine Gibson, Matija Strlic
Heritage Smells! Analysis of VOC emissions from historic plastics using SPME-GC/MS

14.55 Wim Fremout and Steven Saverwyns
Contemporary art materials studied by pyrolysis-GC/MS: selected case studies

15.20 Clara Granzotto, Caroline Tokarski, Christian Rolando, Carlo Barbante
Plant gums in artworks: study of glycoprotein content using adapted methodology based on size exclusion chromatography and MALDI-TOF mass spectrometry

15.45 Tea

Session 2 (cont'd): Material characterisation and degradation studies. Chair Ken Sutherland

16.15 Caroline Solazzo, Jolon M. Dyer, Stefan Clerens, Jeff Plowman, Matthew J. Collins
Proteomic studies of textiles

16.40 M. Virgolici, P. Selleri, E. Ribechini, M.P. Colombini, E.S. Teodor, A.V. Medvedovici, M.M. Manea, M. Cutrubinis
Establishing an archaeometrical method for discrimination of Romanian amber with chromatography and mass spectrometry

17.05 Sabina Rutkowska and Marianne Odlyha
MALDI-TOF-MS of dammar varnishes on exposure to acetic and formic acids

18.30 Buses leaving for Conference Dinner

Thursday, 6 June

Auditorium of Opera della Primaziale Pisana, Piazza Arcivescovado

Session 3: Understanding material formulations and technologies. Chair: Klaas Jan van den Berg

9.35 Patrick Dietemann
Correlating colloid properties of paints with analysis

9.50 Patrick Dietemann, Wibke Neugebauer, Irene Fiedler and Ursula Baumer
Tempera painting around 1900: comparing the composition of samples from paintings and commercial tube paints

10.15 Ursula Baumer, Patrick Dietemann, Irene Fiedler and Mark Peez
Binding media and original varnishes of late 13th century polychromy in the Cologne cathedral

10.40 Coffee

Session 3 (cont'd): Understanding material formulations and technologies. Chair: Chris Maines

11.10 Anna Lluveras-Tenorio, Leila Birolo, Eugenio Galano, Catharina Blaensdorf, Ilaria Bonaduce and Maria Perla Colombini
A first insight into the Asian clay sculptures painting technique and materials: combined use of GC/MS and proteomics on the Western and Eastern Buddhas of the Bamiyan valley (Afghanistan)

11.35 Nobuko Shibayama and Elisa Gagliardi Mangilli
Analysis of natural dyes used on 16 - 18th century Persian/Safavid and Indian/Mughal velvets by high performance liquid chromatography with photodiode array detector (HPLC-PDA)

12.00 Corina Rogge
The varnished truth: recipes and reality of tintype coatings

12.25 Lunch and **Poster session part II**

Session 3 (cont'd): Understanding material formulations and technologies. Chair: David Peggie

14.30 Sophie Tirat, Jean-Philippe Echard, Sylvie Gomes and Agnès Lattuati-Derieux, Jean-Yves Le Huerou and Stéphane Serfaty
New methods for molecular study of oil-resin varnishes

14.55 Václav Pitthard, Silvia Miklin-Kniefacz, Sabine Stanek and Martina Griesser
Asian lacquers on wooden decors, furniture, and decorative objects from European noble houses

15.20 Steven Saverwyns and Marc Vermeulen
The chemical characterisation of European lacquers from the collection of the Royal Museums for Art and History

15.45 Tea

16.30 Chair: Chris Maines
MaSC business meeting
Database discussions

17.30 Close

POSTERS

Pedro Caetano Alves, Maria Helena Florêncio and Jaap Boon
Triacylglyceride Profiling by LC-MS/MS in Oil for paint use: Progress and pitfalls

Laura Degani, Oscar Chiantore, Monica Gulmini, Daniela Gastaldi, Federica Dal Bello, Paola Iacomussi, Gabriele Piccablotto and Anna Pellegrino
HPLC evaluation of natural dyes degradation induced by white led illuminators

Ilaria Bonaduce, Leslie Carlyle, Maria Perla Colombini, Celia Duce, Carlo Ferrari, Erica Ribechini, Paola Selleri, Maria Rosaria Tiné
New insights into the chemical modification of linseed oil in paintings

Maria Perla Colombini, Celia Duce, Sibilla Orsini, Erika Ribechini and Maria Rosaria Tinè
Mass spectrometry (GC/MS) and thermal analysis for wood and bark pitch characterization

Annegret Fuhrmann, Johanna Lang and Christoph Herm
Investigations on historical wax objects and their surface phenomena

Jing Han and Anita Quye
The Historical and Chemical Investigation of Dyes in High Status Sixteenth Century to Eighteenth Century Chinese Costume and Textiles of the Ming and Qing Dynasties by UPLC

Laurens Thissen, Hadi Özbal, Ayla Türkekul Bıyık, Fokke Gerritsen and Rana Özbal
The land of milk? Approaching dietary preferences of Late Neolithic communities in NW Anatolia

Kamilla Kalinina and Ekaterina Dolbunova
Characterization of Lipid Material in Archaeological Vessels from Rakushechny Yar using GC-MS

Francesco Saliu, Jacopo La Nasa, Ilaria Degano, Francesca Modugno, Erika Ribechini and Maria Perla Colombini
Tag profiling by HPLC/MS for the identification of lipids in historical samples

Jacopo La Nasa, Sibilla Orsini, Ilaria Degano, Francesca Di Girolamo, Francesca Modugno, Ilaria Bonaduce and Maria Perla Colombini

Characterization of low molecular weight paint varnishes by analytical techniques based on mass spectrometry

Adele DeCruz and Alessia Androtti

The Restoration of a Roman Urn with the Erbium: YAG Laser. Science in the Service of Art

Shuya Wei, Valentina Pintus and Manfred Schreiner

Analytical Characterization of Synthetic Organic Materials in Contemporary Art Works and Study of Their Ageing Properties by Py/GC/MS, GC/MS and FTIR Spectroscopy

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Abstracts

Comparison of MALDI-TOF and nanoLC-ESI-Q-TOF mass spectrometry on identification of proteins in cross-sections of a medieval sculpture

Stepanka Kuckova[a,b], Irina Crina Anca Sandu[c], Michaela Crhova[a], Radovan Hynek[a], Igor Fogas[d], Vania Solange Muralha[e] and Andrei Victor Sandu[f,g]

[a] Department of Biochemistry and Microbiology, Institute of Chemical Technology, Prague, Czech Republic
[b] Department of Chemistry and Chemical Education, Charles University, Prague, Czech Republic
[c] REQUIMTE and Department of Conservation and Restoration, Faculty of Sciences and Technology, Nova University of Lisbon, Caparica, Portugal
d Moravian Gallery Brno, Czech Republic
[e] Research Unit VICARTE: Vidro e Cerâmicapara as Artes, Faculty of Sciences and Technology, NOVA University of Lisbon, Portugal
[f] Gheorghe Asachi Technical University of Iasi, Faculty of Materials Science and Engineering, Romania
[g] Romanian Inventors Forum, Iasi, Romania

The reliable identification and localization of proteinaceous binders is essential for proposing valid restoration and conservation treatments of painted or polychrome artworks. The challenge for analytical chemists and conservation scientists is the availability of such methods that are able to simultaneously identify individual binders in mixtures, to map the presence of the binders in multi-layered samples and to use a very small amount of sample from the studied art object.

This work proposes an innovative protocol of simultaneous mapping and identification of protein- and oil-based binders in cross-sections of paint materials using staining tests assisted by optical microscopy under visible and fluorescent light, MALDI–TOF and nanoLC-ESI-Q-TOF MS. The protocol was successfully applied on cross-sections of a medieval polychrome sculpture. In the presentation, the advantages and disadvantages of the two mass spectrometric techniques will be discussed.

Integrated analytical approach for indoor air quality monitoring of a museum environment by optical and chromatographic techniques

L. Cartechini[a], S. Castellini[b], B. Moroni[b], F. Scardazza[c], B. Sebastiani[d], R. Selvaggi[b], R. Pellegrino[b], N. Mancinelli[b], B.G. Brunetti[b,e] and D. Cappelletti[c,e]

[a] CNR-ISTM, UOS di Perugia, Italy
[b] Dipartimento di Chimica, Università degli Studi di Perugia, Perugia, Italy
[c] Dipartimento di Ingegneria Civile ed Ambientale Università degli Studi di Perugia, Perugia, Italy
[d] Dipartimento di Specialità Medico Chirurgiche, Università degli Studi di Perugia, Perugia, Italy
[e] Centro di Eccellenza SMAArt, Università degli Studi di Perugia, Perugia, Italy

Preliminary results of a seasonal monitoring campaign of indoor air quality in an Umbrian museum painting collection are presented. The study encompasses different analytical techniques to integrate thermo-hygrometric trends with airborne particulate matter characterization and gaseous pollutants determination. Information on number concentration and size distribution of aerosol and on the organic and inorganic content of particulate matter and gaseous pollutants were obtained using both on-line optical measurements and off-line chemical analysis, including chromatographic, SEM-EDS and ICP-AES techniques. The cross-correlated analytical data available from the integrated analytical approach has been crucial to estimate the level of pollution and its potential impact on the museum collection, to provide information on the sources of pollutants and to establish strategies to improve air quality.

MALDI-TOF-MS and THM-Py-GCMS analysis of a Karel Appel (1949) painting

Inez D. Van der Werf[a], Klaas Jan van den Berg[b], Cosima Damiana Calvano[a], Hannie Diependaal[c] and Luigia Sabbatini[a]

[a] Department of chemistry, University of Bari, Italy
[b] Cultural Heritage Agency of the Netherlands, Amsterdam
[c] University of Amsterdam, the Netherlands

"Man and Animals" (CoBrA painter Karel Appel) was created for the first *International exhibition of experimental art* in 1949 in the Stedelijk Museum (Amsterdam).

Three paint samples were taken for binding medium analysis. Data from Thermally assisted Hydrolysis Methylation (THM) - Py-GC/MS analysis point to an egg- or oil/egg-based binding medium, as testified by the presence of pyrolysis protein markers and by the azelaic/palmitic acid (A/P) values.
Lipid and protein fractions of the same paint samples were extracted and analyzed by MALDI-TOF-MS. Egg white, egg yolk and mammal collagen peptides were identified. Mass spectra relevant to the lipid fraction clearly indicate that oil and egg are present in different proportions. This result, in agreement with the A/P values, shows that MALDI-TOF-MS is very useful in providing precise information about the nature of binding media.

Species level identification of proteins in cultural heritage

Daniel P. Kirby[a] and Ellen Promise[b]

[a] Straus Center for Conservation, Harvard Art Museums
[b] Peabody Museum, Harvard University

Proteins are found in cultural heritage objects in many different forms: as paint binders, coatings and adhesives in artworks; as parchment and leather in books and documents; and in many components of native clothing, religious artifacts and decorative objects. Identification of the materials used in each of these contexts is an important part of an overall understanding of the artists' choices and techniques.

We have implemented and extensively used peptide mass fingerprinting (PMF) to identify proteinaceous materials at the species level from diverse objects of cultural heritage, such as a 7th century Coptic Codex, a 9th century Blue Qur'an, historic Alaskan kayaks, and a Pennsylvania parchment purse from the 1800s. In this presentation we will describe the analytical method in detail, show examples of current results, and discuss plans to extend PMF and species identification to new, important areas of cultural heritage.

Heritage Smells! Analysis of VOC emissions from historic plastics using SPME-GC/MS

Katherine Curran[a], Mark Underhill[a], Lorraine Gibson[b] and Matija Strlic[a]

[a] Centre for Sustainable Heritage, University College London, UK
[b] University of Strathclyde, UK

Although plastics present conservation problems within heritage collections, on account of their instability and rapid deterioration, there currently exists a relative lack of understanding of plastics degradation within the heritage sector (1). Volatile organic compound (VOC) analysis provides a potentially non-invasive method of understanding the degradation of plastic artefacts in collections.

Over 50 historic plastic objects composed of materials including cellulose acetate, cellulose nitrate, PVC, polyurethane and rubber have been degraded for 2, 4, 6, 8 and 10 weeks at 80 °C and 60% RH and their VOC emissions analysed using SPME-GC/MS at room temperature. This paper reports observed changes during degradation in material composition and in the levels of off-gassed degradation products and discusses how VOC analysis can be used to understand the chemical processes contributing to the degradation of a plastic artefact. The use of VOC analysis for material identification and to inform condition assessment and conservation practices will be discussed.

(1) Y. Shashoua, "Origin and development of conservation of plastics," in *Conservation of Plastics*, Oxford: Elsevier, 2008, pp. 11–15.

Contemporary art materials studied by pyrolysis-GC/MS: selected case studies

Wim Fremout and Steven Saverwyns

Royal Institute for Cultural Heritage (KIK/IRPA), Laboratories department - Painting laboratory, Brussels, Belgium

Modern and contemporary artistic movements have profited from the introduction of several innovative materials (pigments, binders and additives). These materials were seldomly used with a thorough knowledge of their long term durability or compatibility with other materials.

Several years ago the KIK/IRPA laboratory began a research program concerning the analytical study of the materials encountered in modern and contemporary works of art, with an initial focus on paints (and to a lesser extent plastics). Existing analytical methods and libraries have been adapted and refined in order to study these materials and to understand their degradation.

Py-GC/MS has emerged as the workhorse technique for the identification of paint binders, additives and polymeric materials. A double measurement protocol, both with and without thermochemolysis, and the use of AMDIS as an aid in the interpretation of the data, have proven effective to better understand the composition of modern paints and plastics, and the causes of their degradation. This will be illustrated by a number of case studies.

Plant gums in artworks: study of glycoprotein content using adapted methodology based on size exclusion chromatography and MALDI-TOF mass spectrometry.

C. Granzotto[a,b,c], C. Tokarski[b], C. Rolando[b] and C. Barbante[c]

[a] Department of Molecular Sciences and Nanosystems, Ca' Foscari University, Venice, Italy
[b] Miniaturisation pour l'Analyse, la Synthèse & la Protéomique (MSAP), USR CNRS 3290, and Protéomique, Modifications Post-traductionnelles et Glycobiologie, IFR 147, Université de Lille 1 Sciences et Technologie, France
[c] Institute for the Dynamics of Environmental Processes IDPA-CNR, Venice, Italy

Plant gums are complex proteoglycans which contain a small but significant amount of proteins. The aims of this research are to fractionate plant gums, evaluate their molecular weights and protein distributions and, identify glycoproteins using adapted strategies based on MALDI-TOF-MS. Raw samples of arabic, tragacanth, karaya, guar, ghatti, locust bean and fruit tree gums have been separated by high performance size exclusion chromatography coupled to a UV detector. The elution profiles of each gum, obtained by monitoring gum protein component at 214 and 280 nm, highlighted the presence of distinct fractions with MWs ranging between 2-3x10^6 and 2x10^3 Da. Three typical arabinogalactan, arabinogalactan-protein complex and glycoprotein-fractions of gum arabic were distinguished. Glycoprotein fraction was analyzed by proteomics. Results obtained so far have shown that plant gums have a characteristic MW distribution with different relative intensities due to differences in protein content. This methodology is currently being optimized for artwork investigation.

Proteomic approach to species identification and protein degradation in archaeological keratinous artefacts

Caroline Solazzo[a, b], Jolon M. Dyer[c], Stefan Clerens[c], Jeff Plowman[c] and Matthew J. Collins[a]

[a] BioArCh, Department of Archaeology, University of York, UK
[b] Smithsonian's Museum Conservation Institute, Suitland, MD
[c] Proteins and Biomaterials, AgResearch Lincoln Research Centre, New Zealand

In ancient and/or damaged artefacts containing keratinous materials, the species of origin of the materials can be difficult to identify through visual examination, therefore a minimally destructive methodology for species identification is required. While hair fibres from some species have seen substantial characterisation, others such as horn or baleen have received little or no attention, or lack protein sequences allowing formal identification using proteomics techniques. Using Peptide Mass Fingerprinting with MALDI-TOF-MS, we have catalogued and identified diagnostic peptide markers up to the genus level. A theoretical database generated by substituting amino acids in known keratin sequences allowed the characterisation of new peptides in species with little genetic information. However, understanding the mechanisms that lead to the loss or modification of diagnostic markers is essential for a reliable identification of artefacts.

We demonstrate here some of the modifications occurring at the amino acid level in wool specifically. Peptide markers with photo-oxidative damage and deamidation modification were identified using a combination of techniques (nanoLC separation, MALDI-TOF-MS and ESI-MS/MS), showing that progressive changes in the proteins can be used to assess degradation and ageing of textile fibres. Proteomic techniques can provide useful information to understand the nature of the materials and the factors that influence protein degradation, offering new methodologies to assess the condition and preservation of materials of cultural heritage.

Establishing an archaeometrical method for discrimination of Romanian amber with chromatography and mass spectrometry

M. Virgolici[a], P. Selleri[b], E. Ribechini[b], M.P. Colombini[b], E.S. Teodor[c], A.V. Medvedovici[d], M.M. Manea[a] and M. Cutrubinis[a]

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[b] University of Pisa, Dipartimento di Chimica e Chimica Industriale, Pisa, Italy
[c] Romanian National History Museum (MNIR), Department of Informatics, Bucharest, Romania
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The Romanian museums hold collections of thousands of adornment items, most of them recovered from funerary contexts. A special category of items is represented by amber objects, generally beads. There is a vast amount of literature regarding archaeological amber, as well as many speculations concerning its origin and commercial routes.

Documented trade routes since prehistoric times and the geological occurrence of fossil resins in the European region suggest that Romanian amber (also known as Rumanite or Romanite) and Baltic amber (also known as Succinite) are the most probable raw materials used for the manufacture of archaeological amber artifacts found on Romanian territory. The present project proposes the chemical characterization of fossil resins by means of a combination of pyrolysis, thermal desorption and GC/MS analysis for characterization of the macromolecular fraction of amber, ICP-MS for analysis of inorganic inclusions, and data treatment with multivariate analysis techniques to extract information related to the geological origin of the fossil resins.

Acknowledgement: This work was funded by the UEFISCDI, Human Resources Program, Postdoctoral research project PN-II-RU-PD-2011-3-0274, contract no. 99/2012.

MALDI-TOF- MS of triterpenoid varnishes after exposure to acetic and formic acids

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Preliminary investigations using MALDI-TOF-MS were made on triterpenoid resins in the framework of the MEMORI project (http://www.memori-project.eu/). One of the aims is to determine how acetic and formic acids affect triterpenoid varnishes and to evaluate threshold limits of damage. Some preliminary work on the effects of acetic acid has been reported (1) and this work continues within MEMORI. Fragmentation patterns of unaged samples showed previously reported peaks (2,3). Samples aged in organic acids were studied together with thermally aged samples. The effects of ageing showed as differences in peak distribution and peaks with higher m/z values. They are considered to be trimers, tetramers and pentamers of the polymeric fraction of dammar (2) and crosslinked dimerised triterpenoids or highly oxidised triterpenoids. It is the first time that the effect of formic acid on dammar varnish has been studied and it appears to act in a similar manner to acetic acid.

(1) Bonaduce,I. et al 2013. *Analyst 138* 487-500
(2) Scalarone, D. et al., *J. Mass Spectrom.* 2003, *38*, 607-617.
(3) Dietemann,P . et al., *Journal of Cultural Heritage* 2009, 10 30-40.

Tempera painting around 1900: comparing the composition of samples from paintings and commercial tube paints

Patrick Dietemann, Wibke Neugebauer, Irene Fiedler and Ursula Baumer

Doerner Institut, Munich, Germany

In the context of a large research project on German tempera painting around 1900, several paintings could be demonstrated to be painted with specific brands of commercial tube paints due to written evidence. These are one painting each by Franz von Stuck (1894, Syntonos paints), Fritz Overbeck and Paula Modersohn-Becker (both 1907, Wurm tempera). Wurm tempera was an important commercial tube paint produced by Richard Wurm in Munich. Syntonos was produced by the Munich painter Wilhelm Beckmann and became famous after a polemic controversy in the *Technische Mittelungen für Malerei*.

The composition of Syntonos is described in a patent (gum arabic with linseed oil and several additives, wax, tallow, green soap and glycerol). The Syntonos samples were compared with samples from original tubes from Whistler's paint box generously made available by the Library of Congress. The Syntonos samples showed to be largely in accordance with the composition described in the patent, but egg proteins as well as ricinoleic acid were found in addition.

The composition of Wurm tempera was always a secret. The samples of the Wurm paintings (both from 1906/07) showed a very similar composition and consist mainly of oil with only a trace of Karaya gum.

Binding media and original varnishes of late 13th century polychromy in the Cologne cathedral

Ursula Baumer[a], Patrick Dietemann[a], Irene Fiedler[a] and Mark Peez[b]

[a] Doerner Institut, Munich, Germany
[b] LVR-Amt für Denkmalpflege im Rheinland, Pulheim, Germany

Around the year 1290, over life-size stone statues of the 12 apostles, Christ and the Virgin were placed on the piers of the inner choir of the Cologne Cathedral. The original colourful polychromy of the statues was only overpainted once, in 1842. In the context of an ongoing conservation project, samples of nearly untouched 13th century paints and varnishes from these statues were analysed.

The paints contained linseed oil with small additions of resins (presumably pine resin) as well as proteins (egg). A recent edition of a manuscript from Montpellier (*Liber diversarum arcium,* c. 13th to 14th centuries) sheds more light on the occurrence of proteins in oil paints. Original varnishes of the 13th century are extremely rare and there are few previous examples where they have been identified. The composition of the varnishes as well as the paints will be discussed in the presentation and compared to late medieval panel painting on wood.

A first insight into the Asian clay sculptures painting technique and materials: combined use of GC/MS and proteomics on the Western and Eastern Buddhas of the Bamiyan valley (Afghanistan)

Anna Lluveras-Tenorio[a], Leila Birolo[b], Eugenio Galano[b], Catharina Blaensdorf[c], Ilaria Bonaduce[d], Maria Perla Colombini[d]

[a] Consorzio Interuniversitario Nazionale per la Scienza e Tecnologia dei Materiali, Firenze, Italy
[b] Dipartimento di Scienze Chimiche, Università di Napoli Federico II, Napoli, Italy
[c] Technische Universitaet Muenchen, Chair of Restoration, Munich, Germany
[d] Dipartimento di Chimica e Chimica Industriale, Università di Pisa, Italy

Painted clay sculptures are found in Buddhist sanctuaries all over Central and South-East Asia, but very little is known about the painting techniques used. This work describes the results obtained from the characterisation of the organic materials in paint samples from Western and Eastern Buddhas from the Bamiyan valley in Afghanistan.

An analytical procedure employing GC/MS was used for the characterization of lipids, waxes, proteins, and resinous materials in the same paint microsample. Results showed that saccharide and proteinaceous materials have been used as binders on clay sculptures. In order to establish the source of the proteinaceous binders, a database of materials, not commonly used as binders in Europe, was established. Moreover, Proteomics was applied to micro samples from the Buddha statues showing to be essential for the evaluation of the original materials used on the sculptures by the painters.

Analysis of natural dyes used on 16th - 18th century Persian/Safavid and Indian/Mughal velvets by high performance liquid chromatography with photodiode array detector (HPLC-PDA)

Nobuko Shibayama[a], Mark Wypyski[a] and Elisa Gagliardi Mangilli[b]

[a] Metropolitan Museum of Art, New York, NY, USA
[b] University of Udine, Palazzo Caiselli, Udine, Italy

Analyses of natural dyes used on 16th-18th century Persian/Safavid and Indian/Mughal period velvets in the textile collection of The Metropolitan Museum of Art were performed by HPLC-PDA. The purpose was to investigate whether or not the dyes might help differentiate velvets from each of the two cultures, along with other analyses and information including the metal of the metal-wrapping threads, weaving techniques, iconography, and historical background. This paper focuses on the results of dye analysis. There does appear to be a type of dye characteristic of one of the two cultures. There also may be a type of dye whose presence points to an exchange of dyeing materials and/or techniques between the cultures. Because of these factors, differentiation based only on a specific dye would not properly categorize the velvets. However, examination of the range of dyes used on the velvets does provide some clues to their origin. And, in addition, information gathered on the dyes, combined with knowledge gained of weaving techniques and metal threads, lead us to better understand and probably determine basic

The varnished truth: recipes and reality of tintype coatings

Corina Rogge

Department of Art Conservation RH 230, Buffalo State College, Buffalo, NY

The tintype was the most popular photographic format in the USA from 1856-1900, when millions of these objects were created. While the fundamentals for this photographic process were largely invariant, historical documents recommended a wide variety of varnish materials. To correlate the identity of varnishes used with recipes from the historical literature, a study collection of 221 tintypes was analyzed using Py-GC/MS, which revealed that while the resins detected were all recommended in the literature, only 24% of the combinations determined correspond to historical tintype varnish recipes. However, 44% of the tintypes have varnish constituents consistent with formulations recommended for ambrotypes or wet collodion negatives, sister techniques of the tintype. The remaining 32% have no direct literature equivalents, and nearly all of these images were varnished with mixtures of shellac and *Pinaceae*, relatively inexpensive resins, and thus this group may represent the 'patent' varnishes that were commercially available through photographic supply companies.

New methods for molecular study of oil-resin varnishes

Sophie Tirat[a,b,c], Jean-Philippe Echard[a], Sylvie Gomes[b], Agnès Lattuati-Derieux[b], Jean-Yves Le Huerou[c] and Stéphane Serfaty[c]

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During the 16th to 18th centuries, linseed oil and *Pinaceae* resin was probably the most commonly used mixture for varnishing musical instruments. To enhance knowledge of the composition and degradation of historical varnishes, we first performed a qualitative study of the molecular composition of model varnishes, using Py-GC/MS. We are now taking an approach towards quantitative analysis since, to our knowledge, such work has not yet been carried out.

Several micro-samples from model varnishes made with linseed oil and colophony in different proportions were prepared and analyzed using Selected Ion Monitoring (SIM) and full scan MS methods. From the qualitative data, we showed that the relative amount of oxidized compounds within colophony was linked to the initial oil/colophony proportions. In addition this approach using model mixtures allows us to assess the proportions of components within oil-colophony varnishes.

An approach towards quantification of a few markers compounds for oil and for colophony using Py-GC/MS-SIM has also been initiated. We will discuss to assess the potentials and limitations of this approach to measure markers compounds of oil and colophony.

Asian lacquers on wooden interiors furniture, and decorative objects from European aristocratic houses

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Collecting Asian artefacts has been fashionable in European aristocratic houses since the 16th century. Because European imitations and replicas were produced later, it is fundamental for assigning the provenance of these objects to accurately define not only the coating technique, but also the composition of the surface varnishes and lacquers. This paper gives an overview of results of a comprehensive, on-going research project, which includes scientific investigations on the coatings and the subsequent conservation of selected exceptional pieces of Asian interiors and richly decorated furniture. The objects studied include two lacquer cabinets from the collection of the Princes of Liechtenstein, Vienna; the Namban lacquer cabinet from the collection of the Kunsthistorisches Museum, Vienna; two Japanese lacquer cabinets from the Imperial Furniture Collection, Vienna; three lacquer boxes, a table and a cabinet from the Esterházy Castle collection, Eisenstadt, Austria; and a series of decorative wooden panels from a Chinese pavilion in Drottningholm Castle, Stockholm, Sweden. Investigations by means of optical and scanning electron microscopy and Py-GC/MS helped to identify both the stratigraphy and the composition of the studied coatings, and revealed that the objects of Asian origin were often restored using European materials. Furthermore, pieces of Asian artefacts were incorporated during production of the European imitations.

The chemical characterisation of European lacquers from the collection of the Royal Museums for Art and History

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European lacquerware has been produced since the 16th century in response to the high demand in the West for Oriental lacquers. European lacquers are essentially varnishes with complex composition, consisting of mixtures of natural resins and filling agents in varying amounts and applied in successive layers. The quest to imitate Oriental lacquer has resulted in an immense variety of recipes. In spite of, or maybe because of this vast number of recipes and variations in composition and technology, often little is known about the technical and chemical aspects of European lacquers in museum collections.
The characterisation of four pieces of furniture with European lacquerware from the collection of the Royal Museums for Art and History (Brussels, Belgium) formed the basis of a pilot study focussing on the identification of the chemical components of European lacquers. The principal analytical technique used was Py-GC/MS, with and without thermochemolysis, complemented by additional analyses on cross-sections with scanning electron microscopy, energy dispersive spectroscopy and Raman spectroscopy. Results of this study will be presented.

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Poster Abstracts

Triacylglyceride Profiling by LC-MS/MS in Oil for paint use: Progress and pitfalls

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The composition of vegetable oils is rather complex. Drying oils suitable for oil paints are a sub class of vegetable oils with a high concentration of linoleic and linolenic fatty acids. The interest is not only to determine the constituent triglycerides but also to find out how the oil composition changes as a result of cleanup treatments, heat treatments and oxidation/cross linking during drying. The analytical strategy chosen is mass spectrometric profiling – mostly direct ESI- MS- followed by target analytical techniques such as LC/MS and MS/MS techniques.

The poster will first present data on the composition of the oils after ESI-MS in positive and negative ion trap mode. The nominal mass spectra in the mass range of m/z 800 to 1000 will be tentatively identified. The LC/MS will at first be used as a profiling method using the mass chromatograms of the pseudomolecular ion peaks of the triacylglycerides to evaluate the number of isomers in the oils. This second profiling method should give therefore a rapid insight into the complexity and increased complexity due to treatments intended to modify the oils as known from literature from traditional paint making.

HPLC evaluation of natural dyes degradation induced by white led illuminators

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The colour degradation of natural dyes exposed to different white LED illuminators has been investigated in order to ascertain the validity and limits of such light sources for museum applications. Taffeta textile samples were dyed with some natural dyestuffs according to traditional ancient recipes. The textile samples were exposed at 10 Klx illuminance, under three different white LED lamps, with three different Correlated Colour Temperature (2600K, 2800K, 3900K) for about 7000 hours: the total lighting dose at the end of the ageing was 68 Mlx h.

The CIE L*a*b* coordinates of the samples have been monitored before and during the light aging period. After exposure, the colouring agents have been extracted and analysed by HPLC-DAD-MS. The decay of the different chromophores contained in each dyed textiles was determined and the results highlighted the different behaviour of the chromophores exposed to different spectral irradiance distribution of the LEDs.

New insights into the chemical modification of linseed oil in paintings

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A combined approach including chromatographic-mass spectrometry (GC/MS), direct exposure mass spectromety (DE/MS) and thermogravimetric analysis (TGA) was used to investigate paint reconstructions prepared with linseed oil that had undergone typical 19th century treatment prior to paint making. The oil was mechanically extracted from one seed lot and then processed by various methods: water washing, heat treatments, and the addition of driers, partly applying heat. Lead white and vine black were used as pigments.

The oxidation and hydrolysis state of the oil binder in paint replicas, as well as the formation of reaction products between the binder and the pigment were characterized.

Different pre-treatments induced various chemical modifications in linseed oil, but these differences were lost after the paint layer's curing and ageing process.

The study enabled us to quantitatively demonstrate that the parameters used to identify drying oils are deeply influenced by the paint's history.

Mass spectrometry (GC/MS) and thermal analysis for wood and bark pitch characterization

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Investigations into natural resinous materials are essential to identify the bio-molecular markers to be used for their identification in archaeological findings. A collection of more than 30 tar and pitch replicas was prepared from wood and barks of various deciduous trees (genus *Betula*) and conifers (genus *Pinus*) using a traditional preparation procedure.

Experiments were carried out on this collection by using an analytical procedure based on both GC/MS and differential scanning calorimetry (DSC). GC/MS, having been applied successfully previously in the characterization of organic residues from archaeological findings, was used here to identify a series of species acting as markers of botanical origin. DSC was used to study the thermal decomposition of selected pitch samples.

Acknowledgements: This work was supported by the Italian Ministry of University and Research (Project No. 2010329WPF_001). The tar and pitch collection was provided by A. Kurzweil, Berlin.

Investigations on historical wax objects and their surface phenomena

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The “Deutsche Hygiene-Museum Dresden“ owns a considerable collection of wax objects originating from an anatomical cabinet. The majority of these objects were produced by a Dresden wax modeler around 1900. They are embodiments of human anatomical and pathological phenomena.
In preparation of an intended presentation, the conservation status of this collection is being investigated and conservation is taking place. In this context it was possible to sample some models, concentrating on objects with peculiar surface phenomena.

The samples were analyzed by means of GC/MS and FTIR spectrometry, pigments were identified by PLM. Interpretation of the results was enabled by combination of these analytical methods.

The investigation results revealed that the surface phenomena such as blooming, exudations and films are components of the bulk wax, colour layers or later treatments of the surface (e.g. colour refreshment with body powder).
The poster will introduce selected objects and present analytical results.

The Historical and Chemical Investigation of Dyes in High Status Sixteenth Century to Eighteenth Century Chinese Costume and Textiles of the Ming and Qing Dynasties by UPLC

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In ancient China, colour was seen as a symbol of status, therefore dyeing played an important role in traditional handicraft. Until recently, historical Chinese dyes have rarely been analysed, let alone systematically studies from a chemical composition perspective. It is known that during the Ming and Qing dynasties, dyeing techniques such as over-dyeing and mordanting flourished, but there has been no specific study of the primary Chinese literature sources for the dyeing history or connections among dyes, dyeing activities and ideas and regulations of colours, especially red and yellow during this period.

This new PhD project aims to prepare natural yellow and red Chinese textile dyes from historical recipes and chemically characterise them using ultra high performance liquid chromatography (UHPLC) with photodiode array and appropriate mass spectrometric detection methods. The analysis of selected historical Chinese textiles from Glasgow Museums and other collections will then follow to reveal the truth of dyeing activities during the sixteenth to eighteenth centuries in ancient China.

The dyes of interest come from madder species, safflower, sappanwood, turmeric, pagoda tree bud, gardenia, amur corktree, smoketree, etc. These are mainly anthraquinone, flavonoid, neo-flavonoid and isoquinoline alkaloids.

Some results of dyed models, aged dyed models and dyes in historical textiles were obtained by HPLC in a previous project. This research begins with the evaluation and development of suitable extraction and analytical methodologies to analyse unknown dyes in historical Chinese textiles. Artificial ageing experiments will be undertaken to study changes in the chemical characteristics of dyes cause by fading during light exposure. Based on the above research, chemical profiling of reference dyes and aged reference dyes will be identified and an analytical database for the dyes will be established.

In the analysis of dyes in historical Chinese textiles, what dyes and dyeing techniques were used, how they changed over time and the relationship with colour will be studied.

The land of milk? Approaching dietary preferences of Late Neolithic communities in NW Anatolia

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Recent work by Richard Evershed and colleagues published in Nature in 2008 concerning residue analysis on ancient potsherds has provided clear evidence for milk processing and dairying in Anatolia and SE Europe in the Neolithic. Good results were acquired from ceramic samples taken from late 7th millennium cal BC sites in NW Anatolia. The investigation also suggestively linked the dominance of cattle in the bone assemblages of these sites to dairying. Building on this pioneering work, a new research project takes these primary results to the level of the pottery assemblages themselves. Integrating the residue analysis with ceramic studies, we regard residue analysis sampling specific vessel categories as an important step into assessing pottery function and meaning in prehistoric assemblages. This paper presents background and first results of the research, and will focus on the NW Anatolian key area seen by Evershed as favourable to Neolithic milk processing.

Characterization of Lipid Material in Archaeological Vessels from Rakushechny Yar using GC/MS

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The investigation of the chemical composition of organic residues associated with archaeological vessels is important for understanding the vital activity of populations in ancient periods of time. Analysis of the lipid components of such residues by GC/MS permits different kinds of plant oils and animal fats to be distinguished.

This report concerns the investigation of organic crust composition on a vessel dated to the early Neolithic period from the site 'Rakushechny Yar'. This site, which is situated in the south of Eastern Europe, on the River Don, is one of the most ancient early Neolithic sites of this region. Thick cultural stratification from early Neolithic to the Bronze Age has been traced here. Its lowest layers are dated to the VII mil calBC. Bones of wild and domesticated animals, as well as fish bones, were found in the lower layers. Finds of fishing net sinkers and objects specific to the flint industry indicate the significant role of fishing in the economy of these ancient people. Elaborate, complex ceramics are represented here with various pottery forms. The organic crust can be observed only on a few of the vessels, which can be explained either by the state of preservation or by their functional use. The organic material was taken from the inner part of the rim of an open vessel, about 26 cm in diameter, found in the lower layer, and was examined using GC/MS.

GC/MS analysis performed on the black crust revealed a series of fatty acids: high amounts of even carbon numbered saturated fatty acids with a small abundance of odd numbered fatty acids (C15 & C17). Apart from the predominance of the saturated homologous series, the GC/MS profile also revealed the presence of various positional isomers of monounsaturated C18 fatty acids, although the double bond positions have yet to be confirmed. The value of the P/S ratio was about 2.0–2.7. In addition, erucic acid was identified in the analyzed mixture at low abundance. Results from GC/MS analysis of the fats of different kinds of fish from this area show a presence of erucic acid in low abundance, depending on the kind of fish. A narrow P/S value (of 2.1–2.9) appeared to be characteristic of fish fats from sheat-fish or burbot. This supposition was confirmed by the presence of a rather great amount of sheat-fish bones on the site. Analysis of organic crusts from three pots dated to the middle-late Neolithic from the Dnepr-Dvina region was also made. Similar acids were traced here and may be evidence for their use in cooking fish.

Tag profiling by HPLC/MS for the identification of lipids in historical samples

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The identification of the source of lipid materials in archaeological or historical objects can yield an important contribution to the knowledge of past technologies and to the planning of conservation strategies. Oils and fats were used not only as food but also as paint media, illuminants or ingredients of cosmetics and medicines. A promising approach for the identification of their raw source is based on the determination of the overall triglycerides (TAGs) profile by HPLC/MS. We compared two detection systems for the analysis of the TAGs profiles: HPLC-APCI/MS and HPLC-ESI-Q-ToF-MS. In both instrumental assets positive-ion ionization was used, and the recognition of individual TAGs was based on the extracted ion chromatograms of the parent ion mass ($[M+H]^+$ for APCI, $[M+Na]^+$ for ESI) and of known fragments deriving from each TAG. We analyzed reference oils and fats, and organic residues found in XVI century historical ointments (Museo Aboca, San Sepolcro, Arezzo).

Characterization of low molecular weight paint varnishes by analytical techniques based on mass spectrometry

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Over the last few decades the number of synthetic paint materials used by artists and restorers has been increased and includes a variety of synthetic resins used as paint varnishes. Low molecular resins produce varnishes with similar properties with respect to natural varnishes, ensuring a good color saturation and gloss of the paint, and are easily removed without using polar solvents. We tested a multi analytical approach based on three different mass spectrometric techniques to characterize the molecular structure of three low molecular weight resins recently diffused as paint varnishes: the hydrocarbon resin Regalrez 1094, the aldehyde resin Laropal A81, and the ketone resin MS2A. To date, the molecular structures of these resins have not been completely elucidated. We combined the information obtained by GC/MS, Py-GC/MS, and electrospray ionization (ESI) Q-ToF-MS to implement the available knowledge of the chemical composition of these materials.

The Restoration of a Roman Urn with the Erbium: YAG Laser. Science in the Service of Art.

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A Roman Cinerary urn, 67-100 CE, was acquired by the St. Louis Art Museum in 1922 and never exhibited because of the intractable encrustation on the surface of the marble. The Roman urn was incrusted with an intractable layer of calcite that covered the decorative area of the body of the marble surface. None of the traditional conservation methods for cleaning the surface was effective in removing the dark crystal structure.

The marble urn was sent to the Duke University conservation laboratory for testing to remove the calcite without changing the marble substrate or disturbing the original polished surface of the marble. Tests were carried out with Er:YAG laser at 2.94 μm to determine whether the calcite could be removed by laser ablation.

Analytic testing demonstrated that the encrustation was a combination of organic materials, which over the millennia had transformed to oxalates. The Er:YAG laser removed the encrustation.

Analytical Characterization of Synthetic Organic Materials in Contemporary Art Works and Study of Their Ageing Properties by Py-GC/MS, GC/MS and FTIR Spectroscopy

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Specimens of polyvinyl acetates (PVAc), acrylics and alkyd paints used in the art field from different companies (Lascaux, Liquitex, Lukas, Rembrandt, Schmincke, and Winsor & Newton) were subjected to accelerated aging by a Xenon arc solar simulator. Unaged and aged specimen were analyzed by Py-GC/MS with double-shot/single-shot techniques, Thermally assisted Hydrolysis and Methylation (THM) GC/MS and GC/MS with derivatization using the reagent Meth-Prep II methods, as well as Fourier Transform Infrared Spectroscopy-Attenuated Total Reflectance (FTIR-ATR). After UV exposure, several alteration processes with consequent formation of volatile compounds or new products were observed by the techniques, especially the double-shot technique of Py-GC/MS. The various analytical techniques provided complementary and useful information for a better understanding of the drying and degradation properties of the synthetic organic materials (PVAc, alkyds, and acrylates) in contemporary art works.

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