

# **39<sup>th</sup> International Symposium on Archaeometry**



# **39<sup>th</sup> International Symposium on Archaeometry**

28 May - 1 June 2012  
Leuven, Belgium



Programme and Abstracts

## **Organizing Institutions**

KU Leuven  
Departement of Earth and Environmental Sciences  
Centre for Archaeological Sciences  
Divison Geology

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# Organization

## Standing Committee

### President:

M.S. Tite (Oxford)

### Chairman:

Y. Maniatis (Athens)

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## Organizing Committee

Patrick Degryse (KU Leuven) – chairman  
Dennis Braekmans (KU Leuven) – secretary  
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Koen Janssens (U.Antwerpen)  
Peter Vandenabeele (U.Gent)  
David Strivay (U.Lg.)

## Convenors

### Stone, Plaster and Pigments (Technology and Provenance)

Yannis Maniatis and Robert H. Tykot

### Archaeochronometry

Marco Martini

### Radiocarbon and Historical Chronologies

Christopher Ramsey

### Metals and Metallurgical Ceramics (Technology and Provenance)

Thilo Rehren

### Biomaterials and Bioarchaeology

Henk Kars

### Ceramics, Glazes, Glass and Vitreous Materials (Technology and Provenance)

Josefina Pérez-Arantegui and Michael Tite

### Remote Sensing, Geophysical Prospection and Field Archaeology

Luis Barba

### Human-Environment Interactions

Mark Pollard

### Colour and Culture

Patrick Degryse





## Introduction

Dear Colleagues,

Let me, on behalf of the local organizing committee, the standing committee and the organizing institutions, welcome you to Leuven for the 39<sup>th</sup> International Symposium on Archaeometry!

This edition of ISA celebrates 50 years of our meetings, and does so with celebratory events, special guests and several key note speakers looking back- and forward. Some 400 abstracts were submitted to ISA 2012, divided over 10 sessions.

Also, the opportunity was provided for 15 students to attend this conference through an ISA 2012 grant, kindly sponsored by the Comité de Gestion du Bulletin-Comité van Beheer van het Bulletin, the intermediate between the two Belgian chemical societies (Koninklijke Vlaamse Chemische Vereniging and Société Royale de Chimie) and ChemPubSoc.

Organizing this event would not have been possible without the support of our colleagues in the local organizing committee, the time of the conference secretary and the effort of the academic staff of the archaeometry laboratory of the division Geology and the Centre for Archaeological Sciences of the KU Leuven.

A particular word of gratitude is owed to the session convenors, handling all abstracts, and the administrative and IT units of the department of Earth and Environmental Sciences of the KU Leuven, handling all registrations and financial matters.

Also the support of our private sponsors is greatly acknowledged, as is the logistic, administrative and technical support of the technical services of the KU Leuven.

I wish you all an enjoyable and successful conference!

Patrick Degryse, Leuven, 2012



# 39<sup>th</sup> International Symposium on Archaeometry

Leuven, 28 May - 1 June, 2012

## PROGRAMME OUTLINE

### Sunday, 27 May

16:00 - 19:00 Registration desk open at conference venue

### Monday, 28 May

8:00 - 18:00 Registration desk open  
9:00 - 9:30 Opening ceremony  
9:30 - 10:55 **Session:** *Stone, Plaster and Pigments 1*  
10:55 - 11:25 *Coffee break*  
11:25 - 12:45 **Session:** *Stone, Plaster and Pigments 2*  
12:45 - 14:00 *Lunch*  
14:00 - 15:00 **Poster Session 1**  
15:00 - 16:05 **Session:** *Archaeochronometry*  
16:05 - 16:35 *Coffee break*  
16:35 - 18:00 **Session:** *Radiocarbon and Historical Chronologies*  
18:00 - 19:00 **Key Note** Professor Colin Renfrew (Lord Renfrew of Kaimsthorn)  
**Excavating Keros: The place of archaeometry in a holistic approach**  
19:00 Opening Reception

### Tuesday, 29 May

8:00 - 18:00 Registration desk open  
9:00 - 11:05 **Session:** *Metals and Metallurgical Ceramics 1*  
11:05 - 11:35 *Coffee break*  
11:35 - 12:55 **Session:** *Metals and Metallurgical Ceramics 2*  
12:55 - 14:00 *Lunch*  
14:00 - 15:00 **Poster Session 1**  
15:00 - 16:40 **Session:** *Metals and Metallurgical Ceramics 3*  
16:40 - 17:10 *Coffee break*  
17:10 - 17:55 **Key Note** Professor David Killick  
**Invention and Innovation**  
17:55 Presentations for the 2014 venue  
19:00 "50 years of ISA" reception

## Wednesday, 30 May

8:00 - 13:00	Registration desk open
8:30 - 10:15	<b>Session:</b> <i>Biomaterials and Bioarchaeology 1</i>
10:15 - 10:45	<i>Coffee break</i>
10:45 - 11:30	<b>Key Note</b> Professor Richard Evershed – <b>After the Revolution: Progress and Prospects for Archaeological Biomarkers</b>
11:30 - 12:30	<b>Session:</b> <i>Biomaterials and Bioarchaeology 2</i>
12:30	<i>Lunchbox</i>
14:00	Excursions

## Thursday, 31 May

8:00 - 18:00	Registration desk open
9:00 - 10:25	<b>Session:</b> <i>Ceramics 1</i>
10:25 - 10:55	<i>Coffee break</i>
10:55 - 12:35	<b>Session:</b> <i>Glazed Ceramics and Vitreous Materials</i>
12:35 - 14:00	<i>Lunch</i>
14:00 - 15:00	<b>Poster Session 2</b>
15:00 - 16:20	<b>Session:</b> <i>Glass 1</i>
16:20 - 16:50	<i>Coffee break</i>
16:50 - 17:50	<b>Session:</b> <i>Ceramics 2</i>
18:00	Open meeting of the Society for Archaeological Sciences
19:30	Conference Dinner

## Friday, 1 June

8:00 - 18:00	Registration desk open
9:00 - 10:05	<b>Session:</b> <i>Remote Sensing, Geophysical Prospection and Field Archaeology</i>
10:05 - 11:30	<b>Session:</b> <i>Human-Environment Interactions</i>
11:30 - 11:55	<i>Coffee break</i>
11:55 - 13:00	<b>Session:</b> <i>Colour and Culture 1</i>
13:00 - 14:00	<i>Lunch</i>
14:00 - 15:00	<b>Poster Session 2</b>
15:00 - 16:00	<b>Session:</b> <i>Colour and Culture 2</i>
16:00 - 17:20	<b>Session:</b> <i>Glass 2</i>
17:20	Prizes and Closing Ceremony

# 39<sup>th</sup> International Symposium on Archaeometry

Leuven, 28 May - 1 June, 2012

## DETAILED PROGRAMME

### Monday, 28 May

9:00 - 9:30 Opening ceremony

9:30 - 10:55 **Session:** *Stone, Plaster and Pigments 1* (Yannis Maniatis)

1. **Advantages and disadvantages of pXRF over other elemental methods of analysis for obsidian sourcing and studying ancient trade**  
Robert H. Tykot
2. **Petrological characterization of the Neolithic-Chalcolithic basalt extraction site of Giv'at Kipod (Galilee/Israel): A basis for provenance analyses of basaltic tools from Southern Levant**  
Tatjana Mirjam Gluhak and Danny Rosenberg
3. **Non-destructive study of the green stone artifacts of the tomb of the Maya ruler Pakal**  
José Luis Ruvalcaba-Sil, Mayra Manrique, Angélica García Bucio, Valentina Aguilar Melo, Edgar Casanova and Laura Filloy
4. **Using Pb and Sr isotopes to infer the source of turquoise at the Aztec Templo Mayor**  
Alyson M. Thibodeau, Leonardo López Luján, David J. Killick, John T. Chesley and Joaquin Ruiz

10:55 - 11:25 *Coffee break*

11:25 - 12:45 **Session:** *Stone, Plaster and Pigments 2* (Robert Tykot)

5. **Microarchaeology of floor and wall plasters from the Aceramic Neolithic site of Aşıklı Höyük, Turkey**  
Susan M. Mentzer and Jay Quade
6. **Characterization of plaster on stone and mud walls from ancient Pella**  
Safaa Abd El Salam, Yannis Maniatis and Ioannis Akamatis
7. **Geology, petrography, mineralogy, geochemistry of natural Fe-based pigments from Verona province (Italy)**  
Giovanni Cavallo and Roberto Zorzin
8. **A mineralogical, petrographic and C-O isotopic approach for the provenance determination of the raw materials from the Kastro-Palaea building constructions, in Volos of Thessaly, Central Greece**  
Markos Vaxevanopoulos, Vasilios Melfos, Evangelia Skafida and Artemis Karnava

12:45 - 14:00 *Lunch*

14:00 - 15:00 **Poster Session 1**

S1-S39: Stone, Plaster and Pigments

A1-A14: Archaeochronometry

R1-R5: Radiocarbon and Historical Chronologies

M1-M66: Metals and Metallurgical Ceramics

B1-B43: Biomaterials and Bioarchaeology

15:00 - 16:05 **Session:** *Archaeochronometry* (Marco Martini)

9. **Progress of archaeomagnetic dating in Western Europe: Examples from sites in Belgium**

Jozef Hus, Souad Ech-chakrouni and Simo Spassov

10. **New perspectives on Amino Acid Racemisation dating: Breaking the egg**

Beatrice Demarchi, Molly Crisp, Julia Lee-Thorp, Curtis Marean, Matthew Collins and Kirsty Penkman

11. **Rehydroxylation (RHX): A universal method for dating archaeological ceramics**

Moir A. Wilson, Andrea Hamilton and Catherine M. Batt

16:05 - 16:35 *Coffee break*

16:35 - 18:00 **Session:** *Radiocarbon and Historical Chronologies* (Christopher Ramsey)

12. **Bayesian modelling of an absolute chronology for the 18<sup>th</sup> Egyptian Dynasty**

Anita Quiles, Eric Aubourg, Emmanuelle Delqué-Količ, Geneviève Pierrat-Bonnefois, Guillemette Andreu-Lanoë and Christophe Moreau

13. **AMS radiocarbon dating of ancient iron alloys**

Stéphanie Leroy, Emmanuelle Delqué-Količ, Philippe Dillmann, Jean-Pascal Dumoulin and Christophe Moreau

14. **Quantitative approach to ancient diet reconstruction and reservoir effect correction**

Ricardo Fernandes, Bernard Meijlink, Olaf Nehlich, Andrew Millard, Marek Brabec, Pieter M. Grootes and Marie-Josée Nadeau

18:00 - 19:00 **Key Note** Professor Colin Renfrew (Lord Renfrew of Kaimsthorn)  
**Excavating Keros: The place of archaeometry in a holistic approach**

19:00 Opening reception

**Tuesday, 29 May**

9:00 - 11:05 **Session: *Metals and Metallurgical Ceramics 1*** (Thilo Rehren)

**15. The emergence of tin bronzes c. 7000 years ago**

Miljana Radivojević, Thilo Rehren, Duško Šljivar, Julka Kuzmanović-Cvetković, Marija Jovanović, Ernst Pernicka and Peter Northover

**16. Mineralogical and isotopic characterization of the Late Chalcolithic slags from Bressanone/Brixen (Northern Italy)**

Ivana Angelini, Filomena Gallo, Gilberto Artioli, Paolo Nimis, Umberto Tecchiati and Benno Baumgarten

**17. Arsenical copper in Southwest Asia: Alloying technology and intentionality determined by the chemical analysis of production remains**

Loïc Boscher, Thilo Rehren, Ernst Pernicka, Ulf-Dietrich Schoop and Barbara Helwing

**18. Lung-powered copper smelting of the Sican period (900-1375 CE), North Coastal Peru**

David Killick and Frances Hayashida

**19. When material science meet ceramic sociology: An archaeometallurgical study of crucibles from Mapungubwe, South Africa**

Shadreck Chirikure

**20. Why is there a Bronze Age from the Atlantic to the Pacific? Analysing the earliest appearance and widespread adoption of copper alloys and production techniques across Eurasia**

Benjamin Roberts

11:05 - 11:35 *Coffee break*

11:35 - 12:55 **Session: *Metals and Metallurgical Ceramics 2***

**21. The metallurgical inventory from Tell Chuera (Syria) – a direct comparison between quantitative pXRF and qualitative WDS data**

Kristina A. Franke

**22. Minoan metal vessel manufacture: Reconstructing techniques and technology with experimental archaeology**

Christina Clarke

**23. A preliminary report on the study of Cypriot bronzes dating to the Early Iron Age**

Andreas Charalambous, Vasiliki Kassianidou and George Papasavvas

**24. Oodles of copper for the Chinese Emperors: The case of Sijiawan plant**

David Larreina Garcia, Kunlong Cheng, Thilo Rehren and Yanxiang Li

12:55 - 14:00 *Lunch*

14:00 - 15:00 **Poster Session 1**

S1-S39: Stone, Plaster and Pigments

A1-A14: Archaeochronometry

R1-R5: Radiocarbon and Historical Chronologies

M1-M66: Metals and Metallurgical Ceramics

B1-B43: Biomaterials and Bioarchaeology

15:00 - 16:40 **Session: Metals and Metallurgical Ceramics 3**

**25. Making weapons for the Terracotta Army: Technology, standardization and logistics**

Marcos Martín-Torres, Xiuzhen Janice Li, Andrew Bevan, Yin Xia, Kun Zhao and Thilo Rehren

**26. A multidisciplinary investigation of bloomery iron smelting**

David Dungworth, Brice Girbal, Zoe Hazell, Paul Linford, Sarah Paynter and Harriet White

**27. Ore, slag and inclusion: Measuring variability in the direct process and assessing its implications for provenancing iron using the SI method**

Thomas Birch, Peter Crew, Zsolt Kasztovszky, Boglárka Maróti and Tim Mighall

**28. Identifying the construction phases of gothic cathedrals through their metallic supply using iron and lead analysis by LA-ICP-MS**

Maxime L'Héritier, Adrien Arles and Bernard Gratuze

**29. Quantitative 3-D mapping of Japanese ancient blades through Energy-Resolved Neutron Imaging**

Filomena Salvemini, Francesco Grazzi, Steven Peetermans, Francesco Civita, Riccardo Franci, Stefan Hartmann, Eberhard Lehmann and Marco Zoppi

16:40 - 17:10 *Coffee break*

17:10 - 17:55 **Key Note Professor David Killick  
Invention and Innovation**

17:55 Presentations for 2014 venue

19:00 "50 years of ISA" reception



## Wednesday, 30 May

8:30 - 10:15 **Session:** *Biomaterials and Bioarchaeology 1* (Henk Kars)

30. **Stable isotope analysis of multiple tissues from Peruvian mummified remains: Investigating tissue-spacing and dietary life histories**  
Lauren Cadwallader and Tamsin C. O'Connell
31. **Dental Calculus: A novel reservoir of health- related biomolecules**  
Christina Warinner, Enrico Cappellini, Kai Yik Teoh, Natallia Shved, Matthew Collins, Tom Gilbert and Frank Rühli
32. **Dairying in the early Mediterranean Neolithic**  
Cynthianne Debono Spiteri, Italo M. Muntoni and Oliver E. Craig
33. **Characterization of maize and manioc residues in prehistoric potteries: The contributions of GC and NIR spectroscopy to Archaeology**  
Fernanda Silva, Ingrid Weber, Fernando Hallwass, Cláudia Oliveira, Fernanda Pimentel and Simone Simões
34. **Extracting biogenic information from archaeological bone carbonate; The potential of density separation to assess the original composition**  
Ji Young Shin and Robert Hedges

10:15 - 10:45 *Coffee break*

10:45 - 11:30 **Key Note** Professor Richard Evershed –  
**After the Revolution: Progress and Prospects for Archaeological Biomarkers**

11:30 - 12:30 **Session:** *Biomaterials and Bioarchaeology 2*

35. **Paleodiet and human mobility in the oasis of Quillagua (northern Chile) during the Late Intermediate Period: A reconstruction through stable isotope analysis**  
Francisca Santana, Mark Hubbe and Mauricio Uribe
36. **Uneven distribution: Asymmetrical mobility patterns and shifting kinship structures in Neolithic and Early Bronze Age Cis-Baikal, Siberia**  
Ian Scharlotta and Andrzej Weber
37. **Applying multiple isotope analyses to the archaeological study of migration and mobility in the Caribbean**  
Jason Laffoon, Roberto Valcarcel and Corinne Hofman

12:30 *Lunchbox*

14:00 *Excursions*

## Thursday, 31 May

9:00 - 10:25 **Session:** *Ceramics 1* (Josefina Perez-Arantegui)

**38. Sr isotope analysis for the provenance study of ancient ceramics: An integrated approach**

Christina Makarona, Karin Nys and Philippe Claeys

**39. Ceramics and palatial power: The identification and characterization of a ceramic production installation in Late Bronze Age Attica through thin-section petrography**

William Gilstrap, Peter M. Day, Noemi Müller, Elina Kardamaki and Konstantina Kaza

**40. Amphora production in Hellenistic Rhodes – patterns, matter and performance**

Anno Hein, Vassilis Kilikoglou, Aggeliki Giannikouri, Fani Seroglou and Charikleia Palamida

**41. Archaeometry in Vesuvian Area: Technological features of thin-walled ware**

Lorena Carla Giannossa, Guiseppe Egidio De Benedetto, Rocco Laviano and Annarosa Mangone

10:25 - 10:55 *Coffee break*

10:55 - 12:35 **Session:** *Glazed Ceramics and Vitreous Materials*  
(Yona Waksman)

**42. Lead-tin glazed ceramics from Southern Italy: Evolution of production technology by archaeometric investigation**

Marianna Acquaviva, Lorena Carla Giannossa, Sabrina Loperfido, Rocco Laviano and Annarosa Mangone

**43. The pigments applied on the Minai wares and the correlation with Chinese blue-and-white porcelain**

Rui Wen and Mark Pollard

**44. Technical studies on medieval Islamic glazed tiles from Northern India**

Maninder Singh Gill and Thilo Rehren

**45. New data on the soda flux used in the production of Iznik glazes**

Michael Tite, Andrew Shortland, Nadine Schibille and Patrick Degryse

**46. High Mg-faiences from Fulda (Germany)**

Marino Maggetti, Gregor Stasch and Vincent Serneels

12:35 - 14:00 *Lunch*

14:00 - 15:00      **Poster Session 2**

V1-V106: Ceramics, Glazes, Glass and Vitreous Materials

G1-G11: Remote Sensing, Geophysical Prospection and Field Archaeology

H1-H15: Human-Environment Interactions

C1-C22: Colour and Culture

15:00 - 16:20      **Session: Glass 1** (Michael Tite)

47. **Technological change or consistency: Strontium isotope analysis of Egyptian faience beads dating from the Middle Kingdom to the New Kingdom at Abydos, Egypt**

Esme Hammerle, Jane Evans and Matthew Ponting

48. **Late Bronze Age glass from Nippur – a new cobalt colorant from the ancient Near East**

Marc Walton, Katherine Eremin, Andrew Shortland, Patrick Degryse and Susana Kirk

49. **Isotopes on the beach – Sr and Nd isotopic analysis for provenancing Roman glassmaking**

Dieter Brems, Monica Ganio, Kris Latruwe, Lieve Balcaen, Mike Carremans, Domingo Gimeno, Alberta Silvestri, Frank Vanhaecke, Philippe Muchez and Patrick Degryse

50. **Roman glass across the Empire: An elemental and isotopic characterization**

Monica Ganio, Sara Boyen, Frank Vanhaecke, Kris Latruwe and Patrick Degryse

16:20 - 16:50      Coffee break

16:50 - 17:50      **Session: Ceramics 2** (Marino Maggetti)

51. **Tracing changes in technology of Athenian Red-Figure Slips with  $\mu$ -XRF**

Marvin Cummings, Marc Walton, Giulia Poretti, Karen Trentelman, Jeff Maish and David Saunders

52. **Redefining Byzantine ceramics: 10 years of research at the “Laboratoire de Céramologie” in Lyon**

Yona Waksman

53. **Life goes on: Understanding the craft organization and economy of Early Postclassic Maya Lowlands through the analysis of Zakpah pottery from Marco Gonzalez, Ambergris Caye, Belize**

Carmen Ting, Marcos Martín-Torres and Elizabeth Graham

18:00      Open meeting of the Society for Archaeological Sciences

19:30      Conference dinner

## Friday, 1 June

9:00 - 10:05      **Session:** *Remote Sensing, Geophysical Prospection, Field Archaeology* (Luis Barba)

- 54. **Beyond the crucible: Field based methods for identifying primary metallurgy**  
Ryan Eldridge, Roger Doonan and Colin Merrony
- 55. **Automatic photogrammetric reconstruction: A new survey method for mining archaeology**  
Adrien Arles, Patrick Clerc, Florian Téreygeol and Jürgen Heckes
- 56. **New developments in wheeled multi-channel geomagnetic systems: Large-scale and high-resolution prospection with LEA D2**  
Cornelius Meyer, Henning Zöllner, Rudolf Kniess and Dana Pilz

10:05 - 11:30      **Session:** *Human-Environment Interactions* (Mark Pollard)

- 57. **Late Pleistocene to Holocene climate and seasonality in North Africa from the stable isotope analysis of marine and terrestrial mollusc shells (Haua Fteah, Libya)**  
Amy L. Prendergast, Rhiannon E. Stevens, Tamsin C. O'Connell, Graeme Barker and Christopher Hunt
- 58. **Human impact on soil formation and the change from natural to cultural landscapes in NW-Germany**  
Eileen Eckmeier, Peter Fischer, Alexandra Hilgers and Renate Gerlach
- 59. **3000 years of erosion and sedimentation in the Sagalassos territory: Man, climate and soil degradation**  
Bert Duser, Koen D'Haen, Johan Bakker, Gert Verstraeten, Bastiaan Notebaert and Marc Waelkens
- 60. **Spatial variations in sulfur isotope composition across Britain and its implication in archaeological provenance studies**  
Carolyn Chenery, Angela Lamb, Joseph Warham and Jane Evans

11:30 - 11:55      *Coffee break*

11:55 - 13:00      **Session:** *Colour and Culture 1* (Patrick Degryse)

- 61. **Red window glass in the Medieval period: Rediscovery of a lost technology**  
Jerzy J. Kunicki-Goldfinger, Ian C. Freestone, Iain McDonald, Jan A. Hobot and Andrew D. Smith
- 62. **Colour and the chemistry of alchemy in Hellenistic and Roman painting**  
Ioanna Kakoulli and Sarah Lepinski
- 63. **The colour of magic: Analysis in the investigation of ritual practice in Late Bronze Age glass**  
Andrew J. Shortland

13:00 - 14:00      *Lunch*

14:00 - 15:00      **Poster Session 2**

V1-V106: Ceramics, Glazes, Glass and Vitreous Materials

G1-G11: Remote Sensing, Geophysical Prospection and Field Archaeology

H1-H15: Human-Environment Interactions

C1-C22: Colour and Culture

15:00 - 16:00      **Session:** *Colour and Culture 2* (Dennis Braekmans)

**64. Comparing painting pigments and subjects: The cases of Maclear area (South Africa) and Metolong dam (Lesotho)**

Adelphine Bonneau, David G. Pearce and Daniel Arsenault

**65. Late Mousterian red pigment proceeding in Les Bossats, Seine-et-Marne (France)**

Hélène Salomon, Pierre Bodu and Stéphanie Geurten

**66. Pigments and tiles from the master potter Ali Muhammad Isfahani: The production of decorative underglaze painted tiles in the cultural context of 19<sup>th</sup> century Iran**

Ina Reiche, Lore Troalen, Jim Tate, Stefan Röhrs, Hélène Rousselière, Boris Pretzel, Bhavesh Shah, Graham Martin and Friederike Voigt

16:00 - 17:20      **Session:** *Glass 2* (Andrew Shortland)

**67. Trace Element Analysis of Islamic Glasses from Egypt, Syria and Israel: Evidence for Provenancing and Mixing of Ancient Glasses**

Julian Henderson, Simon Chenery, Edward Faber and Sophie Bertier

**68. Elemental & isotopic analyses of Roman, Vandal and Byzantine glass from the Bir Messaouda site at Carthage, Tunisia**

Thomas R. Fenn, Patrick Degryse, Andrew J. Shortland and Roald Docter

**69. Byzantine glass bracelets excavated on Romanian territory investigated using external IBA methods**

Roxana Bugoi, Ingrid Poll, Gheorghe Mănuclu-Adameşteanu, Thomas Calligaro, Claire Pacheco and Laurent Pichon

**70. Swabian and Mamluk gilded and enameled glass objects: Technological affinity through archaeometric investigation**

Maria Cristina Caggiani, Rocco Laviano, Nicoletta Ditaranto, Lorena Carla Giannossa, Annarosa Mangone and Philippe Colombari

17:20 Prizes and Closing ceremony



## Poster Sessions Summary

### Poster Session 1

S1-S39: Stone, Plaster and Pigments

A1-A14: Archaeochronometry

R1-R5: Radiocarbon and Historical Chronologies

M1-M66: Metals and Metallurgical Ceramics

B1-B43: Biomaterials and Bioarchaeology

### Stone, Plaster and Pigments

- S1 **Physical and Mechanical Properties of Qusayr Amra Building Materials in Jordan: Towards Characterization and Documentation**  
Firas Alawneh, Fadi Balaawi and Yahya Alshawabkeh
- S2 **Technology and Pathology of Previous Restorations of Imam Mosque's Dome in Isfahan, Iran**  
Hesam Aslani and Zohre Motalebi
- S3 **Material Characterization and Technology of Stucco Decorations of Jurjir Façade (10<sup>th</sup> Century A.D) in Isfahan, Iran**  
Hesam Aslani and Vale Vafaei
- S4 **Scientific Approach of Materials and Pigments in Some of Painting in Third Millennium BC in Iran**  
Roya Bahadori and Faranak Bahrololoomi
- S5 **Identification of Pigments in Two Iranian Historical reverse Glass Painting**  
Roya Bahadori and Atieh Mozafari
- S6 **Case Studies on a Non-Destructive SEM-EDX Analytical Method for Polished Stone Tools and Gems**  
Zsolt Bendő, István Oláh, Bálint Péterdi and Eszter Horváth
- S7 **Danube: The Big Prehistoric Conveyor Belt**  
Katalin T. Biró, Sándor Józsa, Katalin J. Szabó and Zsuzsanna M. Virág
- S8 **Features of Roman Ceiling Plasters (*Cremona* - Italy)**  
Roberto Bugini, Luisa Folli, Elena Mariani and Lynn Pitcher
- S9 **Geomineralogy and Geochemistry of the Sandstone Used at the *San Giovanni Battista* Baptistery in Oggiono - Italy**  
Giovanni Cavallo, Guido Corredig and Elisa Anna Figus
- S10 **Petrographic Evidence of Intercultural Trade During the Chalco-lithic. Examples from Neamț County, Romania**  
Otis Crandell and Vasile Diaconu
- S11 **Geological origin of ochre pieces from the Middle Stone Age site of Diepkloof Rock Shelter (South Africa): reconstruction of supply strategies**  
Laure Dayet, François-Xavier Lebourdonnec, Floréal Daniel, Pierre-Jean Texier and Guillaume Porraz
- S12 **Archaeometry of Pre-European Hand Millstones Quarries in Gran Canaria. Applications for the Case of the Lapilli Tuff Instruments**  
Miguel del-Pino-Curbelo, Amelia Rodríguez-Rodríguez, Jaume Buxeda-i-Garrigós, Luis Hernández-Gutiérrez, Rafael Fort and José Mangas

- S13 **Archaeometric Evidence of Trade of Leucite-Bearing Volcanic-Made Roman Rotary Millstones of Pompeian Style in Hispania (Spain)**  
Domingo Gimeno, Meritxell Aulinas, Guillem Gisbert, Montserrat Pugés and Daniela Novembre
- S14 **Rare Earth Elements and Sr-Isotopic Geochemistry: Tools to Determinate the Geological and Geographical Origins of Neolithic Fluorites**  
Johan Honings, Eric Goemaere, Françoise Bostyn, Cécile Monchablon, Emmanuelle Leroy-Langelin, Patrick Degryse, Mark Golitko, Hélène Collet and Ivan Jadin
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Rita Giannini, Jeffrey Maish, Simon Garrett and Marc Walton
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- M30 **A Characterisation of Roman Age Refractory Ceramics from Autun/France**  
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- M33 **Bronze Age Plain Axes From the Portuguese Territory: An Archaeometallurgical Study of Bujões and Barcelos Types**  
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- M34 **Shaft and Bowls! A Comparative Analysis of Experimental Iron Smelting**  
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Maria Laura Mascelloni, Giorgio Cerichelli and Stefano Ridolfi
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- M42 **'The Objects that Are More than their Analyses': An Archaeometallurgical Investigation and its Implications for the Study of Ancient Technologies**  
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- M49 **Mankinds Earliest Iron - Really Meteoritic?**  
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Aaron Shugar, Robert Thornton and Jonathan Thornton
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Jessica Slater and Roger Doonan
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Jonathan Thornton, Paul Mardikian and Aaron Shugar

- M61 **Archaic Goldwork from the Portuguese Area: Alloys, Technologies and their Relation to the Atmospheric Corrosion**  
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- M62 **Complex Archaeometrical Examination of Iron Tools and Slag from a Celtic Settlement in the Carpathian Basin**  
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- M63 **Finger-Impressed Phoenician-Punic Hearth Ceramics: Testing for Potential Metallurgical Usage**  
Robert H. Tykot
- M64 **The Evolution of Copper Metallurgy in the South-Western Iberian Peninsula – New Evidences from the Pre and Proto-Historic Site of Torre Velha 3 (Southern Portugal)**  
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- M65 **How Can 17<sup>th</sup> Century Bronze Cannons be Magnetic?**  
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Jan Baeten, Michael Paul, Elena Marinova, Stan Hendrickx, Tanja Pommerening, Dirk De Vos, Johann Jauch and Renée Friedman
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Julia Beaumont, Julia Lee-Thorp and Janet Montgomery
- B4 **A Reconstruction of the Living Space in a Community of Early Bronze Age in Sicily: Results of a Multidisciplinary Study**  
G. E. De Benedetto, R. Montesana and E. Margapoti
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Jo Buckberry, Janet Montgomery and Julia Lee-Thorp
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Bea De Cupere and Jeroen Poblome
- B10 **Palaeodietary Reconstruction in the Human Remains Recovered from Roopkund Lake through Elemental Analysis and Estimation of Carbon and Nitrogen Isotope Ratios**  
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Ricardo Fernandes, Pieter M. Grootes and Marie-Josée Nadeau
- B12 **Influence of Cooking on the Isotopic Signals of Fish Species**  
Ricardo Fernandes, John Meadows, Pieter M. Grootes and Marie-Josée Nadeau
- B13 **Paleogenetic Evidence from Dental Calculus: A New Approach to Archaeological Populations**  
Sergio Flores, Constanza De La Fuente, Catalina Fernandez, Fabián Póntigo and Mauricio Moraga
- B14 **Oral Microbiome Evolution in Prehistoric Chilean Populations Using Scanning Electronic Microscopy and Isotopic Analyses**  
Sergio Flores, Constanza De La Fuente, Alfredo Linossier, Mauricio Moraga, Tomás González and Sebastián Krapivka
- B15 **Bees Wax and Propolis as Sealant of Funerary Chambers During the Middle Bronze Age in South-Western Iberian Peninsula**  
José Frade, António Soares, António Candeias, Isabel Ribeiro, Teresa Ponte, Miguel Serra and Eduardo Porfírio
- B16 **Identification of Heme in Aged Blood by Direct Analysis in Real Time Mass Spectrometry**  
Daniel Fraser and Ruth Ann Armitage
- B17 **Stable Isotope Ratio Analysis Elucidates Urban Subsistence and Changing Animal Husbandry Practices at Ancient Sagalassos (SW Turkey)**  
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- B18 **Stable Isotope Studies on Humans and Animals from Tell Tweini, Syria (2600-550 BC)**  
Benjamin T. Fuller, Veerle Linseele and Joachim Bretschneider
- B19 **Microstructural (SEM), Chemical and Molecular-Biological Investigation of Glass Decay at the Medieval Stained Window Glasses of Tarragona Cathedral and Santa Maria del Mar Church (Barcelona), NE Spain**  
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- B20 **Human Mobility and Diet in Iron Age The Netherlands**  
Coen Geerdink, Lisette M. Kootker, Gareth Davies and Henk Kars
- B21 **A Dietary Point of View of Neolithic-Bronze Age Transition: Stable Isotope Analysis of Central France Populations**  
Gwenaëlle Goude and Estelle Herrscher
- B22 **Phosphate Extraction from Enamel and Cementum: A Revised Method**  
Hilary Gough, Brandi Shabaga, Mostafa Fayek and Robert Hoppa
- B23 **Potential of Direct Analysis in Real Time Mass Spectrometry for Rapid Characterization of Organic Residues on Ceramics**  
John Hopkins and Ruth Ann Armitage



- B24 **Isotope Geochemistry in Dutch Archaeology. The Application of Strontium Isotopes as a Proxy for Migration**  
Lisette M. Kootker, Gareth Davies and Henk Kars
- B25 **Herculaneum Conservation Project: Characterisation of Archaeological Waterlogged Wood from Ercolano Site by Pyrolytic and Mass Spectrometric Techniques**  
Jeannette Jacqueline Łucejko, Francesca Modugno, Diego Tamburini and Maria Perla Colombini
- B26 **Subsistence Stability in the Syrian Coastal Area from 2600-550 BC Inferred by Archaeobotanical and Stable Isotope Evidence from Tell Tweini**  
Elena Marinova, Simone Riehl, Ben Fuller and Joachim Bretschneider
- B27 **Paleomicrobial Lipidomics: Mass Spectrometry-Based Discovery of Ancient Mycolic Acids**  
Laszlo Mark, Gabor Maasz and Janos Schmidt
- B28 **Linking Milk Processing to Pottery Function in Prehistoric Anatolia: Diachronic and Regional Perspectives**  
Hadi Özbal, Ayla Türkeku-Bıyık, Laurens Thissen, Turhan Doğan, Fokke Gerritsen and Rana Özbal-Gerritsen
- B29 **Exploring Wine Production and Consumption in the Roman Villa of Sa Mesquida (Mallorca, Balearic Islands)**  
Alessandra Pecci, Verónica Martínez, Catalina Mas<sup>1</sup> and Miguel Ángel Cau
- B30 **Morphometric Data on Bovine Remains (*Bos taurus* and *Bos primigenius*) Found in Chalcolithic Settlements from South-Eastern Romania**  
Mariana Popovici, Balasescu Adrian, Simina Stanc and Luminita Bejenaru
- B31 **Dietary Differences between two Postmedieval Nunnery Sites from the Southern Low Countries: An Investigation Using ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) Stable Isotope Ratio Analysis**  
Kim Quintelier, Benjamin T. Fuller, Anton Ervynck, Gundula Müldner, Michael P. Richards and Wim Van Neer
- B32 **Dietary Patterns in the Mixed Lay and Monastic Population from the Postmedieval Carmelite Friary Burial Grounds at Aalst (Flanders, Belgium), and their Relationship with DISH**  
Kim Quintelier, Benjamin T. Fuller, Gundula Müldner, Wim Van Neer, Michael P. Richards and Anton Ervynck
- B33 **Paleodiet and Provenance of Marbles: Case Studies by Using Stable Isotope at the CIRCE Laboratory**  
Paola Ricci, Carmina Sirignano, Mauro Rubino and Carmine Lubritto
- B34 **Paleodiet in two Archaeological Sites in the Atacama Oasis Inferred by Isotopic Analysis**  
Francisca Santana, Mauricio Uribe, Mauricio Moraga, Anahí Maturana, Pamela Cañas, Francisca Concha and Sergio Flores
- B35 **Tracing Mobility in Early Medieval Populations Using Strontium and Oxygen Isotopes – A Case Study from South-West Germany**  
Christine Schuh, Cheryl Makarewicz and Claus von Carnap-Bornheim
- B36 **Isotopic Life History of Neolithic People from Busan Gadeokdo Janghang Site, Korea**  
Ji Young Shin, Da Young Kang, So Young Kang, Sang Hyun Kim and Eui Do Jung
- B37 **Morphometric Data for Suines (*Sus scrofa domesticus* and *Sus scrofa ferus*) in Chalcolithic Period from Romania**  
Simina Stanc, Mariana Popovici, Balasescu Adrian and Luminita Bejenaru

- B38 **The Use of Intra-Tooth Enamel  $\log(\text{Sr}/\text{Ca})$  &  $\log(\text{Ba}/\text{Ca})$  Sequences to Assess Animal Foddering Strategies**  
Carlos Tornero, James Burton, Douglas T. Price and Maria Saña
- B39 **Non-Destructive Trace Element Analysis of Human Bones to Examine Diet and Mobility**  
Robert H. Tykot
- B40 **A View into the Lives of the Early Christians of Stavanger, Norway: A Palaeodietary Reconstruction Using Multiple Stable Isotopes (C, N, H and S) of Bone Collagen**  
Laura van der Sluis, Hege Hollund and Henk Kars
- B41 **“For Dust thou Art and ...” with Dust You Can Cure**  
Michał Wasilewski
- B42 **Diagenetic Assessment of the Trace Element Composition of Classical Period Bones – A Case Study From Apollonia Pontica (Bulgaria)**  
Boika Zlateva-Rangelova, V. Lyubomirova and D. Lesigyski
- B43 **Residual Analysis of Hellenistic Time Ammoraes from Apollonia Pontica (Bulgaria): A Preliminary Results**  
Boika Zlateva-Rangelova and Miroslav Rangelov

## Poster Session 2

V1-V106: Ceramics, Glazes, Glass and Vitreous Materials

G1-G11: Remote Sensing, Geophysical Prospection and Field Archaeology

H1-H15: Human- Environment Interactions

C1-C22: Colour and Culture

### Ceramics, Glazes, Glass and Vitreous Materials

- V1 **Production, Exchange, and Technological Identity: A Study of Islamic Umayyad pottery from northern Jordan**  
Firas Alawneh
- V2 **A Geometrical Sintering Model (GSM) of Archaeological Ceramics for the Study of Transverse Rupture Strenght (TRS): Preliminary Results**  
Ignazio Allegretta and Giacomo Eramo
- V3 **PIXE Analysis of Decoration Pixels in Classical Attic Pottery**  
Eleni Aloupi-Siotis, Artemi Chaviara, Robert Huszánk, Tassos Lagoyannis, L. Csedreki, E. Furu, Zs. Kertész, Á.Z. Kiss, A. Simon, Zs. Török, I. Uzonyi and Z. Szikszai
- V4 **Rediscovering Brockley Hill: A Compositional and Technological Study of Verulamium White Ware**  
Silvia Rita Amicone
- V5 **Among the Aspects of the Past : The Detection of Tin Foil in Hellenistic Terracotta Figurines**  
Eleni Asderaki-Tzoumerkioti, Manos Dionysiou, Argyroula Doulgeri-Intzesiloglou and Polyxeni Arachoviti
- V6 **Analysis of Lusted Ceramics of the Galleria Regionale Di Palazzo Bellomo di Siracusa**  
Marc Aucouturier, Anne Bouquillon, Renato De Vincolis, Anna Gueli, Giuseppe Politi, Giuseppe Stella, Sebastiano Olindo Troja and Carmela Vella
- V7 **The Technology of Historical Basque Pottery Production During the 14<sup>th</sup> to 17<sup>th</sup> Centuries**  
Cristina P. Barrachina, Peter M. Day, Sergio Escribano Ruiz, Jaume Buxeda i Garrigós and Marisol Madrid i Fernández
- V8 **Chemical Characterization of Medieval Glass Coming from two Stained Glass Windows from Girona Cathedral (NE Spain)**  
Flavia Bazzocchi, Domingo Gimeno and Meritxell Aulinas
- V9 **Basic Research to Establish the Use of p-XRF in Pottery Provenance Studies**  
Sonja Behrendt, Oliver Mecking and Dirk Paul Mielke
- V10 **Archaeometric Investigation of Phoenician Pottery from the Iberian Peninsula**  
Sonja Behrendt and Dirk Paul Mielke
- V11 **Investigating the Influence of Neutron Activation Analysis on European Trade Glass Beads**  
Adelphine Bonneau, Jean-François Moreau, Ron Hancock, Réginald Auger and Bertrand Emard
- V12 **Characterization and Interpretaipon of Roman Ceramic Manufacture in the *Civitas Tungrorum*, Belgium**  
Barbara Borgers, Marc De Bie, Patrick Degryse and Patrick Sean Quinn

- V13 **Pottery Production and Regional Distribution at Düzen Tepe-Sagalassos and the Lake Burdur Area (Southwest Turkey) During the Classical and Hellenistic Period**  
Dennis Braekmans, Patrick Degryse, Jeroen Poblome, Bert Neyt and Marc Waelkens
- V14 **Roman Amphora Production at Casa Valentini (Central Italy) During the Late Republican and Early Imperial Period**  
Dennis Braekmans, Patrick Monsieur and Patrick Degryse
- V15 **Raw Materials for Roman Glass Production in the Western Mediterranean**  
Dieter Brems and Patrick Degryse
- V16 **Sr and Nd Isotopic Analysis of Homemade Roman Natron Glass**  
Dieter Brems, Monica Ganio, Rebecca Scott, Frank Vanhaecke, Kris Latruwe and Patrick Degryse
- V17 **Black-Appearing Roman Glass: A Continued Research**  
Simone Cagno, Peter Cosyns, Karin Nys, Andrei Izmer, Frank Vanhaecke and Koen Janssens
- V18 **Compositional Analysis of 14<sup>th</sup> Century English Stained Glass and Characterization of Corrosion Bodies via Synchrotron-Based Techniques**  
Simone Cagno, Gert Nuyts, Marine Cotte, Lukas Helfen, Kristel De Vis, Joost Caen, Simone Bugani and Koen Janssens
- V19 **Production and Distribution of Cooking Wares in the Early Islamic Vega of Granada (Spain)**  
José C. Carvajal and Peter M. Day
- V20 **An Ethnoarchaeometric Study of the Cooking Ware Production of Pòrtol (Mallorca, Balearic Islands)**  
Miguel A. Cau-Ontiveros, Peter M. Day, Catalina Mas-Florit, Noémi Müller and Evanthia Tsantini
- V21 **Inorganic and Organic Characterisation of Late Roman Pottery from the Site of Can Muntanyans (Palma de Mallorca, Balearic Islands)**  
Miguel A. Cau-Ontiveros, Evanthia Tsantini, Alessandra Pecci and Catalina Mas-Florit
- V22 **Optical Spectroscopy and LA-ICP-MS for the Characterization of Roman Glass**  
Andrea Ceglie, Wendy Meulebroeck, Peter Cosyns, Karin Nys, Herman Terryn and Hugo Thienpont
- V23 **ICP-AES Analysis of Ancient Glass from Qiemo Tomb Sites on the South Line of the Silk Road in China**  
Qian Cheng, Bo Wang and Jinlong Guo
- V24 **Pottery on the Periphery: A Technological Characterization of Stonepaste Ceramics from Middle Islamic Tall Dhiban, Jordan**  
Bryan Cockrell and Benjamin Porter
- V25 **X-ray Computed Tomography Applied in the Study of Ancient Ceramics Objects**  
Florin Constantin, Carmen Pavel, Cosmin I. Suci and Roxana Bugoi
- V26 **Macroscopic and Microscopic Aspects of Polishing Ceramic Pots Using Water Worn Pebbles**  
Otis N. Crandell, Corina Ionescu and Volker Hoeck
- V27 **The Origins of Pottery Technology in the Cantabrian Region (5000 cal BC). Mineralogical and Geochemical Analysis**  
M. Cubas and P. Arias
- V28 **Distant Neighbours: Early Helladic Ceramic Production in the Nemea Valley and Korinth**  
Clare Burke Davies, Peter Day, John F. Cherry, Daniel Pullen and James Wiseman

- V29 **'Marbrite Fauquez' Opalescent and Marbled Glass: A Material-Technical Study**  
Liesbeth Dekeyser, Ann Verdonck, Hilde De Clercq and Gaia Ligovich
- V30 **Development of an Appropriate Isolation Procedure for Boron from Soda-Silica-Lime Glass**  
Veerle Devulder, Patrick Degryse and Frank Vanhaecke
- V31 **Chinese Porcelain Ordered for the Portuguese Market During the 16<sup>th</sup> Century: Study on the Compositional Differences by Neutron Activation Analysis and Indirect Provenance Issues**  
M. Isabel Dias, M. Isabel Prudêncio, M. Antónia Matos and A. Luisa Rodrigues
- V32 **Chemical Signatures of Coimbra and Lisbon Early Portuguese Faience Productions (17<sup>th</sup> – 18<sup>th</sup> Cent.)**  
M. Isabel Dias, M. Isabel Prudêncio, Alexandre Pais and A. Luisa Rodrigues
- V33 **Chemical Fingerprinting of Hungarian and Slovakian Obsidian Using three Complementary Analytical Techniques**  
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- V34 **QXRD, ESEM and DSC Applied to the Study of Pottery Manufacturing Process from Tappeh Zaghe (5100-4900 BC), Iran**  
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- V35 **The Analysis of the Viking-Age Glass Gaming Pieces from the Graves and Settlements in Russia and Ukraine**  
Natasha Eniosova, Lyubov Pelgunova, Tamara Pushkina and Ekaterina Stolyarova
- V36 **Characterization of Late Iron Age Ceramic Sherds and Clay Samples from Zamala (Northern Cameroon, Diamaré Division, Central Africa)**  
Zoila Luz Epossi Ntah, Robert Sobott and Klaus Bente
- V37 **Characterization of Obsidian Sources from Colombia Using Electron Spin Resonance**  
Jhon Escobar, Ovidio Almanza and Alí Duran Öcal
- V38 **Electron Microprobe and Petrographic Analysis of Later Prehistoric Granodiorite-Tempered Pottery from the East Midlands, UK**  
Eddy Faber, David Knight, John Carney, Julian Henderson and Patrick Marsden
- V39 **Archaeometric Characterisation of Late Roman Amphorae from Sant Martí d'Empúries (Catalonia, Spain)**  
Leandro Fantuzzi, Miguel A. Cau-Ontiveros, Evanthia Tsantini and Xavier Aquilué
- V40 **Application of TOF-SIMS to Ancient Glasses**  
Sarah Fearn and Katherine Eremin
- V41 **The Mechanical Properties of 16<sup>th</sup> Century Transport Jars from Panamá and Seville**  
Samantha G. Ferrer, Noémi S. Müller and Vassilis Kilikoglou
- V42 **Distinguishing Handmade and Wheel Thrown Pottery Using X-Ray Diffraction**  
Lesley D. Frame, Sarah Doherty and Ian C. Freestone
- V43 **Non-Destructive Analysis of Chinese Porcelain from the Mexico City Colonial Period (XVI to XVIII Centuries)**  
Gabriel Alejandro Funes and José Luis Ruvalcaba-Sil
- V44 **Geochemical Investigation of Roman Coloured Glass from North-Eastern Italy**  
Filomena Gallo, Alberta Silvestri, Gianmario Molin, Alessandra Marcante, Patrick Degryse and Monica Gano

- V45 **Iron Age Vessels from the Archaeological Museum of Adria (North-Eastern Italy): A Textural, Chemical, and Mineralogical Study**  
Filomena Gallo, Alberta Silvestri, Gianmario Molin, Alessandra Marcante and Paolo Guerriero
- V46 **A Reliable Protocol for the Isolation of Nd from Low Content Archaeological Glass**  
Monica Ganio, Kris Latruwe, Dieter Brems, Frank Vanhaecke and Patrick Degryse
- V47 **Pompeii and Herculaneum: Differences and Similarities through the Chemical and Isotopic Composition of Glass**  
Monica Ganio, Marc Walton, Rita Giannini, Andrew J. Shortland, Kris Latruwe, Frank Vanhaecke and Patrick Degryse
- V48 **The Stained Glass Window from San Antonio di Padova Capella (1525), San Petronio Basilica, Bologna, (Italy): Chemical Evidence of a Northern Europe Glass Influence**  
Domingo Gimeno, Meritxell Aulinas, Flavia Bazzocchi, Maite Garcia-Valles, Elena Basso, Bruno Messiga, Maria Pia Riccardi and Camillo Tarozzi
- V49 **Characterisation of Glazed Tiles with EPMA and Mobile XRF for the Development of Adapted Conservation Materials**  
Rena Gradmann, Ulrich Schüssler, Paul Bellendorf and Jasmin Badr
- V50 **'Pottery from the Underworld'. A Petrological Analysis from a Selected Group of Neolithic-Middle Bronze Age Ceramics from the Caves of Central Sardinia**  
Maria Giuseppina Gradoli
- V51 **Archaeometric Characterization of a Furnace for Bricks Near the Castle of Zena (Piacenza, Northern Italy)**  
Sabrina Gualtieri and Bruno Fabbri
- V52 **Ninth-Century Glasses from Samarra (Iraq) in the Context of Medieval Near Eastern Glass Production**  
Nadine Schibille, Jens Kröger and Mariam Rosser-Owen
- V53 **Of Time and Shapes: Compositional Variation in Post-Medieval Glass from the Netherlands**  
Hans Huisman, Bertil van Os, Guus Lange and Fokko Post
- V54 **The Strange Case of 60 Frothy Beads: Puzzling Early Iron Age Glass Beads from the Netherlands**  
Hans Huisman, Bertil van Os, Joas van der Laan, Dominique Ngan-Tillard, Ineke Joosten and Bert Fermin
- V55 **Mobility and Pottery Production in Panama (ca. 4500-3200 BP): Petrographic and Geochemical Research**  
Fumie Iizuka
- V56 **Ceramic Technologies Among Arctic Foragers: A Pilot Study from Nunivak Island, Alaska**  
Ana Jorge, Noémi Müller and Richard Knecht
- V57 **Waste from a Glasswork at Glargaarde in Northern Jutland**  
Arne Jouttijärvi
- V58 **Physical Chemistry Analysis of Roman Glass Fragments from Thessaloniki Agora**  
Natasa Kalogiouri, Ioannis Nazlis, Despina Ignatiadou, Ioannis Stratis
- V59 **Glassware from Building of XVIII c. of Kazan Kremlin**  
Rezida Khramchenkova and Airat Sitdikov

- V60 **An Archeometric Investigation of Some Byzantine Ceramics Case Study: 12<sup>th</sup>-13<sup>th</sup> Century Glazed Ceramics from Kuşadası Kadıkalesi/ Anaia**  
Burcu Kırmızı, E.Hale Göktürk, Asuman G.Türkmenoğlu, Lale Doğer and Zeynep Mercangöz
- V61 **Cassiterite in Tin Glaze of Anabaptist Faience from Hungary: Morphological and Crystallographic Characterization by SEM and TEM**  
Viktória Kovács Kis and Bernadett Bajnóczi
- V62 **Ceramic Technological Traditions from the Early to the Late Neolithic in Hungary**  
Attila Kreiter, György Szakmány, Nándor Kalicz, Katalin Kovács, Zsuzsanna Siklósi, Orsolya Viktorik, Katalin Vanicsek and Ferenc Horváth
- V63 **The Chemical Composition of Glass from the Hungarian Glasshouses and Glass Utilized in Hungary from the 14<sup>th</sup> Century to the 19<sup>th</sup> Century**  
Jerzy J. Kunicki-Goldfinger, Edit Mester and Ian C. Freestone
- V64 **Study of the Production of an Archaeological Funerary Urn via Electron Paramagnetic Resonance and Mössbauer Spectroscopy**  
Giovana M. Manguiera, Rosane Toledo, Simonne Teixeira, Pablo Munayco, Rosa B. Scorzelli and Roberto W.A. Franco
- V65 **Mesolithic and Neolithic Pottery Production at Al Khiday Sites (Khartoum, Sudan)**  
Lara Maritan, Gregorio Dal Sasso, Sandro Salvatori, Claudio Mazzoli and Gilberto Artioli
- V66 **Production of Glass Beads in Merovingian Times: Study of the Artefacts Coming from the Excavation of Grez-Doiceau Necropolis**  
François Mathis, Olivier Vrielynck, Romain Elias, Amandine Leroy and David Strivay
- V67 **Medieval Lead Glass in Central Europe**  
Oliver Mecking
- V68 **The Tombs of Ayia Kyriaki and the Circulation of Pottery in EMI-II South-Central Crete**  
Roberta Montesana, Peter M. Day, Evangelia Kiriati, Simona Todaro and David E. Wilson
- V69 **Bronze Age Glass Between the Alps and the Baltic Sea. Studies on Manufacture and Distribution of the Oldest Glass in Central Europe**  
Stephanie Mildner, Ulrich Schüssler and Frank Falkenstein
- V70 **Colour and Microstructure in Brown and Green Decorated Spanish Tin Glaze Pottery (10<sup>th</sup> to 17<sup>th</sup> Centuries AD)**  
Gloria Molina, Judit Molera and Trinitat Pradell
- V71 **Classification of Archaeological Pottery from Amazon Basin by Mössbauer Spectroscopy and Neutron Activation Analysis**  
Pablo Munayco, Rose Mary Latini, Alfredo Bellido and Rosa B. Scorzelli
- V72 **Mixing Traditions – Mixing Cultures, Technological Choices for the Production of ‘Kampos Group’ Pottery in Prepalatial Crete**  
Eleni Nodarou and Yiannis Papadatos
- V73 **Technological and Provenance Study of Archaic Glassy Materials from Rhodes Island Using XRF and SEM/EDX Analysis**  
Artemios Oikonomou, Konstantinos Beltsios, Nikolaos Zacharias and Pavlos Triantafyllidis

- V74 **Black- and Red-Slipped Pottery from Ancient Cassope (NW Greece): Inference of Provenance and Production Technology Based on a Multi-Analytical Approach**  
Artemios Oikonomou, Christina Papachristodoulou, Konstantina Gravani, Konstantinos Stamoulis and Konstantinos Ioannides
- V75 **XRF Spectrometry and Late Bronze Age/Early Iron Age Pottery Within Interpretative Framework: An Outline of the Project**  
Monika Okupniak
- V76 **Characterization of Early Neolithic to Calcolitic Pottery from Los Cascajos Settlement (Northern Spain)**  
Luis Angel Ortega, Maria Cruz Zuluaga, Ainhoa Alonso-Olazabal, Xabier Murelaga, Carlos Olaetxea, Jesús García Gazólaz and Jesús Sesma
- V77 **Technology and Composition of Early Anglo-Saxon Glass Beads from Eriswell, Suffolk**  
James R. N. Peake
- V78 **Defining Local Ceramic Production at Hellenistic Syene, Upper Egypt**  
Lisa Peloschek, Roman Sauer and Sabine Ladstätter
- V79 **Making Pottery by the Lakeshore: The Case of the Lake Karla Settlements During the Neolithic (Thessaly, Greece)**  
Areti Pentedeka
- V80 **Portuguese Glazed Tiles (16<sup>th</sup>-18<sup>th</sup> Centuries): INAA, XRD and Luminescence for Raw Materials Characterization and Production Technologies of the Ceramic Bodies, and Chronology**  
M. Isabel Prudêncio, M. Isabel Dias, Christopher I. Burbidge, Lurdes Esteves, M. José Trindade, Rosa Marques, Guilherme Cardoso and Dulce Franco
- V81 **Developing a Novel Method to Identify Salt Production Pottery via the Chemical Release and Detection of Chloride and Sodium**  
Danielle R. Raad and Rowan K. Flad
- V82 **Archaeometric and Archaeomagnetic Measurements on Greek Ceramics and Baked Clays: A Promising Combination**  
Christina Rathossi, Despina Kondopoulou and Evdokia Tema
- V83 **Producing Black Glass During the Roman Period (Notes on a Crucible Fragment from Serdica, Bulgaria)**  
Thilo Rehren and Anastasia Cholakova
- V84 **Using Lead Isotopes for Cypriote Pottery Provenance - Signature of Cypriote Clay Sources and Comparison with Late Bronze Age Pottery from Cyprus**  
Virginie Renson, Jan Coenaerts, Ariane Jacobs, Nadine Mattielli, Karin Nys and Philippe Claeys
- V85 **Petrographic Analysis of Ceramics from the Oyo Ceramic Complex at Ede-Ile (Nigeria)**  
Dana Drake Rosenstein, Akin Ogundiran and Bolanle Tubosun
- V86 **Geometric Features Extraction of Ancient Pottery Decorations through Multiresolution Laser Scanning**  
Giuseppe Salemi, Lara Maritan and Sandro Salvatori
- V87 **Salt Damage Related to Physical Properties of Ceramics**  
Brunella Santarelli and Nancy Odegaard
- V88 **Primary Glass Production in Roman “Kastro Palaia” – Iolkos**  
Thilo Rehren, Eleni Asderaki-Tsoumerkoti and E. Skafida



- V89 **Tracing Roman Glass: The Use of Trace Elements to Determine Primary Roman Glass**  
Rebecca Scott, Patrick Degryse, Monica Ganio and Dieter Brems
- V90 **Danger! High Voltage! The Application of HH-XRF to Different Archaeological Materials; Pitfalls and Potentials**  
Rebecca Scott and Dennis Braekmans
- V91 **The Use of Handheld XRF for the Quantitative Analysis of Archaeological Materials**  
Aaron N. Shugar
- V92 **Roman Grey-Clay Tableware “Imitations” of Late Republican Italic Models from Southwest Iberia: A Preliminary Chemical and Mineralogical Characterization**  
V. Soria, A.M. Arruda, N. Schiavon and J. Mirão
- V93 **Constructing a Database for pXRF, XRD, ICP-MS and Petrographic Analyses of Bronze Age Ceramics and Raw Materials from Failaka Island (Kuwait)**  
Ciprian C. Stremtan, Hasan Ashkanani, Robert H. Tykot and Cristina M. Puscas
- V94 **Craft Production of Pottery from a Bronze Age Site in North-Eastern Italy: New Results from Petrographic Analysis**  
Marta Tenconi, Lara Maritan and Giovanni Leonardi
- V95 **Portable XRF Analysis of Sources and Distribution of Obsidian Artifacts in Iran and Syria**  
Yukiko Tonoike
- V96 **Non-Destructive Sourcing of Prehistoric Pottery from the SouthEastern United States Using pXRF**  
Robert H. Tykot, Martin Koppe, Jeffrey DuVernay and Nancy White
- V97 **Assemini Traditional Pottery Making Village in South Sardinia: Ethnoarchaeology and Ethnoarchaeometry**  
Evanthia Tsantini, Miguel Àngel Cau Ontiveros and Giuseppe Montana
- V98 **Islamic Glass Weights from Egypt: A Non-Destructive Study by  $\mu$ -XRF**  
Gloria Vaggelli, Roberto Cossio, Valeria Lovera and Piero Mirti
- V99 **Geobiochemical Features of Source Materials in Glass of Volga Bulgaria**  
Svetlana Valiulina
- V100 **Comprehensive Investigation and Reconstruction of a Pottery Workshop Used for Production of Glazed Ceramics in Bilyar City Excavation**  
Svetlana Valiulina and Maria Iassonova
- V101 **Home Sweet Home. Identifying Ceramic Production in the Burdur Plain, S.W. Turkey**  
Ralf Vandam, Dennis Braekmans, Eva Kaptijn, Patrick Degryse, Jeroen Poblome and Marc Waelkens
- V102 **Iron Age Glass in the Netherlands: XRF-Analysis of La Tène Bracelets**  
Joas van der Laan, Hans Huisman, Bertil van Os, Nico Roymans, Henk Kars and Louis Swinkels
- V103 **Colorants and Opacifiers in Late Antique – Early Medieval Mosaic Glass from Saint Severo (Classe – Ravenna, Italy)**  
Mariangela Vandini, Rossella Arletti and Cesare Fiori
- V104 **Preliminary Results of Late Neolithic Ceramic Analysis from Hódmezővásárhely-Gorzsa and Aszód-Papi Földek (Tisza Culture, SE Hungary)**  
Katalin Vanicsek, György Szakmány, Ferenc Horváth, Attila Kreiter, Nándor Kalicz, Katalin Kovács, Zsuzsanna Siklósi and Orsolya Viktorik

- V105 **Hyperspectral XRF Imaging of Ancient Athenian Pottery: New Insights on the Production Technology by Utilizing the Full Spectrum at Every Pixel**  
 Marc Walton, Marvin Cummings, Giulia Poretti, Karen Trentelman, Jeff Maish and David Saunders
- V106 **A Technological and Provenance Study of Two Mycenaean Glass Collections Using X-Rays and Ion-Beam Analyses**  
 Nikolaos Zacharias, Maria Kaparou, Zsolt Kasztovszky, Boglárka Maróti, Konstantinos Beltsios, Imre Kovács, Zoltán Szőkefalvi-Nagy, Joanne Murphy, Vasilike Kantarelou and Andreas Germanos Karydas

### **Remote Sensing, Geophysical Prospection and Field Archaeology**

- G1 **Using Photogrammetry and Laser Scanner for Digital Documentation of Architectural Remains in Ancient Jerash City of Jordan**  
 Yahya Alshawabkeh
- G2 **Integrated Prospection Project at Migdal, Israel**  
 Luis Barba, Jorge Blancas, Austin Ortiz, Marcela Zapata and Linda R. Manzanilla
- G3 **Out of the Furnace and into the Field: The Implications of Reconceptualising Metallurgy as Practice**  
 Lucy Cheesman, Brad Comeau, Michael Romano, Jessie Slater and Roger Doonan
- G4 **Multi-Element Soil Analysis on two Classical-Hellenistic Sites in South-Western Turkey as a Proxy for Ancient Human Activity**  
 Katrijn Dirix, Philippe Muchez, Patrick Degryse, Eva Kaptijn, Elvira Vassilieva, Branko Mušič and Jeroen Poblome
- G5 **Mapping Prehistoric Settlement Patterns. The Application of pXRF in Combination with Geophysical Prospection Techniques**  
 Roland Gauss and Knut Rassmann
- G6 **Different Use of Magnetometric Field Methods in Czech Archaeology**  
 Roman Křivánek
- G7 **3D Laser Scanning: The Digital Imaging of Fort Conger, Nunavut**  
 Richard Levy, Peter Dawson and Chris Tucker
- G8 **Advancements in Handheld XRF Technology for Field Archaeology**  
 Kimberley A. Russell
- G9 **Magnetic Gradiometry at Oğlanqala, a 1<sup>st</sup> Millennium Iron Age Fortress Site, Naxçıvan, Azerbaijan**  
 Rob Sternberg, Stella Dee, Travis Johnson, Veli Bakhshaliyev and Lauren Ristvet
- G10 **Long Distance Import of Polished Stone Artefacts: HP Metamorphites in Hungary**  
 György Szakmány, Katalin T. Biró, Ferenc Kristály, Zsolt Bendő, Zsolt Kasztovszky and Norbert Zajzon
- G11 **What is Left on the Floor? An Interdisciplinary Archaeological and Archaeometric Study of a Hellenistic House Floor at Düzen Tepe (SW Turkey)**  
 Kim Vyncke, Patrick Degryse and Marc Waelkens

## Human-Environment Interactions

- H1 **Site Catchment Analysis and Non-Invasive Approaches in Romanian Chalcolithic. Case study: Cucuteni Settlements from Valea Oii river Basin**  
Andrei Asăndulesei, Robin Brigand, Ionuț Cristi Nicu and Vasile Cotiugă
- H2 **From Heating Marks to Intensity of Fires: Thermal Characterization by Thermoluminescence of Reddened Wall in the Chauvet Cave and Fire Experimentations**  
Aurélie Brodard, Pierre Guibert, Catherine Ferrier, Bertrand Kervazo, Evelyne Debard and Jean-Michel Geneste
- H3 **Hearths in the Cave of Les Fraux (Dordogne, France): Thermal Characterization (Thermoluminescence and Magnetic Susceptibility) and Experimentation**  
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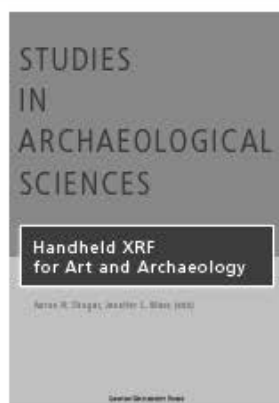
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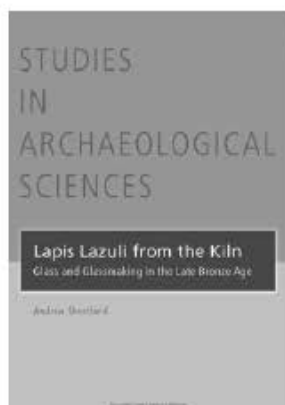


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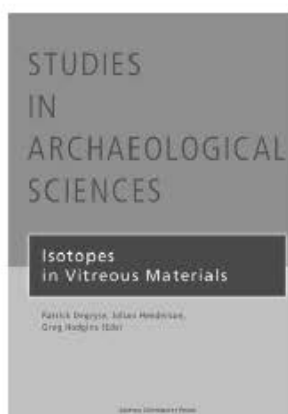


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### 1 **Advantages and Disadvantages of pXRF over other Elemental Methods of Analysis for Obsidian Sourcing and Studying Ancient Trade**

**Robert H. Tykot**

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Elemental methods of analysis have been successfully used for obsidian sourcing for nearly 50 years, using a variety of instrumental methods including optical emission spectroscopy (OES), atomic absorption spectroscopy (AAS), neutron activation analysis (INAA), electron microprobe (EPMA), scanning electron microscope (SEM), X-ray fluorescence spectrometry (XRF; both energy- and wavelength-dispersive), inductively coupled plasma – optical emission spectroscopy (ICP-OES), and ICP mass spectrometry (usually with laser ablation). For many prior studies, the destructive nature and per-sample time and cost of analysis has limited the number and/or physical size of the artifacts tested. Over the last several years, however, portable XRF instruments have been developed and become widely popular, allowing non-destructive analyses to be conducted in museums and other places, on virtually any size obsidian artifact, producing data for one hundred or more per day. Presented here are the advantages and disadvantages of this type of instrument based on my usage over the past five years on thousands of artifacts from around the world. Specifically addressed are precision and accuracy, and how to compare pXRF data, e.g. on artifacts, with results from other methods on geological source samples. This includes a direct data comparison, for the exact same artifacts, between pXRF and INAA, ICP-MS, microprobe, and ED-XRF. Other issues include the limitations of surface analysis and the area being tested, instrumental settings, and manual analysis of each sample. Also included are examples of how pXRF analyses of very large numbers of obsidian artifacts in Europe and the Mediterranean significantly enhances our understanding of trade and contact in the ancient world.

## 2 Petrological Characterization of the Neolithic-Chalcolithic Basalt Extraction Site of Giv'at Kipod (Galilee/Israel): A Basis for Provenance Analyses of Basaltic Tools from Southern Levant

Tatjana Mirjam Gluhak<sup>1</sup> and Danny Rosenberg<sup>2</sup>

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The Giv'at Kipod („hedgehog hill“) belongs to a row of isolated Miocene basalt hills at the southern margin of the Yizreel Basin (Galilee). The Late Neolithic-Chalcolithic extraction site of the Giv'at Kipod is the first known quarry for the production of basalt artefacts south of the Turkish part of Thrace (Erdogu, 2000; Ozbaek, 2000; Shimelmitz et al., 2005; Milevski, 2008; Rosenberg et al., 2008; Rosenberg & Shimelmitz, 2010). This raises questions concerning the distribution of the products and the significance of the Giv'at Kipod for the early societies of the region. However, the distribution of the products can only be determined by provenance analyses based on petrological-geochemical features of the raw material. The deposit provides for the first time the possibility of a raw-material centered provenance study in the Levant. The geochemical-mineralogical variability of the Giv'at Kipod has to be defined and to be distinguished from other comparable Miocene basalt deposits of this region. Twenty two outcrops in the complete Yizreel Basin and from the neighbouring Jordan valley were sampled (in total 104 samples). Considering the following artefact analyses analytical methods were chosen which get along with a small amount of sample material. Each sample was crushed and ground to powder. 0.4 g of powder was used to produce glass beads for XRF analyses, to determine the major element composition and an internal standard element for the following Laser-ICP-MS measurements. A maximum of 0.5 g of powder was needed to determine the loss on ignition. About 0.2 g was used to produce glass beads for bulk rock trace element analyses by Laser-ICP-MS. The analyses of the field samples were supported by thin section studies. The Giv'at Kipod rock is a compact fine-grained basanite with abundant olivine and smaller clinopyroxene phenocrysts in a matrix of plagioclase, sanidine, opaques, lesser clinopyroxene and olivine. It has a well constrained geochemical composition and can be easily distinguished from the other Miocene basaltic/bsanitic rocks of the region already by its SiO<sub>2</sub>-, Fe<sub>2</sub>O<sub>3</sub> (t)-, and MgO-content. All archaeological samples (so far 16 samples of pestles, mortars and hand axes) are analyzed by the same methods. One piece of every sample (if a sufficient amount available) is kept for thin section analyses. The artefact affiliation is supported by cluster and discriminant analyses.

ERDOGU, B., 2000. The problems of dating prehistoric axe factories and neolithisation in Turkish Thrace. *Documenta Praehistorica* **27**, 155-166. ; MILEVSKI, I., 2008. The Exchange of Ground Stone Tools and Vessels during the Early Bronze Age in the Southern Levant. In: ROWAN, Y.M. AND EBLING, J.R. (Eds.) *New Approaches to Old Stones: Recent Studies of Ground Stone Artefacts*, 116-129. ; ROSENBERG, D. AND SHIMELMITZ, R., 2010. Giv'at Kipod - A Basalt Quarry and a workshop for the production of Bifacial Tools in Israel. *The Quarry* **5**, 7-14. ; ROSENBERG, D., SHIMELMITZ, R. AND NATIV, A., 2008. Basalt bifacial tool production in the southern Levant: a glance at the quarry and workshop site of Giv'at Kipod, Israel. *Antiquity* **82**, 367-376. ; SHIMELMITZ, R., ROSENBERG, D. AND NATIV, A., 2005. Giv'at Kipod: a basalt quarry and a workshop for the production of bifacial tools in the Manasseh Hills, Israel. *Neo-Lithics* **1/05**, 9-12. ; OZBAEK, O., 2000. A prehistoric stone axe production site in Turkish Thrace: *Hamaylitarla. Documenta Praehistorica* **27**, 167-170.

### **3 Non-Destructive Study of the Green Stone Artifacts of the Tomb of the Maya Ruler Pakal**

**José Luis Ruvalcaba-Sil<sup>1</sup>, Mayra Manrique<sup>1</sup>, Angélica García Bucio<sup>1</sup>, Valentina Aguilar Melo<sup>1</sup>, Edgar Casanova<sup>1</sup> and Laura Filloy<sup>2</sup>**

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K'inich Ja'naab Pakal was the ruler of the Maya city of Palenque during its splendor from 603 to 683 A.D. His spectacular tomb was discovered by archaeologist A. Ruz in Palenque, Chiapas, Mexico, in 1952. A mask with the features of Pakal and a complete set of green stone artifacts and other objects of other materials were found in the burial. They can be considered among the most important green stone pieces of Mesoamerica. Most of the artifacts discovered in the tomb and in the burial are kept in the National Museum of Anthropology in Mexico City. Recently, most of the tesserae of the Mask and green stone pieces of the tomb of K'inich Ja'naab Pakal were studied in situ using a non destructive methodology and portable equipments. For the characterization, some imaging techniques using UV and IR lighting, colorimetric measurements, as well as analytical techniques, such as X-ray fluorescence, Raman and FTIR spectroscopy were applied on most of the pieces. The objects were studied in the Museum in collaboration with the conservation department in order to determine the mineral identification, the elemental composition and the probable provenance of the green stone materials used for the Pakal's trousseau and the offerings of the tomb. The results of this research indicate that the tesserae correspond to jadeite but also other unexpected green stone minerals were found. A set of the jadeite pieces may come from the Motagua sources of Guatemala but other non-identified beds are pointed out as well. The colorimetric analysis shows a careful selection of the pieces in order to compose the mask and the most outstanding pieces of the Pakal's burial such as the rings and ear ornaments. This study provides outstanding information about the used materials for the Pakal tomb, as well as new data about manufacturing and trade routes of green stone artifacts in the Maya area. This research has been supported by Mexico PAPIIT UNAM grant IN403210 and CONACyT 131944 project.



#### 4 Using Pb and Sr Isotopes to Infer the Source of Turquoise at the Aztec Templo Mayor

Alyson M. Thibodeau<sup>1</sup>, Leonardo López Luján<sup>2</sup>, David J. Killick<sup>3</sup>, John T. Chesley<sup>1</sup> and Joaquin Ruiz<sup>1</sup>

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Turquoise was highly valued in late Postclassic Mesoamerica, especially by the Mixtecs and Aztecs, who used it as part of elaborate mosaics and other ceremonial and status objects. Because turquoise mineralization in North America is primarily distributed across the present-day southwestern United States (U.S.) and northern Mexico, archaeologists have identified these regions as likely sources of the turquoise found in Mesoamerica, and, in particular, used by the Aztec empire. Extant tribute lists indicate that turquoise used by the Aztec elite came from the southern (Mixteca) area of the empire and from one province in the empire's northeastern corner (Berdan, 1987). However, it is not known if these provinces acquired turquoise through trade or directly mined it from local (but presently unknown) sources of the mineral. Despite the common speculation among archaeologists that Mesoamerican societies acquired turquoise through trade with the societies of the southwestern U.S. and/or northern Mexico, chemical data to support this idea has never been published. To evaluate the source(s) of turquoise used by Aztec empire, we apply lead and strontium isotopic measurements to over 50 individual tessera recovered from multiple offerings at the Templo Mayor. We directly compare these measurements to the lead and strontium isotopic compositions of turquoise from over 16 mines across the southwestern U.S. and northern Mexico to determine if there is any isotopic evidence to support the hypothesis that Mesoamerican societies acquired turquoise through long-distance trade. In addition, we also consider the isotopic ratios of the turquoise tesserae in the context of existing geological and geochemical knowledge of mineral deposits throughout Mexico in order to indirectly evaluate the possibility that the turquoise may have come from undocumented mines or outcrops within or on the periphery of the Aztec empire. These data, which represent the first isotopic measurements of Mesoamerican turquoise, provide an alternative perspective on turquoise procurement in Postclassic Mesoamerica, and should spur future research on the topic of turquoise mining and exchange in the Prehispanic Southwest U.S. and Mexico.

BERDAN, F., 1987. The Economics of Aztec Luxury Trade and Tribute. In: BOONE, E. (Ed.) *The Aztec Templo Mayor*. Dumbarton Oaks Research Library and Collections, Washington D.C., 161-183.

## **5 Microarchaeology of Floor and Wall Plasters from the Aceramic Neolithic Site of Aşıklı Höyük, Turkey**

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Aşıklı Höyük is an Aceramic Neolithic tell located in the Central Anatolian plateau. The site presently contains three major occupation levels with the oldest excavated deposits in Level 4 dating to ~10 ky BP. Residential structures are present in all three levels. Level 2 contains both a residential sector and a “special function” area. Oriented block samples of floor and wall plasters were collected from residential structures in Levels 4-2, and “special function” structures from Level 2. The blocks were impregnated in polyester resin and processed into petrographic thin sections for micromorphological and Fourier transform infrared microspectroscopy ( $\mu$ -FTIR) analyses. The remaining portions of the blocks were first analyzed for elemental composition using microscopic x-ray fluorescence ( $\mu$ -XRF), and later, individual (0.5-2 mm) plaster layers were micro-drilled for stable carbon and oxygen isotopic analyses. Thin section micromorphology and FTIR mineralogical analyses of floor and wall plasters from both areas of the site indicate that plaster compositions ranged from pure lime plaster to pure mud plaster. Mud plasters were tempered with plant fibers and range in color from brown to red. Ashes are also present within or on top of floor repaving layers. Stable carbon and oxygen isotopic measurements indicate that carbonates used to manufacture some plasters source from a local lacustrine limestone. This source is supported by micromorphological observations of partially slaked lime containing *Chara sp.* fossils. Slaked lime plasters are present in the earliest phase of occupation of the site. Despite standardization in building architecture, plaster floor manufacturing techniques are variable throughout the site and between construction phases.

## **6 Characterization of Plasters on Stone and Mud Walls From Ancient Pella**

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The city of Pella in Western Macedonia, Greece, was an important centre of civilization from 400 BC up to 168 BC. During that time it was the capital of Macedonia, which had been transferred from Aegae. The city served as the seat of Philip II of Macedon, ruler of the unified Greek states, and Alexander the Great who reached the Siberian steppes, Hindukush River and Egypt, and created the Hellenistic World. The aim of this work was to characterize the plasters and to identify the raw materials and mixtures used in the manufacture. In addition, the pigment materials and techniques of application were also investigated and characterized. The samples consist of a series of plasters on stone and mud walls from different areas and rooms of the Ancient Agora of Pella. Some of them have painted surfaces in red, yellow, and black colours and some are throughout red or grey.

A range of techniques and methods were applied for the examination and analysis of plasters and pigments: 1) Low and high power optical microscopy for the examination of the fragments as received and at polished cross-sections in order to recognize minerals and identify the layering structure of the plaster and pigments, 2) Analytical Scanning Electron Microscopy (SEM-EDXA) for the examination of the micromorphology and chemical composition (overall and particular) of plaster layers and pigments, 3) Thin-section analysis on selective samples for recognizing specific crystallization and dissolution features, 4) X-ray powder diffraction (XRD) to identify the molecular structures of minerals when necessary, 5) Micro-chemical analysis to identify the presence of calcium alumina silicates and water soluble salts, 6) Standard wet chemical analysis, and 7) Grain-size analysis of the non-carbonate inclusions after full dilution in acid of the plaster fragments.

The combined results indicated that the technologies used for making the stone-wall plasters and mud-wall plasters were quite different from each other. Similarities in the technology used were found between some stone-wall plaster samples, but even then the type of inclusions added was different. A rather surprising result was that although samples no. 1, 2 and 3 come from the same building in the Pella Agora, they were made in a different way regarding both the microstratigraphy of the layers applied, and in the mixture used. Even the thicknesses of the plasters were different. Regarding the pigments the artist's palette included red ochre (iron oxides) and yellow ochre (iron hydroxides) as well as charcoal black for the black and grey colours. Traces of lead were detected in the red iron oxides.

## 7 Geology, Petrography, Mineralogy, Geochemistry of Natural Fe-based Pigments from Verona Province (Italy)

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Verona province (NE Italy) played in the past and till the 1950s an important role in the exploitation, trading and processing of natural Fe-based pigments. Celadonite from *Monte Baldo* was well known and popular many artists and painters (Grissom, 1986).

Despite the vast availability of yellow, brown, orange, red and black Fe-based pigments in the studied area, only a few researches were carried out in the past (Federici, 1948) and in the recent years (Cavallo & Zorzin, 2008; Zorzin et al., 1992; Zorzin, 2005).

The samples coming from spots, mines representing the filling of paleokarst caves and deposits associated with volcanic terrains, were analyzed by means of optical microscopy (OM), X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF).

Goethite, Hematite and Goethite plus Hematite are the colouring minerals detected in the yellow and red ochres. Accompanying minerals are related with the host rocks and with the processes forming the earthy pigments. The association Hematite, Kaolinite and Anatase is linked with the alteration of basic rocks (*Val d'Ilasi*); Goethite is mainly associated with dolomite and subordinately calcite in the *San Bortolo* outcrop whilst the association Goethite plus Quartz is typical in the samples coming from *Ponte di Veja*.

Black earths samples coming from the area close to the well known fossiliferous district in *Bolca* are associated with eruptive materials (basalts and basaltic lava, basaltic tuffs and basaltic breccias). Calcite is associated with Montmorillonite; Pyrite is an accessory mineral and amorphous or very poorly crystalline Fe-oxides/hydroxides complete the sequence. Under OM carbon particles were detected as well. According to the composition these materials are black shale.

The research allowed a precise characterization of the raw materials used as pigments since Roman times; minerals assemblage reflects the geological environment and it could be used as marker for provenance studies.

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**8 A Mineralogical, Petrographic and C-O Isotopic Approach for the Provenance Determination of the Raw Materials from the Kastro-Palaea Building Constructions, in Volos of Thessaly, Central Greece**

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The "Kastro-Palaea" archaeological site, at a still inhabited hill in the old part of Volos city in Thessaly, presents a multi-temporal stratigraphic continuity since the Middle Helladic period and the Mycenaean times (2<sup>nd</sup> mil. BC) until today. In this study we focus on the determination of the provenance of the diachronous raw materials which were used for the building constructions. The applied methods include optical microscopy, X-ray diffractometry and C-O isotopes.

Thirty eight samples of the rock building materials, such as marbles, travertines and oolitic limestones, were collected from the Mycenaean settlement, the Archaic temple, the Roman edifices and the Byzantine fortification, all situated on the "Kastro-Palaea" hill. Potential sources at the adjacent areas of Volos, such as the Pilion mountain, as well as other areas in Thessaly, were sampled for possible correlations with the studied building materials. In addition published results were used for establishing data-bases of all the applied methods. Thin sections were prepared in order to investigate the mineralogical composition and the petrographic features and verify the different rock types of the materials used. The C-O isotopes of the carbonate stones confirmed with a high accuracy the locations of the sources.

The first results enable us for a sufficient determination of building material provenance and reveal a new material source of travertine in the nearby area, which was not referred before. The analyses confirm the geographic locations from which the building materials of the "Kastro-Palaea" hill originated, identifying a material distribution throughout Thessaly.

**9 Progress of Archaeomagnetic Dating in Western Europe: Examples from Sites in Belgium**

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The geomagnetic field varies in space and in time. At present, the spatial variation is well known by direct observations. However, instrumental measurements of declination (D) go only back to the XVI<sup>th</sup> century, for inclination (I) to the XVII<sup>th</sup> century and for intensity (F) to the first half of the XIX<sup>th</sup> century. Fortunately, baked clays and solid rocks record the field direction and intensity during the cooling process under the form of a remanent magnetization. Many field directions for the last three millennia are available yet in Europe, based on the thermoremanence (TRM) of independently dated baked clays uncovered in archaeological sites. They allow constructing regional standard secular variation (SV) diagrams, visualizing past field directional changes and that can be used to date baked clays that remained in situ after heating. In Western Europe, standard diagrams for I and D have been published for several countries, the earliest ones for France and the United Kingdom, based on data reduced respectively to Paris and Meriden as reference localities.

For archaeomagnetic dating of baked structures discovered in Belgium we refer to the standard diagrams of France for the last three millennia that were obtained on dated baked structures found mainly in France (Gallet et al., 2002). This is justified as the data include Belgian sites and because of the proximity of the reference locality Paris. For certain time periods these reference SV diagrams enable us to provide archaeomagnetic dates with a precision better than half a century. Several examples, covering different time periods, will be presented for Belgian sites.

An archaeomagnetic investigation consists of the following steps: (a) sampling of oriented burnt materials that remained in situ after burning; (b) measurement of the remanent magnetization; (c) randomizing of remanence components acquired after baking by partial alternating field or thermal demagnetization; (d) calculation of the mean direction and comparison with the reference SV curves.

The precision of an archaeomagnetic date depends, besides the quality of the record and the precision of the reference SV curves used, also on the rate of field changes, the presence of non-dipole components and the distance between the examined site and reference locality. We plan to construct standard SV diagrams for Belgium, based on data available within an area of 500 Km radius centered on Uccle (Brussels).

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## 10 New Perspectives on Amino Acid Racemisation Dating: Breaking the Egg

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A robust and secure chronological framework helps develop a clearer picture of early human evolution and dispersal. Amino acid geochronology is able to span the whole Quaternary and can be applied to a range of common materials which are directly related to the human occupation of an archaeological site: mollusc shells and ostrich eggshells. These are also preserved in sediments which accumulated as a response to global climatic pulses, during the Pleistocene and beyond. Therefore, amino acid geochronology has the potential to be widely applicable to the chronology of human evolution, as well as to the geological record. However, for the use of amino acid racemisation (AAR) as a reliable dating tool, analysis of proteins from a closed system within fossils is vital. The intra-crystalline fraction within ostrich eggshell (Brooks et al., 1990), and more recently from terrestrial and marine molluscs (Penkman et al., 2008; Demarchi et al., 2011), have been found to provide robust closed system protein, allowing significant increases in the resolution and reliability of AAR geochronology. Our research has focused on building chronological frameworks on a wide spatial and temporal scale. Beginning from the Lower Palaeolithic in Northern Europe, we are making our way down to the Mediterranean rim, focusing on key cave sites such as those in Gibraltar and Southern Spain, Libya and Morocco. Whilst mollusc shells are commonly found as sub-fossil remains in these sites, ostrich eggshell (OES) is the most common biomineral found in the African archaeological record: early humans used them as water containers, as personal ornaments and engraved them as a form of symbolic communication. OES is also known to be one of the best substrates for AAR dating – the ultimate egg timer. Here we present novel results on the application of AAR dating to OES from the sites at Pinnacle Point, South Africa, where one of the earliest evidences for modern human behaviour has been recently unearthed (Marean et al., 2007). By combining AAR dating, stable isotopes analyses and state-of-the-art mass spectrometric techniques, we are beginning to understand the complex network of factors driving protein breakdown in OES; this is allowing us to build a clearer chronological framework for one of the crucial events in human evolution: the appearance of modern behaviour.

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## 11 Rehydroxylation (RHX): A Universal Method for Dating Archaeological Ceramics

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Following removal from the kiln, fired clay ceramic begins to chemically combine with atmospheric moisture (rehydroxylation). This results in a super-slow, progressive increase in mass which occurs in two distinct stages. The RHX rate constant is defined as the gradient of the linear second-stage mass versus (time)<sup>1/4</sup> graph and is unique to any particular material (Wilson et al., 2003). Following reheating at 500°C, which removes all the chemically combined water (dehydroxylation), the material once again gains mass according to the (time)<sup>1/4</sup> law and this occurs at the same rate as in the freshly fired material (Tosheva et al., 2010). This is the basis of RHX dating (Wilson et al., 2009): the mass of water removed at 500°C is measured; from the RHX rate constant the time that it would take to recombine with that mass of water is calculated and this is equivalent to the age of the sample.

The two stage form of the data is explained. The effects of environmental conditions on the RHX rate constant are examined. It is shown that the RHX reaction is temperature dependent and that it obeys the Arrhenius rate law; it is shown that the RHX reaction is completely independent of relative humidity.

The implications of these findings for RHX dating methodology and the potential of widespread application to archaeological ceramics of all periods are discussed in detail. Recent results from a validation study of known dated materials are presented.

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### 12 Bayesian Modelling of an Absolute Chronology for the 18<sup>th</sup> Egyptian Dynasty

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The establishment of an absolute chronology for Ancient Egypt is an ambition that concentrates efforts of scholars since the beginning of Egyptology. The present relative chronology was built thanks to historical documents and archaeological evidences. Nonetheless, going ahead to fix absolute chronological points is making difficult because the discount of the years restarted at the year 1 at the beginning of each reign. Thus, the only way to get an absolute chronology would be to know the exact succession of all the kings and their precise length of reigns.

Only a couple of astrophysical points and synchronisms listed in texts have given anchor points that have led to establish the backbone of an absolute chronology.

At first, we will see how we can re-calculate these anchor points by using Sothic dating and modelling lunar dates with a Bayesian approach. In particular, a Sothic Equation is engraved on the Elephantine Calendar. The study of the archaeological context on which this document was discovered led to precise during which period this observation was done, according to the 54 years of reign of Thutmoses III.

Then, a hundred radiocarbon measurements were carried out at the Laboratoire de Mesure du Carbone 14, on Egyptian short life materials (textiles, plants, leaves, twigs) sampled at the Louvre Museum (Département des Antiquités égyptiennes). Basketries found inside tombs excavated in the Eastern Cemetery of Deir el-Medineh and archaeologically attributed to Thutmoses III were first analyzed. In particular, this study allows to propose a reconstitution of the chronology of these tombs. Flower bouquets found inside the Sennefer's tomb of Deir el-Medineh were secondarily dated. With a Bayesian approach, these analyses were combined with the known succession and length of reigns. Kinds of distribution's laws expressing lengths of reigns were chosen following our historical state of knowledge. Sothic and lunar dates were finally incorporated as a *prior* in the model, to constrain radiocarbon data by archaeological evidence. This led to the proposal of an absolute chronology for the 18<sup>th</sup> Egyptian dynasty.

Lastly, the agreement between new results and archaeological evidence as well as the question of the extension of such a model to older periods will be discuss.

### 13 AMS Radiocarbon Dating of Ancient Iron Alloys

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The dating of archaeological iron objects is of particular interest in studies relative to the history of steel industry and the technology of ancient societies. Radiocarbon dating, largely used for organic samples, can also be applied to the ancient iron alloys thanks to the carbon entrapped in the ferrous matrix during the reduction process. Carbon is provided by wood charcoal used as fuel in the process at least until the 19<sup>th</sup> century.

If we assume that relative young wood was generally chosen, we can deduce that the carbon entrapped in the iron is contemporaneous with the manufacture of the ferrous object. First attempts of dating iron has been performed in the nineties, but the need of high carbon amounts (at least 0.5 g of carbon) discouraged for further studies. With the Accelerator Mass Spectrometry, 1 mg of carbon only is now required, offering new perspectives to materials with low carbon content such as iron. Nevertheless, others limitations appeared in radiocarbon dating of ferrous alloys. The material is highly heterogeneous and presents various carbon concentrations from less than 0.2 % of carbon in soft irons to more than 0.8 % of carbon in the hypereutectoid steels. Furthermore, iron ores in the form of iron carbonate (siderite  $\text{FeCO}_3$ ) contains carbon of geological origin that can therefore age the radiocarbon date.

A special feature of our approach is to undertake a metallographic study of the object to determine the distribution of carbon and target the area's most carburized. To validate our methodology, archaeological objects of different carbon contents and of known age were dated. In a second part, the question of carbonated ore has been studied on iron alloys elaborated from experimental smeltings with contemporary charcoal and siderite. The radiocarbon analyses bring new elements to progress on this subject.

Finally, as part of the problem of the iron use in medieval Gothic buildings, radiocarbon datings were carried out on reinforcement buttresses of the Beauvais and Bourges cathedrals (France). The results significantly refine our vision of the iron use in this type of monument.

## 14 Quantitative Approach to Ancient Diet Reconstruction and Reservoir Effect Correction

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Stable isotope analysis is one of the main analytical tools used within archaeological research for ancient human diet reconstruction. However, several issues need to be addressed so that a truly quantitative diet reconstruction becomes possible. A three step approach is hereby proposed:

1) Characterization of the isotopic baseline, i.e., identifying possible food sources and characterizing corresponding isotopic signals. 2) Use of multiple isotopic signals for which dietary routing mechanisms have been well established. This implies knowledge on the dietary routing of food macronutrients (carbohydrates, lipids, and proteins) and biochemical components (e.g. amino acids). 3) Use of statistical models that allow integration of all sources of data variability (e.g. uncertainty in isotopic enrichment, isotopic variability in the food source).

An illustrative example of a quantitative approach to ancient diet reconstruction is hereby presented. Individual human remains, in an excellent state of preservation, were collected from the medieval cemetery of Oude Markt in Vlissingen (The Netherlands). Stratigraphically associated fish and animal material located in an ancient cesspit permitted a characterisation of the isotopic baseline. Archaeological data was complemented with historical information on dietary habits of medieval Dutch coastal populations. Compiled information was used to define food groups (e.g. cereals, land animals, freshwater fish, and marine fish). Estimates of food nutrient composition and isotopic variability relied on previously published data. A Bayesian mixing model was designed to take into account the dietary contribution of different food nutrients and components. For each measured isotopic signal ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  in bone collagen,  $\delta^{34}\text{S}$  in collagen's methionine, and  $\delta^{13}\text{C}$  in bioapatite) weight contributions of the different food nutrients and/or components were determined from an analysis of compiled results of controlled animal feeding experiments (e.g. Warinner & Tuross 2009, Froehle et al. 2010). Model output provided, for each individual, probability distributions and confidence intervals on the consumption of each food group. These results were used to provide an estimate of each individual's reservoir effect. The reliability of such an estimate was assessed by comparison with age offsets observed between collagen radiocarbon dates and the dating (dendro and radiocarbon) of wooden caskets in which the individuals were buried.

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**15 The Emergence of Tin Bronzes c. 7000 Years Ago**

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The earliest tin bronze artefacts in Eurasia appear c. 5000 years ago, and their production has been tentatively linked to the emergence of copper alloying in the Near East. Although most archaeologists agree that tin ores for alloying were probably coming from Afghanistan (Pigott, 1999), the widely established model of early bronze making by alloying remains difficult to demonstrate, and the emergence of tin bronze is still a challenging issue in the evolution of Eurasian metallurgy. The recent excavations in Pločnik, a Vinča culture site in south Serbia, yielded a tin bronze metal sheet in the vicinity of a copper workshop at the site. This object is securely dated to c. 4650 BC, which makes it the earliest known tin bronze object to date. Here we present results of compositional analysis which indicate its origins not by alloying, but from smelting a complex tin ore, such as stannite, while microstructural examination demonstrates the craftsman's awareness of the material properties of the newly acquired metal. We suggest that decisions on selecting complex copper and tin ores for both copper and bronze smelting were based on the appeal of black and green ores, a visual aspect already confirmed as decisive for ores smelted to extract copper, c. 5000 BC (Radivojević et al., 2010). Moreover, this discovery prompted revisiting fourteen insufficiently contextualised Chalcolithic tin bronze artefacts from the Balkans, which set them analytically firmly into the 5<sup>th</sup> millennium BC. These bronze artefacts from the 5<sup>th</sup> millennium BC Balkans extend the record of bronze making by nearly two millennia, challenging the conventional narrative of the evolution of metallurgy.

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## 16 Mineralogical and Isotopic Characterization of the Late Chalcolithic Slags from Bressanone/Brixen (Northern Italy)

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In 2008 the excavation activities for the constructions of the Bressanone/brixen (North-Eastern Italy) west ring-road allowed recovery of an archaeometallurgical site. The finding of Late Chalcolithic slags in the site is particularly interesting because it follows the recent discovery of two nearby coeval occurrences: Millan/Milland and Gudon/gufidaun (Colpani et al., 2009). In order to investigate the nature of the extractive process and the type and origin of the minerals used at Bressanone/Brixen, fifteen slag samples were analyzed. The slags are small, mostly with irregular subrounded shape; only three slags are nearly flat with thickness of 1-2 cm. Polished thin sections were analyzed by transmitted and reflected light optical microscopy, textural and chemical analyses were obtained by SEM-EDS, mineralogical phases were identified by XRPD and semi-quantitatively evaluated by the Reference Intensity Ratio method. The samples show rather heterogeneous textures and similar compositions, with features encompassing: high amount of unreacted quartz, often associated with the high temperature polymorph cristobalite; abundant fayalitic olivine displaying euhedral to skeletal morphology in the same slag; widespread occurrence of unreacted or poorly reacted Cu-sulphides; ubiquitous presence of variable amounts of Fe-oxides; widespread occurrence of small copper prills. The chemical analyses show a substantial Zn content in the Bressanone/Brixen slags, as previously observed in the Millan/Milland and Gudon/Gufidaun materials. The immature slags from Bressanone/Brixen (partially molten, not fully reacted) suggest the use of a low efficiency copper smelting process, similar to those observed at Millan/Milland and Gudon/Gufidaun, and in line with the "Chalcolithic process" defined by Bourgarit (2007). Pb isotope ratios were measured on 10 slag samples from the 3 sites and the results, compared to the geochemical database of the alpine Cu-mineralisations (Alpine Archaeocopper Project - <http://www.geoscienze.unipd.it/aacp/welcome.html>) allow safe identification of the ore source in the post-Variscan polymetallic vein deposits of the Trentino-Alto Adige area (Nimis et al., 2011). Specifically, the isotopic data are fully compatible with those measured on the ores from the relatively near Montefondoli/Pfunderer Berg mine (Chiusa/Klausen, Bolzano/Bozen). The isotopic and trace element data measured on a raw copper fragment found in association with the Millan slags are discussed.

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## 17 Arsenical Copper in Southwest Asia: Alloying Technology and Intentionality Determined by the Chemical Analysis of Production Remains

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While arsenical copper has long been recognized as the alloy of choice of many late Chalcolithic and Early Bronze Age cultures of Southwest Asia (Charles, 1967; Heskel, 1983; Craddock, 1995), little is as of yet known about its production technology. For decades scholars have debated the intentionality of the alloying process, basing their inferences largely on finished objects often lacking archaeological context (Eaton & McKerrell, 1976). Not only is this dataset somewhat dubious chronologically and contextually, but it also holds limited information on the primary smelting and alloying stages of arsenical copper production. However, the analysis of finished artefacts is largely due to the scarcity of excavated archaeological sites containing primary smelting and alloying remains of arsenical copper production, which has meant that little other material has been available for study. However, recent discoveries at two archaeological sites dated to the mid to late 4<sup>th</sup> millennia BC (Schoop, 2009; Vatandoust et al., 2011) are providing new information on the topic and should shed new lights on the various technological pathways by which ancient metalworkers produced arsenical copper. Initial results of archaeometric research focusing on the production remains (slags, technical ceramics, furnace fragments, ores, and tailings) from the two sites of Arisman in West Central Iran and Çamlıbel Tarlası in Central Turkey will be presented here. The analysis, conducted mainly using XRF and SEM-EDS, has yielded conclusive evidence of the intentional alloying of copper and arsenic using both natural and artificial arsenic bearing minerals. In addition, it will be shown that the alloying process was conducted both as a single step process as part of a co-smelting operation as well as a separate crucible-bound operation. These results will be presented within the context of Late Chalcolithic and Early Bronze Age cultural shifts towards craft specialization and urbanization and explore the implications of such an advanced metallurgical technology on early societies. In addition, although the two sites are widely spaced geographically, it will be shown that comparing them highlights both their differences and striking similarities in their approach to the production of arsenical copper, which may be suggestive of cultural interactions between the two regions.

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## 18 Lung-Powered Copper Smelting of the Sican Period (900-1375 CE), North Coastal Peru

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The smelting of copper with blowpipes during the Middle Sican Period was previously reported by Shimada and Merkel (1991) and Epstein (1993) from excavation at Cerro de Los Cementerios near Batan Grande, Lambayeque Province. Further banks of well-preserved furnaces, and other evidence of smelting and subsequent processing, were recently noted during systematic field surveys, led by Hayashida, in the Pampa de Chaparri, about 30 km west of Batan Grande. We report here on the analysis of ores, slags, metal droplets and metalworking scrap recovered during survey. Bulk analysis of slags was by x-ray fluorescence, microchemical analysis of metals and ores by scanning electron microscopy and electron microprobe, and mineralogical identification by optical petrography, ore microscopy and Raman microscopy.

While the smelting technology is very similar to that reported for Cerro de los Cementerios, and may have used the same ores, the secondary processing technique used in the Pampa de Chaparri is distinctive. Separation of copper from slag in the furnaces was evidently relatively poor, so crushed slag bearing small prills was piled on concave ceramic platters and heated from above to promote further separation of copper and slag.

The copper produced is arsenical, and we will consider in some detail whether the arsenic derived from the use of arsenic-bearing copper ores, or alternatively from the deliberate addition of some arsenical mineral to the furnace charge.

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## **19 When Material Science Meet Ceramic Sociology: An Archaeometallurgical Study of Crucibles from Mapungubwe, South Africa**

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Mapungubwe (AD1220-1290) is perhaps the first known state system in southern Africa. It had a vibrant economy that was anchored by a robust ferrous and non-ferrous metallurgical industry. Recent work on the metallurgy of Mapungubwe has however focussed on impressive and spectacular objects such as the golden rhino (Miller et al., 2000). This enchantment had the consequence that less glamorous artefacts such as crucibles were marginalised. A fair amount of crucibles was recovered from the top of Mapungubwe Hill. Compared to crucibles documented elsewhere in the world that were custom made combustion vessels (Martinon-Torres et al., 2008), Mapungubwe crucibles are outwardly indistinguishable from domestic pottery in terms of surface finish and decoration. Inwardly, they are slagged and often contain cuprous prills. This paper reports the results of archaeometallurgical studies carried to characterise the crucible and domestic pottery mineralogy and chemistry. Thin sections of the crucibles and domestic ceramics were studied in transmitted polarised light. Polished blocks of the same specimens were studied in the QEMSCAN to identify the minerals and to understand their chemical composition. Additional SEM-EDX studies were carried out to cross check the accuracy of output from the QEMSCAN. The results revealed that the crucibles were used for making tin bronzes. This makes Mapungubwe one of the earliest known bronze making sites in southern Africa. Comparisons of modal mineralogy in the QEMSCAN revealed that the clays used to make crucibles were similar to those used for making domestic pottery. However, three types of clays with varying degrees of kaolinite, quartz and ilmenite were detected, pointing to possible differences in clay sources. The sociological implications of these results are far reaching. Ethnographies from the 19<sup>th</sup> century indicate that metalworking and pottery making were gendered pursuits. Men invariably worked metals and were responsible for making infrastructure and tools of their trade. The same applied to pottery making which was the preserve of females. Taboos prevented women from metal working and men from pottery making. The presence of typical female material culture, pottery in a male activity at Mapungubwe suggests that the gender division of labour may not have been as strict as currently believed. In fact, women played an important role in metallurgy as makers of crucibles. This observation dismisses as simplistic notions of gender exclusivity in Iron Age technological activities currently propounded by some Iron Age researchers.

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## **20 Why is there a Bronze Age from the Atlantic to the Pacific? Analysing the Earliest Appearance and Widespread Adoption of Copper Alloys and Production Techniques across Eurasia**

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From Ireland to Thailand, archaeologists continue to employ the concept of a Bronze Age when classifying societies in the 3<sup>rd</sup>, 2<sup>nd</sup> or 1<sup>st</sup> millennium BC. The societies which fall under this classification are exceptionally diverse, ranging from the urban kingdoms of Egypt or Mesopotamia to the nomadic pastoralists of the Eurasian Steppes and to the villages of Europe. Despite the production, use and deposition of tin-bronze continuing to be a major regional research focus, the pan-Eurasian metallurgical perspective has been largely ignored. This paper analyses the evidence for the earliest appearance and subsequent widespread adoption of arsenical copper and tin-bronze across an area from the Atlantic to the Pacific. Where possible, it also analyses the pan-continental earliest appearance and subsequent widespread adoption of the identifiable copper alloy production techniques such as casting of sockets and long blades, lost-wax casting and complex sheet metalworking. Contrasting the earliest and widespread dates in each region throughout Eurasia allows the investigation of the long-held ideas that the Bronze Age at its widest spatial and temporal scale represents a period of both increasing metallurgical innovation and interaction. It also allows the investigation of the degree of distinctly regional metallurgical innovation within the broader pan-continental framework.

## **21 The Metallurgical Inventory from Tell Chuera (Syria) - A Direct Comparison between Quantitative pXRF and Qualitative WDS data**

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Tell Chuera, located in ancient Upper Mesopotamia (NE Syria), is a major site and a long term excavation. Therefore, the site offers a wide range of metallurgical material, not only in terms of artefacts but also in terms of different materials from various archaeological contexts. More than one hundred metal artefacts and metallurgical finds have been studied via on-site qualitative pXRF analysis. Additionally, a limited number of samples had been taken for invasive quantitative WDS analysis. pXRF analysis became extremely popular within the last 10 years due to not only its possibility to analyse on site without the need to sample, but also due to limited costs especially in comparison to lab-based methods. While we are roughly aware of the limitations of both methods in analysing metals, hardly any study has been undertaken to directly compare pXRF analysis and lab-based compositional analysis. However, especially in times of a decrease in sponsorship for archaeological work it seems essential to be aware of advantages and limitations of each method for a best possible choice. This study points out the specific concerns of each methods and, more importantly, examines in which way they both support each other to answer archaeological questions.

## **22 Minoan Metal Vessel Manufacture: Reconstructing Techniques and Technology with Experimental Archaeology**

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Metal vessel production flourished in Crete during the Protopalatial, Neopalatial and early Postpalatial periods. Almost all of these vessels were hammered, largely from tin bronzes, and include various pans, cauldrons, hydrias, pitchers, lekanes, lamps and cups. Vessels in precious metals are also extant from Crete, but not in large numbers.

The equipment and processes used to manufacture these vessels have not previously been investigated in detail. The aim of this study was to reconstruct the process used to create these vessels. An interdisciplinary approach was taken, combining archaeological research with practical metalsmithing. Initially, a comprehensive study of Minoan metallurgical technology was carried out and some of the vessels examined for evidence of their manufacture. Subsequently, Minoan metallurgical equipment such as hammers, an anvil and a hearth were replicated and used to create Minoan vessel forms including bowls, a hydria and a lekane.

The results indicate that the simple tools found at many Minoan metallurgical sites are very effective for vessel making and that unhafted stone hammers, a common Minoan tool, are particularly suitable for making Minoan vessel forms. The process of creating these forms revealed that, unlike those of contemporary cultures in the region, Minoan vessels were formed predominantly by sinking, where the vessel is formed by hammering from the inside, and that only a limited amount of raising, working the vessel from the outside, was carried out. This appears to be a result of technological limitations.

## 23 A Preliminary Report on the Study of Cypriot Bronzes Dating to the Early Iron Age

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The present preliminary study concerns the interdisciplinary investigation of more than 150 archaeological bronze artefacts, coming from the necropolis of Palaepaphos-Skales. The site is situated near the modern village of Kouklia, in the Paphos district, on the South-west coast of Cyprus. The necropolis, which was in use for many centuries, starting from the 11<sup>th</sup> century B.C. down to the Classical and Hellenistic period was excavated by Vassos Karageorghis (Karageorghis, 1983). His excavations brought to light some very important metal artefacts. These include several types of bronze tools such as knives, saws, chains and needles, weapons, such as spearheads, utensils and vessels, such as, *obeloi*, tripod stands, strainers, cauldrons and bowls, and ornaments such as pins, fibulae, rings, and ear-rings. The material is published and there have been some specialized studies on the tripod stands and the metal vessels (e.g. Papasavvas, 2001, 2004; Matthäus, 1985). However, this important assemblage of Early Iron Age Cypriot metalwork has never been analysed as a whole and thus very little is known about the alloys used to produce these artefacts or indeed any bronzes dating to this period. For the specific study, a portable XRF was used for the determination of the chemical composition of the bronze artefacts, after the removal, using a torque tool, of a small surface area (3x3 mm) of the corrosion layers. Quite interesting and also important, is the comparison of the “clean”, after the removal of the surface corrosion products, and “unclean” bronze surface of the studied artefacts. This comparison showed significant differences in the chemical composition of the alloys. In some cases and for some elements, such as Sn, this compositional difference is quite impressive. The presence of Fe in significant amounts in the “unclean” surface was also noted and is believed to be probably due to the taphonomic conditions and the incorporation of soil in the corrosion layers. The aim of this research project is the chemical analysis of the objects in order to ascertain the types of alloys used and whether there is a differentiation in composition depending on the type of object. Furthermore, as this is part of a diachronic study of Cypriot metalwork undertaken by the authors, possible changes in the composition of copper alloys used in different periods on the island will also be investigated. One of the issues that will be of particular interest will be the determination of the tin content in the bronze artefacts of the specific period in Cyprus. It has been argued in the past that due to the collapse of the Late Bronze Age palace societies a significant disruption in trading networks had occurred. This may have led to a shortage in the supplies of tin in the Eastern Mediterranean including Cyprus, and eventually to the transition to the use of a new metal – iron. Whether this model holds true remains to be seen.

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## 24 Oodles of Copper for the Chinese Emperors: The Case of Sijiawan (四家湾) Plant

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The Tang (618-907 AD) and Song Dynasties (960-1279) were largely a period of progress and stability with special flourishing of Chinese culture (poets, painters) as well as science, technology and medicine. Metal plays a crucial double role in this brilliant period: on the one hand metal continues to play its usual economic part present in tools, weapons, coins and other commodities; on the other hand, the most important attributes of the lifestyle of the aristocracy are made of metal, with the particularity that there is virtually no association of precious metals -silver and gold- with the elite (Marshak, 2004), since wealth, prosperity and cultural traditions were reflected in the use of bronze and jade.

Sijiawan (四家湾) mining and smelting site -located south-eastern of Shanxi Province, Central China- offers a new picture of this large consumption of copper and copper alloys. The project explores the foundations of this metallurgy by looking at the primary production of copper, the main ingredient for bronze production (Watt, 2004), providing the first archaeometrical approach to this question.

The preliminary investigation points out a remarkably massive scale production that exploited efficiently the original sulphidic ore. Tonnes of slag, furnaces, heaps of gangue and other evidences of the metallurgical activities draw a complex industrial landscape. The focus of the study is on the remains of the production; mainly slag lining, technical ceramics and several intermediate products since these are well established as research material (Bachmann, 1982). The reconstruction of these activities is also analysed through the comparison within the description of the process collected by written sources from the Song and Ming Dynasties, specifically the *大冶赋* (Rhapsody of the Great Smelting) and the *菽园杂记* (Shuyuan Miscellanies).

Provisionally Sijiawan has been chronologically framed in the Tang Dynasty based on the typology of domestic pottery but the observed ample volume of activities carried on there points out to prolonged exertions in time. Radiocarbon dates results are expecting at the time of writing the current abstract.

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## 25 Making Weapons for the Terracotta Army: Technology, Standardisation and Logistics

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The mausoleum of the First Emperor of China is a superb example of the logistical organisation and immense productive forces under the command of an emerging imperial power. Built in just four decades in the 3rd century BC, it covers 56 square kilometres and encases a funerary pyramid together with numerous pits of life-sized servants, acrobats and musicians; wildlife scenes, carriages and much more. The Terracotta Army is the most extensively investigated area. Excavations in Pit 1 have so far recovered 2,000 ceramic warriors heavily armed with hundreds of bronze weapons or parts thereof, such as crossbow triggers, swords, halberds or spears, in addition to over 40,000 arrowheads.

Here we report on a project focused on the crafting methods and logistical organisation behind the manufacture of the numerous weapons buried with the warriors. The methods employed involved wide-ranging typological and metric studies, archaeometallurgical analyses and spatial statistics. The analytical techniques employed included optical and electron microscopy and microanalysis (SEM-EDS, WD-EPMA) in addition to extensive use of portable XRF.

This work has allowed us to gain insight into several aspects of the technological skills of the Qin weapon-makers, including the casting of bi-metallic swords, the application of surface tinning for decorative purposes, or the earliest large-scale use of rotary devices for sharpening blades.

At a more fundamental level, it has been possible to reconstruct the model of craft organisation that allowed the efficient production of such a large number of weapons in a relatively short period. Based on the identification of individual chemical batches corresponding to single crucible loads, combined with metric and spatial analyses, we propose that the weapons were manufactured in a cellular production system. Unlike single flow-line production and assembly lines, cellular models engaged smaller groups of semi-autonomous, versatile workers, that produced finished items (such as a quiver containing 100 finished arrows) instead of unfinished parts (such as an arrowhead tang). It appears that the army pit was divided in 'activity areas' that were allocated to various cells operating in parallel. This system was more adaptable to changes in the masterplan but it required sharing models to ensure standardisation. Yet the standardisation achieved across the whole weapon assemblage is outstanding, as demonstrated by low metric coefficients of variation.

This project exemplifies the role of archaeological science in reconstructing organisational aspects of imperial craft production systems, as well as new avenues for the application of portable XRF.

## 26 A Multidisciplinary Investigation of Bloomery Iron Smelting

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The basic principles of bloomery technology employed for the manufacture of iron before the medieval period are well established (eg Pleiner, 2000). The technology has been studied through the furnace structures which survive in the archaeological record as well as the associated waste materials (slag). Considerable insight has also been gained through the study of broadly similar technologies in Africa and India (eg Schmidt, 1997). Further testing of the nature of bloomery iron smelting technology and its associated structures and waste products has also made use of experimental reconstructions (eg Crew, 1991; Tylecote et al., 1971).

This paper will explore several current research themes relevant to the reconstruction of early bloomery iron smelting through multidisciplinary analysis of materials associated with an experimental reconstruction of this process. The experimental furnace was built of clay and charged with charcoal and iron ore. We employed a relatively fast blowing rate (following Sauder & Williams, 2002) and succeeded in producing a 3.2kg bloom of iron. The post-mortem analysis of samples of raw materials, waste products and iron (at various stages of refining) making use of optical microscopy, XRF and SEM-EDS is underway. The chemical composition of SI in bloomery iron has increasingly been investigated in the hope that this will allow the identification of the site/region of manufacture (eg Blakelock et al., 2009). The chemical analysis of bloomery iron smelting waste materials has also been used to identify the efficiency of the process (eg Crew, 2000). Our analyses will contribute to both of these topical research themes.

Samples of charcoal were taken; some unused, some partially burnt and some quenched. The examination of these samples aims to identify characteristics which can then be sought in archaeological samples. The results will allow the better identification of industrial (rather than domestic) use of charcoal. Samples of the clay-built furnace have also been taken in order to determine its magnetic characteristics and the extent that these have been affected not just by the high-temperatures but also by the fact that iron has been produced inside the furnace. The impact of the results will be discussed in relation to the archaeomagnetic dating of iron smelting furnaces.

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## **27 Ore, Slag and Inclusion: Measuring Variability in the Direct Process and Assessing its Implications for Provenancing iron Using the SI Method**

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Recent investigations into provenancing iron using the slag inclusion (SI) method have yielded promising results (Desaulty et al., 2009). This paper aims to further validate the SI method of iron provenancing by addressing the issue of compositional variability. In order to establish a relationship between ore and artefact, production slag and entrapped inclusion, some issues remain to be fully addressed. What is the compositional variability within a geological ore body, within a smelt, and between SI? Here, we present the results from the analysis ore, slags and a bloom cross-section from two smelting experiments (XP61, XP90). The samples selected were six bog ores from the same geological deposit, nine production slags from a single smelt, a over 400 SI from a cross-sectioned bloom. Variability of major and minor oxides, along with a suite of trace elements, was measured using a combination of SEM-EDS, PGAA and LA-ICP-MS. This work aims to further clarify the reliability of the SI method for provenancing iron.

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## 28 Identifying the Construction Phases of Gothic Cathedrals through their Metallic Supply Using Iron and Lead Analysis by LA-ICP-MS

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Art history and written sources were for long the only disciplines used to study the construction phases of ancient buildings. Recent research on medieval architecture has shown that iron and lead are among the main construction materials used in the structure of gothic monuments. Their chemical composition has therefore already provided useful information on the quality of the raw materials used in these prestigious constructions (L'Héritier et al., 2010) and also on their provenance (Dillmann & L'Héritier, 2007). New developments in LA-ICP-MS analysis allow to include these materials within the discussion on the construction phases of these monuments, giving therefore new insights on their history. This approach was applied on two emblematic gothic cathedrals of the French kingdom: Chartres and Bourges to disentangle controversies about their early construction phases in the 13<sup>th</sup> c.

The analytical protocol aims to characterize, with the same LA-ICP-MS technique, the iron armatures placed during the construction of the monument and the lead joints used to seal them. Regarding iron, trace element composition of slag inclusions entrapped in the metallic matrix was considered to discuss their provenance, as proposed by Desaulty *et al.* (2009) and Leroy *et al.* (in press). Regarding lead, trace element characterization of the matrix was also used to define groups of different supply. The proposed protocol does not require any heavy preparation and can therefore be routinely applied for the first time to lead-based archeological samples. LA-ICP-MS analysis gathers two main advantages for this purpose: a small spot size (50-100 µm) which is required for slag inclusions analysis and a good sensitivity for trace element analysis. Thanks to the use of adequate reference materials (NIST 610 for slag inclusion, MBH 83X PR2 and PR7 for lead) the analytical precision in these two matrices is below the ppm level for a large collection of elements.

A corpus of 135 iron armatures and 90 lead joints was sampled on two specific locations of the cathedrals: the upper bays of Chartres and the clerestory chain of Bourges. Thus, almost all the available samples in these buildings parts were analyzed. The collected data allows to discuss the supply in metal of these two monuments and to propose a renewed approach on their chronology by confrontation with the archaeological observations. In the cathedral of Bourges, three different phases can be highlighted for the chain, which indicate a carefully thought-out north-south management of the building yard.

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## 29 Quantitative 3-D Mapping of Japanese Ancient Blades through Energy-Resolved Neutron Imaging

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The study of sword-forging techniques, and their time-evolution, represents one of the most interesting topics in the investigation of Japanese blade manufacturing techniques. In this work, we present novel results from a non-invasive approach to the study of five Japanese sword fragments pertaining to Koto (987-1596) and Shinto (1596-1781) times.

The studied samples are large sword fragments, broken at approximately 10-20 cm from the tang, made available through the Stibbert Museum in Florence. All of them are signed and the authorship and attribution can be accurately identified. Even though an invasive approach could have been used, given the nature of the present samples, we have deliberately chosen to apply a non-invasive technique, in order to demonstrate the validity of neutron tomography methods. Thanks to their high penetration power, neutrons represent an almost unique probe for noninvasive characterization of the bulk microscopic structure of massive metal objects (Squires, 1996; Sears, 1992). These techniques have been recently applied to the study of metal objects of archaeological (Siano et al., 2002) and historical (Grazzi et al., 2009) origin, giving detailed information on bulk properties (e.g. phase composition, texture, residual strain distribution) and allowing to obtain information on the manufacturing techniques, through identification of peculiar features related to these processes. On a different length scale, neutron tomography techniques can add useful information on the bulk conservation status (Lehmann et al., 2005) and the inner structure (when present) of the artefact (Lehmann et al., 2010). We discuss, here, the results of a neutron imaging experiment, carried out at the ICON beamline operating at the neutron source SINQ (CH), applied to the aforementioned sword fragments, using both, white and energy resolved, incident neutron beam. The tomographic reconstruction of the white beam images has permitted to identify some peculiar characteristics (e.g. slag inclusions, cracks, type of temper, alterations extending under the surface) related to the forging methods that were used by the different schools and traditions in Japan. The subsequent experimental investigation, using the energy resolved tomographic method (Josic et al., 2010) gave us the possibility of increasing the image contrast for a selected phase, taking advantage of the abrupt change of the attenuation coefficients at the so-called Bragg cut-off. We have applied this method to maximize the ferrite contrast and to map the distribution of this phase in the bulk of the measured samples.

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### 30 Stable Isotope Analysis of Multiple Tissues from Peruvian Mummified Remains: Investigating Tissue-Spacing and Dietary Life Histories

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Carbon and nitrogen isotopic analysis has been performed on multiple tissues types (dentine, bone, skin and hair) from mummified human remains from the south coast of Peru. Remains from 7 looted cemeteries (c. 100 BC – 1400 AD) in the lower Ica valley have been sampled from an area that previous research has shown to have witnessed a radical shift in food production and management over approximately 1000 years (Beresford-Jones et al., 2011a, 2011b). For forty-eight individuals at least three of these tissue types were sampled. The aim of this research is to 1) investigate to what extent the tissue spacing seen in these mummies reflects our current knowledge of  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  diet-tissue spacing; and 2) investigate if any discrepancies between these can be explained by taking the archaeological record and dietary life histories as a whole of particular individuals into account. Collagen has been extracted from dentine, bone and skin, which reflects diet at different life stages: childhood, long-term average and pre-mortem life stages. Segmental data from keratin analysis of the hair samples provides a month by month analysis of diet over a pre-mortem period representing, in these samples, between 6 and 40 months. We investigate the comparability of the three collagen sources with each other as well as the correlation between skin collagen and hair keratin which both reflect diet during the months of life. The results obtained so far from the bone-skin offset show similar patterns observed by Finucane (2007) in his archaeological population from the site of Vinchos in the Peruvian highlands. The bone-hair offset shows wide variation with little agreement with values published from modern samples (O'Connell & Hedges 2001). In some cases, seasonal dietary variation, as seen in the hair, can be used to explain the discrepancies in short turnover versus long turnover tissues, which in turn reflects the agricultural practices hypothesised for the valley. For individuals where this is not the case we hypothesise the potential factors that may have caused a significant dietary shift between early and late life or reasons as to why our understanding of tissue-spacing is limited.

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### 31 Dental Calculus: A Novel Reservoir of Health-Related Biomolecules

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Archaeologists have long been interested in the health histories of ancient peoples. However, reconstructing these histories using conventional tools can be difficult, indirect, and imprecise. Recent microscopy investigations of dental calculus have shown that this mineralized biofilm is a long-term reservoir of food remnants and bacteria (e.g., Fox et al., 1996; Linossier et al., 1996; Hardy et al., 2009; Henry et al., 2011). In this paper we present new data demonstrating that dental calculus is also a robust reservoir of ancient biomolecules, including ancient DNA.

Dental calculus samples from three individuals with evidence of moderate to advanced periodontitis ( $\geq 4$ mm attachment loss) from the Dalheim Monastery (c. AD 1100) were investigated for human and bacterial DNA. Targeted sequencing of the bacterial 16S rRNA gene reveals the preservation of a diverse range of commensal and pathogenic oral taxa, and metagenomic shotgun sequencing further characterizes periodontal infection at the time of death. Many of the bacteria detected in the dental calculus are implicated in modern cases of periodontitis, indicating long-term continuity in the etiology of periodontal disease. Today, periodontal disease affects approximately 50% of adult Europeans and is a leading cause of antemortem tooth loss (Bourgeois et al., 2007).

Given the near ubiquity of dental calculus in the archaeological record, the discovery of well-preserved biomolecules within dental calculus promises to greatly expand our inquiry into the history of periodontitis and open up new a new line of research in the field of paleopathology.

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## 32 Dairying in the Early Mediterranean Neolithic

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The origin of dairying in Europe has been subject to much debate. It has been suggested that secondary products, including milk production, only developed at the end of the Neolithic period (Sherratt, 1997). However more recently, zooarchaeological evidence has pointed to a much earlier origin, pushing back the dates to the early 8<sup>th</sup> millennium BC (Mid-PPNB) in the Near East, and the mid 6<sup>th</sup> millennium BC in Mediterranean Europe. The former has been directly confirmed by organic residue analysis (ORA), where dairy residues were identified on pottery dating to the 7<sup>th</sup> millennium BC, based on structural and isotopic characteristic of individual lipid compounds using Gas Chromatography-Mass Spectrometry (GC-MS) and GC-combustion-Isotope Ratio Mass Spectrometry (GC-c-IRMS). Residue analysis offers the only method of providing direct evidence for the presence of dairy products, which complements zooarchaeological data and adds confidence to the economic interpretation of a site when the faunal assemblage is not preserved or is too fragmented for mortality patterns to be conclusive. However, very few residue analysis studies have been conducted on early pottery from farming sites in the Mediterranean, despite the fact that the speed and nature of the spread of pastoralism through this region has been much debated and might be quite different to other parts of Europe.

To address this issue, here we present a systematic study of pottery use at early farming sites from the central Mediterranean. We have improved the isotopic criteria for distinguishing ruminant products in this region by extending the reference data set with authentic modern lipid samples, sourced from the study area. Using these criteria we have classified lipid residues extracted from early Impressed Ware vessels, excavated from Apulia (Italy; 6100-5600 BC), Malta (5000-4300 BC), Catalonia (5470-5300 BC) and Croatia (5600-5159 BC). A significant number of samples matched the criteria for dairy products, showing that dairying was an important part of the early Neolithic economy and may have been crucial in sustaining pioneering farmers as they rapidly spread through this region.

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### 33 Characterization of Maize and Manioc Residues in Prehistoric Potteries: The Contributions of GC and NIR Spectroscopy to Archaeology

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Archaeometry, a promising connection between Chemistry, Physics, Materials Science and Archaeology, constitutes a significant advance in archaeological studies. Classes of artifacts like stone objects, metal containers, coprolites and potteries can be studied, providing very useful information to archaeologists. Data obtained by chemical analyses of organic residues impregnated in pottery, for example, can give to archaeologists a set of information which can help them to verify some archaeological hypotheses as well as understand different aspects of prehistoric populations. Maize and manioc (cassava) are typical foods of prehistoric tropical populations. So the characterization of their residues in potteries is of first interest for archaeological studies. Our group have been studied these residues by gas chromatography (GC) and by NIR spectroscopy - as a non-destructive, more sensible and faster tool to characterize residue containing pottery. Initially, an extraction procedure was tested employing different solvents (hexane, chloroform and methanol 2:1 v/v and dichloromethane) to extract characteristic substances of maize and manioc. Alternatively, some laboratory-prepared samples were produced adding maize and manioc, in the ratio of 10 wt %, to pottery powders. Then, the samples were analyzed and the data were treated by bidimensional graphics, Principal Component Analysis method (PCA) and Soft Independent Modeling of Class Analogy method (SIMCA). Supplemental analysis were realized adding different maize and manioc proportions to pottery powders (0,1%, 0,25%, 0,5%, 1%, 2,5%, 5%). Moreover, some samples of historical importance (couscous kettle and manioc meal house piece) and some archaeological samples were selected to test the methodology. It was observed that among the tested solvents, chloroform and methanol (2:1 v/v) provided the best result, allowing the maize and manioc extracts identification. Employing this solvent it was possible to distinguish between potteries containing or lacking the residues and even to distinguish between potteries containing maize or manioc residues. The NIR spectroscopy results indicated a good differentiation between the simulated samples composed by ceramics and maize or manioc residues. The PCA scores allowed to differ between "organic residues free" and "organic containing" in samples and the SIMCA classification and the Coomans Graphic were sufficient to relate clearly the manioc meal house piece to manioc laboratory-prepared samples (99% Confidence Level). This approach gives a new tool for a better understanding about the Tropical Forest human life in the Prehistory. Work supported by CNPq and FACEPE.

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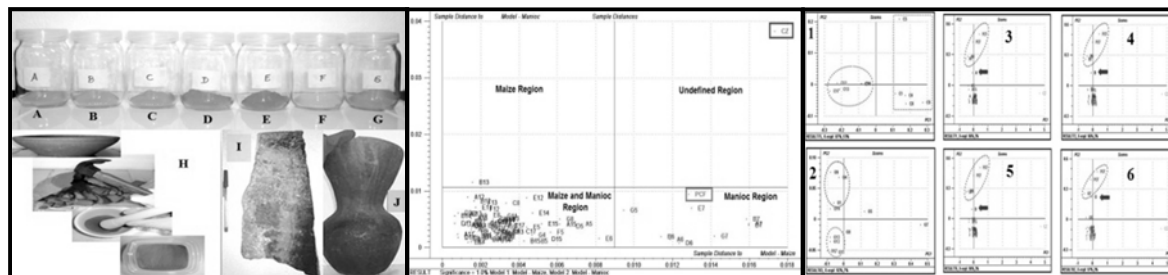


Fig. 1: Different clays used in this study to produce the laboratory-prepared samples - (A) (B) (C) (D) (E) (F) (G); Laboratory-prepared samples (H) and test samples - couscous kettle (I) and manioc meal house piece (J).

Fig. 2: Principle components analysis (PCA) scores of laboratory-prepared (1 and 2) and test samples (3 to 6) showing manioc grouping (blue lines) and maize grouping (green lines) and the relation between different clays used - 1 (C), 2 (G), 3 (A), 4 (B), 5 (D) and 6 (G).

Fig. 3: Coomans Graphic showing four regions of classification: maize region, manioc region, maize and manioc region and undefined region. The graphic shows the inclusion of the manioc meal house piece (PCF) in the manioc region (99% Confidence Level). Confirming the PCA results and the SIMCA classification table, the couscous kettle sample could not be associated with maize region.

### 34 Extracting Biogenic Information from Archaeological Bone Carbonate; The Potential of Density Separation to Assess the Original Composition

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We present a new approach to the isotopic measurement of diagenetically altered archaeological bone apatite carbonate. Bone carbonate provides an invaluable palaeodietary information. There are two main advantages of using bone collagen and bone carbonate together: that is, the different dietary information and the different survival period in the burial environment. However, the usage of bone carbonate has been questioned to date due to the difficulty of removing diagenetic carbonate and the lack of any suitable test for validity. In order to remove the diagenetic carbonate and to recover the biogenic signal from archaeological bone, an understanding of the characteristic differences between biogenic and diagenetic carbonate is essential (Garvie-Lok et al., 2004; Zazzo & Saliège, 2011). We describe how the existing differential dissolution method may be combined with a new approach to remove diagenetically reformed material on the basis of its greater specific gravity (Bell et al., 2001; Shin et al., 2004). We show that heavier, more diagenetically altered fraction has a higher (altered)  $\delta^{13}\text{C}$  and also increased crystallinity within one individual. In addition, we proposed one potential tool to check the validity of bone carbonate by comparing bone collagen, enamel carbonate and bone carbonate values of  $\delta^{13}\text{C}$  and radiocarbon content from the same individual.

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### 35 Paleodiet and Human Mobility in the Oasis of Quillagua (Northern Chile) During the Late Intermediate Period: A Reconstruction through Stable Isotopes Analysis

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Quillagua is a small oasis located at the margins of the Loa River in northern Chile, 70 km from the Pacific coast. It has been intensively occupied since the Late Formative Period, and archaeological research in the area has recovered evidence of significant cultural heterogeneity in funerary contexts of the Late Intermediate Period (AD 1000-1400). This heterogeneity has been interpreted as a result of the direct influence of two different cultures in the oasis: the Tarapacá culture, which originated on the coast north of Quillagua; and the Atacameño culture, developed in highland areas to the southeast of Quillagua. This situation has been studied through analyses of textiles and pottery on the so-called Eastern Cemetery site ("Cementerio Oriente"). Here, we present the results of carbon, nitrogen and oxygen stable isotope analyses on collagen and apatite of bone and tooth from 23 individuals recovered from two sectors (Upper sector n=10; Lower sector n=13) of the Quillagua's Eastern Cemetery. Our objective was to test whether the described cultural heterogeneity was reflected in aspects of local diet and mobility patterns. Results from carbon and nitrogen isotopes show that there was an important consumption of marine proteins in the majority of the individuals, with some of them also showing high C4 plant (maize) consumption in both the Upper and Lower sectors of the cemetery ( $\delta^{13}\text{C}$  average = -12,5‰ and -12,6‰;  $\delta^{15}\text{N}$  average = 17‰ and 18‰ respectively). Oxygen isotope results show very low  $^{18}\text{O}$  values for four individuals in the sample, two from the Upper sector ( $\delta^{18}\text{O}$  = -8,4‰ and -8,3‰) and two from the Lower sector ( $\delta^{18}\text{O}$  = -10,1‰ and 8,3‰) suggesting a possible highland origin, while the remaining individuals show  $^{18}\text{O}$  values consistent with coastal origins (Upper and Lower sector  $\delta^{18}\text{O}$  average = -4,8‰ and -5,1‰ respectively). The consumption of marine C4 plants in the cemetery is confirmed by the presence of dry fish, shellfish and maize on the burials from the site. Together, these results support the idea that the Quillagua oasis represented a relevant interaction area, with close contact with the coastal environment and a significant presence of people from the interior.

### **36 Uneven Distribution: Asymmetrical Mobility Patterns and Shifting Kinship Structures in Neolithic and Early Bronze Age Cis-Baikal, Siberia**

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Previous geochemical work in the Lake Baikal region has demonstrated the effectiveness of Sr isotope analysis in interpreting mobility patterns among Early Bronze Age hunter-gatherer groups and helped to confirm the validity of using  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios for mobility research (Haverkort et al., 2008). This research has focused on the Khuzhir-Nuge XIV (KNXIV) cemetery due to its large size; however, numerous smaller cemeteries have been excavated throughout the Cis-Baikal region. Lacking the number of interments necessary to support broader interpretations, individuals from these cemeteries are analyzed to provide broader geographic framework for the interpretation of the larger sites. First, second and third molars of 16 individuals from 6 cemeteries throughout the Cis-Baikal region were microsampled for  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios, and rare earth and trace element concentrations using ICP-MS in order to generate mobility data with improved temporal resolution. Geochemical signatures for 363 water, plant and animal bone samples were found to be far more variable across the region than predicted based on the age and type of geologic formations (Scharlotta, 2010).  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios for cultural micro-regions proved to overlap significantly and required trace element data to identify more discrete geochemical groups. The level of hunter-gatherer mobility in the Cis-Baikal region was significant with individuals recovered far from Lake Baikal showing contact with areas along the western coast of the lake. Mobility patterns show preferential cultural contact and exchange between select micro-regions. The new mobility data further support the identification of different kinship structures in operation during the Early Neolithic and Late Neolithic/Early Bronze Age.

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### **37 Applying Multiple Isotope Analyses to the Archaeological Study of Migration and Mobility in the Caribbean**

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In the Caribbean, archaeologists have often interpreted spatial-temporal distribution of cultural traits as resulting from various forms of inter-community interaction between island and mainland populations. Proposed forms of interaction during the pre-Columbian period include trade, exchange of marriage partners, bride capture, village relocation and fissioning, residential mobility and migrations at multiple scales: intra-island, inter-island, and island-mainland. Later, during the contact and colonial periods the scale of these spheres of interaction expanded as the Caribbean region experienced large-scale migrations of peoples from all over the world including not only Europe and Africa but also from other parts of the Americas. Here we present the preliminary results of an isotopic study of patterns of ancient residential mobility in the Caribbean region. We tested the utility of combining strontium (Sr), oxygen (O) and carbon (C) isotope analyses of human dental enamel ( $n = 50$ ) to identify first-generation immigrants from several archaeological skeletal assemblages representing different geographic, temporal, and cultural contexts. The Sr isotope results indicate varying proportions of nonlocal individuals amongst these populations. The carbon and oxygen isotope results displayed reduced variance for the majority of the sample population, with the exception of a few nonlocals from the protohistoric site of El Chorro de Maíta, Cuba. The combined isotope results 1) confirm the presence of non-Antillean immigrants at this site, including one each originating from Mesoamerica and Africa; 2) support independent assessments of their foreign origins based on osteological and archaeological evidence; and 3) constrain assessments of their possible geographic and cultural origins. The isotope data produced by this study also contribute to the development of empirical models of the spatial patterning of Sr, O, and C isotopes within the wider Caribbean region.



**38 Sr Isotope Analysis for the Provenance Study of Ancient Ceramics: An Integrated Approach****Christina Makarona<sup>1,4</sup>, Karin Nys<sup>2,4</sup> and Philippe Claeys<sup>3,4</sup>**<sup>1</sup> Faculty of Sciences, Vrije Universiteit Brussel, Pleinlaan 2, 1050, Brussels, Belgium<sup>2</sup> Mediterranean Archaeological Research Institute, Department of Art Sciences and Archaeology, Vrije Universiteit Brussel, Pleinlaan 2, 1050, Brussels, Belgium<sup>3</sup> Isotope Geology and Evolution of the Paleoenvironments Research Unit, Department of Geology, Vrije Universiteit Brussel, Pleinlaan 2, 1050, Brussels, Belgium<sup>4</sup> NARNIA-ITN, EU Marie Curie Training Network, <http://narnia-itn.eu/>*cmakaron@vub.ac.be*

The potential of Sr isotopic analysis in provenance studies of ancient ceramics has been explored in only a limited number of case studies (Carter et al., 2011; Li et al., 2005). The current project constitutes a novel application of Sr isotopic analysis for the provenance study of ancient pottery, within the scope of an integrated analytical approach. The technique was applied to both pottery sherds and clay sediment samples from Cyprus as an effort to link the isotopic fingerprint of the raw materials to that of the final product. The results were meant to be compared to and complement pre-existing Pb isotopic analysis carried out at Vrije Universiteit Brussel (Renson et al., 2011), while an array of additional analytical techniques was considered in order to ensure a more holistic examination of the samples. Our initial motive was to determine the applicability of Sr isotopic analysis within the scope of provenance studies and investigate to which extent these results could be used to disambiguate previous Pb isotopic analysis research on the same samples. To this end a preliminary test group of a total of 46 samples from the archaeological site Hala Sultan Tekke (Dromolaxia – Vyzakia, Cyprus) was selected: 27 ceramic sherds, covering different classes of local and imported pottery (plain white wheel-made, coarse hand-made, coarse wheel-made, white slip II, Canaanite), and 19 clay sediment samples, covering a variety of soils within relative proximity to the area of production. The samples were analyzed using a MC-ICP-MS in order to determine their characteristic <sup>87</sup>Sr/<sup>86</sup>Sr ratio.

The data resulting from the Sr isotopic analysis were assessed in parallel with the previously obtained Pb isotopic data, allowing a more clear distinction between the isotopic fingerprint of the raw material sources. Moreover, an attempt was made to investigate the use of the Sr data in conjunction with petrographic examination in order to reach conclusions regarding the nature of the materials added to the raw clay (temper). In this manner we hoped to achieve a mapping of the deviation of the final ceramic product from the raw material fingerprint composition, in relation to the technology used for its creation.

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RENSON, V., COENAERTS, J., NYS, K., MATIELLI, N., VANHAECKE, F., FAGEL, N. AND CLAEYS, PH., 2011. Lead isotopic analysis for the identification of Late Bronze Age pottery from Hala Sultan Tekke (Cyprus). *Archaeometry* **53**, 37-57.

### 39 Ceramics and Palatial Power: The Identification and Characterisation of a Ceramic Production Installation in Late Bronze Age Attica through Thin-Section Petrography

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Previous petrographic study of Late Helladic IIIB (c. 1200-1100 BCE) ceramics from Kanakia, Salamis have revealed an *entirely* imported assemblage, with kitchenware fabrics from Aegina and other vessel shapes assumed to be brought in from Attica. Intriguingly, these sources are shared by contemporary material from Greek Archaeological Service excavations at the site of Plaka, below the eastern face of the Athenian Acropolis. These wide links and large-scale movement of pottery have provided a catalyst for the investigation of specific centres of ceramic production in the area of the Saronic Gulf, specifically coastal Attica.

This paper investigates pottery from the site of Alimos located south of Athens on the Saronic coast. Material evidence at Alimos suggests several craft production installations, including pottery manufacture, were present at the pinnacle of the Late Bronze Age, a time epitomised by early state organisations, with a degree of centralised control over the production and movement of material and other goods by Mycenaean palatial centres. Ceramics from Alimos, including over-fired kiln wasters, were sampled for analysis by a combination of a multi-technique chemical analytical protocol and thin-section petrography with the aims of testing the hypothesis that identifies Alimos as the producer of ceramics found in the neighbouring sites of Plaka and Kanakia, a statement that is strongly supported by macroscopic fabric analysis. This paper focuses on the results of analysis by thin-section petrography, in order to locate ceramic production centres, and to interrogate inter- and intra-regional movement of products during the Mycenaean period in Attica. Results from this study are used to synthesise technologies of local pottery production, patterns of movement and consumption and regional evidence of political economies within an area where controversy centres around the way in which the exchange and distribution of craft goods, most significantly pottery, can be taken to reflect political boundaries and economic power.

#### 40 Amphora Production in Hellenistic Rhodes – Patterns, Matter and Performance

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In antiquity, amphorae were the most common containers for transportation and storage of a large variety of fluid or solid commodities. They were used for transport of these goods over long distances usually by shipping them throughout the Mediterranean, the Black Sea and neighboring regions. For this purpose the ceramic vessels had to fulfill considerable requirements in terms of mechanical performance. Eventually, the failure of an amphora implied not only the damage of the vessel but also the loss of its content, which was in most cases more valuable.

Rhodes was part of the Eastern Mediterranean trading network and therefore an important amphora production place fabricating characteristic vessel types. In 2002 the Archaeological Institute of Aegean Studies initiated a program concerning the study of Rhodian stamped amphorae, the number of which amounts up to today to 100.000 roughly. These vessels constitute an important evidence of the scale of Rhodian trade during the Hellenistic period as it is proved by the vast majority of stamped amphorae or handles that have been found all over the ancient world.

In the framework of the research project “Rhodian Amphorae” it was decided that for full comprehension of this amphora type physicochemical analysis of selected samples was essential. The aim of the present study was to characterize the amphora production in Rhodes during the Hellenistic Period, in terms of raw material selection, production technology and functional use of the transport vessels. Therefore an integrated approach was employed, which has recently been established for the study of the Hellenistic amphora production on the neighbored island of Kos (Hein et al., 2008). This approach comprises apart from chemical trace element analysis with neutron activation analysis (NAA) the study of the mineralogical composition and of the microstructure and material testing in order to investigate the mechanical performance. The determined material properties, finally, are used as input parameters for digital models of the vessels which are tested under simulated loads with regard to their shapes.

The results of the present study reveal an interesting picture of a specialized production of a particular type of functional ceramic vessels, with high requirements concerning performance and standardisation. On the basis of the trace element compositions of new reference groups the dissemination of Rhodian amphorae in the Mediterranean can be investigated. The study of the production technology and the functionality of the Rhodian amphora constitute a further piece in the puzzle of the organization of production and trade of amphorae in the Hellenistic period.

HEIN, A., GEORGOPULOU, V., NODAROU, E. AND KILIKOGLU, V., 2008. Koan amphorae from Halasarna – Investigations in a Hellenistic amphorae production centre. *Journal of Archaeological Science***35**, 1049-1061.

#### 41 Archaeometry in Vesuvian Area: Technological Features of Thin-Walled Ware

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Thin-walled pottery, a fine tableware, represents a widespread class in Roman Mediterranean area between 2<sup>nd</sup> cent. BC and 3<sup>th</sup> AD. Production marks are few, so production centers are usually hypothesized on the base on quantity and homogeneity of retrieved material in different archaeological sites, or comparing samples of certain provenance with unknown ones. Up to now, this class has only sporadically been evaluated archaeometrically (Montana et al., 2003; Morandi et al., 1999; Fabbri et al., 1989). Therefore, this study is aimed at defining the compositional and structural characteristics of Roman thin-walled pottery recovered in the Vesuvian area – Herculaneum and Pompeii – to validate the archaeological hypothesis of local production and to identify production technology. A collection of 24 shards have been analysed by Optical Microscopy (OM), Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS) and Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) with statistical treatment of ceramic bodies compositional data.

Obtained results, besides to confirm the previously established archaeological hypothesis about campanian production, indicate the existence of two different production technologies. Indeed, the result of the multivariate statistical treatment highlights two markedly distinct groups, which correspond to the different finding sites of the fragments. Both clusters split themselves into two subgroups. To identify the reasons of the splitting, the fragments were examined from a morpho-mineralogical point of view.

Results by OM and SEM-EDS analyses showed that finds belonging to every subgroup distinguish themselves for their surface finishing. Moreover, the presence in every sample of volcanic minerals comparable with eruptive products from Vesuvio-Monte Somma complex proves the archaeological hypothesis of a local production for this ceramic class.

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FABBRI, B., CASADIO, R. AND PEDELI, C., 1989. Studio tecnologico di ceramiche romane a pareti sottili rinvenute ad Aosta. *Miner. Petrogr. Acta* **32**, 223-30.

**42 Lead-Tin Glazed Ceramics from Southern Italy: Evolution of Production Technology by Archaeometric Investigation**

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Three ceramic classes (*protomaiolica*, *smaltata di transizione*, *smaltata*), dating from the 13<sup>th</sup> to the 16<sup>th</sup> century, coming from the archaeological site of Castello del Monte in Montella (Avellino, Italy), were investigated.

The aim of the work is to confirm the archaeological hypothesis that the *smaltata di transizione*, produced between the 14<sup>th</sup> and 15<sup>th</sup> centuries, though presenting many points of contact with the *protomaiolica*, represents a version of the tin enameled ceramic (*smaltata*) characterized by morphological and ornamental diversity (Rotili, 1999).

It is known that the evolution of production technology of glazed ceramics has led, in time, the modification of glaze composition (particularly, the increase of tin amount is generally considered a typical indicator of this evolution).

In this work archaeometric and stylistic time-related information has been achieved both on glaze and on ceramic bodies. In particular, glaze composition was determined with the aim to evaluate lead and tin amounts, whereas chemical composition of the ceramic body was quantified with the aim of identifying groups of objects distinguished on the basis of their compositional features (Bruno et al., 1994; Neff, 1992), and of evaluating the relationship between clusters identified and the composition of the glaze. Different complementary techniques -Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with multivariate statistical treatment of the compositional data matrix, Portable X-Ray Fluorescence (PXRF) and  $\mu$ -Raman spectroscopy- were used for the analyses.

The results obtained show the formation of three distinct clusters, which correspond, except for a few outliers, to the different ceramic classes of fragments: *protomaiolica*, *smaltata di transizione* and *smaltata* and seems to confirm the archaeological hypothesis that the *smaltata di transizione* is a ceramic class with transitional features (different raw materials and technological procedure of glaze application) between *protomaiolica* and *smaltata*.

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#### 43 The Pigments Applied on the Minai Wares and the Correlation with Chinese Blue-and-White Porcelain

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Persian painted *minai* ware is a group of ceramics adorned the figural or geometric design in the middle of the vessel with multi-coloured overglazes on an opaque white or turquoise base glaze. The term *minai* means enamel in Arabic and the less usual name *haft rang* which means many coloured in Persian (Mason et al., 2001). Little is known about its production because no contemporary ancient literature describing the production process has been found. These often finely painted wares are believed to have originated in Seljuq Iran during the late twelfth to the early thirteen century. The *Minai* wares declined when the Mongol invaded Persia in the 1220s. Abul Qasim of Kashan says that in his time (1301 A.D) the 'seven-colour wares' were no longer made (Allan, 1973). However, the blue-and-white porcelain re-flourished in China when the Mongol controlled the porcelain manufacture centre, Jing-de-zhen. The high quality blue pigment for the blue-and-white porcelain manufacture was imported from the west. The two different ceramics system was connected once again by the Mongol.

Fifteen *Minai* sherds and two Chinese Yuan dynasty (1271-1368 A.D) blue-and-white porcelain sherds from the Victoria & Albert Museum were analyzed by X-ray fluorescence to characterize and identify the colourants, particularly the blue pigments applied on them were discussed and grouped based on their chemical composition characteristics. Furthermore, the previous published results for Yuan blue-and-white porcelain were compared with V&A analysis results and the chemical compositions show that the *Minai* wares and Yuan blue-and-white porcelain shared the same cobalt deposit. It is highly plausible came from the Qamsar village of Kashan. The special blue pigment exported to China for the blue-and-white porcelain manufacture continued until Ming dynasty (1368-1644). However, the trading in Ming dynasty was interrupted several times and Chinese local cobalt ores were substitutes when the imported blue was unavailable.

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#### **44 Technical Studies on Medieval Islamic Glazed Tiles from Northern India**

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Following the advent of Islam in India, the use of glazed tiles for the embellishment of buildings, by then already a characteristic feature in Western/Central Asia, was introduced and adopted by the Muslim rulers in the sub-continent. Although evidence of its early use is scarce it is clear that by the 15<sup>th</sup> century and thereafter until the mid-17<sup>th</sup> century, buildings in virtually every province were being decorated with glazed tile-work. In spite of their very perceptible presence on standing monuments, tiles from medieval Islamic India are relatively less well studied especially in their comparison to the ceramic traditions of contemporaneous Western and Central Asia. Preliminary analyses by scanning electron microscope on tile specimens from three 16/17<sup>th</sup> century monuments across northern India shed light on their material composition and character. All tiles comprise quartz rich stonepaste bodies with little interstitial glass and alkali glazes. Tiles bodies are divided over more than one group based on varying quartz grain size and distribution. The presence of slips of finer-grained quartz in some tiles lends an apparently higher degree of opacity to their glazes. Glazes are marked by notable variances in alumina content. Lead detected in the yellow and green glazes is attributable to particles of lead-tin yellow dispersed within, acting as a colorant. Copper imparts a turquoise colour by itself and green in combination with lead-tin yellow. Dark blue is from cobalt present in low concentrations. No colorant was detected in white glazes, the colour apparently resultant from the induced opacity in a transparent glaze through a basal layer of fine-grained silica. The presentation will discuss the existence of compositional variances, which within an overall largely uniform technology are suggestive of organized production and the occurrence of more than one production centre. Overall similarities of the tiles with their counterparts elsewhere in the Islamic world allude to the close politico-cultural relationships in the region at the time of their manufacture, enabling us to use the glaze compositions as a reflection of these wide-ranging interactions.



## 45 New Data on the Soda Flux Used in the Production of Iznik Glazes

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Iznik pottery which represents the peak of Islamic pottery production was produced in Ottoman Turkey from towards the end of the 15<sup>th</sup> century onwards, initially as a substitute for imported Chinese blue-and-white porcelain. The Iznik glazes, which are of the lead-soda type, are characterised by a very high purity, such that their potash, lime, magnesia, alumina and iron oxide contents are each typically less than about 1 wt%. As a result, the standard soda-rich plant ashes used for Islamic alkali and lead-alkali glazes, which contain several wt% each of potash, lime and magnesia, could not have been used in the production of the Iznik glazes. Since it is generally accepted that natron from Wadi Natrun in Egypt was not being used for glass or glaze production at this period, Paynter *et al.* (2004) suggested that either some, as yet unidentified, source of inorganic soda was being used, or soda-rich plant ash was being purified prior to its use, as in the case of Venetian *cristallo* glass production. More recently, Schibille (2011) has similarly argued, on the basis of their low potash and magnesia contents, that it was unlikely that a standard soda-rich plant ash was used in the production of Middle and Late Byzantine glasses from Pergamon in Turkey. In this case, LA-ICP-MS has shown that these glasses contained high levels of boron (600-1800 ppm B) and lithium (15-440 ppm Li), as compared to their levels in both natron and standard soda-rich plant ash glasses (50-200 ppm B, 3-8 ppm Li). As a result, Schibille suggested that the source of the soda flux used in the production of these Byzantine glasses was linked in some way to the extensive borate sources in western Anatolia. The present paper will present the results of LA-ICP-MS undertaken on a small group of Iznik glazes. These analyses have shown that, as in the case of the Byzantine glasses from Pergamon, the Iznik glazes contained high levels of boron (300-1600 ppm B) and lithium (50-180 ppm Li), and as a result, strongly suggest that the same, or a related, source of soda was used in the production of both the Byzantine glasses and the Iznik glazes. The potential role of the borate sources in Anatolia in providing this soda will be considered.

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## 46 High Mg-Faiences from Fulda (Germany)

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In 1996, archaeological excavations close to the ancient Fulda faience manufacture site discovered a rich deposit of faience wastes (biscuits, faiences, technical ceramics). The manufacture was founded 1741 by prince-abbot Amand von Buseck and closed down in 1761. Adam Friedrich von Löwenfinck, a famous painter from the Meissen porcelain manufacture, became director of the Fulda manufacture in the years 1741-1744. This first archaeometric study of a German faience manufacture included 31 samples, six with painted years 1742-1748, the rest datable to the period 1741-1760. Analytical techniques were optical microscopy, X-ray fluorescence, X-ray diffraction (XRD) and scanning electron microscopy, coupled to an energy-dispersive X-ray spectrometer (SEM-EDS). Biscuits and faiences are MgO- (5-13 wt.%) and CaO-rich (9-20 wt.%), easily distinguishable from the actually known two French Mg-rich productions of Granges-le-Bourg by its significantly lower K<sub>2</sub>O and Fe<sub>2</sub>O<sub>3</sub>, and higher TiO<sub>2</sub>, and of those of Varages by its higher TiO<sub>2</sub>, Ba, Zn and Zr contents. We can therefore define the first German faience chemical reference group. Three samples show high P<sub>2</sub>O<sub>5</sub> (2.6-3.3 wt.%). Such unusual concentrations are not due to the admixing of crushed bones to the clay during processing, or to one of the well known post firing secondary contamination processes, but are caused by the presence of sharp edged, rhomboedric fragments with sizes around 20-30 µm and an overall chemical composition of apatite, as evidenced by SEM-EDS examination. These fragments are interpreted to be remnants of primary phosphoritic elements, present *ab initium* in the clay, and give some hints on the origin of the raw materials used. Phosphoritic layers can be found in the German Trias, mostly in dolomitic marls of the middle Keuper. Such marls form the basement on which Fulda is built and could easily be extracted by the Fulda manufacture. The high MgO values of the faiences can therefore be linked to the presence of dolomitic grains in the plastic raw material, corroborated by the positive MgO/CaO correlation. Firing temperatures of the faiences were, according to their XRD patterns, mostly between 950-1050°C. SEM observations of the opaque white glazes showed variable thicknesses (50-250 µm). The absence of an interaction layer between glaze and body indicates that the glaze was applied on a biscuit fired body. The tin-oxide crystals (cassiterites), responsible for the opacification, are heterogeneously distributed, often grouped into clusters. SEM-EDS analyses of representative glaze areas show relatively high SnO<sub>2</sub> concentrations (9-11 wt. %).

**47 Technological Change or Consistency: Strontium Isotope Analysis of Egyptian Faience Beads Dating from the Middle Kingdom to the New Kingdom at Abydos, Egypt**

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Faience material is considered to be the first 'high-tech' non-clay ceramic (with a chemical composition of soda-lime-silica) and has been prolifically produced for over 6,000 years throughout Egypt and the Near East. The objectives of the research presented here is to understand how the technology and chemical composition of faience in Egypt changed from the Middle Kingdom (2040-1640 BC) to the New Kingdom (1570-1070 BC) by analyzing beads from tombs at Abydos. Another objective is to develop a methodology for determining the raw material sources utilized in the production of faience, specifically the colorant, silica and alkali sources using several different analytical methods, i.e. scanning electron microscopy coupled with an energy dispersive spectrometer (SEM-EDS). However, these methods brought up several other questions regarding the alkali sources.

After reviewing several successful cases of strontium isotope analysis on Bronze-age soda-lime-silica glass, this research set out to recreate this success with Egyptian faience. Strontium isotope analysis has the potential to provide significant information on the provenance of silica and alkali sources used in the production of faience.

This is the first time strontium isotope research has been conducted on faience and will form an integral part of the development of a scientific methodology for its study. As part of this process the project aimed to investigate the alkali and silica sources utilized in the production of ancient Egyptian Faience. This is a pilot study funded and supported by the Natural Environmental Research Councils Isotope Geosciences Laboratory (NIGL). The project tested two hypotheses: whether the significant differences in strontium isotopes determined in soda-lime-silica glasses can also be determined in faience (a material of similar composition) and that the differences identified are attributable to the same basic reasons; the different origins of the soda and the lime. The second hypotheses tests whether the origins of the soda and lime in Egyptian faience differ between samples of faience made during the Middle Kingdom and faience made in the New Kingdom.

#### **48 Late Bronze Age Glass from Nippur- A New Cobalt Colorant from the Ancient Near East**

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Here we report on the unique composition of a group of Late Bronze Age (LBA; 2<sup>nd</sup> millennium B.C.) glasses colored with cobalt. These glass axe heads and other artifacts are the first significant group of glasses colored with cobalt to be identified from the Near East. The axes were excavated from the site of Nippur in present day Iraq. Several are incised with the names of three kings which date the material to the 14<sup>th</sup>-13<sup>th</sup> century B.C. Analysis by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS), indicates that the glass had high magnesia (MgO) and potash (K<sub>2</sub>O) associated with a plant ash flux and colored blue by copper or a combination of copper and cobalt. These glasses are similar, but not identical, in major element composition to blue colored glasses manufactured in ancient Egypt and elsewhere in Mesopotamia at the same period. However, the Nippur cobalt and copper colored glasses exhibit significantly different trace elemental compositions compared to Egyptian glass colored with cobalt showing that the ancient Near Eastern glass-makers had clearly identified and utilized a distinctive cobalt ore source for the coloring of this glass. Since previously it was thought that the only cobalt ores exploited in the LBA were exclusively of Egyptian origin, this new finding provides interesting insights on the origins of glass and how it was traded during the Bronze Age period.

#### 49 Isotopes on the Beach – Sr and Nd Isotopic Analysis for Provenancing Roman Glassmaking

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Provenancing Roman natron glass is one of the most challenging problems in the field of archaeometry. The elemental composition of this glass type is relatively uniform and specific objects can almost never be uniquely assigned to their origin. Although the use of Sr and Nd isotope ratios as an indication of provenance has proven promising, there are still unknown factors. In this study, we evaluate the applicability of Sr and Nd isotopic analysis for provenancing Roman glass and we present a database of Sr and Nd isotopic compositions of possible sand raw materials from the western Mediterranean, as a means of comparison for the growing number of isotopic studies on ancient glass. It is shown that the  $^{87}\text{Sr}/^{86}\text{Sr}$  ratio in natron glass is significantly influenced by the silicate fraction of the sand used and does not always provide a clear indication of the lime source used. The  $^{143}\text{Nd}/^{144}\text{Nd}$  isotope ratio of sands is a good indicator for their geological (and sometimes geographical) provenance. The variation of the isotopic composition of Nd in beach sands along the Mediterranean coasts of Spain, France and Italy is presented, and the relation between the Nd isotopic signatures and geologically distinct regions is explained. The use of the isotopic signature of Nd as a proxy for the source of silica in glass is, however, not as straightforward because of the possible overlap of signatures from different suppliers. Nevertheless, the technique has proven its value and when used in combination with, e.g., trace element geochemistry, many remaining questions concerning the organisation of the Roman glass industry may be answered.

## 50 Roman Glass across the Empire: An Elemental and Isotopic Characterization

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This study focuses on natron glass, dated from the 1<sup>st</sup> to 5<sup>th</sup> century AD, excavated in different areas of the Roman Empire. The eastern side of the Roman Empire is accounted for by three archaeological sites, Petra and Barsinia, both in Jordan, and Gonio, in Georgia. The Italian peninsula is represented by the *Iulia Felix* and *Embiez* shipwrecks, and by samples from Augusta Praetoria. Samples from Sant Boi de Llobregat in Spain are examples of the western side of the Empire, and the sites of Tienen and Oudenburg represent the northern provinces.

No clear distinctions in glass composition and origin between the different regions of the Roman Empire can be made based on the major element compositions. However, the western, central and eastern Roman Empire samples can be divided into two groups, coloured and colourless, which also is reflected in the K<sub>2</sub>O contents. Sr-Nd isotopes, used for provenancing geological raw materials in primary glass production, indicate an eastern Mediterranean origin ( $\epsilon$ Nd between -2.5 and -6.0) for most of the samples. Conversely, samples with more negative  $\epsilon$ Nd, between -7.02 and -10.8, indicate a western Mediterranean origin. By applying this technique on samples from well known archaeological contexts, this study demonstrates that several primary glass factories were located throughout the Roman Empire. This contrasts with current interpretations of late Roman to early Byzantine data.

**51 Tracing Changes in Technology of Athenian Red-Figure Slips with  $\mu$ -XRF****Marvin Cummings<sup>1</sup>, Marc Walton<sup>1</sup>, Giulia Poretti<sup>1</sup>, Karen Trentelman<sup>1</sup>, Jeff Maish<sup>2</sup> and David Saunders<sup>2</sup>**<sup>1</sup> Getty Conservation Institute<sup>2</sup> J. Paul Getty Museum*mcummings@getty.edu*

In this study, we analyzed the composition of over seventy Athenian sherds spanning the 6<sup>th</sup> to the 4<sup>th</sup> centuries B.C. (Greek classical period) utilizing *in-situ* micro x-ray fluorescence ( $\mu$ -XRF) spectroscopy. The goal of this study was to examine a wide breadth of Athenian sherds produced during the classical period and determine how the technology may have developed from the beginning of the period to the end. Using a variety of selection criteria (e.g., contour line drawing, clay slip thickness / texture / color, and quality of applied line and tool markings), sherds that best represent classic-period Athenian pottery production were chosen from the J. Paul Getty Museum collection. Results from compositional analysis show that clay slip materials (sherds) produced in the early 5<sup>th</sup> century B.C.— during the height of Athenian red figure vase production in terms of both aesthetics and the sheer number of vessels produced — possess a remarkable degree of homogeneity in the slip composition. These findings suggest the artists were likely able to refine the clay slip materials with a relatively high degree of consistency. Nonetheless, subtle, yet meaningful, variations in the composition were identified. In particular, we note that sherds produced around the 5<sup>th</sup> century B.C. show strong correlations in the clay slip material composition between iron and titanium. Conversely, potassium-content in the black gloss regions also shows an inverse correlation to both Fe and Ti, indicating the possibility of K-addition to black-gloss clay slip regions. We interpret this as indicating a potassium-containing material (e.g., potash) was deliberately added to the raw materials either as a clay-dispersant, to aid in deflocculating, or as a sintering agent. The correlations above, however, do not hold for clay-slip materials produced much earlier or later in the 6<sup>th</sup> and 4<sup>th</sup> centuries B.C., respectively; as sherds produced outside of the 5<sup>th</sup> century do not show similar trends in the clay slip composition. The implications of these results will be highlighted in the presentation, as they reflect possible shifts that may have occurred in the clay slip production technology and clay sourcing around the 5<sup>th</sup> century.

## 52 Redefining Byzantine Ceramics: 10 Years of Research at the "Laboratoire de Céramologie" in Lyon

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Byzantine ceramics, especially tableware of the Middle and Late Byzantine periods, have long been defined mainly according to techniques of decoration and to stylistical features. Other approaches, based on fabrics and laboratory analyses, and providing a definition of ceramics in terms of *productions*, are still rare. They are however seen as a first step when the circulation of wares and its quantitative aspects are considered. For the last 10 years or so, chemical analyses have been carried out by WD-XRF at the "Laboratoire de Céramologie" in Lyon in this perspective. A number of workshops in Turkey, the Crimea, Greece, Cyprus ... were characterized, which helped identifying their repertoire, studying their diffusion and the evolution of production in time (e.g. Waksman et al., 2009; Waksman, 2007; Sauer & Waksman, 2005). Wares from as yet unlocated workshops were investigated as well, showing the association of different types in a same production, contributing to our knowledge of their areas of distribution, as well as providing indirect information on chronological and typological aspects (e.g. Waksman & Teslenko, 2010; Waksman & von Wartburg, 2006; Waksman & François, 2004). Technological issues constituted another line of research (e.g. Waksman et al., 2007). Some of these results will be discussed, especially those emerging from the recent rescue excavations in Istanbul, which unearthed the first archaeological evidence of ceramics production in the Byzantine capital.

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**53 Life Goes On: Understanding the Craft Organisation and Economy of Early Postclassic Maya Lowlands Through the Analysis of Zakpah Pottery from Marco Gonzalez, Ambergris Caye, Belize**

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Ranging from such labels as ‘the dark age of Maya civilization’ to ‘commercialisation’ and ‘standardisation’, the nature of the Early Postclassic period (AD 950/1000 to 1200) in the Maya Lowlands is subject to much controversy and debate among archaeologists. The recovery and study of Zakpah pottery, however, is beginning to shed light on crafts and economies that were far from stagnant. This paper focuses on the analyses of two types of Early Postclassic fine wares from the archaeological site of Marco Gonzalez on Ambergris Caye, Belize: pedestal-based composite silhouette dishes - called ‘chalices’ - and pedestal-based jars. Both belong to a group of orange-red, slipped pottery known as ‘Zakpah’, reported from a dozen sites, including the port and trading community of Marco Gonzalez, which yields one of the largest assemblages. Analyses of Zakpah chalices and jars by visual examination, thin-section petrography, ED-XRF, and SEM-EDS aimed at documenting their technological and compositional variations, determining their provenances, reconstructing their manufacturing technologies, characterising their craft organisation, and more importantly, providing information that reflects the socio-economic developments that enabled this island community to thrive during the Early Postclassic period.

Five compositional groups were identified, each corresponding to a ceramic recipe used to manufacture both chalices and jars. Only one of these groups is consistent with the geology of Ambergris Cay, thus implying local production, whereas the provenances of other groups scatter along various coastal and lacustrine locations in northern Belize. Based on the technological variability, it seems that producers aimed to manufacture vessels with particular forms and surface treatments that would be recognised by consumers over a broad geographic region, but there was greater flexibility in how these vessels were produced. The present findings, which contrast with the popular belief of the occurrence of a ‘dark age’ following the Maya collapse, demonstrate continuity in the organisation of producing fine wares from the Terminal Classic to Early Postclassic, although there was greater tendency of using local raw materials during the Early Postclassic period. On the other hand, the production of chalices and jars was far from ‘standardised’ and ‘commercialised’, as evident in the highly variable technological and compositional attributes. In fact, the production of Zakpah pottery may indicate the emergence of new elite class that was engaged in tribute system, which characterises the Early Postclassic economy in the Maya Lowlands.

**54 Beyond the Crucible: Field Based Methods for Identifying Primary Metallurgy****Ryan Eldridge, Roger Doonan and Colin Merrony**

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Primary evidence for copper smelting remains vastly underrepresented in the British Bronze Age. The analysis of recovered copper and copper alloy objects has built a database of metal types and isotopic signatures, but has been unable to penetrate the archaeological record to pinpoint the contexts of primary production. This paper details a prospection method aimed at identifying the traces of copper smelting within the landscape, the issues and the implications.

The reduction of mineral ore to copper metal may occur within a furnace or crucible, but primary metallurgy is more than a chemical process; it is a social practice that is embedded in the landscape. As such, metallurgy draws together skilled individuals and chosen resources at specific locations and at certain times. Recognizing the spatiality of metallurgical processes opens up new opportunities for archaeometric investigation and new ways of addressing long standing problems in the study of British metallurgy. The methodology discussed within this paper utilizes a combination of visual, geochemical survey, and geophysical survey within the hinterland of identified Bronze Age mineral exploitation.

Finally, the methodology will be examined using a case study. Ecton in North Staffordshire has been radiocarbon dated to the early Bronze Age (Barnatt & Thomas, 1998) and provided a testing ground for the proposed methodology. Geochemical survey was conducted using portable X-ray Fluorescence (pXRF) while the geophysical survey was conducted with a fluxgate gradiometer. Survey results were tested through excavation. The scientific results of this study are used to address our conventional knowledge of how best to study the evidence of early metallurgy.

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## 55 Automatic Photogrammetric Reconstruction: A New Survey Method for Mining Archaeology

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Photogrammetry is a technique used to reconstruct three-dimensional scenes only from a series of photographs taken within defined conditions. This process is used for many years by the French National Geographic Institute to produce topographic maps entirely from aerial photographs, e.g. However, the protocols previously used were extremely concentrated on specific needs. Today, with the significant development of digital photography and the computer power, the production of three-dimensional data from photographs can be almost automated (Lowe, 2004; Rejas et al., 2008). Then, we like to propose a new protocol using a photogrammetry to study the ancient mines. In mining archaeology, access to three-dimensional data is a particularly important to understand the organization of a mine because such structure are developed in the space (Téreygeol, 2007; Weisgerber & Willies, 2001). From a three-dimensional model of a part of a mine, it is possible to extract data previously collected on maps from archaeological surveys. However, the record of sections of a gallery, profiles of a working place and tool marks can be increased thanks to the study of a 3D model. Moreover, we also have access to new data about the volumes extracted and the space management in a mine. And 3D photorealistic models can be used as a tool for cultural heritage visualization and give the opportunity to present to the public archaeological mines, places usually dangerous and difficult to access. Finally, given the simplicity of our original survey method that only requires the use of a camera, a flashlight and spatial references, it is of great interest to be systematically implemented in mining archaeology. The purpose of this paper is to present our image-based method and its application on the study of archaeological mines. We will expose our experiments in different medieval French mines to validate the efficiency of our process on different cases of study (digital reconstruction of narrow and large spaces, gallery, shaft, working place...). The good accuracy of our 3D models will be proved in comparison with the traditional archaeological survey plan. And finally, different examples will demonstrate the relevance of using a digital model on the study of mines: tool marks, fire setting technique and for curatorial purpose.

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## **56 New Developments in Wheeled Multi-Channel Geomagnetic Systems: Large-Scale and High-Resolution Prospection with LEA D2**

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A new multi-channel digitizer for fluxgate gradiometer arrays is presented. It is characterized by a very high measuring resolution, broadband ADCs of 24 Bit bandwidth, sampling rates up to 500 Hz and flexible GPS interfaces. Extensive field tests with several sensor types have been realized since 2010. The ruggedized and waterproof digitizer was successfully applied in several large-scale archaeological prospection projects. The advantages of the new geomagnetic system are shown by large-scale prospection examples of archaeological structures of very low magnetization. Especially sites situated in sandy environments are often characterized by unfavorable conservation conditions for organic remains due to an increased acidity of the soil. But compared to other fluxgate arrays even very small magnetic anomalies in the range of  $\pm 1$  nT can be detected in these soil types. Case studies from several sites in Germany (Early Medieval sites in Brandenburg), Portugal (the Roman city of Ammaia – project RADIO-PAST) and Turkey (Prehistoric sites near Pergamon) are presented. The economic advantages of fluxgate magnetometers especially in large-scale prospections of archaeological sites and landscapes provided the starting point of the efforts to improve multi-channel fluxgate systems. In contrast to alternative magnetometer types applied in archaeological research like cesium (Cs) probes, fluxgate magnetometers can be assembled to large arrays (6 to 16 probes) with comparatively low costs. Only such arrays allow the fast and efficient prospection of large areas. The development of a flexible lightweight cart with flexible width is another advantage of the presented system. It allows to carry 2 to 10 gradiometer probes adjusted to the required profile distance and the terrain conditions. Most important precondition for the successful application of fluxgate arrays in archaeological research is a high-quality data logging exploiting the dynamic range and the maximal resolution of the probes to a maximum extend. Using a high-resolution broadband digitiser with high sampling rates (up to 1000 Hz) the lower measuring accuracy of fluxgate sensors compared to Cs sensors can be fully compensated. The reason for this is, that the crucial factor for the successful detection of archaeological structures and objects is the ratio of their proper and the ambient magnetisation. The ambient magnetisation (caused by geological and anthropogenic structures) superimposed by the magnetic anomalies of the archaeological targets determines the practical range of the required measuring resolution. Due to this limitations the dynamic range of archaeological magnetograms rarely drops under  $\pm 1$  nT. Below this range of  $\pm 1$  nT the magnetograms are usually oversaturated i. e. geological and anthropogenic influences dominate the results. From 2009 to the beginning of 2011 the new multi-channel digitizer LEA D2 for geophysical measuring systems was developed. The adjustment to different sensor types and even other methods, like seismic measurements is planned. The development project was funded by the ZIM program of the German Federal Ministry of Economy. By the first quarter of 2012 the digitiser LEA D2 will be ready for a broader commercial exploitation.

**57 Late Pleistocene to Holocene Climate and Seasonality in North Africa from the Stable Isotope Analysis of Marine and Terrestrial Mollusc Shells (Haua Fteah, Libya)**

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The Haua Fteah cave in Libya contains one of the longest and most complete sequences of human occupation in North Africa. This rich archaeological assemblage occurs in tandem with abundant material for paleoenvironmental reconstruction. In this study, stable isotope and element ratio analyses of the archaeological mollusc assemblage from the Haua Fteah have allowed the reconstruction of paired marine and terrestrial climate records that extend from c.22,000 to 5,500 cal BP. In the marine topshell *Osilinus turbinatus*,  $\delta^{18}\text{O}$  and Mg/Ca ratios record fluctuations in sea surface temperature. In the terrestrial mollusc *Helix melanostoma*,  $\delta^{18}\text{O}$  varies according to the water ingested by the animal as the shell grows, which in turn is linked to water and air temperature at the moment of precipitation whilst  $\delta^{13}\text{C}$  provides a proxy for palaeovegetation patterns and water stress. Intrashell stable isotope series from these shells record snapshots of sub-seasonal climatic variations covering rapid and profound climatic fluctuations from MIS 2 to MIS 1. This high-resolution climatic framework coupled with the well-dated record of cultural change, allows an examination of human-environment interactions during critical periods of late Pleistocene to Holocene climate change.

## **58 Human Impact on Soil Formation and the Change from Natural to Cultural Landscapes in NW-Germany**

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During Late to End-Neolithic (3500-2200 BC), agricultural practice in the Lower Rhine Basin (NW-Germany) changed from ploughless agriculture to a presumably fire-based livestock farming. These changes were connected to a strong human impact visible in nearly all terrestrial archives (e.g. extensive changes in tree species composition). The natural landscape was transformed into a cultural landscape. We found that those environmental changes were also recorded in soils.

Dark soil horizons (Bht horizons, Luvic Phaeozem) are common features of the loess areas of the Lower Rhine Region, and they are always connected to man-made pits. We investigated these Bht horizons and pit fillings by combining methods from (geo-) archaeology (geographical distribution within the landscape, shape of the pits, soil texture), geochemistry (carbon, pyrogenic carbon, nitrogen and lipids), palaeobotany (species determination of charcoals), AMS <sup>14</sup>C measurements and luminescence dating.

Dark soil horizons and associated pits occurred as a patchwork of kilometer-sized islands independent of natural conditions (e.g. slope, parent material). The soil material itself was characterized by specific patterns of lipid compounds and high contents of charred organic matter, which are relics of biomass burning. Considering its Holocene age, determined by radiocarbon dating, the occurrence of the finely distributed charcoal can only be explained by human-caused vegetation fires because temperate deciduous forests could not be easily ignited naturally.

IRSL and OSL ages showed that the Bht horizons did not form in Pleistocene loess parent material, but in colluvial sediments that date to the Younger Neolithic (4400-3500 BC). Thus, the history of human induced soil erosion and the accumulation of correlating colluvial sediments in the investigated area started more than 1000 years earlier than assumed before.

There is archaeobotanic evidence for sustained fire-based agricultural practices in the Baltic Sea area and the Alpine Foreland since ca. 4400 BC. We concluded that the investigated dark soils are relics of prehistoric agricultural burning activities in NW-Germany, possibly providing the basis for the change to a cultural landscape in Late-Neolithic. They are not, as was presumed before, relics of naturally occurring Early Holocene steppe-soils (Chernozems).

## 59 3000 Years of Erosion and Sedimentation in the Sagalassos Territory: Man, Climate and Soil Degradation

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In order to reconstruct historical landscape changes in the territory of the ancient city of Sagalassos, SW Turkey, a multidisciplinary KU Leuven team has continued earlier palaeoenvironmental research in the area by focusing on the 260 km<sup>2</sup> Büğdüz River system draining towards Lake Burdur. A holistic approach is applied, whereby geomorphology, sedimentology, palynology and sediment fingerprinting are integrated. Since 2006, about 180 sediment cores have been retrieved from both the Büğdüz River alluvial deposits and some small intramontane basins for detailed analysis, while also topsoil sediments have been collected on the catchment's hillslopes. Apart from textural, mineralogical and geochemical analyses, ~178 AMS radiocarbon ages were obtained, and selected cores were used for very detailed palynological analysis, focusing on the post-Beyşehir Occupation Phase period, thus supplementing previous research.

Results reveal that there was a major pulse in sediment accumulation in the upper valleys during the middle 1<sup>st</sup> millennium BC. Pollen research has shown that this sedimentation pulse coincided with the first major disturbance of the landscape. A geomorphic modeling study with WATEM/SEDEM, incorporating the abovementioned results, demonstrated that land use changes are the main driving forces of hillslope erosion and hence valley sedimentation. However, after the mid-1<sup>st</sup> millennium BC sedimentation rates declined. According to the modeling results, this is due to hillslope soil depletion starting to become significant.

In the lower reaches of the Büğdüz River the sedimentation pulse is delayed compared to the upper reaches. Here, sedimentation generally peaks during the Middle Ages. In the Büğdüz River catchment also sediment fingerprinting research has been conducted. It has been shown that a large proportion of the alluvial sediments have a local origin. Sediment transport along the river valley is generally limited in the Büğdüz River system. These observations will also be incorporated, along with the small scale results, in a larger scale modeling study on the entire Büğdüz River catchment. Moreover, the significance of colluvial deposition will also be assessed.

## 60 Spatial Variations in Sulfur Isotope Composition across Britain and its Implication in Archaeological Provenance Studies

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Sulfur isotope analysis of human and faunal tissues is a relatively new application to palaeodietary studies and is used primarily in conjunction with carbon and nitrogen isotopes to distinguish marine, freshwater and terrestrial food sources (Richards et al., 2001; Privat et al., 2007). However, the interpretation of sulfur isotopes ( $\delta^{34}\text{S}$ ) in relation to diet is less well understood in comparison to carbon and nitrogen isotopes. This is because  $\delta^{34}\text{S}$  baseline data sets are typically small and mainly limited to faunal remains. The relationship between bio-available sulfur and plant  $\delta^{34}\text{S}$  is not fully defined. The aim of this study is to define the relationship between sulfur isotopes in vegetation and underlying soil and bedrock for Britain. These data should improve our interpretation of terrestrial diets and thus provide information on migration and place of origin. This study presents the first comprehensive data reference set for bio-available  $\delta^{34}\text{S}$  across Britain. The sample set is comprised of over 80 vegetation samples (grasses and herbaceous plants), previously analysed for strontium isotopes ( $^{87}\text{Sr}/^{86}\text{Sr}$ ) (Chenery et al., 2010, 2011; Warham, 2012), growing over a wide variety of soil types and geologic substrates (chalk, sandstone, mudstone, limestones and meta-sediments) across Britain. Analyses were carried out on freeze-dried powdered vegetation samples for  $\delta^{34}\text{S}$  by Continuous Flow Isotope Ratio Mass Spectrometry (CFIRMS). The instrumentation is comprised of an Elemental analyser (Flash/EA) coupled to a ThermoFinnigan Delta Plus XL isotope ratio mass spectrometer via a ConFlo III interface. In this study we compare  $\delta^{34}\text{S}$  to  $^{87}\text{Sr}/^{86}\text{Sr}$  and define plant  $\delta^{34}\text{S}$  ranges in relation to geologic terrains and soil types within these ranges. We consider the influence of sea spray sulphate across coastal transects and compare values for coastal and inland sites with similar geologic substrates.

Our results show that: British vegetation  $\delta^{34}\text{S}$  values for inland terrains fall between -10.5 and +7‰; The maximum value for coastal regions is +18‰ compared to a value of +20.3‰ for seawater; Effects of sea salt sulphate drop off significantly within 2km of the coast; Chalk and limestone bedrock lithologies have a distinctively narrow range between 3.4 and 6.3‰ (  $+5 \pm 0.8\text{‰}$   $1\sigma$ ,  $n=16$ ) compared to sandstones which have successively broader ranges; Type of geologic superficial cover (excluding soil) does not clearly influence  $\delta^{34}\text{S}$  values for vegetation growing over bedrock types; Soil chemistry appears to have strong influence on  $\delta^{34}\text{S}$  values for vegetation growing over mudstone and sandstones.

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**61 Red Window Glass in the Medieval Period: Rediscovery of a Lost Technology**

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Energy dispersive X-ray analysis (SEM-EDXA) of 132 examples of ruby red glass from medieval church windows reveals the presence of approximately 1% copper oxide in all cases. SEM and TEM of selected samples confirm the presence of Cu nanoparticles. Two structural categories of red glass sheet are identified. Sheets comprising a single layer of red glass from a few tens to around 300  $\mu\text{m}$  thick overlying a supporting substrate of white glass, with or without a protective cover of white (colourless) glass, are typically found from the fourteenth century onwards. However, in 12<sup>th</sup>-14<sup>th</sup> century England, France and Spain, perhaps elsewhere, typical red glass sheets have a complex microstructure comprising multiple coloured striations about 1  $\mu\text{m}$  thick in a white background. In some earlier studies, failure to section glass sheets perpendicular to the surface and to examine in a high powered light microscope has resulted in the description of this structure as comprising alternate stripes of red and white glass. In fact it comprises alternate stripes of high-Cu and low-Cu glasses, both of which are pale in colour, and the red striations are confined to the low-Cu glass.

SEM-EDXA, TEM, LA-ICP-MS and synchrotron X-Ray absorption spectroscopy have been used to characterise and investigate the technologies of the two types of red glass. The single-layered glasses were produced using an approach analogous to that of copper red glass in the modern period, where a red glass is flashed onto a colourless base. In contrast, the multi-layered glasses were formed by the incomplete mixing of an oxidised high-Cu and a reduced low-Cu glass and their subsequent heat-treatment. The red colour formed due to the diffusion of oxidised copper into the reduced glass and the nucleation and growth of metallic copper during heat-treatment. This represents a previously unrecognised medieval glass technology, where red was created by mixing two weakly coloured glasses, an arcane procedure which must have re-enforced the exclusivity and mystique surrounding the craft. The occurrence of the technique has implications for dating windows and the identification of glass which has been inserted in early restorations and repairs, for the trade in coloured glass and for the transmission of glassmaking technologies in medieval times.

## 62 Colour and the Chemistry of Alchemy in Hellenistic and Roman Painting

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Painting during the Hellenistic and Roman periods was more than captivating compositions of colours. Ancient painters employed complex chemical practices to render specific optical and physical qualities in their paintings. They used natural minerals, dyes and plant gums and also produced artificial colour composites with the desired properties in the absence of natural materials.

Pigments and colorants recovered from archaeological deposits and surviving paintings on walls, panels and architectural elements, provide evidence of the varied materials in the ancient Hellenistic and Roman painting palette. These included natural and artificially-produced pigments such as cinnabar, red and white lead, verdigris (Laurie, 1910; Caley, 1945, 1946), Egyptian blue (Pradell et al., 2006; Hatton et al., 2008; Kakoulli, 2009a) and organic dyes, employed as 'lakes' involving a process in which a dye is rendered insoluble with the reaction of a metal ion to form an organometallic complex (Gettens & Stout, 1966; Van Elslande et al., 2008; Miliani et al., 2010). The variability and chemical complexity of these colouring materials demonstrate a well-established colour industry with strong connections to metallurgy, glassmaking, wine and perfume manufacture, dying, and alchemy focused on transmutation, regeneration and symbolic representations (Caley, 1926, 1927, 1928; Sherwood, 1937; Herschbell, 1987; Papathanassiou, 1990). These connections facilitated the development of pictorial effects such as shading, translucency and transparency, innovations that characterize the art of these periods (Kakoulli, 2002, 2009b).

Here we explore the intrinsic chemical and microstructural properties in ancient colouring materials in the context of the early history of chemistry and alchemical practices in the Hellenistic and Roman period (Platnauer, 1921; Caley, 1928; Nock, 1929; Sherwood, 1937; Herschbell, 1987; Papathanassiou, 1990; El Khadem, 1995; Mertens, 2006) and question how these properties contributed to the paintings' qualities and aesthetics. Our study derives mainly from direct scientific investigations on material culture and from the writings of ancient writers such as Dioscorides, Theophrastus, Vitruvius and Pliny that describe the chemical arts of colour. However, whilst the information contained in these texts is informative, the knowledge draws from indirect sources. These well-known texts are complemented by two third-century A.D. manuscripts (Greek papyri) known as the Leyden Papyrus X (Caley, 1926) and the Stockholm Papyrus (Caley, 1927), which provide the earliest surviving direct evidence on ancient chemical practices. The integration of scientific analyses of archaeological materials with textual evidence significantly enhances our understanding of the Hellenistic and Roman colour technology and its use in ancient art.

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### **63 The Colour of Magic: Analysis in the Investigation of Ritual Practice in Late Bronze Age Glass**

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Late Bronze Age glass from Egypt and the Near East has been well studied particularly over the past 20 years by a number of research groups. Study of ancient texts and iconography has shown that glassmaking and glass use was a highly ritualized material, involving ritual practices that imbue glass objects with various powers. Analysis has determined the raw materials of the glasses, and given details of production technologies from a functional and practical point of view. However, these two sources of information, ritual and practical; religious and scientific, have sat relatively isolated from each other, each unable to add much to the other's interpretation of glass and glassmaking. This paper attempts to draw them closer together firstly by looking very carefully at the analyses of the glass and deducing small compositional anomalies in the compositions that hint at ritual activity. Secondly, it discusses colouring strategy and shows that certain workable recipes for creating colours are apparently deliberately avoided. Thirdly, it discusses the wider use of glass not only as a physical object, but as a ritual material with applications in medicine and protective magic and considers what physical properties of the glass might impact on its ritual as well as practical use. It emphasizes that of all the physical properties of the glass, colour is perhaps the most important in its ancient use and should therefore be a major feature of the modern interpretation of that use.

## 64 Comparing Painting Pigments and Subjects: The Cases of Maclear Area (South Africa) and Metolong Dam (Lesotho)

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The Later Stone Age rock art of southern Africa is probably the best understood tradition of rock art in the world. This understanding comes from detailed interpretative work using the extensive San ethnographies available. The meaning of the art, in broad strokes, is well established.

In contrast, the pigments used to produce these paintings are poorly known. Different colours were used: black, red, yellow, white, and of different hues. Previous studies were done since the 1990s such as Bonneau *et al.* (in press.), Tournié *et al.* (2010) and Prinsloo *et al.* (2008), identifying different kind of pigments and suggesting provenance. However, none of them compares the representation and the pigment used. Our study aims to identify the pigments used by the San artists and to compare the pigments used between the representations. Is the red pigment used to paint an eland the same as the one used for a human? We will present the preliminary results on the black, white and red pigments in two areas: Maclear, Eastern Cape, South Africa, (only the black paints were studied there) and the Metolong Dam, Lesotho.

We examine whether different 'recipes' of paint were used to depict different subjects and whether this may be linked to the symbolism of different subjects. We also examine whether the way paint was made differed between the two areas.

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## **65 Late Mousterian Red Pigment Proceeding in Les Bossats, Seine-et-Marne (France)**

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Les Bossats, near Ormesson, is a newly discovered late mousterian site dated around 47.000 B.P. by thermoluminescence. The archaeological level, fossilized by loess, revealed a rich industry based on the discoide mode, associated with numerous fragments of red pigment rocks. The geological sources were identified by means of SEM-EDX, XRD, FT-IR, PIXE and by petrographical observation of thin sections. The past mechanical and morphological modifications of the pigment blocks were characterized by macro-photography, microscopy and topographical micro-measures of the used surfaces.

It was thus possible to demonstrate that the colouring materials were brought to the site by the Neanderthals and the supply in raw material was local. Eleven blocks show different use marks such as facets, grooves and scars. The colouring materials employed by the Neanderthals on the camp site were used by different process (scraping, rubbing, crushing and grinding) in order to obtain red powder.

The archaeological remains reveal an organized proceeding sequence of red pigment. During the late Mousterian a great phenomenon in expansion in western Europe is remarkable by the much wider exploitation of mineral red and black pigments corresponding to technical modifications and divers utilizations under development. As such, it questions our perception of the humanity of Neanderthal. Did he produced symbol by using pigments or were these minerals part of the economy of subsistence?

**66 Pigments and Tiles from the Master Potter Ali Muhammad Isfahani: The Production of Decorative Underglaze Painted Tiles in the Cultural Context of 19<sup>th</sup> Century Iran**

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Polychrome tilework is a distinguishing feature of Iranian architecture. For centuries it was one of the main art forms to express the Islamic world view and values of Iranian culture in geometric, floral and figurative patterns and inscriptions. From travel accounts we learn about how people engaged with the impressive tile decoration of buildings, referring to their practical use or highlighting the artistic skills of the potters. Each period in time seems to have favoured a different technique of tile making; the 19<sup>th</sup> century brought underglaze painting to maturity. This technique allows a very fine drawing of richly coloured figures and patterns and the sheer number of preserved tiles suggests that they were the most suitable form of visual expression in architectural ceramics in the 2<sup>nd</sup> half of the 19<sup>th</sup> century. Tiles and panels painted with figurative scenes provided a medium to display socially and politically relevant topics of that time (Reiche & Voigt, in press).

While we can reason the social relevance and creative development of the tiles from viewing them, qualitative and quantitative data are needed to confirm the way how compositions of body materials, glazes and most of all paints were amended and improved by the potters to achieve the visual effects wished. Assuming that material and process are intrinsic parts of an artwork we are combining art historical and analytical methods to investigate the change in recipes and to identify aesthetic values of that time.

Recorded information on the manufacture of Persian pottery by indigenous craftsmen is very rare. Fortuitously, however, in 1887 Sir Robert Murdoch Smith, Director of the Edinburgh Museum of Science and Art, commissioned the Tehran master potter 'Ali Muhammad Isfahani to write a treatise on the process of tile making. In a British-Franco-German research program we have brought together the potter's description with the samples (Troalen et al., 2010) he also gave to illustrate his treatise (today in the collection of the Victoria and Albert Museum, London) and tiles from his workshop from British and German collections. They were studied by different non-invasive techniques ( $\mu$ PIXE/PIGE,  $\mu$ XRF, Raman,  $\mu$ XRD, SEM-EDX and Vis spectroscopy) (Reiche et al., 2011). Through this unique combination of materials and analyses, we



are aiming to study the provenance of the raw materials, the exact production processes of tiles in an individual workshop and the cultural importance of underglaze painted tiles in 19<sup>th</sup>-century Iran.

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67 **Trace Element Analysis of Islamic Glasses from Egypt, Syria and Israel: Evidence for Provenancing and Mixing of Ancient Glasses****Julian Henderson<sup>1</sup>, Simon Chenery<sup>2</sup>, Edward Faber<sup>1</sup> and Sophie Bertier<sup>3</sup>**<sup>1</sup> Department of archaeology, University of Nottingham, University Park, Nottingham NG72RD, UK ; <sup>2</sup> British Geological Survey, Keyworth, Nottingham, NG12 5GG, UK ; <sup>3</sup> IFAPO, Damascus, Syria*Julian.henderson@nottingham.ac.uk*

Scientific analysis of Islamic Middle Eastern glasses has tended to focus on the presence of major and minor levels of chemical compounds (Henderson et al., 2004), and on isotopic analysis (Henderson et al., 2009). Both of these approaches have provided information about the raw materials used to make the glasses and glass provenance. The present study focuses on the application of electron probe microanalysis and LAICPMS to the analysis of 10 glass bowl samples excavated from 11<sup>th</sup>-12<sup>th</sup> century contexts in the Damascus citadel (Syria), a set of 10 12<sup>th</sup>-14<sup>th</sup> century mosque lamps some with inscriptions stating that they were made in Cairo (Egypt) and a group of 14 samples of early 8<sup>th</sup> (Umayyad) and 13<sup>th</sup>-14<sup>th</sup> century (Mamluk) vessels excavated from Khirbat al-Minya (Israel). The latter group consists of window glass and fragments of flasks and bowls. All of the glasses from Damascus and Cairo are of a plant ash glass type; those from Khirbat al-Minya are of both plant ash and natron glass types. The provenance of natron glass has been quite successful due to the discovery of primary glass making sites in Egypt and on the Levantine coast together with the scientific analysis of the glass found on them. Fewer primary plant ash glass making sites have been discovered. It is therefore necessary to carry out scientific analyses in order to attempt to provenance such glass. One research aim is threefold: 1) to define analytical evidence for the use of different silica sources in a more sensitive way than is possible using major and minor oxides; 2) to investigate whether similar silica sources were used to make natron and plant ash glasses; 3) to identify the use of different plant ash sources of alkali. The 8<sup>th</sup> century glass was of both natron and plant ash compositional types for a time when natron glass was soon to be replaced by plant ash glass. Natron glass compositions were compared with available trace element data published by Freestone *et al.* (2000) for Levantine primary glass making sites. A series of plots of trace element concentrations and ratios has revealed not only evidence for the use of distinct silica sources in the eastern Mediterranean to make plant ash compositional glass types but also evidence of glass mixing/ recycling. These results will be compared with existing isotopic and major/minor elemental evidence and interpreted in a broad regional context.

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## **68 Elemental & Isotopic Analyses of Roman, Vandal and Byzantine Glass from the Bir Messaouda Site at Carthage, Tunisia**

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In recent years, the production and trade of glass in the ancient Mediterranean has received growing attention, particularly for the Near East. However, there are still significant gaps in our knowledge from other parts of the Mediterranean basin, with North Africa probably representing the least well-known region with regards to ancient glass production. Limited archaeological evidence suggests secondary glass working occurred in the region by at least the late Roman period, raising questions concerning origin(s) of the worked glass (i.e., locally produced or imported), but at present little evidence for primary production is documented from greater ancient Carthage. To address this disparity and to explore evidence for ancient production, trade and consumption of glass in North Africa, a collection of glass originating from Roman, Vandal and Byzantine contexts excavated at the Bir Messaouda site of Carthage, Tunisia, were analyzed. The chemical compositions of the glass are determined by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) to identify major, minor and trace element patterns among the samples. Strontium and neodymium isotopic ratios are determined for a sub-set of samples, and provide evidence on the provenance of raw materials used to produce the original raw glass, while lead isotopic ratios determined from the same sub-set of samples can inform on additions to the glass during secondary working (e.g., [de]colorants, opacifiers). These differing datasets also can provide information concerning the potential for glass recycling as well. Preliminary results suggest that ancient Carthage received imported raw or finished glass from production sites likely in the Near East, but that other regions and production loci also supplied the city, and some glass was probably produced in North Africa if not in Tunisia or Carthage itself. Since this glass originates from contexts spanning several centuries, changes in importation, production and utilization patterns of glass can be examined through time.

## 69 Byzantine Glass Bracelets Excavated on Romanian Territory Investigated Using External IBA Methods

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Some tens of colored glass bracelets fragments excavated in three Byzantine sites – Nufăru, Păcuiul lui Soare, Isaccea - located in nowadays Romania, dated from the 10<sup>th</sup> to the 13<sup>th</sup> century A. D. were investigated using Ion Beam Analysis (IBA) techniques. The measurements were directly and non-destructively carried out on the artifacts using the external 3-MeV proton micro-beam of the AGLAE accelerator facility of Centre de Recherche et de Restauration des Musées de France (C2RMF) in Paris, in the frame of CHARISMA EU FP7 project. The composition of the glass samples was determined by proton bombardment of each measured fragment, simultaneously using three ion beam techniques, respectively Particle-Induced X-ray Emission (PIXE), Prompt-Induced Gamma-ray Emission (PIGE) and Rutherford Backscattering Spectrometry (RBS). PIXE and PIGE techniques provided the glass bulk composition, whereas RBS allowed the identification of possible surface alteration.

The main purpose of this study was the determination of the chemical composition of the bracelets fragments, in order to get some hints about the employed glass recipes. The external IBA data showed that analyzed Byzantine glass bracelets can be divided into two main groups: 90% of them are soda-lime-silica glasses, while 10% of the analyzed fragments, all from the same site - Isaccea, are leaded glasses. Most of the investigated soda-lime-silica Byzantine glass bracelets pertain to mixed *natron*-plant ash category, a glass type encountered mostly in artifacts dated to the end of the 1<sup>st</sup> millennium A.D. In turn, leaded glasses can be further subdivided in two categories: lead-silica and potash-lead-silica glasses. The determined bulk compositions suggest the manufacturing of the glass bracelets using different raw materials, and possibly in different workshops. Glass recycling procedures seem to have been practiced by the Byzantine artisans who manufactured the glass bracelets.

The compositional IBA data also evidenced the chromophores providing glass color – Mn, Fe, Co, Cu ions, and provided some hints regarding the mineral pigments used to decorate the external surfaces of some of these archaeological artifacts – e.g. lead white, lead-tin yellow, and, in a special case, metallic gold-silver alloy.

Some glass corrosion phenomena, such as alkali leaching and silica enrichment of the weathered zone, were evidenced by comparing the compositions obtained on cleaned and un-cleaned areas of the same bracelet fragment.

## 70 Swabian and Mamluk Gilded and Enamelled Glass Objects: Technological Affinity through Archaeometric Investigation

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Gilded and enamelled glass objects coming from a 13<sup>th</sup> century landfill in Melfi's castle (PZ, Italy), a Frederick II fortress, and Islamic Egyptian and Syrian Mamluk Mosque Lamps (13-14<sup>th</sup> cent., - Department of Islamic Art of Musée du Louvre in Paris) were investigated (Colomban et al., submitted). The comparison arose from the resemblance in style of the objects and was carried out with the aim of understanding the links between the Swabian glasses and the Eastern models.

Both groups were studied by Raman spectroscopy, the Italian samples were also studied by Optical microscopy, Scanning Electron Microscopy and X-Ray Photoelectron Spectroscopy thanks to their fragmentary condition.

The bodies of the objects of both groups are comparable (soda-lime-silica glass). As far as enamels and gilding are concerned, similarities in the raw materials and in the technological devices can be highlighted: a hematite and lead-rich red enamel below the gilding, gold layer applied by means of an organic medium, lapis lazuli employed for at least some of the blue enamels and in some cases for the green, calcium phosphate for the white and Naples yellow for the yellow-green ones. Some peculiar features were observed in the Italian samples, such as the exclusive use of cobalt in some blues and of copper in some greens.

The set of archaeologic-archaeometric results, that highlights a strong influence of the Eastern production on the Italian one, leads to the hypothesis that the latter, after an early stage of importation from the Syrian-Palestinian area, became more and more local thanks to the probable presence of skilled Islamic artisans at the court of Frederick II.

COLOMBAN, Ph. et al., Submitted. Pigments and enamelling/gilding technology of Mamluk mosque lamps and bottle. *Journal of Raman Spectroscopy*.

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**S1 Physical and Mechanical Properties of Qusayr Amra Building Materials in Jordan: Towards Characterization and Documentation****Firas Alawneh, Fadi Balaawi and Yahya Alshawabkeh**

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The current study is a comprehensive investigation of the chemical and physical properties of the stones building materials of Amra palace, one of the main World Heritage Site in Jordan. The chemical weathering effect of salt crystallization on these stones was evaluated using a thermodynamic approach based on Runsalt software. In order to achieve that, different analytical techniques and tests were conducted. For authenticity purposes a very small samples were used. The Thermodynamic data from the tested samples showed that Halite, Sylvite and Calcium Nitrate were the main potential soluble salts at the studied samples within the current environmental conditions. Moreover a 3D laser scanning system GS100 was applied to produce a 3D model for better monitoring and conservation purposes. The results of this research shows that a slight change to current environmental conditions could have a positive impact on the building form the major deterioration factor in this site which is salt crystallization and distribution. Knowing the composition of painted plaster is of great importance for conservation; an understanding of the ancient materials and technology is necessary when creating new plasters or mortars to be used in restoration. There is little knowledge of the specific techniques used in those plasters and particularly the combination of various materials used to create more sophisticated plaster. This paper presents a complete technological study of the Qusayr Amra painted plaster in Jordan. It also attempts to propose sustainable general preservation strategy, based on a holistic methodology, integrating novel scientific techniques for noninvasive in situ examination with state-of-the art non-destructive analysis of micro samples at their molecular level. 15 samples of plaster pigments were collected from various locations in the palace, which represented all deterioration features and pigments. The methodology used to identify and investigate the painting plasters, involved the use of different techniques, namely, electron microprobe analysis, X-ray diffraction, scanning electron and optical microscopy analysis with SEM-EDX, and in addition, a micro-chemical analysis. The examination and analysis of the plasters/mortars revealed the preparation techniques used for the walls. The analysis of different samples of mortars and plasters from different areas indicated the manufacture of the mixtures used. Quantitative and qualitative analysis has shown variation in composition. A detailed macroscopic study of the painted surface revealed several features indicating that the technique of al fresco painting was employed extensively similar to those traditionally used in ancient times.

## **S2 Technology and Pathology of Previous Restorations of Imam Mosque's Dome in Isfahan, Iran**

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Imam mosque is located in southern of Naghshe Jahansquare in Isfahan with other historical buildings as a complex which is recognized original art as well as culture of Safavis dynasty. Isfahan Imam mosque in 5<sup>th</sup> January 1931 by NO.107 registered in the list of national cultural heritage works.

Domes of this mosque were damaged a few years ago. Indeed, 3 parts from 16 parts of this dome demonstrated distance of edge as well as dislocation which cause in the near future some demolition, accordingly. Direct contact with weathering agents, and previous restorations carried out in it, increase deterioration in this dome. Existed damages, hence to recent repairing need more investigation for better interpretation of the damaging process. The main reasons of these damages appeared due to mortar which is used in it. As a matter of fact, incorrect use of iron plate in the structure could be a reason for existed high distance between tiles and structural surface of the dome.

The usage of good arcing for replacing the glaze tiles could be a better idea for fixing the tiles on the dome. As a matter of fact the gypsum mortal used in the structure must have the good workability due to water/gypsum ratio in the mortal.



### **S3 Material Characterization and Technology of Stucco Decorations of Jurjir Façade (10<sup>th</sup> Century A.D)in Isfahan, Iran**

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Jurjir façade is the only remain of the Jurjir mosque in Isfahan, which is one of the few artworks of Buyids dynasty that dates back to the 10<sup>th</sup> century A.D. The Jurjir façade which stands as an early landmark in decorating portals of mosques has typical brickworks and also stucco decorations with unique motifs which probably haven't been made just in one era.

In this article, the technology of its decorations and differences between their materials will be discussed. In line with this, several samples from different parts of stucco decorations were taken for analyzing. Wet chemical tests and X-ray diffraction (XRD) were applied to identify the elements of decorations. Also, scanning electron microscopy (SEM) was used to characterize the crystalline properties, microstructures and consequently better identification.

The decoration techniques and the materials that were used are not the same throughout this façade. It is also demonstrated that a thin layer of plaster was used on the surface of brickworks. As a matter of fact, the brickworks always covered with a thin layer of plaster for obviating their aesthetic problems in appearance and give a uniform color throughout this façade. This study leads us to this fact that the decorations were created in different eras.

#### **S4 Scientific Approach of Materials and Pigments in Some of Painting in Third Millennium BC in Iran**

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Color has always played an important part in improving the quality of our environment. Many of the artifacts that we hold in the museum are decorated with a wide variety of different dyes and pigments (Roy, 1977). The fragments of mural painting and mosaic in archaeological site of Shahdad in Kerman, color traces in Tappeh Zaghe in Ghazvin and mural painting in Malian in Fars were analyzed in this work. All of these paintings belong to 3<sup>th</sup> millennium BC in Iran.

Light microscopy (LM), X-ray diffraction (XRD), scanning electron microscopy in combination with energy dispersive X-ray microanalysis (SEM-EDX) and Fourier transform infrared spectroscopy (FTIR) were used for the characterization of the structure of the paint layers of a specimen taken from mural paintings and mosaic (Stuart, 2007).

According to the obtain results, in Shahdad painting, red pigment is ochre, white pigment is calcite and black pigment is mixture of calcite and quartz. Analysis of stones in decorative mosaic showed that red, blue and white stones are ochre, azurite and calcite, respectively. In color traces in Tappeh Zaghe, three colors were used, that red and yellow are ochre and white is calcite. In Malian painting ochre and calcite used as red and white pigments. In ground layer of all painting were used of calcite, dolomite and quartz. The black material used as adhesive for mosaic include calcite, dolomite and an organic material that FTIR showed, a type of natural resins was used in this sample.

ROY, A., 1977. *Artists Pigment a handbook of their history and characteristics vol.2*. National gallery of art, Washington D.C.

STUART, B., 2007. *Analytical Techniques in Materials Conservation*. John Wiley & Sons, Ltd.

## **S5 Identification of Pigments in Two Iranian Historical Reverse Glass Painting**

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Reverse painting on glass is a type of painting where the object being painted is transparent and the image is intended to be viewed from the opposite or reverse side. The origins of this craft are not very well known (Aiken, 1982). Ancient Rome and China both lay claim to the idea that reverse painting on glass was born in their backyards. Roman dishes dating back to the third century that feature gold painting between layers of glass remain the oldest surviving example of glass painting (Caldararo, 1997).

Reverse glass painting is one of the used decorations in Rashvand house in Ghazvin. These painting are belonged to Qajar period (19<sup>th</sup> century) which has been severely destroyed. Several samples were taken from painting for material analysis. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy in combination with energy dispersive X-ray microanalysis (SEM-EDX) were used for characterization of a specimen taken from painting.

According to the obtain results, red lead, ochre, ultramarine, basic copper acetate (verdigris) and mixture of two metals, copper and zinc were used in this painting. Also, existence of gypsum, quartz and oil has been proved in background of painting. In reverse glass painting of National Museum of Iran, copper and zinc alloy, Verdigris, and basic lead carbonate (lead white) were used as gold, green and white. Mixture of gypsum and calcite were used as background layer and oil was used as binder and varnish.

AIKEN, C., 1982. Care and Conservation of Reverse Paintings on Glass. *Art and Antiques***6**, 44-46.

CALDARARO, N., 1997. Conservation and Treatment of Paintings on Ceramic and Glass: Two case studies. *Studies in Conservation***42**, 157-164.

## **S6 Case Studies on a Non-Destructive SEM-EDX Analytical Method for Polished Stone Tools and Gems**

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As spectacular, very well elaborated, intact polished stone tools represent significant archaeological value the investigation of their mineral chemistry and texture have always been problematic for geologists: in such cases only non-destructive methods (eg. Raman spectroscopy, PIXE) can be used. Although conventional non-destructive investigations in themselves are generally not enough to determine the artifacts' provenance area. Recently we have developed a new method in our laboratory, which makes in situ mineral chemical and textural examination of such artifacts possible. In our laboratory there is an AMRAY 1830 type SEM available. It is equipped with a sample chamber suitable for receiving oversized samples. After cleaning and sample preparation (selected area carbon coating) the artifacts are inserted into the scanning electron microscope, where photos are taken and measurements are carried out on previously chosen original surface areas. An aluminum foil is applied to conduct electric charge. During the measurements international standards were used in every case. In some cases there were thin sections available from the investigated artifacts and these were used as reference: the results of the investigation of the thin sections were compared to that obtained by analyzing original sample surfaces (Oláh et al., in press). Gemstones mounted into jewellery can neither be investigated separately, although their chemical composition and inclusion content could give information on their origin and provenance area. By using the sample preparation method mentioned above, mounted gemstones can be well studied without doing any harm to them or modifying their state (Horváth et al., in press).

Chemical examinations can be correlated with the sample's texture in each case which is a big advantage of the method. Several additional information about individual minerals, their textural habit, zonation, inclusions can be obtained. These make it easier to clarify the conditions of their formation and restrict possible provenance areas.

OLÁH, I., BENDŐ, ZS., SZAKMÁNY, GY., SZILÁGYI, V. AND PÉTERDI, B., In press. Results of the archaeometric analyses of stone implement preforms from Veszprém-Kádárta (W-Hungary). *Acta Archeologica*.

HORVÁTH, E., BENDŐ, ZS., MAY, Z., In press. Materials technological characteristics and workshop affinities of the polychrome style metalwork from Gáva. Wandering and settled Barbarians in the Carpathian Region and neighboring areas (1<sup>st</sup>-5<sup>th</sup> cent.). *Proceedings of the international archaeological conference held in 2010 in Nyíregyháza and Satu Mare*.

## S7 Danube: The Big Prehistoric Conveyor Belt

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The Danube is one of the largest rivers in Europe. It is transecting several countries and mountains from its sources in the Black Wood till its estuary at the Black Sea. Along the Danube, there was always a great movement of people as well as mineral substances. Recent investigations into the load of the river (gravel beds) fortunately met with petroarchaeological investigation of prehistoric settlement along the Danube in the environs of Budapest.

On the Budapest-Nánási út settlement of the younger period of the Middle Neolithic TLPC culture, an unusually large amount of pebble material was found clearly in secondary, anthropogenic context. Many of them showed intensive traces of utilisation. The variety of pebble material allowed us to include the Danube as a major transporter and source of raw materials. Recent petrographic and morphologic characterisation studies of the Danube pebbles at the gravel pits of Dunavarsány offer the possibility to compare lithological character and morphological features of the outcrops and the site.

MÁTÉ, L., 2005. *A dunavarsányi Aqua Kft. kavicsbányájában termelt felső-pleisztocén folyóvízi eredetű kavicsos összlet andezitekavicsainak statisztikai és közettani vizsgálata = Statistical and petrographical investigation of andesites from upper Pleistocene fluvial gravel from gravel pit of Aqua Kft., in Dunavarsány*. MSc Thesis, ELTE, Dept. of Petrology and Geochemistry, Budapest, pp. 136.

VIRÁG ZS.M., 2009. Újkőkori és középső rézkori települések maradványai a Nánási úton = Remains of Neolithic and Copper Age settlements in Nánási Road. *AqFüz* **15**, 97-109.

## S8 Features of Roman Ceiling Plasters (Cremona - Italy)

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Roman plasters used as ceiling coat are rarely preserved in Lombardy and their features are almost unknown on scientific criteria. Many fragments of ceiling plasters were unearthed during excavations in the old town of Cremona, a roman *castrum* on the river Po (Lombardy, Northern Italy). The ceilings are related to roman *domus* built after the devastation made by Vespasian's soldiers (69 CE): the first one (via Cadolini, mid 3<sup>rd</sup> century CE) includes three rooms with opus sectile floors; the second one (via Bella Rocca, 3<sup>rd</sup>-4<sup>th</sup> century CE) includes two rooms with mosaic floors. Ceilings plasters lay on bound reeds and they were impressed with a geometric pattern then painted with few pigments (cinnabar, green earth, yellow and red ochre). Seven plaster samples were chosen in order to investigate the features, using petrographic methods (X-ray diffraction on powder, optical microscopy on thin cross section).

Both sites show a plaster made of two lime coats with siliceous sand, covered by a thin lime coat supporting the pigments. The most interesting feature, marking a difference between the sites, is the aggregate composition of the supporting coat. The Cadolini's coat is 0.5-1.5 mm thick, the aggregate is made of angular dolomite grains (single crystal or polycrystalline, grain size 0.1-0.6 mm, close packing) and the same features are present in the supporting coat of plasters on the walls. The Bella Rocca's coat is 1-3 mm thick, the aggregate is made of sub-angular quartz grains with undulose extinction (grain size 0.2-0.8 mm, very loose packing) but the supporting coat of plasters on the walls contains calcite crystals with sharp corners and loose packing.

The disparity of the aggregate composition involves a very different way of raw material supply: the siliceous sands with metamorphic quartz were supplied by the alluvial sediments of the river Po, close to Cremona; the dolomitic grains, on the contrary, show the features (i.e. sharp crystal corners) of a manual crushing. Crushing tests made on different Prealpine dolomite formations reveal the use of "Dolomia di Zandobbio", a recrystallized dolomite (grain size 0.4-1.2 mm, Rhaetian - Hettangian) of the lower Bergamasc Prealps, some 65 km North of Cremona. This kind of aggregate was already detected in the painted plaster of some sites closer to the outcrops (i.e. Brescia - *domus* near St Giulia, late 2<sup>nd</sup> - early 3<sup>rd</sup> century CE).

## S9 Geomineralogy and Geochemistry of the Sandstone Used at the *San Giovanni Battista* Baptistery in Oggiono - Italy

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*San Giovanni Battista* Baptistery in Oggiono (Lombardy, Italy) dates back to the 11<sup>th</sup> century and represents an important testimony of Romanesque architecture.

It was built almost entirely with a bluish-grey fine-grained sandstone called locally *Pietra Molera* (Mill stone) exhibiting in many cases a superficial yellowish or reddish-brownish chromatic alteration. This sandstone was quarried in some areas close to Oggiono town (*Canali* quarry, *Molera* quarry and *Bomboldo* quarry), and belongs to the Lombard Flysch Formation (LFF). The term LFF is an informal denomination indicating many different stratigraphic units of Cretaceous age, all having turbiditic origin (Bini et al., 2010; Bersezio et al., 2010; Gaetani et al., 2010; Bichsel & Häring, 1981). The goal of the research is the characterization of the natural building materials in order to try a possible correlation between its petrographic, mineralogical, composition and those of the lithostratigraphic units belonging to the LFF; this also to establish the provenance also taking into account the historical and archival documentation. Samples collected from the monument and the local quarries were analyzed adopting optical microscopy (OM), X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF). The analyzed samples are very similar each other having the following mineralogical composition in order of decreasing abundance: quartz, quartz-feldspar rock fragments (magmatic and metamorphic rocks), chert fragments, fragments of micritic-bimicritic-microsparitic limestone or sparitic-microsparitic dolostone, intraclasts and peloids, K-feldspar and subordinate plagioclase, micas (biotite, often altered and in association with Fe-Oxides, muscovite and chlorite), opaque minerals, glauconite, fossil fragments (foraminifera) and zircon, garnets and tourmaline in traces. The cement is composed of sparitic and subordinate micritic calcite with variable amounts of microcrystalline silica and clay minerals (mainly Kaolinite). The detrital mode was also estimated and the sandstones were classified as Feldspathic Litharenites-Litharenites in according to the compositional triangular diagram (QFL) by Folk-Krynine (Bosellini et al., 2000). Mineralogical and chemical data of the samples examined so far are in agreement with the composition of *Pietra di Sarnico* Formation (Coniacian Age) as well as with those of the samples collected from the local quarries.

BERSEZIO, R., BINI, A., GELATI, R., WITH CONTRIBUTION OF BERETTA, GP., CORBARI, D., FERLIGA, C., FORNACIARI, M., ROSSI, F. AND TUCCI, G., 2010. Note illustrative della Carta Geologica d'Italia alla scala 1:50'000, Foglio 098 Bergamo. ; BICHSEL, M., AND HÄRING, M.O., 1981. Facies evolution of late Cretaceous Flysch in Lombardy (Northern Italy). *Eclogae Geologicae Helveticae* **74**, 383-420. ; BINI, A., SCIUNNACH, D., BERSEZIO, R., SCARDIA, G., TOMASI, F., WITH CONTRIBUTION OF BERETTA, GP., CARCANO, C., ROGLEDI, S., ROVIDA, A., STRINI, A., STUCCHI, M., AND MILETTA, S., 2010. Note illustrative della Carta Geologica d'Italia alla scala 1:50'000, Foglio 096 Seregno. ; BOSELLINI, A., MUTTI, E. AND RICCI LUCCHI, F., 2000. *Rocce e successioni sedimentarie*. UTET, Torino. ; GAETANI, M., SCIUNNACH, D., BINI, A., ROSSI, S., WITH CONTRIBUTION OF CORBARI, D., 2010. Note illustrative della Carta Geologica d'Italia alla scala 1:50'000, Foglio 076 Lecco.

## S10 Petrographic Evidence of Intercultural Trade During the Chalco-lithic. Examples from Neamț County, Romania

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During the late Neolithic and Chalcolithic, the Moldavian plain and Eastern Carpathians were occupied mainly by the Precucuteni-Cucuteni (a.k.a. Trypillian or Tripolye) culture. Previous researchers have proposed the possibility of intercultural trade between Cucuteni settlements and those of neighbouring cultures but empirical research to support this is very limited. In this study we focus specifically on possible interaction with the Gumelnița culture to the south, in the Dobruja-Danube area. Within the Precucuteni-Cucuteni cultural territory, there were several raw materials suitable for knapping. The best quality of these is Miorcani type flint, found in Upper Cretaceous chalky marl throughout the Moldavian plateau between the Upper Prut and Upper Dniester rivers and as fluvial deposits south of this area. There are also numerous sources of different knappable materials in the Eastern Carpathians. To the south, within the Gumelnița culture territory there are large sources of so-called Balkan flint. We analysed 507 artefacts from three sites in the Sub-Carpathians - Săcălușești-Dealul Valea Seacă, Topolița - La Ilioi and Bețești - all situated within 50km of each other. These sites are approximately 5 to 20km from various types of raw material sources in the Carpathians, 100 to 120km from sources of Miorcani flint (in the Prut river) and 190 to 240km from the nearest sources of Balkan flint (in the Danube). Twenty-five artefacts suspected of being Balkan flint were thin sectioned for petrographic analysis to confirm or redetermine sources. Based on macroscopic and microscopic analysis, the primary material used at these sites was Miorcani type flint (representing approximately 68%). Various Carpathian materials represented 29% of the assemblages. Approximately 2% appears to be Balkan flint. This study indicates that intercultural trade definitely took place in this region as early as the Neolithic. The study was financed by ID-2241/2008 and PN-II-ID-PCE-2011-3-0881 projects, granted to CI (Romanian Ministry of Education and Research).

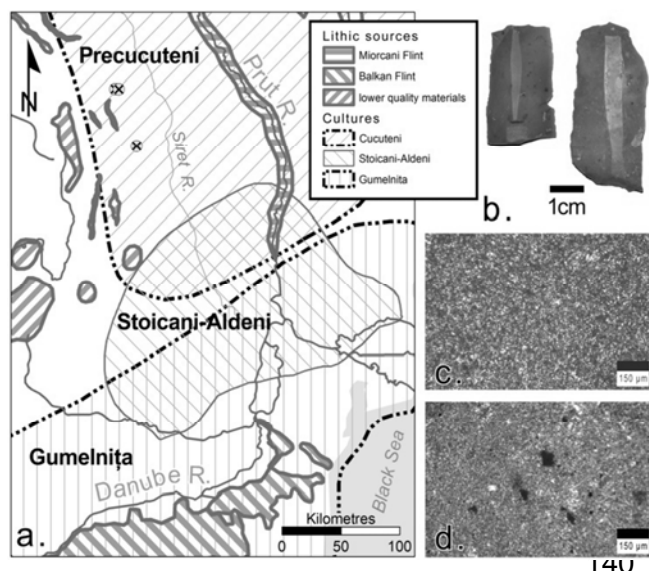
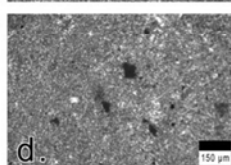
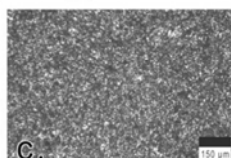
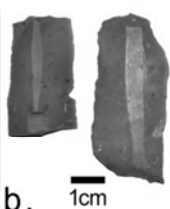


Figure. a. Map showing the location of the main lithic resources and sites from this study; b. Examples of Balkan flint tools; Thin sections of (c.) Miorcani flint and (d.) Balkan flint (+P).





## **S11 Geological origin of ochre pieces from the Middle Stone Age site of Diepkloof Rock Shelter (South Africa): reconstruction of supply strategies**

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Although no paintings are associated to archaeological contexts during the second part of the Middle Stone Age, hundreds of ochre pieces were discovered on numerous southern African sites suggesting a strong tradition of ochre use (Watts, 2002, 2009, 2010). Since there is a lack of evidence on how such materials were used, the pieces of ochre showing signs of use-wear are the main witnesses of MSA people activities involving ochre. Several questions are addressed as regard to the ochre pieces: how they were selected? Did the procurement of the raw materials require long-distance supply? Do the supply strategies of ochre procurement depend on cultural changes over time? The MSA site of Diepkloof Rock Shelter offers a unique opportunity to discuss such questions over a long diachrony.

Geological surveys were carried out around Diepkloof in order to document sources of iron-bearing rocks. The mineralogical and geochemical features of the geological sources were compared by using SEM-EDS, XRD, ICP-AES and ICP-MS. Shale and ferricrete were the main raw materials identified. Both mineralogical and geochemical features allow discriminating shale sources separated by only 10 km. No relevant mineralogical differences were observed between the ferricrete outcrops studied. According to previous works (see e.g. Popelka-Filcoff et al., 2008) we came to the conclusion that the statistical analysis of the ratios of the trace elements positively correlated to iron and iron was the more powerful method to discriminate all the sources. On the other hand, the macroscopic observation of the Diepkloof ochre pieces reveals some distinct categories of shales and ferricretes. Given the best results obtained on the geological samples, XRD and ICP-MS analyses on some archaeological samples are in progress in order to confirm these first observations. We will then be able to discuss ochre supply strategies at Diepkloof Rock Shelter.

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## **S12 Archaeometry of Pre-European Hand Millstones Quarries in Gran Canaria. Applications for the Case of the Lapilli Tuff Instruments**

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Our team develops a research project on the reconstruction of the social relationships of the production in Gran Canaria during the preeuropean time. Based on this aim, we have carried out several archaeometric studies with two objectives: 1) to know the qualities and features of different lithic raw materials, and 2) to identify the nature of the distribution networks where they are included. This work specifically regards to the case of the grinding tools which were made of vesicular volcanic flows or of lapilli tuffs, in different proportions depending on the sites.

Up to this time five hand millstones have been found associated with strombolian cones of Post Roque Nublo Cycle (Pliocene and Quaternary ages), containing welded lapilli's tuff with a composition of basanite-nephelinite and basalt, while the exploitations of lava flows have not been documented yet. Due to this we have focused on the tuff's quarries, applying several techniques of analysis (petrography, petrophysics and geochemical analysis by NAA, ICP-MS and ICP-OES).

The results obtained allow us to assert several questions: 1) the tuffs in every quarry have petrographic and geochemical homogeneity, what facilitates to constitute a reference group with each one. (Rodríguez Rodríguez et al., 2010). 2) The geochemical analysis of larger elements permits to discriminate between some of them, but the petrographic data and some trace elements are necessary to depict their origin categorically (Mangas-Viñuela et al., 2008). 3) The petrophysical tests (specific weight, porosity, erosion, compression resistance, water absorption, etc.) enable us to explain the causes of their selection as grinding tools whereas those made of lava are more resistant and lasting.

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### **S13 Archaeometric Evidence of Trade of Leucite-Bearing Volcanic-Made Roman Rotary Millstones of Pompeian Style in Hispania (Spain)**

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The leucite-bearing phonolitic and tephryphonolitic rocks of the ultraalkaline potassic province of Italy constitute a very characteristic volcanic suite unique in the circummediterranean region. These porphyritic rocks are easily recognizable in macroscopic samples by the presence of milimetric to centimetric-sized phenocrysts and glomerules of leucite, and under the microscope and with EMPA can be well characterized attending to their texture and to the mineral chemistry of their green clinopyroxenes. These kind of cereal roman millstone have been detected in a large number (more than twenty) at the Pompei excavations, (Buffone et al., 2003) and are widespread over the italian peninsula (Renzulli et al., 2002; Santi et al., 2003), South of France (Oliva et al., 1999) as well as also noticed in some north african roman sites (Antonelli et al., 2005). In North Spain their presence has been postuled (Peacock, 1986) in two very distant sites (Asturica Augusta in NW Spain and Emporium in NE Spain) but, as far we know, never proved archaeometrically or documented archaeologically. The recent findings at 3 different sites at Barcelona downtown excavations (former Barcino) have been petrographically proved and compared (by EPMA study of the mineral chemistry of green clinopyroxenes) of the known outcrops of these types of lava flows in Italy (volcano of Vico, Bagnoreggio, Orvieto, Roccamonfina). All available data strongly supports an origin of the volcanic rocks employed in the millstones at the Vulsino Volcanic District (Northern Roman Volcanic province, Italy), a fact that implies an specific and important trade way of heavy industrial instruments around I-II centuries AC. Also, the petrographic identification of findings in a rural villa near to Caesaraugusta (now Zaragoza, Ferreruelo & Mínguez, 1992), and the macroscopic identification of leucitic porphyritic rocks in millstones of Cartagonova (Cartagena), Emporium (Empuries) and in a rural villa near to Terrassa (this study) confirms that trade of these Pompeian-style millstones was widespread, at least in eastern Spain. The findings at Emporium and Terrassa provides evidence of a curious late sanitary re-use of the pierced upper part of consumed millstones as water collectors.

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#### **S14 Rare Earth Elements and Sr-Isotopic Geochemistry: Tools to Determinate the Geological and Geographical Origins of Neolithic Fluorites**

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Fluorite (CaF<sub>2</sub>) in Belgian and French Neolithic sites is a mineral not so frequently encountered. Nevertheless, several pieces of worked fluorite have been identified in excavations of sites. This has lead to the question where the mineral originate, as fluorite outcrops were not always identified in the immediate surroundings of the find spots. Was prehistoric man willing to put time and effort in procurement of the material? Was this a precious material exchanged over a significant distance? Or was the mineral always available in the proximity of its use? In trying to find an answer to these questions, two different techniques were used: isotopic <sup>87</sup>Sr/<sup>86</sup>Sr-ratios (MC-ICP-MS) and REE-contents (LA-ICP-MS). Sixteen pieces of fluorite, including a fully shaped bead, excavated in a total of eight sites were analyzed. Five sites in Belgium, namely Neufvilles (4 samples), Thieusies (6), Spiere (1), Spiennes (1) and La Houssaie (1), and three sites in France, namely Carvin (1), Lowin-Planque (rue Jaques Cartier) (1) and Lowin-Planque (ZAC) (1). All eight sites were occupied during the Michelsberg culture (4.400 – 3.500 BC) in the Middle Neolithic period.

As reference material, eleven geological fluorites samples were collected over a wide geographical range in southern Belgium and northern France (Seilles, Gimnée, Doische, Foisches, Chérq, Chokier, Lavaux-Sainte-Anne, Neufvilles and Sclayn). In these outcrops, grain-size and color match the fluorite grains excavated in the archaeological sites. The results from the archaeological samples will be compared to this geological reference material, and eventually to fluorites analyzed from Upper Paleolithic sites in Belgium (Spy, Magrite & Chaleux caves). This will show us where the raw material might have come from and hence give us important information on travel/trade distances, and possibly even point to a shift in primary source between the Upper Paleolithic and the Middle Neolithic.

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## S15 Recent Provenance Study of Obsidian Artefacts Found in Central Europe

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Obsidian was one of the most popular raw material for prehistoric stone objects (arrow heads, blades, etc.). In Europe, the main raw material sources are well known and characterised. Carpathian obsidian (consisting of C1, C2 and C3 subtypes) is the most significant mainland type among the so-called „Mediterranean” sources.

We have performed a provenance study of archaeological finds from the territory of today's Hungary, Croatia, Bosnia-Herzegovina, Serbia, Romania and Ukraine, using Prompt Gamma Activation Analysis (PGAA) at the Budapest Neutron Centre. The PGAA method is able to quantify all the major components and some important trace elements in a non-destructive way. It is a bulk method, providing composition data of a few cm<sup>3</sup> irradiated volume. About 75 archaeological pieces and 30 geological references were analyzed.

According to our earlier studies, PGAA is suitable for separating obsidians of different origin. Among the components, trace elements B and Cl found to be the most distinctive, but iron- and alkali content is also informative. Based on PGAA, it was possible to prove the Carpathian subtypes among Hungarian, Romanian, Serbian and Ukrainian archaeological finds. Moreover, we were able to distinguish between obsidians of different provenance in Croatia and in Bosnia-Herzegovina (originating from Lipari Isles and the Carpathian Sources, respectively).

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## **S16 The Pigments of the Medieval Painters in Fribourg : Investigation of a Top Quality Mural Painting from the Cordeliers Church in Fribourg**

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During the 80's, thousands of fragments of mural painting dating back to the late medieval period (15<sup>th</sup> century AD) were found during restoration works in the church of the Cordeliers in Fribourg (Switzerland). For 25 years, they remain unexploited at the Archaeological Service of Fribourg until their extraordinary esthetic quality was recognized.

Since 2009, an interdisciplinary team of researchers has been set up and a research project has been supported by the Swiss National Science Foundation (B. Pradervand). It includes the reconstruction of the entire scene -20'000 fragments- and the art historical and the technical studies. The technical study aims the identification of the materials (pigments, binders, supports) and the description of the painting techniques.

As this major work of art is already fragmented, it offers an exceptionnal opportunity for the systematical sampling of the whole painted layer and the production of numerous small polished sections for microscopic examination. On the other hand, the burial of the fragments under the ground inside the church about 200 years after the painting has been made, allows a very good preservation of the very fragile painted layer.

A panel of analytical techniques XRF, XRD, SEM, optical observation at various scales and under different lights (all performed at the university of Fribourg), Raman Spectroscopy (performed at the Department of Analytical Chemistry / University Gent with P. Vandenabeele) have been applied to the fragments.

The medieval artist, still unidentified, was using a large variety of mineral pigments, including high quality ones, like red cinnabar, blue azurite, artificial lead tin yellow and even metallic gold. His technique was highly sophisticated with pigments mixtures for the production of specific shades and superposition of layers to obtain complex visual effects. First mineralogical results on this material will be presented.

## S17 Non-Destructive Analytical Techniques as Tools for the Attribution of Paintings Related to Luis de Vargas and his Followers

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Luis de Vargas was an important Spanish painter active in the 16<sup>th</sup> century. In the Fine Arts Museum of Seville several of his paintings are conserved and some of them form part of the permanent exhibition. On the other hand, in the museum's collection there are also some paintings attributed to this artist, however the authorship is not definitive. They might be as well artworks carried out by painters from his circle or related to him in some way (Izquierdo & Muñoz, 1990; Valdivieso, 1992). The aim of the analysis carried out on several paintings selected by conservators and restorers of the museum was to obtain more information on materials and pigments applied. The comparison of the results could help at the attribution of analysed artworks. The research work, carried out in the collaboration between the curators of the Museum and the CNA research team has started with the exam of *The Purification*, painted by Vargas himself, and with *Last Supper*, whose authorship is not certain. Other paintings are scheduled to be analysed soon.

As the artworks cannot be removed from their location, only non-destructive techniques have been used, which allow the analysis *in situ*. First, the paintings were observed with ultraviolet light which revealed later interventions. Next, infrared reflectography was applied in order to exam possible under-drawings on both paintings. The material analysis has been carried out with X-Ray Fluorescence (XRF), using a portable device composed of X-ray generator RX38 and a SDD detector. The elemental results allowed us to identify inorganic pigments (West Fitzhugh et al., 1987-2007).

The results showed that the pigments applied were common for that period of time: lead white (Pb), yellow and red ochres (Fe), lead-tin yellow (Pb, Sn), vermilion (Hg), a green based pigment (Cu), azurite (Cu), smalt (Co, Ni, As, Bi), umber (Mn, Fe) and an organic black pigment, probably bone black (Ca). Some differences in the choice of pigments between both paintings have been observed, which point out that the *Last supper* was probably carried out by another, still anonymous, painter. The UV light revealed small areas of later interventions, confirmed by XRF analysis that show Zn peaks, which identify the use of modern pigment zinc white.

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## **S18 Provenance Study of Tritone Barbato Marble Statue Taken from Grotta Azzurra Capri (Italy)**

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White marbles have been very popular since ancient times. Amongst the most aesthetically valuable stones used in antiquity and in recent times for relevant architecture and decorative work, marbles played an outstanding role, particularly under the Romans.

Blocks of marble were transported far from quarries and spread over the Mediterranean archaeological sites. This study was focused on the study of the provenance of the marble statue "Tritone barbato", recovered underwater in the 60s from "Grotta Azzurra", Capri Italy. Together with other statues the archeological find was a decorating element of the cave-nymphaeum of a maritime imperial villa in Roman age. The villa was attributed to Emperor Tiberius. Several analytical techniques were utilized to solve problems of provenance assignment of marble. In particular petrographic analysis of the marble made it possible to determine textural characteristics and evaluate maximum grain size (MGS) for formulate some hypotheses about their provenance. Geochemical methods have been applied for trace and ultratrace element determination, precisely: laser ablation inductively coupled plasma mass spectrometry (LA- ICP/MS) and cathodoluminescence (CL) tests for determination of the amount of Mn were carried out.

Finally, <sup>87</sup>Sr/<sup>86</sup>Sr isotopic ratios and also O and C isotopes Isotope determinations were performed for the discrimination of marble locality.

## **S19 Archaeometric Investigation of Antimony Sources by Multi-Collector ICP - Mass Spectrometry**

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Production sites and trade routes of Roman glass have received much attention over the past decade. It is assumed that raw glass was produced in primary workshops near the raw material sources used, to be transported to secondary glass houses. Colourless glass was a particularly prestigious material in this process, difficult to make. It has been looked at from the perspective of the provenance of its sand and flux, but rarely from the perspective of the origin of the decolourizing material. In effect, for the production of early Roman colourless glass, antimony was used, deliberately added under the form of Sb-bearing minerals. Isotopic analysis of Sb ores could help identify the origin of the decolorizing agent present in Roman glasses and, consequently, to reconstruct how such material was traded and transported, and how this can be integrated in the network of primary and secondary glass producers. In this work, variations in the isotopic composition of Sb in different ore sources (stibnites) are explored using multi-collector ICP - mass spectrometry. A new method is proposed, where Sb is directly analysed for its isotopic composition using MC-ICP-MS after chromatographic isolation of the target element from a sample digest. The isotopic composition of the selected materials shows variations up to 6 ε-units relative to an antimony standard solution. Indium was used as internal standard for correction for instrumental mass discrimination and an external precision for the <sup>123</sup>Sb/<sup>121</sup>Sb ratio of 0.01% RSD was obtained.

## **S20 Technological Examination of Pigments from a Late Minoan Sacrificial Alter at Chania, Crete**

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At the Kastelli Hill in the city of Chania and in the area of the Minoan Palace complex, an important and unusual find was discovered during the excavations directed by the archaeologist Mrs Maria Vlazaki. Inside an outdoor or perhaps semi-covered area with plastered floor, close to the gate of the Palace a rectangular construction with a recession on one side came to light. The vertical sides of it were covered with plaster and decorated with red and an unusual grey/blue colour and spiral designs. The shape and position of this structure together with the findings next to it: a human female skeleton, animal bones and seeds indicated that this was probably a sacrificial altar on which a human sacrifice had probably also taken place. The activity was dated by the excavator to within the 1<sup>st</sup> half of the 13<sup>th</sup> century BC.

Samples of the red and the grey colour that had an unusual blue hue were received and subject to examination and analysis. A set of analytical techniques were used to examine and characterize the pigments: 1) Optical microscopy on fractured samples and polished cross-sections, 2) Analytical Scanning electron microscopy with energy dispersive x-ray analysis system, and 3) Microscopic Raman Spectroscopy.

The red pigment is produced with iron oxides (hematite) mixed with lime. However, the grey/blue pigment layer is very unusual and seems composed of three ingredients; a mixture of very finely ground black charcoal/soot particles, silver mica (muscovite) and lime. The charcoal/soot particles are finely dispersed and in many cases produce coatings on the mica particles. Charcoal alone in a white lime matrix produces a grey coloured pigment. However, mixed with a phyllosilicate flaky mineral, like this silver/grey mica, it produces coatings on the mica flakes and as it appears it gives reflections and shades of a more bluish appearance. As for the materials, the soot/charcoal is obtained by combusting wood of various trees and plants and the mica particles are most probably obtained from schist metamorphic rocks rich in muscovite which are abundant in the area.

It is the first time we identify a pigment produced with a combination of finely ground charcoal/soot and silver mica particles giving a unique grey-blue macroscopic colour. However, in order to prove the use of this innovative technology in the 13<sup>th</sup> century BC at Chania beyond any doubt a supplementary sampling of the plaster with this grey pigment and more analytical work will be necessary.

## **S21 On the Determination of Mineral Phases in Rock Thin Sections by Means of Object Based Image Analysis**

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Knowing the material of archaeological objects is of utmost importance in archaeology. One of the best and cheapest ways of identifying mineral phases is by optical microscopy. On the other hand this can be a time-consuming process especially if dealing with a high number of objects and associated thin sections.

This contribution presents how object based image analysis (OBIA) works and the possibilities it can attribute to the identification of mineral phases in rock thin sections. OBIA as a method for image analysis has been originally introduced in the realm of remote sensing. Due to the fact that with higher spatial resolution classic methods of pixel based image processing and analysis were no more satisfying, object based approaches approved to be an alternative. In contrast to pixel based approaches OBIA uses spatially contiguous image objects as the building blocks for image analysis. For the generation of these image objects arbitrary methods of image segmentation can be used. In GIScience, image analysis of remote sensing data is widely used to generate or update geo-datasets stored in geo-information systems (GIS). Meanwhile OBIA is routinely applied in a variety of image analysis fields, such as life sciences, medical image analysis and materials science.

In fact, one of the strengths of OBIA is to analyse image objects beyond their spectral properties, that is, by their shape properties and their spatial relationships to other image objects (in the current context: grain shape, type of neighbouring minerals, border characteristics). Additionally, spatial hierarchical relationships between image objects can be used for analysis, such as being-part-of or consists-of-relationships to quantify a mineral's internal fabric. As object recognition is defined and controlled by a dedicated programming language (we used Definiens and the Cognition Network Language), the mineral recognition process can be stepwise enhanced by focusing on dedicated petrographic properties.

In the context of petrography a particular advantage of OBIA is given by diverse shape describing parameters that allow a more objective and thus a more comparable description of grains in a specimen.

## **S22 Inorganic Pigments of Post-Byzantine Portable Icons from SW Greece: Non-Destructive Analysis by Means of X-Ray Fluorescence**

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The occupation of the Constantinople by the Ottomans in 1453 sets the beginning of the post-Byzantine period which lasted several centuries. During this period the artistic and spiritual life in Greece remained vivid; hundreds of churches and monasteries were build and decorated with splendid frescoes, while at the same time thousands of portable icons were painted. The materials and methods used for the production of these artifacts are described in a few texts written by contemporary artists, such as the monk Dionysius who wrote around 1730 the “Hermeneia” (interpretation) of the art of painting (Dionysius, 1909).

During the last few years the interest for the materials and techniques used by byzantine and post-byzantine artists has increased significantly. It is indicative that the relevant literature is constantly enriched by studies in which modern analytical tools are employed for the identification of the painting materials of icons and wall paintings (Civici, 2006; Kouloumpi et al., 2007; Daniilia et al., 2008). As regards probing the requirement for a non destructive approach prevails. X-Ray Fluorescence (XRF) is one of the most powerful analytical techniques used for the identification of inorganic materials. The major advantages of the method are the non-destructive character, the portability and flexibility as well as the quick acquisition and interpretation of the data. Given that many of the pigments used in icons are of inorganic nature the XRF may well be used for their identification.

In this paper, the results from the study of a population of post-byzantine icons from SW Greece (Epirus) are presented. The icons are exhibited in the Byzantine Museum of Ioannina and are dated to the 17<sup>th</sup> and 18<sup>th</sup> century AD. During this period, traders from Epirus were traveling through the Balkans and East Europe; the wealth produced by their trade was invested back to their land and this is one of the reasons for the rich artistic production of the period. For the identification of the pigments a portable, milli-XRF was used and the icons were studied in situ in the museum of Ioannina. Tiny samples of pigments were also selectively collected for further analytical studies (SEM-EDAX, Raman etc) and for comparison of the p. XRF results with the laboratory ones. Ongoing studies show that painters used a variety of inorganic pigments such as lead white, copper green (verdigris), copper blue (azurite or verditer), iron ochres (red and yellow), cinnabar etc. There are also indications of the use of organic pigments (such as carbon black). The results are described in detail, while the advantages and limitations of the method are also discussed.

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**S23 Raw Materials and Inorganic Pigments in Magna Graecia: Their Experimental Reproduction by Ochres from Monasterace and Rocca Imperiale (Calabria, Southern Italy)**

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Rocca Imperiale and Monasterace, two built-up areas located in Calabria Region (Southern Italy), have Pliocene clay outcrops rich in iron nodules of a range of colours, varying from brown to red. It is likely that the inhabitants of some colonies in Magna Graecia - who already used the clay deposits to produce bricks (De Francesco et al., 2009) - had detected the presence of the above-mentioned raw materials and decided to use them to produce mineral pigments and to decorate polychrome artefacts. The present study was devoted to show how brown Monasterace and Rocca Imperiale nodules may be used to produce mineral pigments, varying in colour from brown to red. Each nodule was pulverised in an agata mortar and heated in air at a controlled temperatures (275°C, 400°C, 550°C and 750°C) for 8 hours and subsequently analyzed through SEM-EDS, XRPD and colorimetric analysis. The heating of the powders leads to significant chromatic variations, mainly due to the formation of hematite, which takes place after heating at 275°C. Hematite relatively increases up to 750°C, shifting the colouring of samples to red. However, the idea that Greek coloners have used these nodules to decorate polychrome artefacts remains a hypothesis. Only future comparisons between archaeological pigments and pigments produced by Monasterace and Rocca Imperiale nodules, will lead to the solution of the archaeometric problem (Miriello et al., 2010).

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## S24 Mortars and Stone Materials from Marina el-Alamein (Egypt)

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Archaeological site of Marina el-Alamein (northern Egypt) is the location of a *Greco-Roman town* (Medeksza et al., 2008). The remains unearthed by Polish scientists are represented mainly by buildings and tombs on different state of preservation (Czerner, 2009). The investigations included the analysis and petrographic observations of mortars and stone material used for construction and architectural decoration purposes. The study of geological surroundings of the archaeological site showed that raw material originated from a local supplies.

The research included petrographic observation on thin section and SEM-EDS morphological and micro-chemical analyses. Additionally for marbles - carbon and oxygen isotopic ratio and CL color images were assigned. In case of mortars the radiocarbon dating was conducted. Samples were collected from various parts of the houses and public objects (like walls, pavements and other artifacts).

The state of local oolitic limestone used as main building material is variable including different forms of alteration and degradation (Mrozek-Wysocka & Zambrzycki, 2010). The provenance of another stones have been also indicated including Mons Porphyrites, Proconessos and Carrara quarries. Composition of mortars is mainly carbonatous and gypsum with different portions of aggregate and charcoal fragments occurrence.

The results of the research in Marina el-Alamein archaeological site are due to be applied in the future conservation and restoration works and also for establishing the chronology of the buildings. The absolute age determinations by  $^{14}\text{C}$  will be compared with relative chronology of the walls (Nawrocka et al., 2009).

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## **S25 Inventory, Mapping and Multidisciplinary Study of the Ancient Quarries of the Sinis Peninsula (West Sardinia, Italy)**

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In recent years, the ancient stone quarries along the Mediterranean coasts are increasingly emerging as significant archaeological sites, being manifest sources of building materials employed in past urban areas, and also historical “industrial” sites that show large evidences of mining techniques and stone extraction activities in the antiquity. In Sardinia, the ancient coastal quarries are probably mostly of Punic and Roman epochs, and exploited late Pleistocene marine and aeolian sandstone deposits, extended along the Southern and Western coasts of the island, where several major extraction sites and minor quarries have been yet identified. Our studies are focused on the Sinis Peninsula, in Western Sardinia, an area where several ancient quarries are known, probably related to the ancient city of Tharros. The city, located in the Southernmost part of the Sinis Peninsula, was an important port in ancient Sardinia. During the history of the city, the late Pleistocene carbonate sandstones that crop out along the Sinis coasts have been largely quarried, being an easy-to cut material, well suitable for stone blocks productions destined to military and civil works, and for civil and religious buildings. The main extraction areas have been identified in three sectors of the Sinis coasts: 1) a quarry complex, extended for over 0.5 ha, located about 1.5 km NW from Tharros (39°15'19.87"N – 8°25'48.41"E), close to the present village of San Giovanni di Sinis; 2) a quarry complex, apparently of the same size, located at Punta Maimoni (39°54'33.43"N – 8°23'48.94"E), about 3 km NW from Tharros; 3) a more articulated and extended (well over 3 ha.) quarry complex, located in the Is Arutas area, about 6 km NW from Tharros, including several extraction sites from the Monti Corriglias (39°56'29.85"N – 8°24'19.44"E) to Nuraghe Muras area (39°57'43.6"N – 8°24'14.03"E). The study comprises field surveys and mapping of the quarries, and integrates archaeological data with the basic geological/geomorphological features of the deposits, the mineralogical/petrographical characterisation of the stones, and the functional study of the features related to past quarrying activities (layout/structure of the quarries, extraction fronts, spoil heaps, working areas, paths of transport of stone blocks; loading and harbouring facilities, etc.). This multidisciplinary approach is finalised to obtain a complete framework of the sites, including their dating, essential for recognising their role in the local economies, and for further reconstructions of the historical diffusion and uses of building materials in the region.

**S26 The Piscinnì Quarry Complex (Southern Sardinia, Italy): Geological/Mineralogical Characterisation and Functional Study of a Punic-Roman Stone Extraction Site**

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The site of Piscinnì (Gulf of Teulada, Southern Sardinia coast) is one of the largest and most significant ancient quarry complexes of Sardinia. The quarries produced sandstone blocks; they were presumably set during the Punic period (before the 238 B.C., in Sardinia), being largely expanded during the Roman age, according to the needs of construction materials in the nearby areas, as the port of Malfatano (Bithia Portus), where the use of stone blocks coming from Piscinnì for breakwaters and jetties has been documented. Despite its relevance, the site is not dated, and has never been the object of systematic archaeological studies. As part of a University of Cagliari project for study and cultural valorisation of ancient quarry landscapes, funded by the Geo-mineral, Historical and Environmental Park of Sardinia, the quarries of Piscinnì have been studied in order to define their relevance as geosites of historical mining. The data acquired during this study provide a basic support for further archaeological surveys. Sandstones quarried in Piscinnì are lithologically similar to those widely extracted and used in Punic-Roman ages both in Sardinia (e.g., in Sinis peninsula) and in Tunisia (e.g., El Haouaria/Cap Bon); the stone blocks were extracted from Quaternary (late Pleistocene) marine/coastal and aeolian sedimentary deposits, including a thick sequence of cross-bedded, bioclastic carbonate sandstones. The quarry complex can be divided into three sectors, located along 1 km of the coastline, with a total extension of over 3 ha; the volumes of extracted materials are of several tens of thousands of m<sup>3</sup>. The most structured sites, with several evidences that allow functional interpretations of the past activities, are located in the Porto di Piscinnì (38°54'32.59"N, 8°46'46'.17"E) and Torre di Piscinnì (38°54'13.84"N, 8°46'45.30"E) areas. They include different types of quarry works (large open casts; trenches, vertical fronts, etc.), with extraction fronts up to 3-4 m high, marked by negatives of blocks (prevailing module of about 90x50x50 cm) and by abundant toolmarks. As usual in the ancient Sardinian sandstone quarries, the extraction techniques were a mix between channelling and rock splitting by wedges: in the working yards close to the sea, the postholes of derrick cranes used for handling and loading the stone blocks have been individuated; spoil heaps and block fragments are scattered in the whole area. Despite coastal erosion and weathering, the sites are well preserved, and offers many opportunities for observation of the main features of an ancient stone quarry.

## **S27 Characterization of Lead White and Lead-Tin-Yellow Pigments in Palette of Mediaeval Gdańsk Pomerania Painting**

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We propose of instrumental neutron activation analysis and SEM-EDS to the study of lead-based pigments (lead white and lead tin-yellow) in mediaeval Gdańsk painting. The investigation aims, among other is the creation a database of historic pigments originated from different regions and periods.

In the second half of the XV century, Gdańsk enjoyed renown as one the most significant cities of Europe. The city belonged to Hanzeatic League and operated extensive trading activities. Its favorable economic condition promoted the development of the city. The rich society founded both sacred and secular buildings decorated with numerous works of art.

Being a significant commercial centre, Gdańsk maintained wide contacts with a number of cities Europe-wide. The Gdańsk art of those times was particularly influenced by the Netherlands and Northern Germany. Gdańsk panel paintings is a non-homogeneous combination works of art. A part of the altars might have been imported, while another was created by foreign artists working in Gdańsk or by local painters.

The concentration of trace elements in an pigment ( so-called 'finger print') depends not only on the place where the samples was taken, but also on the technological process used in its production. The combination of these accumulated databases with powerful multivariate statistical methods (i.e., principal components analysis, factor analysis, discriminant analysis, and cluster analysis) allows many materials to be sourced with a high degree of confidence. The main objective of the study is characterisation and identification of lead pigments in the 15<sup>th</sup>-17<sup>th</sup> century panel paintings from Gdańsk School on the basis of elemental composition. Additionally for the comparison will become represented findings of West-European pictures being found in Polish crops: the Netherlandic painting represents triptych of Hans Memling - Last Judgement (at nowadays of the National Museum in Gdańsk), and Hans Pleydenwurff German Master painting from the former main altar from the church St. of Elisabeth in Wrocław (at nowadays National Museum in Warsaw).

These analyses make it possible to work out a model of the painter's palette, revealing a great deal of information on the paintings techniques used, the school the painting belongs to and the ways in which the structure of painting was achieved. The variation seen in trace element compositions according to region and time, proper authentication would require compiling a library of analyses of carefully documented paintings with which to compare statistically the painting in question.

**S28 Analysis of Grounds from Icon Painting (Korytniki Orthodox Church, SE Poland)**

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The main aim of this study is petrological, geochemical and micropaleontological analysis of grounds from the icons of ST Demetrius orthodox church at Korytniki near Przemyśl (SE Poland), now collected at the Orthodox Art Department at the Castle Museum in Łańcut. The samples of grounds were taken from the 14<sup>th</sup>-18<sup>th</sup> centuries icon paintings representing the so-called South-Eastern Polish school. We had sampled the natural outcrops of chalk located in Carpathian Mountains and near Przemyśl. The following icons were selected for analysis: *Chrzest Chrystusa*, *Św. Paraskewa*, *Św. Jan Ewangelista* and *Sąd Ostateczny*. These paintings of high artistic value are representative for this regional iconographic style.

We had applied for analysis the following methods: 1) optical microscopy – for description and identification of grounds and painting layers as well as for characterization of potential source rocks (chalk) collected from the outcrops located in SE Poland; 2) scanning electron microscopy (SEM-EDX) – for morphology, distribution and chemistry of minerals as well as for identification of microfossils in grounds and chalk; 3) instrumental neutron activation analysis (INAA) – for identification and concentration of elementary and trace elements and 4) multi-parameter statistical analysis – for identifying the degree of similarity between the analyzed objects and the potential sources of the grounds.

## S29 Pigments of Iron Age Painted Pottery from Garvão Votive Site (SW Iberia)

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In 1982 an impressive votive deposit was discovered in Garvão (Southwest Iberia) attributed to the second Iron Age period (Beirão et al., 1985). The recovered materials (especially ceramics) were intentionally deposited, carefully arranged in order to optimize the available space. Some complete pieces and some shards are from painted pottery, decorated with black, white and red-brown pigments. Most of the decoration found in the pottery is characterised by the presence of geometric drawings. Parallel and horizontal bands are the main motives. There are only two main forms of painted containers, ovoid vases and plates, the latter form presents only decoration of red coloured circles. The complete characterization of the archaeological site and of the materials as testimony of an ancient Iberian society must provide information about the cultural links and the capability to use raw materials. The main purpose was the characterization of Garvão votive deposit decorated pottery by determination of the chemical and phase composition of pigments and identification of the phases in the ceramic. The methodology for the chemical and mineralogical characterization involved several complementary techniques. The texture of the pigments was registered by stereomicroscopy. The phase identification was provided by  $\mu$ XRD and Raman spectroscopy, useful for non-crystalline compounds. The chemical composition was attained by *in-situ*  $\mu$ XRF, considering the dissimilarity with the ceramic body and by SEM-EDS. This technique can provide also textural information and spatial distribution and associations of the chemical elements. The abundance of iron detected by  $\mu$ XRF and the presence of hematite, clay and quartz are strong evidence that red color is attained due to ochre pigments. The results of SEM-EDS analysis of black pigment show the presence of manganese particles, consistent with  $\mu$ XRF data. Nevertheless, the  $\mu$ XRD do not show the presence of crystalline manganese compounds. Since Raman spectroscopy is sensitive to short-range order it can show that the black color is due to the presence of pyrolusite like compounds. The XRF is unable to provide a chemical signature of the white pigments and distinguish it from the paste. By SEM-EDS, the chemical composition and the texture showed that color is attained by white clay material. The abundance of potassium, aluminum and silicon make illite a very probable choice. This hypothesis is confirmed by  $\mu$ XRD.

It is expected that these archaeometric results can contribute to a better understanding of the Garvão votive deposit in the context of Iberian Iron age.

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### **S30 “Marble” Luxury in the Public Buildings of Colonia Ulpia Traiana, Xanten**

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Colonia Ulpia Traiana (CUT) near modern Xanten was founded at the beginning of the 2<sup>nd</sup> century AD by the Emperor Trajan and belonged administratively to the Roman province Germania Inferior. During the whole 2<sup>nd</sup> and the first half of the 3<sup>rd</sup> century colonia underwent a continuing process of urbanization and monumentalisation, during which it was provided with the main public buildings like Capitolium, Harbor-Temple, Baths and Forum.

Recent archaeological excavations in CUT brought numerous fragments of veneering slabs to light, which once lavishly decorated walls and floors of the public buildings. CUT has no on-site quarries producing white or colored marbles. For that reason colonia had to cover its needs for desirable decorative stones through import from regional or foreign sources. It was considered until now, that the cities in Germania Inferior mainly imported decorative stones from the affordable regional sources, whereas the Mediterranean marbles for a long time stayed a costly rarity.

Therefore the main aim of this study was to characterize and determine the types of stones used for the wall and floor veneerings in CUT, in order to identify the provenance of the studied samples.

Approximately 3250 fragments were inventoried and classified into different material groups, from which a representative number of samples (≈250 Fragments) were selected for different analytical methods to pinpoint the origin of marbles. The methods applied were petrographical investigations (optical microscopy on thin section, XRD), chemical analysis (RFA, EPMA) and isotopical analysis for the determination of O & C stable isotope ratios.

The preliminary results point out that a substantial higher quantity (≈ 80 %) of stones used for the interior decorations of public buildings in CUT originates from different Mediterranean sources. White and gray marble varieties were imported from Thasos, Penteli, Carrara, Proconnesos, Ephesos, Hymettos. As the most popular colorful “Marble” types can be mentioned Cipollino Verde, Verde Antico, Lapis Lacedaemonius, Africano, Pavonazzetto, Fior di Pesco, Breccia di Settebasi, Rosso Antico, Porfido Rosso and others. Only a quite low percentage of decorative stones (≈ 20 %) can be subscribed to the regional quarries: Trachyt from Drachenfels and Berkum, Ruhr sandstones, black limestones from Belgium and Aachen, red limestones from Belgium, white marbles from Odenwald. Some of the mentioned local varieties were quarried on the “barbarian” side of Limes.

### **S31 Preliminary Investigation into the Materials and Technology of Romano-Egyptian Mummy Portraits at the J. Paul Getty Museum**

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Mummy portraits from Roman Egypt are well known for their beauty and significance as a snap shot of the people who lived 2,000 years ago. The incorporation of the Greco-Roman tradition of painted portraits into an already ancient Egyptian funerary practice of mummification represents a major cultural melding. Along with this Western artistic influence, comes the introduction of new materials and techniques previously unknown in Egyptian art. However, the production technology and the application techniques of these materials remain to be fully understood today.

The J. Paul Getty Museum, in collaboration with the Getty Conservation Institute and the British Museum, has launched a multidisciplinary project investigating the materials and production methods of 10 portraits on wooden panels and one complete portrait mummy, dating from the 1<sup>st</sup>-3<sup>rd</sup> century A.D. in the Getty collection. The initial study of these mummy portraits began with elemental analysis using: x-ray fluorescence (XRF) spectroscopy, Raman microspectroscopy and inductively couple plasma time of flight mass spectrometry (ICP-TOFMS) revealing that the pigments are unique and chosen with a specific purpose. One example is the pigment red lead, introduced by the Romans, and imported from Spain (Walton & Trentelman, 2009). The importation of materials goes beyond pigments however and confirms a sophisticated understanding of not only the wood selected for preparing the portraits (Cartwright et al., 2011) but the methods of producing the portraits themselves. These materials also function in concert with known Egyptian funerary beliefs.

Published research on the materials and technology used for creating these objects is limited and typically isolated to only one or a few portraits. Even the investigation of the Getty group is too small for a greater understanding of mummy portrait production. It is for this reason that the Getty will launch a database of these unique artifacts, inviting international museums with mummy portrait collections to contribute data with the goal of creating an interactive and dynamic repository of information that can be continuously built upon. Through this database, a larger quantity of portraits can be examined and compared as well as easily accessed providing a greater understanding of the materials and techniques for these striking precursors to the Western painting tradition.

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CARTWRIGHT, C., ROSA SPAABECK, L. AND SVOBODA, M., 2011. Portrait Mummies from Roman Egypt: ongoing collaborative research on wood identification. *The British Museum Technical Research Bulletin* **5**, 49-58

### **S32 Long Distance Import of Polished Stone Artefacts: HP Metamorphites in Hungary**

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Polished stone tools made of HP metamorphites constitute one of the most spectacular items on the list of prehistoric long-distance trade goods in Europe. As the rocks are attractive, and of high quality, they were distributed over large distances mainly to W-Europe and scarce to E-Europe. In Hungary only 3 pieces of these tools known until now. It is, however, not easy to identify them unambiguously among “greenstones”. The first suspect pieces were located in the course of macroscopic and petrographic investigation of large collections on polished stone tools in the Veszprém Museum and the Hungarian National Museum. Recently, with the possibility to use non-destructive analytical techniques for chemical composition (PGAA), mineralogical composition (parallel-beam X-ray Diffraction) and crystal chemistry (EPMA) a series of “greenstone” axes were analysed and the first authentic pieces were demonstrated. Our results show that the raw materials of analysed “greenstones” are various metamorphites: greenschist-metabasite, serpentinite as well HP-rocks, nephrite and jadeitite/omphacite.

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SZAKMÁNY GY., FÜRI, J. AND SZOLGAY, ZS., 2001. Outlined petrographical results of the raw materials of polished stone tools of the Mihálydy-collection, Laczkó Dezső Museum, Veszprém (Hungary). In: REGENYE (Ed.) *Sites and stones: Lengyel culture in western Hungary and beyond A review of the current research. Lengyel'99 and IGCP-442 Conference, Veszprém 1999*. Veszprém 109-118.



### S33 Whetstones in Gaul: Building a Typology

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Tool of the tool, whetstones are meant for the sharpening of metal utensils: knives, scythes, weapons... Used in the context of a profession or more simply objects of the daily life, they are often rediscovered in excavations, sometimes in great number. However, scientific interest for these whetstones is minimal. Often quoted, sometimes described, they encounter an astonishing void in literature. To study and characterise this unpublished material is really important. It will as well deal with the raw material, manufacturing, trade and typology of these tools. Thinking about the tool, its place in the operative chains of agricultural, craft and domestic activities will reveal a testimony to the knowledge of Roman society in northern Gaul. The originality of this work lies in the combination of archaeometric analyses and archaeological processes. Building a typology is the first step of this study, based on three selected sites: Rues-des-Vignes (France), Arlon (Province of Luxembourg, Belgium) and Nereth-Baelen (Province of Liège, Belgium). The Rues-des-Vignes site is a potter's workshop located in the south of *Civitas Nerviorum*. The occupation of the whole settlement covers two centuries from about 65-70 to 270-280 A.D. (Deru, 2005). Several excavations were achieved in Arlon over the last years. They revealed houses and craft building from the settlement of *Orolauno Vicus* which are dated from the 2<sup>nd</sup> to 3<sup>rd</sup> century (Henrotay, 2007). The settlement of Nereth near Baelen in the North West of Belgium provides also a great number of whetstones. At Nereth, a large settlement from the Late Antiquity follows a rural settlement from the Early Roman Empire. The site proves the presence of one or more Germanic families during the 4th century (Hannut et al., accepted).

The material is divided into six groups based upon shape: pebble, clog-like, parallelepiped rectangle, shuttle and cylinders with elliptic cross-section or circular cross-section. The nature of the material ranges from detrital sedimentary rocks of different granulometry between siltstones and medium sandstones (Goemaere, 2010) to low-grade metamorphic quartzites.

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### **S34 An Isotope Database of Marble Quarry Sources in Algeria and Tunisia**

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Between 2005 and 2008, we conducted a detailed survey and scientific sampling of nine Algerian and four Tunisian marble sources, most of which exhibited signs of use in pre-modern times. With the assistance of the Algerian Ministry of Energy and Mines, we collected more than 300 geological samples, aiming at a minimum of 20 samples from each quarry. At the University of South Florida, stable carbon and oxygen isotope analysis has been done on more than 250 samples. In addition, maximum grain size measurements have been made and X-ray diffraction or X-ray fluorescence conducted on many samples.

These Algerian and Tunisian quarries produce a variety of marbles, which we have separated into groups based on their macroscopic characteristics. White and light gray marbles were produced at Mt. Filfila, Cap de Garde, and Mt. Mahouna. Dark gray and black marbles were also produced at Mt. Filfila, and they can be compared isotopically with similar stones from Tunisia, Greece, and Turkey. Red breccias were produced at Kristel, Cape Chenoua, and Kheddal. To greater or lesser degrees these stones resemble *breccia corallina* from Vezirhan (Turkey). Yellow breccia was quarried at Kristel, Chemtou and Mt. Ichkeul. Layered marble (travertine or onyx marble) was produced at Mt. Mahouna, Aïn Smara, Bou Hanifia, and Aïn Tekbalet.

Our analyses clearly indicate that many Algerian and Tunisian sources can be distinguished from each other and from other known Mediterranean sources. Notably, despite their apparent heterogeneity the breccias and the layered travertines exhibit no greater isotopic variation than the white marbles. Our isotopic data, combined with those of Antonelli *et al.* (2009) on 20 samples from Cap de Garde, Agus *et al.* (2006) on 46 Tunisian black limestones, and Norman Herz (unpublished) for 11 Chemtou samples, provides an extensive database for scholars working on marble potentially from North Africa.

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ANTONELLI, F., LAZZARINI, L., CANCELLIERE, S. AND DESSANDIER, D., 2009. Minero-petrographic and geochemical characterization of 'Greco Scritto' marble from Cap De Garde, near Hippo Regius (Annaba, Algeria). *Archaeometry***51**, 351-365.

### **S35 The Golgotha in the Church of the Holy Sepulchre. Loss and Damage of a Holy Place**

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The Church of the Holy Sepulchre was first built over the (supposed right) place of crucifixion of Jesus (Golgotha) about 325 AD. During the centuries many destructions, reconstructions and additions went over one of the main places of Christianity until now. The Rock of the Calvary is fully covered with glass since some years. Especially at the bottom the powdery result of a continued destruction of the rock can be observed. With scientific investigations the origin of the decay had to be found to recommend concepts for the future. This work is supported by the Greek Orthodox Authorities and the Fondation Heritage Orthodoxe, Geneva.

The scientific methods were X-ray diffraction, thin section with polarized microscope, scanning electron microscopy with EDX, measuring of porosity, absorption spectroscopy and climate measurement by data logger.

The rock of the Calvary is a relic of a limestone quarry, this remaining material was found not to be good for use. This is one of the reasons for the actual state. The limestone has a very low porosity and a high part of small pores. It contains traces of hematite and dolomite, which makes the typical reddish colour. The sand-like loss of the solid material has a high amount of nitrates and chlorides, the solid material is free of salts. That means the decay starts at the surface with an intrusion of salts in the inter-crystalline space, followed by a destruction of the crystalline parts. The origin of the salts seems – at the moment – to be in the use of holy water, which contains sodium chloride for avoiding growth of algae.

A short climate check showed a quite constant temperature inside the glass covering (19 – 20 °C), but the humidity changed at the same time with a range unto 15 % relative humidity (30- 46 % rH, and 52 – 64 % rH depending on the place).

At the actual state of the investigations we can consider that the original stone material of the Calvary is the basis for an attack of ritual ingredients used for long time, supported by climate changes. In the future the aim is to avoid larger ranges of humidity to create a constant climate inside the glass covering of the Golgotha.

### S36 16<sup>th</sup> Century Flemish-Portuguese Painting: The Pigments Behind Master Frei Carlos Technique

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Painting techniques and styles were changing in Portugal in the early 16<sup>th</sup> century, mainly due to the influence of the intense trade with the Flanders that brought in the new artistic movement which was developing there. Artistic items were being imported from Bruges and Antwerp, while Portuguese and Flemish masters were traveling between the two countries, bringing the Flemish style and technique to Portugal. "Portuguese-Flemish Painting" is a common expression used in the history of Portuguese painting of the first third of the sixteenth century and in its most basic meaning designates the work of Flemish masters who settled in Portugal during the reign of King Manuel (1495-1521) contributing decisively to the process of renewal of Portuguese painting at the time (Santos, 1971).

Frei Carlos, one of the most important "Portuguese-Flemish Painters", working in the region of Évora (active between 1517 and 1539-40), left a large number of works mainly that come from the Espinheiro's Convent where he made the Profession of Faith (in 1517).

This paper presents the comparative analytical study of three paintings attributed to Frei Carlos that come from the Espinheiro's nuclei, namely "Nativity", "Good Sheppard" and "Lamentation". The microsampling of representative areas was supported by physical imaging techniques namely macrophotography, visible light photography, raking light photography, UV-vis fluorescence photography, Infrared reflectography and X-ray radiography (Candeias et al., 2011). The collected microsamples were mounted in resin to show all the stratigraphic structure (cross-sections), making the interior layers available for further analysis by Raman spectroscopy and scanning electron microscope coupled with energy dispersive X-ray spectrometry (SEM-EDS). The material characterization was also performed by micro-FTIR allowing the identification of most pigments and binders applied in the technical execution of the paintings. Whenever necessary the mineralogical characterization was performed by micro X-ray diffraction. The combined analysis of the paintings allowed the identification of the main painting palette: lead white, yellow and red ochre, vermilion, azurite, lead tin yellow and carbon. Unusual green copper pigments were also identified namely brochantite and antlerite, which are usually associated with verdigris copper pigment (Hommes, 2004). In this case however the granulometry along with pigment morphology suggest a natural origin rather than an alteration product. High amounts of gypsum were found in the preparatory layers,

which is associated to the southern Europe technique. The study of this master work is fundamental to understand the profound changes that occurred in the Portuguese art during the reigns of Kings Manuel and João III.

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### **S37 The Difference Between Red, Red and Red on Unknown Walls of a Roman Villa. A Reconstruction through Combination of Mortar and Plaster Analysis**

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In 2008 and 2009 a Merovingian burial site at Borgharen, near Maastricht, the Netherlands was excavated. The Merovingians used the ruins of a Roman villa for their burials, which is situated on a high gravel bed, in the flood plain of the river Meuse.

During the excavation many small fragments of mortar and plaster were found. The study of fragmentary paintings can be helpful for the interpretation of provincial-Roman interior decoration in the Low Countries. It allows for the comparison between Roman villae in this area, which is important for the process of Romanization of agriculture in the first and second century A.D.

About 200 plaster fragments were investigated, varying from 1 cm to 5 cm in diameter, of which about 50 were described. On the basis of the structure of each fragment (thickness and composition of mortar layers, inclusions such as organic material, lime, minerals, colour, grain size) several types and subtypes could be distinguished. Of the additional 150 non-identified fragments, the painted upper lime coat layer was analyzed with hand-held xrf.

The results show that there is a good correspondence between the mortar type and the chemical analysis of the painted lime coat. Several types of ochre and other pigments could be distinguished, applied on at least two different types of mortars. The red pigments used were limonite rich clay, two different kinds of hematite and red lead.

This study shows that the chemical characterization of plaster can aid the identification of fragments in addition to the traditional macroscopic study of colours, structure and texture of the plaster and mortar layers. In the case of Borgharen, it was possible to categorize the remaining non-identified mortar pieces, which greatly improved the possible interpretation of how the villa walls were decorated. In situations where material is very fragmented, almost identical colours were used and only small parts of the plaster have survived, rapid, non destructive chemical characterization may be a valuable addition to the palette of the archaeologist.

### S38 Ancient Mortar Production in Ostia, Italy

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This paper presents a study of mortar samples from seven brick masonry structures in Ostia, Italy, built between 117 and 160 CE. The sample set includes public, private, domestic, and monumental buildings, representing small- and large-scale construction projects undertaken by multiple groups of builders identified by DeLaine (2002). Variations in mortar mix design and durability could represent specific technical decisions with an intended result, opening the discussion of the agency, intent, and expertise of the ancient builders (Jackson et al., 2009).

Material analysis can determine whether mortars were produced in a way that likely would have fostered beneficial properties or limited deleterious features. The geological provenance and particle size distribution of the aggregate have been identified and quantified with thin section petrography and image analysis; the nature, micro-structure, and geochemical profile of the binder and cementitious components have been investigated with X-ray diffraction and scanning electron microscopy. Analytical results are under review to determine the relative quality and durability of the ancient mortars.

Preliminary results reveal three distinct mortar mix designs within the sample suite. Although all samples contain multiple types of volcanic materials, each mix displays a strongly dominant aggregate material from different lithological units local to Ostia and Rome in central Italy. The selection of these particular materials is significant because they contain natural alteration products that contribute to the strength and durability of the mortar (Moropoulou et al., 2004). Several samples also contain fibrous cements formed within the vesicles of aggregate clasts during later-stage cementation. Similar aggregate profiles and cementation products have been found in mortar samples from contemporary structures in Rome, suggesting the possible spread of specific design mixes and processing techniques across the region (Jackson et al., 2010).

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### S39 Carbon and Oxygen Isotope Signatures of Historical Lime Mortar

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Historical lime mortar represents a record for environmental conditions. The chemical and isotopic composition of mortar provides information about raw materials provenance, processing and weathering. The stable isotope ( $^{13}\text{C}$  and  $^{18}\text{O}$ ) analysis allows establishing environmental conditions during calcite formation and secondary processes responsible for the material degradation (Kosednar-Legenstein et al., 2008; Dotsika et al., 2009).

The old lime mortars are basically a mixture of lime, water and sand. During binder formation atmospheric  $\text{CO}_2$  is fixed in the calcite and isotope fractionation starts mostly due to the hydroxylation of  $\text{CO}_2$ . The  $\delta^{13}\text{C}$  of binder depends on the isotopic values of  $\text{CO}_2$  and water. The relative  $^{13}\text{C}$  enrichment in gaseous  $\text{CO}_2$  and calcite is related with the kinetic isotope fractionation during binder formation.

Isotopic data of the calcite from mortar represents non-isotopic equilibrium explained by a continuous enrichment of  $\delta^{13}\text{C}$ . Considering values for  $\delta^{13}\text{C}_{\text{CO}_2} = -6\text{‰}$  and  $-9\text{‰}$  of the Earth's atmosphere (Eiler & Schauble, 2004), the isotopic composition is  $\delta^{13}\text{C}_{\text{calcite}} = -24\text{‰}$  and  $-27\text{‰}$ . Variations from ideal isotopic behavior can be because of secondary effects as  $\text{H}_2\text{O}$  evaporation, biogenic origin of  $\text{CO}_2$ , relicts of unburned limestone, and calcite recrystallization.

This study is focused on the isotopic composition of datable pure binder of carbonate mortar of roman, medieval and modern periods from historic buildings of the northern Spain, the church of Santa Maria la Real (Zarautz), Isturitzaga Tower (Andoain) and the church of Santabata (Mutriku). Isotopic values of calcite of historical lime mortar comprise a range of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  values from  $-9.4\text{‰}$  to  $17.4\text{‰}$  and  $-12.7\text{‰}$  to  $-18.1\text{‰}$ , respectively. Local limestone which was the main source of raw material for lime production has  $\delta^{13}\text{C}$  between  $1.8\text{‰}$  and  $2.3\text{‰}$  and  $\delta^{18}\text{O}$  between  $-2.8\text{‰}$  and  $3.6\text{‰}$ . Therefore the isotopic values of binder of lime mortar are isotopically lighter compared to local limestone used for burning.

The study is supported by IT315-10 research project of the Basque Country Government and EHU10-32 of the Basque Country University.

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**A1 Review of the Dating Methods Applied to Building Archaeology**

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The integration of building material dating methods into building archaeology has resulted in the advance of qualitative and quantitative information available for the history of the building.

In order to examine the chronology of historical buildings and consider the question of the origin of building materials, a European group of research entitled “Ceramic building materials (CBM) and new dating methods” is involved in the archaeological study of early medieval buildings (5<sup>th</sup> – 12<sup>th</sup> century) in France, England and Italy, all displaying CBM in their masonry structures. The team is made up of European specialists in the fields of building archaeology, history of art and chronology. The combination of luminescence and archaeomagnetism dating methods applied on CBM and radiocarbon dating on charcoals from within the mortar and dendrochronology on wood beams, has helped to shed light on the reuse of Roman *spolia* (mainly in Anglo-Saxon and Norman parish churches) and/or early medieval materials (Savigny’s abbay church), *ad novo* materials (Notre-Dame-sous-Terre in Mont-Saint-Michel, Saint-Martin at Angers in France, Castelletto priory in Italy, ...) or even a combination of both in the same structure (St-Irénée’s church, Lyons). In the case of production contemporary to the building, it is therefore possible to precisely map the chronology of buildings leading to, in some cases, a need to reconsider the traditional chronological attribution based on written sources or a chronomorphological approach. Furthermore, it has unveiled unexpected evidence of the early medieval brickmaking process.

Finally, since the chronological studies allow the architecture to be dated with *Termini Post Quem* by examination of the building materials, a ‘next-level’ approach is already being investigated: the direct dating of the edification. This can be seen with the emergence of OSL on mortar, which is the next step to investigate and develop in building chronology studies.

## **A2 Ceramic Residue Dating: Predictive Testing as a First Step in Selecting Appropriate Samples**

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Burned or charred ceramic residue has gained a reputation for producing dates that are “too old”. Then, again, some dates seem to fit the local sequence. Determining when to trust ceramic residue dates and when to avoid them is one of the most engaging questions in archaeochronometry today. Identifying the contents of the residue is likely to be extremely important in the quest for reliable dates on ceramic residues. Studies and tests to identify the problem have pointed to old carbon introduced from aquatic resources as a major problem in dating ceramic residues from northern Europe. Where else might the same source of old carbon be contributing to unreliable dates? Dates on charred ceramic residues from North America are shown to be identical to dates on annuals at some sites and discrepant by up to a few hundred years at other sites. What factors contribute to this difference?

We examine the archaeological record for evidence of fish or other aquatic products, identify organic residues in the ceramic residues and the fabric of the ceramics looking for evidence of fish or other aquatic products, then present paired dates. Which of the tests at our disposal are relevant to predict whether or not the dates on charred ceramic residue will be similar to those on annuals? How reliable are the tests? We have examined and continue to work with both isotope and FTIR analysis. Our current research continues to examine evidence from the central portion of the United States as a test case to develop predictive tests. After developing predictive criteria that have focused on presence or absence of evidence for fish in the residue and use (or absence) of shell-temper in the ceramic fabric, we have dated both ceramic residues and annuals from sites with these characteristics and sites without any evidence of fish or shell-tempered pottery. This paper presents our most recent results, which includes a set of residue and annual dates that all calibrate to the same age from one site that had no fish identified in the residue and no shell temper. Anomalously old dates are reported from other sites on residue that contained a signature consistent with the presence of fish and/or shell-temper in the ceramics. As our predictive criteria and tests are refined, our ability to predict which sherds to avoid improves.

### **A3 Evaluation of the Internal $\gamma$ Dose by Means of Monte Carlo Simulation in TL, OSL and ESR Dating**

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The absolute dating methodologies mainly used in archaeometry are based on luminescence technique, thermoluminescence (TL) and optically stimulated luminescence (OSL), and on electron spin resonance (ESR). In all of the cases the evaluation of annual dose is a crucial step above all when it is impossible to evaluate experimentally the internal dose contribution due to gamma emissions of U, Th and K chains present in the sample to be dated. This often happens for different samples and it making not datable the event of interest, unless we accept approximations properly justified. This approach is based on regular geometry considerations that it is difficult to relate to real cases.

In this paper we propose a methodology for the internal gamma dose evaluation of a sample of any geometry, using the Monte Carlo simulation code MCNP5. An important step of the procedure regards the 3D virtual geometry reconstruction in MCNP format through processing DICOM files obtained from the CT slices performed on the same sample. The 3D virtual geometry of the sample, the density and the elemental concentration of the materials, together with gamma spectra emitted by radioelements of the chains and their probability distributions, represent the input data for the simulation procedure. The Monte Carlo simulation allows an estimate of the absorbed dose in any volume of the structure considering the real geometry.

In this occasion we present the results obtained on test structures, first to validate the methodology of calculation, then to assess the internal doses of samples actually dated.

## **A4 Influence of Fired Clay Ceramic Composition and Contaminants on Rehydroxylation Dating**

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Fired clay ceramics undergo a slow rehydroxylation process (Wilson et al., 2003) as the ceramic recombines with environmental moisture which is the basis of a new dating technique for archeological ceramics (Wilson et al., 2009). Dating is carried out by first dehydroxylating the sample at 500 °C followed by measuring the slow mass gain in a controlled environment (Savage et al., 2008). We show in detail how the refractory and non-refractory mass components of a ceramic piece contribute to the calculated age and how to both measure and account for these components in calculating the age of a ceramic. In particular we emphasize non refractory components (organic carbon) which are present in many excavated ceramic objects and show how analysis of the organic carbon content before and after dehydroxylation at 500 °C can have an impact on the calculated age of the object. We show compositional analysis and calculated ages for two very different ceramics: A coarse grained, Anglo-Saxon, fired clay loomweight from West Berkshire, UK and more finely grained, Werra earthenware, sherds from a wastepit excavated at Enkhuizen (NL) in 1979. Both objects have been dated using independent methods including radiocarbon (the loomweight) and an inscribed date (Werra earthenware). We show how slow mass gain data is analyzed and discuss statistical uncertainties associated with analysis of the data and the impact on calculated sample age.

WILSON, M.A., HOFF, W.D., HALL, C., MCKAY, B. AND HILEY, A., 2003. Kinetics of moisture expansion in fired clay ceramics. *Physical Review Letters* **90**, 125503.

WILSON, M.A., CARTER, M.A., HALL, C., HOFF, W.D., INCE, C., SAVAGE, S.D., MCKAY, B. AND BETTS, I. M., 2009. Dating fired clay ceramics using long term power law rehydroxylation kinetics. *Proceedings of the Royal Society A* **465**, 2407-2415.

SAVAGE, S.D., WILSON, M.A., CARTER, M.A., MCKAY, B., HOFF, W.D. AND HALL, C., 2008. Mass gain due to the chemical recombination of water in fired clay bricks. *Journal of the American Ceramic Society* **91**, 3396-3398.

## A5 Investigation of Lime Mortars and Plasters using Electron Paramagnetic Resonance

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This study presents the preliminary results of investigation of the types and dynamics of paramagnetic centres in lime mortars and plasters from two sites: Sveta Petka church in Budinjak (Croatia) and archaeological excavation in Hippos (Israel), using Electron Spin Resonance (ESR) spectroscopy, in order to characterize the building material and to evaluate the possibility of dating them. ESR is widely used in dosimetry and dating of geological and archaeological materials (Ikeya, 1993, 2004). Lime mortar is valuable but problematic material for luminescence and radiocarbon dating (Goedicke, 2011; Nawrocka et al., 2005, 2007, 2009). It has not been dated before using ESR; therefore, careful studies are required to identify the useful paramagnetic centres. The excavation in Budinjak discovered a very unique four lobed plan object Sveta Petka, with no additional finds or reliable historical records about the time of its construction. ([http://www.park-zumberak.hr/destinacije/budinjak\\_en](http://www.park-zumberak.hr/destinacije/budinjak_en)).

The ancient settlement Hippos (Sussita) situated on the east shore of the Sea of Galilee functioned from the 3<sup>rd</sup> cent. BC until it was destroyed by the earthquake in 749 AD. Samples were taken mainly from NW church and surrounding area. The petrographical analyses showed carbonate and locally gypsum character of binder and different kind of aggregate. Chosen samples in different fractions were dated by AMS and GPC technique. (Nawrocka et al., 2005, 2007, 2009). All samples were  $\gamma$ -irradiated in <sup>60</sup>C bomb with the doses of 1, 10, 20, 50, 80 and 100 kGy. The measurements were performed at room temperature using EPR spectrometer working at 9 GHz frequency. In all spectra signals from Fe<sup>3+</sup> and Mn<sup>2+</sup> ions have been observed. Paramagnetic centres which give the ESR signals may be interpreted as CO<sub>2</sub><sup>-</sup>, CO<sub>3</sub><sup>-</sup>, CO<sub>3</sub><sup>3-</sup>, HCO<sub>3</sub><sup>2-</sup>, SO<sub>2</sub><sup>-</sup>, SO<sub>3</sub><sup>-</sup>, PO<sub>2</sub><sup>-</sup> and PO<sub>3</sub><sup>2-</sup> species (Barabas et al., 1992; Polikreti et al., 2004; Seletchi & Dului, 2007; Wencka & Krziminiewski, 2004). In case of samples from Budinjak the changes in ESR signals amplitude measured at magnetic field range about 3440 – 3450 G were analysed versus the dose of irradiation, using Mn<sup>2+</sup> signals as a reference. For samples from Hippos amplitudes were measured after subtraction of non-irradiated samples spectra. In both cases exponential growth of the curve and saturation for doses above 20 kGy were observed; therefore, irradiation with smaller doses is required. Differences in presence of some paramagnetic centres and the level of dose saturation between the investigated samples are connected with different age of samples, their localization and preparation technology.

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## A6 The Chiavenna Evangelistary Cover: A Radiocarbon Dating of a Supposed Ottonian Goldsmiths' Work

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The so-called "Pace di Chiavenna" is an Evangelistary cover, stylistically and iconographically attributed to the Ottonian age (950-1050). It is composed of a wooden plate, covered with crafted gold sheets, gems and *cloisonné* enamels, the latter fixed to the structure by a beeswax compound. There were no clues of the exact date of production of this work, the most similar Evangelistary cover being the one given to the Milanese Church by archbishop Ariberto da Intimiano (1018-1045) (Maggioni, 1996, 2011).

A restoration campaign, promoted by Catholic University of Milan among research project 'La Pace di Chiavenna svelata', carried out in the last two years allowed us to take two samples from the wooden frame, and a sample from the wax compound. The samples were treated to be analyzed via AMS, to perform a radiocarbon dating. In particular, it was possible to wiggle-match the two wooden samples, since they came from the same plank, in which the rings were clearly distinguishable. The obtained radiocarbon dates were calibrated to obtain a probability density on a chronological scale, allowing the determination of the crafting period.

The materials used in the construction of the cover are dated in a period in good agreement with the historical setting.

MAGGIONI, C., 1996. Un capolavoro dell'oreficeria ottoniana milanese: la Pace di Chiavenna. *Arte Lombarda* **116**, 8-18.

MAGGIONI, C., 2011. L'Evangelario erratico ora a Chiavenna, La bellezza nella Parola. Il nuovo Evangelario Ambrosiano e capolavori antichi. *Catalogo della mostra*, 32-38.

**A7 Archaeometric Studies of the Graphics of the Russian Artist P.N. Filonov**  
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Analysis of grounds (consisted of analysis of chemical composition, color and structure) of 23 works of the well-known Russian artist O. A. Kiprensky (1782-1836), carried out earlier (Mazina et al., 2008), made it possible to divide them into three different periods of creation reliably. These investigations showed that the results of studies of materials' science can be used in archaeometric and also in a number of cases in the differentiation of the works of one author from another.

The studies of paper and pigments used by Russian artist P.N. Filonov (1883-1941) in his 23 graphics were made using the same approach.

In our investigation the following methods were used: 1) a study in the ultraviolet rays (max = of 365 nm) and the neighbor IR range of the spectrum (800 - 1000 nm); 2) determination of the optical characteristics of paper (color, character of the luminescence, excited by ultraviolet emission); 3) local X-ray spectral analysis; 4) determination of the composition of the fibrous components of paper and 5) micro-chemical analysis.

Based on the conducted investigations the following conclusions have been made:

1. 4 works out of 23 were not painted by P. N. Filonov as they do not correspond to the time of their creation (1900-1920-th years) – because of a presence of pigments, synthesized later in 1930-th years.
2. 12 works out of 23 were created on the paper basis, which were prepared until 1950 (corresponding fibrous semifinished products were used).
3. 11 works out of 23 might be created in the period after 1934 as a result of presence of pigments, synthesized in 1930-th years.

MAZINA A.YA., GOROKHOVA G.N., KONONOVICH M.G. AND SOKOLOV S.V., 2008. Study of the painting materials, used by Russian artist Kiprensky, at different periods of his creative work. *Program and abstracts of the 37<sup>th</sup> International Symposium on Archaeometry*. Siena, Italy.



## **A8 Optically Stimulated Luminescence Chronology and Characterisation of Pottery Sherds from Maligrad, Albania**

**Artemios Oikonomou<sup>1</sup>, Konstantinos Stamoulis<sup>2</sup>, Petrika Lera<sup>3</sup>, Stavros Oikonomidis<sup>4</sup>, Aris Papayiannis<sup>5</sup>, Akis Tsonos<sup>6</sup>, Christina Papachristodou-lou<sup>7</sup> and Konstantinos Ioannides<sup>2,7</sup>**

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This work reports the results of an ongoing research using the optically stimulated luminescence (OSL) chronometry methodology and the radioisotope-induced energy-dispersive X-ray fluorescence (EDXRF) spectroscopy for the characterization of pottery sherds recovered from Maligrad, Albania. Maligrad is a rocky islet of karstic tectonic formation at the westernmost edge of the Great Prespa Lake in SE Albania. The Institute for Transbalkan Cultural Cooperation (ITCC) and the Institute of Archaeology of Tirana are conducting a multi-disciplinary archaeological research on Maligrad since 2007. The main objectives of this Greek-Albanian archaeological expedition are the combined study of both the islet and the neighboring area along the coastline of Prespa and the definition of the life span of this specific area (Lera, 2009, 2010, 2011). Fifty five pottery sherds and surrounding sediment samples were collected from Maligrad. Also ten pottery sherds were recovered from neighboring areas. All samples were sent for analysis to the laboratories of the Archaeometry Center of the University of Ioannina, Greece. The samples were dated using the Riso TL/OSL DA-15C/D reader. The single-aliquot regenerative-dose (SAR) protocol was followed for the equivalent dose (De) determination. The OSL natural signal was obtained by stimulating the samples with blue LEDs. Also each sample was repeatedly irradiated for various periods of time with a <sup>90</sup>Sr/<sup>90</sup>Y source with a calibrated dose rate of  $0.0982 \pm 0.0002$  Gy/s. To determine the dose rates, the natural radioactivity of sediments from the surroundings of the original sample location, due to potassium 40 (<sup>40</sup>K) and the uranium (U) and thorium (Th) series was assessed, using gamma spectrometry with a counting system based on a HPGe detector. A first result for the estimated age of the sherd samples is 190 BC  $\pm$  150 years. According to archeological data collected during the seasons 2009-2011, the oldest findings belong to the Middle Bronze Age and are related to a settlement of the same period. Finally the elemental composition of the ceramic bodies is studied using radioisotope-induced EDXRF spectroscopy. The multivariate statistical treatment of the elemental data will reveal if different compositional groups exist and also the differentiation between local sherds and sherds collected from the neighbouring to Maligrad locations.

LERA, P., OIKONOMIDIS, S., PAPAYANNIS, A. AND TSONOS A., 2009, 2010, 2011. *Annual Reports of the Foreign Archaeological Missions in Albania*. Tirana.

## A9 Optically Stimulated Luminescence Dating of Samples from Tall al-Kafrayn, Jordan

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Tall al-Kafrayn is an isolated cone-shaped site, rising 35 m above the Jordan valley floor. The small, flattened summit of this natural rock-hill has been used during the last century as a cemetery. In ancient times it was the site of a fortress or fortified village, commanding a strategic location astride an established communication caravan route. The ancient habitation of the site is attested by visible architectural remains of several buildings, which suggest the existence of an important settlement. In 2000, a Hellenic research excavation project directed by two of the authors (Papadopoulos & Kontorli-Papadopoulou, 2007) commenced in Jordan. The present work aims at the dating of sherds and sediments recovered from trenches during the excavation campaign in spring 2011, using the Optically Stimulated Luminescence (OSL) methodology (Aitken, 1998). Samples from three trenches near the top of the Tall (hill), were collected and sent to the laboratories of the Archaeometry Center of the University of Ioannina, Greece, where they were analyzed. The samples, sherds and sediments, were dated using the Riso TL/OSL DA-15C/D reader. The single-aliquot regenerative-dose (SAR) protocol was followed for the equivalent dose (De) determination. The OSL natural signal was obtained by stimulating the samples with blue LEDs. Also each sample was repeatedly irradiated for various periods of time with a <sup>90</sup>Sr/<sup>90</sup>Y source with a calibrated dose rate of  $0.0982 \pm 0.0002$  Gy/s. To determine the dose rates, the natural radioactivity of sediments from the surroundings of the original sample location, due to potassium 40 (<sup>40</sup>K) and the uranium (U) and thorium (Th) series radioisotopes was assessed, using gamma spectrometry with a counting system based on a HPGe detector. The estimated ages of the sherd samples varied from 1400 BC  $\pm$  800 years to 2700 BC  $\pm$  1700 years, while the ages determined for the sediment samples ranged from 720 BC  $\pm$  630 years to 1100 BC  $\pm$  200 years. An ongoing research, using analysis techniques like the radioisotope-induced energy-dispersive X-ray fluorescence (EDXRF) spectroscopy and X-ray diffraction (XRD) hopefully will reveal the sources of the observed age variation and the differences between the ages derived for sediments and the ages for sherds surrounded by the same sediments.

PAPADOPOULOS, T. AND KONTRORLI-PAPADOPOULOU, L., 2007. The Hellenic Archaeological Project of the University of Ioannina in Jordan: A Preliminary Synthesis of the Excavation Results at Ghawr as-Safi and Tall al-Kafrayn (2000-2004). In: FAWWAZ AL-KHRAYSHEH (Ed.) *Studies in the History and Archaeology of Jordan*. Department of Antiquities, Amman, Jordan.

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## **A10 Analytical Study of Ancient Artifacts-Bone, Glass, Pottery and Pigments from Archaeological Site of *Tell al-Husn* and *Khirbeted-Darieh* in Jordan**

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This work presents an idea about the role of scientific techniques in the cultural heritage and archaeological materials. These techniques which comes mainly from physics and chemistry give the trend to conserve and preserve the tangible objects in Arts and Cultural Heritage such as glass, ceramics, coins, monuments, landscapes, and all other artifacts in the Museums for supporting the continuity of the intangible items as languages arts, music, etc. It focuses on the main tasks of the museums which are taking care of artifacts, handling the object and restoring them when needed in the appropriate store, providing the proper environment.

During the excavation phase of the work adopted by the team in Yarmouk University in 2008 at *Tell al-Husn* and *Khirbeted-Darieh*, with the support of the Jordanian Department of Antiquities archaeological. This site is located in the northern part of Jordan, a significant set of artifacts, bone, glass, ceramics, pigments objects found in the sites. New trend of Raman dating method of bone objects is very important and not expensive method to reveal the historical and archaeological aspects of these significant sites in Jordan. Raman spectroscopy technique is a good method for studying several types of bone and it is particularly a new trend for dating of bone. A collection of bone fragments was collected and prepared for measurement using this new technique from both sites. It shown that bone objects are dated back around to 2000 years (Roman Age). This age is consistent with that estimated by archaeological (typology) studies. Also this study will cover several methods and techniques which I have used at the university of Jena in Germany in the last summer (from 01.06.2011 to 01.09.2011) on the characterization of several artifacts samples, dated from prehistoric to Roman times. In particular, X-ray Diffraction (XRD), SEM-EDX coupled with Energy Dispersive X-ray system, BSE, were used for the determination of the morphological, chemical and mineralogical characteristics of the artifacts from the sites of *Tell al-Husn* and *Kherbat Al Darieh* in Jordan.

## **A11 Historical Building Dating: The Multidisciplinary Study of the Convento de São Francisco (Coimbra, Portugal)**

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The radiometric dating of a building by stimulated luminescence is currently based on the chronology obtained by Thermoluminescence and/or Optically Stimulated Luminescence dating of bricks. The assumption is that the manufacturing of a terracotta brick happened almost contemporary to or, at least, not much earlier than the moment of its laying in the building's construction.

This represents an obvious limitation of the method, especially when the studied building presents a complex history of diachronic construction/modification phases. In fact, TL dating of bricks can fail to identify the period of construction and/or successive modifications of the buildings if there was no reuse of older materials.

In order to overcome these limitations in dating historical buildings, it is possible to date the mortar (the binder used to connect the building's elements). The natural dosimeters are quartz or feldspar in the sand fraction and the zero event can be related to exposure at daylight during the mixing and laying of the mortar.

The possibility to use mortars to evaluate the time of construction, repair works or modifications of a structure, represents an important break-through in dating historical buildings.

This, with the application of alternative techniques can solve the problem of reuse materials, but however not solve the problem related to low statistical sampling. The result is a higher reliability of individual data.

Such cases demand for cross-checking between (1) dating results, (2) macroscopic interpretation of the structure and (3) microscopic and colorimetric characterization of the studied materials.

In the case of Convento de S. Francisco study, the results of optically stimulated luminescence dating using mortar and bricks were crossed with optical microscope observations, ICPMass results, mineralogical characterization by XRD and colorimetric data.

This multidisciplinary approach has allowed us to overcome the limitations of applying a single technique such as stimulated luminescence dating for the solution of a chronological problem, thus improving our ability to precisely reconstruct the historical events that have affected the building.

## **A12 New Archaeointensity Results from Greek Ceramics and their Contribution on the Intensity Secular Variation in Greece**

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Archaeomagnetic data are an important source of information in order to retrieve the variations of the Earth's magnetic field during the past few millennia. However, a large number of well dated and high quality archaeomagnetic results are necessary to reconstruct the detailed geomagnetic field variations in order to be used for archaeomagnetic dating applications. We present here, new archaeointensity results from ceramic collections coming from four different archaeological sites in Greece. The ages of the ceramic fragments, based on archaeological constraints and radiocarbon analysis, range from 2200 BC to 565 AD.

The new absolute archaeointensity data are based on several archaeointensity determinations per ceramic fragment and per site and were obtained using the Thellier classical method with regular partial thermoremanent magnetization checks. The effect of TRM anisotropy and cooling rate upon TRM acquisition has been investigated in all the specimens. Therefore, the new data can be considered as reliable markers of past geomagnetic field intensity in Greece. The obtained mean site intensities *in situ* range from  $53.6 \pm 4.1$  to  $69.3 \pm 3.9$   $\mu\text{T}$ .

The new data have been compared with other available data from Greece as well as with the Greek and Balkan reference secular variation curves and regional and global geomagnetic field models. Combined with previous published data from the area, they confirm that important changes of the Earth's magnetic field intensity occurred in Greece during the last five millennia. Nevertheless, for some periods, the available archaeointensity data for Greece, even if numerous, are quite dispersed. Undoubtedly, this shows that more well dated archaeointensity data are still necessary in order to restrict the error uncertainties of the Greek secular variation curve that subsequently will result to the increase of the archaeomagnetic dating accuracy in this area.

### **A13 Archeomagnetic Dating in Italy Based on the Full Geomagnetic Field Vector: New Results from Italian Kilns**

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Archaeomagnetic dating is based on the comparison between the geomagnetic field vector recorded on baked archaeological material during their last firing and the reference secular variation curves that describe the temporal changes of the local geomagnetic field in the past. However, in most of the up to now published studies, only two of the geomagnetic field elements (declination and inclination) are used. In this study we present the dating of three Italian kilns inferred from archaeomagnetic measurements of the full geomagnetic field vector (declination, inclination and intensity). The three kilns (Ascoli Satriano, Vagnari and Fontanetto Po kiln) have been studied for archaeointensity determination with the Thellier modified by Coe method. We identified magnetite and Ti-magnetite as the main magnetic minerals, with some minor hematite only in few cases. Magnetic susceptibility versus temperature shows a good thermal stability of the samples. The intensity results have been corrected for anisotropy of the thermoremanent magnetization and cooling rate effects. The archaeodirections of the Ascoli Satriano and Vagnari kilns have been previously studied and published by Tema *et al.* (2006). For the Fontanetto Po kiln only the archaeomagnetic inclination has been defined because the presence of methane metallic tubes in a depth around 1 m below the kiln, and the bad weather conditions during sampling, prevented the use of magnetic compass and the sun orientation of the samples, respectively (Tema *et al.*, 2011). The directional results of the three kilns together with the new intensity determinations have been used for comparison with the reference secular variation curves. The archaeomagnetic ages of the kilns have been obtained with the Matlab Tool developed for archaeomagnetic dating by Pavón-Carrasco *et al.* (2011) and the reference secular variation curves calculated directly at the sampling sites by the SCHA.DIF.3K regional geomagnetic field model. The final dating intervals have been calculated after combination of temporal probability density functions of the three geomagnetic field elements, except for the Fontanetto Po kiln for which only inclination and intensity values were available. The ages obtained are in good agreement with the archaeological evidences and suggest that, when it is possible, the full geomagnetic field vector must be used for archaeomagnetic dating.

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#### **A14 Chronological Evidence for the Mesolithic Archaeological Site of Damnoni at Plakias (Crete) Using OSL Dating**

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The Plakias Archaeological Survey conducted by the American School of Classical Studies and the Ephoreia of Palaeoanthropology-Speleology of Southern Greece discovered Mesolithic and Palaeolithic artifacts in datable geologic contexts. Following that systematic survey, an excavation was conducted at the site of Damnoni in the summer of 2011 that provided evidence of manmade tools which are morphologically similar (e.g. microliths, scrapers, etc..) to the Mesolithic industry that dates to ca. 9000-7000 B.C (Strasser et al., 2010, 2011).

The aim of the study is to present the chronological data derived from the application of luminescence dating on sediments from the excavated trenches. Three plots were chosen to take six samples in total that follow identical stratigraphic units. Single aliquot and single-grain OSL dating (Murray & Wintle, 2000; Zacharias et al., 2008, 2009) was used on pure quartz aliquots recovered from the sediment material. Field radiation was monitored *in situ* by placing sensitive dosimetry phosphors; additional mineralogical and chemical analyses was used to assist the environmental dosimetry studies. The results verified for a single and closely bracketed event that dates to the Mesolithic Era, highlighting the importance of the Damnoni site to Aegean archaeology.

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### R1 Collagen-Bioapatite Radiocarbon Age Differences Linked to Reservoir Effect Ricardo Fernandes, Pieter M. Grootes and Marie-Josée Nadeau

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Radiocarbon dating represents the most widely used method for the establishment of absolute chronologies, often relying on the dating of human bone material. Reservoir effect is the expression used to denote the presence of a radiocarbon-depleted signal within an aquatic reservoir. Humans with an aquatic diet will incorporate a reservoir effect signal. Radiocarbon dating of this bone material results in apparently older chronologies.

The main organic fraction of bone, collagen, is usually preferred for radiocarbon dating. In contrast, bone's inorganic fraction, bioapatite, is less often radiocarbon dated due to its high susceptibility to diagenesis and contamination. Bioapatite carbonate is in equilibrium with blood bicarbonate, which is derived from respired CO<sub>2</sub> (Jim et al., 2004). Thus, bioapatite carbon is routed from the entire dietary mix (Froehle et al., 2010), mostly represented by carbohydrates. In contrast, bone collagen will reflect primarily protein intake. As fish is particularly rich in protein and depleted in carbohydrates, collagen-bioapatite radiocarbon age differences should be observed in fish consumers and provide an estimate of reservoir effect.

Based on recently published compiled results of controlled feeding experiments of animals (Froehle et al., 2010), a simple algebraic model is presented establishing the routing of dietary protein and energy (carbohydrates and lipids) towards bone collagen and bioapatite.

This model was applied to recently published data (Cherkinsky, 2009), consisting of stable isotope analyses ( $\delta^{13}\text{C}$ ) and radiocarbon dates of both collagen and bioapatite, on well preserved human remains found in Stone Age shell middens in the Western Cape Province of South Africa. The model was capable of establishing a clear linear relationship between dietary energy intake and individual reservoir effect. It was also capable of accurately predicting the local reservoir effect and the local plant isotopic signal. These results provide the first evidence for collagen-bioapatite radiocarbon age differences linked to reservoir effect, and open the possibility of using the presented model to improve radiocarbon-based chronologies.

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## **R2 Dating Mortars: Three Medieval Spanish Architectures**

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One of the major issues in building archaeology is finding the age of elements and structures discovered. Mortars represent a class of material basically constituted by a mixture of different phases (i.e. binder, aggregates, water) and are widely used for constructive uses and artworks. Current scientific literature regarding the possibility of accurate radiocarbon dating for mortars reports different and still contradictory results.

In this study, a new protocol for radiocarbon dating of mortar developed at the Centre for Isotopic Research on Cultural and Environmental heritage (CIRCE) is used to perform <sup>14</sup>C measurements on archaeological mortars coming from three medieval architectures of northern Spain (two churches and the walls of a castle). Results observed will be discussed and compared with independent age estimations (i.e. radiocarbon dating performed on organic materials found in the same study site, archaeological analyses) in order to frame experimental observations in the actual site knowledge by means of a multidisciplinary approach.

### **R3 Radiocarbon Dating of Mortars: A New Reliable Protocol**

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To date, mortar radiocarbon dating represents one of the main “open issues” involving the whole radiocarbon community because of the discrepancies observed over the almost 4 decades of experimentations. The technology used for the production of aerial mortar, even if modified over time, preserves the potential to establish the absolute chronology of studied artifacts because the setting process is primarily addressable to the atmospheric CO<sub>2</sub> adsorption (carbonatation). This potential was firstly guessed by Delibrias & Labeyrie (1965), which began a series of measurement campaigns to assess the feasibility of the methodology but, to date, the clues about the effective accuracy of the mortar dating remain still unclear.

Since about one year the CIRCE (Centre for Isotopic Research on Cultural and Environmental heritage) <sup>14</sup>C group started an intense experimental campaign to develop a protocol able to guarantee a reliable mortar dating bypassing most of the literature observed pitfalls. Among the different sources of contamination, C of geological origins (primary) has been reported as one of the major sources introducing a sensitive ageing effect. <sup>14</sup>C ageing of mortars is due to this source of C introduced when different aliquots of primary carbonate survives to burning of limestone (calcination) and/or when carbonatic sand is used in the mortar production process.

CIRCE proposed methodology (CryoSoniC Cryobreaking, Sincation and Centrifugation), instead, is based on a sequence of physical separations.

First step of validation procedure was performed on a series of synthetic laboratory mortars produced without the aggregate addition and applying different calcination temperatures. Observed results showed a successful binder selection efficiency allowing unbiased dating for mortars produced at functional calcination temperatures. CryoSoniC selectivity was then tested and implemented on: i) mortar lime lumps commonly addressed as the unique feasible alternative to the purification procedures; ii) real mortars coming from a series of chronologically constrained archaeological sites with the aim to check also for the influence of different mortar typologies on the effectiveness of the procedure.

This contribution aims to propose a definitive preparation pathway to follow in order to achieve the accurate dating of analyzed mortars.

DELIBRIAS, J. AND LABEYRIE, G., 1964. Dating of old mortars by the carbon-14 method. *Nature* **201**, 742.

#### **R4 Bones, Wood and Charcoal from Sowinki Medieval Cemetery (Central Poland) in Radiocarbon Dating**

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In Sowinki village, located in central Poland, the traces of human presence from different chronological period were noticed. Archaeological site, where the investigations were carried out, was classified as an early Medieval. The analyses were performed for the samples from skeletal cemetery.

The main aim of the research was to reconstruct the different stages of the cemetery existence and its surroundings. The radiocarbon dating included 15 different samples: the human bones, the fragments of wood, charcoal and the leather, dated by accelerator mass spectrometry (AMS).

The archaeometric research, especially petrographic and SEM observations of samples enable to choose the most appropriate fragments for radiocarbon dating. The identification of wood and charcoal fragments was also performed.

The <sup>14</sup>C dating results were compared with the relative chronology, established by archaeologist for 10<sup>th</sup> to the first half of 11<sup>th</sup> century AD. The preservation stage, environmental conditions on the site and different laboratory pretreatments were taken into account for the reason of verifications of the results.

The research was supported by the Polish Ministry of Science and Higher Education, within the grant no IP2010 027870 and Archaeological Museum in Poznań.

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## **R5 Radiocarbon Measurement in Photographic Materials**

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In some circumstances it is important to determine when a photograph was created. The nature of photographic reproduction means that images can be printed from negatives even decades after their original exposure, making the process of reconstructing the historical sequence of events in a photograph's creation challenging. As culturally and historically important photographs increase in value, the appearance of late 20<sup>th</sup> Century prints of early 20<sup>th</sup> Century photographs presents a vexing problem to historians of photography. Primacy is generally given to photographs both taken and printed by the same photographer, but other modes of creation exist, both legitimate and sinister.

The purpose of this research is to investigate whether radiocarbon measurement can be used to identify the manufacture date of photographic materials: negatives, photographic paper, and film stock. The approach focuses primarily on late 20<sup>th</sup> Century materials, as it is tracking the appearance of elevated environmental radiocarbon levels associated with above ground nuclear testing in photographic paper and emulsions. The pattern of appearance differs in these two materials due to their differing biological sources.

Although this approach cannot alone establish when such materials were used to make an image, it can provide strong terminus post quem data when examining putative 19<sup>th</sup> or early 20<sup>th</sup> Century photographs. Radiocarbon measurements on "known age" reference materials will be presented as well as measurements on questioned photographs from museum collections.

**M1 Final Bronze Age Copper Slags from Transacqua and Segonzano (Trentino, Italy)****Anna Addis, Ivana Angelini and Gilberto Artioli**

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The Trentino area (North-Eastern Italy) shows diffuse evidence of protohistoric copper smelting sites dated to the Recent and Final Bronze Age (Weisgerber & Goldenberg, 2004; Cierny, 2008; Bellintani et al., 2009). Recent archaeological excavations in the Transacqua and Segonzano sites by the “Soprintendenza per i beni librari archivistici e archeologici di Trento” (Dr. Paolo Bellintani, Dr. Elena Silvestri) yield abundant copper smelting slags, which are the object of the present study. Over 130 slags were fully investigated in order to understand: 1) the technological development of the smelting processes performed, 2) the possible use of different working-steps in the metal production process, 3) the ore source of the smelted minerals. On the basis of the macroscopic features the slags were grouped into two morphological types, coarse/cake slags and flat/Plattenschlacke slags in agreement with the literature, as well as a separate class here referred as massive, according to the recently defined groups of slags found in Luserna (Addis et al., submitted). The most representative slags of each group were analysed by X-ray powder diffraction, optical microscopy and electron microscopy. The result of the minero-petrographic analyses of the Transacqua slags indicate that the coarse slag samples, showing a quartz content of about 60 wt% and the presence of partially reacted chalcopyrite and copper sulphides, may be associated with the first matte-producing step of the smelting process. The massive slags, assumed to be the result of the second production step, can be divided in two sub-groups ( $M_1$  and  $M_2$ ).  $M_1$  is mostly composed by quartz (50 wt%), fayalite (31 wt%) and magnetite (9 wt%). The  $M_2$  group presents a higher content of fayalite (59 wt%) and magnetite (34 wt%), with quartz near or below 5 wt%. The  $M_1$  and  $M_2$  groups may be associated to successive steps of refinement of the matte, to matte produced with charges having different Cu contents, or to different conditions in the processing. The Cu-rich matte was then re-processed to produce raw metal and flat slags, which display a very high amount of fayalite (81 wt%) and the widespread occurrence of magnetite, matte, and copper prills. The results of analyses of the Transacqua and Segonzano smelting slags indicate that the copper extraction process was performed in at least three processing steps, maybe four in Transacqua, related to different levels of matte enrichment, copper reduction and separation efficiency.

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## **M2 Experimental Archaeometallurgy: Towards the Understanding of the Late Bronze Age Cu Extraction Process in the Eastern Alps**

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The Recent and Late Bronze Age were periods of marked copper extraction activities in the Eastern Alps. Several furnaces and countless sites are known with abundant smelting slags of various type, including the Plattenschlake of well-defined composition and morphology (Anguilano et al., 2002; Weisgerber & Goldenberg, 2004; Cierny, 2008). The reconstruction of the Late Bronze Age smelting process are still debated, especially concerning the number and nature of the different high temperature working-steps employed in the process. The detailed investigation of the copper slags found in several Eastern Italian Alps LBA sites suggest that the smelting process was performed in at least three standardised steps related to different levels of copper extraction efficiency (Addis et al., submitted). To the purpose of verifying the working steps assumed on the basis of the analyses of the archaeometallurgical slags, two seasons of copper smelting experiments were performed in the Summer of 2010 and 2011. The first session of experiments principally aimed to explore the effect of different roasting cycles on the process. A number of twenty roasting experiments starting from three different types of commercial chalcopyrite were carried out using different strategies: charge roasting in crucibles heated in a furnace, and a open air roasting on piles of wood. X-Ray powder diffraction analyses on the starting materials and on the roasted products were performed at different stages during the process, which was repeated several times. These experiments allowed to select the most suitable charge to be used for subsequent smelting, to understand the efficiency of roasting strategies, and to assess the degree of transformation in the ores. Several smelting experiments were performed using a charge of roasted/unroasted chalcopyrite plus quartz, inserted in crucibles within a furnace heated by artificial air source and coal. The mineralogical associations and the copper enrichment of the matte obtained during the transformations were measured. These analyses allowed stoichiometric optimization of the chalcopyrite/quartz ratio in the charge. The last experimental session was devoted to produce a highly copper-enriched matte from the optimized charge, and to understand the latter steps of the process including final copper extraction and formation of Plattenschlake-like slags. Based on the results of the experiments and in agreement with the observed features of the archaeological slags, a working 3-step process is proposed as the basic scheme for LBA copper extraction activity: (1) repeated roasting of the charge, (2) mass production of copper-enriched matte with formation of coarse and massive slags, (3) re-processing of the matte for the extraction of copper, with multiple production of flat slags.

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### **M3 Morphological Reconstruction of Roman Sagittae from *Iulia Concordia* (North-Eastern Italy)**

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*Iulia Concordia* (Venice, Italy) is a Roman settlement that assumed an important strategic role at the time of barbaric invasions (III-IV cent. A.D.). The city is known to have hosted important military forces and at least one weapon workshop, although to date the location of the archaeometallurgical site is undetermined. During the excavations of the old theatre area, evidence of metal working activities and about 40 *sagittae*, ancient metal arrows used by Roman archers, were found (Di Filippo Balestrazzi & Vigoni, 2009). A project has been developed in cooperation with the “Soprintendenza dei Beni Archeologici del Veneto” (E. Pettenò, F. Rinaldi and V. Tinè) in order to investigate the conservation status, the metal characteristics, the production and working processes and, possibly, the relationship between the different typologies of the *sagittae* and their practical functions. For our investigation 18 *sagittae* were selected from the 3 main typology groups, defined on the base of the arrowhead section shape: flat; triangular and quadrangular. The analytical work has been divided in two phases: 1) non invasive investigation of the morphology and conservation status of the *sagittae*; 2) selection of a few samples for invasive mineralogical, chemical and metallographic analyses. Here we will report the results of the first part of the project. Owing to their penetration power in dense matter (Squires, 1996), thermal neutrons are ideal tools for the bulk characterization of metal artefacts. Neutron techniques have been widely used to investigate the inner part of metal objects of archaeological (Siano et al., 2002) and historical (Grazzi et al., 2009) interest, obtaining information on phase composition and distribution of texture and residual strain. Moreover neutron tomography allows the reconstruction of the detailed 3D internal morphology. We will present here the result of a neutron imaging experiment carried out at the NEUTRA beamline (Lehmann et al., 1996) operating at the spallation neutron source SINQ in Switzerland. The 3D tomographic reconstructions of the *sagittae* allow a clear distinction of the main observed structural components, namely: well preserved residual iron metal, iron oxide alterations, internal cracks, and slag inclusions. The quadrangular section arrows generally show a good conservation status, with weathering and small cracks only visible in the proximity of the surface. On the contrary the *sagittae* with flat and triangular sections present a higher content of iron oxides and often exhibit intense fracturing, with cracks parallel to the surface and distributed through the core of the objects.

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#### **M4 The Metallurgical Workshop of Piazza Madonna di Loreto (Rome)**

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The site of Piazza Madonna di Loreto, in Rome, recovered through a commercial archaeology excavation during the works for the Construction of Metro C, uncovered a surprising metallurgical workshop of around 1500m<sup>2</sup>. The workshop, dated to the VI century AD is located in the Adrian *Atheneum* in the Trajan Forum (Serlorenzi, 2010). The excavation demonstrated the presence of several furnaces of different typologies, and the richness of the metallurgical debris and metal artefacts (ingots) also pointed towards several processes being carried on at the site at the same time. These considerations demonstrate the importance of this workshop within the Early Middle Ages. Also reflecting on the central position of the site we hypothesise that a highly important state activity was performed in this workshop. This paper will focus on the first archaeometric investigation carried on a small fraction of the material recovered at the site indicated that at least seven metallurgical processes were performed at the site: alloying to produce plumbiferous bronze; refinement of copper; cupellation; production of lead; silvering of the bronze alloy; assaying of copper ores; “re-working” of iron objects. The bronze alloy seems to derive from a mixture of copper, both recycled and derived from the smelting of ores probably carried out somewhere else after assaying at the site); tin inserted in the alloying process directly as cassiterite, and lead deriving from the reduction of the litharge resulting from the cupellation process. The silvering process seems to be performed with the use of silver chloride. Testing using Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is being performed to determine if this process involved a hot bath of cerargyrite (Cope, 1972) or the use of an amalgam including mercury (Vlachou, 2002). Overall this first screening of the material seems to indicate that the main production was of plumbiferous bronze and this was found in small ingots of rectangular shape (ten centimetres long times one by two centimetre as rectangular base), indicating that the final objects to be produced were of small dimensions. One planchet was also found at the site suggesting the possibility that the small objects finally produced at the site were coins.

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## M5 Characterization of Ancient Japanese Tsubas through Time of Flight Neutron Diffraction

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This work presents a systematic study of Japanese *Tsubas* (sword hand guards) carried out by means of the non-invasive technique of thermal neutron diffraction. The *tsuba* appears from the 10<sup>th</sup> century to protect the hand of the warrior during the fight. In contrast to Chinese and European equivalents, here the guard is made of a metal plate, detachable from the blade, so that its production could be accomplished by specialized craftsmen aiming to optimize this sword component characteristics. The first *tsubas*, produced with the main purpose of exploiting the best functional use only, were forged in iron or steel with a very simple shape. In the Edo age (1600-1867), we assist to a progressive change, with the *tsuba* becoming a status symbol, and the increasing use of copper, or copper alloys, and the corresponding concentration of the steel component in the core. At the same time, an extensive use of gold and silver becomes apparent to decorate the surface of the *tsuba*. The decorations became more valuable and were miniaturized as naturalistic representations, fantastic animals, or else. Being highly artistic objects, *tsubas* cannot be studied, by means of the traditional metallographic techniques, both on the surface (because of the always present patina) and in the bulk (sampling is strictly forbidden). Thus we have proposed an innovative approach, to the quantitative characterization of the bulk of such artistic samples, using Time of Flight Neutron Diffraction (ToF-ND). Here we report the results of a number of experiments carried out on several *tsubas* from different periods that were made available by the Japanese Section of Stibbert Museum (Florence) and private collections. Measurements were executed at the INES diffractometer (Grazzi et al., 2006) which operates at the ISIS pulsed neutron source (UK). The ToF-ND technique permits to quantitatively determine the phases present in the sample in a gauge volume variable from a few mm<sup>3</sup> up to some cm<sup>3</sup> (Grazzi et al., 2009). Neutron diffraction data have been refined through the Rietveld method, using the GSAS code (Larson & Von Dreele, 1994), in order to quantify the phase distribution and the grain size of the metal phases. Semi-quantitative indexes of the strain level and texture intensity were also determined for all the samples. The results reveal the high quality of metals both in the steel and copper alloy components with noticeable changes in composition and working techniques, depending on the place and time of manufacturing.

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**M6 Using PGAA to Determine the Composition of Experimental Iron Smelting Residues: Strengths and Limitations of a Non-Destructive Analytical Technique**

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This poster will show the results obtained by prompt gamma activation analysis (PGAA) of experimental iron smelting residues. The aim of this poster is to display the applicability and limitations of this neutron-based analytical technique for the purpose of determining the composition of ore, clay, slag, iron and charcoal. PGAA is a non-destructive bulk analytical method, based on the detection of characteristic prompt-gamma photos emitted after radiative neutron capture (Révay, 2009). It provides an average composition of the irradiated volume. PGAA proved to be very informative for determining the major element composition of charcoal.

Samples of charcoal were analysed for 12hrs in overnight runs, to acquire the data for C, H and O. This allows a calculation of the volatile material, fixed carbon and ash content of the charcoal. Comparison of this data with analyses of charcoal ash, produced at different temperatures, gives an accurate estimation of the losses of K<sub>2</sub>O and CO<sub>2</sub> from the decomposition of the carbonates. The analysis of a sample of fine magnetic ash, collected in a trap above the furnace, shows that it is a combination of charcoal ash and partly reduced ore.

The analytical work was made possible through the CHARISMA project (Cultural Heritage Advanced Research Infrastructures: Synergy for a Multidisciplinary Approach to Conversation/Restoration).

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## **M7 Forging Ahead: Using Experiment to Augment our Understanding of Ferrous Artefacts**

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Metallography is a powerful tool for investigating metal artefacts of all types. The problem for the archaeometallurgist is that they are faced with making inferences from a material which is initially poorly understood and derived from processes about which they are uncertain. The interpretation of archaeological metal artefacts is further complicated by a lack of control samples as many processes which were used in times past are now no longer practiced. The paper concentrates on the metallographic examination of a set of ferrous artefacts that have a well understood history. Artefacts were forged from bloomery iron produced in an experimental bloomery and smithed using a traditional open hearth charcoal fueled hearth. The study aimed to characterize a number of microstructures which had resulted from a variety of working techniques including bloom consolidation, stock reduction, and fire welding. In addition a number of 'subtle' smithing techniques were investigated to see if they could be realized in metallographically. The paper concludes with a discussion of how experimental archaeology, especially, when a known ore is reduced to bloom and subsequently smithed into artefacts, can be used to help better interpret archaeological artefacts.

## **M8 Technological Study of a Collection of Hellenistic and Roman Jewellery Items of The Musée Royal de Mariemont, Belgium**

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A collection of gold jewellery items dated to the Hellenistic and Roman periods was purchased in 1970 by the Musée royal de Mariemont, Belgium. There is no information about the archaeological context of these objects. This collection has been analysed by PIXE-PIGE and SEM-EDS to better understand their origin. The purpose of this research was threefold. It first consisted in characterising the different materials to investigate refining quality and technological choices. The various techniques used in the manufacture of these objects were identified to understand the 'chaîne opératoire' of each object. Finally, we tried to determine the material history of the items by looking not only at usewears and ancient repairs but also at evidence of modern conservation treatments and reconstructions.

The results of the elementary analysis show that there are two groups of gold alloy. The first one presents rather pure gold with low amounts of silver and a few traces of copper whereas the other group shows a poorly or not refined gold with much higher silver and copper concentrations. This difference in the alloy composition does not seem to be related to a particular technological choice or an intentional colour appearance. These two types of composition might be interpreted as the result of the use of different sources of gold (native ores, recycled metal scraps, etc.) which were widely available after Alexander's conquest.

The microscopic examination has enabled to identify the main manufacturing and decoration techniques of Greek and Roman jewellery. The objects were shaped by hammering, repoussé or casting. Several types of wire have been used for chains and filigree such as twisted and beaded wires. Granulation, gilding, intaglio and enamelling have been used to decorate the surface of some of these items.

Usewears are visible on some of the objects and confirm their functional purpose. It is also believed that some objects have been repaired in Antiquity, as well as recently. Some links of a chain seem to be repairs as they are shaped in a different way. Evidences of conservation treatment have also been identified on two earrings. Whereas the other items were covered by sediments, those objects show a very clean surface with an intergranular corrosion which could be due to an acid treatment. Moreover, the combination of a Roman locket and Hellenistic chain seems to have been done in modern time in order to create a complete necklace.

## **M9 Evolving Complexity? A Comparative Analysis of Smelting Copper Oxide and Sulphide Minerals in a Bowl Hearth**

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There is a common assumption in the study of ancient technology that processes increase in sophistication and complexity through time. Whilst some scholars have questioned how we conceptualize complexity and in turn how such ideas inform understandings of progress (Pfaffenberger, 1992) there remains a tendency within archaeometallurgy to continue with such evolutionary schemes. In some ways these ideas are reinforced in the study of early copper metallurgy because of the geological circumstances associated with the formation of copper deposits, namely that oxide ores are generally believed to be more accessible in early periods than sulphide ores. The relative complex chemistry of sulphide smelting has often been seen to demand an equally complex technology. However, this paper argues that it is misguided to assume that complex chemical processes necessarily correspond with complex technologies.

A campaign of experimental archaeology sought to assess the relative ease with which two ore types, sulphide and oxide, could be converted to copper in a simple bowl hearth. Experiments employed a crucible to contain ore charges in a hearth fueled with charcoal. Simple bag bellows were employed to induce a draught in to the furnace. Results from the experimental campaign illustrate how assumptions that equate chemical complexity with technological sophistication are most likely misguided. The implications for our understanding of the development of early metallurgy are discussed.

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## **M10 Thermoluminescence (TL) Dating of Ancient Metal Manufacturing Furnaces Found in Northeastern Provinces of Thailand**

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Thermoluminescence (TL) dating method is applied to determine the age of ancient metal furnaces found in northeastern provinces of Thailand such as Mukdahan, Buri Ram and Surin. Scanning electron microscope coupled with energy dispersive spectroscopy (SEM-EDS) and particle induced X-ray emission spectroscopy (PIXE) were also carried out to characterize chemical composition of metal slag discovered nearby these furnaces. It was revealed that the furnaces were built on the same age, between Historic to Protohistoric Ages. It was also found that the main composition of slag samples was iron. It can be assumed that these areas were the ancient iron production.

## **M11 Technological Traces as Travelogue: An Analysis of Metal Objects from the Cenote Sagrado, Chichén Itzá**

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This project proposes that the metals, jades, textiles, and other objects recovered from the Cenote Sagrado, a water-filled limestone sinkhole at Chichén Itzá in Yucatán, Mexico, are the product of multiple ritual depositions from the 8<sup>th</sup> century A.D. into the Spanish Colonial period (Coggins & Shane, 1984; Coggins, 1992; Piña Chán, 1970, 1998). Building on prior studies (Contreras et al., 2007; Franco Velázquez & Grinberg, 2002; Lothrop, 1952), this research focuses on the Cenote's metals, including bells, rings, anthropomorphic figurines, and sheet. The lack of metallurgical debris at Chichén, the absence of ore in Yucatán, and similarities in macroscopic style between these objects and those of distant regions suggest foreign origins for these objects. Visitors to the Cenote commemorated their journeys by burning, breaking, and/or crumpling and then depositing materials they brought with them. These materials' 'technological styles' may have allowed Cenote visitors to communicate their origins. This research involves the construction of object "life histories," which describe the processes leading from the extraction of the objects' raw materials through the objects' use and deposition, and then, comparison of life histories to create "technological groups" for objects with similar macro- and microscopic attributes. Groups will be compared with known ancient metallurgical traditions of regions such as West Mexico, Oaxaca, and the Central American Isthmus in order to propose provenances for objects. The metals are held in Mexico and the United States in three museums: the Museo Nacional de Antropología, the Museo Palacio Cantón, and the Peabody Museum of Archaeology and Ethnology. The investigators' initial studies at each museum suggest that the objects are predominantly copper and gold alloys often with surface enrichment in gold. The surfaces display tool impressions as well as porosity, fracture, discoloration from burning, and several types of corrosion patinas. To achieve the project's aims, metals will be analyzed non-destructively and non-invasively. Their surfaces will be documented through stereoscopy with digital photography. Surface alterations, arising from ancient manufacturing processes, physical manipulations of the objects before deposition, and/or modern conservation treatments, will be pinpointed through the application of Ultraviolet and Infrared light. Bulk elemental composition will be determined through portable Energy-Dispersive X-Ray Fluorescence Spectrometry (ED-XRF). Future analysis may incorporate the microscopic documentation of surfaces with Scanning Electron Microscopy (SEM) and the characterization of surface enrichment with Particle-Induced X-Ray Emission and Rutherford Backscattering Spectroscopy (RBS). Preliminary compositional results from analysis of the Peabody Museum objects will be presented.

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## **M12 Elements of Personal Style: A Technological Analysis of Tweezers from Chincha, Peru**

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Excavations of Chincha Valley cemeteries, dating from AD 1000/1100 to the early 15<sup>th</sup> century, yielded ceramic, textile, and metal burial accoutrements, including 22 complete tweezers (Kroeber & Strong, 1924; Lothrop, 1937; Uhle, 1924). These tweezers, in addition to 32 tweezer fragments, are now housed in the Phoebe M. Hearst Museum of Anthropology. Spanish-Colonial period documents related to the Andes, including the treatise of Guaman Poma, lack illustrations of indigenous peoples adorned with these depilatory tools. This project asks: if tweezers are not displayed actively in daily life, but deposited with deceased individuals in burial, how can they communicate personal style? Following the proposal of Kroeber & Strong (1924) that these burials were used over generations, do the tweezers evince multi-generational or familial style? This stylistic preference may be expressed through the tweezers' shapes and elemental compositions. This research, building on limited analysis by Root (1949), aims to reconstruct patterns in fabrication and use practices as a route to investigating the preferences of tweezer producers and consumers. Initial examination of the tweezers yielded a coherent classification that suggested a relatively limited diversity of shapes. While many tweezers display extensive corrosion, cleaned tweezers show copper, gold, and silver surface colors. First, the tweezers were examined with stereoscopy and digital photography. Documentation of textiles and beads attached to the tweezers revealed the ways in which they may have been worn and potentially concealed. Then, Ultraviolet (UV) and Infrared (IR) light were applied to their surfaces in order to reveal manufacturing defects, such as porosity, the extent of surface corrosion, and/or the locations of modern conservation treatments. IR light can reveal marks of cutting tools, which, in many cases, have left sharp, but uneven edges on the tweezers. Portable Energy-Dispersive X-Ray Fluorescence Spectrometry (pXRF) was employed to determine bulk elemental compositions of the tweezers, accounting for the contributions of natural corrosion and conservation treatments to these compositions. Even if metal tweezers were not visible in public settings, their shape and elemental composition, in addition to their color and association with textiles and beads, may have expressed individual tastes of their producers and consumers. Considering the proximity of copper, silver, and gold smelting, indicated by excavations at the Chincha Valley site of Tambo de Mora (Alcalde Gonzales et al., 2002), and the easy access to maritime trade (Hosler, 1994), Chincha consumers might have been able to exert careful control over the tweezers they received.

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### M13 Analysis of Celtic Coins from Northern Italy

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The study of the coinage of the Celtic tribes of northern Italy began only recently being the first work published in 1966. Thanks to the development of the modern archaeology and to the increasing knowledge of the Celtic world, few years ago E. Arslan brought to a new, proofed and widely accepted classification and attribution of these coins (Arslan, 1995). Two of the main questions of the numismatists nowadays concern the silver content in the different emissions minted between IV and I century B.C. and the provenance of the metal used to struck those coins. Few compositional analysis have been carried out so far and only few specimens have been analyzed. Recently, in order to solve these archaeometric problems, a wide number of dracmas coming from the Antiquities Museum of Turin (Italy) have been analyzed by means of XRF technique (X-Ray Fluorescence). The aim of this project was to investigate both the silver content and the presence of minor elements, such as Au and Pb, that are considered as provenance indicators. Since the presence of a surface silver-enriched layer is quite common in objects made of a silver-copper alloy (Beck et al., 2004), data obtained for silver cannot be considered as the bulk composition of the coins until the presence of enriched layers is confirmed or not: further analysis will be carried out with bulk techniques. The most interesting data obtained so far from the analyses concern the minor elements, in particular Au and Pb, which are not subjected neither to enrichment nor to depletion. In fact, a plot of Au versus Pb shows small scale composition differences between coins attributed to different Celtic tribes. The coins are grouped in different clusters: one group collects all the coins minted by the tribe of Libui (settled near Vercelli, in Piedmont), while the other coins, attributed to Insubri and Boico-Cenomani tribes (Lombardy and Emilia) are split, but mixed, into two groups. Thanks to these results, we can reasonably suppose that the raw material used for the coins come from different sources. Moreover, in the case of Insubri and Boico-Cenomani coins, we cannot highlight differences between them: this could mean that the silver mineral in both cases come from a common source. Similar results have been achieved recently on Ptolemaic silver coinage (Kantarelou et al., 2011).

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## **M14 The Evolution of Iranian Metallurgy: Deriving Archaeological Information from a Large and Varied Data-Set of Compositional Analyses**

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In the past century a number of research teams have analysed altogether over 2000 Iranian copper based objects for their chemical composition. This wealth of information was originally scattered in about 30 different publications and has never before been studied as a whole. In order to better understand changes in metalworking in Iran and in particular the transition into the Iron Age all the results were regrouped in one database. A chronological and a geographical framework were then defined to bind the data into one workable set.

Tin was the first element to be studied in details. There are only very limited tin resources in Iran and today most scholars agree to say that Afghanistan is a likely provenance for this resource. For that reason, objects from regions located on potential tin trade routes (overland or through the Persian Gulf) were added to the database: over 2500 compositional analyses from Bactria, Afghanistan, the Indus Valley, the Oman Peninsula and Mesopotamia were assembled and added to the Iranian set. Preliminary results show a clear East/West divide in Iran. In the West, tin was routinely used as an alloying element from the mid 3rd millennium, including in the first centuries of the Iron Age. In the East however copper is mostly devoid of tin throughout the Bronze Age.

The other elements to be studied in details were arsenic, antimony, silver and nickel in the hope that they could give us some information on the provenance of copper and patterns of re-melting and recycling. The objects were divided in groups of similar composition and each group was then studied separately. This method, along with the analysis of tin levels, shows evidence of trade of copper, bronze and tin along the Persian Gulf and south-western Iran, however the direction of each one of these trades is still to be determined. The analysis has shown the region of Luristan in western Iran, already well known for its decorated bronze-work, to be a region of primary production of bronze, possibly using a local source for both copper and tin (Nezafati, 2006) that could have been in competition in western Iran with the Afghani sources.

NEZAFATI, N., 2006. *Au-Sn-W-Cu-Mineralization in the Astaneh-Sarband Area, West Central Iran*. Ph.D. dissertation. Earth Sciences Faculty, Eberhard-Karls University Tuebingen.

## **M15 Analysis of Metallic Objects of the Moche Priest Warrior from the 14<sup>th</sup> Tomb of Huaca Rajada-Sipan, Lambayeque, Peru**

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The Moche culture is one of the most fascinating ones of peruvian territory. Developed in the northern coast of Peru, it is characterized by its great development of ceramics and metallurgy, which can be seen in the artifacts found in the distinct tombs of political and religious authorities that we can find in the Sipan sector of the Huaca Rajada-Sipán archeological complex. The present work is part of a research project sponsored by the Peruvian and Italian governments, in which the evolution of metallurgy in pre-colombine cultures in the north of Peru is studied, with a special interest in golden and gold-coated objects. The study of metallic artifacts from the 14<sup>th</sup> tomb of the Huaca Rajada-Sipán archeological complex at is presented, a burial corresponding one of the four political-religious ledaer of the Mochica culture, known as the Priest Warrior, as well as the study of a fragment from a hanging object belonging to his metallic clothing corresponding to the middle Moche period, aproximately between 300 – 600 d.C. The artifacts have been studied through the non destructive X-Ray Fluorescence (XRF) technique obtaining crucial information of alloys known as “golden copper” or “arsenical copper”, in which copper is found in a higher amount. We also observed and analyzed the internal section of the fragment corresponding the metallic clothing, which was done at a Metallography Examination Facility (MEF), appreciating the different phases of corrosion of the piece, determined by its coloring, redish and brown for copper oxides and greenish tones for chlorides, which complements the analysis of the Scanning Electron Microscope (SEM) and the Energy-Dispersive X-ray Spectroscopy (EDS). Their results show the morphology and elemental composition from inside the fragment, showing that it is a Copper, Gold, Silver alloy, and determining the width of 0.5  $\mu\text{m}$  of gold coating in the fragment. At the patina of the metallic clothing's fragment impurities like Br, Si, Fe, Al were found, corresponding the mineralogical phases found through X-Ray Diffraction (XRD) of the tomb's soil, like quartz and albite in higher amounts, this due to the long period of burial of the piece. Also at the X-Ray Diffractogram of the fragments patina, higher proportions of copper oxide like Cuprite and Atacamite hydroxychloride were found.



## M16 New Insights on Medieval Iron Metallurgy in Lorraine

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Precedent studies (Leroy, 1997) have high-lighted the scale of iron-production activities in Lorraine during the Middle Ages, and especially for the early medieval period. According to archaeological data, those activities seem to have benefitted of very important iron ore deposits, and specifically a particular one, the *Minette Lorraine*. For this context, the iron production organization was well studied last years through archaeometallurgical works led on some production sites. Nevertheless, the becoming of the metal produced is not yet documented. The question of its spreading modalities remains open. The scale of iron distribution circuits remains unknown for this area. Moreover, although some textual data evoke some metal quantities produced in several sectors for the late medieval period, the relative importance of the different production zones in Lorraine during ancient times is not defined for now. The work presented aims to treat these questions by complementary approaches. Following the way given by the studies of Leroy *et al.* (in press), a geochemical approach which consists in establishing the chemical signature of the production area by performing major and trace analyses using ICP-MS on ore and smelting slag samples is applied. Then, to establish a link between resources and products, representative iron production sites have to be studied by archaeometallurgical studies. To do so, a specific methodology, mainly based on metallographic examinations and chemical analyses led on artifacts samples stemming from consumption sites, is developed. Different metallurgical wastes categories from production sites are studied. Experimental protocols were established to refine archeological interpretations. Confronted to this reference data set, artifacts from consumption sites, such as the Metz Cathedral and a Carolingian bridge, are studied following a process including macroscopic and microscopic analyses, the latter coupled with metallographic observation, followed by chemical analyses for major (SEM-EDS) and trace (LA-ICP-MS) elements. First results show that the main types of iron ore, and therefore the main iron production zones in Lorraine, can be globally discriminated from each other on a chemical point of view, particularly using statistical multivariate approaches. Moreover, the study of a reference site presenting remains related to both smelting and smithing stages in a *Minette*-smelting context allowed characterizing the evolution of the original chemical signature during all different operating steps. These observations let us expect, despite an increase of the variability of the signature along the operating chain, that a reliable chemically-based discriminating tool could be developed to evaluate the iron products' spreading in the area considered.

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## **M17 Chemical-Mineralogical Studies on Iron Crucibles from Chahak, Iran**

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Crucible steel was the material used to produce famous swords' as well as sabers' blades, so-called "Watered or Damascus steel". The method of production causes some features such as flexibility, sharpness and sturdiness upon striking. Crucible processes used to make metal swords in various locations in central Asia, India and Iran. One of the recently excavated area is Chahak, in southwest Iran. According to a historical manuscript, Chahak was an important crucible steel production site in Iran since 11<sup>th</sup> century. Inspection of site displays hundreds of cylindrical crucibles, so that all of them are fragmented. Moreover some of them have massive slag cakes remained inside. Accordingly, surveying on the site shows some slag heaps or scatters with different densities, crucibles' lids and several fragmented pottery.

The chemical- mineralogical investigation carried out with respect to the constituents phase composition of objects. XRD, XRD and optical microscopy was the base scientific methods that lead us to understand about the extracted material (namely Iron) in this site. According to analytical methods distribution of major, minor and trace elements determined and the crystalline constituents were investigated also by refining the phases. These investigations can be the base analysis on Chahak and will focus also about the provenance study of investigated materials in the future.

As a matter of fact, to understand the essence of structure and the technology of crucible steel as a valuable heritage, this paper will focus on the crucibles' structure as well as their microstructure, in order to discover the technique of making crucible steel and extracted metal as well.

## **M18 Late Bronze and Early Iron Age Smelting Debris from the Black Sea Coast of the Republic of Georgia**

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The Late Bronze Age to Early Iron Age transition in the greater Near East was marked by a series of dramatic social and technological changes. Over the course of several hundred years, from about 1200 to 800 BC, iron changed from a high status material to a ubiquitous material used for utilitarian tools and weapons. Over the same period, the social and political configuration of this region changed dramatically, with the collapse of the Hittite Empire at the beginning of this period and rise of the Urartian and Neo-Assyrian Empires toward the end. Though iron's role as a prime causative factor in these transformations has been heavily criticized, many questions remain about the relationship between the rise of iron production and the social-economic changes during this period. Though many scholars see the discovery of iron as emerging from experience smelting and working copper, many theories contrast the organization of iron and bronze production.

The lack of detailed study of primary production contexts, especially for iron, is problematic for understanding this transition. While new research has begun to change this (Veldhuijzen & Rehren, 2007), the relative organization of copper-alloy and iron production remains unclear in many regions. A large number of metallurgical furnaces in Western Georgia, with dates ranging from about 1800-600 BC (Khakhutaishvili, 2009), offers a prime opportunity to understand the relationship between these two industries. Previously published results (Nielsing, 2009; Tavadze et al., 1982) suggest that both iron and copper smelting may have occurred in this region, though the exact timing of these activities is unclear. This talk presents the preliminary results of optical and scanning electron microscopy on a set of slags collected by a joint Oxford-Georgian Expedition. Particular attention is given to determining the kind of metal produced, and the relative atmospheric conditions within the furnace.

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## **M19 Metals and Metallurgy at Shanga, Kenya, ca. 750-1200 CE: Technology Transfer within an Islamic World System**

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The emergence of Swahili ethnicity on the east coast of Africa between ca. 700 CE and ca. 1500 CE was linked to the development of trade routes connecting India, Arabia/Middle East and the African interior. Swahili towns – the first to appear on the coast south of Somalia – served initially as places where African ivory was exchanged for Indian and Middle Eastern manufactures, including cloth, glass and metalwares. After 1100 CE Swahili towns also served as entrepôts for the transfer of southern African gold to the Islamic world, and many grew rich in this role. This paper reports on a study of metals and slags excavated by Mark Horton from the Swahili port of Shanga in the Lamu archipelago. These samples range in age from ca. 750 to 1200 CE.

The metallurgical materials from these sites were subjected to a range of analytical techniques, including optical petrography and metallography, SEM, EPMA, and MC-ICP-MS. The compositions of the metals examined are compared to those reported from contemporary sites in southern Africa, the Middle East and India, and the evidence for technology transfer is discussed. Lead isotopic ratios were measured by MC-ICP-MS for non-ferrous metals and the results compared with existing datasets to examine provenance of imported metals. Connections to Arabia, the Middle East, India and even China are presented and implications of these connections within the greater Indian Ocean trade and interaction sphere, and the Islamic World System are discussed.

## **M20 Elemental and Lead Isotopic Compositions of Metals from Fraga dos Corvos (N Portugal): First Results of an Integrated Study**

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Fraga dos Corvos is one of the only Bronze Age settlements with metallurgical operation evidences in the Northeast of the Portuguese territory which is being subject to archaeometallurgical studies. The site is composed by (1) a habitat area from the 1<sup>st</sup> Bronze Age that has been under archaeological excavations since 2003 where various metallurgical remains and artefacts have been recovered (Senna-Martinez et al., 2010), (2) a shelter that has been excavated between 2003 and 2006 where some bronze artefacts with orientalising features were recovered (Figueiredo et al., 2009), and (3) a Late Bronze Age occupation area detected in 2011, from which some artefacts, including an Acebuchal fibulae type, were recovered. Based on the material cultural evidences it is suggested that the site had different occupation periods, from the second quarter of the 2<sup>nd</sup> millennium BC until Late Bronze Age. Among the metallic artefacts are both objects with an “Atlantic” typology (such as torques) and objects with a “Mediterranean” affiliation (such as fibulae). Other materials, related to metallurgical operations and attributed to the 1<sup>st</sup> Bronze Age are crucible and open vessels fragments, metallic droplets, a mould fragment and some metallic bars. For the present work micro-EDXRF analyses were performed to study the elemental composition of the metallic artefacts and macro-EDXRF analyses were performed to identify metallurgical vestiges, on the crucible, open vessels and mould. Additionally, ICP-MS analyses were performed in some selected metallic artefacts to determine the Pb isotope ratios. The combination of the results will reflect the homogeneity or heterogeneity of the assemblage, giving further information about the metallurgy in different periods of the site. The overall results will provide relevant information on metallurgical processes undergoing Bronze Ages, particularly in what concerns bronze metallurgy. Elemental analyses by micro-EDXRF show that most of the artefacts are made of bronze (exception for a copper bar) with Pb contents <4wt.%. The macro-EDXRF analysis show that the crucible and open vessels have been used for bronze metallurgy, namely for bronzes with some Pb content, and thus in agreement with the metal artefacts found at the site. The ICP-MS analyses provide the first results of Pb isotopic composition of bronzes from the Portuguese territory, and the discussion of the results will include comparisons with isotopic data of bronzes from the nearby Spanish territory. This research work has been financed by the Portuguese Science Foundation (FCT-MCTES) through the EarlyMetal project (PTDC/HIS-ARQ/110442/2008) and the grant SFRH/BPD/73245/2010 (EF). The financial support of CENIMAT/I3N through the Strategic Project-LA25-2011-2012 (PEst-C/CTM/LA0025/2011) is also acknowledged.

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## M21 Variation in Roman Period Iron Smelting: A Case Study in SouthWest England

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Iron smelting in Roman period Britain typically occurs in clusters of relatively small sites across the iron-rich areas of the Kent and Weald, Forest of Dean and Mendips, east Yorkshire, Midlands, and northwest Norfolk (Paynter, 2007). Here bloomery iron was produced in furnaces with highly variable morphology, measuring from 30cm to over a 1m in diameter. Compared to concentrated large-scale production in other parts of the Empire (cf. Cech, 2008) Romano-British production in most cases appears to have served local needs, with some suggestion of occasional military involvement (Cleere & Crossley, 1995). However there has been limited technological characterization of smelting processes or craft organization in the UK beyond the identification of slag-tapping and local ore use. Recent excavations in the Exmoor region of southwestern England have revealed a new region of Roman iron smelting activity, with numerous dispersed production sites operating between the late first through to fourth centuries AD (Bray, 2006). Production debris, including tap slag, ceramics and ore excavated from a stratified slag heap at one of these sites have enabled a more detailed characterization of the production processes utilizing optical microscopic, microanalytical and bulk compositional techniques. Besides confirming the production of bloomery iron using high grade ore and local raw materials, in line with practice seen in other parts of England, the analyses concentrated on identifying and explaining variation in the major element compositions of tap slags. Tap slag compositions across the period of occupation at the site showed no discernable pattern even when interrogated using multivariate statistical techniques. Whilst debris from some contexts shows some standardization, others show significant variation, and across the 400-year period of production there is no clear evidence that production moved towards the most efficient process, despite suggestions that this should occur even unintentionally (Charlton, 2006). Whilst all producers active during the occupation period of the site shared a similar broad methodology, similar production was only replicated within individual periods of slag deposition. Breaks in deposition may therefore argue for periodic exploitation, or relatively regular changes in producers present on the site. This has implications for our understanding of socio-economic influences on the craft organization and presents new definition in the wider context of Roman iron production.

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**M22 Is it by design? An Investigation into whether the Black Colored Copper Sulfide Surface on a Roman Leaded Bronze Statue is Corrosion or a Manmade Patina**

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In this study we characterize the chemistry and morphology of patches of a black surface crust found on an ancient Roman (1<sup>st</sup> century AD) bronze Eros statue in the collection of the J. Paul Getty Museum (JPGM 93.AB.53). The primary aim was to determine whether the layers were from an intentionally produced patina or the result of natural corrosion processes. Although ancient sources describe sulfur patinas, no clear correlation has been established with extant examples of black patina. Black patinas have also been associated with sulfur-reducing bacteria in archaeological burial environments and more recently, with historic bronzes exposed to outdoor sulfur-containing pollution. This study describes a sulfide-rich surface that is dark olive to black in color with a compact and remarkably smooth and reflective surface. In cross-section the surface appears to have a well-defined structure composed of two layers of chalcocite (Cu<sub>2</sub>S). The linear arrangement of the chalcocite layers and its reflective appearance suggest a deliberately applied patina as opposed to formation through natural corrosion processes. However compositionally distinct strata, composed of lead, chlorine and tin-rich phases, were also observed beneath the chalcocite layers. The chemistry of these subsurface layers suggests that further natural corrosion occurred in oxidizing conditions and in a Cl-rich environment. Studies were conducted to understand the chemical relationships in this complex layering system (reduced sulfur complexes in the upper layers and oxide layers in the lower layers) as well as, on a fundamental level, how aqueous and gaseous sulfur reacts with leaded bronze surfaces. Based on comparisons to experimental replicates, we discuss the correlation between the observed microstructural features and the elemental composition of each of these layers, as well as the association between the chalcocite and corrosion structures. These studies will provide a better understanding of sulfur-containing surface layers on bronze, and in the end, of the sculpture's original appearance.



## M23 Non Destructive Characterization of two Japanese Swords through Time of Flight Neutron Diffraction

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Historical Japanese swords are interesting objects for researchers, as they represent some of the best examples of historical metalwork and are among the most rewarding artefacts for studying the evolution of metallurgy. However, a non-destructive approach is mandatory, as sampling of such rare blades is never permitted. Here we report the result of an investigation on two Japanese long swords in the Pitt Rivers Museum collections, University of Oxford. These are a Chu Ao School *tachi* (chivalry sword) signed blade of 13<sup>th</sup> century and a Bungo School *katana* (infantry sword) signed blade of 17<sup>th</sup> century. The second sword is attributed to (the Mino tradition). Both blades, according to the makers' signature and the exterior characteristics, are very high-grade artifacts. The Ao *tachi* blade appears in good conditions, not too different from the original, in spite of its ancient age and an evident slight shortening. Therefore, its study provided meaningful data for the characterization of ancient blades. The Bungo *katana*, instead, appears intact in its original shape, so that its characterization assumes also a paramount importance. Neutron diffraction has been applied on the two samples using the INES diffractometer at the ISIS pulsed neutron source (UK) (Grazzi et al., 2006). Measurements have been performed on the average gauge volumes in the tang, and in selected parts of the blade, in order to determine the quantitative distribution of the phases. The comparative analysis of the phase distribution, in the two samples, has permitted the identification of characteristics peculiar to the forging methods (e.g. the carbon content, the fayalite amount, the presence and distribution of wuestite and troilite). In particular, we could quantitatively determine the carbon content of the various measured volumes from the cementite to ferrite ratio (Grazzi et al., 2011). A diffraction scan was also performed (dividing the blade into three different sections: the edge, the core and the ridge) to determine the inner phase distribution and confirming the highly differential specialization of the parts of this kind of sword. From the shape analysis of the ferrite peak, we have been able to determine, semiquantitatively, important microstructural properties, like the texture level, the strain level, and the domain size of the grains. This information is important to increase our knowledge about the several forging methods used by the different schools and traditions in Japan.

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## **M24 Women's Sacred Silver Jewels from Patagonia (19<sup>th</sup> Century): Mapuche-Tehuelche Alloys and Technologies**

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Indigenous silver jewellery blossomed in Southern-central Chile (Araucania region) and in Argentinean Patagonia during the 19<sup>th</sup> century. Reserved to women, this jewellery provided fecundity, protection, and a support for codified messages referring to their owner's social status. As a consequence of groups' migration, reduction and adaptation to the new political and economic situation in the past century, the meaning of some of these codes as well as the technologies, the type of alloys produced and the origin of the metals faded in people's memory.

The aim of this work is to shed new light on the Mapuche-Tehuelche jewellery through an approach based on the scientific study – by PIXE, XRF, optical microscopy, SEM and X-radiography – of a group of composite jewels (such as earrings which are said to suffer an evolution during women's life corresponding to precise events) of known context of collect. The studied items belong to the *Museum für Völkerkunde's* Schythe collection (finalised in 1867) in Berlin and the *musée du quai Branly's* La Vault collection (1896-1898) in Paris.

The study clearly pointed out the use of the same workshop practices and traditions: the objects are produced by cutting plates obtained by hammering; several plates are joined by using suspension rings applied by perforation of the plates; very few elements present signs of soldering; the decoration motifs are obtained by repeated punching of chisels. Nevertheless we could observe the wide variety of workshops having participated to the production of jewels present in a same camp, which confirms the exchanges that took place between communities and questions the status of the indigenous silversmith.

The objects are manufactured with Ag-Cu alloys with variable Cu contents and sometimes containing Zn and Ni, whose highest concentrations are reached for alloys close to commercially available nickel silver (maybe use/reuse of coins). Composite objects present diverse compositions according to the constituent parts, which could in the case of the earrings indicate an addition of parts along the objects' life. The concentrations of trace elements present in the silver alloys show the exploitation of very different sources of metals and a large use of re-melting.

## M25 An Approach to Inca Metallurgy: Votive Gold and Silver Figurines from the Empire (1400-1532)

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Miniature figurines produced in gold, silver and copper alloys representing human beings of both sexes and camelids account for one of the most representative metal production in Inca times. Although most specimens hosted in museum collections have lost their context of provenience, some items have been found in archaeological contexts, in high-altitude sites of the Andean *cordillera*, where children were sacrificed following the *capacocha* Inca rite. In the latter, some figurines were found wearing miniature textiles and feather head-dresses, as well as miniaturised silver pins (*tupus*), bracelets (*chipana*) and gold diadems (*canipus*).

Few science-based studies were carried out on these gold and silver figurines produced either with joined hammered metallic foils or by casting. While their iconography is very codified, the differences in their fabrication technique, as well as the human body's proportion and the surface treatment of the figurines, may reflect the existence of diverse workshops traditions and question the data contained in the chronicles, which inform on royal workshops settled in Cuzco, held by requisitioned Chimú goldsmiths from the Peruvian Northern coast. The corpus of 9 figurines from the *musée du quai Branly* collection studied in this project reflects this diversity, which may suggest a wider geographical or temporal variability.

The aim of this work is to present the analytical study with non-destructive methods – elemental analysis, X-radiography, SEM and optical microscopy – of a representative corpus of Inca figurines, to determine whether their fabrication technologies and alloys correspond or not to equivalent workshop practices. In order to approach these questions in a more general way, our results are compared with the few published data on the figurines from Cerro El Plomo (Chile) (Mostny, 1957), the Cerro Aconcagua (Argentina) (Bárcena, 2004); the Dumbarton Oaks' collection (WDC) (Boone, 1996); and the Museo de América (Madrid) (Rovira, 1994).

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## **M26 Ball Site Copper-Based Metals: Where were they Discarded or Lost?**

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The Ball site is located in the Huronia region of southern Ontario, about 13 km south of Georgian Bay and 96 km north of what is now Toronto. It was occupied in the first half of the 17<sup>th</sup> century, and is the only completely excavated village site in Ontario. It was a palisaded village that started in the northwest of the full site, and more than doubled its area during its lifetime by expanding simultaneously to both the south and east.

Since copper and brass samples were recovered in both the “old” and “new” areas of the site, it was hoped that different metals, or different metal chemistries, might be concentrated in different parts of the village, giving us an insight into a micro-chronology of the village.

The Ball site metals that were analysed by INAA included 1 native copper sample, 205 European copper samples, 91 red brass (high-Sn, lower Zn) samples, and 129 yellow brass (high Zn) samples. Each of the European metal types were subdividable into at least 6 different multi-sample chemical groupings, using either bivariate plots or PCA, together with up to half a dozen more single or double sample chemistries.

The European copper samples were recovered 68% from the old part of the village, with most of the chemical groupings behaving this way. The red and yellow brass samples were recovered at a rate of 59% and 61% from the old part of the site, with chemical groups biased more towards the newer part of the site. The percentage of metals recovered from inside houses was 52% for European copper; 50% for red brass; and 39% for yellow brass.

All of this implies that the new part of the village was not occupied by new arrivals who brought their own, chemically different, copper-based objects with them and did not share them with the original members of the village, but points toward a population-increase, forced expansion of the village.

The difference in the distributions of coppers and brasses in the village, may confirm that copper kettles were traded to these Hurons before brass kettles.

## **M27 Bullets over Gennep: Using Compositional Variation in Lead Musket Balls in Battlefield Archaeology**

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The archaeology of battlefield is hampered by the ephemeral state of the locations of many armed engagements. Many historical battlefield sites only contain scatters of metal objects that are difficult to interpret. One of the most common finds are lead bullets. The spatial distribution of such bullets may elucidate the progression of a battle. Unfortunately, it is rarely possible to identify which bullets were fired by which of the warring parties.

In this study we wanted to investigate whether compositional analysis of lead bullets from a 17<sup>th</sup> century battlefield site (Gennep castle) could be related to the warring parties. Gennep castle – and its earthen fortifications - was besieged in 1641, the final phase of the 80-year (Spanish-Dutch) war. After three months of siege warfare, the Spanish-paid mercenaries surrendered the castles to the Dutch forces. The fighting has been recorded in great detail, and several plans of the battlefield and siegeworks are available.

In a plan to redevelop the castles ruins and its surroundings, the moats of several of the defensive works around the castles were dug out. This gave the opportunity to systematically gather and record lead bullets. In addition, bullets were collected from a besiegers' artillery battery site. In total, ca. 1500 lead bullets were collected. Three main calibers were identified. Bullets of c. 12 mm probably originated from pistols. 16 mm and 19 mm bullets were probably derived from different types of muskets.

Their chemical composition was determined by portable XRF. Within the group of 12 mm bullets, a subgroup shows elevated tin contents. Within the 19 mm group, a subgroup shows elevated antimony contents. All calibers were found on all locations. The bullets with high tin contents also showed up everywhere. However, the 19-mm bullets with elevated antimony contents only occurred in the vicinity of the castle, and not in the besieger's artillery battery. Also, their distribution on the earth ramparts – where both parties had been fighting - shows irregular distributions that may be related to the progression of the siege. It is therefore most likely that the elevated-antimony bullets formed part of the ammunition store of the besiegers, and that they can be used as fingerprint for some of their ammunition.

## **M28 The Production of Chainmail**

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Examination of Iron Age chainmail from a number of sites in Denmark has revealed that welding was not used for the non-riveted rings. The structure and distribution of slag inclusions shows that they must have been punched from a sheet of iron, probably by the use of two different sizes of hollow punches. A number of the rings still show the burrs produced by the punching. Some of the rings might afterwards have been smoothed by polishing to remove the burrs that might otherwise harm the bearer or the underlying clothing.

The finest of the mail-shirts were made from a 0.9 mm hammered wire riveted with rivets made from 0.7 mm cut wire.

In the medieval period, the chain mail was made exclusively from riveted rings, probably due to the growing production of drawn wire making the process of punching obsolete.

## **M29 Crucibles and Ladles. The Casting of Copper Alloys in the 15<sup>th</sup> – 16<sup>th</sup> Century**

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Finds from Danish excavations has recently shown two different technologies used for casting of copper alloys in the 15<sup>th</sup> century. From Elsinore, Copenhagen, Ribe and other places, fragments of imported, probably hessian, crucibles are known. Often used for the casting of tin-bronze or leaded tin-bronze. Opposed to these highly refractory crucibles stands a number of apparently very large crucibles made from ordinary clay. Impressions in the clay shows that it was applied as a lining within a kind of basket made from iron, and the vitrification of the surface reveals that they were heated from above. The remains are interpreted as a kind of ladle described by Biringuccio in *De la pirotechnia*, published in 1540. The ladle served both as a container for the molten metal and as a sort of furnace.

All of the examined fragments of Biringuccio type ladles have been shown to having been used for the casting of a bronze with a fairly low tin content and a high content of antimony. Some of the bronzes were heavily leaded.

### **M30 A Characterisation of Roman Age Refractory Ceramics from Autun/France**

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Brass-making crucibles, double-layered metal-melting crucibles as well as fragments of moulds found in an archaeological excavation site of the Lycée militaire (Autun/France), which dates to the Gallo-Roman period, were analysed with a series of classical mineralogical techniques in order to obtain knowledge about the raw materials of the individual ceramics. This study focuses on the usage of the crucibles and moulds as well as technical aspects of their production. The different fragments were studied by using petrographical (optical microscopy), elemental (SEM-EDS, EMPA, XRF-WDS) and mineralogical (XRD) techniques.

In general, the metal-melting crucibles show two main layers, which are made of high refractory, kaolinite-based ceramic with crushed granite added as temper. The analytical and petrographical results show remarkable differences between these two layers. The inner layer is characterised by a non-vitreous matrix with a high content of orthorhombic mullite,  $\beta$ -cristobalite and  $\alpha$ -quartz. In contrast, the outer one is dominated by a high content of mullite-bearing glass and contains analcime which was formed during the burial stage. An innermost protecting layer is detectable in the majority of these fragments. Electron microprobe data indicate that the crucibles were used with copper-based alloys (mainly brass). The mineral composition allows an estimation of the firing temperatures, which have reached approximately 1300 to 1600 °C (König & Serneels, submitted).

The brass-making crucibles are mainly single layered. Only the topside shows a second ceramic layer, which were added to close the space between the lid and the ceramic body to avoid a loss of the volatile phase. The purple colour of these crucibles is indicative for a high amount of zinc within the ceramic related to gaseous zinc during usage. The composition of these crucibles is similar to the inner layer of the double-layered crucibles. However, the amount of silicon is 10 to 15 % lower and the amount of zinc is 100 times higher than in the other studied refractory ceramics.

The moulds of Autun/France are lost-wax moulds and are mainly used for spring-claps fibulae from which five different types are known (Chardron-Picault & Pernot, 1999). The majority of these fragments consist of a homogeneous ceramic body containing quartz, two types of feldspar, illite and meta-kaolinite. The preliminary XRF analyses show a similar composition like the other refractory ceramics from Autun/France. Further results of the brass-making crucibles and the moulds will be given.

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### **M31 ICP-AES/ICP-MS Analysis of Bronze Objects Found in Ancient Necropolis from Apollonia Pontika, South-Eastern Bulgaria**

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Information which is obtained in the archaeometric study of archaeological artifacts is an important part of understanding and knowledge of the ancient world. It is therefore essential that such studies be conducted. This report gives preliminary results of a study of bronze artifacts (clippers, buttons, mirrors, tools, jewelry, arrowheads, etc.) found in several ancient necropolis (6<sup>th</sup>-4<sup>th</sup> centuries BC) near the town of Sozopol on the Black Sea coast (South-Eastern Bulgaria).

Sozopol (Apollonia Pontika then) was founded as a polis in the Greek colonization of the west Black Sea coast along with some other poleis and quickly acquired great importance due to its strategic location. To the Greek colonists joined many residents of the local Thracian tribes and this contributed significantly not only economic but also cultural development. Trade with luxurious bronze objects flourished and this led to the need of raw materials for their preparation. It is thus important to compare the chemical and isotopic composition of the bronze pieces found with the composition of the ore extracted from those in the vicinity deposits. The study of the chemical composition of the findings may also provide important information about the technology of ore processing and production of artifacts.

In the initial stage of this study were analyzed fifty-five samples of bronze artifacts and the concentrations of fifteen elements were determined (Al, As, Bi, Co, Cr, Cu, Fe, Mn, Ni, P, Pb, Sb, Se, Sn, and Zn). The analytical techniques used for analysis were ICP-AES (inductively-coupled plasma atomic-emission spectroscopy, Perkin-Elmer Optima 7000 DV) and ICP-MS (inductively-coupled plasma mass-spectrometry, Perkin-Elmer ELAN DRC-e). The obtained analytical data were processed using statistical program SPSS 16.0 for Windows and were subjected to cluster analysis. Results provide some useful information on craft production and processing of metals in the ancient town of Apollonia Pontika. However, further investigations are still required such as chemical and lead isotopic analyses of the ore which is supposed to have been used for the production of the bronze alloy.

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## **M32 Copper Processing in Oasis Sites of Northwest Arabia. The Evidence from Tayma and Qurayyah**

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The oasis sites of the northwestern Arabia peninsula are important nodal points in the overland trade routes between south and north Arabia as well as markets for the exchange of goods (MacDonald, 1997). Despite this importance, copper metallurgy in this area has remained a rarely studied topic. Until now, scholars have known relatively little about the alloy types and copper processing technology used by Copper and Iron Age people from this region.

The excavation of two Iron Age oasis sites in northwest Arabia, Tayma and Qurayyah, has revealed the first evidence for copper metallurgy in this area. Crucible fragments, slag pieces and metal dross have been analyzed in order to reconstruct the metallurgical activities of these two sites. This research has enhanced our knowledge of the nature of ancient metallurgical technology of northwest Asia and provides a foundation for further archaeometallurgical and archaeological research on this topic. The analytical results show that metallurgists in Qurayyah and Tayma were using different metal processing technologies and producing different types of copper alloys from each other. In Qurayyah, arsenic was widely detected in the slag and metal prills suggesting that arsenical copper was the dominant alloy used at this site. In addition, refining and smelting activities were identified in the Qurayyah samples and there is evidence that a silica rich material was used as a fluxing agent in the refining process to facilitate the separation of slag and metal. However, in Tayma, tin bronze and leaded tin bronze played an important role and an alloying process was detected in most slag samples. A fluxing agent may also have been used to reduce the iron content in copper during alloying. The presence of tin in Tayma may indicate that it had higher economic and political status than Qurayyah, because tin was scarce in ancient Arabia (Weeks, 2003; Muhly, 1985) and it is suggested that Babylonian occupation of Tayma, during the middle of the first millennium BC may be one of the causes for this difference in status.

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### **M33 Bronze Age Plain Axes From the Portuguese Territory: An Archaeometallurgical Study of Bujões and Barcelos Types**

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During First Bronze Age (1<sup>st</sup> BA) in western Iberian Peninsula most metal artefacts were still produced in copper or arsenical copper. The first bronze productions did probably occur during 1800-1600 BC. The present study concerns eleven plain axes found in several archaeological sites from central and northern Portugal, attributed to 1<sup>st</sup> BA. These plain axes are of Bujões and Barcelos types, typologies that are frequently associated to the early stages of bronze metallurgical technology in the Portuguese territory (Senna-Martinez, 2007).

Conventional and micro-energy dispersive X-ray fluorescence (EDXRF) were used for elemental analyses. Microstructural characterization of the axes was made under optical and scanning electron microscope (SEM-EDS) in order to determine the metallurgical manufacturing techniques and identify different metallic phases and inclusions. Microstructural observations by optical microscopy were usually made in two areas, over the butt and the blade of the axes.

Results show that artefacts are made of a copper–tin alloy, frequently with lead and arsenic impurities. The composition of all axes is rather homogeneous, with Sn contents around 10wt.%, except for one axe that has a lower amount of tin. These results point to a bronze composition independent of the typology or origin of the artefacts. Additionally, they suggest some production control in the manufacturing of these first bronzes.

Metallographic results show that the plain axes were manufactured using mould cast technology followed by some thermo-mechanical processing. Some of the axes present microstructural differences between the butt and the blade, which in some cases might indicate a final sharpening operation of the blade or be the consequence of their use as cutting tools.

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### **M34 Shaft and Bowls! A Comparative Analysis of Experimental Iron Smelting**

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Experimental approaches have always played a significant role in archaeometallurgy, with a key focus for experimenters being prehistoric iron smelting. Most experiments have focused on reconstructing shaft furnaces with little recent attention paid to smelting iron in bowl furnaces. In light of recent work highlighting the apparent disregard for bowl furnaces (Dungworth, in press; Dungworth, forthcoming; Girdals, forthcoming) and the potential for iron being produced in bowl furnaces, a program experimental iron smelting in bowl furnaces was undertaken.

The aim of the experimental campaign was twofold. Primarily it was to explore the feasibility of iron reduction in a bowl furnace using high grade ore. Secondly, it was to contrast the use of space and the group dynamics experienced with the use of a bowl furnace with that of a shaft furnace.

This paper presents the results of this experimental campaign and argues that whilst the production of iron in a bowl furnace appears feasible it is much less efficient than that encountered with a shaft furnace for a range of reasons. However, despite differences in efficiency and scale of production, the shift from bowl to shaft furnace may not have had dramatic implications on how labour was organized in the Iron Age.

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### M35 High Carbon Steel and Cast Iron in Africa: Reality or Myth?

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The technology of iron production is generally considered to be a two stage (smelting and smithing) process, but in central and eastern Africa there is another and separate bloom refining (not primary smithing) stage that was situated in between smelting and smithing processes (Greig, 1937; Davison & Mosley, 1988). The presentation aims at verifying the presence and technological function of this second stage of iron production microscopically, because macroscopically, Mapunda (2010) points out, that bloom refining sites can be differentiated from ore smelting sites by examining the composition of the sites, nature of the tuyères, texture and size of the slags, and size of the furnaces.

Microscopically iron refining slags systematically contain iron metal grains as droplets, but the smelting slags consistently constitute irregular iron particles. The droplet shape indicates that iron metal produced possibly got a liquid stage, which further suggests that this was a more reducing or efficient process. The proposition of liquid iron production has been tested by analysing the iron droplets in the slags using the electron microprobe (EPMA) method, which indicates that the production of high carbon steels or/and cast iron was achieved. The fact that the refining slags are different from the smelting slags can be used to verify the presence of the bloom refining process, and presence of droplets in the former and absence of these in the latter process can be used to suggest that the bloom refining process aimed at producing a heterogeneous product of high carbon steel with cast iron, instead of a bloomery iron that more likely was produced during ore smelting stage.

The presence of this new stage both in the field and in the laboratory, therefore, can be used to argue that some traditional ironworking societies in Tanzania (and central and eastern Africa) practised a three stage iron production process, and that bloom refining process was meant to produce better quality product and tools than the bloomery iron. It is imperative, based on these results, that should look for the iron refining tradition elsewhere in central, southern, and eastern Africa as comparative case studies.

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### **M36 Approaches to Ancient Metallurgy in the Atacama: Scientific Analyses of Metal Artefacts from Oasis Sites in Northern Chile**

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The San Pedro de Atacama region is home to some of the most important archaeological sites in northern Chile, having experienced continuous human occupation for more than 4000 years (Agüero, 2005; Llagostera & Costa, 1999; Núñez, 1995). Most investigations of these settlements have built on information from over 3000 burials, excavated in 47 cemeteries by the Belgian priest Gustavo Le Paige over the course of nearly three decades (Le Paige, 1957-58, 1963a, 1963b, 1964a, 1964b, 1970, 1974). Different categories of artefacts including implements such as axes, maces, chisels, and tweezers, as well as adornments which include pins and metal discs, have been recovered from funerary contexts of thirteen small agricultural oases of San Pedro. Over a thousand metal artefacts from the Le Paige collection have been recorded and saw-cut samples and drill shavings were obtained from 175 of these objects. The examined samples have been analysed by XRF. The saw-cut samples have in addition been studied by metallography and SEM-EDS, and characterised chemically by electron microprobe. Twenty samples, including both shavings and fragments, have been further selected for lead isotope analysis.

The results obtained so far have been extremely valuable. XRF analysis has enabled us to characterise the different elements present in the metal objects. These results might provide information on the nature of the raw materials used. Metallographic and mineralogical results demonstrate the occurrence of both oxides and sulphides in the samples. Because northern Chile is very rich in mineral sources and oxides tend to be abundant, it had been assumed that pre-Columbian miners focused largely on these easy-to-work minerals. The results of our analyses, however, indicate that they were in fact making use of different types of ores. Finally, lead isotope analyses are being used to characterise potential ore sources in northern Chile. Lead isotope analysis has been one of the most important methods developed for the research of the movement of metal ores, ingots, and finished metal products in ancient economic systems (see Rehren & Pernicka, 2008; Yi et al., 1999). Our focus on process reconstruction builds on the experience made elsewhere in the world, while taking into account the specific environmental and climatic situation of the Andes, as well as the unique economic and political revolutions of the second millennium AD. This research thus represents a first step towards a large-scale map of prehistoric copper production and exchange across the Andean region.

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### **M37 The Disc Phenomenon. A Multidisciplinary Approach to the Study of 50 Sporadic Protohistoric Bronze Discs with Concentric Decoration**

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Discs, referred to in classical terms as 'armour-discs', are by bronze, bronze/iron or iron, and they were used for centuries by all the Italic cultures. All the discs are sporadic but 42 reports Abruzzo as provenience, and 31 a small area, Fucino, where till the end of the IXX c. there was a lake.

Research explores dynamics related to production, use and management of discs, alongside relationships between production centres, local workshops, and technological changes from bronze to iron and viceversa.

#### Methodology and discussion

- Quantification of finds and statistical treatment of data.

- Technological and traceological analysis, by stereo Optical Microscope (80x), of 54 discs: 26 discs, the whole collection of the Pigorini Museum of Rome, 27 discs from the Bellucci Collection of the Archaeological Museum of Perugia, 1 unpublished disc from Ortucchio of the Prehistoric Museum of Celano.

- Chemical characterization, by portable ED-XRF, of 27 discs.

50 discs present concentric decorations and 4, figurative ones. Research focuses on discs with concentric decoration and figurative samples are used for data comparison. Contextual discs with concentric decoration, all the times gender is recognizable, come from feminine burials. We propose a diachronic analysis of discs and we explore the hypothesis that discs with concentric decoration have both independent origin and different function from either armour pectorals than figurative samples. We retain that discs with concentric decoration are material to a feminine sphere and, in order to process them in this sense, we investigate their dress code through the study of decorations and suspension systems. About 25% of discs have been repaired during their life as objects. Particular attention is given to chemical compositions and technologies of the various constituent elements or parts of reparation, in order to compare data both inside the same disc and among those showing similar interventions. Tecno-traceological and chemical aspects suggest local workshops operated in the area with own strategies either compensating the absence of raw materials than adapting technology to commissions. At the same time, technology and chemistry of discs show production was performed by highly specialized workshops, hardly conceivable in protohistoric Abruzzo at the state of knowledge. Composition groups of discs are also recognisable.



**M38 A Multidisciplinary Approach to the Study of an Assemblage of Prehistoric and Protohistoric Bronzes from the Fucino Area, in the Aquila Province of Abruzzo**

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This study proposes a broader re-evaluation of all the resources available for the interpretation of -out of context- metal finds. The project aims to characterize an assemblage of sporadic finds known as 'Fucino bronzes', owned by the Pigorini Museum of Rome and the Archaeological Museum of Perugia. The project includes also the analysis of 48 contextual bronzes, owned by the Celano Prehistoric Museum, from the Final Bronze Age site of Celano 'Paludi'. The research explores dynamics related to production, use and management of objects, alongside relationships between production centres and local workshops.

Methodology and discussion

- Technological and traceological analysis by stereo Optical Microscope (80x)
- Quantification of finds and statistical treatment of data
- Chemical characterization of selections of objects by 2 portable ED-XRFs. Bronzes are selected on the basis of their typological, technological and territorial relevance: 27 sporadic bronze discs, 20 sporadic bronzes: 5 *aes rudae*; 7 beads; 1 spiral; and 8 bronzes reporting provenience Celano, 48 contextual bronzes from Celano.

Bronzes are studied as both groups and complex. Dynamics related to use and consumption of metals in the area and homogeneity degree in composition and technology of objects are also discussed. Inventory follows the general trend regarding Bronze Age. Ornaments and accessories are the most large category but, going back through the time, always more tools and, earlier, over all weapons are present. Chemistry of different objects show compositions are variable but consistent among functional classes. Analysis of the Celano materials indicates a large use of recycling for the metal supply. The inventory of the Fucino bronzes shows two opposite typological and technological tendencies: an obsessive traditional trend and a relevant presence of foreign suggestions, mostly referable to northern *European cultures*. Traceo-technological and chemical aspects of bronzes suggest local workshops operated in the area with own strategies. Both the Final Bronze Age materials of Celano and the Middle Iron Age management of discs stress occurrence of continuity in the metallurgical tradition of the Aquila province.

### **M39 A Hidden Wonder of Ancient Technology: The Roman Cart – The Metallographic and Energy Dispersive X-Ray Analysis (SEM-EDX) of Iron Parts of Carts from the Roman Empire**

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In the north-western part of Pannonia inferior province, which was situated in the present territory of Hungary, the Romanized native aristocracy pursued a very rare and spectacular burial rite for 150 years on the territory of the Eravisci tribe (Mráv, 2001; Kiss, 1989). Amongst the rich grave goods, they also placed two-and four-wheeled carts, and the bodies of the horses which pulled them along with their sets of harnesses in the graves. These horse-drawn vehicles served as a means of transport to the other world. Several archeologically analysed and reconstructable cart finds have emerged from the more than 30 rich wagon graves known in Pannonia at present, which supply an inexhaustible and so far unexploited source of the history of ancient technology (Kiss, 1989; Röring, 1983; Raepsaet, 1982; Венедиков, 1960). The excavations have provided the most new data to enhance our knowledge of the two-wheeled vehicle, thus we have chosen the material analysis of the iron parts of the two-wheeled carts found in these graves as the topic of our presentation (Thiele et al., in press). We have conducted metallographic and energy dispersive X-ray (SEM-EDX) analyses of 50 iron parts from 5 two-wheeled carts of different ages and constructional mechanisms. Analysing the results of the material analyses, we can get an insight into the material quality of the iron used by the wagon smiths of the Roman Empire. Furthermore, by introducing an engineering aspect, we can make a new and complex picture of the selection of sufficient iron material and the application of an effective forging and heat-treatment technology against the mechanical strain at the time. As a result of our research, we can get a deeper understanding of a long-forgotten industry: the craft of the ancient cart smiths.

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## **M40 Set in Stone: A Technical Study of Casting and Inlay on Chinese Ceremonial Weapons**

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Some of the oldest and most recognizable objects from ancient China include the bronze weapons and ritual implements from the Three Dynasties (Xia, Shang and Zhou), ca. 1900 to 256 BCE. The technical study and scholarship of Chinese bronzes in the past half century has answered early questions and corrected misunderstandings about vessel casting technology, but rarely includes weapons or the inlay techniques and materials used to decorate them. This study investigated both the bronze casting and the mineral inlay that decorate the ceremonial weapons and riding implements from the Grenville L. Winthrop bequest at the Harvard Art Museums. Twenty-three bronzes, the majority with jade blades and turquoise-inlaid bronze handles, that range in date from the 19<sup>th</sup> – 11<sup>th</sup> centuries BCE, were selected for examination.

Characterization of the inlay technology included an extensive investigation of bronze casting evidence observed on the weapons' handles, and concluded that the mold-making process used for casting weapons is distinct from that used for early vessels. Unlike the shallow, decorative carved patterns prolific on early vessels, the weapons display tall, cloison-like decorative bands that were cast integrally with the handles. These cloisons protrude from opposing sides of a bronze floor, and evidence suggests they were carved into molds with the use of a stencil or stamp. The turquoise stones and other inlay materials set between the bronze cloisons were categorized into four groups through observations of shape, size, and finishing marks. The adhesive joining the inlay to the bronze was identified as a fruit tree gum, not lacquer as speculated in most literature. Evidence observed on the objects along with casting replication experiments suggest that early Chinese production for inlaid weapons began with jadeworking, followed by bronze casting, and finalized with decorative turquoise inlay. The primary scientific methods employed for this investigation include examination with the stereo binocular microscope, ultra-violet (UV) light examination, X-radiography, X-ray fluorescence (XRF), Reflectance Transformation Imaging (RTI), Raman Spectroscopy, Fourier transform infrared spectroscopy (FTIR), and Matrix-assisted laser desorption ionization (MALDI).

## **M41 Investigation of Copper and Early Copper Alloy Axes from the Carpathian Basin**

**Ivan Ordentlich<sup>†</sup>, Sarel Shalev<sup>1</sup>, Tibor Kovács<sup>2</sup>, Katalin T. Biró<sup>2</sup> and Florin Gogaltan<sup>3</sup>**

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On the initiative of Ivan Ordentlich in 2001 17 copper- and early bronze axes were selected for analysis from the Prehistoric Collection of the Hungarian National Museum. At the same time a similar pilot project was started in Romania with the help of Florin Gogaltan, extending over 16 axes. The aim of the analyses was to increase knowledge on early metallurgy in the Carpathian Basin in the framework of a „pilot project”, to be followed by further and more extensive analyses. The evaluation of the two lots was planned to be synchronized. These plans were sadly interrupted by the sad and unexpected death of Ivan Ordentlich. The existing results are presented here in memory and appreciation of Ivan and in accordance to the original idea: give more information of early metallurgy of the region and with hope to finish in the future the work that he started.

The axes were sampled using a very thin file with a hand jeweler piercing saw. On the fresh metal surface, after mounting and polishing, Wavelength-Dispersive X-Ray Spectroscopy (WDS) was performed at the Dept. of Materials Oxford Univ. U.K.

Five measurements on each sample were performed. The metal composition was assessed based upon the normalized results. The normalized point analyses were then averaged and subjected to preliminary basic statistical tests.

Some of the axes were most probably analysed by OES in the series SAM. Part of the axes analysed were further investigated using TOF/ND for structural information.

The earliest samples are seemingly made of almost pure copper. Part of the axes contain less Cu but still contain no Sn. The rest contains considerable amount of Sn (3-10 %). Interrelation of type /composition of alloy was examined in function of chronological position.

SARIEL, SH., KOVÁCS T. AND BIRÓ, K.T., In press. Korai rézötvözetek vizsgálata a Magyar Nemzeti Múzeum gyűjteményéből = Investigation of early copper and copper-based alloys from the collection of the Hungarian National Museum. *Archeometriai Műhely*.

**M42 'The Objects that Are More than their Analyses': An Archaeometallurgical Investigation and its Implications for the Study of Ancient Technologies**

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Metallographical investigation of the bronze votive offerings deposited at the sanctuary of ancient Pherae in Volos took place in order to explore aspects of Early Iron Age (EIA) technology in Greece\*. The sanctuary itself and the two gods (Enodia and Thavlios Zeus) to which it was dedicated were of great importance not only for Thessalian cult, but for the whole of the mainland Greece and the Aegean as well. Over this period and with an emphasis on the 8<sup>th</sup> and 7<sup>th</sup> centuries BC, several thousands of small bronze artefacts, mainly dress ornaments, jewellery and other decorative objects, have been dedicated to the sanctuary. The material has been recovered from two deposits in close proximity to the actual temple.

A sample of 250 objects has been selected for surface and invasive analysis in order to investigate the nature of raw materials, metals and alloys used, as well as the metalworking techniques employed in the production of the bronzes. Analytical techniques include optical microscope, XRF, SEM-EDS and EPMA. Results are statistically processed and then tested against the artefacts' established typology and their attribution to regional workshops established in different geographical regions in eastern Mediterranean.

The examination of the bronze technology employed at the workshops of early Greece in one particular chronological period, i.e. the Geometric, reveals aspects of EIA technological knowledge and the way this was perceived, transferred and adopted by the different communities. Such an approach moves beyond traditional points of view which often adopt an evolutionary perspective focusing on the development of technology over time, thus treating ancient technology as a continuous series of technological achievements. Shifting the focus of research reveals the relationships between these communities, as well as the ways in which technology was transferred and embedded in them.

\* Results so far have been obtained as part of a still ongoing PhD project undertaken by S.O. under the supervision of Prof. Thilo Rehren and Dr Corinna Riva at UCL Institute of Archaeology.

**M43 The Origin and Chronology of Selected Medieval Silver Coins from Poland and Central Europe Based on the Chemical Composition, Raw Materials Sources and Technology**

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The purpose of this work was to determine the provenance and dating of a few groups of the early medieval Central Europe coins. The widely known and already described types of coins have been chosen. However, an attribution and chronology of them often constitute a serious problem for historians and numismatists. The main focus of this study was on early medieval items, mainly Sachsenpfenning struck from the mid tenth century till the end of the eleventh century. Moreover, the Saxon coins, so called the Otto and Adelheid denarii as well as the Polish ones, the Władysław Herman and Bolesław Śmiały coins were examined as a comparative material. Totally, about two hundred coins was studied. The ores from selected sources (Olkusz – Poland and Rammelsberg –Hartz) were also analyzed to determine a provenance of metals used. SEM-EDS and X-ray fluorescence analysis (EDXRF, TXRF) as non-destructive methods were chosen to study a large number of samples. Apart from composition study of coins was carried out analyses of lead isotopes ratios by means of LA-ICP-MS method. Interpretation of the results of the statistical methods was allowed to differentiate the artefacts in relation to the various production centres, various recipes as well as various raw materials and methods of their purification used. Database for different types of coins is result of these studies.

#### **M44 Reconstructing/Dating Technologies in Prehispanic Gold Metallurgy**

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<sup>3</sup> Fundación Museos Banco Central de Costa Rica

<sup>4</sup> Museo Arqueológico Nacional, Madrid

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Combining different analytical techniques and systematic research has been the methodology applied to the study of one of the most important prehispanic gold collections in Europe kept at the Museo de América in Madrid: the Quimbaya treasure from Colombia, and the Costa Rica Collection, with a total of near two hundred objects. We have completed a three years research program, funded by the Spanish Ministry of Science and Innovation (Ref.: HAR2009-09298), with the aim of studying gold alloys, manufacturing techniques, surface treatments and state of conservation combining Optical Microscopy, XRF, SEM-EDS and PIXE-RBS. In some unexpected cases we were able to obtain organic material from the interior of vessels that were analysed by AMS, an important event due to the lack of archaeological contexts and chronological references in pre-Columbian metallurgy. This complementary research action was again funded by the Spanish Ministry of Science (HAR2011-12809).

We present the first results and discuss the problems we had to tackle with, both from the analytical point of view and from the inadequate cleaning of the objects through their actual life since 1893, when they entered the Museum.

PEREA, A., FERNÁNDEZ-ESQUIVEL, P., ROVIRA-LLORENS, S., RUVALCABA-SIL, J.L., GARCÍA-VUELTA, O. AND CUESTA-GÓMEZ, F., 2011. Prehispanic gold metallurgy: The Arqueometal research project. *Archaeometallurgy in Europe 2011. 3rd International Conference*. Bochum, Germany.

PEREA, A., FERNÁNDEZ-ESQUIVEL, P., ROVIRA-LLORENS, S., RUVALCABA-SIL, J.L., GARCÍA-VUELTA, O. AND CUESTA-GÓMEZ, F., 2011. Combining SEM-EDS, PIXE and XRF techniques for complex analytical problems: depth profile characterization of prehispanic gold. *Microscopy & Microanalysis 2011 Annual Meeting*. Nashville, Tennessee.

## **M45 Metallurgical Production Evidences in Castro de Vila Nova de São Pedro (Azambuja, Portugal)**

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The Castro de Vila Nova de São Pedro (VNSP) is an emblematic settlement located at Azambuja (Portugal), occupied during the third and second millennia BC, predominantly during the Chalcolithic period. The present study focused on the chemical and microstructural characterization of selected artefacts and metallurgical remains from the VNSP collection, including crucibles, slags and metallic debris aims to contribute to a better comprehension of the copper-based metallurgy on the Portuguese Estremadura. A diversified collection of 53 copper-based artefacts, fragments of artefacts and several metallurgical production remains (8 crucibles and 12 metallic nodules - slags and droplets) belonging to VNSP were characterized by using different analytical techniques: EDXRF spectrometry, micro-EDXRF spectrometry, optical microscopy and SEM-EDS. The combination of these techniques allowed the determination of the alloy composition and structure, as well as the identification of the thermomechanical processes applied to the shaping of the artefacts. Vickers micro-hardness measurements were carried out to establish the actual effectiveness of the thermomechanical processes in the hardness of the artefacts. Results show that the artefact collection is mainly composed of copper or arsenical copper being 38% of the artefacts copper alloyed with arsenic (As>2%). A statistically significant association was found between copper alloyed with arsenic and artefacts classified as weapons (blades, arrowheads and daggers). In several cases, the presence of arsenic rich phases in the microstructure indicates the absence of proper thermo-mechanical control in order to achieve chemical homogeneity during the processing. Microstructural analyses also show that the majority of this artefactual group was shaped with forging plus annealing operation cycles and 23% of the artefacts received a final forging treatment. This final treatment was associated to artefacts presenting higher arsenic contents. Preliminary results on the elemental composition of the metallurgical production remains are consistent with copper and arsenical copper artefact production. Further analysis, involving optical microscopy and SEM-EDS, will help to determine if these remains are related with melting or smelting operations (of copper ores or copper with arsenic ores) in crucibles. This research work has been financed by the Portuguese Science Foundation (FCT-MCTES) through the EarlyMetal project (PTDC/HIS-ARQ/110442/2008) and the PhD Grant SFRH/BD/78107/2011 (FP). The financial support of CENIMAT/I3N through the Strategic Project-LA25-2011-2012 (PEst-C/CTM/LA0025/2011) is also acknowledged.



## M46 A GIS Database to Study the Evolution of Copper Metallurgy in the Alpine Region

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This work is about the beginning of metallurgy in the Alpine region. The period from the Chalcolithic to the Early Bronze Age is critical for metallurgy's development. In this region, metal production ranges from pure copper objects to bronze objects with an almost standard amount of tin, to very different copper alloys. Many compositional analyses have been done, such as the fundamental work of Otto & Witter (1952), Junghans *et al.* (1960, 1968, 1974) and Ottaway (1982).

I propose to use these data to understand how the composition of metal objects changes in space and time and to speculate on some possible metal trade routes.

To achieve these aims GIS (ArcGIS10) is the most suitable research tool. All data have been grouped according to the site of provenience of the objects. Sites have been georeferenced with GIS to create a connection between archaeometrical data and their geographical position. Moreover, using a GIS instrument even topographical information can be achieved. Finally, time dimension have been obtained using different layers.

Preliminary results show spatial distribution of different groups of objects defined by their composition.

A further step has been treating data to understand the frequency of occurrence of each compositional group of material, and how this frequency changes in space and time. This kind of analysis allows the possibility of identifying which are the original centres of production of each group and where the groups have spread over time.

Hypotheses on trade routes can then be pursued, taking into consideration not only the geography, but also the topography of the territory, highlighting the role of rivers, valleys and mountain passes.

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## **M47 Into the Crucible: The Impact of Ceramic Temper Choice on Metallurgical Practice**

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This paper focuses on a series of experiments aimed at exploring the smelting of sulphide ores in prehistory, primarily through the matte conversion process. The specific goal is to test the impact that metallurgical ceramics have on the successful production of a copper matte. Attempts to reconstruct the matte conversion process have often encountered problems as the low viscosity of liquid matte causes it to be absorbed into porous ceramics. Archaeologically this problem is particularly evident in Middle Bronze Age, Sintashta communities found on the Southern Ural Steppe where metallurgists are known to have exploited sulphide ores, but metallurgical ceramics show little evidence of adherent matte. The experiments outlined in this paper aim to explore the impact that various tempering methods and recipes have on the ability of a crucible to retain a liquid matte whilst remaining comparatively unaltered.

The experimental program consisted of a series of controlled lab test and full reconstruction smelts that employed a selection of archaeologically relevant tempers and recipes. The tempers and recipes that were selected were drawn from studies of prehistoric refractory ceramics in areas which are known to have exploited sulphide ores. The levels of absorption and ceramic/slag interaction were assessed in both sets of experiments using a combination of analytical techniques that included optical microscopy, scanning electron microscopy and x-ray fluorescence. The results show that temper choice has a significant impact on the ability of a crucible to retain a liquid matte with results ranging from a complete absorption of the charge to a near perfect retention. Microscopic and chemical analysis allowed inferences to be drawn regarding the mechanics of these results as well as to suggest a framework for better understanding Sintashta material.

## M48 Ancient Urban Copper Metallurgy in Qantir (Pi-Ramesses)

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The main goal of the research project presented here is the investigation of the large Late Bronze Age copper-alloying complex that has been excavated in Qantir (ancient Pi-Ramesses) in the eastern Nile Delta (see e.g. Pusch, 1990, 1994; Pusch & Rehren, 2007), to assess its position within the urban context, as well as within the larger Eastern Mediterranean region. On the one hand, this will be done by looking at the technical processes that were used within the different areas in the metal-working zone in Qantir. To assess these, the excavated crucible fragments will be studied using reflected light microscopy and SEM, as well as analysed chemically using SEM-EDS. Looking at the internal variation of metallurgical technology within the site will shed light on the organisation of the different areas within the complex. On the other hand, the metallurgy complex will be investigated by attempting to provenance the metal artefacts and processing remains, using lead-isotope analysis. This will help bring understanding to how the metallurgical installation in Qantir was supplied and how this compares to other contemporaneous sites.

The project will give a deeper understanding of LBA urban copper metallurgy, specifically in Egypt, but it will also attempt to give a better insight into the complex copper trade relations during this period in the whole of the eastern Mediterranean.

This investigation is part of the PhD research performed by the author. By the time of the conference, the author expects to be able to present preliminary findings of this research, consisting of metallography and SEM results, as well as an outline of the further work and expected outcome.

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#### **M49 Mankinds Earliest Iron - Really Meteoritic?**

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The earliest known iron objects are a set of originally nine elongated iron beads, excavated a hundred years ago by Petrie and Wainwright at Gerzeh, Egypt. Forming part of a tomb dated to about 3,300 BC and associated with numerous other tubular beads made from lapis lazuli and agate, and other materials such as a stone mace head, a copper harpoon, a fish-shaped slate palette, and numerous ceramic vessels, they are archaeologically well contextualised. Predating the invention of bloomery smelting by nearly two millennia, they are commonly assumed to be made from meteoritic iron; full chemical analyses of two of these beads in the 1920s, however, failed to provide convincing evidence for or against this hypothesis.

The present study uses a combination of non-invasive surface and bulk body analytical methods to determine various diagnostic chemical and structural features unique to meteoritic iron to test this hypothesis once more. Neutron imaging and ToF-Neutron Diffraction were used to characterise the overall internal structure and texture of the beads, looking for specific intermetallic phases and the overall texture of the iron metal, should any have survived. Prompt Gamma Activation Analysis provided detailed information about the major and trace element composition across the entire body of the beads, while PIXE and XRF were used to determine their surface composition.

For comparison, partly corroded fragments of an iron meteorite of similar 'Earth Age' from Peru and a fully corroded medieval arrow head from the Levant were analysed using the same range of methods, supplemented by optical and scanning electron microscopy on polished cross sections to aid in the interpretation of the non-invasive analytical results. The presentation will summarise and discuss the results obtained, and offer a comprehensive interpretation of these beads based on analytical data, and their significance for the history of iron working and jewellery development.

## **M50 Assay of Metallurgical Investigations in Hungarian Cannon Casting Workshops**

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The casting of brass cannons covers larger historical period in the area of Hungary than in the areas of western Europe (15<sup>th</sup>-17<sup>th</sup>. century). In this era three cannon casting workshops were present in the area of today's Hungary. One was at Buda in the 15<sup>th</sup> century during the reign of King Mátyás I, one at Miskolc-Diósgyőr in the 15<sup>th</sup> century as well and one at Sárospatak in the middle third of the 17<sup>th</sup> century. According to the contemporary documents and knowledge the archeological revelation of the cannon casting workshops step in the final stage. A large amount of metallic founding was revealed in every cannon casting workshop. The remains of the melting furnaces were also found during the excavation of the workshops. At the digging site of Sárospatak it was found in a especially good condition. Furthermore, remains of casting dies and casting sockets were also found besides the metallic items. Bell casting was certainly present besides the cannon casting in the workshops of Diósgyőr and Sárospatak. The foundings confirm this at the workshop of Diósgyőr, while no foundings refer to this in the workshop of Sárospatak, but the contemporary documents report about bell castings too. No evident of bell casting was found at the workshop of Buda so far.

The Instrumental examination of the mentioned systemized foundings ensures to study the metallurgical and casting processes and the evolution of the technologies of these casting workshops. The composition, microstructure of the metallic foundings, the mineral composition of the oven components and casserole materials and the remains of the interaction between the melt and the casserole materials reveal the technologies of the alloy production, melting, the conditions of the castings. Some items from the foundings were subjected to optical microscope and scanning electron microscope examinations. X-ray diffraction examinations were carried out on the non-metallic technological foundings connected to the cannon casting.

The results of the examinations on the metallic foundings connected to the cannons – cannon production will be presented. Cannon pieces prepared for remelting, melt drops and melt pieces, adhered pieces solidified at the bottom of the cast die were examined. The metallurgical properties, the quality of the metallurgical processes were investigated through the comparison of the results. The method of the alloying was revealed by combining our results with contemporary documents. The accuracy of the alloying technique was revealed by our results. The conclusions of the different workshops were combined to get a whole picture about the contemporary brass metallurgy and its evolution in the area of today's Hungary. These relationships will also be presented besides the experimental results.

## **M51 Specialised Choices? – The Manufacture of Clay Moulds in Late Prehistoric Scotland**

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Detailed studies of the technology of Bronze Age and Iron Age clay moulds used in the casting of non-ferrous metals have so far been limited; either looked at the utilization and the moulds impact on the ready cast, or made generic statements of the manufacture. Such studies are important, but have to a large degree ignored the question of the selection of raw materials and how the ceramic material was prepared. In the past decades have theoretical and material studies demonstrated that individual and social choices had an important role in the development and construction of technology (cf. Sillar & Tite, 2000).

This presentation, using ceramic petrography combined with SEM-EDX investigates the ceramic technology of Scottish late prehistoric casting moulds. The aim is to examine choices and practices made in the production sequence of moulds from the collection of appropriate clay to the preparation of the finished mould. A diachronic perspective of production at a number of sites gives the possibility to highlight technological developments and investigate variation of individual practices. It is clear from the current study that in the production of ceramic moulds in late prehistoric Scotland the craftworker predominately local clays, but it is also clear that there is a broad variation in the preparation of ceramic paste. Some of this is related to chronological and environmental differences, but it is also clear that there is some variation in technology related to difference in mould type and/or individual practices.

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## **M52 Metallographic Evidences of Bronze Casting Conditions in Early Iron Age**

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The Moscow region which is situated in Volga-Oka interfluvium was inhabited by the tribes of the Dyakovo archaeological culture in the period of the Early Iron Age. The studies of the archaeological material have allowed reconstructing some of the aspects of the population's material culture. Thus it is known that above all the inhabitants were processing nonferrous metal – were making melting and casting – in the territory of the site. There are some views on the existence of the nonferrous metal processing in the studied territory in the academic literature inter alia on the jewellery. Whether making jewels is the population's occupation (so to say "woman's occupation") in their free-from-agricultural-works time or this is a specialized branch of the craft with all the assistant attributes (craftsmen, traditions and workshops). Metallographic evidences supplemented by the studies of the chemical composition of nonferrous metal and the evidences of the method of jewellery making allow getting a foretaste of the foundry conditions and general development of the bronze casting in the period of the early Iron Age. It also allows studying of the traditions in the territory in question. This research presents another version of the nonferrous metal processing viz. jewellery in the territory of the Moscow region in I b.c.- I a.d. It is likely that making jewels as a domestic craft was coexisting that period with the jewellery which itself was the independent branch of craft. Moreover there was the import of jewels produced in the other jewellery tradition.

### **M53 Trace-Elements and Lead Isotope Ratios for Provenancing Early Medieval Silver Coinage**

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Tracing ancient silver can be achieved using two complementary approaches: trace element analysis and lead isotope ratios analysis. Recent developments allow to characterize silver by laser ablation ICP-MS and to determine the concentration of numerous trace elements susceptible of being used for provenance studies (Sarah et al., 2007; Sarah, 2010): they were contained in the metals constituting the matrix and not added on purpose by the ancient metallurgists. The content of some of them remained unchanged during the metallurgical processes which occurred to produce metal from the ore, so their determination is susceptible of revealing the source of the precious metal. The measurement of the lead isotope ratios is another powerful way to discriminate coins made of silver from different origins, and possibly to trace the mine it was extracted from (Cattin et al., 2009).

For both trace elements and lead isotope provenancing, limitations have to be borne in mind, especially due to possible remeltings or identical fingerprint for different sources. But the combination of these two approaches, after an accurate study of the historical context, appears to be extremely promising for tracing silver.

A project named FAHMA (for Filière de l'Argent au haut Moyen Age, or Silver processing during the early Middle Ages) has been launched a few years ago, which aims at combining the skills and knowledges of historians, numismatists, archaeologists, archaeometallurgists and chemists. In this project the emphasis is on the medieval silver mine at Melle in France, which was the only of any significance which is known to have been active in Western Europe during the early Middle Ages (Sarah et al., in press; Téreygeol, 2010). Archaeological artefacts have been studied (galena ore, lead, vitreous slags) and their elementary and isotopic fingerprint are known. A wide range of Carolingian coins has also been analysed, firstly by LA-ICP-MS, and the lead isotope ratios have been measured for a significant part of them.

The results obtained have been compared with numismatic and historical data. This interpretation reveals changes in the provisioning of silver for some mints located in modern France regarding the trace element and lead isotope fingerprint of coins minted there. Although the results underline the significance of Melle, other cities, especially Clermont and Toulouse, used silver which is different from both trace elements and lead isotopes fingerprinting. This study confirms the great potential of combining those two approaches for provenancing silver, and provides significant information concerning the circulation of silver in Western Europe during the early Middle Ages.

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SARAH, G., GRATUZE, B. AND BOMPAIRE, M., 2007. Application of laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) for the investigation of ancient silver coins. *Journal of Analytical Atomic Spectrometry* **22**, 1163-1167.

TÉREYGEOL, F., 2010. Y a-t-il un lien entre la mise en exploitation des mines d'argent de Melle (Deux-Sèvres) et le passage au monométallisme argent vers 675 ? In: BOURGEOIS, L.(Ed.) *Wisigoths et Francs autour de la bataille de Vouillé (507). Recherches récentes sur le haut Moyen Age dans le Centre-Ouest de la France*, 251-261.

#### **M54 Early Iron Age Phoenician Bronze Ex-Votos from Southwestern Iberia: A Preliminary Study by EDXRF and BSEM+EDS**

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As part of a large multidisciplinary study aimed to identify common elements in ancient metal artifacts influenced by Phoenician-Punic presence in the Mediterranean area and to reconstruct routes of technological transfer from primary centers of production in the East to secondary centers in Western areas (i.e. Sicily, Sardinia, Morocco and Iberian Peninsula), 8 bronze ex-votos of unknown provenance, composed of 4 anthropomorphic figures (three male and one female) and three zoomorphic representations (two goats and a cow), and by an anthropomorphic tripod, from the Museum of Evora, (Portugal), archaeologically dated to the Late Bronze Age- Early Iron Age have been investigated. This type of artifacts, widely present not only in the Portuguese Alentejo region but also in Spain, could be linked to the local cult of the goddess Adaegina, although, typologically, reference to the Phoenician gods Baal and Astarte could not be excluded. The aims of the archaeometric research were to assess whether the production could be identified as Phoenician, Iberian, or Orientalizing, to identify the local area of provenience of the bronze ex votos, and to eventually restore the historical significance of the entire Evora collection. For comparative purposes, a geo-referenced database was created (using the ArcGIS software) based on published archaeological and archaeometric data on Phoenician sites in Portugal (Arruda, 2000; Figueiredo et al., 2011 and references therein) coupled with geological data on ancient metal mines active during the Phoenician period. The chemico-mineralogical study of the Evora collection was performed by Stereomicroscopy, X-ray Fluorescence Spectroscopy (XRF) and Scanning Electron Microscopy and Energy Dispersive X-Ray Spectrometry on cleaned areas of the statuettes. The alloy chemical composition (bronze with lead) of the statuettes suggests an Early Iron Age production period. The presence of iron, higher than the LOD further suggest that the production was not Iberian but Orientalizing Iron Age. Querying the GIS database on the presence of archaeological sites with Phoenician presence, in which metallic ex votos were found, the Portuguese site of the Orientalizing area was located at Alcacer do Sal, where similar offerings were found.

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## **M55 Composition and Microstructure of Silver from Hoards in Tel Beth-Shean, Tel Dor and Tel Migne**

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Chemical composition and microstructure of objects excavated in three different hoards from Late Bronze Age to the Iron Age are studied using ED XRF method and optical microscopy. The objects are obviously of industrial origin (scrap of jewelry pieces) and different shaped ingots (like hackzilber mainly in shape of chopped chocolate bars in various sizes). The typical composition is Ag with several percent of Cu and Au and traces (in less than percent) of Pb. The concentrations of these metals are significantly different from one hoard to another. The microstructure of the hackzilber ingots shows a typical dendritic as-cast structure and a cold work deformation of the upper edges caused probably by chiseling the ingot piece out from a bigger cast bar. Higher concentration of Au, like in the above samples, was observed previously in Egyptian ancient silver, and differ from the low content of Au detected in Greek ancient silver.

## **M56 Iron Smelting in South Africa: New Finds and a Reassessment of the Landscape**

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New finds along the Komati River and at Machadodorp in South Africa suggest that developed iron smelting was taking place in the region. Evidence identified at this point in time includes extremely high grade iron ore (haematite and magnetite, with red and yellow ochre and other iron-rich minerals), a tuyere fragment, vitrified ceramics, and iron slag. Initial investigation of the slag shows a well-developed fayalite structure with wustite dendrites. The included metallic iron is hyper-eutectoid cementite rich. Analysis and characterization of these materials is being performed using OLM, Raman, XRF, and SEM.

These finds are offering a new interpretation for the thousands of complex stone-built structures along the escarpment in Mpumalanga Province, indicating that they may have been primarily used in the iron ore extraction and smelting process rather than for farming.

## **M57 Envaluing Past Practice: A Framework for the Spatial Analysis of Metal Production in First Millennium BC Britain**

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This paper critically reviews current investigations into metal production in Britain in the first millennium BC and offers a new framework for the study of craft practice which integrates theoretical concerns with emerging scientific methods. The inception of iron metallurgy in Britain has often been described as the result of a wave of technological change sweeping across Europe after 1000 BC (Pleiner, 1980). The advent of iron metallurgy in Britain was accompanied by a shift from a metallurgical technology centring on distinct distant copper deposits, to one which seems to have exploited local deposits, with production often located at conspicuous points in the landscape. Such changes in practice represent dramatic changes in the way metallic substances are valued and socially constituted. Instead of conceptualising iron metallurgy as a chemical process, this study sees metallurgical practice as a defined set of social acts which are located in space, involving the exploitation of knowledge, materials and skilled individuals. It is this dimension of metallurgy which to date has been underexplored and remains open to significant archaeometric investigation. The pioneering work of Mills & McDonnell (1992) showed how existing techniques, such as magnetic susceptibility, could effectively map ironworking micro-debitage in space. Despite the example of this early application, the lack of a coherent theoretical framework meant that the interpretive potential of such an approach remained unrealised. More recently, detailed spatial mapping of archaeological and experimental contexts associated with metal production has been undertaken utilising portable x-ray fluorescence and magnetic susceptibility. These data when contrasted with the technological choices evidenced from the macroscopic and microscopic analysis of production debris offer the ability to bring new understandings to metallurgical contexts and renegotiate the role of craft practice in our understanding of societal development.

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## **M58 Bell Beaker Gold Foils from Perdigões (Southern Portugal) – Manufacture and Use**

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The archaeological site of Perdigões is one of the most important chalcolithic settlements in Southern Iberia due to its size, architecture, spatial organization and recovered archaeological material. The settlement occupies an area of 16 ha delimited by concentric ditches. The outer ditch was enlarged to enclose a graveyard with several collective tombs. In one of them, the Tomb 2, displaying a circular chamber, a short corridor and an atrium, several phases of funerary use were identified, namely one belonging to the Bell Beaker period. Several ivory lunulae, V-perforated buttons and a dozen of fragments of gold foil can be ascribed to that Bell Beaker context dated to the third quarter of the 3<sup>rd</sup> millennium B.C. The thickest recovered gold foils – rectangular plaques with a thickness of c. 200 µm and 7-9 mm wide – are strong enough to support their own weight and could be used as diadems or other kind of ornament without need of backing material. The remnant gold foils, with thicknesses between 40 and 200 µm, in a very fragmentary state, present all of them a shiny golden surface (the obverse) while the reverse is invariably dull, suggesting that these gold foils were used attached to some sort of a fabric backing. EDXRF and PIXE microanalysis were used to determine the elemental composition of the gold ornaments recovered at Tomb 2 of Perdigões. The results point out to a rather pure native gold used in their manufacture – Ag contents from 0.5% to 4.9% while Cu is less than 0.08%. The microstructure of the gold foils was studied using Optical microscopy (OM). Twinned grains indicate that several cycles of hammering followed by annealing took place to obtain the foils. Inclusions observed by OM were also analyzed by SEM allowing the identification of Si and O, also suggesting the use of native gold in the manufacture of these jewels. In order to explain the dully surfaces of the foils additional chemical analysis by µ-FTIR were performed on the material scraped from those surfaces of two foils. The spectra allow the identification of kaolinite, traces of calcium carbonate, and an organic material, a wax, i.e. probably a mixture of clay and bees wax were used. As far as we know this is the first time that the material applied for the attachment of such thin foils to a fabric backing is identified. This research work has been financed by the Portuguese Science Foundation (EarlyMetal Project - PTDC/HIS-ARQ/110442/2008). The financial support of CENIMAT/I3N through the Strategic Project LA25/2011-2012 (PEst-C/CTM/LA0025/2011) is gratefully acknowledged.

## **M59 Technology of Iron Metallurgy in Early Medieval Hungarian Bloomery Workshops – Comparing Material Investigations of Slag, Iron and Ore Samples from Excavated and Reconstructed Smelting Experiments**

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There are different types of the early medieval bloomery furnaces located in the Carpathian Basin. One of the built-in type furnaces is the so-called Fajsz-type. This kind of bloomery from the 10<sup>th</sup> century AD – named after Somogyfajsz, the most widely known site of this type (Gömöri, 2006) – was the first one whose use was a characteristic feature of the Hungarian conquerors. The Fajsz-type furnace was approximately 70-100cm tall and it was built into the side wall of a workshop pit with its full height. In our research we tried to answer some basic questions regarding the early medieval technology of iron metallurgy in Pannonia. What kind of iron ores were used in the bloomery? What was the available productivity of bloomeries and the quality of iron blooms like? What kind of technological parameters were suitable for a successful metallurgical technology? During the past three years an engineering team set out to discover this medieval technology in a collaborated study with archaeologists using experimental archaeology involving more than 30 reconstructed smelting experiments in the Fajsz-type furnace (Thiele, in press). In these experiments the main influencing parameters of the technology (i.e. temperature distribution in the furnace, air supply, gas composition, quality and quantity of iron ore and charcoal, etc.) were measured. The raw materials and samples of the obtained iron bloom and slag were examined by chemical (ICP), metallographic, energy dispersive X-ray (SEM-EDX) and mineralogical (X-ray diffraction) analysis. Chemical and metallographic investigations of iron ore and slag finds found in excavation sites of Fajsz-type bloomeries and workshops (Ágh & Gömöri, 1999) and from other medieval sites in the Carpathian Basin (Török, 2010) were carried out as well. The experiences of experimental smeltings have been compared with the archaeometallurgical remains of Pannonia. These comparisons provide guidance to further understanding the subject. By these results the physicochemical and metallurgical parameters of the technology will be more approachable. Contemporaneously the reconstructional experiments serve as feedback concluding the technical characteristics of Hungarian iron metallurgy of the 10<sup>th</sup> century as well as the materials testing of the finds.

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## **M60 Recreating a Roman Stoneworkers Tool**

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During excavation in 1992 of the Porto Novo, a 1<sup>st</sup> century A.D. Roman shipwreck discovered off the coast of Corsica, numerous large ferrous concretions were recovered. X-radiography of one of the concretions revealed a cavity in the shape of a hammerhead and a partially preserved wooden handle. After careful excavation of the concretion, a silicone cast was made of the cavity followed by an epoxy replica. In 2010, the epoxy cast was used as a template to forge a mild steel reproduction of the hammerhead to a shape and weight that matched the epoxy replica. This poster reports on the process of replication of the hammerhead including an interpretation of the handle and the hafting technique.



## **M61 Archaic Goldwork from the Portuguese Area: Alloys, Technologies and their Relation to the Atmospheric Corrosion**

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The abundance of gold and silver deposits in the Iberian Peninsula led, since the Chalcolithic, to a rapid and significant development of goldsmith's techniques in the Portuguese territory that attained the most important turning points in the Early and in the Late Bronze Ages (Canosa, 2000). The National Archaeology Museum exhibits in Lisbon the most relevant Portuguese archaic gold jewellery. However, during the last years, several objects developed corrosion, which can be related, in part, to the exhibition conditions (Liang et al., 2011). On one hand, the scarce publications on analytical studies of Portuguese archaic gold jewellery disrupted insight knowledge on materials and technologies and on the other hand, the knowledge on environmental parameters and display materials is also insufficient to tackle the questions raised by the artefacts corrosion. In fact, the corrosion products patterns and distributions suggest a relationship between corrosion and fabrication technologies.

By using optical microscopy, XRF and XRD, this work gives the first data obtained for gold archaic jewellery from the collection of the National Archaeological Museum, assembling the study of the goldsmiths' techniques and the evolution of the base-alloys to the identification of the atmospheric corrosion products and their localisation on the objects surface according to the mounting and decoration techniques applied. A first set of artefacts was selected in the showcases displaying the Chalcolithic and the Late Bronze Age jewellery. From the technological point of view these objects represent the first technological transition – foil and wire are replaced by casting – while from the corrosion point of view they present rose alteration products with apparent patterns that could be related to the fabrication techniques and the silver contents present in the alloys. In order to evaluate the exhibition room environment and to identify the pollutants sources, a set of silver *cupons* was inserted in the showcases and Oddy tests were carried out for the showcase materials. The XRD analysis identified the presence of Ag<sub>2</sub>S and AgCl at the environment and the materials test showed the presence of Ag<sub>2</sub>S and AgI, allowing considering them as a source of pollutants.

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## M62 Complex Archaeometrical Examination of Iron Tools and Slag from a Celtic Settlement in the Carpathian Basin

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Traces of a diversified settlement system of the middle and late La Tène period (3<sup>rd</sup> century BC – 1<sup>st</sup> century AD) were found recently in South-West Hungary. The site of Ordacsehi-Csereföld is one of the largest sites of celtic settlements of the Carpathian Basin due to its 26 buildings, kiln for baking pottery, further objects and its huge amount of findings of local iron metallurgy (Gallina, 2000). Some of the celtic villages lying south to the Lake Balaton could keep on living thus continue its iron production during the age of the Roman culture up to the 2<sup>nd</sup>-3<sup>rd</sup> century (Gabler, 1989). Ten slag samples, nine iron tools and a bloom from the mentioned site were examined by our research team. Chemical analysis were carried out on the slag samples by ICP and wet assay furthermore, they were examined from a mineralogical point of view by XRD as well. The samples of iron items were examined by optical microscopy and SEM-EDX besides hardness test. The main objectives of examinations closing at the beginning of 2012 are to reveal the structural properties of the slag and metal items, the definition of the metallurgical functions of the different pieces of slag, the exploration of the traces of processing and probable heat treatments on the iron tools in order to characterize the technology of local smithing and to compare the microstructures of the bloom and iron tools as intermediate and final products. The present results can be well compared to our previous results on similar findings from the latter (7<sup>th</sup>-9<sup>th</sup> century) times of the same area (Gömöri & Török, 2002; Török, 2010; Török & Kovács, 2011), as well as to results of celtic iron items from other European sites of the same period (Sievers, 1996; Buchwald, 2005). The composition of the examined slags is very inhomogeneous and in many cases they contain higher amount of CaO than the slags from the early middle ages of the same area. In many cases reoxidized traces of previously metallized iron grains were found - which is typical of forge slag – which is also confirmed by the hematite content of the slag. During the metallographic examinations of the iron items it was conspicuous that the carbon content of the samples varied in a wide range, which result was also seen on the microstructure. Furthermore, the slag inclusions besides the ferrous matrix gave a hand in characterizing the production technology more accurately.

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GÖMÖRI, J. AND TÖRÖK, B., 2002. Technical Examination of the Early Medieval Ferrous Metallurgical Finds from Hungarian Sites. In: JEREM, E. AND BIRÓ, K. (Eds.) *Archaeometry 98, Proceedings of the 31st Symposium*. Archaeolingua, British Archaeological Reports (BAR) International Series **1043**, 375-381.

SIEVERS, S. 1996. Manching im Lichte neuer Grabungsergebnisse. In: JEREM, E., LEEB, K., NEUGEBAUER, A., URBAN, W. AND HERMAN, O. (Eds.) *Die Kelten in der Alpen und an der Donau*. Archaeolingua, Studien zur Eisenzeit im Ostalpenraum **I**, 327-333.; TÖRÖK, B.,

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## **M63 Finger-Impressed Phoenician-Punic Hearth Ceramics: Testing for Potential Metallurgical Usage**

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Structural hearth ceramics found in Sardinia and Tunisia at Phoenician-Punic archaeological sites were tested using a variety of methods to determine whether or not they were used for metallurgical production. Some of this material was first excavated at the Nuragic site of Ortu Comidu (Sardara) by A. Taramelli in the early 20<sup>th</sup> century, who identified four hearths as copper smelters. The site was reexcavated in the 1970s by M. Balmuth, revealing the ceramic hearths, each about 1 meter in diameter, with no bottom, and probably originally higher than the 30 cm to which the walls were preserved. They have numerous finger-impressions (*ditata*) found on the exterior surface, traces of burning on the inside and a layer of ash at the bottom.

Ceramic hearths have also been found in Punic contexts in Carthage, including a nearly complete installation on the Byrsa, which was initially identified as a bread oven or *tabun* because similar structures are still used for bread-making in rural areas of North Africa and the Near East. More recent excavations on the Byrsa, however, uncovered these same finger-impressed hearth ceramics in association with bowl furnaces and metallic slag, and some fragments were associated with tuyeres. The excavator published reconstructions in which the finger-impressed ceramics form a superstructure over the bowl furnace.

The contextual association of these ceramics with metalworking in Carthage thus required a reassessment of the initial interpretations made for Ortu Comidu. I used petrographic examination and refiring experiments to estimate the highest temperatures reached within these hearths, along with scanning electron microscopy and X-ray fluorescence analysis to find any copper-based or other metallic residues. These analyses were conducted on numerous fragments of finger-impressed ceramic hearths from Ortu Comidu, as well as Tharros, Nora, Monte Sirai, Senorbì, San Sperate, and Guspini in Sardinia, and on several samples from Carthage, to determine whether there was any evidence for a metallurgical function to these materials.

The low temperatures determined for both ash and ceramic samples, and the absence of copper or other metallic residues on the hearth ceramics, along with insignificant finds of metal or slag, reinforce Balmuth's interpretation that Ortu Comidu was not a Nuragic bronze foundry as initially proposed by Taramelli. The *ditata* finds at Punic sites in Sardinia were most like used as domestic ovens.

**M64 The Evolution of Copper Metallurgy in the South-Western Iberian Peninsula – New Evidences from the Pre and Proto-Historic Site of Torre Velha 3 (Southern Portugal)**

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Over the last few years several archaeological research projects have allowed to redefine the knowledge about the Pre-History of the South-western Iberian Peninsula. At the archaeological site of Torre Velha 3 (TV3), Serpa, on the left bank of the Guadiana river, besides several pits filled up with earth containing probable kitchen refuse, fragments of pottery, metallic artefacts and, in some of them, human burials, a large funerary hypogea assemblage was found. While the first negative structures reveal the large diachronic occupation of the site – Late Chalcolithic to Late Antiquity, with a large hiatus – the hypogea present a chronology of the second quarter/beginning of the third quarter of the 2<sup>nd</sup> millennium BC. Grave goods recovered from hypogea are meat offerings together with pottery and metal artefacts – probably personal belongings. In fact, the majority of metals have common typologies that were indiscriminately used along several millennia (awls, chisels and saws). On the opposite, other items exhibit narrower chronologies: Palmela points are very common in Late Chalcolithic and Early Bronze Age contexts, contrary to daggers with rivets that seem to be absent until the Middle Bronze Age or double-spring fibulae that are typical of later periods, namely Late Bronze Age and Early Iron Age. Nevertheless, several radiocarbon dates have been obtained from bone samples closely associated with metallic artefacts, establishing a reliable chronological framework regarding the typological and metallurgical evolution recorded at TV3. Elemental compositions determined by micro-EDXRF analyses showed the compositional diversity of metallic offerings and other artefacts from TV3 – copper, arsenical copper and copper-tin alloys with low amount of impurities. It was also possible to ascribe the introduction of bronze alloys in this western end of the Iberian Peninsula to the Middle Bronze Age, since several hypogea grave goods were found to be composed of bronze. Results were compared with other Iberian pre and proto-historic metal collections that allow a better knowledge about the metallurgical development for this Iberian region. Finally, microstructural characterization involving optical microscopy and SEM was made in order to establish the *chaîne opératoire* and also to identify common inclusions present in metal artefacts. The efficiency of forging and annealing operations utilized during manufacturing was ascertained, producing unique data regarding the Middle Bronze Age in this region. This research work has been financed by the Portuguese Science Foundation (EarlyMetal Project - PTDC/HIS-ARQ/110442/2008). Financial support of CENIMAT/I3N through the Strategic Project LA25/2011-2012 (PEST-C/CTM/LA0025/2011) is gratefully acknowledged.

## **M65 How Can 17<sup>th</sup> Century Bronze Cannons be Magnetic?**

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During investigations to the cause and type of corrosion of salvaged 17<sup>th</sup> century cannons, it became apparent that some of the bronze pieces were (ferri or ferro) magnetic. A magnet would stick at the surface of, what should be, a bronze cannon. What could be causing such a phenomenon?

The hypotheses are: 1) During manufacturing the mold for the cannon, an iron frame is constructed and added for strength. If this frame is close to the surface, the cannon could get magnetic. 2) Iron particles or scrap could be included in the bronze melt. 3) Magnetic iron minerals (magnetite, greigite) may have been formed during the stay on the seafloor.

We investigated the composition of eight 17<sup>th</sup> century cannons with hand held XRF. The results indicate that the magnetic cannons have higher iron contents than non-magnetic cannons. In addition, the tin content of the bronze was in some cases lower as was described or ordered in the 17<sup>th</sup> century. Was some iron added to the cannon in order to disguise the deficiency of tin? Material science research is currently being performed to confirm the addition of iron to the bronze cannons.

## **M66 The 9<sup>th</sup> Century Bloomery Iron Smelting in Northeast Thailand: A Case Study from Ban Kruat, Buriram Province**

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The study of Thai ferrous extractive archaeometallurgy suffers from an almost total lack of research. Of numerous archaeologically documented slag mounds in Thailand, only two sites have been archaeometallurgically explored; both of which revealed a practice of bloomery smelting (Nitta, 1991, 1997; Suchitta, 1983). Moreover, data is only limited to the prehistoric and early historic periods. Recently, the work of the Living Angkor Road project in the Ban Kruat area, Buriram province offered archaeologists an opportunity to explore one of largest iron production areas in Thailand (Lertlum et al., 2008). Ban Khao Din Tai, one of 67 surveyed slag mounds, was chosen to represent the local iron production. The excavations uncovered large numbers of metallurgical components ranging from furnace remains, technical ceramics, and slags to probable laterite ores. Thermoluminescence analysis of the furnace walls indicated that the site dated to the 9<sup>th</sup> century AD (1,111-1,135 BP). Nineteen samples covering all materials mentioned above were subjected to commonly applied archaeometallurgical instruments (optical microscopy, Scanning Electron Microscope-Energy Dispersive Spectroscopy (SEM-EDS), and Polarising Energy Dispersive X-ray Fluorescence ((P)ED-XRF) in order to reconstruct the extractive activities conducted on site. The analytical results confirmed the excavations data which suggested that the smelting took place in a shaft furnace with a probable slag-pit or slag tapping hole. All technical ceramics were produced by the same clay and paste and probably the same manufacturing technique. In terms of iron ores, there is still much debate surrounding the use of laterite, and whether it was or can be used at these sites. The slags were macroscopically classified into three groups, whereas microstructural and chemical analysis suggested just two groups. The smelting operation, which was conducted at a temperature around 1,150-1,200°C, was fairly consistent and efficient based on chemical composition, uniformity of slags, and the absence of free iron oxides. This pilot study is part of ongoing research which aims to use a technological approach to identify not only the iron production technology, but also the scale of production and organisation, and identify the intended market for such products. Also, these technological results may help explore the socioeconomic relationships amongst the region's iron makers and contemporary civilisations.

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**B1 Analysis of the Conservation State, the Traces of Polychromy and Gilding of Ancient Ivories****Marie Albéric, Katharina Müller, Dounia Large and Ina Reiche**

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Elephant ivory used for sculpted, colored and gilded museum artefacts is a fragile biomaterial subjected to complex physico-chemical alteration mechanisms occurring over time. The aim of this study is to present a combination of analytical methods allowing the precise evaluation of the preservation state of ancient ivory taking into account alterations in the burial environment and former restorations. Additionally, it allows in some cases the determination of original polychromy and gilding, if present, as well as identifying possible sediment traces that are characteristic of the burial conditions.

On the basis of the study of museum and archaeological objects by UV photography, X-ray radiography and tomography, electron microscopy, Visible, infrared and microRaman spectroscopies, microXRD as well as microPIXE at the AGLAE accelerator installed at the C2RMF the potential of these methods is highlighted in order to gain new insights into the complex history of the ivory museum objects (Fontan & Reiche, in press) or archaeological ivory remains (Large et al., in press).

FONTAN, E. AND REICHE, I., In press. Les ivoires d'Arslan Tash (Syrie) d'après une étude de la collection du Musée du Louvre : mise en oeuvre du matériau, traces de polychromie et de dorure, état de conservation. *Archéosciences* **35**.

LARGE, D., MÜLLER, K. AND REICHE, I., In press. Approche analytique pour l'étude des ivoires archéologiques. Les défenses d'éléphant du site de *Jinsha* (1200-650 BC, Sichuan, Chine). *Archéosciences* **35**.



## B2 Grave Goods in Predynastic Hierakonpolis, Egypt: Botanical and Molecular Approaches to Identify Plant Substances

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Botanical remains as aspect of Predynastic grave goods have often been neglected in the past, mainly due to poor preservation. However, they may add considerably to the interpretation of funerary equipment when they are well preserved (Buchez, 1998; Fahmy, 2005). Hierakonpolis is the largest Predynastic site known and consists of settlements and cemeteries, such as the elite cemetery HK6 (Friedman et al., 2011) and the “working class” cemetery HK43 (Friedman et al., 1999). Both of them were in use during the early Naqada II period (ca. 3700-3600 BC). Among the more puzzling finds from these cemeteries are a number of clay cones, often found in combination with leather and organic remains. These objects have also been found occasionally at other cemeteries and were considered as models of poppy-heads (Hartung, 2011). However, this interpretation is highly unlikely, if only because neither *Papaver somniferum* sp. *somniferum* nor *Papaver somniferum* sp. *setigerum* grew in Predynastic or Early Dynastic Egypt. In order to unravel the function of these cones, parts of their organic embedding were subjected to a multidisciplinary study involving both macrobotanical analysis and molecular techniques. Among the macrobotanic finds were pieces of coniferous wood, diverse plant tissues and resin-like red crystals. The latter were investigated by several analytical techniques: infrared microscopy, thin layer chromatography (TLC), high performance liquid chromatography (HPLC) and gas-chromatography coupled to mass spectrometry (GC-MS). By comparison with modern resin references, it was found that the resinous plant material most likely originates from myrrh (*Commiphora myrrha*). The modern geographical distribution of myrrh is situated in Southern Arabia and the Northeast of Africa, more than 2000 km southeast of Hierakonpolis. Considering both the long distance import of myrrh and the presence of other fragrant substances in the embedding of the cones, it would not be surprising if these objects are cheap models of the highly valuable myrrh resin. This would furthermore correspond well with imitations of other goods found in Predynastic tombs (cf. Hartung, 2011).

BUCHEZ, N., 1998. Le mobilier céramique et les offrandes à caractère alimentaire au sein des dépôts funéraires prédynastiques: éléments de réflexion à partir de l'exemple d'Adaïma. *Archéo-Ni* 8, 83-103.

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### **B3 Investigating Childhood Dietary Variation Using Stable Isotope Analysis of Incremental Dentine Sections**

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In human permanent teeth dentine is secreted and fully mineralized in approximately 3-8 days (Dean & Scandrett, 1995): because the age at which teeth develop is well-established (Hillson, 1996), high temporal resolution can be achieved when constructing isotopic profiles from dentine. The use of stable isotope ratios obtained from dentine to investigate diet has been used in a number of studies (e.g. Müldner et al., 2009). High-resolution dentine sampling offers the opportunity to investigate childhood dietary variation in individuals who survived childhood thus avoiding the need to consider pathological changes in the period immediately prior to death, i.e. the “osteological paradox” (Wood et al., 1992). Comparison of the isotopic profile of individuals who survived childhood can then be made with those who did not, to investigate dietary and metabolic differences over the same age range.

Carbon and nitrogen isotopic profiles were produced using incremental dentine collagen from individuals where historical, archaeological and documentary evidence suggests significant dietary variation or deprivation. These include individuals from cemeteries dating from the period of the Great Irish Famine (1845-1852) in London and Kilkenny, and marginal environments in prehistoric Scotland. For permanent teeth, this yielded approximately 10-20 increments, each representing an average of 9 months. Both deciduous and permanent teeth from sites in the British Isles ranging from prehistoric to 19<sup>th</sup> century were investigated. For some individuals, two or more teeth were examined, extending the period of life studied. Results are presented and compared to evaluate the potential information contained within dentine, both for dietary studies and for investigating the effect of stress on the individual. The profiles reveal variations in the isotope ratios that are consistent with expected dietary variation, however, they also suggest evidence for physiological variation in the nitrogen isotopes. The differences between individuals who died in childhood and those who survived, may allow interpretation of the state of health of individuals in these early years.

DEAN, M.C. AND SCANDRETT, A.E., 1995. Rates of Dentine Mineralization in Permanent Human Teeth. *International Journal of Osteoarchaeology* **5**, 349-358.

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MÜLDNER, G., MONTGOMERY, J., COOK, G., ELLAM, R., GLEDHILL, A. AND LOWE, C., 2009. Isotopes and individuals: diet and mobility among the medieval Bishops of Whithorn. *Antiquity* **83**, 1119-1133.

WOOD, J.W., MILNER, G.R., HARPENDING, H.C. AND WEISS, K.M., 1992. The Osteological Paradox: Problems of Inferring Prehistoric Health from Skeletal Samples. *Current Anthropology* **33**, 343-358.

**B4 A Reconstruction of the Living Space in a Community of Early Bronze Age in Sicily: Results of a Multidisciplinary Study**

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The main aim of this paper is to trace the use and the organization of space in Early Bronze Age Sicilian (2200-1450 BCE) societies. The purpose is to identify the connection among types of food consumed in relationship to vessel shape, in addition to characterising indicators of food preparation and the space in which these activities take place. This study incorporates both organic chemistry and GIS spatial analyses in order to understand vessel use in direct correlation to food preparation space in a settlement context.

The hill site of Santa Febronia, contains the remains of an Early Bronze Age hut that was destroyed by a fire leaving a sealed deposit with a large quantity of artefacts in their original position. The deposition creates an ideal scenario for GIS spatial analysis of the artefact distribution within the structure in contrast to areas of storage. Chemical residue analysis was carried out with GC-MS to extract and identify absorbed lipids within ceramic vessels (cf. Eerkens, 2005). The gas chromatographic results are used to establish relations between the food processing and the depositional space. The study reveals a multi-functionality of both space and pottery and further confirms the distinction of storage areas and in particular areas dedicated to water storage.

EERKENS, J.W., 2005. GC-MS. analysis and fatty acid ratios of archaeological potsherds from the western Great Basin of North America. *Archaeometry* **47**, pp. 83–102.

## **B5 Stable Isotope Analyses ( $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ ) of Degraded Bone Collagen and Hair/Wool Keratin by Using Cross Flow Nanofiltration as a Cleanup Step**

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Degradation and/or contamination with organic material of bone collagen and hair/wool keratin is demonstrated by measuring the C/N ratio. For bone that has not been significantly affected by diagenesis, the atomic C/N ratio should lie between 2.9 and 3.6 (De Niro, 1985) while the accepted atomic C/N ratio for hair/wool keratin is between 2.9 and 3.8 (O'Connell & Hedges, 1999). Ultrafiltration of bone collagen, dissolved as gelatin, is an effective method of removal of low-molecular weight contaminants from bone collagen but it does not remove high-molecular weight contaminants, such as cross-linked humic collagen complexes (Brock et al., 2007). However, comparative dating studies have raised the question whether this cleaning step itself may introduce contamination with carbon from the filters used (Bronk Ramsey et al., 2004; Brock et al., 2007; Hülts et al., 2007, 2009). In this study a cross flow nanofiltration method was developed using a ceramic filter as a means to avoid extraneous carbon contamination via the filter. This method should be applicable on various protein materials e.g. collagen, hair, silk, wool, leather and should be able to remove low-molecular and high molecular weight contaminants, depending on the choice of molecular weight cut off (MWCO) of the membrane. Here a filter membrane with a cutoff of 200 Dalton was used in order to collect the amino acids, released by hot acid hydrolysis of the protein material, in the permeate (MW of amino acids varies between 75.07 and 204.23 Dalton) and the contaminants (> 200 Dalton) in the retentate. Stable isotope analysis was done on the bulk material of well preserved and degraded archaeological bone collagen and hair/wool keratin, pre-treated with the conventional methods, and on the crossflow nanofiltrated amino acids. Quality assessment of the samples was performed by measuring the C/N ratio.

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**B6 Death During the Scottish Wars of Independence: An Osteological and Isotopic Analysis of Medieval Individuals from Stirling Castle, Scotland**

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The Scottish Wars of Independence have long ignited public interest, however to date little skeletal material relating to them has been excavated or analysed. In 1997 a small population radiocarbon dated to the 14<sup>th</sup> and 15<sup>th</sup> centuries was excavated in a lost royal chapel at Stirling Castle, one of the key garrisons of the wars. Shortly after the excavation skeletal analysis revealed that one individual suffered healed sharp force trauma to the frontal bone and a second had possible peri-mortem puncture wounds to the cranial vault. Our re-analysis has identified an abundance of previously unidentified peri-mortem trauma within this small group. Most of this is blunt force trauma, with small numbers of sharp force and penetrating trauma present. One individual suffered over 80 peri-mortem fractures, many to his post-cranial skeleton.

This paper will present the evidence of trauma from Stirling Castle, highlighting the diagnostic criteria for peri-mortem blunt force post-cranial injuries, which are rarely reported on in palaeopathology. While many of the fractures found in isolation could easily be attributed to accidents, a small number of sharp-force injuries clearly relate to inter-personal violence. It is argued that this, alongside the significant burial location within a royal castle, indicates that these are high-status individuals who died in encounters relating to the Scottish Wars of Independence.

Carbon and nitrogen isotope analysis of bone collagen indicated these individual enjoyed a mixed diet, with ratios similar to other notable high-status medieval populations. Oxygen and strontium isotope analysis of tooth enamel was undertaken to investigate the origins of all individuals with surviving teeth. Stirling Castle changed hands repeatedly during the course of the wars of independence, so one of our main aims was to establish if these people were English or Scots.

## **B7 Ancient Maritime Pitch and Tar: A Multi-Disciplinary Study of Sources, Technology and Preservation**

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Tars and pitches are black sticky substances manufactured by destructive distillation of wood and bark from soft and hardwood trees such as pine, spruce and birch. Tar is the initial liquid pyrolysate, while further distillation produces more viscous pitch, which is solid at ambient temperatures and must be re-heated prior to use (Evershed et al., 1985; Beck et al., 1994). These materials have a long history of use as waterproofing agents and timber preservatives, especially in maritime contexts: they have been used by shipbuilders and seafarers to caulk the seams of vessels and to waterproof rope and tarpaulins (Langenheim, 2003). In the medieval period, their role in ship building and maintenance led them to acquire crucial strategic and political importance for the developing European seafaring economies and naval fleets.

Material from the Newport Ship discovered in 2002 on the right bank of the river Usk in Newport, Wales is central to this study. The wreck constitutes the most complete example of a fifteenth century clinker-built vessel ever found in the UK. The dating evidence (from dendrochronological analysis and coinage) indicates that it was constructed after AD1445 and came to rest in Newport soon after AD1468, while associated finds suggest contacts with the Iberian Peninsula (Jones, 2005, 2009). The ship predates the carvel-built Mary Rose by some 50 years and represents a late survival of northern European shipbuilding traditions (Evershed et al., 1985). A systematic study to map the use of tars and pitches over the whole vessel is currently underway to further the understanding of the construction and later repair of the ship and also to examine the economics behind the use of these substances in the shipyards of medieval Europe.

Interpretation of the Newport material is supported by the analysis of comparative material from further archaeological sites (the Roskilde wrecks, Denmark; St Peter Port wrecks, Guernsey; Doel cog, Belgium) with different preservation conditions enabling the impact of both intra- and inter-site variability in burial environment to be compared.

GC/MS analysis of the Newport tars has shown them to be conifer-derived but of variable composition and different in some ways to tars reported from other maritime contexts (Robinson et al., 1987; Colombini et al., 2003; Connan & Nissenbaum, 2003). Light stable isotope analysis ( $\delta D$  and  $\delta^{13}C$ ) have also been undertaken in order to refine origin of these tars. The first results of this study will be reported here.

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**B8 Archaeobotanical Study of Ancient Food and Cereal Remains at the Astana Cemetery, Xinjiang, China**

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Analysis of starch grain, phytolith and cereal bran fragments were undertaken to identify the food remains including cakes, dumpings, as well as porridge unearthed from the Astana Cemetery in Turpan of Xinjiang, China. The results suggested that cakes were made from *Triticum aestivum* while dumpings were from *Triticum aestivum*, along with *Setaria italica*. The ingredients of the porridge remains came from *Panicum miliaceum*. Moreover, we present direct macrobotanical evidence of the utilization of six cereal crops such as *Triticum aestivum*, *Hordeum vulgare* var. *nudum*, *Panicum miliaceum*, *Setaria italica*, *Cannabis sativa* and *Oryza sativa* in the Turpan region during the Jin and Tang dynasty (about 200-900AD). All of these cereal crops not only provided food for the survival of the indigenous people, but also spiced up their daily life.

## **B9 What Did they Eat in that Dining Hall? Evidence from Faunal Remains and Material Culture at Late Antique Sagalassos (SW-Turkey)**

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Ongoing excavations of the Roman imperial Baths at the archaeological site of Sagalassos (SW Turkey) have revealed the modification of a large-scale *frigidarium* annex *apodyteria* into a dining hall in late Roman times. Along the south façade of the building, as well as in specific locations inside, a series of stratigraphical deposits was identified containing considerable amounts of food waste materials (animal remains) associated with concentrations of tableware for food and beverage consumption. These dumps illustrate the relative chronology of the use of the dining hall.

The archaeozoological analysis revealed that the faunal assemblages of these dumps largely consisted of sheep and goat remains, while pig and cattle remains were less numerous. Further, the skeletal element distribution of the goats and the sheep was very peculiar: the majority of the identified bones could be attributed to the hind leg, more precisely the meat bearing parts (pelvis, femur and tibia). All other skeletal elements, such as cranial fragments, vertebrae, ribs and elements of the fore limb were heavily under-represented; canon bones and phalanges were also mostly missing. In addition to this body part selection, it appeared that the hip joint (proximal femur) and the knee joint (distal femur, patella and proximal tibia) were also almost completely absent. Therefore, the faunal material evidenced that mainly special cuts of goat and sheep meat were served in the late Roman dining hall of the Roman Baths.

The study of the material culture from the relevant layers provided the chronological framework for these practices. Moreover, these collections represent a rare archaeological opportunity to document communal dining practices in action. The deposits are considered to have formed as a direct result of the festivities, representing not only a potential to reconstruct functional tableware sets, but also to extract information on social practices of the local community when wining and dining together. Apart from patrician diner settings, communal dining traditions represented the socio-cultural framework of eating habits in late antique society.

## **B10 Palaeodietary Reconstruction in the Human Remains Recovered from Roopkund Lake through Elemental Analysis and Estimation of Carbon and Nitrogen Isotope Ratios**

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Present study is carried out in the bone samples collected from Roopkund Lake in district Chamoli Garhwal, Uttarakhand which is located at 5029 meters from main sea level in between Nanda Ghunghti and Trishuli peak. This historical site belongs to 9<sup>th</sup> century A.D. All the samples selected for the study were dried in room temperature as well as hot air oven at 32 degree Celsius. Cleaning, pretreatment and digestion process of faunal remains was followed through established scientific methods. Chemical analysis i.e. concentration of different elements such as calcium, strontium, barium, magnesium and zinc as well as isotopic ratios of Carbon and Nitrogen was estimated with the help of Inductively Coupled Plasma Spectroscopy (ICP) and Atomic Absorption Spectrophotometer (AAS).

The results obtained from the chemical analysis are significant. On the basis of concentration of different elements and ratios of Nitrogen and Carbon isotopes, the dietary habits of the peoples buried in the Roopkund Lake are identified, which is differing from sample to sample person to person. Besides this, the results are also significantly helpful for knowing the preservation status of faunal remains in Roopkund Lake. Finally this study also indicated the potentiality of chemical analysis for reconstructing the palaeodiet behaviour and preservation status of bone remains.

## **B11 EDTA-Solution Based Protocols for the Cleaning of Ancient Bone Bioapatite**

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Isotopic signals obtained from ancient bone material are of great importance in archaeological research, providing information on chronology, dietary habits, and mobility. Typically, radiocarbon dating and stable isotope analysis is performed on bone collagen. The inorganic phase of bone, bioapatite, is often not considered for analysis as the isotopic signal may be influenced by the deposition of foreign minerals, while bioapatite itself may undergo recrystallization and ionic exchange (Hedges, 2002). Bone cleaning protocols using a weak acid, normally acetic acid, are capable of removing foreign calcite deposits, which are more soluble than bioapatite. However, acetic acid is incapable of selectively leaching less soluble mineral deposits such as fluorapatite. The complexing agent ethylenediaminetetraacetic acid (EDTA) provides an alternative to acetic acid. The fibrillar structure of collagen (Nudelman et al., 2010) ensures the relative protection of bioapatite crystallites in a basic EDTA solution. Furthermore, in an EDTA solution the dissolution rates of geologically deposited minerals, including fluorapatite, are much higher than that of bioapatite. The present study focusses only on bone material containing a significant amount of collagen, as recent research results have demonstrated, at the osteon level, the association between collagen and bioapatite preservation (Reiche et al., 2010). Different EDTA protocols have been tested on powdered bone material. These include, variations in solvent, working pH, dissolution times and temperature. Comparison of EDTA and acetic acid based protocols was done using attenuated reflectance fourier transform infrared spectroscopy (ATR-FTIR). Study of the infra red spectra clearly demonstrates that EDTA is less aggressive towards bone's collagen. The infra red splitting factor (IRSF), a crystallinity parameter calculated from the phosphate peaks in the spectrum, is commonly used to assess the degree of crystal growth and recrystallization (Weiner & Bar-Josef, 1990). Acetic acid treatments resulted in higher IRSF values, denoting incapability in leaching less soluble foreign mineral deposits. In contrast, bone powder treated using an EDTA solution presented lower IRSF values, close to those observed in fresh bone material. This study will also present the first preliminary radiocarbon dates on bone bioapatite using an EDTA protocol. Bone material, containing collagen, from a variety of time periods and locations was selected for comparative dating of collagen and EDTA-treated bioapatite.

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## B12 Influence of Cooking on the Isotopic Signals of Fish Species

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Stable isotope analysis represents the principal scientific technique used in the reconstruction of ancient human diet. Characterization of human diet, based on stable isotopes, requires that the isotopic baseline, that is the isotopic signals of the consumed food groups, is established. Ideally, the isotopic baseline for mammal or fish species, consumed by humans, is based on isotopic data from bones of these species found in association with the human remains.

Different animal tissues will present varying isotopic signals (del Rio et al., 2009). The isotopic signal of fish meat will depend on its nutrient (carbohydrates, lipids, protein) and biochemical composition (e.g. amino acids or fatty acids). Fish, a major protein source, may also have high lipid content, with lipids having a significantly depleted  $\delta^{13}\text{C}$  signal when compared with protein (Post et al., 2007).

Cooking may alter the isotopic signal of meat (Warinner & Tuross, 2009), not only through fractionation processes, but also through selective loss of macronutrients or biochemical components possessing different isotopic signals.

We investigated the influence of cooking on the stable isotope values of meat for two fish species (mackerel, which has a very high fat content, and haddock, which is low in fat) using three potential prehistoric cooking methods. The fish were boiled in a pot, grilled beside an open fire, or wrapped in leaves and clay and baked in hot sand. Cooking times and temperatures were monitored, and the cooked fish was tasted for palatability.

Stable isotope ratios ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) were measured on the meat and bone collagen of raw fish and compared with measurements obtained after cooking using the three different methods. Results will be presented together with a discussion of possible implications for the reconstruction of ancient human diet.

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### **B13 Paleogenetic Evidence from Dental Calculus: A New Approach to Archaeological Populations**

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Both the diversity and evolution of human populations have been systematically analyzed through polymorphisms in the human genome and microbiome, with several microorganisms successfully analyzed to infer microevolutionary events of their host. The aim of the present investigation is to contribute to the study of human bacterial flora as indirect genetic markers of their host by evaluating a new source of genetic material: dental calculus. Electron microscopy studies have shown the presence of bacteria preserved in samples from different geographical and chronological context, stimulating the evaluation of this material from a genetic approach. Recent and archaeological DNA samples, some of them as old as 4,000 YBP, were successfully extracted and amplified following all the necessary control procedures for ancient DNA. Previous to the DNA extraction, the samples were treated with sodium hypochlorite to eliminate surface contaminant. The demineralization was performed with 0.5 M EDTA and the proteins digested in a buffer with proteinase-K. Protein and cell components were eliminated by phenol:chloroform:isoamyl alcohol (25:24:1) extractions and the DNA was finally precipitated with isopropanol. Species-specific PCR primers were designed to identify five bacterial species (*Actinomyces naeslundii*, *Fusobacterium nucleatum*, *Streptococcus gordonii*, *Streptococcus mutans* and *Porphyromonas gingivalis*). *F. nucleatum* was the most frequently observed species in all of the recent samples and in almost 40 percent of the ancient samples. In *F. nucleatum* we also observed the presence of two or more genetic variants from a single human host, which were identified by DNA cloning and sequencing. Given that the highest presence and variability was detected in *F. nucleatum*, this species emerged as a valuable target for future analysis.

In summary, genomic analysis of bacteria from dental calculus is a promising source of evidence for paleopathological and microevolutionary studies, focused either on microorganisms or on their human hosts.

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## **B14 Oral Microbiome Evolution in Prehistoric Chilean Populations Using Scanning Electronic Microscopy and Isotopic Analyses**

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The human oral microbiome is composed by a myriad of bacteria species, representing an enigmatic ecological community. The oral microbiome emerges, consequently, as a valuable model to inference of diet, human populational genetic structure, oral health features, and microevolutionary processes, among other factors associated to this ecological system. In this study we explored the association among geographical distribution, paleodiet and bacterial communities in human populations from different archaeological contexts in Chile: Solcor 3 (San Pedro de Atacama, Northern Chile); Cuchipuy (Central Chile); Chonos (Southern Chile), in addition with the analysis of samples from living population. The main goal of this study is to evaluate the relative frequencies of bacterial morphotypes as predictors of diet and geographical distribution of the prehistoric populations. We tested the relative frequency of three bacterial morphotypes, using scanning electronic microscopy (SEM). Dietary regimes were inferred from archaeological evidence and from isotopic analysis ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) performed in this study, as well as from previous works. Differentiation in the oral microbiome was detected at the inter-populational level for filamentous bacteria, which are most frequently found in San Pedro de Atacama samples. The causal explanation of this differentiation is still unresolved. Loss of differentiation of other morphotypes in relation to dietary or geographical distribution, including populational genetic structure, refutes our hypotheses, opening new insights on microbiome evolution in past human populations: taxonomic units (species), instead of morphotypes, should be the target of evolutionary, ontogenetic and ecological processes affecting oral microbiome, as a consequence of changes in cultural practices and genetic differences among past human populations. Grants support: Fondecyt 1100643, Fondecyt 1110461, Anillo ACT-096.

## **B15 Bees Wax and Propolis as Sealant of Funerary Chambers during the Middle Bronze Age in South-Western Iberian Peninsula**

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Recent archaeological excavations in the Portuguese region of Baixo Alentejo, carried out under the implementation of the irrigation project connected with the Alqueva Dam, have brought to light important finds dated to the South-western Iberian Bronze Age. Among those new finds are funerary hypogea, dated from the second quarter of the 2<sup>nd</sup> Millennium BC, which show some similarities with those from the Argaric Culture (South-eastern Iberian Peninsula), namely as far as rituals, architecture and grave goods are concerned. Each funerary structure is composed by an atrium connected with a chamber (an artificial cave cut into the rock) closed by vertical slabs. These slabs are involved by a black earth with a greasy aspect, suggesting an organic origin. It was already known that some stone cists, another kind of grave in use during the Middle Bronze Age in South-western Iberia, also present a similar black greasy earth covering the cist stone lid (Paço & Leal, 1962/63). Earth of one of these graves was sampled and analyzed using infrared spectroscopy and gas chromatography. IR analysis indicated the presence of organic substances being the main constituent a fatty material of animal origin. The identification of cholesterol also proves that origin. The chromatograms obtained were compared with those of well known samples, indicating that probably the fat detected in the earth is that of swine (Ribeiro & Soares, 1991). The black earth with a greasy aspect that involved the slabs closing the chamber of three funerary hypogea from the archaeological sites of Horta do Folgão and Torre Velha 3, both near Serpa, were sampled and analyzed using a FTIR spectrometer. The spectra obtained were compared with reference spectra. Two materials were identified: bees wax and propolis. As far as we know is the first time that a mixture of bees wax and propolis with a clayey earth used to seal a funerary structure was identified in the archaeological record, suggesting the importance that the Middle Bronze Age people in South-western Iberia put in the preservation of the corpses buried in their graves.

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## **B16 Identification of Heme in Aged Blood by Direct Analysis in Real Time Mass Spectrometry**

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Identification of blood on or in archaeological materials is often done using presumptive tests adapted from forensic applications. Clinical test strips, designed for the detection of blood in urine, are arguably the most utilized of such presumptive tests in archaeological science. In forensic science, however, presumptive tests must be followed up with confirmatory tests; presumptive tests suffer from high rates of false positives due to the presence of contaminants. Confirmation that a stain or residue is blood generally requires biological methods, such as DNA or immunological tests. We report here a novel method for identification of blood through mass spectrometric analysis of the heme moiety from the blood and muscle proteins hemoglobin and myoglobin.

Direct analysis in real time (DART) ionization combined with high-resolution time of flight mass spectrometry provides exact mass information about molecules up to approximately 1000 Daltons. While DART is not applicable directly to intact proteins, a novel sample preparation method developed in our laboratory yields a molecular ion for the permethylated heme porphyrin at  $m/z$  644.2085. Blood samples applied to surfaces and weathered for multiple years produced reliable heme signal. Control materials without heme produced no heme signal. Further studies are currently underway to determine the applicability of this method to blood on lithic materials and we are optimizing collection of residue samples from archaeological materials.

# **B17 Stable Isotope Ratio Analysis Elucidates Urban Subsistence and Changing Animal Husbandry Practices at Ancient Sagalassos (SW Turkey)**

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The present study is based on the analysis of the stable isotope ratios of carbon and nitrogen in both human (n=49) and animal (n=465) bone samples. These samples were obtained from the site of Sagalassos (Burdur Province, SW Turkey) and Düzen Tepesi (at 2km SW of Sagalassos) and are ranging in date from the Classical-Hellenistic period (400-200 BC) up to the Middle Byzantine period (800-1200 AD). Therefore, they witness a long span of occupation at Sagalassos and its immediate surroundings.

The aim of the research is to gather information on human dietary patterns and livestock management practices (such as herding, grazing and foddering) in the past within the territory of Sagalassos. While the human diet seems to be based on predominantly C3 plants and terrestrial (domestic) animals, the data of the animal bones samples show shifting  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values.

Diachronic comparisons of the stable isotopes ratios were carried out for the main domestic mammals, i.e. dog, cattle, pig, sheep and goat, and show how different grazing areas were used through time. In addition, the consumption of C4-plants by cattle is highlighted. These results are discussed in the context of previous interdisciplinary research (including archaeozoological, archaeobotanical and geochemical analyses) to elucidate the urban subsistence and the changing animal husbandry practices at Sagalassos.

**B18 Stable Isotope Studies on Humans and Animals from Tell Tweini, Syria (2600-550 BC)**

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Tell Tweini is a coastal Syrian site with settlement remains of diverse periods between the Early Bronze Age and the Iron Age (2600-550 BC). Inside urban contexts in Field A at the site, eleven burials have been unearthed. Most date to the Middle Bronze Age (2000-1600 BC), including one collective grave. Stable isotope ratio analysis ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) was carried out on human remains from these Middle Bronze Age graves in order to reconstruct human dietary practices. In addition, a large sample of faunal remains from the major periods of occupation at Tell Tweini, Early Bronze Age (2600-2000 BC), Middle Bronze Age (2000-1600 BC), Late Bronze Age (1600-1200 BC) and Iron Age (1200-550 BC) have been subjected to stable isotope ratio analysis. The large dataset on animals is the first of its kind for the period and region. The results add to a reconstruction of the human diet. More importantly, they allow us to make diachronic inferences on livestock management practices and the natural environment. The results will be discussed in the context of data on the faunal composition through time at Tell Tweini as well as that of the palaeo-environmental data which points to an abrupt climatic change at the transition between the Late Bronze Age and the Iron Age.

**B19 Microstructural (SEM), Chemical and Molecular-Biological Investigation of Glass Decay at the Medieval Stained Window Glasses of Tarragona Cathedral and Santa Maria del Mar Church (Barcelona), NE Spain**

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The medieval stained window glass of two religious medieval buildings (the Cathedral of Tarragona and the Church of Santa Maria del Mar in Barcelona), have been investigated and provided evidence of biodeterioration (Garcia-Vallès & Vendrell, 2002; García-Vallès et al., 2003). These biodeterioration was active under Mediterranean climate and affected more selectively potash-lime glass of inferred foreign origin than the local sodium glass of mediterranean tradition (Aulinas et al., 2009). Biological activity in both sites showed to be fossil and only remains of this activity, as orange patinas, bio-pitting and mineral precipitation, was observed. Glass surfaces were investigated by Scanning Electron Microscopy (SEM), Energy Dispersive Spectrometry (EDS) and X-Ray Diffraction (XRD). Their chemical composition was investigated using wavelength-dispersive spectrometry (WDS) microprobe analysis. The microbial diversity was investigated by molecular methods: DNA extraction from glass samples, amplification by PCR targeting the 16S rRNA and ITS regions, and fingerprint analyses by denaturing gradient gel electrophoresis (DGGE). In parallel clone libraries containing either PCR fragments of the bacterial 16S rDNA or the fungal ITS1 region were screened by DGGE. Clone inserts were sequenced and compared with sequences listed in the EMBL database. Similarity values ranged from 89 % to 100 % to known bacteria and fungi. Analyses revealed complex bacterial communities consisting of members of the phyla Proteobacteria, Bacteroidetes, Firmicutes and the dominance of Actinobacteria. Fungi showed lower diversity than bacteria and species of the genus *Cladosporium* were dominant. The detected Actinobacteria and fungi may be responsible for the observed bio-pitting phenomenon. Moreover, some of the detected bacteria are known for their capabilities of mineral precipitation. Sequence results also showed similarities with bacteria commonly found on deteriorated stone-monuments, what supports the idea that medieval stained glass biodeterioration in the Mediterranean area shows pattern comparable to those developed on stone.

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## **B20 Human Mobility and Diet in Iron Age The Netherlands**

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In The Netherlands cremation is the most general practice for the burial of the dead from the Late Bronze Age (1100 BC) up to the 3<sup>rd</sup>-4<sup>th</sup> century AD. However, in recent years dozens of Iron Age inhumation graves were discovered amidst cremation graves in the Dutch Lower Rhine area. In this specific area inhumation of the dead seems to be a more common practice than elsewhere in The Netherlands. Until now, the presence of these Iron Age inhumations has had two different explanations: 1) the arrival of immigrants from the Middle-Rhine area (Early Iron Age) and 2) a broader cultural influence spread from the northern France Marne-Aisne area (Middle Iron Age). Evidence of exchange and trading has already been identified from the material culture. However, isotopic evidence for human mobility has not been assessed.

This project investigates whether these buried individuals were of local or non-local descent. In addition, carbon and nitrogen isotope analyses were conducted to investigate palaeodiet. Strontium isotope analyses are reported on 26 Iron Age individuals, mostly from burial sites in the Dutch river area. Preliminary results show that migration of people has taken place. Further research will try to identify possible areas of origin. The results of the isotopic analyses will be compared with archaeological data and interpretations from the grave-sites and with relevant previous Iron Age studies concerning subsistence economy and diet. The outcome of this research gives additional insight into the burial culture and quantifies the role of mobility within these Iron Age populations.

## **B21 A Dietary Point of View of Neolithic-Bronze Age Transition: Stable Isotope Analysis of Central France Populations**

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This paper presents the study of diachronic dietary patterns of Prehistoric populations from Central France. Stable isotope measurement ( $\delta^{13}\text{C}$ ,  $\delta^{15}\text{N}$ ) on bone collagen is a relevant tool to understand, at individual and group levels, the environment from which dietary resources come from and the importance or not of animal protein intake. Following a research programmes started few years ago on West-Mediterranean populations (e.g. Goude, 2007; Vaquer et al., 2008), new stable isotope analysis were performed on Middle Neolithic and Early Bronze age (EBA) human groups located in Auvergne region (Research funded by Nestlé France Foundation; 2009-2011). Added to human, faunal remains were also analysed in order to define the environmental isotopic baseline for each period concerned. Auvergne region has recently seen development of bio-anthropological works thanks to several excavations, highlighting numerous tombs and well preserved osteological material. Combined to stable isotope analysis, environmental and archaeological data, anthropological features (e.g. sex, age at death, stature) allow understanding food choices within social and cultural aspects. Both adults (n=74) and subadults (n=45) from 4 archaeological sites, close from each other, gave well preserved collagen.  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values of animal indicate a possible specific feeding between EBA bovine and caprine, and a distinction between the two period, specifically for  $\delta^{13}\text{C}$ . This  $\delta^{13}\text{C}$  shift is also recorded between humans with highest values among EBA individuals. No  $\delta^{15}\text{N}$  difference is highlighting between human groups; dietary resources, dominated by herbivores meat, and probably including aquatic fish for some individuals, seems to be equal whatever the period. These results could support a local environmental  $\delta^{13}\text{C}$  modification between these periods, with more open fields (deforestation?) at the EBA.

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VAQUER, J., GANDELIN, M., REMICOURT, M. AND TCHEREMISSINOFF, Y. (Eds.), 2008. Défunts néolithiques en Toulousain. *Archives d'Ecologie Préhistorique*, 228 p.

## B22 Phosphate Extraction from Enamel and Cementum: A Revised Method

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The isolation of phosphate from biological and inorganic materials for the purposes of oxygen isotopic analysis has been employed in a variety of archaeological studies (e.g. Evans et al., 2006; Kolodny et al., 1983). Migration, dietary and paleoclimate investigations employing oxygen isotope analysis have benefited greatly from the isolation of phosphate, which is argued to be resistant to diagenetic processes (Wiedemann-Bidlack et al., 2008). Advancements in extraction techniques have been significant, primarily with the transition from isolating  $\text{PO}_4$  as  $\text{BiPO}_4$  to isolating it as the much more stable and non-hygroscopic  $\text{Ag}_3\text{PO}_4$  (Firsching, 1961). Although methods have been developed for use on calcified biological materials (e.g. Crowson et al., 1991) and inorganic phosphate minerals (Vennemann et al., 2002), there is no agreement on the best technique. The extraction of purified  $\text{Ag}_3\text{PO}_4$  from sub-milligram size samples of organic-rich material was recently published by Wiedemann-Bidlack *et al.* (2008) where they obtained biogenic oxygen isotopic compositions from <1mg samples. Here we report on a simplified method for chemical precipitation of  $\text{Ag}_3\text{PO}_4$  from small (5mg) samples of enamel and cementum. The isolation of phosphate from cementum is an avenue for accessing late-life oxygen isotopic compositions, and expands on previous studies, which primarily focus on enamel and dentine. Our method has been tested on modern cows (*B.p. taurus*), and pre- and post- purified sample collection is being optimized for application on archaeological human teeth. Analysis of the resultant  $\text{Ag}_3\text{PO}_4$  is ongoing and will be employed to create a standard for comparison between *in situ* micro-analytical oxygen isotope results obtained by Secondary Ion Mass Spectrometry (SIMS) and bulk analytical results obtained by Thermal-Combustion Elemental Analyzer (TC-EA)-gas source mass spectrometry for each tissue. Ultimately, this comparison should facilitate the comparison of enamel to cementum using the less destructive SIMS micro-analytical technique.

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## **B23 Potential of Direct Analysis in Real Time Mass Spectrometry for Rapid Characterization of Organic Residues on Ceramics**

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GC-MS analysis of ceramic residues is a time consuming and expensive process that has the potential to yield important information about ancient diet and materials utilization. With the widespread availability of GC-MS instrumentation, more extractions and analyses are being carried out on large collections of ceramic sherds, often yielding results of questionable utility. A screening method for selecting only the most promising samples for rigorous analysis would save both time and money.

We report here on a preliminary study utilizing direct analysis in real time mass spectrometry (DART-MS) to identify specific biomarkers on ceramics. DART-MS is an ambient ionization method widely used for identification of small (<1000 Da) molecules on surfaces without sample preparation. Food materials – garum, olive oil, wine, chocolate, chili pepper, and corn-based beer – were applied to ceramic surfaces, dried, and divided. Half of the ceramic was buried for up to six months. Samples were analyzed using DART-MS to look for biomarkers of each material, specifically pyroglutamic acid, fatty acids and diglycerides, tartaric acid, theobromine, capsaicin, and maltol.

Sample handling was critical for the characterization of surface residues. Squalene from fingerprints and erucamide slip agent from plastic bags were the most common materials observed in the DART spectra, even when care was taken to wear gloves during processing and use foil for packaging. In some cases, burial and subsequent cleaning obscured or obliterated the surface residues. Other samples, especially the olive oil, were resistant to water-based cleaning, and were readily identified even in the presence of soil. Future work will involve authentic ceramics from archaeological contexts in Central and South America and ancient Rome.



## **B24 Isotope Geochemistry in Dutch Archaeology. The Application of Strontium Isotopes as a Proxy for Migration**

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Migration has been subject to research and debate for many decades and has become a key component of archaeological thinking. The original concepts of waves of migration in archaeology were based upon the dispersal of cultural artefacts. This approach led to an active debate about the extent to which the archaeological record represents the actual movement of people or the diffusion of ideas. A new perspective on this debate is provided by the discipline of archaeological science. In addition to aDNA studies, the application of isotope ratios, in particular those of strontium, of mineralized tissue (bone, dentine (ivory) and enamel) is used to study migration, specifically at the individual level. Isotope research in archaeology has matured over the last three decades, proved its potential in numerous studies, and is nowadays one of the most innovative research fields in archaeological science.

Despite its international success and proven potential, isotope research has rarely been applied in Dutch archaeology. The major contributing cause is the absence of a bioavailable strontium isotope distribution map of The Netherlands, which is a fundamental component for data interpretation. This PhD project therefore focuses on the collection of data to create this isotope distribution map. Based on this map, the spatial variation in bioavailable  $^{87}\text{Sr}/^{86}\text{Sr}$  in The Netherlands will be evaluated and the applicability of isotope geochemistry as a proxy for interregional mobility will be assessed. The data and the bioavailable strontium distribution map ultimately will lead to more insight into the cultural diversity of ancient populations throughout The Netherlands.

## **B25 Herculaneum Conservation Project: Characterisation of Archaeological Waterlogged Wood from Ercolano Site by Pyrolytic and Mass Spectrometric Techniques**

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The Roman city of Herculaneum, Campania, Italy, in the shadow of Mt. Vesuvius was discovered accidentally in 1708 by a farmer digging a well. Herculaneum was a smaller town with a wealthier population than Pompeii at the time of the eruption. Initial excavations revealed only a few skeletons; it was long thought that nearly all of the inhabitants had managed to escape. It was not until 1982, when the excavations reached boat houses on the beach area, that this view changed. In the suburban area, archaeologists discovered several hundred skeletons huddled close together on the beach and in 12 boat houses facing the sea. Further excavations in the 1990s confirmed that at least 300 people had taken refuge in those chambers, while the town was almost completely evacuated.

The Herculaneum Conservation Project (since 2000) was undertaken in the conviction that the extraordinary challenges of conservation of the Vesuvian sites are an international responsibility and cannot be shouldered by the national heritage authority alone. The project is the initiative of the Packard Humanities Institute (Los Altos, California, USA) and aspires to conservation and to create new archaeological knowledge of the site, and to extend the appreciation of the site among the professional communities and the general public.

Our aim in this project is the chemical characterization of waterlogged archaeological wood. Chemical characterization is crucial to assess the decay of archaeological or historical wood. Traditionally, wood analysis and determination of the content of lignin and holocellulose is performed by means of reagent- and time-consuming wet chemical methods, which require a consistent amount of sample. Recently, increasing attention is given to instrumental analysis based on analytical pyrolysis and mass spectrometry applied to the characterization of archaeological wood. The evaluation of the degradation state of the analyzed wood is based on the comparison with sound wood of the same species by Pyrolysis-Gas chromatography/Mass spectrometry (Py/GC/MS), an analytical approach that achieves semi-quantitative results on the content of lignin and polysaccharides in degraded wood, on syringyl vs. guaiacyl ratio, and on the chemical structure of lignin, avoiding the long wet-chemical procedures that are commonly used in wood analysis, and allowing us to use a minimal sample size.

Part of the analysed wood structures presented residues of the original paint in different colours (white, blue red or gold). Characterisation of the organic binders was also performed by GC/MS in order to reconstruct the painting technique.

## **B26 Subsistence Stability in the Syrian Coastal Area from 2600-550 BC Inferred by Archaeobotanical and Stable Isotope Evidence from Tell Tweini**

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The study focuses on the evidence of continuity and stability of the ancient subsistence in the period between Middle Bronze Age and Iron Age (2600-550 BC) in the Syrian coastal area. The main sources of evidence are the plant macrofossil assemblages, and stable isotope analyses of charred caryopses of cultivated cereals extracted from the cultural layers of Tell Tweini. The archaeobotanical assemblages are generally dominated by cultivated plants like wheat, pulses and especially fruits, namely grape and olive. The high amounts and abundance of grape and olive indicate clearly that they were of great importance for the plant economy of the site. Together with the remains of cultivated plants also a variety of wild growing plants, originating from wet environments, open habitats etc. was recorded. The stable carbon isotope analyses of cereal crops from the same assemblages show relatively little general variation of  $\delta^{13}\text{C}$  throughout time. There is however variation of  $\delta^{13}\text{C}$  within periods considering different crop species. Particularly during the Late Bronze Age  $^{13}\text{C}$  is strongly depleted in free-threshing wheat compared to barley and emmer. This could indicate different crop production technology, such as irrigation of free-threshing wheat or even import of free-threshing wheat from other regions.

The plant subsistence and its relation to environmental change of the studied area is also considered in regional context using the evidence from the archaeobotanical database of Eastern Mediterranean and Near Eastern sites (Riehl & Kümmel, 2005).

RIEHL, S. AND KÜMMEL, C., 2005. The archaeobotanical database for Eastern Mediterranean and Near Eastern sites: <http://www.cuminum.de/archaeobotany>

## **B27 Paleomicrobial Lipidomics: Mass Spectrometry-Based Discovery of Ancient Mycolic Acids**

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The *Mycobacterium* genus, which is hallmarked by mycolic acids, comprises several human pathogens such as *Mycobacterium leprae* and *Mycobacterium tuberculosis*. It is estimated that more than one-third of the worldwide human population is *Mycobacterium tuberculosis* infected, with nearly two million yearly deaths caused by infections. The mycobacterial cell-wall skeleton is consists of various long chain lipid, glycolipid, arabinogalactan and peptidoglycan structures. Mycolic acids (MAs) are major (40-60% of the cell dry weight) and unique fraction of the cell envelope of mycobacteria that include the ancient causative agents of tuberculosis and leprosy.

In the present study, we successfully analyzed the presence of free MA fraction and their derivatives by various analytical techniques as well as matrix-assisted laser desorption/ionization tandem time-of-flight (MALDI TOF/TOF), liquid chromatography coupled electrospray ionization mass spectrometry (LC ESI MS and nanoUPLC-nanoESI MS), thin layer chromatography (HPTLC) and MALDI LTQ Orbitrap imaging mass spectrometry (UV-MALDI IMS).

## **B28 Linking Milk Processing to Pottery Function in Prehistoric Anatolia: Diachronic and Regional Perspectives**

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The organic residues in Prehistoric Anatolian pottery sherds primarily from the Neolithic and Chalcolithic periods have been previously studied by Richard Evershed and colleagues (Evershed et al., 2008). Results of this groundbreaking work provided clear evidence for milking processing and dairying from 7<sup>th</sup> millennium BC sites in Northwestern Anatolia. Our previous study of Barcın Höyük pottery which is a Neolithic/Late Chalcolithic NW Anatolian site confirmed the results of Evershed. Building on this pioneering work, our application proposes to take the results to the level of pottery assemblages themselves. This has not yet been done for the region concerned. Correlation of milk processing and its products with specific vessel categories will be the first reliable step into assessing pottery functions and meaning in prehistoric assemblages. The extracted organic residues are mainly lipids that have been preserved in the porous matrix of potsherds. The detection and identification of the lipid components has been accomplished by using high temperature gas chromatography (HTGC) and gas chromatography mass spectrometry (GC/MS). (GC-IRMS) is used to determine the compound-specific stable carbon isotopes ( $\delta^{13}\text{C}$ ) of major n-alkanoic acids. Approximately 20 per cent of analyzed potsherds yield significant amounts of lipid residues. GC analysis yielded abundant distribution of saturated free fatty acids and minor amounts of di- and triacylglycerols indicating the presence of degraded animal fats. The GC-IRMS results of Barcın potsherds indicate that the majority of detected lipids originated from dairying fats with only few originating from ruminant adipose fats. The thin walled, mineral tempered ceramics from Neolithic Barcın Höyük is very suitable to sustain and regulate heat while cooking, and the processing of milk into curds, butter or yoghurt may well have been of crucial importance for the adoption of pottery in NW Anatolia. The first results of our project already indicate that it is foremost S-shaped globular cooking pots with flat bases and four vertically pierced knob handles placed on the belly diameters that give evidence of milk residues. Interestingly, also a small drinking cup is yielding unequivocal traces of milk residue.

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## **B29 Exploring Wine Production and Consumption in the Roman Villa of Sa Mesquida(Mallorca, Balearic Islands)**

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Sa Mesquida is a Roman villa occupied in the Early and Late Roman periods. The presence of wine Roman amphorae suggested an important trade of this product with other Roman provinces. The most common types are the Dressel 1 and the Pascual 1 amphorae which are supposed to be produced in Italy and in NE Spain respectively. In addition, the presence of a deposit for liquids opened the possibility of the existence of a domestic/local wine production.

In order to confirm the presence of wine in the amphorae and to determine the substance contained in the deposit and therefore to know its function a new methodology for the identification of wine residues with gas chromatography-mass spectrometry (GC-MS) has been applied.

The identification of wine markers in the amphorae type Dressel 1 type and in the plastered coating of the vat allowed demonstrating that wine was both imported and produced at the site.

Furthermore, to better understand the origin of these amphorae, a set of five Dressel 1, one Tarraconense 1, and six Pascual 1 pottery sherds were characterised by X-Ray Fluorescence, X-Ray Diffraction and optical microscopy by thin section analyses. The results were compared with the large analytical database on amphorae available at the ERAAUB. They reveal that Dressel 1 amphorae were produced in several pottery workshops located at the Tyrrhenian coast of Italy whereas the two other types came from several workshops placed at the central coast of Catalonia, in NE Spain.

In short, this contribution combines organic and inorganic analyses of pottery in order to understand wine production and consumption in a small rural Roman site yet well connected to the mainstream commercial dynamics of the Empire.

**B30 Morphometric Data on Bovine Remains (*Bos taurus* and *Bos primigenius*) Found in Chalcolithic Settlements from South-Eastern Romania**

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The results of osteometric analysis of the bovine remains from the archaeological sites from Romania are reported. These sites dated from Chalcolithic period (5000-3500 BC) and belong to the following cultures: Precucuteni and Cucuteni from east, Boian and Gumelnita from south and south-east of country. The linear measurements (variables) were defined according to von den Driesch (1976) for anatomical elements as: humerus, radius, metacarpus, metatarsus, tibia, astragalus, phalanges. Variables used in this analysis are based on breadth of bones especially. Our study focuses two aspects: sexual dimorphism and body size in populations while trying a demarcation of the wild of domestic forms; the distribution of measurements tends to be bimodal, reflecting the existence of two distinct populations: one domestic and the other wild. The sizes of bovines from different sites are compared by histograms using logarithm size index and coefficient of variation. Although it is difficult to clearly assign individual specimens as wild or domestic bovines, the general patterns of distribution of measurements suggest that bovines from assemblages of Precucuteni and Cucuteni cultures are more robust than those from Gumelnita culture (e.g., higher values for Bd and Bp recorded on metacarpus). An insignificant difference was revealed between cattle from Precucuteni-Cucuteni cultural complex and Boian culture.

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VON DEN DRIESCH, A., 1976. A guide to the measurement of animal bones from archaeological sites. *Peabody Museum Bulletin* 1. Peabody Museum of Archaeology and Ethnology, Harvard University, Cambridge.

### **B31 Dietary Differences between two Postmedieval Nunnery Sites from the Southern Low Countries: An Investigation Using ( $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ ) Stable Isotope Ratio Analysis**

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The aim of this project is to examine past diet diversity by comparing stable isotope ratio data ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ), measured from human bone collagen. The skeletons that were studied (N=69) are derived from the cemeteries of two synchronous, postmedieval (16<sup>th</sup> to 18<sup>th</sup> century) female monastic sites that were excavated in the southern part of the Low Countries in present-day Belgium. The sites represent religious communities belonging to different orders, inhabiting different environments (i.e. urban vs. rural).

The first site investigated is the Poor Clare nunnery, located in the centre of the former and present town of Brussels. The second is the Cistercian nunnery of Herkenrode, located in the countryside, in the province of Limburg, Belgium. Both sites were built and developed by contemplative orders. As known from historical sources, the Poor Clares at Brussels followed a rule of absolute poverty. They could not become, or remain, wealthy, or possess property, and were living from alms given by the local people. The almost opposite situation is true for the Cistercian nuns from Herkenrode, who, based on historical and archaeological sources, benefited from a rich lifestyle, possessing ample land and livestock.

Based on the results of the bio-archaeological study of the human remains, clear differentiations in demographic (age-at-death) structure, appearance and frequency of palaeopathological lesions were already noticed between the two sites. Possible inter-population dietary diversity will be further evaluated by comparing the ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) stable isotope ratio data from both sites.



**B32 Dietary Patterns in the Mixed Lay and Monastic Population from the Postmedieval Carmelite Friary Burial Grounds at Aalst (Flanders, Belgium), and their Relationship with DISH**

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Although studies on diet reconstruction using carbon and nitrogen stable isotope signatures ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) are numerous, little research is done on Belgian archaeological populations. The focus of this study is twofold. The first goal is to examine diet composition for a postmedieval mixed lay and monastic population, evaluating possible intra-population diversity by comparing carbon and nitrogen ( $^{13}\text{C}$  and  $^{15}\text{N}$ ) stable isotope data from different social, sex and age groups. The second goal of this research is the  $^{13}\text{C}$  and  $^{15}\text{N}$  stable isotope analysis of skeletons that display *Diffuse Idiopathic Skeletal Hyperostosis* (DISH), a condition of the vertebral column of uncertain aetiology, of which the most prevalent theory states that it is caused by a diet rich in animal protein. Stable isotope analysis is used to test whether such a high trophic level diet can indeed be linked to the prevalence of DISH.

### **B33 Paleodiet and Provenance of Marbles: Case Studies by Using Stable Isotope at the CIRCE Laboratory**

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Stable isotopes of the four most abundant elements of living matter (oxygen, carbon, hydrogen and nitrogen) are tracers of many processes for a wide range of applications in the field of anthropology, ecology, food industry, archeology. In this paper different examples illustrating studies recently conducted at the Center for Isotopic Research for Cultural and Environmental heritage (CIRCE) of the Second University of Naples, will be showed. In detail, for different archeological contexts (Spanish and Italian Middle Age sites, Iron Age Italian site, Roman Imperial site), studies concerning analysis of the diets of populations of the past and the identification and characterization of the origin and provenance of marble artifacts will be presented. Carbon and nitrogen isotope analyses have been applied to collagen extracted from human remains of a necropolis, in order to verify the hypothesis of presence of meat or fish in the diet of populations and we have compared these findings with studies on the coevals sites. Moreover we present the results obtained from measurements of the isotopic ratios of carbon and oxygen of a group of 20 epigraphic artifacts, found in the early Christian church of Abellinum (AV) in order to determine the area of origin of lithoid and try to draw meaningful information for the reconstruction of the history of the church. All these studies have in common the use of these powerful natural tracers, which, with appropriate techniques, can be followed to access an infinite store of information.

### **B34 Paleodiet in two Archaeological Sites in the Atacama Oasis Inferred by Isotopic Analysis**

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The Middle Horizon period in the Atacama oasis has been characterized mainly by the influence of the Tiwanaku state. Even though there is no evidence related to presence of Tiwanaku migrants in the archaeological sites of the Atacama oasis (Central-South Andes), there is evidence suggesting improvement of health and nutritional conditions of the local population during this period. At the same time, studies on the burials of some of the cemeteries from this period show important differences, in the offerings of a few individuals suggesting possible social status differentiation within groups of the Atacama culture.

The aim of the present work is to characterize the diet of individuals from the archaeological sites Solcor 3 and Coyo 3, and to evaluate the association of dietary patterns with social status according to offerings in their burials. Evidence from carbon and nitrogen isotopes would indicate ingestion of terrestrial diet, mainly based on the ingestion of rodents, camelids and vegetables such as maize, algarrobo (*Prosopis* sp), chañar (*Geoffrea decorticans*) and some beans. Qualitative diet differences between high and low status individuals, with the former consuming more protein resources and the second ingesting a higher amount of maize is tested. Physiological adaptation to arid conditions or by the consumption of animals that have gut bacteria, that tend to increase the values of nitrogen on their tissues, are potential factors explaining the isotopic values founded and are here discussed.

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### **B35 Tracing Mobility in Early Medieval Populations Using Strontium and Oxygen Isotopes – A Case Study from South-West Germany**

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In the second half of the 5<sup>th</sup> century AD, a new burial practice emerged in Central and Western Europe that entailed the creation of fresh burial grounds with inhumation graves oriented in an east-west orientation, and the placement of rich grave goods with interred individuals. There remains an ongoing debate regarding the origin and meaning underlying this burial rite. More traditional interpretations consider the richly furnished graves as remains of ‘Germanic’ immigrants (Bierbrauer, 2004). Newer perspectives criticize a sheer ‘migrational’ explanation and argue this new kind of burial practice is indicative of cultural reorientation of the local population in response to the social and political dynamics of the time (Fehr, 2008). In order to find out if the ‘founders’ of the cemeteries were immigrants, we use strontium ( $^{87}\text{Sr}/^{86}\text{Sr}$ ) and oxygen ( $\delta^{18}\text{O}$ ) isotope analyses of tooth enamel and dentine recovered from individuals of different early medieval cemeteries in the region Palatinate in south-west Germany. Human isotopic values are compared to those obtained from domestic animals- also recovered from graves-, which serve to define the local strontium signature at each respective site.

First results of the  $^{87}\text{Sr}/^{86}\text{Sr}$  isotope ratio measurements using MC-ICP-MS indicate that the values of the individuals dating to the earliest period of the cemetery Eppstein fall outside the local range. In the following period already about half of the sampled individuals fall into the local range while in the next period most seems to be of local origin. These results suggest that population movement and reorganization was somehow tied to the emergence of a new burial rite in the early Middle Ages.

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**B36 Isotopic Life History of Neolithic People from Busan Gadeokdo Janghang Site, Korea**

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This paper presents the dietary lifestyle of Neolithic people in Korea. Recently, a large number of human bones (46 individuals) were found in excavated cemeteries at Busan Newport, Gadeokdo Janghang site, which is an outstanding discovery in Korean Neolithic archaeology. There are also supposed grave goods such as pottery, red pigment, shell bracelet and jade accompanying the burial. We extracted isotopic information from human bone collagen using stable carbon and nitrogen isotope analysis, which was carried out on recently established stable isotope analysis laboratory (National Research Institute of Cultural Heritage, Korea). Isotopic results show the highly marine-based diet, as we expect the dietary input in this coastal area is mainly based on marine resources. Our isotopic findings will provide the invaluable information on subsistence economy, which may be based on fishing, animal hunting and plant gathering in this period. In addition, two different burial types are investigated at the site, and one type shows that individuals are found in bended position. Stable nitrogen isotope result implies that there is trophic level difference between different burial types. Although this is the preliminary study on this site, our study will open up potential to understand the 8000 year long isotopic dietary history of Korean Neolithic people.

**B37 Morphometric Data for Suines (*Sus scrofa domesticus* and *Sus scrofa ferus*) in Chalcolithic Period from Romania**

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This study is based on suines pig remains recovered in excavations of Chalcolithic (5000-3500 BC) assemblage from Romania belonging the following cultures: Petresti, Precucuteni, Cucuteni, Tiszapolgar, Cotofeni, Salcuta, Boian, Gumelnita. The discrimination between domestic and wild forms is often difficult due to the coexistence of these two forms in Neolithic period and it is possible that in archaeological samples occur hybrid forms which make complicated the identification. Our results reveal biometric data for suines remains and intend to characterize and distinguish interpopulational differences. We used relevant measurements recorded on different anatomical elements which were undergone statistical analysis. Linear measurements were defined according to von den Driesch (1976). A statistical analysis of recorded measurements has been attempted using bivariate and principal components analysis. The complete metapodials providing data on withers height are few therefore the withers height was established by means astragalus and calcaneus; the great wither height was detected in domestic pigs from Tiszapolgar Culture, for the other cultures were not find significant differences on this issue. The high degree of variability in characters emphasizes the presence of pig regional structures whose size varies but that fits the "palustris" type which characterized the Neolithic period.

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VON DEN DRIESCH, A., 1976. A guide to the measurement of animal bones from archaeological sites. *Peabody Museum Bulletin* 1. Peabody Museum of Archaeology and Ethnology, Harvard University, Cambridge.

**B38 The Use of Intra-Tooth Enamel  $\log(\text{Sr}/\text{Ca})$  &  $\log(\text{Ba}/\text{Ca})$  Sequences to Assess Animal Foddering Strategies**

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Sequential  $\log(\text{Sr}/\text{Ca})$  and  $\log(\text{Ba}/\text{Ca})$  measurements of enamel samples from caprines have been examined in order to provide information on paleodiet and past foddering strategies. Faunal remains recovered from Pre-Pottery Neolithic-B site of Tell Halula (Middle Euphrates Valley, Arab Republic of Syria) were analyzed to help understand changes in feeding strategies during the earliest development of domestication and herding strategies. Modern plant samples from the tell Halula site and surroundings were analyzed as well in order to evaluate specific dietary categories. Results show that elemental concentrations cycle through the growth axis of the tooth. Some observed patterns could likely be explained in terms of seasonal variation in the availability of plants while other patterns additionally suggest herd management strategies (human control of animal food, foddering herds during periods of less available resources), with distinct individual histories. The data contain significant information for understanding the kind of management strategies developed for the first herds of domesticated animals. Moreover, this work shows the potential of the qualitative information provided by alkaline-earth elements in intra-tooth variation studies from hypsodont mammal species.

### **B39 Non-Destructive Trace Element Analysis of Human Bones to Examine Diet and Mobility**

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While the theoretical basis for elemental analysis of human bone to study diet and mobility is well-established, the number of studies done is few and far between, especially with the increasingly wide application of carbon, nitrogen, oxygen, and strontium isotope analysis over the last 25 years. Nevertheless, elemental analysis of barium, strontium, and other trace elements can support isotope-based interpretations, and be of greater use in cases where bone collagen is not preserved. One potential obstacle for any elemental analysis research project is degradation and contamination of the mineral portion of bone. In virtually all previous studies the samples have been well-cleaned, ashed, and put into solution for analysis by ICP spectrometry.

Presented here are new studies done using X-ray fluorescence (XRF) spectrometry to non-destructively measure Sr, Ba, and other trace elements, along with major elements including calcium and phosphorus. This research was initiated by several osteoarchaeological cases where isotope analysis was not permitted due to its being destructive to human remains, an increasingly common issue in the Americas and other parts of the world. The first project was on 30 individuals from 4 prehistoric native American sites in peninsular Florida. The small amounts of variability among individuals at each site suggest little contamination, while the clear differences between the sites are most likely due to varying proportions of seafood in the diet.

This was followed by analyses of several hundred archaeological skeletal remains from several sites in Belize, one in Colombia, two in Peru, several in Bolivia, and a large number in both Chile and Argentina, all previously analyzed isotopically, and with interpretations made about the dietary importance of maize and/or seafood along with patterns based on sex and status. In comparison to Florida, these regions have much greater geological-based variation in Sr and Ba concentrations, which is partly assessed by analyses of faunal remains. Multiple analyses were done on each sample, both on the original surface and on a cross-cut section when possible, to check for potential contamination and variability. XRF results for samples with noticeable iron and/or zinc peaks are carefully studied since there could also be a related contamination effect on Sr and Ba values. Tooth enamel was also tested for some individuals. Both the reliability of this method of elemental analysis of skeletal remains, and its utility in studying ancient diets will be discussed.



## **B40 A View into the Lives of the Early Christians of Stavanger, Norway: A Palaeodietary Reconstruction Using Multiple Stable Isotopes (C, N, H and S) of Bone Collagen**

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Stable isotopes chemically record information in bone about numerous aspects of past human lives, such as diet, residence and possible migration. Stable isotopes of carbon and nitrogen are widely applied for dietary reconstructions. Hydrogen appears to be a reliable independent trophic level indicator (Reynard & Hedges, 2008), while sulphur can indicate possible migrants in the population (Vika, 2009) and aid in diet studies (Nehlich et al., 2010). Stable isotope datasets are expanding as the method is increasingly applied, which carries importance for comparison between other regions or archaeological periods. However, few stable isotope studies have been carried out for palaeodietary reconstruction using Norwegian material (Johansen et al., 1986), and none from the south west coast. In 1968, excavations revealed Christian burials predating the construction of the Stavanger cathedral, dating to the Iron Age, Viking Age and Middle Ages. The Iron Age individuals, being the earliest Christians found in Norway, raise questions about their origin and the expansion of Christianity. Additionally, excavations in 1995 in front of the cathedral yielded post-reformation burials. As the human remains from 1968 became commingled after excavation, the contextual information was lost. Still, this assemblage comprises the opportunity to draw more information from these bones using archaeometric analyses, which can provide insight in past life at individual and population level in the Stavanger region through time. Stable isotopes of carbon, nitrogen, hydrogen and sulphur were analysed on bone collagen of 18 humans and 37 animals, including a wide range of species, such as cattle, sheep, chicken, seal and various fish species. Preliminary results from carbon and nitrogen stable isotope analysis indicate the presence of marine protein in human diet. Additionally, a clear trend of enrichment in both  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  is visible from the Iron Age to the Middle Ages, although the consumption pattern changes again during the post-reformation period. The production and trade of stockfish during the Middle Ages possibly contributed to this trend. The pigs display a large variation in nitrogen isotope ratios combined with narrow carbon isotope ratios. The enrichment in  $\delta^{15}\text{N}$  may be induced by the proximity to the sea, e.g. salt spray or coastal salt marshes, possibly affecting human isotope ratios. The hydrogen and sulphur stable isotope analysis is ongoing. These isotopes are expected to aid in quantifying sources of human diet (Parnell et al., 2010). Overall, this provides unique first insights into both animal isotope value ranges of the region, and changes in dietary habits through time.

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## B41 "For Dust thou Art and ..." with Dust You Can Cure

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In traditional pharmacopoeias approximately 10-15% of pharmaceuticals are of mineral origin (Ackerknecht, 1971; Jeszke, 1996). The majority of them are the clay minerals (Wasilewski, 2009). These very widespread minerals were and still are widely used in traditional pharmacopeas. In some societies particular groups of people specialize (or specialized) in this kind of healing (Czubala, 1984). In anthropological studies the eating clay minerals phenomenon is usually called geophagy and was often described (e.g. Reid, 1992; Abrahams & Parsons, 1996). However the mineralogical and biological studies on this matter still are very scarce. The subject is still interesting enough to be explored, described and explained.

This study is based on the instrumental analysis (X-ray diffraction) of several mineral drugs from different parts of Egypt, Peru, Romania and Poland. The aim of the research was to compare the mineral composition of the traditional and academic medicaments and to confront it with the proposed use of them. Together with the detailed bibliographical query (e.g. Halstead, 1968; Wilson, 2003; among others) it gave the answer to several question about the reasons for using of particular clays, the efficacy of such medicaments, the mechanisms of internal and external reactions with human organism, the use-risk and the reasons for traditional restrictions, etc.

It is significant that clays and clay minerals were and are still used for the same purposes in different times and regions. This diachronic and synchronic cultural convergence together with our scientific knowledge on their biological action can prove the so called "folk wisdom" in medicine and indicate its rational rather than superstition basis.

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## **B42 Diagenetic Assessment of the Trace Element Composition of Classical Period Bones – A Case Study From Apollonia Pontica (Bulgaria)**

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Bone and other calcified tissues can be important archives of environmental and paleodietary exposures. Bone analyses based on archaeological sample material always hold the risk of generating invalid data, since the bones might have undergone severe decomposition processes during their inhumation period. A too advanced degree of diagenetic alteration reduces the success rate of usable archaeometrical results. In this study we analyzed trabecular bones, belong to individuals who had lived in Apollonia Pontica (Bulgaria) during mid 5<sup>th</sup> – 4<sup>th</sup> century BC. Using ICP-AES and ICP-MS the concentration of Al, As, Ce, Eu, Fe, Hg, K, La, Mg, Mn, Mo, Pb, Se, Sb, Sr, V, Y and Zr had been determined both in bones and soils samples collected from excavated site. Bone surface contamination was evaluated using sequential acid leaching that included distilled H<sub>2</sub>O, 0.1 M acetic acid, and microwave digestion in concentrated HNO<sub>3</sub> as this procedure was used parallel for upper layer of bone's surface removed previously by drilling in order to estimate soil effect into bones in depth. The results show a selected group of metals to be enriched by up to factor 5 in the bone surface, indicating that these elements may have a higher contamination component. However, the results of sequential acid leaching experiments indicated that mechanical removing of upper bone layer and single acid leaching step was effective in removing most surface-enriched contaminants associated with soil.

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### **B43 Residual Analysis of Hellenistic Time Ammoraes from Apollonia Pontika (Bulgaria): A Preliminary Results**

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The Greek colonization is ubiquitous Panhellenic phenomenon in 8<sup>th</sup> – 6<sup>th</sup> century BC. Many Greeks settled in non Greek territories where colonies were founded. The Greek colony of Apollonia Pontika (today Sozopol) was founded at 610 BC by settlers from the Greek city of Miletus (nowadays Turkey) and according to ancient sources from 5<sup>th</sup> – 4<sup>th</sup> century BC the number of the inhabitants is about 3000. The ancient necropolis, which is located on the shore, 2.5 km south of the city, consists so far of over 1500 burials which are studied over the last 15 years. Grave goods consist mainly of pottery, with predominance of lekythoi and occasionally other vases for scented oils and wines.

In the framework of a larger project devoted to paleodiet, technology of production of metals (mainly bronze) and glass artifacts in Greek colonies in the Black Sea coast funded by the National Scientific Fund, some of the amphorae with resin residue found at the bottom of these amphorae were sampled to carry out chemical analysis. High Performance Liquid Chromatography (HPLC, Waters "Aliace") and Nuclear Magnetic Resonance (NMR, Bruker, 250 MHz) had been used for the determination of organic residues with the intention of recovering further information of the use of amphorae under this study. In particular analysis of the resin residues was aimed at understanding content of the amphorae and to verify hypothesis on the transport of wine, named "Retsina". "Retsina" is well-known Greek wine with three-thousand year old tradition which is prepared with adding of extract from resin of Aleppo pine (*Pinus Halepensis*) (Manassis, 1996). Additionally this hypothesis was confirmed by such kind of analysis of modern resin residue from Aleppo pine (*Pinus Halepensis*) growth in Attica region (Greece).

Our previous analysis of resin residues from an ancient Greek amphora, found near Sborjanovo (NE Bulgaria) also shows trade contacts of Thracians with ancient Greek towns for wine supply (Surowiec et al., 2006; Zlateva et al., 2007).

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**V1 Production, Exchange, and Technological Identity: A Study of Islamic Umayyad pottery from northern Jordan****Firas Alawneh**

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This study investigates exchange and production of Islamic Umayyad ceramics from Jordan. Regional production and exchange of ceramic goods are integral to economic systems and political and social processes. Archaeological study in Jordan has traditionally focused on understanding the organization of a single component (production or distribution) within a regional ceramic economy. In recent years, however, researchers have suggested that a more integrated approach to the study of production and exchange is needed to elucidate the broader social contexts in which these economic activities took place. This research explores the connections and interrelationships between production and distribution systems in the northern regions of Jordan during the Umayyad period (A.D. 661-750). Focusing on Umayyad pottery, this comparative study evaluates organizational variability in regional economic systems in order to understand how social relations structure, and are structured by, different production and exchange practices. Three dimensions of regional ceramic economies are considered –production, exchange, and social identity. Although these dimensions involve intertwined social and economic processes, aspects of each may be delineated through the consideration of a few key parameters. Samples were analyzed by optical and electron microscopy to study their mineralogical and structural features, with the aim of defining the nature of the paste and recognizing inclusion types, and also by powder X-ray diffraction and X-ray fluorescence to determine production technology and possible provenance. Analytical data were statistically processed with multivariate analysis using SYSTAT 11 software 2006. To obtain further information about possible source areas of raw materials used in ceramic production, clays were also sampled in areas surrounding the archaeological sites. Some firing experiments were carried out on clays with compositions comparable with those of ceramic sherds, to define better the technological features of the firing process used to produce the pottery. The multifaceted analytical approach has revealed important information on ceramic production in Jordan. Amman, Jarash, Beit Ras and Umm Qaies are possibly just a few important production centers during this period. The study shows a multidirectional socio-cultural exchange and economic trade patterns within each region and between adjacent regions, as well. Also, importation from adjacent provinces cannot be excluded for certain samples. Despite the different archaeological levels to which these samples belong to, this study illustrates some similarity in technological features and chemical composition. This in turn suggests that technological identity is rather the trend in ceramic tradition of the society during the Umayyad period.

## **V2 A Geometrical Sintering Model (GSM) of Archaeological Ceramics for the Study of Transverse Rupture Strenght (TRS): Preliminary Results**

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Ceramic physical properties are affected by raw materials used and producing cycle. If on the one hand a great quantity of works in the field of new composite materials have been published (e.g. Kingery et al., 1976; Kuang et al., 1997), on the other hand less attention has been paid to archaeological ceramic sintering and the way it influences physical properties. A geometrical sintering model (GSM) of traditional ceramics has been developed and has been applied to study the transverse rupture strength (TRS) of two different kind of transformation vessels (glass-melting crucibles and pots). The model describes the sintering process between the ceramic change point and the maximum densification point (Hamer & Hamer, 2004). The model takes into account three different phases: matrix, temper and pores. Matrix is composed of equal-sized spherical grains, arranged according to the highest possible packing (tetrahedral coordination). Temper is characterized by spherical-shaped of fixed grain-size distribution and, as a whole, a volume fraction between 0 and 40 % of the ceramic body was considered. While for matrix, morphological changes have been allowed, no shape deformation has been assumed for temper. It has been fixed that the temper/matrix grain diameter ratio must be greater than or equal to twenty in order to have a nearly flat temper-matrix interface. Neck boundaries have been assumed cylindrical. Porosity is evaluated in dependence of sintering level, temper quantity and granulometry. Quartz has been considered as temper and its  $\alpha$ - $\beta$  transition has been taken into account. In the case of pots, the model also involved secondary shrinkage porosity and coarser grain-size distribution of temper. The model has been developed in three steps: (1) evaluation of matrix morphology and porosity according to the sintering level; (2) estimation of the porosity at the rim of a temper grains; (3) combination of the two effects. A comparison between the TRS output of the model and data of the literature (e.g. Kilikoglou et al., 1998) shows a good fit for high sintered ceramic without secondary porosity like glass-melting crucibles, and a large bias for low sintered pots. A finite element modeling (FEM) approach should be developed to simulate the mechanical behavior of pots.

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### V3 PIXE Analysis of Decoration Pixels in Classical Attic Pottery

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The paper focuses on the study of special techniques of the Athenian vase-painters making use of the advantages of non destructive, scanning  $\mu$ -PIXE (micro-proton induced X-ray emission) topography and 2D tomography. The experimental work was performed at the ATOMKI<sup>1</sup> Accelerator Centre (Institute of Nuclear Research of HAS, Debrecen) in Hungary focusing on the surface analysis of specially chosen decorated sherds from recent excavations in the slopes of the Acropolis and the area of Kerameikos, where an ancient neighbourhood and the potters' quarter located in Classical antiquity. The sherds analysed in the first experimental run (April 2010) are decorated with very fine relief lines of black glaze (red figure style) of the order of 0,2-1mm wide with occasional presence of added colours (white, purple and coral red). The trace element analysis of the black glaze, especially with respect to the Zn content, points to the use of at least two different clay sources for the preparation of the slip that eventually becomes black following a 3-stage firing under OxidisingReducingOxidising conditions. This is the first analysis of the attic black glaze combining quantification of trace elements in conjunction with both major and minor elements. The second experimental run (January 2011) focused on the detailed mapping with a submicron spatial resolution of the BG trace element content on 25 attic sherds. Differentiation patterns with respect to trace elements composition i.e. Ti, Mn, Ni, Co, Cu, Zn, As, Pb were observed with local concentration of certain elements extending over areas of 5-10 $\mu$ m. This led to a geological survey of iron rich illitic clays covering a plateau at the NW part of Attica (Panakton area) near the borders with Boeotia. The samples collected were processed at the THETIS laboratory for the preparation of clay slips, i.e. suspensions of illitic clays in water. The next experimental run (January 2012) will focus on the surface analysis of the black glazed laboratory specimens produced with firing of these clay slips under a 3\_stage cycle imitating the ancient, so-called, iron reduction technique. This will allow a direct comparison of the archaeological sherds with laboratory specimens bearing geological information with a view to address for the first time the long standing archaeological question on the provenance of the clay(s) used for the decoration of attic pottery.

<sup>1</sup>For the period 2010-11 the analysis was carried out in the framework of CHARISMA (Cultural Heritage Advanced Research Infrastructures: Synergy for a Multidisciplinary Approach to Conservation/Restoration), an EU-funded integrating activity in FP7/ Capacities Specific Programme(<http://www.charismaproject.eu/>); for 2012-14 the project is supported by NARNIA Marie Curie Initial Training Network (FP7-PEOPLE-2010-ITN)<http://narnia-itn.eu/>



#### **V4 Rediscovering Brockley Hill: A Compositional and Technological Study of Verulamium White Ware**

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This project focuses on the technological and compositional characterization of well dated groups of pottery from Brockley Hill, one of the most extensively excavated romano-british kiln sites of the Verulamium region industry (Seeley & Thorogood, 1994; Castle, 1976). This large pottery industry developed along Watling Street in the area between *Verulamium* (St. Albans) and *Londinium* (London) and was one of the major suppliers of Romanised coarse pottery for southeast England between the late 1<sup>st</sup> and early 2<sup>nd</sup> AD.

An integrated plan of petrographic and chemical analysis (Energy Dispersive X-Ray Fluorescence) was applied to the Verulamium Region White Ware (VRW), the most common ware produced at Brockley Hill (Tyers, 1996). The data obtained was processed through different kinds of statistical analyses, hereby providing valuable information about a variety of technological aspects related to the VRW production. Within that it was possible to define a clear chemical and petrographic fingerprint for the VRW produced in Brockley Hill. This will allow to clarify issues related to the VRW production and distribution. In fact, given the similarities between products created by this industry, only chemical and petrographic analysis can provide a reliable tool to identify products manufactured in the different kiln sites.

Furthermore this research allowed to clarify the broader socio-political contexts under which this pottery were manufactured through the reconstruction of the organization of production. The results revealed a scenario of strong persistence of technological traditions and denotes Brockley Hill as a large scale production site, where potters of different origins (Swan, 1984) were working while sharing similar sources of raw materials and technological choices throughout the period in which the kiln site was in function.

Moreover a programmed geological survey in the area during the next months will attempt to individualize outcrops of clay likely to be used for pottery production in Brockley Hill, but also in the other kilns sites. This will allow to have a better understanding of the clay sources exploited by that time and to clarify the issues raised by the petrographic and chemical analyses.

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## V5 Among the Aspects of the Past : The Detection of Tin Foil in Hellenistic Terracotta Figurines

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It is known that the ancient city of Pherai was an important centre of a major local industry producing several types of clay objects, including terracotta products (Doulgeri-Intzesiloglou, 1992). Plenty of such material was collected as a result of the excavations carried out systematically, over thirty years, in Pherai (Doulgeri-Intzesiloglou, 2000; Doulgeri-Intzesiloglou & Arachoviti, 2006).

This material became the focal point of a research which started in 2008 and is still ongoing. The main purpose of this study was to better understand the techniques which were developed for the manufacture of these objects, the detection of the pigments which were survived on their surface, as well as other materials that were used to decorate them. Non-destructive surface analysis using X-ray fluorescence spectrometry operating in air, X-ray equipment and UV light were the main methods used in the first stage of this study. More than two hundred objects have been analysed so far and most of the pigments have been determined. However, these techniques were able to help us identify important aspects of the decoration of these figurines that to date had escaped notice.

During the analyses of the pigments, high levels of tin were detected in some areas. After a careful cleaning of the surface of the objects, fragments of metal tin foil appeared (Asderaki-Tzoumerkioti & Doulgeri-Intzesiloglou, 2010). In most cases the foil is poorly preserved and covered with a thick layer of soil and salts, which made it invisible. After a more careful examination of the rest of the figurines, tin foil has been identified so far in 19 fragments of different pieces. This was an unexpected find; the use of tin foil for decorating ceramic figurines was not known in the literature and its identification now raises an issue of major conservation, archaeological and analytical interest. However, we do know that tin foil decoration was used both on Mycenaean and Hellenistic ceramic vessels (Holmberg, 1983; Gillis, 1991; Gillis & Bohm, 1994; McCarthy, 2003; Kotitsa & Schussler, 2002), which opens up interesting possibilities for comparative technical and art historical studies.

This discovery encouraged us to further examine selected areas of the figurines with environmental scanning electron microscope-EDS. This confirmed that the presence of the tin foil is beyond any doubt. Its thickness has been measured and the organic binding medium with which it was adhered to the surface of the figurines has been determined.

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## V6 Analysis of Lustred Ceramics of the Galleria Regionale Di Palazzo Bellomo di Siracusa

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Lustre was one of the most important decorative techniques of medieval and renaissance pottery of the Mediterranean basin. It produces brilliant reflections of different colours and iridescence, and it is characterised by a heterogeneous metal–glass film, some hundreds of nanometres thick, essentially made of silver and copper nano-particles dispersed within the layers of the glaze.

Several fragments of lustred pottery from the collection of the *Galleria Regionale di Palazzo Bellomo*, mainly excavated in Syracuse, were studied through techniques of ion beam analysis in the *Centre de Recherche et de Restoration des Musees de France*, located at the *Louvre Museum* in Paris. The study of the composition of the terracotta and glaze and of the structure of the lustre was proposed in order to have more information about these objects, up to now never studied from this point of view, whose provenance and dating are not certain, as based only on the aesthetic and stylistic observations.

The elemental compositions of pottery and glazed parts were obtained by PIXE analysis, while the composition and stratigraphy of the lustred decorations were analyzed by the RBS technique. The results obtained, crossed with those from XRF application of spectroscopic techniques, Raman and optical properties, have provided important information on dating and origin of the objects. A more comprehensive study is currently using the application of additional analytical techniques.

## V7 The Technology of Historical Basque Pottery Production During the 14<sup>th</sup> to 17<sup>th</sup> Centuries

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The Basque Country was a dynamic production area of Majolica pottery, together with other centres of the Crowns of Castile and Aragon (Sevilla, Talavera de la Reina, Barcelona, Muel, etc.). Alongside this production, a wide range of coarse glazed and unglazed pottery, which would have been used as tableware, cooking ware and storage vases, was most likely made in those workshops, which remain as yet unexcavated.

In order to define this production and establish the technology used for their manufacture, material has been examined from sites located in the Basque Autonomous Community dated back to the 14<sup>th</sup> and 17<sup>th</sup> centuries. The techniques used are thin section petrography, X-ray diffraction, X-ray fluorescence and scanning electron microscopy to investigate issues of raw material provenance and choice, as well as the technology of production.

In the first phase of research, the petrographic study has identified the presence of several technologically different products. While the picture remains complex, three main fabric groups have been defined. The first is a low-fired low calcareous pottery which is rich in white mica, primarily present in glazed ceramics (plates, serving dishes, large earthen jars, etc.). The second comprises a high-fired calcareous ceramic, mostly yellow/grey in colour and, less frequently red, mainly majolica (*escudillas*, plates, pitchers, etc.). The remaining group is a red ferruginous clay, a low-fired low calcareous pottery, rich in quartz and micas and with frequent clay pellets. This is present in coarse unglazed ceramics (large earthen jars, pitchers, etc.). It should be noted that, in general, the clays used were rich in quartz and iron.

The characterization of these groups by petrography and XRF is the first step in an effort to define production at a number of locations in the Basque country found in first contact North America. The presence of pottery of Basque origin has much to offer the study of Basque settlement and trade on the Atlantic coast of Canada, on account of the Basque fishing industry which was so important in the 16<sup>th</sup> century.

## V8 Chemical Characterization of Medieval Glass Coming from two Stained Glass Windows from Girona Cathedral (NE Spain)

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The chemical composition of 15 medieval stained glass fragments from several panels of stained glass windows coming at least from two separate chapels of Girona Cathedral (now conserved at the cathedral archive) has been conducted by HR-ICP-MS-LAM (the 10 main constituent elements of the glass, and up to 49 trace elements). This set of samples includes uncolored and colored glass: deep green, yellow, silver yellow over uncolored glass, pink, deep blue, purple, and red plaque.

The gothic Girona Cathedral was built in the XIV<sup>th</sup> century over the precedent roman one. The studied stained glass windows can be attributed, respectively, to the Master of the Presbiterio (the older ones, beginning of XIV<sup>th</sup> century) and the normand Guillem de Letungard (documented working on site at 1357).

Attending to the main elements composition, 10 glass fragments are sodic glass of Mediterranean tradition (Gimeno & Pugès, 2002; Gimeno et al., 2008, 2010), while the other 5 are lime-potash showing the two well-known compositions of this type of glass in northern Europe during medieval times. Four of these lime-potash glass fragments are plaqué (3 red glass and the other blue) a technology till not developed in Southern Europe at that time. In particular, the presence of red (plaqué) glass fragments are widespread in Mediterranean medieval stained glass windows mostly made with sodic glass and is considered the product of trade with northern Europe. The color in red plaqué glass was obtained as usual at that time with thin strata of dispersed copper microcrystals in uncolored glass. The blue plaque lime-potash glass was obtained with cobalt, as well as massive lime-potash blue purple glass.

The color recipes in sodic glass have been also deciphered. The four blue fragments were also obtained with addition of cobalt, the green glass fragments by adding copper and iron (a well know recipe known i.e. at Pedralbes Monastery's church at Barcelona Gimeno & Pugès, 2002), the brown-amber was obtained with iron adding to the composition of uncolored glass, the yellow silver was obviously obtained with this element. All the color recipes are in good agreement with the coeval ones previously described in the Mediterranean area (Gimeno & Pugès, 2002; Gimeno et al., 2008, 2010).

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## **V9 Basic Research to Establish the Use of p-XRF in Pottery Provenance Studies**

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In a pioneering stage of application, portable X-ray fluorescence analysis (p-XRF) has been used in a number of recent projects to analyse pottery, especially for provenance studies. Various research groups have shown that in principle, accurate measurements can be obtained using p-XRF. However, compared to classical laboratory analytic methods, p-XRF can only measure a limited set of chemical elements, and the measurements have a greater standard deviation. To investigate the influence of the lower number of elements and larger standard deviation, we examined a series of ceramic ensembles (approximately 500 samples), ranging in origin from the Neolithic to the Iron Age, using both classical laboratory methods (XRF, ICP-MS, NAA) and p-XRF. The results were subsequently analyzed and critically compared using multivariate statistical methods. Despite the successes achieved to date, some fundamental questions about the use of portable x-ray fluorescence devices remain to be adequately clarified.

Our study focused furthermore in particular on examining the effects that the temper has on measurement quality. To address existing desideratum in our understanding in this area, we began by manufacturing more than 100 ceramic samples with varying degrees and forms of temper in different grain sizes, and subsequently analyzed them. Furthermore, we compare these results with theoretical derivation of the influence of the temper (grain size and quantity) for the quality of the measurement. Having conducted a precise analysis of these types of effects, we now hope to present the results of our research for the purpose of critical discussion. Thanks to the comprehensive basic research we have conducted, the advantages and limitations of p-XRF are now more precisely understood. Our work thus represents an important step in determining the appropriate role of p-XRF in pottery provenance studies.

## V10 Archaeometric Investigation of Phoenician Pottery from the Iberian Peninsula

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The Iberian Peninsula was the most important target of the Phoenician expansion at the beginning of the 1<sup>st</sup> millennium BC. From the 9th century onwards the newcomers from the Levant established a dense network of settlements on the southern coastal parts of the Iberian Peninsula why this region was culturally part of the Orient for a time of more than 200 years. For a better understanding of the cultural and economic development of these settlements a new large-scale pilot project for the archaeometric investigation of Phoenician pottery was initiated. Until today more than 1000 pottery samples of all known wares and types from about 60 different sites were analyzed. The pottery covers a time span from the 9<sup>th</sup> to 6<sup>th</sup> century BC. Thanks to the great amount of analyzed pottery the project offers for the first time a solid scientific basis for the reconstruction of the economic network between the settlements and also of its supraregional connections.

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BEHRENDT, S., MIELKE, D.P. AND TAGLE, R., 2010. Provenienzanalysen im Vergleich - Neue Ansätze zur Klassifizierung von Keramik. In: HAHN, O., HAUPTMANN, A., MODARRESSI-THERANI, D. AND PRANGE, M. (Eds.) *Archäometrie und Denkmalpflege 2010. Jahrestagung im Deutschen Bergbau-Museum Bochum, 15.-18. September 2010*. Metalla, **Sonderheft 3**, 113-115.



## V11 Investigating the Influence of Neutron Activation Analysis on European Trade Glass Beads

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Intensive chemical analyses of trade glass beads discovered in Quebec have been performed since the 1990's by Ron Hancock and Jean-François Moreau using neutron activation analysis (NAA) (Hancock et al., 1996; Moreau & Hancock, 2010). This work has resulted into the creation of a large database that may be used as a tool for dating beads by comparisons of their chemical compositions. However, new questions are being asked by the archaeologists. The two most important are: how were these beads made? Is it possible to make sub-groups in the dating groups in order to follow the penetration of beads from their coastal point of arrival into the interior of North America? Raman spectroscopy may be used to investigate the structure of the glass and thus to answer the question about the manufacture of beads. That method has already proven to be very good at finding the "shape" of the opacifiers, to identify the type of glass and to determine different groups of beads based on their glass type (Tournié, 2009; Colomban, 2004). Since a large number of glass beads have been analysed with NAA, a method which use a neutron beam to excite the atoms and turn a small fraction of them into radioactive isotopes, that method may interfere with our interpretation. The transmutation of some atoms, and the beta and gamma emissions of the radioisotopes produced, may introduce changes in the glass structure. Our study aims to verify the possibility of permanent modification of the glass structure as a result of the use of NAA and thus the possibility to apply Raman spectroscopy to pre-analyzed beads.

COLOMBAN PH., 2004. Raman spectrometry, a unique tool to analyze and classify ancient ceramics and glasses. *Applied Physics A: Materials Science & Processing* **79**, 167-170.

HANCOCK R.G.V., AUFREITER S., MOREAU J.F. AND KENYON I., 1996. Chemical Chronology of Turquoise Blue Glass Trade Beads from the Lac-Saint-Jean Region of Québec. In: ORNA M.V. (Ed.) *Archaeological Chemistry: Organic, Inorganic, and Biochemical Analysis*. American Chemical Society, ACS Symposium Series **625**, 23-36.

MOREAU J.F. AND HANCOCK R.G.V., 2010. Un siècle d'approvisionnement : 1550-1650 : de la préhistoire à l'histoire au site du poste de traite de Chicoutimi. *Archéologique* **23**, 84-98.

TOURNIE, A., 2009. *Analyse Raman sur site de verres et vitraux anciens: modélisation, procédure, lixiviation et caractérisation*. Université Pierre et Marie Curie, Paris VI, Thèse, p. 227.

## **V12 Characterization and Interpretation of Roman Ceramic Manufacture in the *Civitas Tungrorum*, Belgium**

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The *Civitas Tungrorum* is known to have become an important pottery production center during the Roman period, with numerous kilns already identified in the region. Roman ceramic technology arrived in the mid 1<sup>st</sup> century A.D., and represents a cultural marker used to separate the Historic Period from the preceding Iron Age. Beyond the classification of ceramics into broad shape categories and hand-specimen fabric types, little is known of how they were integrated in local manufacturing traditions. This thesis study examined the evidence for economy, marketing and distribution and society at this time, with an integrated compositional focus on the important pottery industries (using petrography and geochemistry). By focusing on the raw materials and the techniques by which they have been manipulated, it has been possible to define a much higher diversity of paste preparation techniques than previously thought. The analysis of changes in the operational chain of ceramic manufacture has clarified to what extent these matched changes in patterns of ceramic consumption, potentially shedding light on continuity as well as discontinuity in potting groups, or in the organization of ceramic production. By placing ceramic compositional groups in their landscape, we hope to provide a firm basis with which to interpret aspects of ancient potters' craft practice within these northern fringes of the Roman Empire.

### **V13 Pottery Production and Regional Distribution at Düzen Tepe-Sagalassos and the Lake Burdur Area (Southwest Turkey) During the Classical and Hellenistic Period**

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This paper wishes to present the results of a high resolution archaeometric study of the ancient ceramics in the later territory of Sagalassos dated to the Classical and Hellenistic periods (5<sup>th</sup>-2<sup>nd</sup> century BC). This study specifically focuses on the ceramics from the periods directly preceding the mass production of Roman imperial ceramics at Sagalassos. Up till now, no systematic interdisciplinary research on these assemblages was undertaken.

Central in the debate is the site of Düzen Tepe, located 1.8km South from Sagalassos, excavated since 2006. The ceramics of this site are compared to pottery of Archaic, Classical and Hellenistic sites in the later territory of Sagalassos (Düver, Belören, Seydiköy, Hisar, Aykırıkça, Suludere, Gavur Evi, Bereket, Kökez, Kepez Kalesi, Taşkapı Kale and Körustan), the survey remains from smaller sites within the Ağlasun Valley itself and finally with the Hellenistic ceramics of Sagalassos.

Questions remain towards the understanding of the variability in these wares, as macroscopic classification from a fabric point of view proves to be highly erratic for a reliable mapping of these ceramics. Also, the provenance question needs addressing, collecting information on the possible common origin of the pottery from different sites and/or periods. Related to the question of local production is the circulation of wares and the transfer of technological solutions.

In order to create both a typo-chronological and archaeometric framework, a set of 361 archaeological samples was collected. The analytical procedure followed included optical microscopy and chemical analysis (ICP-MS, EMPA). Major and trace element diagrams and NASC-normalized multi-element diagrams were edited in order to determine the variability within the ceramics. This study reflects the ability to establish whether the main types of pottery appearing in the Archaic, Classical and Hellenistic period in the Sagalassos area are locally produced or imported. These results can add to the discussion on changes in craft activities, and by extension possible cultural changes in Sagalassos, Pisidia and the area of Southwest Turkey in general.

## **V14 Roman Amphora Production at Casa Valentini (Central Italy) During the Late Republican and Early Imperial Period**

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Since 2002, an interdisciplinary project of Ghent University, Belgium focused on reconstructing settlement history within the Potenza Valley, Italy. To the south of the modern city of Ancona several amphora workshops were discovered in the vicinity of ancient *Potentia*. Major and trace-element concentrations determined by inductively coupled plasma-optical emission spectrometry (ICP-OES) show that three pottery groups can be distinguished involving the characterization of new amphora production sites at the Adriatic coast. This paper focuses on four different research questions: (1) characterize the amphora production at Casa Valentini (Central Italy), (2) identify possible chronological differences in raw material use, (3) outline morphological diversification in function of resource selection and (4) define the relation of the ceramics with the excavated kilns and samples clay and soil material.

The selection of raw materials is chronologically diversified and corresponds with the local production of Greco-Italian amphora (175 – 125 BC) and Dressel 6A/B amphora (25BC -25 AD). Two groups of Dressel 6A/B pottery from Casa Valentini, Casa Alvata and Colle Bruchio are easily distinguished using SiO<sub>2</sub>, CaO, Sr and Zr data combined with statistical factor and cluster analysis. Using geochemical analysis in determining pottery production proved to be successful in determining local signatures of ceramics and clays. In this study alkaline earth minerals (e.g. Ba and Sr) and high field strength elements (e.g. Zr) are considered useful for provenance studies taking into account to variety of clays and non-plastic material used in manufacturing ceramics.

## **V15 Raw Materials for Roman Glass Production in the Western Mediterranean**

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During the Late Roman and Byzantine period, natron glass was made from its raw materials in a limited number of primary production centres in Egypt and Syro-Palestine. For the earlier Hellenistic and Roman period, no primary furnaces have been found and the location of primary production during this era remains unclear. Ancient authors such as Strabo and Pliny the Elder suggest that glassmaking sands were found near the River Belus (Israel), in Egypt, near the mouth of the Volturno River (Italy) and also in Spain and France. However, primary production in the western part of the Mediterranean is not supported by any direct archaeological evidence and possible sand raw materials from these regions have never been evaluated for their suitability to produce glass.

In this study we investigate the possible existence of a Roman primary glass industry in the western Mediterranean, based on the occurrence of suitable sand raw materials. 178 beach sands from Spain, France and Italy are evaluated for their suitability for glass production by calculating the composition of hypothetical glasses made from these sands and comparing them to Roman natron glass. The results show that good glassmaking sands are far from common. Only a very limited number of the 178 analysed beach sands would produce a glass with major and minor elemental compositions within the ranges of Roman imperial natron glass. The rest of the analysed sands are unsuitable for glass production in their present form due to their insufficient  $\text{SiO}_2$ , high  $\text{Al}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  and either too low or too high  $\text{CaO}$  contents. If the sand raw material was too low in  $\text{CaO}$ , extra lime could be added to the glass batch in the form of shell or limestone. This was taken into account in a second calculation.

Overall we were able to define six limited areas where suitable sand raw materials would have been available to the Roman glassmaker. Good glassmaking sands occur in the Basilicata and Puglia regions (SE Italy), and Tuscany (W Italy). After the addition of an extra source of lime also sands from the Huelva province (SW Spain), the Murcia region (SE Spain) and from the Provence (SE France) would produce glasses with a typical Roman composition.

## V16 Sr and Nd Isotopic Analysis of Homemade Roman Natron Glass

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Ancient natron glass was essentially a mixture of three components: quartz sand, fluxed with a soda-rich mineral matter called natron, and stabilised with lime. The natron was derived from evaporitic lake deposits rich in sodium carbonates with minor sulphates and chlorides, and was a fairly pure soda source. The major source of lime would have been calcium carbonate, which either was added deliberately to the glass batch as a separate component or accidentally as particles of shell or limestone in the sand used as the source of silica.

The elemental composition of Roman glass is relatively uniform, which makes it very difficult to link specific objects to their (primary) production location. During the past decade, however, studies on the provenance determination of ancient glass have revived due to the introduction of Sr and Nd isotopes. While Sr isotopic ratios are considered to be an indication of the source of lime in the glass, Nd isotopes are a tracer for the sand raw material. More and more isotopic data of ancient glass is being published and recently a database of Sr and Nd isotopic signatures of possible sand raw materials became available for comparison (Brems et al., Submitted). Because of their relatively high masses and low relative mass differences, Sr and Nd isotopes are expected not to be fractionated during glass melting and therefore the isotopic composition of the glass artefact would be identical to that of the raw materials from which it was derived. But is this really true? To our knowledge, this has never been verified experimentally.

In this study, the influence of primary melting on the isotopic composition of glass is studied by performing a series of melting experiments. Naturally occurring calcareous beach sands and synthetic sodium carbonates were used to produce glass with a typical Roman natron glass composition. The influence of the three different basic constituents (i.e. sand, lime and natron) on the final isotopic signature of the glass is studied and an isotopic mass balance is constructed. It is shown that the isotopic signature of the experimental glass can not always be explained by simple binary mixing between the calcareous sand and the soda. The differences between the isotopic signatures of the sand and the glass might be attributed to the fact that not all mineral phases present in the sand have been completely dissolved in the glass.

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## V17 Black-Appearing Roman Glass: A Continued Research

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A large collection of archaeological black-appearing glass originating from various areas of the Roman Empire and dated to the 1<sup>st</sup> - 5<sup>th</sup> century AD has been gathered over the past years with the aim of performing a thorough historical-archaeological and chemical-physical characterization in order to extract information that can help our understanding of the organization of glassmaking, trade and glass-working in that era.

Black glass enjoyed a changing popularity throughout the Roman Imperial period, and an empire wide consumption based on a regional distribution pattern (Cosyns, 2011). This makes it an ideal subject of study. The glass samples analyzed are all macroscopically black, but when the glass is looked through in thin layers, a distinction appears between the different colors applied to obtain black appearing glass. This means the adoption of various recipes to produce black glass artefacts, which were verified to detect regional and chronological idiosyncrasies.

The investigation of these glass finds involved the quantification of the chemical composition down to the trace element level of over 400 relevant black-appearing Roman glass fragments. The analyses were performed by means of a Scanning Electron Microscope coupled with Energy-Dispersive X-ray spectroscopy (SEM-EDX) for the major-minor element composition and Laser Ablation – Inductively-Coupled Plasma – Mass Spectrometry (LA-ICP-MS) for the trace elements.

Different recipes have been discovered that were in use for the production of black glass, with two major changes, one in the second half of the 2<sup>nd</sup> century AD and the second around the mid 4<sup>th</sup> century. In the second half of the 2<sup>nd</sup> century a transition is registered between black-appearing glass made with impure Fe-rich sand without deliberate addition of coloring agents and natron-based Syro-Levantine glass, colored with a high amount of iron (4-10%) (Van der Linden et al., 2009). A second major change occurs around the mid 4<sup>th</sup> century, when this Levantine glass got substituted by black-appearing glass manufactured by means of HIMT base glass and high amounts of iron (Cagno et al., accepted).

Our results support the hypothesis that in a first phase, the craftsmen knowledge in the western provinces was limited to the production of objects by re-melting imported naturally colored and deeply colored raw glass. In a later phase at about the mid 2<sup>nd</sup> century AD, the expertise got enriched with the acquaintance in how to obtain independently the color of the re-melted glass batch: in this case naturally colored glass was rendered black by addition of iron. This hypothesis is further corroborated by the subsequent appearance of the HIMT glass colored in the same way.

CAGNO, S., COSYNS, P., VAN DER LINDEN, V., SCHALM, O., IZMER, A., DECONINCK, I., VANHAECKE, F., NOWAK, A., WAGNER, B., BULSKA, E., NYS, K. AND JANSSENS, K., Accepted. Composition data of a large collection of black-appearing Roman glass. *Open Journal of*

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## V18 Compositional Analysis of 14<sup>th</sup> Century English Stained Glass and Characterization of Corrosion Bodies via Synchrotron-Based Techniques

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In the framework of the investigation of medieval archaeological stained glass excavated in the Sidney Sussex College, Cambridge (UK) and dated to the 14<sup>th</sup> century, the composition of the unaltered bulk glass was determined and the superficial alteration layer of these glass finds was characterized by means of different synchrotron-based techniques.

These glass finds are affected by what is known in glass conservation as *manganese-browning*. Manganese-browning is caused by the presence of dark-colored Mn-rich stains in the alteration layer of buried glass artifacts that turn the surface brown/black (Cooper et al., 1993; Domenech-Carbò et al., 2006; Schalm et al., 2011). Manganese browning is treated by glass restorers by means of mild reducing/chelating agents (Fitz, 1981; Torge et al., 1993).

The composition of the archaeological fragments was determined by means of Scanning Electron Microscope coupled with Energy Dispersive X-ray spectroscopy (SEM-EDX) analyses. Subsequently the alteration layer, made by leached-out glass and Mn-rich corrosion bodies (Schalm et al., 2011), were examined by means of synchrotron radiation-based techniques, namely microscopic X-ray Absorption Near-Edge Spectroscopy ( $\mu$ -XANES) and microscopic X-Ray Fluorescence ( $\mu$ -XRF) and with high resolution Computed absorption Tomography ( $\mu$ -CT). Moreover, the glass fragments were monitored during conservation treatments performed with different chemical reagents.

The SEM-EDX results show that the investigated samples are made of potash-lime-silica glass (Cagno et al., 2011). By means of  $\mu$ -XANES it has been verified that, while manganese is present in the glass the +II or +III oxidation states, in the Mn-rich bodies manganese is in a higher oxidation state (+IV) (Schalm et al., 2011; Cagno et al., 2011). Thanks to the  $\mu$ -CT the total volume originally occupied by the leached out glass and the Mn-rich bodies could be determined. Finally, the evaluation of the conservation treatment allowed us to calculate the speed of Mn-removal for different products and evidence the negative effects created by the treatment itself (Cagno et al. 2011). These findings are useful for optimizing the modalities in which the treatment is performed, as well as minimizing its unwanted results.

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## **V19 Production and Distribution of Cooking Wares in the Early Islamic Vega of Granada (Spain)**

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In 711 CE the Muslims invaded the Iberian Peninsula, which comprised for many years the frontier of the Islamic Empire in the West. Although there is abundant scholarship on the study of the adaptation of Islam to al-Andalus, very little has been written on production and distribution of everyday goods.

In this study we analyze cooking wares from 6<sup>th</sup> to 12<sup>th</sup> centuries from seven sites in the area of the Vega of Granada, in south-eastern Spain. Supported by abundant archaeological research in the Vega itself (Carvajal, 2008, 2009), this paper examines cooking wares of the region with petrography. We deal not only with the two towns of the region, Ilbirah and Garnatah, but our study is extended to contemporaneous sites of the Vega and all the results are brought together.

Our work combines insights from morphological typology, technological choices and micro-provenancing of fabrics. The same basic principles in typologies and choices of raw materials for technological recipes are shared all over the Vega during the time-span under study, showing a common cultural development of the human communities. The micro-provenancing studies however show that production and distribution are managed at very local level until the late 10<sup>th</sup> or early 11<sup>th</sup> centuries, which accounts for a lack of economic regional structuration in spite of the shared cultural elements. After 1000 CE, our study shows how the production of an urban centre, Garnatah, overtakes the formerly local production of the site of Nivar, thus allowing us to speak of an economic change at regional level.

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## **V20 An Ethnoarchaeometric Study of the Cooking Ware Production of Pòrtol (Mallorca, Balearic Islands)**

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The Balearic Islands, in the Western Mediterranean, have a long pottery-making tradition normally divided into two main groups of pottery. On the one hand the unglazed ceramics mainly for water purposes and on the other hand glazed production including the cooking wares.

Within the framework of a larger project on Late Roman Cooking Wares in the Mediterranean (LRCWMED), one of the aspects considered was the study of living pottery making traditions, particularly of cooking wares, to undertake an ethnoarchaeological approach integrated with the application of physicochemical and minero-petrographic techniques in order to test assumptions in archaeometry of ancient ceramics. From a broader perspective the intention was also to record a pottery tradition that is rapidly vanishing.

This paper introduces the on-going project in and around the village of *Pòrtol* on the island of Mallorca, in the Balearic Islands. After an exploratory visit in 2003, two brief study seasons were carried out during 2010 and 2011.

Our aim is to present the analytical results obtained. Both raw materials and pottery from different workshops were characterised using a combination of techniques (XRF, XRD, OM by thin-section analysis). This has allowed to obtain the reference group for several workshops and to understand the *chaîne opératoire* as well as to examine the dynamics of ceramic traditions, especially those which pertain to cooking vessel production in the context of economic change which has taken place on this Mediterranean island.

## **V21 Inorganic and Organic Characterisation of Late Roman Pottery from the Site of Can Muntanyans (Palma de Mallorca, Balearic Islands)**

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Within the framework of a larger project on Late Roman Cooking Wares (herein LRCWs) in the Mediterranean (LRCWMED), several sites have been selected in order to characterised the main LRCWs and coarse wares both local and imported.

Archaeological excavations in the Roman and Late Antique city of Palma (underneath the modern Palma de Mallorca) in the island of Mallorca (Balearic Islands), have documented two ceramic deposits that can be provisionally dated to the Vandal (455-534 AD) and Byzantine (534-902/903 AD) periods, respectively.

The paper provides the first analytical results of the archaeometric characterisation of some LRCWs of these deposits, obtained by a combination of techniques: X-ray fluorescence, X-ray diffraction, and optical microscopy by thin-sections analysis for the petrographic characterisation.

The results show the presence of a good number of hand-made cooking wares fabrics from South-western Spain and the Central Mediterranean islands and no local products have been identified. Coarse Wares are dominated by the regional products of Ibiza with additional imports of North African coarse wares.

Moreover, some of the materials, particularly LRCWs, were sampled to carry out chemical analyses of the organic residues with Gas chromatography–Mass spectrometry in order to obtain information on the organic residues contained in some of the ceramics to better understand the food prepared and consumed at the site.

Therefore, the study present a combination of inorganic and organic analyses carried out on the same samples from the same Late Antique deposits, ranging from the mid 5<sup>th</sup> to the mid 6<sup>th</sup> centuries AD from the city of Palma in the Balearics.

## V22 Optical Spectroscopy and LA-ICP-MS for the Characterization of Roman Glass

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In archaeometry several analytical tools have been applied for the chemical, structural and physical characterization of ancient glass. Among these, the most common are SEM-EDX, EPMA and LA-ICP-MS (Brill, 1988; Cagno et al., 2010; Freestone et al., 2002; Garcia-Heras et al., 2005; Jackson, 2005; Kato et al., 2009, 2010; Schibille et al., 2008). These techniques provide precious information concerning the raw materials and their provenance. However, an analytical method which has the potentiality to be very beneficial for the study of ancient glass materials is optical spectroscopy (Schreurs & Brill, 1984; Green & Hart, 1987; Mirti et al., 2000, 2002; Meulebroeck et al., 2010a, 2010b; Ceglia et al., 2011). This work concerns the study of approximately two hundred colourless or “naturally” coloured Roman glass fragments found at Treignes (Belgium) using the following approach. First, all material is analysed by means of optical spectroscopy. Several parameters are helpful to subdivide the original set of fragments in different groups: the determination of impurities ( $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$  and  $\text{Co}^{2+}$ ), the UV-absorption edge and the colour coordinates. In particular cobalt may indicate the use of recycled glass, while the ratio of the oxidation states of iron gives an insight into the technological aspects of production. The second step is the selection of a subset samples from each group and their chemical characterization using LA-ICP-MS. The results provide important information about the proportion between different compositional groups available in this site, as well as about glass recycling. A comparison between the analytical results obtained and published data helps the identification of the provenance of the primary glass. Furthermore, it is shown how optical spectroscopy represents a powerful method for first-step screening of large set of glass fragments, in order to reduce the number of samples to be analysed chemically.

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## **V23 ICP-AES Analysis of Ancient Glass from Qiemo Tomb Sites on the South Line of the Silk Road in China**

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Zhaguluke tomb sites are located at Qiemo country, the south boundary of Tarim Basin in west of China. Qiemo was an ancient country on the ancient south line of Chinese Silk Road between the 1<sup>st</sup> ~5<sup>th</sup> century AD. These glass finds from tomb sites reflect on the relationship between China and the West. These glasses including only colorless glass vessel and more than 100 monochrome beads in brown, blue, dark blue, yellow, green, white and gold color have been excavated in 1998. A project has been carried out to analyse about 40 samples by X-ray fluorescence (XRF), laser ablation inductively coupled plasma atomic emission spectrometry (LA-ICP-AES) and scanning electron microscope (SEM-EDS). New analysis has discovered that glass finds are especially valuable as a source of information on chronology, economic, cultural and social history of ancient Qiemo country.

According to chemical composition of the colorless glass cup by ICP-AES analysis, it shows that the cup was probably produced in a glass workshop of the syro-palestine coast during the Roman period. Manganese (Mn) was used as decolorant for colorless glass working. Moreover, facet-cut decoration on the surface was typical style of Roman glass. A few delicate trace lines between the facet-cut patterns seemly indicate the process of manufacture or use of the cup. All of evidences have shown that the glass cup was probably produced in east coast of Mediterranean Sea during the Roman Period and was transferred to the Qiemo country alone the south line of the Silk Road.

About 40 monochrome beads were scientifically studied by LA-ICP-AES with corning glass as reference. According to results of chemical composition by LA-ICP-AES, those beads are all soda-lime silica glass which possibly came from the West and were divided into two groups of HMG (high magnesia glass) and LMG (low magnesia glass). Majority of beads are plant ash glass which suggested possibility of production in Sasanian period. However, those LMG beads which shows transparent dark blue appearance were probably close to characteristic of Roman glass.



## **V24 Pottery on the Periphery: A Technological Characterization of Stonepaste Ceramics from Middle Islamic Tall Dhiban, Jordan**

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Stonepaste ceramics were recovered from the final phases of the Middle Islamic agro-pastoral settlement of Tall Dhiban, Jordan, radiocarbon-dated to the late 14<sup>th</sup> and early 15<sup>th</sup> centuries CE. In the previous century, Dhiban's economy intensified through the Mamluk Empire's increased commercial interests in the Levant (Porter, 2010; Walker, 2011). This project, using Dhiban as a case study, proposes that a technological investigation of stonepaste technologies can offer insight into the transformations of local and regional craft economies under these new economic traditions. From what technological traditions did producers draw in their vessel crafting, and how standardized were their vessels? Variation in stonepaste recipe may indicate that Dhiban residents exercised greater choice in pottery acquisition than might be expected for a rural settlement in an imperial periphery. This research aims for a comprehensive chemical and structural characterization of the glazes, glaze-body interaction zones, and bodies of the stonepaste specimens and, through comparisons among specimens, a determination of the standardization of the imported stonepaste. Comparing this data to known technological attributes of stonepaste ceramics from other Middle Islamic settlements will allow Dhiban's consumption practices to be situated in a broader context. Initial study of the Dhiban stonepaste specimens with optical microscopy has revealed glazes of varying thicknesses, from 0.3 to 0.7 mm, as well as apparent glaze-body interaction zones. Reflectance Transformation Imaging (RTI) illustrated that the extent of fusion between the glaze and body of the stonepaste is variable even within a particular specimen. Bulk compositional analysis of specimens (n=10) with Energy-Dispersive X-Ray Fluorescence Spectrometry (ED-XRF) showed that glazes are Ti, Cr, Mn, and Cu-based, but despite an orientation of specimens to their glazed sides for analysis, body and glaze compositions were largely irresolvable. Following the suggestion of Freestone *et al.* (1985), the significant presence of P<sub>2</sub>O<sub>5</sub> in the bodies (ca. 2-7 wt%) may have arisen from interaction with the stonepaste's depositional contexts. Field Emission Scanning Electron Microscopy (FE-SEM) has been employed to compositionally characterize the glaze and glaze-body interaction zones of sections of the stonepaste specimens. Another aim of this analysis is to evaluate microstructure, paying attention to the relationship between vitreous phases and quartz particles, following Tite *et al.* (2011), and characterizing the presence of phosphate. Thinned samples of the stonepaste were ion-milled for diffraction analysis with Transmission Electron Microscopy (TEM), permitting the identification of individual glaze compounds through comparison with known lattice spacings (Groot *et al.*, 2006).

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## V25 X-ray Computed Tomography Applied in the Study of Ancient Ceramics Objects

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X-ray Computed Tomography (CT) is a non-destructive technique useful to visualize the inner features within solid objects, helping the analyst to obtain information on their 3-D geometries and properties. Up to now, X-ray CT was successfully applied in various fields, such as medicine, industry, material science and - last but not least! - archaeometry.

Recently, a specially designed X-ray computed tomographic device has been developed at “Horia Hulubei” National Institute for Nuclear Physics and Engineering, with the purpose of performing imaging studies of cultural heritage artifacts made out of low-Z materials (ceramics, clay, bone, wood) (Constantin et al., 2010). This CT device contains a 160 kV X-ray tube, a sample positioning system and a Varian PaxScan® flat panel detector. Using this home-made CT device and the corresponding reconstruction algorithms, small archaeological objects (20×20×20 cm<sup>3</sup>) made out of light materials can be imaged with a spatial resolution around 300 μm.

This communication will present the results of the studies performed with the help of this CT machine on several ceramics archeological objects belonging to different collections and deposits of Romanian museums and dating from the Neolithic, Chalcolitic and Bronze Age periods. By performing the X-ray tomographic scans, the internal structure of the investigated objects was put in evidence, revealing - in some cases - their inner content. Thus, for the so-called “rattles”, enclosed mobile or fixed balls/bodies were observed, while in the case of some ceramics objects, cracks or fissures in their structure were observed – information potentially useful for conservation/restoration purposes. For some composite artifacts – e.g. statuettes representing Fertility Goddesses - the analysis of the obtained tomographic images provided some hints on the way the ceramics objects were manufactured.

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## **V26 Macroscopic and Microscopic Aspects of Polishing Ceramic Pots Using Water Worn Pebbles**

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A famous pottery called “Marginea ceramics” is produced nowadays in small workshops in NE Romania. It is black on the surface and grey in fresh break. The decoration is rather simple and is restricted to few shining lines, obtained by circular movements using a water worn pebble or “polishing stone”. As many of the prehistoric potshards found at several excavations show polishing, our study focused on methods that may have been used to produce this effect. Stereoscopic microscopy, polarized light optical microscopy, scanning electron microscopy (SEM) and back scattered electrons (BSE) images were employed in the study.

The black Marginea ceramics is obtained from a Miocene illite-rich clay, in quite primitive kilns, with no control of temperature. The raw material consists of illite, muscovite, feldspar, quartz, chlorite/kaolinite, Fe oxi-hydroxides and carbonate.

Rubbing the surface while the clay is still in the so-called “leather hard stage” induces the smoothing of the rough surface, closure of the pores and a more compact ceramic body. The polishing also aligns the platy minerals (e.g. mica) within a thin layer, which causes the shiny surface of the lines drawn by water worn pebbles. Because the surfaces of the platy minerals are aligned, most of them will reflect light at the same time when viewed from the right angle. Stereoscopic microscopy of the pot surface shows fine striations due the movement of the polishing stone. Although stones used for burnishing tend to become apparently very polished themselves, under the microscope it is possible to see many parallel scratches on their surface. The more used stones had the most scratches. Simple unused river stones were less polished and had far fewer scratch marks.

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## **V27 The Origins of Pottery Technology in the Cantabrian Region (5000 cal BC). Mineralogical and Geochemical Analysis**

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The aim of this paper is to discuss the results of a programme of systematic analysis of the earliest pottery production in coastal northern Spain (Asturias, Cantabria and the Basque country), dated to the fifth millennium cal BC. Our approach departs from the understanding of the pottery as manufacture, the result of a technological fabrication process (*pottery production sequence*) which covers a set of operations that transform a raw material into a product with certain physicochemical properties which practically eliminate its original characteristics.

The main pottery ensembles come from three cave sites dated to the fifth millennium cal BC, located along the region: from west to east, Los Canes (Asturias), Los Gitanos (Cantabria) and Kobaederra (Biscay). In each case, a characterisation of the manufacturing sequences has been proposed. Moreover, an attempt has been made to determine whether the raw materials were local or foreign origin, in order to identify technology transfers. Finally, the relationship between the appearance of this technology and other economic and social processes in the framework of the transition to the Neolithic is also explored.

The samples were studied considering each sherd as a unit of analysis, since its fragmentation impeded the reconstruction of the vessels. As a preliminary macroscopic analysis, they were classified attending to their technological, morphological and decorative features. The selection of samples for later petrographic analysis was performed based on the observed technological variability. The thin-section petrographic analysis focused on the description of the texture and mineralogical features of the samples. A distinction was made between the detrital fraction, naturally present in the sediment, and materials that were added intentionally (temper). A second selection of samples followed, in this case for mineralogical analysis, using X-ray diffraction (XRD) and chemical analysis, using Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS).

The main conclusions reached in the study are related to aspects of the pottery manufacturing sequence and their contribution to the study of these societies during the fifth millennium BC in this geographical area.

## **V28 Distant Neighbours: Early Helladic Ceramic Production in the Nemea Valley and Corinth**

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Immediately preceding the foundation of the Palaces of Bronze Age Greece, the third Millennium BC has received considerable attention. A key aspect of this research has been the intense discussion regarding the nature of and changes in ceramic technology across the Aegean, relating trends to wider discussions of societal organisation and development.

Forming part of a broader programme of analysis of Early Bronze Age ceramics from Korinthia and the Argolid, this paper examines results of macroscopic and petrographic analysis of Early Helladic II ceramic material from Corinth and Nemea. Analysis was undertaken on 144 samples from the Gymnasium area, Ancient Corinth, 28 samples from Nemea Valley Archaeological Project Survey Site 204 and 114 samples from the settlement site of Tsoungiza, Nemea.

Located less than 19km from each other, these two areas show significant variability in technological practice, as revealed through analysis by thin section petrography, between Corinth and the sites in the Nemea Valley. Whilst all sites have groups which testify to local production, the Gymnasium material at Corinth displays considerable variability in paste recipes, including acid igneous imports most likely from the island of Aegina, located c.55km to the East.

In contrast the Tsoungiza and NVAP material has more standardized paste recipes, derived from a limited number of geological resources/production centres. This evidence suggests that the EHII population of Corinth had access to a wider variety of producers than the sites within the Nemea Valley and was participating in wider networks of exchange, with consequent implications for our understanding of the size and role of these settlements.

These results demonstrate the variation in craft practice and participation in ceramic exchange that can be found between contemporaneous small-scale communities who, whilst sharing the same typological repertoire suggestive of shared consumption practices, were participating in different networks of production and exchange.

## V29 'Marbrite Fauquez' Opalescent and Marbled Glass: A Material-Technical Study

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The development of innovative materials like *Marbrite Fauquez* opalescent glass characterize the early 20<sup>th</sup> century. The Walloon glassworkshop S.A. *Verreries de Fauquez* produced this marbled glass from 1922 onwards. Due to their low maintenance requirements and their appropriate properties in terms of hygienic aspects, these mass coloured glass plates – available in about 36 shades – were preferably used as finishes for bathrooms, kitchens, hospitals, stores,... Later on, Belgian architects like Henry Lacoste (1885-1968) have applied *Marbrite Fauquez* glass as decorative panels in external façades. Since the production of *Marbrite* glass stopped in the 1960s, replacement of its applications in the framework of restoration procedures is almost impossible. Moreover, knowledge on the detailed composition and production techniques of this opalescent glass was till now very limited. Hence questions arise on how to deal with these at that time innovative glass plates as part of young built heritage during restoration. In order to improve the existing knowledge on composition, production and application techniques of these colourful finishes, a material-technical study was conducted based on several sources including patents, interviews with the former director of the glassworks, literature, building specifications, building plans, journals, advertisements, etc. were considered and valued. Moreover, onsite lifted samples of *Marbrite* glass were submitted to a laboratory investigation using optical and scanning electron microscopy. The results of both historical sources and lab analyses revealed crucial information about the original preparation formula and application method. This paper discusses the origin, development and composition of *Marbrite*, a peculiar Belgian opalescent glass. The research aims to improve the existing knowledge on *Marbrite* glass in order to develop and optimize future repair and renovation techniques.

### **V30 Development of an Appropriate Isolation Procedure for Boron from Soda-Silica-Lime Glass**

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Provenancing of the raw materials used to produce ancient glass is of paramount importance, not only to gain more insight into the glass production process, but also to understand trade routes in ancient times. The raw materials used to manufacture Roman natron glass include beach sand (silica source) and natron as a flux. Mainly based on the writing of Pliny the Elder and geochemical evidence (Shortland et al., 2006), the major source of this flux is thought to be the evaporite deposits of the Wadi Natrun region in northern Egypt. However, Pliny also suggested other possible sources, such as lake Pikrolimni in Greece (Dotsika et al., 2009). Although geochemical research related to these natron sources was already carried out, no isotopic analysis has been done so far. Isotopic analysis has already been proven to be a useful tool for provenance determination of glass raw materials (Degryse & Schneider, 2008). One element present as a trace element in glass (Wedepohl et al., 2011) and in natron is boron. The isotopic composition of boron is easily affected by physical processes, such as precipitation and evaporation (Vengosh et al., 1992). Consequently, boron isotope ratios, determined by means of multi-collector ICP-MS (MC-ICP-MS), could possibly be used to discriminate between different sources of flux, as different lakes can have a different boron isotopic signature. To accurately determine isotope ratios with MC-ICP-MS, adequate correction for mass discrimination is necessary. Mass discrimination is a phenomenon causing the ion transmission efficiency to be different for different analyte masses. Hence, the measured ratio is not the true ratio. Mass discrimination is not only influenced by the mass of the ions analyzed but also by matrix elements present. As a result, separation of the analyte from the matrix is recommended (Vanhaecke et al., 2009). For this reason, a separation procedure for the isolation of boron from glass was developed. This procedure uses three different ion exchange columns, each providing a boron recovery of about 99% and thus avoiding problems related to possible on-column fractionation. B was isolated from glass standards and a few Roman natron glass samples and analyzed for its isotopic composition and these results will be presented.

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**V31 Chinese Porcelain Ordered for the Portuguese Market During the 16<sup>th</sup> Century: Study on the Compositional Differences by Neutron Activation Analysis and Indirect Provenance Issues**

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The existing documentary history of Chinese porcelain ordered for the Portuguese market (16<sup>th</sup> cent.) is reasonably advanced; nevertheless detailed laboratory analyses able to reveal new aspects like the number and/or diversity of producing centres involved in the trade with Portugal.

Blue and white porcelains are undoubtedly considered the most important of the Chinese porcelains produced along the various dynasties, from the Yuan, Ming to the Qing. One of our purposes is to locate the origin(s), at a regional scale, of the porcelains found in Lisbon and Coimbra archaeological excavations. To do so, it is assumed that there is already a chemical fingerprint of Chinese kilns. On the other hand, if not possible to attain such an ambitious goal, at least contribute to figure out the diversity of productions centers involved in the commercial trade to Portugal.

Chinese porcelain fragments collected during recent archaeological excavations from Portugal (Lisbon and Coimbra) were analyzed by means of instrumental neutron activation analysis (INAA) of core samples taken from the ceramic body, avoiding contamination from the surface layers constituents. Major (a few) and trace elements were determined with very good accuracy and precision (Na, K, Mn, Fe, Sc, Cr, Co, Zn, As, Ga, Br, Sb, Rb, Cs, Ba, La, Ce, Nd, Sm, Eu, Tb, Dy, Yb, Lu, Hf, Ta, Th, U). Samples and standards were irradiated together in the core grid of the Portuguese Research Reactor (ITN, Sacavém) for 2 minutes (short irradiation) and seven hours (long irradiation).

The obtained results point to the existence of a trend of two main chemical groups in Lisbon and Coimbra samples. Besides a few sub-groups appears to exist. A comparison study with several well-known kilns from several regions of China (i.e. Fujian, Yunnan, Jiangxi provinces) during the Ming dynasty was performed. The Chinese porcelain ordered for the Portuguese market during the 16<sup>th</sup> century was done in different kilns/raw materials, and a few allocations may be established.

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### **V32 Chemical Signatures of Coimbra and Lisbon Early Portuguese Faience Productions (17<sup>th</sup> – 18<sup>th</sup> Cent.)**

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The history of the Portuguese faience (PF) has been essentially made on the basis of macroscopic observations of known objects and documentary research. The production of PF in the 17<sup>th</sup> century till the 1st half of the 18<sup>th</sup> century – with particular characteristics and with an identity that distinguishes it from the European contemporary production - has come to be organized on the basis of stylistic resources that are clearly insufficient.

Nevertheless, there are some questions that matters to clarify, namely the differentiation of two great centers of production of faience in Portugal (Lisbon and Coimbra). The setting up of “signatures” to define each production centre is very important, as it can be usefully applied in future provenance/attribution studies of similar ceramics. Early PF samples had been selected from archaeological findings from Central Lisbon excavations (yielded by the company Era Arqueologia SA) and Coimbra excavations (at the Museum of Santa Clara a Velha), and also from the National Museum of Ancient Art collection and excavations. The analyzed shards belong to archaeological contexts, like dumps, collapsed structures of buildings – in the case of Lisbon it was possible to establish the chronological goal of the 1755 earthquake, with materials prior to the earthquake and destroyed by him, and others post-earthquake. Chemical analyses were done by instrumental neutron activation analyses (INAA). Samples and standards were irradiated together in the core grid of the Portuguese Research Reactor (ITN, Sacavém).

Obtained results point to a good chemical based identification of PF, allowing a clear chemical differentiation of Coimbra and Lisbon faience productions, as well as, the definition of two compositional groups for Lisbon production. This geochemical approach prove former assumptions based on the appearance of the faience from Lisbon and Coimbra. Chemical analyses of pastes confirmed the production of faience with “rendas” (laces) in Lisbon, usually assigned to Coimbra. No special correlation between stylistic / decoration and compositional group was found. The chemical homogeneity found for each compositional group may reflect the use of standardized raw materials and probably also recipes for faience production of the studied period.

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### V33 Chemical Fingerprinting of Hungarian and Slovakian Obsidian Using three Complementary Analytical Techniques

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The natural volcanic glass obsidian is one of the classical objects of archaeometrical analyses. Reliable provenancing by means of its highly specific chemical composition, the “chemical fingerprint”, can provide information about economy, policy and the social system of ancient societies. Although Mediterranean obsidian have mainly been the focus of characterization since the pioneer work of Cann & Renfrew (1964), provenancing of Central and Eastern Europe obsidian sources attracts increasing attention in the past decades. Fingerprinting of Hungarian and Slovakian obsidian sources is of great interest especially for Central European sites where obsidian has been widely used (Williams-Thorpe et al., 1984; Kasztovszky et al., 2008; Biró, 2009). The application of three complementary analytical techniques on the same set of raw material samples allows both, a more complete characterization of obsidian sources and a comparison of analytical results. The aim of this multi-methodical approach is to apply three different analytical methods, in particular: Instrumental Neutron Activation Analysis (INAA), Ion Beam Analysis (IBA) comprising of Particle Induced X-ray Emission (PIXE) and Particle Induced Gamma-ray Emission (PIGE), Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS), to detect a maximum element spectrum and to compare element concentrations determined with at least two analytical techniques. This way a check of self-consistency of analytical results is possible. Furthermore, it allows the identification of a maximum of compositional differences between Hungarian and Slovakian sources by revealing the most characteristic “chemical fingerprint” composed of more than 40 elements. For this study, NAA, IBA and LA-ICP-MS measurements are scheduled to be applied to 25 raw material samples from sources from Hungary and Slovakia in cooperation with the Natural History Museum Vienna (Hammer, V. and Seemann, R., Department of Mineralogy and Petrography) and the Vienna Lithothek (Trnka, G., Department of Prehistoric and Protohistoric Archaeology). Up to now, IBA studies have already been carried out using the external 4 MeV proton beam of the 6 MV Tandem accelerator of the Ion Beam Centre of Helmholtz-Zentrum Dresden-Rossendorf. Further NAA investigations will be performed at the TRIGA Mark II 250 kW research reactor of the Atominstitut in Vienna. LA-ICP-MS measurements will be conducted using the Thermo Element 2 ICP-MS coupled to an ArF gas Excimer laser system at the Aberystwyth University.

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### **V34 QXRD, ESEM and DSC Applied to the Study of Pottery Manufacturing Process from Tappeh Zaghe (5100-4900 BC), Iran**

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Ceramic materials from Tappeh Zaghe (5100-4900 BC) reflect careful selection of raw material for pottering in the prehistoric time. The ceramics from different archaeological strata contain distinct manufacturing processes due to usage of different admixtures. They are two types of ceramic products which have been painted with diagonal lines as typically Zaghe, and classified as reddish ceramic (4900 BC) and earth-colored ware (5100). 50 samples from different archaeological strata were subjected to mineralogical, chemical analysis to determine the manufacturing as well as the proportion of their constituent phases for modeling the pottery processing in the two periods of Zaghe. Quantitative X-ray diffraction employed for determining the crystalline phase constituents that calculated also with Rietveld refining method for better crystal structure fitting from measured peaks to calculated peaks in the ceramic's texture. Differential scanning calorimetry utilized for determining decomposition of minerals during the time as well as firing temperature. The results matched with polarization microscopy methods to obtain mineralogical-petrological conclusion (Emami & Trettin, 2010). Environmental scanning electron microscopy concluded the distribution of the major, minor and trace elements in the ceramic's texture, including mapping of them on the ceramics' body. These samples embrace two different types according to CaO- and SiO<sub>2</sub> constituents. The CaO-rich body is covered with well processed crystalline quartz aggregates on the surface like glazing. The CaO-poor ceramics proved impact texture on the surface and the quartz aggregates are processed very carefully in the matrix. The firing temperature was about 900-1050°C. The pottery creation from Zaghe can be well documented by their chemical compositions, which permit to confirm attribution according to their stylistic contentions.

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### **V35 The Analysis of the Viking-Age Glass Gaming Pieces from the Graves and Settlements in Russia and Ukraine**

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Glass gaming sets and single counters were found in the rich graves and on the settlement sites in Norway, Sweden, Russia and Ukraine dated to the 9<sup>th</sup> -10<sup>th</sup> centuries AD. The number of gaming pieces in each archaeological find varies from one to twenty-six. All counters from Eastern Europe apart from the 'king pieces' are of similar size and form, combining inverted truncated cone and hemisphere. They were produced of translucent purple-violet, turquoise, dark green, blue-green or green-olive glass and decorated by gilding or applied spiral glass threads of the second colour. The 'kings' differ in colour, size or shape. Despite the wide spread of board games in Byzantium, Islamic Orient and Medieval Europe, the origin of glass counters is still unclear. This paper focuses on the technological study of the Viking-age glass gaming pieces and analytical study of their chemical composition. Gaming pieces were produced by moulding into open stone or earthenware mould. Standard height of the items indicates to standard portions of glass filled each mould. Considerable part of the counters shows negative marks of a punty rod slightly chipping the top of hemispherical surface. The samples available for non-destructive analysis include 25 gaming pieces. A micro-XRF Tornado spectrometer fitted with a Rh-tube and extra large sample chamber (Bruker AXS) was used to analyse the glass composition in a vacuum. Quantification was performed by FP calculations. Obtained results are not completely representative for the bulk composition of the glass because of alkali leached out from surface leaving behind a body with an outer layer enriched with silicon dioxide. All samples belong to entirely homogeneous low alkali - high calcium group. The presence of P<sub>2</sub>O<sub>5</sub> at level ranging from 0.3% to 0.9% coupled with K<sub>2</sub>O is clear indication of the use of ashes of inland trees appeared in northwestern Europe from the late 8<sup>th</sup> – 9<sup>th</sup> centuries (Henderson, 1993; Wedepohl, 2003). Discussing the origin of the glass gaming pieces we should exclude small counters of opaque glass produced by the provincial Roman workshops up to the end of the 4<sup>th</sup> century AD. The earliest Viking-age glass counters from Norway (Gunnarshaug) and Sweden (Birka) dated to the 9<sup>th</sup> century (Lindquist, 1984). They are totally different from the Roman gaming sets. Gaming pieces of glass dated to the 9<sup>th</sup> -11<sup>th</sup> centuries were found in Iran, Iraq, Afghanistan and Egypt (Kröger, 1995; Carboni, 2001). However, their conical form and mosaic technique differ from the Viking-age glass counters. Chemical composition of the glass gaming pieces from Russia and Ukraine probably confirms assumption about their origin from northern Continental Europe (Arbman, 1937).

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### **V36 Characterization of Late Iron Age Ceramic Sherds and Clay Samples from Zamala (Northern Cameroon, Diamaré Division, Central Africa)**

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Zamala is a village situated in the Far Northern Region of Cameroon at about 40 km NW of Maroua (Capital of the Region) in the Diamaré Division. The aim of this work is to characterize selected proto-historical ceramic sherds and clays of this locality in order to determine the pottery technology. By the application of analytical methods such as X-ray diffractometry, X-ray fluorescence, optical microscopy and differential thermal analysis, an answer will be given to questions about the provenance of the ceramics (local or imported), the temper added or not, the moulding techniques and the firing processes. Sixteen ceramic sherds dated to the period 1289-1614 AD (Delneuf & Otto 1992) and two clay samples from pits in the vicinity of the archaeological excavation were studied. The chemical composition of the ceramics shows a homogeneous group of 15 sherds and one isolated sample, mainly due to its content of silicon oxide and iron oxide. The homogeneous group presents a silicon oxide content from 69 to 73 wt.% and an iron oxide content from 2.8 to 4 wt.%, whereas the isolated sample presents a silicon oxide content of 61wt.% and iron oxide content of 7.5 wt.%. The mineralogical composition of all the ceramics is similar: quartz, K.feldspar (microcline), plagioclase and mica (biotite). The firing temperature is estimated under 900°C due to the presence of mica (biotite). The matrix presents dark grey or brown color and a combination of brown and dark grey color due to the alternation of oxidizing and reducing firing conditions. Bonfire and pit firing systems are suggested. The grain size distribution of the aplastics inclusions is serial, no temper was added during clay paste processing. The irregular shape of the pores in the ceramic bodies suggests a hand modeled process.

The chemical comparison between ceramics and the two clays samples studied shows a clear correspondence. A local production of the ceramics in this region is assumed. The mineralogical phases observed in the clays are quartz, K.feldspar (microcline), plagioclase, mica (biotite) and kaolinite. The absence of kaolinite in the ceramics confirms a firing temperature of the ceramics above 500°C according to the results of the X-ray diffraction of the clays samples fired between 400-1000°C.

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### V37 Characterization of Obsidian Sources from Colombia Using Electron Spin Resonance

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A previous step for determinate the provenance of archaeological obsidians is to characterize the sources for the physical properties or geological parameters which will be measured in the artifacts. Because of iron presence as paramagnetic ions of  $\text{Fe}^{3+}$  in obsidians, is possible to use electron spin resonance (ESR) on geological samples in order to characterize different sources (Duttine et al., 2003). In this work, about thirty obsidians coming from Rio Hondo, Rio Negro and Las Balsas, places linked to the volcanic structure of Paletará caldera, SW of Colombia, are studied by electron spin resonance and they are classified using ESR intensity and line width as parameters. This work is the first effort to use the ESR technique in studies of provenance made within the country, and this is the first step in order to determinate the provenance of obsidians found in the archaeological site of Mesitas, related to the culture of San Agustín, one of the most important cultures of the prehispanic past in Colombia.

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### **V38 Electron Microprobe and Petrographic Analysis of Later Prehistoric Granodiorite-Tempered Pottery from the East Midlands, UK**

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Petrographical studies of later prehistoric pottery from the East Midlands in the UK have identified several distinctive fabrics characterised by angular granitoid inclusions that may derive from the Mountsorrel Complex and South Leicestershire Diorites of Leicestershire (Knight, 2002; Knight et al., 2003). The inclusions suggest granodiorite and quartz-diorite source rocks but, given the coarse-grained nature of these rocks, the few crystals that are present may not always be sufficiently representative to enable robust comparisons to be made by thin section analysis alone. A recent review of later prehistoric granitoid-tempered pottery suggested that the results of thin section analysis should be tested by chemical analyses, although a technique based on whole rock analyses also would not work due to the small size of the pottery inclusions (Knight et al. 2003). This paper presents the results from a study of later prehistoric pottery and rock samples using a combination of thin section petrography and electron microprobe analysis of single crystals. The electron microprobe enables the chemical composition of the individual minerals or phases within the pottery inclusions to be determined, allowing a direct comparison with the composition of similar phases or minerals present in the rock sources and alleviating the problem of representativeness required for whole rock analyses. This was an essential requirement since the individual mineral phases, especially those comprising perthitic alkali feldspars, may only be in the order of a few microns in width. By combining the results of these two analytical approaches, this project has established that it is possible to distinguish between the different outcrops of granitoid rocks in the study area on the basis of individual minerals rather than by whole rock analyses. The project has demonstrated that the inclusions in the pottery originated from a variety of granitoid rock sources, and that it was possible to provenance the inclusions to particular outcrops of granodiorite or quartz-diorite within the East Midlands. Inter-regional exchange has rarely been demonstrated convincingly in the East Midlands before, mainly because diagnostic inclusions that can be tightly provenanced are comparatively rare in prehistoric pottery. These results add significantly to the growing evidence for long-distance ceramic exchange networks in first millennium BC Britain.

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**V39 Archaeometric Characterisation of Late Roman Amphorae from Sant Martí d'Empúries (Catalonia, Spain)**

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The object of this contribution is to present the first results of the archaeometrical characterisation of Late Roman amphorae recovered at the site of Sant Martí d'Empúries. This is one of the case studies within the framework of a larger project on the characterisation of Late Roman amphorae in Eastern Spain and the Balearic Islands (AD 200-400).

This site, located at the southern end of the Gulf of Roses (Catalonia, Spain), is the only sector of the ancient city of *Emporiae* that continued to be occupied during Late Antiquity, corresponding to the so-called *Palaea Polis*. Archaeological excavations conducted between 1994 and 1996 uncovered a substantial body of material of great relevance for understanding the settlement in this place. A continuity of occupation throughout Late Antiquity was documented, as well as an intense port activity, as reflected in the arrival of all the most commonly traded products in the western Mediterranean during this period, including fine wares (ARS D, Late Roman C, DS.P, Late Hispanic Terra Sigillata), coarse wares, cooking wares and amphorae. Amphorae constitute the majority of the ceramic materials recovered from the excavation of a rubbish dump dated to the 5<sup>th</sup> century AD and a series of building structures, silos and burials dated to the 6<sup>th</sup> and 7<sup>th</sup> centuries AD.

A total of 51 amphorae samples, including mainly African and Eastern Mediterranean materials have been analysed using a combination of techniques, including X-ray fluorescence, X-ray diffraction and optical microscopy by thin-sections analysis. The results offer a first insight to the amphorae fabrics and their provenance and subsequently to the commercial trends of San Martí d'Empúries from the 5<sup>th</sup> to the 7<sup>th</sup> century AD.

## **V40 Application of TOF-SIMS to Ancient Glasses**

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ToF-SIMS analysis has been carried out on a variety of Late Bronze Age to Byzantine glasses from the site of Nuzi in modern Iraq and from Lydian to Roman glasses from Sardis in Turkey to investigate isotopic ratios of elements in the glass. The interest in using ToF SIMS to measure isotope ratios is that it is possible to obtain the very low mass elements such as Li, B and C, and O in addition to the higher mass elements such as strontium and lead. All elements can be collected with high mass resolution, and specific areas of interest can be easily selected. Ion distributions may also be mapped with a lateral resolution of 300nm.

Cross sections of the glass samples were mounted in resin and polished to 1 micron grade finish. Mass spectra were then collected from the sample surface in the glass region and in the corrosion crusts. The same ion beam dose was applied in each analysis and was kept low to maintain 'static' conditions. The data was collected from a range of coloured glasses, including amber, white, blue, black and multicoloured (blue-yellow-red and black-yellow). In the multicoloured glasses, measurements were made in the different coloured regions of the glass.

Isotopic ratios for Pb, Sr and O were measured. Initial results indicate a variation of ratios with the different coloured glasses, suggesting production of the primary glass at different glass making sites. However, Nd isotopic ratios could not be measured by ToF-SIMS in these samples since the low concentration of Nd in these glasses and the low ion yield for Nd results in poor sensitivity for this particular element. Variations in transition metals within the glasses were also compared.

## V41 The Mechanical Properties of 16<sup>th</sup> Century Transport Jars from Panamá and Seville

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The sixteenth century is the beginning of the colonization of the American territories as an economic enterprise supported by the Spanish Monarchy. Pottery artisans settled in Seville, Europe's gateway to America. Many workshops were specialized in certain types of ware. The guild of *botijeros*, for example, manufactured large transport jars destined for the commerce of goods with the new foundations in America. One of these foundations is Panamá (Central America), which was established in 1519 and lasted until 1671 when it was destroyed in a raid led by Henry Morgan. Excavations at Panamá Viejo have brought to light European transport jars but also vessels of a different type, *contenedores de pasta roja* (red paste transport jars). The latter appear to imitate European vessels but are manufactured with a local ceramic paste and interpreted as a result of acculturation in Panamá.

Transport jars must be able to keep their contents safe to the final destination. During transport, they should be able to withstand stresses related to weight loads, e.g. from overlying vessel layers, but also stresses arising from collisions, caused by movements of the ship. Fracture strength and toughness describe a material's ability to withstand such mechanical stresses. Fracture strength is related to the stress a material can be exposed to until crack initiate, while toughness gives a measure of the energy required for both fracture initiation and propagation. In order to assess the mechanical performance of transport vessels found at Panamá Viejo, 29 sherds chemically characterized from Seville and 18 sherds from Panamá were subjected to material tests (fracture strength and toughness). In addition, material parameters which would allow making inferences on a ceramic's mechanical performance, such as firing temperature, porosity, and amount of aplastic inclusions were also determined. Finally, the vessel's behavior during transport was simulated by Finite Element Analysis.

Preliminary results show differences in the mechanical performance of Seville vessels as opposed to Panamá vessels. The results of mechanical tests are presented and discussed in relation to the different manufacturing choices which are observed in the production of the two vessel types. The case of Panamá workshops, which appear to have been copying Seville productions is examined, taking into account that while technological choices in manufacture such as the addition of temper or use of different firing regimes affect the mechanical properties of a vessel, there is a plethora of potential reasons behind such choices, functional and other.

## V42 Distinguishing Handmade and Wheel Thrown Pottery Using X-Ray Diffraction

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The potter's wheel is one of the most significant innovations in the history of ceramics. Its adoption has been related to increased craft specialisation and changes in social organisation. However, our understanding of the introduction of the wheel in many cultures is unclear. Characteristic surficial evidence of the wheel has frequently been obscured by finishing techniques, and devices such as the tournette blur the distinction between hand-made and wheel-thrown fabrication methods. Methods proposed to distinguish fabrication techniques include thin-section examination, macroscopic examination and in particular X-radiography. However, all have limitations, which may include a perceived element of subjectivity in the method, the requirement for whole pots or large sherds for examination, and the need for large elongate inclusions or voids in the ceramic fabric which track the applied fabrication pressures. New approaches are required, which are widely applicable, require relatively small and easily transportable sherds, and which offer the possibility of an objective assessment of the likelihood of wheel use.

We have piloted the use of X-ray diffraction in the assessment of pottery fabrication methods, based upon the preferred orientation of platy minerals (e.g. sheet silicates) in the pottery fabric. The relative intensities of the reflections from the 001 crystallographic plane, parallel to the main (perfect) cleavage plane of the mineral, and crystallographic directions perpendicular to this (including 110, and 020 for most sheet silicate minerals selected for this study), may be compared with the relative intensities in randomly oriented grains from powder diffraction data. When analysed along the plane of preferred orientation (e.g. the surface of a wheel-thrown pot), the relative intensity of the 001 reflection increases with increasing alignment of the mineral particles. We have cut sections of archaeological and replica sherds, made using coil, pinch, slow and fast wheel methods, and analysed along six orientations at and below the surfaces of the vessels. Selected reflections were measured using a PANalytical X'Pert Pro X-ray diffractometer (XRD), operating with  $\text{CuK}\alpha$  radiation at 40 kV voltage and 30 mA current, 5mm divergence and 3mm receiving slits, in the 7-26°  $2\theta$  range, scanning at 120s/step, and using a RTMS X'Celerator detector. Results indicate strong orientation at the surfaces of both wheel-thrown and several of the hand-made pots. However, in the centres of the walls, there are clear differences between forming methods. These results call for a systematic evaluation of the enhanced capabilities of non-destructive synchrotron X-ray diffraction in the measurement of preferred orientation in ceramics.

#### **V43 Non-Destructive Analysis of Chinese Porcelain from the Mexico City Colonial Period (XVI to XVIII Centuries)**

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In the historical center of Mexico City, near the main square (Zocalo), an important amount of pottery and other materials were discovered in recent archaeological excavations. Among them, numerous postsherds of Chinese porcelain corresponding to the colonial levels of the archaeological site were recovered. From the style and features of the decorations patterns and the macroscopical features of the pieces, they correspond to XVI to XVIII centuries and various workshops of China. Porcelain objects were highly appreciated in the Mexico's colonial period. They were traded from Filipinas to Spain and Europe across the Pacific and Atlantic oceans though the New Spain.

In order to determine the groups of similar manufacturing and origin, a non-destructive elemental analysis was performed. Considering the style of the postsherds, a set of two hundred pieces was analyzed by portable X-ray Fluorescence (XRF). From the XRF results of white glaze and ceramic paste, a representative selection composed of more than 60 pieces was studied by Particle Induced X ray Emission (PIXE). This analysis included ceramic paste, white glaze, and colored areas of the postsherds. The results of this analysis and a comparison with reported data for the Chinese porcelain are presented in this work. Also, some Majolica local imitation pieces were studied in order to show the material and technical differences in the ceramic production.

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#### V44 Geochemical Investigation of Roman Coloured Glass from North-Eastern Italy

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The Roman period saw a prodigious use of glass in domestic, industrial and funerary contexts. The predominant type of Roman glass is natron glass, produced in so called 'primary' workshops with siliceous-calcareous sands and natron as flux. This raw glass was broken up and traded throughout the Mediterranean as chunks and then remelted and shaped into vessels and other objects in 'secondary' workshops (Foy et al., 2000). The location of primary production in the Hellenistic and early Roman world is still up for intense debate and some authors have suggested that it also took place outside the 4<sup>th</sup>-8<sup>th</sup> primary workshop identified in Egypt and the Levant (Leslie et al., 2006; Degryse & Schneider, 2008). This study is focusing on 34 intensely coloured glasses, dating between the 1<sup>st</sup> and the 3<sup>rd</sup> century AD, and coming from the Archeological Museum of Adria (Rovigo), one of the most important port in northern Adriatic area from the 6<sup>th</sup> century BC until the 2<sup>nd</sup> century AD. The glasses fall into five distinct colour groups: blue, amber, purple, emerald green and black; a great variety of forms is represented both casted and blown. A combined approach, chemical and isotopic, was employed to investigate the type and the provenance of raw materials used in glass production. All the glasses are soda-lime-silica in composition. The vast majority of blue, purple and amber glass has a homogeneous chemical composition, consistent with the typical Roman glass. Only three blue glasses differ in CaO and Al<sub>2</sub>O<sub>3</sub> contents, suggestive of the use of different sands. Minor and trace elements analysis revealed that subgroups can be identified, which appear to correlate with the glass colour but not to archaeological types. However, emerald green and black samples are in stark contrast to the other analyzed glasses since they were produced with a soda plant ash as flux, suggesting they were manufactured in a different production centre with different raw materials. The <sup>87</sup>Sr/<sup>86</sup>Sr ratios of the most part of these glasses range between 0.70884 and 0.70916 and well correspond to the present-day seawater signal (<sup>87</sup>Sr/<sup>86</sup>Sr ~ 0.7092), suggesting the use of beach sand as lime source. The Nd composition generally shows a Nile-dominated signature, with εNd varying between -2.59 and -5.65. Conversely, two samples have lower Nd signatures (-7.41 and -10.04 εNd), not corresponding to sediment signatures from the eastern Mediterranean basin. This suggest that the primary production of the vast majority of Adria glasses likely took place in the eastern Mediterranean, notwithstanding a western production cannot be excluded.

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## **V45 Iron Age Vessels from the Archaeological Museum of Adria (North-Eastern Italy): A Textural, Chemical, and Mineralogical Study**

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During Iron Age radical changes occurred in glass production, in particular in the use of fluxing agents. It is well known that in the Late Bronze Age glass was produced using plant ashes as the batch fluxing component (Angelini et al., 2002), while the Final Bronze Age was characterized by the appearance in Europe of the so-called “mixed alkali glasses” (Angelini et al., 2004). From the 7<sup>th</sup> century BC onwards, glass composition changed radically, and the so-called “natron based” glass became widespread in eastern and western regions.

This research is focused on a small group of Iron Age vessels, dating from the 6<sup>th</sup> to the 2<sup>nd</sup> century BC and coming from the Archaeological Museum of Adria (Rovigo). These glass items include essentially three types of vessels (*oinokai*, *aryballoi* and *amphoriskoi*), produced with the technique of the core-forming. They belong to the so-called ‘Mediterranean Groups’, characterized by an intense transparent blue body decorated with yellow, white and turquoise trails. Notwithstanding the bulk chemistry and the opacifying agents of numerous Iron Age vessels were extensively characterized in recent works (Arletti et al., 2010, 2011), a textural study on the transparent glass body was never performed. In this respect, a complete chemical textural and mineralogical characterization was conducted on the Adria vessels.

All analyzed samples are soda-lime-silica glasses, produced with a siliceous-calcareous sand and natron as flux. Their bulk composition is consistent with that of the coeval vessels of Bologna and Spina (Arletti et al., 2010, 2011) and is also similar to the typical Roman glass. Trace elements analysis revealed that the main chromophore of these glasses is cobalt, associated with copper and iron. Numerous residual phases were observed in all the blue bodies. They likely represent residues of Co-bearing raw materials and are constituted mainly by drops of copper sulphides, sometimes with metallic segregations (Pb, Sb), and by metallic inclusions containing Fe-Co-Ni.

All the white decorations show the presence of euhedral calcium antimonates crystals, dispersed in the glass matrix. The same phases were identified in the turquoise samples, although with an anhedral morphology, suggesting a different production technology. Finally, the analytical data for the yellow glasses show the presence of euhedral and anhedral lead antimonate crystals, with notably iron contents ( $\text{Fe}_2\text{O}_3 = 3.62\text{--}10.16 \text{ wt\%}$ ), suggesting that this element comes from Sb and/or Pb raw materials or, alternatively, that it was added during crystal synthesis in order to modify the colour (Dik et al., 2005).

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#### **V46 A Reliable Protocol for the Isolation of Nd from Low Content Archaeological Glass**

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Objective determination of provenance relies on the assumption that there is a scientifically measurable property that links an artifact to a particular source or production site. Since the elemental composition of Roman glass was found to be relatively uniform, it was proven very difficult to assign specific objects to their manufacturing site. Recent advances using radiogenic isotopes, in particular those of Sr and Nd, have allowed the development of new approaches in the provenance determination of primary glass. Due to their relatively high masses and low relative mass differences, Sr and Nd isotopes are not fractionated at the temperatures involved in glass melting, and therefore, the isotopic composition of a glass artifact should be identical to that of the raw materials from which it is derived. Whereas the extraction chromatographic separation of Sr from complex matrices is relatively easy and well established (De Muynck et al., 2009), this is not the case for Nd. Nd is conventionally separated from a sample solution by a two-step extraction chromatographic separation using TRU and Ln resin (Pin et al., 1994; Pin & Zaldegui, 1997). In a first step, the LREE's are separated from the matrix using TRU resin. In a second step, Ln resin is used for sequential elution of the LREE's. A slightly modified version of the protocol described by Pin (Pin et al., 1994; Pin & Zaldegui, 1997) was used to separate Nd from glass samples from the Roman town of Augusta Praetoria (modern Aosta, NW Italy). This however did not result in a good separation of Nd from the matrix. Furthermore, the final fraction obtained was relatively low in Nd. The existing separation procedures were therefore found to be not suitable for use on glass samples with low Nd concentrations. In this study, a new, simple, fast and reliable analytical protocol for the isolation of Nd from complex glass matrices with low Nd contents was proposed and tested. This method uses commercially available disposable columns and chromatographic TRU and Ln resin. Application of the new analytical protocol to the same glass samples resulted in a better separation of Nd from the glass matrix. Nd isotope ratio measurements with MC-ICP-MS, showed signal intensities 2.5 to 13 times higher than those obtained when measuring the Nd fractions separated using the original protocol. Consequently, the newly proposed method provides better and more reliable Nd isotopic data.

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#### V47 Pompeii and Herculaneum: Differences and Similarities through the Chemical and Isotopic Composition of Glass

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Destroyed in 79 AD by the eruption of Vesuvius, Pompeii and Herculaneum were the favorite residences of wealthy Romans. The two towns were situated in strategic locations: Herculaneum located on elevated ground between two rivers with an important port; Pompeii located at the mouth of the Sarno river with a fertile and economically important agricultural plain in its hinterland (Davenport Adams, 1872; Horne, 1895). Rediscovered in 1738 (Herculaneum) and 1748 (Pompeii), the two towns have been extensively excavated in the last two centuries yielding many important finds among which is the large amounts of glass that is the subject of investigation of this study. This study focuses on 39 glass fragments: 25 colourless examples excavated in Pompeii, and 14 (5 of which colourless and 9 green, blue and yellow) excavated in Herculaneum. Through a combined approach, involving compositional analysis and Sr-Nd isotopic analysis, the primary origin of the raw materials used in glass making is investigated. The isotopic ratios obtained for the glass samples are compared to a sand database (Brems et al., submitted), which includes relevant sands from the regions described by Pliny the Elder, and to the signature of primary glass from known production centers in the eastern Mediterranean. In this way, the technology used for and occurrence of raw glass factories outside the Syro-Palestine and Egypt is investigated. Based on our results we have been able to address fundamental questions concerning the economics of glass making such as where mass producing ‘primary’ factories were situated, and how was the glass materials transported? Also, we address whether glass production in the early Roman days was primarily situated in the Near East or were there western factories also producing natron glass (Freestone & Gorin-Rosen, 1999; Gorin-Rosen, 2000; Nenna et al., 1997).

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**V48 The Stained Glass Window from San Antonio di Padova Capella (1525), San Petronio Basilica, Bologna, (Italy): Chemical Evidence of a Northern Europe Glass Influence**

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The chemical composition of 20 medieval coloured stained glass fragments from the stained glass window of San Antonio di Padova Capella, San Petronio Basilica, Bologna Italy (made at 1525 AD), has been characterized by ICP-MS (main constituent elements of the glass, and up to 42 trace elements). This set of samples includes uncoloured and coloured glass: deep green, olive green, yellow, pink, deep blue, light blue, and red plaque. Attending to the main elements composition, three groups of glass can be distinguished: 1/ a set of 5 samples (deep and olive green, uncoloured, red plaque) are in fact calcium-(sodium-potassium glass), with contents 20-21,5 wt% CaO, 62-64 wt% SiO<sub>2</sub>, and total alkalis lesser than 7,5 wt%; 2/ a set of 3 red *plaqué* fragments, with 70,5-73,5 wt% SiO<sub>2</sub>, 15,5-17,5 wt% Na<sub>2</sub>O, 6,7-11 wt% CaO; 3/ the rest of glass fragments is made by sodium glass of Mediterranean tradition with 63,5-69,3 wt% SiO<sub>2</sub>, 16,5-19 wt% Na<sub>2</sub>O, 6,2-8,2 wt% CaO and 2,3-4,7 wt% K<sub>2</sub>O. The trace elements allow inferences of the origin of raw materials, both network formers and colouring agents.

In set 1/, Rb, Sr, Ba, Ce, Li and Be contents clearly differs of the other glass groups and might be interpreted as associated to the source of calcium (carbonate) to the glass. It is outstanding that in this group CaO probably plays a role of network former more than stabilizer. This set of glass has the chemical character of woodash lime glass produced in northern Europe starting around 1400 (Wedepohl, 1998). The main colouring agent in this group is Cu, diffused in the mesostase of the glass in green fragments and as an enamel in red plaque ones. In set 2/, two fragments have remarkably low contents of K<sub>2</sub>O (lesser than 0,3 wt%) and have again Cu (enamel) as colouring agent, while the third sample holds 3,2 wt% K<sub>2</sub>O and Zn is the colouring agent (with an associated chemical fingerprint of Cd-As-Se; probably introduced in the uncoloured original glass in a K-Si-Al rich frit). In set 3/ the two yellow fragments are silver-yellow glass, uncoloured and pink glass show remarkably low contents of iron (accurate cleaning of the silica source), being pink glass coloured traditionally with relatively low contents of Mn, and Cu (with several trace-element chemical fingerprint) is the main colouring agent in green and blue glass. REE contents of sets 1 and 3 is homogeneous in each group.

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#### **V49 Characterisation of Glazed Tiles with EPMA and Mobile XRF for the Development of Adapted Conservation Materials**

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From the ancient Islamic World, many noteworthy buildings like mosques and medreses are still preserved, but most of them are in need of being carefully conserved. In order to determine the composition of glazes decorating the inner and outer ceramic tiles of this cultural heritage, previous analyses were done by Mason *et al.* (2001) and al-Saad (2002). The database can now be enhanced with new data from Central Asian Islamic buildings. The distribution of the analysed objects allows estimations about the scope of the influence of the Near Eastern cultures and the cultural imprint of the historical Silk Road, respectively.

After preliminary analyses using EDS, the samples are now being analysed with mobile XRF. The comparison of the data from XRF and EPMA shows, that the results from the mobile XRF are less precise. However, with careful calibration on the basis of EPMA-data, mobile XRF can be an important tool for on-site analysis of large quantities of glazed tiles.

The analysis data of the glazes give the opportunity to develop an adapted conservation material, which is chemically similar to the original substrate. It thus presents an art-historically reasonable solution for conservation concepts. The basis of the material is the hybrid-polymer ORMOCER<sup>®</sup>, which was developed by the Fraunhofer-Institute for Silicate Research, and which was already successfully used in different conservation projects, e.g. for glasses and metals as well as glazed tiles. Due to the variable composition of this material, a broad variety of physical properties can be considered, e.g. bonding to substrate, viscosity or surface tension.

In order to imitate the historical glazes, the ORMOCER<sup>®</sup> will be modified with coloured glass, produced according to the chemical analysis of the historical ones. The material is tested in chemical and optical characterisation (EPMA, UV-Vis-spectrometry, refractometry) and in the application on ceramics, weathered in special furnaces. Iterations of characterisation and stress simulations yield a practicable and historically reasonable conservation material. In a long-term experiment, the material should be applied in the original conditions on the original historical buildings.

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**V50 'Pottery from the Underworld'. A Petrological Analysis from a Selected Group of Neolithic-Middle Bronze Age Ceramics from the Caves of Central Sardinia**

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Fifty prehistoric pottery sherds ranging from Late Neolithic to Middle Bronze Age from four caves of the central part of Sardinia, were studied at the petrographic microscope using the method proposed by Whitbread (1989, 1995). The aim of the study was to investigate how the raw materials were selected and mixed by the ancient potters and whether a typical local style, reflecting the social, cultural and natural environment in which they lived, could be envisaged.

Five different fabric groups and classes were identified taking into account the prevalent geological nature of their a-plastic inclusions. They are: Metamorphic (grog), Volcanic, Felsic (quartz in a red matrix), Felsic (with biotite) and Calcareous sand.

The provenance study of the raw materials indicated that they were selected in two streams valleys, nearby the limestone plateaux where the caves open. Refiring tests confirmed that only one type of clay was used in their manufacture.

Experimental tests using the sampled raw materials mixed together in the proportions found at the microscope permitted to state that the area identified was the one where the ancient potters collected the raw materials needed for their vessels.

A peculiar technological style in use among the potters of the area was identified: the tempering of the paste of the new pottery with burnt grog.

During Late Neolithic the Is Janas Cave was used for ritual practises implying the consumption of great quantity of food and the intentional smashing of the pottery during the rites (as proved by the archaeological excavation evidence). Bronze Age sees the abandonment of the Is Janas Cave as ritual place and a different use of the other caves of the area where pottery was not intentionally burnt.

Once the Is Janas Caves became redundant and new rites were performed in other caves during Bronze Age, the most appropriated grog to be used, in perpetuation of this sacral continuity, could have been the Is Janas Cave's one (as confirmed by the study of grog mineralogical composition). Then the potters, periodically, should have re-entered into their ancestors' ritual places to collect the burnt pottery sherds to be included into the new vessels as a linking element incorporating past into their present.

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## **V51 Archaeometric Characterization of a Furnace for Bricks Near the Castle of Zena (Piacenza, Northern Italy)**

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During a recent study for a better valorization and fruition of the medieval castle of Zena (Piacenza, northern Italy), archaeologists have brought to light a furnace for the production of bricks, probably of medieval ages or Renaissance (Cessari & Giunchi, 2007). The structure of the entire firing complex is perfectly preserved, including several bricks interpreted as a residue of the last production of the furnace. This furnace is located a few tens of meters from the castle, therefore it was deduced that it worked just for producing the bricks necessary for construction of the castle and/or for its maintenance.

In order to deepen the knowledge of the furnace, an archaeometric project has been planned, which has been developed in parallel with a wider project for the characterization of the building materials (bricks and mortars) of the castle (Fabbri et al., in press). The first step of our work has been to characterize all the materials present in the furnace: structural parts made of bricks or roofing tiles, residual fired bricks, and concotto from the walls of the furnace, together with samples of local clay collected under the furnace or very close to it.

The results of chemical (WDS-XRF), mineralogical (XRD) and microstructural (Optical microscopy on thin section) analyses have evidenced different characteristics of the investigated materials. At least three different types of composition can be individuated, which are mainly distinguished by their amount of calcium oxide and their microstructure, while the differences of mineralogical composition always can be explained by the effects of firing.

The archaeometric results have been useful for a better interpretation of the construction system of the furnace and for interpreting the differences between the various products in terms of provenance of their raw materials. A part from concotto, the clay of the sediment on which the furnace is established was never used or just in a few cases.

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## V52 Ninth-Century Glasses from Samarra (Iraq) in the Context of Medieval Near Eastern Glass Production

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The Herzfeld excavations at Samarra, the capital of the Abbasid Caliphate between 836 and 892 CE, yielded large quantities of glass (Lamm, 1928). Stylistically some of the vessels exhibit typical early Islamic forms, while some of the finds (e.g. millefiori glass tiles) indicate Roman influences (Carboni & Whitehouse, 2001). The main objective of this project was to determine the chemical composition of the glasses from ninth-century Samarra by EPMA and LA-ICP-MS, to investigate whether the stylistically different artefacts were made from the same or different raw glass and how the Samarra glasses relate to other assemblages from Mesopotamia and the Near East. Specifically, it was envisaged to trace the chronological development in the production of glass (particularly mosaic tesserae) from the Sasanian (Ctesiphon), through the Umayyad (al-Minya) and to the Abbasid (Samarra) period. The analytical results (EPMA, LA-ICP-MS) show that the glasses from Ctesiphon are almost exclusively produced with a plant-ash recipe, the finds from al-Minya, on the other hand, are of the natron-type and the assemblage from Samarra falls into two distinct groups. While the majority of the mosaic tesserae from Samarra have a typical natron-type composition, all other artefacts have a plant-ash signature. These preliminary results have far-reaching consequences for our understanding of the production and supply of glass in first millennium Mesopotamia. Firstly, the plant-ash based technology is attested in fifth to seventh century Ctesiphon and this applies also to the production of mosaic tesserae. Under Abbasid rule two centuries later things have clearly changed. At Samarra, the material for the tesserae has been imported, possibly from the Levantine coast. Finally, the eighth-century glass from al-Minya is of the natron type, reflecting its geographical vicinity to the Levantine coast. Taken together the chemical data recapitulates the political and cultural trends at the time. Whereas the Umayyads were orientated towards the Mediterranean, the centre of the Abbasid world was in Mesopotamia, stimulating a more intense Syria/Palestine – Iraq exchange (Wickham, 2002).

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## **V53 Of Time and Shapes: Compositional Variation in Post-Medieval Glass from the Netherlands**

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Glass is a common occurrence in post-medieval urban archaeological sites. Cess pits from several cities in the Netherlands have yielded a range of glass objects. Based on such finds, a clearly defined typochronological sequence is available. Such a sequence, however, does not provide much information on the raw materials used, and how they varied through time.

In order to test whether changes in raw material use would be detectable, we analyzed the c. 300 glass objects that forms the basis of the National Reference Collection (NRC). They originate from four different cities, and range in age from 15<sup>th</sup> to 19<sup>th</sup> century. The objects are mainly bottles and various types of drinking glasses, but other types of objects (e.g. salt dishes) are present as well.

The chemical composition of these objects was determined using portable XRF. Analysis of the dataset shows that most objects are potash glass, with a smaller group of lead glass objects. Part of the variation in K and Ca contents is probably caused by leaching during burial. However, K-Ca plots show still distinct groups with varying K/Ca ratios. Throughout the ages, bottle glass has a much higher K/Ca ratio than the various types of drinking vessels. This indicates that the production of glass for bottles during the last centuries was separate from the production of glass for luxury items. There also seems to be compositional variation between different luxury items; this would suggest that various production lines for luxury glass existed parallel for centuries. In each of these production lines, different standard recipes for glass production were used.

Although for each of the types of objects the mix of raw material remains more or less the same, variations in Rb and Sr contents indicate that the source for potash and lime may have varied.

## **V54 The Strange Case of 60 Frothy Beads: Puzzling Early Iron Age Glass Beads from the Netherlands**

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During the excavation of a late prehistoric urnfield near Zutphen (NL), a rich Early Iron Age burial was found to contain c. 60 simple small blue-green glass beads and two blue ringaugenperlen. This made this a very special find, as glass from this period in the Netherlands is extremely rare (totalling c. a dozen beads). The simple glass beads have a semi-opaque, almost frothy appearance due to the large amount of small air bubbles inside and on the surface.

Compositional analyses by portable XRF show that the glass was made using soda as a flux. This is remarkable, since the closest known glass production centre (Fratessina, Italy) produced mixed alkali glasses at this time. Some of the beads were found to contain high levels of lead or antimony, which indicates that the beads were not made from a single melt. The color of the glass, however, is dominated by cobalt and very high copper contents (3.5 – 7 %). Study by microscope, electron microscope and micro-CT showed that the air bubbles in the glass can reach an estimated 50% of the total volume of the object. Moreover, the glass mass contained a large number of fine metal particles and some mineral inclusions. These metal particles are identified as copper. Such metallic fragments are known from some types of faience, but have not been observed before in glass. The structural and compositional properties of these glass beads are unique within the spectrum of Bronze Age and Iron Age glass in Europe and the Mediterranean. However, if we disregard the frothiness and the high concentrations of metal fragments, the composition matches that of the Eastern Mediterranean glass production centers. We propose that the Zutphen glass beads are the result of local inexpert reworking of imported glass objects. The air bubbles were the result of inexpert melting of glass fragments or objects; the copper fragments may have been added to homogenize the color.

## **V55 Mobility and Pottery Production in Panama (ca. 4500-3200 BP): Petrographic and Geochemical Research**

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Conducting microbotanical and zooarchaeological analyses, archaeologists now know that during the earliest ceramic period (ca. 4500-3200 BP) in Panama, called Monagrillo, people cultivated plant foods, hunted, and engaged in fishing and coastal shellfish procurement. They occupied inland rock shelters in the Cordillera Central, and coastal shell bearing middens of the Azuero Peninsula (Cooke & Jiménez, 2004, 2008; Dickau, 2010; Piperno & Pearsall, 1998). Nevertheless, archaeologists are not certain whether inland farmers practiced coastal activities seasonally or sedentary communities exchanged materials (Carvajal-Contreras et al., 2008; Peres, 2001). This project investigates the type and degree of residential mobility through studying pottery production and patterns of circulation.

In order to understand production and circulation, the project aimed to identify locally produced vessels and ware that was transported. Ceramic petrography (n=101), bulk chemical analyses via portable x-ray fluorescence pottery (n=145) and raw clayey soil (n=90), and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) of pottery clays (n=162) and raw clayey soils (n=79) were conducted. Ceramics from He5, Pr14, and Pr32 sites on the Pacific coast, Ag13 site on the Pacific plains, Cl1, Lp134, and Sf9 sites on the Pacific foothills, and Lp8 on the Caribbean slopes were examined. The results suggest that: (1) the majority of pottery from the Cordillera was produced in situ having one or possibly two production zones and small numbers of He5 ceramics were transported from the Cordillera zone; (2) most pottery from Pr32 was likely to have been produced in situ; (3) some Cordillera ceramics were transported from Azuero; (4) about half of the Ag13 sherds were from the Cordillera and the other half were from Azuero. It was difficult to source ceramic clays to raw clayey soil. Barium and strontium high in some ceramics from Ag13 and Cl1 indicate diagenesis; hence, these elements were excluded from the sourcing study. In terms of mobility, people moved between Cl1, Lp134, Lp8, and He5 but less often from Pr32 to the Cordillera indicating that, Pr32 population tended to be more sedentary than people living in other sites but others also had enough degree of sedentism for ceramic production. Ag13 probably had frequent visits from both zones or there was a shift from the use of Cordillera to Azuero ceramics or vice versa, over time.

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## **V56 Ceramic Technologies Among Arctic Foragers: A Pilot Study from Nunivak Island, Alaska**

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Integrated ceramic technological analyses have had a limited impact on the study of hunter-gatherer pottery traditions, particularly in northern latitudes. The aim of this pilot study is to assess the potential for the analytical study of technology and provenance of prehistoric Arctic pottery. It fits into a broader research project investigating changes in ceramic technological traditions and scales of interaction among prehistoric marine foragers in coastal Alaska. This paper focuses on the recently excavated site of Nash Harbor, Nunivak Island (c.1800-800 ybp).

Ten samples, corresponding to an equal number of vessels, were selected for the initial analysis. Sampling encompassed both Norton tradition pottery, decorated with check-stamped and curvilinear patterns, and plain pottery of likely Thule affiliation. Thin-section petrography was undertaken as a means to identify the raw materials used and their possible sources, and to characterize the manufacturing process. SEM and FT-IR were combined to provide information on firing regimes.

Petrographic analysis revealed variation in technology and clay sourcing. Two fabric groups have been defined: one characterized by coarse basalt sand in clays also of volcanic origin; the other showing only smaller, natural inclusions of mixed origin. Comparison with samples of Cretaceous fluvio-marine sediments collected at one coastal location suggests these were not exploited. Residual and alluvial materials of volcanic origin are more likely to have been used. Significantly, different technological strategies seem to be associated with different ceramic categories (plain versus decorated).

Preliminary SEM and FT-IR results point to overall intermediate firing temperatures, insufficient to result in vitrification of the clay matrix but higher than the dehydroxylation temperature of clay minerals (c. 550-650°C). Some samples appear to have been exposed to higher temperatures than others, but these differences do not seem to correlate with decoration or fabric category. On the basis of these results, it would appear that the prehistoric Nunivak pottery does not fall into the category of 'unfired' cooking pots documented in Arctic ethnography and later prehistory (Harry et al., 2009).

Future work will expand geological and ceramic sampling; well dated house assemblages will be targeted in order to contextualize the patterns identified so far. The information gained will ultimately help to address issues of tradition, cultural change and social interaction among Arctic foragers. It will also contribute to discussions of function and performance, which have been central to studies of early hunter-gatherer pottery and Arctic ceramics.

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## **V57 Waste from a Glass work at Glargaarde in Northern Jutland**

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Waste found during archaeological investigation of a Renaissance glasswork by Glargaard in northern Jutland have been analyzed. Among the parts from the furnace clay plates that had been used for closing openings in the furnace was found. Some of these were made from ordinary clay, while fireclay was used for another group. This probably reflects the use in different parts of the furnace or different furnaces. The crucibles for melting of the glass were also made from fireclay. As raw clay and unburned crucibles were found, it could be seen, that the crucibles were made at Glargårde from a mixture of fresh clay and grog made from old crucibles.

Frit, the intermediate product of the glassmaking process, was identified as greyish half molten lumps having a composition close to that of the finished glass.

Window glass was apparently the main product of the glasswork, and three types of glass were identified. The light olive-green glass normally associated with "forest glass" did constitute a large part of the glass fragments found, but the glassmaker at Glargårde had also been able to produce nearly colourless glass by decoloration with manganese oxide. A third type of glass was coloured bright green using copper oxide. This type was the only one used for the production of painted window panels. The paintings were primarily made in a brown color, using iron oxide as a pigment, mixed with lead containing glass. The lead-oxide probably made the fusing of the paint easier. A gray color on some glass fragments was identified as a copper oxide. On similar painted window fragments from Ribe to shades of brown was used, produces by the addition of different amounts of iron oxide to a pulverized lead glass.

## V58 Physical Chemistry Analysis of Roman Glass Fragments from Thessaloniki Agora

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This analytical research deals with twelve Roman glass fragments, coming from excavations of the Roman Agora of Thessaloniki, and provided by the Archaeological Museum of Thessaloniki. The samples were studied by means of micro X-ray Fluorescence Analysis ( $\mu$ -XRF), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES), Flame Atomic Absorption Spectroscopy (FAAS), Raman Spectroscopy and the Application of Principal Components Analysis (PCA) on the experimental data. The main purpose of this research was to determine the chemical composition of the glass fragments and give answers to questions concerning their origin and dating, as well as the manufacturing technology of glass. Moreover, different analytical techniques and methods, were compared, according to their efficiency.

The results showed that high percentage of sodium oxide ( $\text{Na}_2\text{O}$ ) and calcium oxide ( $\text{CaO}$ ) indicates that all the samples belong to the category of soda-lime glass. The relatively low percentage of potassium oxide ( $\text{K}_2\text{O}$ ) and magnesium oxide ( $\text{MgO}$ ) shows that natural soda was used as alkali material. Its origins cannot be determined precisely, it is either natron of Egypt or it comes from the region of Macedonia. The study concerning the colours of the samples showed that blue glass was coloured with cobalt oxide ( $\text{CoO}$ ), even at very low percentage. Blue-green glasses were coloured with a mixture of copper oxide ( $\text{CuO}$ ) and cobalt oxide ( $\text{CoO}$ ). The purple colour is due to the high concentration of manganese oxide ( $\text{MnO}$ ). Finally, the combined use of two decolorizers, manganese oxide ( $\text{MnO}$ ) and antimony oxide ( $\text{Sb}_2\text{O}_3$ ) was observed, which is related to geographical, chronological and technological factors. There was a deliberate addition of antimony in the melt to produce a more bright glass, or antimony glass was used as an additional material (cullet) for the production of glass that was decolorized by manganese ( $\text{Mn}$ ). As far as the analytical techniques are concerned, multi-elemental and non-destructive analysis constitutes an objective in archaeometry.

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## **V59 Glassware from Building of XVIII c. of Kazan Kremlin**

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In the 2000 year during the excavations in the Kazan Kremlin timber cellar was discovered. Cellar contains soot soil with inclusions of fritted bricks and large limestone. The building flanked to the basement of governor house, which existed in the first half of XVIII c. and is dated from a silver ruble of 1713 year, which was laid by workers under the angle of the building. This date is corroborated by other coins of the first half of XVIII c. which were found in the cellar.

In the cellar remains of furnace were found. It had size of 200x200 cm. Floor of the frame from the side of hearth-stone was bricked. In the frame beads and bottle glass were discovered among the brick piles, glass slag and limestone. The majority of beads are twice-truncated globular beads; twice-truncated ellipsoid beads have lesser numbers. There is only one ring-shaped bead.

From the chemical analysis of beads structure it was established that most of them had been made of glass which is identical to the bottle glass from this building. Such glass is typical for Russian glass melted with ash in the period of XVII-XVIII c. Thorough investigation of trace elements components showed, that in this workshop remelting of various glass scrap, including old beads and button, was performed. These experiments were conducted to obtain new glassware from the old glass scrap. Analysis of trace components composition revealed that master blended glass which varied in color and composition, and also added ashes of animal bones to obtain different colors of his new beads. Master made large black bead, colored by manganese (1,39%) which has very unusual composition - without main glass-forming components. This glass had unstable recipe that is why it came to us in very bad condition. Chemical analysis showed that glass had been made of quartz melted with milled bones of young animal.

In the same workshop unusual glassware was discovered. It has size of 20x20x10mm and a color of blue. One side of the glassware contains sunked picture made by a sharp tool.

Beside glassware, numerous objects were found in the cellar, including two Dutch pipes, remains of a pistol, etc. From all of the above we can make a conclusion, that the production of beads in this case was probably a hobby of the noble man.

**V60 An Archeometric Investigation of Some Byzantine Ceramics Case Study: 12<sup>th</sup>-13<sup>th</sup> Century Glazed Ceramics from Kuşadası Kadıkalesi/ Anaia**

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Kadıkalesi is a Byzantine fortress on the shore of Kuşadası Bay in Western Anatolia. It was built for the defense of Anaia, a significant port town and an episcopacy center during the Late Byzantine period. Strong evidences for the local ceramic production reveal the position of Kadıkalesi as an important ceramic production center during the 13<sup>th</sup> century. Ceramics analyzed in this study mainly belong to *Zeuxippus Ware Family* ceramics which constitute the most frequent occurrence in the Byzantine culture during the 13<sup>th</sup> century. As their identification is still controversial, the material characterization of them, which is expected to give further information, turns out to be quite important.

Archeometric investigations were carried out on the glaze, slip and body parts of the ceramics by using optical microscopy, XRD, SEM-EDX and ICP-OES /ICP-MS techniques. The glazes have lead-silica compositions with low alkali contents, as typical characteristics of Byzantine glazes. Relatively high amounts of alumina ( $\text{Al}_2\text{O}_3$ , ~ 3-10 %) found in the glazes may be due to the intentional addition of clay to the glaze suspension in order to give plasticity and to facilitate the application of the glaze to the body. Iron-oxides, with concentrations not less than ~ 1 %, seem to be responsible for the observed yellow, brown and some of the green colors of the glazes.

A layer of lead-rich feldspar crystals at the glaze-body interface observed with relatively small thickness possibly suggests that these ceramics were biscuit-fired. Low amounts of calcium oxide and iron oxide were determined in the slip layers which display color tones from white to yellow.

The absence of high temperature phases and partial vitrifications in the body parts indicate the use of relatively low firing temperatures probably not exceeding ~ 850°C. Based on the SEM-EDX and ICP-OES investigations, most of the body parts were found to contain at least around 10 % calcium oxide, indicating the use of calcareous clays in the production. The use of at least two types of clay sources based on the observations of different mica contents in thin sections is also supported by ICP-OES measurements. Furthermore, data are evaluated statistically in order to check the validity of the classifications proposed on the basis of the stylistic analysis of the samples.

## V61 Cassiterite in Tin Glaze of Anabaptist Faience from Hungary: Morphological and Crystallographic Characterization by SEM and TEM

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Dissolution and recrystallization of tin oxide opacifier is a well-known phenomenon to occur during firing of lead glazes (Molera et al., 1999; Tite et al., 2008). Study of morphological and crystallographic characteristics of the recrystallized cassiterite, especially using high-resolution techniques such as transmission electron microscopy, was done only in few cases (e.g. Viti et al., 2003; Giorgetti et al., 2011). During the production technology study of 17<sup>th</sup> century Anabaptist faience (tin-glazed earthenware) artifacts from NE-Hungary (Sárospatak), relatively high tin oxide content was found in the white glaze. The tin-opacified lead-alkali glaze has ~150 to 400 µm thickness and contains high amount of cassiterite inclusions. Bulk EDS analyses indicate 16 to 20 wt% SnO<sub>2</sub> in the glaze. Cassiterite inclusions were analyzed using scanning and transmission electron microscopes. Two cassiterite types are distributed heterogeneously in the vitreous matrix. Larger (up to 10 µm size), angular cassiterite particles are probably the remnants of the tin oxide raw material used for the glaze. Newly crystallized tin oxide inclusions were detected mainly as needles up to 1-2 µm size. Needles preferentially form aggregates reaching 30 µm size, which preserved the original morphology of the tin oxide raw material. Cassiterite needles can also form skeletal crystals. The intimate intergrowth of the two cassiterite types is clearly visible supporting the tin oxide dissolution and recrystallization during firing and cooling of the glaze. (HR)TEM analysis confirmed the presence of two types of cassiterite morphology. The size of the isometric crystals ranges between 200 nm and 1 µm. Ordered single crystals and [101] twinned crystals were found as well. Detailed structural analysis on the atomic scale revealed shear bands associated to the twin plain. The periodicity of the subsequent tin and oxygen planes along the shear bands is inconsistent with cassiterite structure. The possibility of local, nanometer-scale nonstoichiometry is examined.

Needles, hereafter called nanorods, are typically 15-20 nm thick and 200-500 nm long single crystals. The direction of elongation, determined analyzing ca. 20 crystals, can be either parallel or perpendicular to [110], both types occurring with the same frequency. According to Fourier transforms of high resolution images, some nanorods show the 4.7 Å periodicity, which is the forbidden d(100) of cassiterite. Image simulations are used to validate if incomplete unit cells along the thickness of the nanorod can cause such violation of symmetry.

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## **V62 Ceramic Technological Traditions from the Early to the Late Neolithic in Hungary**

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The aim of this study is to assess the ceramic technological tradition of the Late Neolithic, in particular for the Lengyel and Tisza cultures from five settlements in north (Aszód-Papi földek), south-west (Belvárdgyula-Szarkahegy, Szemely-Hegyes, Zengővárkony) and south-east (Hódmezővásárhely-Gorzsa) Hungary. A particular focus is given to Aszód-Papi földek, which constitutes a particular focus for archaeological research since its material culture represents two well-known Late Neolithic cultural groups in Hungary that are the Lengyel and the Tisza cultures.

The Lengyel settlement at Aszód exhibits considerable number of vessels of the Tisza style. The Tisza style vessels at the site are distinct in terms of vessel forms and decorations. By the means of petrographic analysis the ceramic technology of the Lengyel and Tisza style ceramics are examined. The possible ceramic technological similarities and differences between similar vessels types, which represent different cultural groups at the same site provides us with an essential methodological tool to examine the interaction between cultural groups in terms of ceramic technology and can reveal the possible cultural flexibility available to potters facing cultural constraints.

Macroscopic and thin section petrographic analysis is applied to examine the similarities and differences between raw materials, fabric preparations, tempering practices and building techniques. The results from the Tisza and Lengyel sites are compared and it is assessed whether there are particular ceramic technological choices that are peculiar to the Late Neolithic.

The results from the Late Neolithic sites are then compared with Neolithic ceramic technologies from twelve Early and Middle Neolithic sites providing a vivid picture of how ceramic technology changed through the Neolithic in Hungary.

**V63 The Chemical Composition of Glass from the Hungarian Glasshouses and Glass Utilized in Hungary from the 14<sup>th</sup> Century to the 19<sup>th</sup> Century**

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The aim of the project is to outline the development of Hungarian glass and glass utilized in Hungary from the 14<sup>th</sup> century to the 19<sup>th</sup> century. It focuses on chemical composition of material excavated in three glasshouses (in Visegrad and Diósjenő, both dated to the 14<sup>th</sup>-15<sup>th</sup> century and in Gyertyánvölgy, dated to 18<sup>th</sup>-19<sup>th</sup> century) as well as in various places, including royal castles, of medieval and post-medieval Visegrad and Buda. The majority of glass pieces included in the project are of Hungarian origin, however, there are also some Italian, Turkish and central European imports. Totally, about 50 samples have been analysed. They represent colourless, coloured and opaque glass, vessel and window glass, including stained glass, as well as some jewellery items in addition to the glass fragments, such as moils, slugs and tears gathered from the glasshouse sites.

Scanning electron microscopy - energy dispersive spectrometry (SEM-EDS) has been applied to analyze the fragments. A preliminary technological interpretation of the glasses has been discussed together with some results of analysis of about 50 samples and their technological interpretation is shown.

Preliminary conclusions on the glass trade in Hungary in various periods are outlined with special emphasis on the 14<sup>th</sup> and 15<sup>th</sup> century material. Moreover, the results also allow us to discuss the level of development of the domestic Hungarian glass technology through the centuries.



#### **V64 Study of the Production of an Archaeological Funerary Urn via Electron Paramagnetic Resonance and Mössbauer Spectroscopy**

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Electron paramagnetic resonance (EPR) and <sup>57</sup>Fe Mössbauer spectroscopy are excellent microscopic method in the investigation of clay-based ceramics since practically all pottery clays contain iron, usually in concentrations between 1 and 10 wt%. The changes which pottery clays undergo during firing are reflected in the EPR and Mössbauer spectra of the fired ceramics and hence can be used, even after millennia of burial, to obtain information on the original firing conditions and on techniques and abilities of the ancient potters. Here we report on a study on a fragment of an archaeological funerary urn from a site located in the city of Campos dos Goytacazes, Brazil, using EPR and <sup>57</sup>Fe Mössbauer spectroscopy techniques. The cross-section of the pottery wall has a sandwich-like structure bounded by light red edges and dark gray interior. The firing temperature was evaluated, comparing archaeological pottery with clay and comparing changes caused by heat treatment in archaeological samples (Manguiera et al., 2011). The method used consists in monitoring the spectra of iron and the effects of heat treatments in this signal in both spectroscopies.

All EPR spectra show an intense and broad line at  $g = 2$  due to  $\text{Fe}^{3+}$  in octahedral sites, but with different line shape in the dark and red portions of archaeological pottery. The <sup>57</sup>Fe Mössbauer results, at room and low temperatures, evidenced the presence of quadrupole doublets associated with the presence of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  species and additionally two magnetic components associated with hematite and goethite in all the structure. However the Mössbauer spectra show that the light red edges present a higher amount of oxides than the dark grey interior. The strong  $\text{Fe}^{3+}$  presence suggests an oxidant environment in the ceramic production firing technique. These results are complemented by the x-ray diffractograms in which it can be seen the presence of mica only in the dark grey interior being absent in the external and inner parts. The methods complemented each other, providing valuable information about firing techniques of archaeological pottery.

MANGUEIRA, G.M., TOLEDO, R., TEIXEIRA, S. AND FRANCO, R.W.A., 2011. A study of the firing temperature of archaeological pottery by x-ray diffraction and electron paramagnetic resonance. *Journal of Physics and Chemistry of Solids***72**, 90-96.

## V65 Mesolithic and Neolithic Pottery Production at Al Khiday Sites (Khartoum, Sudan)

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The archaeometric study on archaeological materials is normally aimed to define raw materials provenance and production technology. Although in the last years this type of research advanced considerably, issues were often only qualitatively tackled. Quantification of textural parameters in relation to the bulk and single phase chemical composition represents a wide field of investigation, which can provide important information especially in terms of production recipes, technological choice and cultural changes over time. In this frame the study presented here will focus on the analysis of Mesolithic and Neolithic pottery from Al Khiday 1 (16-D-5) and 10-W-4 sites (Khartoum, Sudan), through images collected under scanning electron microscope (SEM) to quantitatively describe the production recipes, especially with respect to the minero-petrographic nature of temper, clay:temper ratio, and textural features of added inclusions in terms of shape, grain size and grain-size distribution. Archaeologically these sites, differently from others formerly investigated in Central Sudan along the Nile Valley, are unique either in this geographic context and age, since they provided well stratified sequences, radiometrically dated (Salvatori et al., 2011; Zerboni, 2011). The stylistic analysis on the pottery showed that, in addition to the characteristic decoration defined *wavy line*, widely attested in numerous Mesolithic Sudanese sites, at least other 10 decorative types were used in this ceramic production (i.e. *dotted wavy line*, *“lunula-shaped”*, *alternately pivoting stamp*, *rocker stamp drops*, *rocker stamp drops deep*, *rocker stamp dotted zigzag*, *rocker stamp plain zigzag*). The distribution of these decorative motives in the stratigraphic sequence show that certain decorative types are predominant in the oldest strata and progressively substituted by others, indicating a well defined stylistic evolution along the time axis, which should reflect also changes in the production technology in terms of ceramic pastes and production recipes. Although many archaeological studies have been done on Mesolithic and Neolithic sites in Sudan and Nubia, archaeometrical researches are rare, limited to small number of samples, often coming from disturbed stratigraphic levels with uncertain dating. The possibility of quantifying elements characterizing the production technology, resulted to be an important tool to determine the evolution of pottery production over the time at the sites of Al Khiday, providing a scientific support to the model of production technology based on archaeological observations.

SALVATORI, S., USAI, D. AND ZERBONI, A., 2011. Mesolithic site formation and paleoenvironment along the White Nile (Central Sudan). *African Archaeological Review***28**, 177-211.

ZERBONI, A., 2011. Micromorphology reveals in situ Mesolithic living floors and archaeological features in multiphase sites in Central Sudan. *Geoarchaeology***26**, 365-391.

## **V66 Production of Glass Beads in Merovingian Times: Study of the Artefacts Coming from the Excavation of Grez-Doiceau Necropolis**

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Merovingian times have often been described as a transition period between well-known cultures without important evolution in manufacturing technology and especially glass technology. However recent excavations of important necropolis led to the discovery of an exceptional corpus of artefacts. Among these objects we can highlight the exceptional production of glass beads which reveal a very fine and complex evolution in style and in shape during all the period.

Thanks to the CEA-IPNAS Ion beam facility we had the ability to study chemically the raw material of these objects, both the glass matrix and the colouring and opacifying agents, in order to confirm or not the hypothesis, often presented as a fact, of reuse of roman glass for the fabrication of beads.

We studied more than 300 beads by PIXE and PIGE techniques in external beam mode. These methods, particularly suitable for archaeological glass analysis, are complementary to each other and do not require any kind of sampling or preparation except for a superficial cleaning of the object. The elemental composition of the sample (from Na to U) given by PIXE with excellent sensitivity (down to trace elements), together with the correction of bulk Sodium concentration supplied by PIGE, allows to assess alteration and provides the chemical signature of the glass. Complementary observations were made by SEM/EDX, Raman spectrometry and  $\mu$ -XRD.

These analyses have evidenced the apparition of a new type of glass, as early as in the very beginning of the 6th century, for the production of black beads in parallel with the recycling of different type of roman glass for the same type of production. Moreover the use of a mix of these two types of glasses has also been observed.

The new glass has apparently also been used for the fabrication of yellow opaque beads. This glass is very rich in lead oxide (more than 40 percent) and possibly find its origins in glassy slag coming from lead primary metallurgy as it has been already observed in Carolingian times (Gratuze et al., 2003). These results indicate a real technological research in the glass fabrication, as early as the beginning of the Merovingian period, and this independently from the supply of Roman glass for recycling.

GRATUZE B, FOY D., LANCELOT J. AND TEREYGEOL F., 2003. Les « lissoirs » carolingiens en verre au plomb : mise en évidence de la valorisation des scories issues du traitement des galènes argentifères de Melle (Deux-Sèvres). In : FOY D. AND NENNA, M.-D., (Eds.) *Echange et commerce du verre dans le monde antique*. Aix en Provence, 101-107.

## **V67 Medieval Lead Glass in Central Europe**

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Wood ash glass was the most common type of glass in Central Europe during the 13<sup>th</sup> Century. In addition to this type of glass, natron glass and lead glass were used in much smaller quantities; this presentation will deal specifically with the lead glass types in detail.

The start of the research was the finding of a glass processing workshop in the Thuringian City of Erfurt. This workshop produced with lead glass beads and rings and dates to the 13<sup>th</sup> century. Two different lead glass types were used in the workshop. The question was: How do these results from Erfurt relate to the results published from other glass findings? To answer this question, the various types of lead glass must be presented in relation to the findings from Erfurt, in order to show that a new type of glass can be identified. So deal this presentation with the different medieval lead glasses from England till Russia. For this presentation will be used 36 analyses from Thuringia and 392 analyses from 93 archaeological sites from the literature. The most important type is the high lead glass which was found from England till Russia. This type was produced from lead, sand and pigments. The three other lead glasses are lead ash glasses. One type – the wood ash lead glass – was produced with leadoxid, sand, wood ash and pigments. It was found in the German speaking area and around the Baltic Sea. A different ash with a small CaO/K<sub>2</sub>O ratio was used for the Slavic lead ash glasses but the amounts of lead are similar to the wood ash lead glass. It was found from Poland till Russia. The same ash as the Slavic lead ash glass but a different amount of lead was used to produce another type of lead ash glass. This newly defined type of glass (Central Europe lead ash glass) was found in the Eastern part of Germany, Poland, Slovakia and the Czech Republic.

Furthermore will be discuss in the presentation the different ash types and technological reasons for the different lead ash types.

## V68 The Tombs of Ayia Kyriaki and the Circulation of Pottery in EMI-II South-Central Crete

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The Mesara Plain in southern Crete is a key area for our understanding of the important social changes that took place in the period prior to the foundation of the Minoan palaces, or court-centred buildings. The region has a very rich pottery tradition whose nature and trajectory have been used to illustrate developments and disruptions in social and economic life. Recently, it has been suggested that important aspects of ceramic craft practice survive the transition from the Early Bronze Age to the Palatial period in the Mesara and that this might inform our consideration of social change (Day et al., 2006). Such technological insights have made a return to understanding the production and distribution of pottery in both periods a matter of priority, in other words to micro-provenancing the ceramic products and examining varied exchange and consumption of ceramics in sites which hosted different activities. This paper rises to that challenge by examining Prepalatial pottery of the crucial Early Minoan I and II phases (c. 3100-2300 BCE) from the tholos tombs of Ayia Kyriaki, situated in the Agiofarango (Holy Gorge) which runs down to the Libyan Sea from the Asterousia mountains. This pottery is compared with two settlements in the Mesara Plain itself, Ayia Triada and Phaistos, which we now know hosted social gatherings, as well as a possible pottery production centre (Todaro, 2012). The funerary assemblage at Ayia Kyriaki is found to have a major part of its pottery from the same centre of production as that of the two settlements in the Plain. Through comparison with regional geology, raw materials and a range of ceramic material, it is concluded that the production centre for these ceramics is located in the Mesara Plain itself, probably at Phaistos, the very site which goes on to dominate the region with the construction of a court-centred building at the start of the second millennium BCE. This has major implications for the consideration of site hierarchy, as well as the movement of goods and people in the Early Bronze Age of Crete. Phaistos may be seen as a social and political *hub*, linked with ceramic and other craft production, ritual activity such as ceremonial feasting, maintaining the social cohesion of the region's communities.

DAY, P.M., RELAKI, M. AND FABER, E., 2006. Pottery Making and Social Reproduction in Bronze Age Mesara. In: WIENER, M.H., WARNER, J.L., POLONSKY, J. AND HAYES, E.E. (Eds.) *Pottery and Society. The Impact of Recent Studies in Minoan Pottery. Gold Medal Colloquium in Honor of Philip P. Betancourt, 104<sup>th</sup> Annual Meeting of the Archaeological Institute of America*. Archaeological Institute of America, Boston.

TODARO, S., 2012. Craft Production and Social Practices at Prepalatial Phaistos: the Background to the First 'Palace'. In: SCHOEP, I., TOMKINS, P. AND DRIESSEN, J. (Eds.) *Back to the Beginning: Reassessing Social and Political Complexity on Crete during the Early and Middle Bronze Age*, 195-235. Oxbow, Oxford.

**V69 Bronze Age Glass Between the Alps and the Baltic Sea. Studies on Manufacture and Distribution of the Oldest Glass in Central Europe**

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Blue glass beads from graves, hoardings and settlements of the middle to late Bronze Age (14<sup>th</sup>-9<sup>th</sup> century BC) are the oldest glass finds in central Europe. Compared to other European regions, e.g. Italy, France or Great Britain, these finds are only deficiently explored. The current project archaeologically and archaeometrically investigates such finds to identify their technological and cultural-historical position in the central European Bronze Age.

The studies include analysis by electron microprobe for major and minor elements and laser ablation ICP mass spectrometry for trace and rare earth elements. The methods are working poorly destructive, requiring on a very small scale the removal of the corrosion layer and polishing of the surface.

Until now almost 100 glass beads from different excavation places in northern and southern Germany and in Tyrol/Austria have been analyzed, covering chronologically the middle and late Bronze Age. These are mainly small annular beads, larger spherical beads and the so-called "Pfahlbauperlen", barrel-shaped beads with a white thread of glass inlay. The colour spectrum of the translucent glass beads range from light blue to bright turquoise and dark blue. From their major and minor element contents the middle Bronze Age samples can be assigned to the group of high-magnesium soda-lime glass (HMG), while the late Bronze Age beads were produced from low-magnesium high-potassium glass (LMHK; Henderson 1988, 1989). In addition, a third and previously unknown group of glass is suggested, with significantly higher contents of potassium compared to the LMHK glass, but still low magnesium and particularly low sodium contents.

Trace elements and rare earth elements bear information about the sands and the colouring agents used. Normalized trace element patterns show some striking differences in the contents of the colouring components, i.e. copper or cobalt, and their accompanying elements. Further trace elements and the rare earths may distinguish between different sands on the basis of their heavy mineral contents. This allows the definition of various sub-groups and therefore gives detailed knowledge about central European Bronze Age glass types and their regional and age distribution.

HENDERSON, J., 1988. Glass production and Bronze Age Europe. *Antiquity* **62**, 435–451.

HENDERSON, J., 1989. The scientific analysis of ancient glass and its archaeological interpretation. In: HENDERSON, J., (Ed.) *Scientific analysis in archaeology and its interpretation*. Oxford, 30–62.

## **V70 Colour and Microstructure in Brown and Green Decorated Spanish Tin Glaze Pottery (10<sup>th</sup> to 17<sup>th</sup> Centuries AD)**

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Tin glaze pottery with brown and green (B&G) decorations started being used in the Islamic kingdoms in Spain during the Caliphal period (10<sup>th</sup>-11<sup>th</sup> century AD), continued later being produced in the Christian kingdoms. The earliest Islamic productions (mainly Caliphal and Almohade) usually show a very poor state of conservation with a sandy, mat appearance which was for some time attributed to the use of a white slip instead of a tin glaze. The quality and conservation state of the B&G decorated ceramics dramatically improved in the Valencian, Aragon and Catalan productions from the 13<sup>th</sup> and 14<sup>th</sup> centuries respectively. By the end of the 14<sup>th</sup> century AD, brown and green decorations were largely substituted in Valencia and Catalunya by blue and blue and lustre. Nevertheless, in Teruel (Aragon) B&G pottery continued being produced until the end of the 20<sup>th</sup> century. Brown and green colours are associated to manganese and copper, respectively. Until recently both manganese and copper were thought to be dissolved in the glaze, and their valence and coordination responsible for the differences in the hue observed between productions. Lately the study of B&G by means of Micro-XRD and SEM demonstrated that this is not always true and, the nature of the compounds identified (kentrolite, johansenite, bustamite and braunite). However, the link between this and the colour differences between the several productions has not yet been investigated. In this study, the relationship between the presence of Mn and Cu bearing compounds and the colour shown by the decorations is attempted from a selection of ceramics covering early 10<sup>th</sup> to late 17<sup>th</sup> centuries. Differences and similarities in the nature, microstructure and colour are related to differences in the materials used and production processes followed. The possible reasons for the sudden change in the quality and conservation state of the B&G produced during the 13<sup>th</sup> century are also discussed.

## **V71 Classification of Archaeological Pottery from Amazon Basin by Mössbauer Spectroscopy and Neutron Activation Analysis**

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Pottery fragments discovered in archaeological sites with and without earth circular structures in the hydrographic basin of high Purus River at Acre state Brazil, were preclassified by archaeologists, using visual inspection or small microscopes, in three archaeological phases denominated as Quinari, Iquiri and Iaco (Dias & Carvalho, 1981). Fragments from another archaeological site, Los Angeles, belonging to the same region were also studied in order to identify their relation with these archaeological phases. The aim of this work is to classify archaeological artefacts on the basis of chemical composition and interpret the results in the light of the geographical origins and find out information on the original firing conditions (i.e. firing temperature and atmosphere) using instrumental neutron activation analysis (INAA), <sup>57</sup>Fe Mössbauer spectroscopy (MS) and X-ray diffraction (XRD), combined with multivariate statistical analysis.

The analysis of the <sup>57</sup>Fe Mössbauer spectra at room temperature was performed in a total of 94 fragments of different archaeological phases.

The data obtained by INAA and Mössbauer were combined in a total of 36 variables and used for the multivariate statistical analysis (SPSS). Cluster analysis methods was used for the classification and for the ordination the principal component method. The results show that the Mössbauer parameters complement the interpretation obtained by INAA and these parameters can also be used for ceramics classification studies since the INAA provides information about the geographic origin (Bellido et al., 2011) and Mössbauer on manufacturing techniques.

DIAS, O., CARVALHO, E., 1981. Aspectos da Arqueologia Amazônica **2**, 21.

BELLIDO, A.V.B., LATINI, R.M., NICOLI, I.G., SCORZELLI, R.B., SOLORZANO, P.M., 2011. XXXIII Brazilian Workshop on Nuclear Physics., AIP Conference Proceedings., 1, 1-5.



## **V72 Mixing Traditions – Mixing Cultures, Technological Choices for the Production of ‘Kampos Group’ Pottery in Prepalatial Crete**

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This presentation deals with the issue of the so called ‘Kampos group’ pottery in Crete (Greece), dated to the end of the Early Bronze I (c. 2900-2800 BC). Although it is widely spread in several sites across the north Cretan coastline, it is considered of Cycladic origin on the basis of pottery shapes and technology of manufacture. From this point of view the ‘Kampos group’ pottery constitutes a key element in the debate of the relationship between Crete and the Cyclades, the presence of islanders in Crete and the character of the ‘International Spirit’, a “panaegean *koine*” that characterizes the Aegean Early Bronze Age.

The combined typological and analytical study of the ‘Kampos group’ pottery assemblage from the cemetery of Gournes, on the north coast of central Crete, contributes new evidence on this issue and allows a reconsideration of the existing interpretations. The cemetery produced vessels of the ‘Kampos group’ alongside vessels following the Minoan repertoire. The typological study of the pottery reinforced the Cycladic character of the ‘Kampos group’ vases but revealed also deviations from the Cycladic prototypes, and a mixture of Cycladic and Cretan typological features. The petrographic and SEM analysis showed that the ‘Kampos group’ vessels were produced locally following techniques known from other sites on the island. Moreover, for the production of many vessels a combination of Cycladic and Cretan technological traditions was followed especially in the selection of tempering materials.

Considering technology as a dynamic cultural phenomenon, technological choices do not depend solely on material properties, distribution of raw materials, and available techniques but primarily on social, political or ideological factors. The archaeological and archaeometric evidence from Gournes reinforces this idea. The combination of different typological and technological traditions for the production of similar vessels seems to represent a conscious effort of the potters to express this mixture of Cycladic and Cretan cultural elements that characterizes many sites along the north coast of Crete. Within this context, the problem of the ‘Kampos group’ pottery seems far more complex than the simple pseudo-dilemma of the Cycladic vs. Cretan origin of the vessels and/or the potters.

### **V73 Technological and Provenance Study of Archaic Glassy Materials from Rhodes Island Using XRF and SEM/EDX Analysis**

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Two archaeological collections of glassy material are introduced and examined in this study, namely a Geometric to Archaic period's (8<sup>th</sup>-7<sup>th</sup> century B.C.) ascribed assemblage excavated from Rhodes island. The first collection consists of 86 glass beads while the second one of 10 faience samples. It has to be emphasised that according to certain ancient-glass scholars Rhodes is an established center for both glass-production and glass-making from end of 2<sup>nd</sup> millennium B.C. till Roman Times. The majority of the beads are plain, translucent, exhibiting the same colouration (blue, dark blue and white) and also transparent samples were selected while the faience samples are shreds of small figurines and vessels with blue-green decorations.

The aim of the study is twofold: to investigate the occurrence and concentration levels of metal oxides responsible for colouration/decolouration of samples and thus give solid answered to technological aspects; and also to attempt a classification of the raw materials used thus determining glass provenance.

The non-destructive examination of the samples was implemented using a portable XRF unit resulted the concentrations of minor and trace elements and SEM/EDX microscopy to provide major element concentrations.

The basic assumption aiming at tackling provenance issues by examining patterns of trace element results from the axiom that a number of these are refractory and non-volatile, meaning not affected by the high temperatures involved in pyrometallurgical processes; moreover their inter-regional variations are usually larger than intra-variations.

The results shed light to the technological and production issues raised in the study aiming also to a first cross-examine presentation of the two sites and to the development of a chemical databank for archaeological glasses.

**V74 Black- and Red-Slipped Pottery from Ancient Cassope (NW Greece): Inference of Provenance and Production Technology Based on a Multi-Analytical Approach**

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The present work reports on the results of a multi-analytical study of 90 pottery sherds recovered from the archaeological site of Cassope (mid-fourth to first century BC), in Epirus (NW Greece). Based on the archaeological criteria of date, style and fabric, the sherds were initially classified as local black-slipped pottery, local red-slipped pottery, eastern sigillata A and western terra sigillata (Gravani, 1994, 2004). The elemental composition of the ceramic bodies was assessed using radioisotope-induced energy-dispersive X-ray fluorescence (EDXRF) spectroscopy. Multivariate statistical treatment of the elemental data revealed different compositional groups and allowed distinction between local and imported products. Mineralogical analysis of the ceramic bodies by X-ray diffraction (XRD) indicated firing temperatures in the range from 800 to 1000°C for most of the sherds, while one group consisted of over-fired items, possibly in excess of 1050°C. Examination of the microstructure and chemical composition of the slip layers is underway, using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX). Preliminary results show that different pottery groups exhibit surface slips of different nature, in terms of thickness, degree of vitrification and elemental composition. Overall, the elemental classification combined with inferences concerning production skills and choices, shed light to different aspects of the political and socioeconomic history of Cassope. As part of an ongoing project (Papachristodoulou et al., 2006, 2010), the present work contributes to developing a compositional databank and establishing reference groups for Hellenistic pottery in the region of Epirus.

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## **V75 XRF Spectrometry and Late Bronze Age/Early Iron Age Pottery Within Interpretative Framework: An Outline of the Project**

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The object of these studies is a social context of diversity of the technological properties of Late Bronze Age/Early Iron Age pottery from Poland (so called Lusatian Culture). The analyzed vessels and fragments of ceramics will come from two different usable categories: everyday used pottery from settlements and cemetery pottery. They were produced for different purpose and there is a reason to consider if source materials and technology were also different.

The aim of the project is to analyze the chemical composition of used clay materials in production of these two categories of pottery. Already widely developed archaeometrical researches are offering few methods. In this project all analysis will be carried out by X-ray fluorescence spectrometry (XRF) which allows non-destructive determination of chemical composition of clay. Moreover, the handheld analyzer which I want to use gives the possibility to analyze outcrops of clay directly during excavations. The analyzed materials will come from two different microregions in Greater Poland so we can compare the results and check their recurrence. This way we will have the characteristic of both groups of pottery.

XRF analysis on prehistoric pottery have a potential also in theoretical considerations about Late Bronze Age/Early Iron Age societies. Pottery (as a most common archaeological material) can give us numbers of interpretative possibilities to understand the human behaviors in the past. Thanks to XRF method we can find out about material (clay) preferences for production of different kinds of vessels from the two categories which I mentioned above. In addition, the symbolic meaning of clay can also be investigated, for example different kinds of clay reserved just for producing grave pottery. These are just few of all the questions which I will try to answer with XRF spectrometry and chemical composition of prehistoric pottery.

## **V76 Characterization of Early Neolithic to Calcolitic Pottery from Los Cascajos Settlement (Northern Spain)**

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Los Cascajos constitutes one of older farmer settlement in the Iberian Peninsula. The site is located in the Odon river terrace a tributary of Ebro River. 65 potsherds of different pottery types of the Neolithic ware have been studied. The pottery correspond to ages ranging from  $6435 \pm 45$  BP to  $5100 \pm 50$  BP although the occupation of the site continues until  $3620 \pm 45$  BP. The studied samples are hand-made pottery, mostly fired under reducing conditions.

The petrographic study allows to determine several paste types and different manufacturing process. Five different petrographic types (TP) have been distinguished. The TP-1 paste has a fine-grained quartz-feldspar matrix with abundant intentionally added sparitic calcite temper. A very clayed fine matrix characterizes TP-2 paste type with almost total absence of quartz-feldspathic minerals and sparitic calcite is also used as tempering agent. The TP-3 paste has a fine-grained quartz-feldspar matrix, with abundant coarse quartz ( $>1$  mm in size) and grogs are used as a common temper. The nature of these grogs is similar in composition to TP-1 and TP-3 paste types therefore are fragments of pottery made these types of raw materials. TP-4 is constituted by a clayed matrix, around 75 vol% by visual estimation, with of minerals and rock fragments of large size ( $> 1.5$  mm) as natural non-plastic inclusions. The TP-5 paste is characterized by the presence of ophitic rock tempers. The petrographic features of TP-1, TP-2, TP-3 and TP-4 types are compatible with sediment collected in the surroundings of the archaeological site. The main differences between potteries of TP-1 and TP-3 consist in the handling procedure of the paste by adding calcite and limestone fragments, or grogs, respectively. Sample from TP-2 correspond to another local raw materials. Only TP-5 type samples (a total of 6 samples) are of foreign origin and exhibit petrological and chemical features similar to those of ceramics from the Iron Age sites found in the Pamplona basin.

The chemical features allow to distinguish two main groups one corresponding to foreign potteries (TP-5) and the other group constituted by all the local potteries (TP-1 to TP-4). Within local pottery several subgroups can be recognized according to the tempering agent.

The study is supported by IT315-10 research project of the Basque Country Government.

## **V77 Technology and Composition of Early Anglo-Saxon Glass Beads from Eriswell, Suffolk**

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The fifth-seventh century Anglo-Saxon cemetery complex at Eriswell, southeast England, is exceptional in that it spans an extensive period of Anglo-Saxon history, and is amongst the largest inhumation cemeteries of the period in the UK (Caruth & Anderson, 2005). A comprehensive range of approximately 400 monochrome and polychrome glass beads from the site have been studied using energy-dispersive x-ray analysis in the scanning electron microscope. The aim of this was to investigate the technology, origins and variability of early medieval glass beads, with hope to gaining a better understanding of the way in which the glass industry was organized during this period. The study has shed new light on the production technology of certain colours, most notably red glass, as well as chronological and typological variations in certain categories of bead.

Distinct compositional groupings have been identified corresponding to different bead types. In particular, tight compositional groups in the translucent blue glass beads, relating to quantities of minor colouring elements, suggest that there were likely to have been different workshops producing specific bead types. Additionally, there appears to be a relationship between the colouring agents employed in the production of certain glass colours and bead date. For example, two different opacifiers have been identified in opaque white glass beads; those beads ascribed to the earlier phases are opacified by a dispersion of tiny bubbles, whereas the later ones are opacified by tin oxide. There is also a strong correlation between the copper contents of opaque orange glass beads and their suspected dates.

The results demonstrate that considerably more information can be obtained by analysing small samples than by using non-destructive surface analysis techniques, as much can be missed in relation to the internal microstructure of materials and what this can tell us about technology.

## **V78 Defining Local Ceramic Production at Hellenistic Syene, Upper Egypt**

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Research on the typology of Hellenistic pottery from Syene (modern: Aswan) in Upper Egypt has already revealed extensive trade relationships with the Mediterranean region. At the same time, local ceramic production began to flourish by imitating the shape repertoire of such foreign vessels.

Our analysis aims to demonstrate how the Hellenisation of Upper Egypt from the 4<sup>th</sup> to 2<sup>nd</sup> centuries BC affected the material culture of Syene from a petrographic point of view. Thin-section petrography will be applied to analyse ceramic samples and raw materials collected in the vicinity of the ancient town. We will examine if and how the available Nile silt was manipulated by the potters of indigenous ethnicity to produce pots that are stylistically identical to those from the Greek cultural area. Our aim is to highlight whether or not a change in the technological choices of the local craftsmen - mainly in the selection of raw materials and paste preparation methods - can be detected when they started to manufacture Greek-Hellenistic vessel types.

Additionally, the adaption of Greek drinking, dining and, especially, cooking culture is a common phenomenon in Syene. In this connection it is of particular interest to focus on the functionality and performance properties of the locally-produced ceramics in order to test, for example, if those vessels were indeed sufficient for their intended Greek-inspired cooking practices.

For the first time, the local pottery production of Syene will be identified and studied in detail using archaeometric methods. The study contributes to the definition of local petrofabrics as well as to the documentation of socio-cultural interactions or acculturation between the Eastern Mediterranean and Upper Egypt.

## **V79 Making Pottery by the Lakeshore: The Case of the Lake Karla Settlements During the Neolithic (Thessaly, Greece)**

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The exact position of the now drained Lake Karla during the Neolithic period is a much discussed issue already since the beginning of the 20<sup>th</sup> c. Accordingly, a number of theories have been put forward on the development of settlement activity in relation to the lake (e.g. Alexakis, 2009). The Fitch Laboratory (British School at Athens) has launched a large-scale research programme on the archaeometric analysis of Neolithic pottery deriving from a large number of settlements in Thessaly, focusing on potting traditions and regional connectivity. This paper will pay special interest to the area of the eastern Thessalian plain where Lake Karla is believed to have been situated.

The integrated approach adopted includes petrographic analysis, refiring tests and raw materials prospection and experimentation, combined with the comparative examination of a large sample dataset (Schneider et al., 1991; Pentedeka, 2008). Preliminary ceramic analysis results from four sites in the vicinity of the lake (Halki 1, Magoula Visviki, Magoula Agrokipiou, Magoula Hatzimissiotiki) were very informative on the organisation of pottery production and ceramic technology. The hydrology of the southern Thessalian plain, the proximity to the lake and the detected fluctuation of the lake's shoreline due to the complex stream network of the surrounding hillsides seem to have a significant influence on the raw material supply for pottery manufacture. This had a major impact on the organisation of pottery production and the technological practices developed by the people residing in a lacustrine microenvironment.

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## V80 Portuguese Glazed Tiles (16<sup>th</sup>-18<sup>th</sup> Centuries): INAA, XRD and Luminescence for Raw Materials Characterization and Production Technologies of the Ceramic Bodies, and Chronology

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Glazed tiles (“azulejos”) are everywhere in Portugal, decorating walls of churches, monasteries and palaces as well as ordinary houses. The cultural materials to be studied in this work are ancient glazed tiles (16<sup>th</sup>-18<sup>th</sup> centuries) belonging to the Museu Nacional do Azulejo (Lisboa) and coming from Lisbon and vicinities.

The highlight of the Museu Nacional do Azulejo (MNA) is a blue and white composition of 1300 tiles, 23m in length, of Lisbon's cityscape (“Grande Panorama de Lisboa”). It was made around 1700, prior to the Great Earthquake (1755), and reputedly the country's longest tile piece (see figure).

Chemical analyses are done by instrumental neutron activation analysis (INAA) using the Portuguese research reactor (RPI, Sacavém); XRD measurements were performed by using a Philips X'Pert Pro diffractometer; and dating by luminescence techniques.

The main goals of this work are the determination of the raw materials variability for the production of the tiles to be painted by the artists of those times, and the technologies of production of the ceramic bodies. The results obtained so far point to several raw materials used in the workshops to produce tiles of different qualities for painting purposes. Among the glazed tiles studied, a special attention is paid to the “Grande Panorama de Lisboa”, classified as national treasure.

This work was conducted as part of the FCT funded Project: RADIART, PTDC/HIS-HEC/101756/2008.



## **V81 Developing a Novel Method to Identify Salt Production Pottery via the Chemical Release and Detection of Chloride and Sodium**

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Our work focuses on the development of an inexpensive technique used to identify salt-production pottery. Horiuchi *et al.* (2011) have recently proposed this method, whereby water-insoluble chloride ions trapped within pottery matrices are released via a chemical exchange reaction, and subsequently measured in solution through the use of a chloride ion selective electrode. Our goal is to both replicate and refine the Horiuchi method using both archaeological and experimental samples. We have produced salt pan in vessels obtained from a local ceramics studio in order to compare the results to a range of archaeological sherds thought to have been used for salt production, salt storage, or not affiliated with the process. Our artifacts come from the Mississippian site of Kimmswick, MO, a prehistoric saline site curated in the Peabody Museum, and two salt production sites in China: Zhongba, along the Yangzi River in the Three Gorges region excavated by one of us (Flad, 2011), and Shuangwangcheng, in Shandong Province (Li *et al.*, 2009). Preliminary results indicate that this method allows us to chemically distinguish certain vessels that were primarily used in the process of salt-making from those that were not.

In order to remove any soluble ions retained in the ceramic matrix, potentially introduced via post-depositional processes, the samples are first thoroughly cleaned by repeated sonication and washings with distilled water. A solution of ammonium fluoride is next used to exchange permanently-bound chloride for fluoride ions. The chloride ions, which had been permanently incorporated into the body of the pot during salt production, are thus released into solution and rendered easily measurable by means of a chloride ion probe. We are also paralleling the Horiuchi procedure by examining whether similar evidence might be observed with a focus on water-insoluble sodium ions, which may also become permanently integrated into the body of the pot alongside the chloride ions. We are pairing our analyses with a more direct identification of salt by elementally mapping cross-sections of samples using SEM-EDS, in order to verify the results of the novel technique with more traditional and costly approaches, as previously employed (Flad *et al.*, 2005). This elegant and cost-effective method has the potential to impact the archaeology of salt production worldwide.

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## **V82 Archaeometric and Archaeomagnetic Measurements on Greek Ceramics and Baked Clays: A Promising Combination**

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The purpose of this work is to link two parallel research fields, the archaeomagnetic and the petrological-mineralogical investigation of ancient ceramic materials. This combination is based on the fact that the determination of archaeointensity is a difficult procedure which requires very cautious evaluation of the suitability of the burnt clay-rich raw material. The quality of the intensity results highly depends on the mineralogical characteristics of the ceramics and one of the most important failure reasons is the bad stability of the samples due to insufficient firing in the antiquity and the burial conditions. These parameters either cannot be directly detected by the corresponding archaeomagnetic measurements, or, this is attempted after bad results have been obtained. On the contrary, relevant important information could be acquired from the petrological (polarizing and electron microscopy) and mineralogical (X-ray diffraction) analysis. Such measurements are able to elucidate the firing conditions (temperature, atmosphere, time) prevailing in the ancient kilns as well as the degree of alteration to which ceramics have been subjected during the post-burial processing.

The studied ceramic sherds and baked clays come from nine archaeological sites, situated in N. Greece, Cyclades and NW Peloponnese. All the sites are archaeologically dated, based on the ceramic typology and age-diagnostic objects. For some cases thermoluminescence, dating results are also available. The ages of the studied sites range from Early Bronze Age to Roman times.

For the majority of archaeological sites, the petrological and mineralogical data indicate that a wide range of firing temperatures varying from 500 up to 1000° C prevailed in their kilns and ceramics. A more narrow range of temperatures, T≈850-1050° C was applied to the kiln from NW Peloponnese. The preservation of primary clay minerals such as smectite, kaolinite and chlorite, representative of the raw materials of ceramic samples in combination with the absence of new-crystallized high-T mineral phases as mullite (Ca-poor ceramics), diopside and gehlenite (Ca-rich ceramics) during firing procedure determined the low temperatures. The presence of analcime and secondary calcite in high fired (T>850° C) samples from Cyclades and Peloponnese are the indicators for post-burial alteration phenomena.

The above results are compared to successful or failed palaeointensity experiments performed on numerous samples from seven out of nine sites. A satisfactory convergence between the two datasets suggests a promising perspective for the combination of the respective disciplines.

### **V83 Producing Black Glass During the Roman Period (Notes on a Crucible Fragment from Serdica, Bulgaria)**

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Recent excavations in the Roman and Late Roman city Serdica (present day Sofia, Bulgaria) prove that a diversity of urban production activities has taken place there, including secondary glass working. A ceramic shard with a thick layer of black opaque glass adhering has been found in an uncertain context, but probably from the Late Roman period of the site. This find demonstrates the technique of glass melting for colour modification, using a wheel-turned ceramic vessel originally made as a household ware. To our knowledge, this is the first direct evidence for black glass making known so far.

Our presentation aims to contribute to the current stage of research on different Roman glass technologies by presenting this crucible fragment, which provides a specific example of black coloured glass recipe.

The fragment was analyzed in the Wolfson Archaeological Science Laboratories in the UCL Institute of Archaeology by OM and SEM-EDS. The results demonstrate that the glass has a proper Roman composition with an excessive amount of iron oxide (ca 10 wt%) which gives its unusual colour. A detailed study of the microstructure of the material allows understanding the way in which the iron has been added to the glass batch, with small FeO particles seen in the glass at the bottom of the crucible.

It can be supposed that such specific black glass was used for shaping beads and / or bangles. An attempt is made in the presentation to discuss that technology in comparison with similar and contemporary finds from other regions of the Balkans.

#### **V84 Using Lead Isotopes for Cypriote Pottery Provenance - Signature of Cypriote Clay Sources and Comparison with Late Bronze Age Pottery from Cyprus**

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A recent study demonstrated the effectiveness of lead isotope analyses for pottery provenance studies (Renson et al., 2011). Late Bronze Age pottery fragments of different wares excavated in Hala Sultan Tekke were distinguished according to their isotopic composition and sherds of Plain White and Coarse wares locally made were linked to different possible raw material developed in the surroundings of the site i.e. Holocene sediments and marls (Renson et al., 2011). The clay sources analyzed were mostly collected in the Circum Troodos Sedimentary Sequence but also in the sediments derived from the weathering of some rocks from the Troodos Ophiolite (Renson et al., 2011). Cyprus is divided in four main geological areas (the Troodos Sequence, the Circum Troodos Sedimentary Sequence, the Kyrenia Range and the Mamonia Complex) of different ages and origins that contain rocks and sediments with variable isotopic signatures. To further investigate the use of lead isotopes to characterize the regional origins of pottery in Cyprus, we analyzed the isotopic composition of clay samples from the two geological zones, which had not been investigated so far, namely the Kyrenia Range and the Mamonia Complex. The isotopic compositions of all four geological areas of Cyprus are thus characterized. These clays are then compared to Plain wares from three Late Bronze Age sites located in the Circum Troodos Sedimentary Sequence (HST, Enkomi and Alassa). Finally, these isotopic results are compared to Base-ring fragments to investigate the interest of this method to discriminate raw materials possibly used for the production of this unique Late Bronze Age Cypriote pottery ware.

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## **V85 Petrographic Analysis of Ceramics from the Oyo Ceramic Complex at Ede-Ile (Nigeria)**

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Petrographic analysis of 50+ ceramic sherds was carried out with the view to understand variations in the mineralogical characteristics of several serving-bowl forms in the Oyo ceramic complex at Ede-Ile, a colony of the Oyo Empire of Nigeria, occupied ca. 1600-1840 CE.

We compare the results of this new petrographic study with information from stylistic and macroscopic analyses (Ogundiran, 2001), testing the agreement between the non-destructive and destructive techniques. In addition, instrumental neutron activation (INAA) analyses (Usman et al., 2005; Cecil et al., 2006) were performed previously on the same samples, producing a robust total dataset. In the earlier research, INAA was used to separate the suite of ceramics into source groups according to major and minor element composition. By identifying the mineral species from which the chemistry is derived, a petrographic study can corroborate or contradict the INAA results.

With this multifaceted approach, we can investigate variations in clay-sourcing strategies, sources of imports, and/or the proximate distribution patterns of the different vessel forms represented in the Oyo ceramic complex. The results are informative of the degree of intra-site variability in ceramic procurement and manufacture in Ede-Ile and can serve as pointer to potential regional distribution networks for specific ceramic forms in the Oyo Empire as a whole.

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## V86 Geometric Features Extraction of Ancient Pottery Decorations through Multiresolution Laser Scanning

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The aim of this paper is to test the utility of intensity data coming from multiresolution laser scanning surveys to extract geometric data and geometric features from ancient ceramics. Three laser scanners with different technical specifications have been tested: Konica-Minolta Vivid 910, Faro CAM2 Laser ScanArm, OGP SmartScope Flash CNC 300. All these type of instruments are very suitable in the framework of Cultural Heritage because they are non-contact instruments with a very high acquisition rate and precision up to 0.0006" (0.016mm) for the OGP. The software environment (Geomagic, Rapidform and MeshLab) is the same for all the dataset, composed by fragments of ancient ceramics coming from the archaeological sites of Al Khiday 1 (16-D-5) and 10-W-4 sites (Khartoum, Sudan), radiometrically dated to Mesolithic and Neolithic, and characterized by incisions of the surface. More than 10 different decorations types were distinguished (*wavy line*, *dotted wavy line*, *"lunula-shaped"*, *alternately pivoting stamp*, *rocker stamp drops*, *rocker stamp drops deep*, *rocker stamp dotted zigzag*, *rocker stamp plain zigzag*), and here geometrically described. Archaeological evidence indicate a defined stylistic evolution over the stratigraphic sequence, probably reflecting also changes in the production technology over the time.

A common data format has been chosen (STL - is a file format native to the stereolithography) in order to gain interoperability among different software platforms. Starting from "low" resolution acquisition the possibility to mosaicking different acquisitions in the same frame has been tested, inserting different scans at micron scale; lacunas can be filled automatically using standard procedures like "filling holes".

On the multiresolution model, where different areas can be investigated at different resolution levels according to different research purposes, some numerical algorithms and filters were applied to investigate the morphology of the surface. The curvature analysis was used to enhance local features, like letters, symbols or single descriptive elements.

## **V87 Salt Damage Related to Physical Properties of Ceramics**

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A pressing concern in the conservation of archaeological ceramics is the damage caused by soluble salts. When salts crystallize in the pores of a ceramic their expansion affects the internal structure of the matrix causing powdering and spalling of the surface, weakening the ceramic body. Salt damage to ceramics is of particular concern to conservators working with ceramics from the Southwestern United States. The Arizona State Museum in Tucson, Arizona has a collection of over 20,000 Southwestern ceramics which has a Save America's Treasures designation; over ten years of survey and study of this collection identified damage caused by active soluble salts as a pressing concern to the preservation of the collection. Certain patterns of damage were observed during this survey, and this research is concerned in identifying the factors that affect these patterns, and how they relate to the preservation state of a collection.

The factors that affect salt damage are the identity of the salt, the technological properties of the substrate and the environment of deposition. A set of Southwestern ceramics representative of the ones with the highest levels of salt damage in the collection of the Arizona State Museum was selected for a study of their material properties and how they relate to degrees of observable damage. A material characterization was carried out to study the physical properties of the ceramics: porosity, pore-size distribution and permeability. These properties relate to the technology of fabrication of the ceramics. Salts were introduced into the ceramic samples and they were run through an accelerated aging experiment to model the effect of extreme environmental fluctuations. Damage to the ceramic samples was assessed quantitatively by percent weight loss and percent increase in porosity. The results from this experiment showed that the physical properties of a ceramic sample determine the degree of damage a ceramic will exhibit from salt action, therefore implying that the technology of production is an important determinant in the degradation of ceramics.

This presentation will explore the pattern of salt damage observed in the pottery collection of the Arizona State Museum, and it will address the factors that cause salt damage with an emphasis on the preservation of Southwestern ceramics. Understanding these different factors can aid in understanding the pattern of degradation observed in collections, and can provide better guidelines for the treatment of ceramics with salt damage.



## V88 Primary Glass Production in Roman “Kastro Palaia” – Iolkos

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This paper is a first attempt to present and interpret new finds from the excavation of a Roman workshop for primary glass production in the important and extensive settlement on the hill “Kastro Palaia” of Volos, Greece. This site, inhabited from prehistoric times to the present, is linked by researchers to the ancient city of Iolkos, known from historical records.

Recent excavation finds from the Roman period of the site, especially the Imperial times (2<sup>nd</sup>-3<sup>rd</sup> centuries AD), attest a large complex of buildings, including bath installations at numerous locations on the settlement, an aqueduct, inscriptions and tombs. It is in this period that the workshop for glass production is dated; it has a paved floor, a well, a semicircular construction with ceramic tiles, coarse sherds of vessels, a deposit of waste, and water pipes as well as specialized structures with artefacts, and raw materials apparently relating to activities involving the “*chaîne opératoire*” for glass object manufacture.

Two pieces of raw glass are direct archaeological evidence for the primary production of glass during the Roman times in Iolkos. They are porous and mostly greenish in appearance. Initial X-ray fluorescence spectrometry showed a typical natron-based glass chemistry to which no opacifier had been added; the apparently milky appearance of the material is probably due to air bubbles and silicate crystal phases.

Evidence for Roman glassmaking from raw materials (as opposed to working glass into artefacts) is very rare, and mostly restricted to the Byzantine large kiln sites in the Levant. This presentation will explore the nature of the evidence excavated at Iolkos in more detail, trying to discriminate between glassmaking and working, and to link the glass to one of the major (late) Roman glass groups.

## **V89 Tracing Roman Glass: The Use of Trace Elements to Determine Primary Roman Glass**

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Samples of Roman glass dating to the 1<sup>st</sup>-6<sup>th</sup> centuries AD, excavated from Sagalassos, Turkey, and 1<sup>st</sup>-3<sup>rd</sup> century AD glass from Oudenburg and Tienen, Belgium were analysed using LA-ICP-MS. The colourless natron glass could be grouped according to the decolourisers used into Mn only, Sb only, and mixed Mn and Sb. The trace elements which can be associated with specific decolourants are identified, including Ba, Sr, V, As, W, and Mo. The coloured glasses can also be split in terms of the Mn and Sb, with the samples from Sagalassos containing little or no Sb and the samples from Belgium having a mixed Mn and Sb signature. The Tienen and Oudenburg samples contain higher amounts of Cu, which is not seen in the Sagalassos naturally coloured or colourless glass. The levels of Cu in these samples are too low to impart colour to the glass, this may, therefore, indicate the deliberate mixing or recycling of two or more different glass types. This suggests that the workshop which created the Belgian glass was either utilising primary glass from at least two sources or was recycling glass made from an alternative composition. The Sagalassos samples all have a very similar composition suggesting a similar primary glass source.

This paper discusses the variations in the trace element composition of Roman glass both geographically and chronologically. The potential minerals which may have been used as the primary decolourants are discussed, along with a consideration of the influence played by the recycling of glass. The contribution that a study of trace elements can make to identifying the potential sources of primary glass and associated raw materials is evaluated.

## **V90 Danger! High Voltage! The Application of HH-XRF to Different Archaeological Materials; Pitfalls and Potentials**

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HH-XRF is growing in terms of its popularity for use in archaeological studies. It has been regularly used for studying soils, obsidians and metals, but to what extent can its use be applied to other archaeological materials, for example glass and ceramics? How reliable are the studies that are undertaken using this technique? And, to what extent is a trained 'expert' needed to conduct the analysis? The influence of size, shape and thickness of an object, in particular, pose some significant problems. The parameters of the HH-XRF, including analysis times, current and voltage settings vary between objects and material types. The depth-dependency of the elemental distribution and the attenuation properties of the sample also require focus. As a result, the implications of qualitative, semi-quantitative (e.g. (standardless) fundamental parameters) and quantitative analysis (the use of regression methods) are addressed. This research looked at four types of archaeological material, obsidian, metal, glass and ceramic, and determined the minimum basic parameters needed for a useful analysis of each. From this it was possible to determine the extent to which these parameters varied between the different materials. HH-XRF has huge potential for *in situ* archaeometrical analyses, but the potential pitfalls for the unwary user can be many and great. This paper seeks to highlight some of the dangers associated with a 'point and shoot' technique. The equipment used for this study consists of a Bruker Tracer III-SD without using a built in quantification program.

## **V91 The Use of Handheld XRF for the Quantitative Analysis of Archaeological Materials**

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With the development of handheld XRF there is an ever increasing use of the instrument by archaeologists. The benefits of having a portable unit in the field to help direct excavation and provide instant data are exciting to say the least.

These instruments, although powerful and useful were not designed to provide quantitative data of unaltered archaeological materials no matter what the manufacturers will tell you.

This paper presents the issues related to acquiring quality data from archaeological materials using handheld XRF and provides suggestions on the best way to achieve these results. Issues related to the analysis of soils, metals, ceramics and organic materials will be discussed.

## **V92 Roman Grey-Clay Tableware “Imitations” of Late Republican Italic Models from Southwest Iberia: A Preliminary Chemical and Mineralogical Characterization**

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Within the framework of research regarding Italic imports to the Iberian Peninsula during the late II and the I century BC, this study focuses on the chemico-mineralogical characterization by OM, SEM+EDS, XRD and Raman Spectroscopy of a specific type of tableware artifacts: the grey clay pottery. Grey clay sherds have been found and unearthed from three Portuguese sites, Santarém, Castro Marim and Faro, in association with other Italic imports (Roman black gloss pottery, thin walled pottery, Italic amphoras and Italic common ware) and Baetican imports (common ware, amphoras). Under visual examination, the ceramic sherds are characterized by a grey clay paste and by a superficial blackish coating which could be used to macroscopically group the sherds into 4 different types (1-4). Among these types, Group 2 is the only one present in all three archaeological sites under investigation. As far as typological and chronological aspects are concerned, this production has often been regarded by the archaeological research community as tableware “imitations” of the Italic black gloss prototypes, which were probably developed within local-regional communities during the process of Romanization. The multianalytical approach adopted in this study has been used to assess the detailed chemical and mineralogical composition of both the grey paste and the dark coating of selected samples from the sites studied with the aim in particular to find, making use also of elemental and phase mapping tools, mineral markers such as, for instance, volcanic and /or metamorphic rock fragments that could be used to link the raw materials to local (or external) geological sources. Due to their proximity to the sea and well established ancient maritime routes, the sites studied are likely to have represented important consumption centers at the time: the archaeometrical approach has been used to determine whether the grey clay tableware were manufactured near the finding sites.

**V93 Constructing a Database for pXRF, XRD, ICP-MS and Petrographic Analyses of Bronze Age Ceramics and Raw Materials from Failaka Island (Kuwait)**

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Ceramic artifacts from Failaka Island (Kuwait) as well as raw materials from the surrounding areas of the archaeological sites were analyzed by means of petrographic thin sections, non-destructive portable X-ray fluorescence (pXRF), and high precision powder X-ray diffractometry in an attempt to fingerprint production centers, raw material sources as well as trade and exchange routes in the Bronze Age.

The study of the bi-phase composition of ceramics (matrix and clasts) is a powerful indicator of raw materials used in the manufacturing process (e.g., clays and tempering materials) and can be successfully deployed in understanding the physical parameters of the techniques of manufacturing. Mineralogical (e.g., the presence of mineral gehlenite, partial decomposition of micas, advanced fissuring in quartz and melting of quartz rims) and textural changes (e.g., developments of secondary pores and contraction voids around large clasts, later filled by melt) observed within the same sample and throughout the sample set, especially in the composition of the matrix, argue for relatively high firing temperatures of at least 800-850°C and oxidizing conditions.

The quantitative and qualitative mineralogical composition of the analyzed clays (e.g., illitic-kaolinitic clays) considered as potential sources of raw materials yielded positive results. However, the lack, in the vicinity of the site, of similar tempering material to the one identified in the artifacts (i.e., intermediate-acidic (meta)igneous rocks - granitoids and gneisses) precludes any unequivocal interpretation as to the origin of the raw materials and further on, to the origin of the ceramics and the location of the production centers.

## **V94 Craft Production of Pottery from a Bronze Age Site in North-Eastern Italy: New Results from Petrographic Analysis**

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Castel de Pedena (Veneto region, North-East Italy), is a fortified mountain village dated between the Early Bronze Age and the Late Bronze Age (XVIII-IX Century b.C.).

Many ceramic remnants have been found during the archaeological excavations in the last few years. Based on variations in shapes and decorations this pottery can be grouped chronologically into three different periods. The oldest vessels belong to the Polada culture (North Italy) dated at the Early Bronze Age, whereas a second phase can be identified between the Middle Bronze Age and the Late Bronze Age, and a third one to the Final Bronze Age/Early Iron Age (Leonardi, 2004). Only few pots are characterised by peculiar shapes referable to the eastern Wiselburg-Gata culture (Hungary and Adriatic area) and to the northern Laugen-Melaun culture (Italian South Tyrol and Trentino, Austrian East Tyrol and Swiss Grisons), respectively.

Archaeological and archaeometric analysis has been carried out with the aim of defining the production technology, to characterize in detail the compositional variability of the pottery in terms of chemistry and petrography, to characterize the type and provenance of the raw materials, to define whether pots referable to Wiselburg-Gata and Laugen-Melaun types were locally produced or imported objects and to define possible trades with these regions. In addition to the archeological finds, a series of clays and sands sampled from potential provenance sites were studied by optical microscopy (petrographic microstructural and textural analysis) and by image analysis on scanning electron microscopy (image and elemental maps), the mineral assemblages have been gained by X-ray diffraction (XRD), geochemical analysis by X-ray fluorescence (XRF) and multivariate statistical analysis.

The high variability in microstructural features and type of inclusions, and the low firing temperature reached suggests a standardised technology for the vessels dated between the Middle Bronze Age and the Late Bronze Age, while a not too standardised production, mostly for the more recent ones. The strong similarity with sands and clays collected induces to suppose that these pots were produced locally or in the proximity of the site.

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## **V95 Portable XRF Analysis of Sources and Distribution of Obsidian Artifacts in Iran and Syria**

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Using a portable XRF, the Bruker Tracer III-V Plus, owned by Yale University Department of Anthropology, obsidian artifacts from various sites in Iran and Syria were analyzed to better understand the pattern of access to obsidian sources.

Based on the results of a pilot project carried out in the spring of 2010 and the winter of 2011 (results presented by Tonoike as a poster at the Society of American Archaeology 76<sup>th</sup> Annual Meeting in spring 2011), the Bruker Tracer III-V Plus was carefully recalibrated using calibration samples borrowed from the University of Missouri Research Reactor Center that were specifically chosen for this assemblage, which seemed to show unusually high concentrations of some elements. This realization had caused some concern that the previous calibrations done based on the traditional MURR calibration samples may be significantly inaccurate. The obsidian samples analyzed were chosen from collections belonging to Professor Frank Hole at the Department of Anthropology, Yale University from various sites in Iran and Syria with a focus on pursuing the question of differences and changes in preferences of obsidian sources through both time and space.

This project is also part of a larger project in cooperation with McMaster University, which aims at comparing data across different laboratories and different analytical methods (EDXRF).



## **V96 Non-Destructive Sourcing of Prehistoric Pottery from the Southeastern United States Using pXRF**

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Ceramic artifacts from pre-Columbian Florida and elsewhere in the Southeastern United States were tested non-destructively using a portable X-ray fluorescence (pXRF) spectrometer to study local production and potential long distance trade prior to European contact. Following a pilot study done on 30 potsherds from five excavated sites in northern Florida, which appeared quite promising and was presented at the 38<sup>th</sup> ISA, this research was expanded considerably with analyses conducted on more than 800 ceramic samples from several counties in Florida as well as in Georgia, Alabama, Mississippi, and Louisiana.

The first part of this larger study was to confirm that it was possible to obtain sufficient and reliable sourcing data by analyzing the surface of native American ceramics, rather than using the established destructive method of extracting and homogenizing powder samples for analysis by XRF, INAA, or ICP-OES. Even though these ceramics were not painted or glazed, pXRF analyses were conducted on both inside and outside surfaces, and occasionally broken edges. The results obtained, for a broad range of major and trace elements, were calibrated against standard reference materials. In part due to the type and small size of temper used in these ceramics, and the 5x7 mm area tested, little variation was found for the same sherd, especially for trace elements.

Examination of the large amount of data for several late prehistoric and protohistoric groups (e.g. Fort Walton, Lamar, Weeden Island, Swift Creek) and ware types (e.g. cob-marked, incised, check stamped) strongly supports the utility of this method of analysis, with the ceramics falling into distinguishable site groups, and most of the artifacts tested apparently coming from multiple local clay sources. Some exceptions include Late Archaic Poverty Point and St. John's type ceramics, which support long-distance exchange between Florida and Louisiana.

While there are limitations with this method, producing trace element results for a statistically significant number of ceramic sherds non-destructively, rapidly and at low cost is clearly useful for addressing questions about ceramic production and trade, and the socioeconomic systems in which they are embedded. Further work is planned for testing of known clay sources.

**V97 Assemini Traditional Pottery Making Village in South Sardinia: Ethnoarchaeology and Ethnoarchaeometry**

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The ethnoarchaeological study of contemporary traditional pottery-making communities can provide knowledge on the clayey deposits and techniques traditionally used for pottery production in a given territory. The application of archaeometric techniques to ethnographic cases, on the other hand, is an excellent way to complement the ethnographic information and it is also useful to test theoretical and methodological assumptions that are on the basis of the archaeometric work.

This contribution presents the study of the pottery making tradition in the area of Assemini (Middle Campidano, Southern Sardinia), one of the main pottery production centres of the Campidano, since the 15<sup>th</sup> century.

The present contribution combines the information from interviews with the still working traditional potters, the existent literary sources and the archaeometric study of traditional common wares and raw materials. All the materials were characterised using a combination of techniques (XRF, XRD, OM by thin-section analysis, study of the grain-size distribution and Atterberg's limits) that enabled the full characterisation of the ceramic production and the raw materials used.

## **V98 Islamic Glass Weights from Egypt: A Non-Destructive Study by $\mu$ -XRF**

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Forty-five Islamic glass weights were analysed in order to determine whether their production was based on the use of an evaporite or plant ash as a fluxing agent. The weights bear an impressed inscription or a symbol, frequently reporting the name of the Imam ruling at the time of their production; this allows dating 38 of them to the Fatimid dynasty which ruled Egypt between the tenth and the twelfth century. Other three might be attributed to the Mamluk period and the remaining four could not be assigned to a definite age.

The weights consist of glass discs, a few centimetres in diameter and a few millimetres in thickness. They could be divided into eight groups on the basis of mass measurement; this also allows establishing a correspondence with coin values, such as the gold dinar or the silver dirham, or fractions of them, which were used throughout the Islamic area.

Since the weights could not be sampled, a non-destructive analytical technique had to be used for the analysis; micro X-ray fluorescence spectroscopy ( $\mu$ -XRF) was chosen at this purpose, due to its relatively low limits of quantification for major, minor and a number of trace elements, and its relatively high spatial resolution, the latter allowing one to analyse spots of unaltered glass.

Seventeen major, minor and trace elements were determined on the studied weights. The obtained results show that their composition is consistent with a production based on the use of sodic plant ash, due to the contents of  $K_2O$  (mainly between 2 and 4 wt.%) and  $MgO$  (mostly between 1.5 and 3.5 wt.%).  $Al_2O_3$  and  $CaO$  contents generally range from 1.5 to 3.5 wt.%, and from 6 to 10 wt.%, respectively, while both  $FeO$  and  $MnO$  levels do not normally exceed 1 wt.% apart from deeply coloured items. As for trace elements, the Sr content is generally below 400 ppm and that of Zr is below 80 ppm. Two opaque weights of late Fatimid production are characterised by significant amounts of lead and tin (15 and 4 wt.%  $PbO$ , and 8 and 16 wt.%  $SnO_2$ , respectively).

These results give further evidence that sodic plant ash had replaced evaporites for glass production in Egypt by the end of the first millennium AD. Similarity in composition among the studied glasses suggests recourse to the same recipe for obtaining reference weights for coinage over a relatively long span of time.

## **V99 Geobiochemical Features of Source Materials in Glass of Volga Bulgaria**

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Local glass-making in Muslim Volga Bulgaria emerged in 12<sup>th</sup> century. This is evidenced by archeological finds of distinct workshops and equipment in the remnants of the capital city of Bilyar, standardization and mass-production of glassware in Bulgar cities, as well as stable chemical composition of the sodium-rich ashen glass (Na-K-Ca-Mg-Al-Si/+Mn+Fe). For the explanation of this specific composition one should consider characteristics of the source materials of the Middle-Volga and Near-Ural regions.

In Western Kama region in many tributary valleys saline and solonetz soils are common. Sodium-sulfide solonetz soils were also investigated in Samara and Ulianovska regions. In tributary streams' low flood valleys the vegetation is represented mostly by halophilic plants, such as *Statice Gmelini*, *Chenopodiaceae*, *Atriplex Tatarica*. These plants generally tend to accumulate higher proportion of sodium over potassium minerals, which allows us to explain why the Bilyar glass is characterized by similarly high proportion of sodium. These geobiochemical features of the

The source of alkali earths was the dolomite limestone. Carbonate s were common in the region and were actively used by Bulgars in everyday activities. High concentration of alkali metal in form of calcium and magnesium oxides, as well as aluminum oxide reduced crystallization of the glass, enabled better thermal and chemical resistance.

Bulgar glass also contains rather high concentration of iron mineral (1.2-1.9%). Most of the Volga Bulgaria territory geologically characterized as part of the Kama and Volga-Kama provinces. Relative concentration of Fe in Bulgar glass is nearly identical to that of the Kama region sands. Presence of iron in glass is common and usually adds greenish tint. Although non-desirable in modern glass, in ancient times the natural color was used for decoration and did not present a functional problem.

Volga sands always contain less iron minerals when compared to Kama sands. As opposed to Bilyar, the city of Bolgar is located within Volga sand province of clean quartz sands with least non-desirable impurities in the region.

## **V100 Comprehensive Investigation and Reconstruction of a Pottery Workshop Used for Production of Glazed Ceramics in Bilyar City Excavation**

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In the 10<sup>th</sup> century in the Middle Volga and Kama region the Volga Bulgaria state was formed. Its peak of development is estimated to be in 12<sup>th</sup> to early 13<sup>th</sup> century, as evidenced from the volume of international trade and high level of craft development, including such technologically demanding arts as glass-making and glazed pottery.

This work presents the investigation of a workshop in the central part of the inner city of Bilyar, the capital of Bulgaria. Stratigraphically the find was located in the top of the cultural layer, undisturbed by any building construction since the destruction of the city as a result of Tatar-Mongol invasion in 1236 AD, thus providing a great opportunity for research and reconstruction. The items made in this workshop had rather original form and decoration not commonly found in Bulgarian ceramics yet bearing obvious similarities with Transcaucasian finds.

The dig area of 60 m<sup>2</sup> revealed full production workshop including dedicated potter's workplace with a two-disk pottery wheel, 100cm wide dual-camera ceramic oven (similar in design to a tadoor) with instruments, as well as separate resource pits, all indicating specialization in glazed pottery making. The workshop was a part of a larger craft complex which included ferrous metallurgy, jewelry and glass making. Stratigraphic data, including finds of Iranian ceramics with luster decoration, minai, glassware, jewelry, weapons and other items, indicates that the workshop can be dated to the early part of the 13<sup>th</sup> century.

Characterization of the materials in the workshop was based on the results of petrographical analysis of the glaze and petrography of the clay. Lead-silica transparent glazes colored with copper- and iron-rich minerals were used. Concentration of artificial additives (both organic and mineral) in the clay dough corresponds well to local resources available in the region.

Production of the workshop shows distinctive similarity to Transcaucasian ceramics on morphological level, which can be explained by the cultural interaction between regions. On the other hand, the resource base and technological process remain consistent with Bulgarian pottery traditions and reflect its further artistic development. In addition, a comprehensive 3D reconstruction of the workshop, including specific tools of the trade such as the kiln and the workplace with the potter's wheel has been performed in order to visualize and aid in comprehension of the development of arts and crafts in Bulgaria in its later years.

## V101 Home Sweet Home. Identifying Ceramic Production in the Burdur Plain, S.W. Turkey

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This paper will discuss the local ceramic production of small prehistoric communities from the Burdur Region, SW Turkey. The studied material was collected during the 2010 and 2011 survey of the Sagalassos Archaeological Research Project of the Katholieke Universiteit Leuven which has been conducting archaeological and interdisciplinary surveys in the territory of ancient Sagalassos since 1993. The 2010 campaign was the first of a series of intensive survey seasons in the Burdur plain which aimed to provide more information about the diachronic use of this plain by ancient communities. Special attention was given to the late prehistoric period in this region, which is one of the best known of South Western Anatolia mainly due to the excavations of Hacilar (Mellaart, 1970) and Kuruçay Höyük (Duru, 1996). However, still little is known about the relation of these large sites to their surroundings. In this respect our survey results and the material studies can contribute to a more complete image of the Late Neolithic to Early Bronze Age (6500 - 2000 B.C.) occupation as they can be contrasted with these large settlements.

The largest number of sherds that was collected during the archaeological survey dates from the Late Chalcolithic (4000-3100 BC) and Early Bronze Ages I - II (3100-2300 BC). The ongoing ceramic study consists of a macroscopic and petrographic fabric characterisation and typo-chronological classification, with the aim to investigate aspects of the technological matrix, craft organisation and product distribution, which can give information about wider socio-economic aspects of the Late Chalcolithic and Early Bronze Age I-II societies. The thin-section sample set contains 36 vessel fragments from similar macroscopic fabrics that occurs at different contemporary sites. Varying amounts of micritic limestone, calcite and chert inclusions are identified as the main aplastic components although the presence of serpentinite and amphibole minerals suggests the use of variable local resources. These results presented in this study give a good insight into the local ceramic production of the different prehistoric sites, but also indicates intra-regional and inter-regional interactions to a certain extent.

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## V102 Iron Age Glass in the Netherlands: XRF-Analysis of La Tène Bracelets

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Glass bracelets occur in abundance in the Late Iron Age in the Netherlands, with the largest concentration in the Eastern Rhine area. Thousands of fragments are known, mainly blue and purple in colour, but green, colourless and amber-coloured fragments also occur. Some bracelet types are decorated with yellow glass lines.

One theory suggests local production of these bracelets, using local raw materials. Another theory suggests the import of bulk glass, which was locally reworked into the bracelets. The aim of this study is therefore to elucidate glass provenance and shed more light on the production methods and raw materials used.

Around 2600 fragments, mainly amateur finds, from the collection of the Valkhof Museum in Nijmegen were analyzed using energy-dispersive hand-held XRF. This method is non-destructive and fast, resulting in a database of over 2900 measurements.

Low potassium values indicate that the glass was made using natron. The only sources for natron known at that time are in the Eastern Mediterranean, e.g. Egypt, making glass production from local raw materials unlikely. Furthermore, different groups could be distinguished on the basis of elements derived from sand and lime, such as Sr and Zr, and colourant elements, such as Cu, Co, Mn, Pb and Sn. Similar patterns were recognized in the elemental composition of Iron Age bracelets from elsewhere in Europe, such as Eastern Austria (Karwowski, 2004) and Southwestern Germany (Burkhardt, 2006). It is therefore most likely that raw glass was traded from the Eastern Mediterranean through Central Europe via the Danube-Rhine axis.

Correlations between Mn and Sr have also been found, indicating possible pitfalls for use of Sr isotope ratios as a proxy for the provenance of the lime fraction in glass.

In addition, the first results of Sr and Pb isotope analysis will be presented.

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## **V103 Colorants and Opacifiers in Late Antique – Early Medieval Mosaic Glass from Saint Severo (Classe – Ravenna, Italy)**

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The Basilica and the Monastery of Saint Severo are archaeological contexts of paramount importance for the study of the monument expansion in Classe. Built at the end of the 6<sup>th</sup> century, the history of the basilica is linked to that of the monastery, raised next to it at the end of the 9<sup>th</sup> century and active until the year 1512. Recent archaeological campaigns (directed by Prof. Augenti - University of Bologna) in the area of the monastery has led to the collection of mosaic tesserae. Most of the material is residual and cannot be precisely dated. Besides, two parts of mosaic floors detached in the 1960s from the Saint Severo area, have been recently restored, showing the presence of a few opaque glass. One of the floors comes from the area of the so-called *sacellum*, the first nucleus of the area (first half of the 5<sup>th</sup> century) and the second from the northern aisle of the basilica (6<sup>th</sup> century), but the two pieces are not exactly contextualised.

As a whole 52 tesserae of different coloring and opacity coming from the excavation of the monastery, from the floor of the *sacellum* and from the floor of the basilica were sampled and analysed. The aims of this work were to identify the raw materials, colourants and opacifiers employed in their production and to compare the three sample sets and confirm, when possible, their different chronology. Chemical analyses were performed by EMPA-WSD and the identification of crystalline phases was performed coupling X-ray diffraction and SEM- EDS.

The chemical analyses allow to classify all the samples as natron based glasses, consistently with their chronology. The use of colorants and opacifiers is rather different in the three glass sets. Lead antimonates have been recognized in the opaque blue and turquoise tesserae along with lead stannate in the green ones. A significant difference between the samples from the two mosaic floors and the tesserae from the monastery is the use of the decolorant in the colourless tesserae: manganese in the first and antimony in the latter.

The differences found in the three sample sets lead to the hypothesis of the use of different raw materials and to the confirmation of the different chronology of the production. The analyses of the archaeometrical data and their comparison with other mosaic glass from the neighboring area (eg Ravenna – Saint Vitale basilica) could give more chronological indications about the site.



**V104 Preliminary Results of Late Neolithic Ceramic Analysis from Hódmezővásárhely-Gorzsa and Aszód-Papi Földek (Tisza Culture, SE Hungary)**

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The aim of this study is to examine Late Neolithic ceramics from a Tisza culture site at Hódmezővásárhely-Gorzsa. Vessels were examined in thin section by polarising microscopy. Ceramics were also compared with the composition of daub and argillaceous soil sediments acquired in the vicinity of Gorzsa. The sediments were obtained by shallow drilling.

The primary aim of the research is to determine possible similarities and differences between raw materials, tempering practices and building techniques between the ceramics from Gorzsa. Moreover, sediments are also examined from the vicinity of Gorzsa in order to assess possible similarities or differences between sediments and ceramic raw materials, and identify potential ceramic raw materials.

The raw materials of the Gorzsa ceramics are very fine or fine (0,025-0,15 mm), the amounts of non-plastic inclusions are common to abundant (20-40%) and comprised of mainly monocrystalline quartz and muscovite mica. Inclusions are moderately to well sorted. The most commonly used tempering material was grog and hard pieces of clay also known as argillaceous rock fragments (ARF). The amount and size of tempering materials show extensive variability.

Some of the argillaceous sediments collected from the vicinity of the Gorzsa site are similar to the ceramics in terms of composition and mixing of different raw materials can also be assumed. The composition of local daubs shows remarkable similarity to the ceramics, however, daubs are very porous and contain phytoliths indicating the use of vegetal tempering.

The ceramic technology of Gorzsa is compared with that of a Lengyel/Tisza site at Aszód-Papi földek in north Hungary. The ceramics from Aszód show very fine to fine-grained raw materials containing significant amounts of non-plastic inclusions. The most common tempering practice at Aszód, similarly to Gorzsa, was grog and hard pieces of clay fragments (ARF). The results show that the ceramics at Gorzsa and Aszód were made in a similar manner, in spite of the significant distance between the two sites indicating a strong social relationship between the north and the south part of the cultural group.

## **V105 Hyperspectral XRF Imaging of Ancient Athenian Pottery: New Insights on the Production Technology by Utilizing the Full Spectrum at Every Pixel**

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We describe the use of hyperspectral XRF imaging to examine the production technology of Ancient Athenian red figure pottery (~5<sup>th</sup> c. B.C.). Typically when imaging using X-rays, regions of interest (ROI) are selected and integrated from the multichannel analyzer (MCA) to correspond to the K, L, or M lines of the elements contained within a sample. These ROIs are addressed with an X-Y position that can be unfolded into an elemental distribution image. In the process of pre-selecting ROIs, however, useful spectral information is thrown away (e.g., beta X-ray lines, Compton and Raleigh scatter, bremsstrahlung shape, etc.). Instead of using ROIs, it is now possible to produce a stack of images corresponding to every channel in the MCA thus producing a hyperspectral image cube. This image cube can be interrogated using a variety of image-based chemometric techniques such as principal component analysis (PCA) and image correlation analysis (ICA). Here we use a scanning micro-Xray fluorescence spectrometer ( $\mu$ XRF; Bruker ARTAX) to capture a full spectrum at every pixel position of slipped areas of Athenian pottery. By using these techniques we have discovered that the ancient potters were deliberately tailoring the chemistry of the slips to produce different decorative effects on these vessels. For instance, on some sherds it was found that the detailed line features contained more potassium and iron than the broad background washes. We show how this the hyperspectral X-ray cube has helped to reveal these new insights that would not have been possible using the standard ROI imaging approach.

## V106 A Technological and Provenance Study of Two Mycenaean Glass Collections Using X-Rays and Ion-Beam Analyses

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The aim of this paper is to present the advantages and limitations in the studies of archaeological glasses using a series of standard and state-of-the-art laboratory practices and moreover, to characterize and provide provenance assignments for the studied collections. Recent studies have shown that it is possible to obtain reliable data on the elemental composition of the glass by using a variety of scientific techniques. These data can provide essential information on past cultures' trade relations. Scientific studies on archaeological glasses should meet requirements of both solely non-invasive and robust analytical techniques (Sokaras et al., 2009). Therefore, the present study uses conventional (OM, SEM/EDS, milli-XRF) and advanced (PGAA, PIXE) analytical tools for the study of 2 archaeological glass collections from key Greek Bronze Age sites in the Peloponnese during the Mycenaean Era (16<sup>th</sup> -13<sup>th</sup> c. B.C.), namely Mycenae, Kazarma, Ancient Assini and Palaia Epidavros in the Argolid (total 57 samples) and from Pylos (total 53 samples) in Messenia. Sampling in these areas was prompted by specific archaeological questions aimed at investigating whether the glass artifacts were simply imported as ingots and/or previously shaped materials via a general network of exchanged goods derived from the established glass-making centers of Egypt and Mesopotamia or if they were produced in situ as part of an indigeneous glassworking and/or glassmaking tradition (Henderson et al., 2010; Walton et al., 2010; Polikreti et al., 2011). Mixed scenarios are also possible; among the various alternatives considered models involving substantial local primary glass production are thought to be the least probable ones.

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**G1 Using Photogrammetry and Laser Scanner for Digital Documentation of Architectural Remains in Ancient Jerash City of Jordan****Yahya Alshawabkeh**

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Photogrammetry is a generally accepted technique for the collection of three-dimensional representations of the environment. For this reason, this technique has also been extensively used to produce high quality 3D models of heritage sites and historical buildings for documentation and presentation purposes. During the last years, the efficiency of traditional image based techniques has been improved considerably due to the availability of systems for digital image acquisition. Additionally, terrestrial laser scanners came on the market, which allow for accurate geometric data collection by a dense measurement of 3D points.

Within the paper, image based techniques will be combined with laser scanning in order to benefit from the complementary advantages of both techniques. Laser scanning is advantageous with respect to accuracy and resolution of 3D point measurement, while image based approaches are superior to provide high quality surface texture for realistic object visualizations. Additionally, the integration of both techniques additionally simplifies and reduce data file size, which is frequently required for efficient 3D virtual presentation. The research aims on the development of tools for the efficient photogrammetric data collection. The applicability of these tools will be exemplarily demonstrated through data collected from different architectural remains in the ancient roman city of Jerash. The data was collected from Artemis Temple, North Theatre, the North Tetrapylon and the remains of the Byzantine church of Sts. Cosmas and Damian.

**Some of the Collected Data**

Figure 1. 3D Models of Artemis Temple

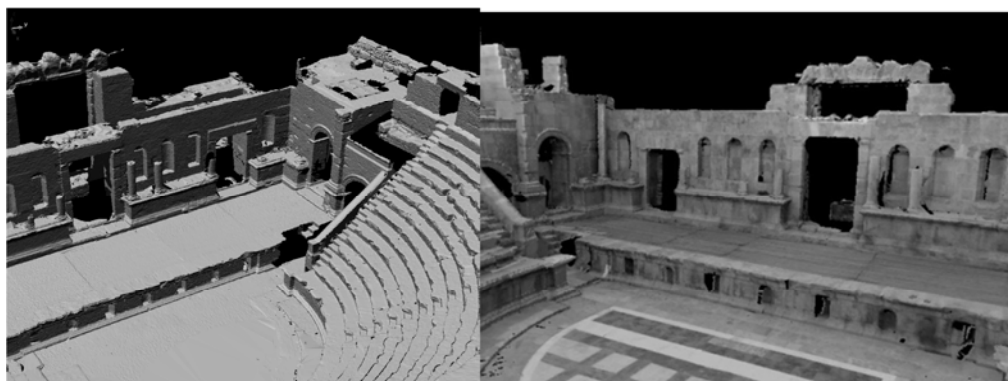


Figure 2. 3D Models of The North Theatre



Figure 3. 3D Models of the North Tetrapylon

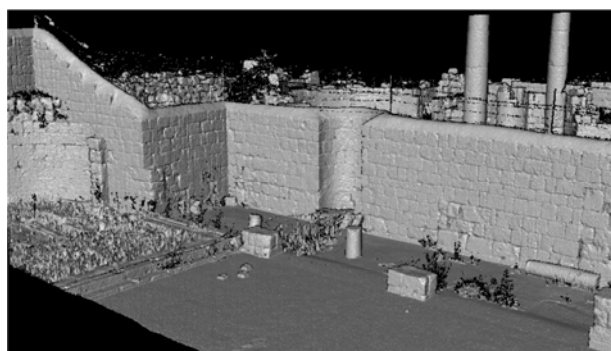


Figure 4. 3D Models of Sts. Cosmas and Damian church

## **G2 Integrated Prospection Project at Migdal, Israel**

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In 2010 the Israel Antiquities Authority, granted a license to Mexico to work on a Biblical Archaeology project. Two academic institutions were involved in partnership, the Anahuac University Mexico Sur and the National Autonomous University of Mexico (UNAM). The Migdal Archaeological Project goal is to develop a project that beginning with the application of the archaeological prospection methodology established by our Laboratory, continues with an integrative methodology based on the extensive excavation, the detection of activity areas, and the processing of all recovered information to reconstruct the history of the ancient Migdal town.

Following our prospection methodology, after analyzing aerial photography, regional topographic map was the first approach and the minor changes of relief were emphasized producing a residual map. The magnetic gradient survey covered 1.6 ha and provided large amount of information. It revealed a noisy map with large amount of magnetic dipoles produced by basalt fragments. In the central and western parts the basalt concentration increased but in the far east of the terrain, the dipoles concentration decreases. In some cases it was possible to recognize some straight alignments and closer to the southwestern limit, rectangular structures were observed. The electrical resistivity survey was conducted in areas where the magnetic gradient showed anomalies but did not defined structures. The electrical resistivity map is smaller than the magnetic but it has higher definition in the shape and dimensions of the buried archaeological structures. In the processed resistivity map we can observe clusters of small rooms. In the southeastern part the presence of two large rectangular structures was confirmed. After the first prospection season, the analysis of magnetic and electric surveys guided the archaeological excavation that confirmed the existence of the cluster of rooms forming architectonic spaces as a part of a Jewish first century town. All geophysical techniques showed the presence of a stone channel. After excavation, the discovered channel was made with basalt slabs and placed 50 cm below the surface. Comparison of the excavation and geophysical techniques showed a very close relationship. Most of the excavated walls have correspondence with the alignments showed by the electrical resistivity map and then it was possible to extrapolate to the non-excavated areas. In the southeastern corner of the land, electrical resistivity map also showed a diagonal anomaly SW – NE, that suggests the presence of a thick wall.

### **G3 Out of the Furnace and into the Field: The Implications of Reconceptualising Metallurgy as Practice**

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The study of ancient metallurgy is often understood as a problem which needs to be understood in terms of process. Thus, many archaeometallurgical studies focus on identifying and characterizing the chemical processes associated with smelting and other production steps. Whilst such studies contribute valuable data to our understandings of past craft practices they tend to focus on production debris rather than production contexts. The implication of such an approach is that contexts associated with production are often ignored in archaeometric analysis.

This paper begins from the perspective that metallurgy cannot be properly understood in terms of a process contained within a crucible or furnace but rather it is a social practice that gathers material resources and skilled practitioners to specific spaces in time. As such the contexts of practice are as open to archaeometric enquiry as are fragments of production debris. The paper illustrates how recent developments in on site analysis can furnish archaeologists with significant insights to how craft practices such as metallurgy are organized. The implications for field strategy, laboratory based analysis and the development of new research frameworks are discussed in light of relevant case studies.

#### **G4 Multi-Element Soil Analysis on two Classical-Hellenistic Sites in South-Western Turkey as a Proxy for Ancient Human Activity**

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In this study multi-element soil analysis is applied to two Classical-Hellenistic to Roman-Late Roman archaeological sites located in the suburban zone of ancient Sagalassos (Taurus Mountains, SW-Turkey). This research aims to evaluate to what extent geochemical analysis of soils and sediments can help to delineate and interpret ancient human activity areas. At the first study area, Çatal Oluk, geophysical research has revealed the presence of buried kilns, showing a spatial association with large concentrations of ceramic tiles and pottery collected during archaeological surveying campaigns. Also on the second study site, an area within the eastern suburbia of Sagalassos (commonly referred to as “the Potters’ Quarter”), geophysical anomalies are present, although their interpretation is more ambiguous.

At both sites, a total of approximately 100 topsoil samples have been collected in a regular grid, together with soil samples taken in several drill cores with depths between 0.7 and 2.5m. Aqua Regia extractions were conducted on the fine fraction (<63 µm) and the elements Al, As, Ba, Ca, Cu, Co, Cr, Fe, K, Mn, Mg, Na, Ni, Pb, P, Si, Sr, Ti, V and Zn were measured by inductively coupled plasma optical emission spectrometry (ICP-OES), using a Varian 720-ES apparatus. At Çatal Oluk, spatial and statistical data analysis revealed a geochemical anomaly of Co, Cr, Ni and Mg at and around the buried kilns. This signal probably reflects the remnant signatures of ophiolitic clays that were transported to the kilns, to be used as a raw material for the production of ceramics. In the Potters’ Quarter, an area with exceptionally high concentrations of Fe, Ti and V is detected. As this element association corresponds with the geochemical composition of iron mineralisations occurring around Sagalassos, these results might indicate that iron metals were processed in the Potters’ Quarter. However, in this case also the possibility of a local change in bedrock geology, causing an enrichment of Fe, Ti and V in the soils, needs to be considered.



## **G5 Mapping Prehistoric Settlement Patterns. The Application of pXRF in Combination with Geophysical Prospection Techniques**

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Chemical analyses of soils have a long tradition in archaeological research. Particularly in combination with other fieldwork techniques, they have a great potential for the identification of habitation patterns within ancient settlements. Since the 2008, the Early Bronze Age fortified settlement of Fidvar (Vráble) has been systematically surveyed and partly excavated by the Roman-Germanic Commission in a multilateral project with various international institutions (Lüth et al., 2008). This included geomagnetic surveys using vehicle-drawn 8-channel and 16-channel magnetometer arrays (MAGNETO®-MX ARCH by SENSYS). A total area of 120 ha were covered by these surveys. We used a new generation of pXRF technology (XL3t GOLDD+, by Thermo Niton) to conduct quantitative chemical analyses of soil samples extracted from representative areas of the site by coring. They were meant to provide an additional analytical perspective for understanding the habitation pattern of the prehistoric settlement and for facilitating functional interpretations of individual structures identified by the geomagnetic surveys.

In a first step, a measurement routine was developed using the above-mentioned pXRF device. It is equipped with a helium purge and thus enabled us to analyse also for light elements, most importantly phosphorous. The raw data are corrected using the fundamental parameter method that comes with the device and additionally calibrated by the measurement of certified reference materials. As an example, in tills phosphorous can be analysed with a relative standard deviation of five per cent at a concentration of about 900ppm (standard “CCRMP TILL-4”).

A number of 252 soil samples were obtained from several parts and depths of the Early Bronze Age site of Fidvar. They were analysed and the data were matched with the geomagnetic data using a geographical information system (gvSIG). Applying models derived from previous anthropological studies (recent overview in Holliday & Gartner, 2007), the dataset enables us to discuss use of space patterns of the prehistoric settlement of Fidvar. For example, a high concentration of phosphorous occurs in the massive fortification ditch of the site as well as in the alleys between the individual house structures, which can be well explained by the presence of human and animal excrements and the disposal of waste in these areas.

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## G6 Different Use of Magnetometric Field Methods in Czech Archaeology

### Roman Křivánek

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Magnetometric survey has been applied as the most frequent and powerful geophysical method in Czech archaeology during more than four decades. During this long time we could observe many quantitative and also qualitative changes of the real possibilities of these measurements. Using of vapour magnetometers or fluxgate gradiometers in 90 of the 20<sup>th</sup> century allowed change to extensive magnetometric surveys of large-sized archaeological sites or landscapes. These results brought from one point of view a large amount of area information about subsurface archaeological situations of more prehistoric or early medieval hillforts, enclosed sites, settlements or production areals (Křivánek, 2003, 2006). Some of these non-destructive results were included in more archaeological projects or changes of protection of archaeological monuments. Much greater density of measured data on the other hand contributed also to better archaeogeophysical interpretation in more detailed scale of particular archaeological features and situations. Due to change of density and sensitivity of measurement we could observe also archaeological sites with majority of small and shallow sunken features as for example graves in funeral areas or post-hole constructions in settlement or other areas (Křivánek, 2004). Another step to detailed archaeological information in scale of individual features or layers than brought an application of detailed measurements of magnetic susceptibility in situ. Simple use of kapameters during archaeological excavation helped to identify more unknown or visually unclear open archaeological situations. Quick use of kapameter during rescue archaeological excavations helped for example to detect horizontal or vertical changes of anthropogenic activity, separation of different magnetic properties of various features or differentiation of magnetic changes inside of fillings of individual features (Křivánek, 2005, 2008). Combination of different magnetometric field methods could be today also used not only for archaeological prospection of sites but also in more detailed systematic study of site and in uncovered terrains during different stages of archaeological excavation.

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KŘIVÁNEK, R., 2006. Magnetometric prospection of various types of large ditched enclosures in Bohemia. *Archaeological prospection* **13**, 25-43. ; KŘIVÁNEK, R., 2008. Detailní měření magnetické susceptibility v odkrytých archeologických situacích = Detailed measurement of magnetic susceptibility in an open archaeological situation. *Archeologické rozhledy* **LXI/4**, 695-724.

## **G7 3D Laser Scanning: The Digital Imaging of Fort Conger, Nunavut**

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This paper reports on the use of 3D laser scanning to document Ft. Conger, one of the most remote scientific stations in the Canadian Arctic. Ft. Congers was established in 1882 by an American First Lieutenant, Adolphus Greely. The wood framed structures at Ft. Conger provided researchers, military officers and staff a permanent base from which to conduct scientific research. Fourteen expeditions were conducted during the First International Polar Year from this site. The fort at Franklin Bay on Ellesmer Island has the distinction of being the place of a desperate escape of 25 members by sled and boat to Pim Island at Camp Clay. Only seven men out of the original 25 would survive their attempt. The structures and artifacts at Ft. Congers, though largely intact because of the cold temperatures, are beginning to experience some deterioration. On the site can be found several wood huts built in 1900 under Robert Perry's direction, using materials taken from the original 1881 structure built by Adolphus Greely. The foundation for the original building (18.5 m x 5.2 m) containing the rooms for staff and scientific work is still a prominent feature on the site along with a variety of preserved objects, including cast iron stoves, clothing, tools and hundreds of tin cans. In 2007, scientists from the Royal Military College of Canada identified the need for remediation at Fort Conger due to the elevated levels of arsenic and mercury present in some soils. The origin of these contaminants seems to be the Lady Franklin Bay Expedition, which used a variety of toxic substances to preserve natural history specimens. When the station was abandoned, the containers corroded and the chemicals leached into the soil. Remediation will be required for the removal of contaminated soil, creating problems for the preservation and protection of structures and artifacts. Laser scanning offered a key tool for digitally preserving the site and its artifacts before potential removal and remediation. In the summer of 2010, using both Z + F Imager 5006i W-Lan and Minolta Vivid 910 laser scanners, an archival 3D image of the site and artifacts was created. This data has served as a basis for a virtual reconstruction of the site from 1881-1900 that will educate the public on the importance of this scientific expedition in North American history.

## **G8 Advancements in Handheld XRF Technology for Field Archaeology**

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Portable XRF is used worldwide for research and discovery in a variety of academic disciplines. Handheld XRFs have been shown to be particularly effective for field archaeological studies as these in-situ, nondestructive analyzers provide fast, simple, accurate and cost-effective elemental and compositional analysis. They also provide immediate and comprehensive information that lead field archaeological research efforts to new levels of discovery.

Recent advancements in Handheld XRF include new tube target materials, optimized beam configurations, detector selection, integrated cameras, collimators, heat dissipation elements, hot swap battery replacement for 24/7 operation, and integrated XRF-GPS-GIS systems.

Advanced analytical calibration models are available that incorporate Compton normalization, fundamental parameters, inverse matrix overlap corrections, and other correction factors for quantitative analysis of elements from parts-per-million (PPM) up to high percentage levels for varying archaeological applications. Qualitative and quantitative analysis of light elements such as magnesium, aluminum and silicon; heavy elements such as lead, mercury and arsenic; precious metals such as gold, silver and platinum; and indicator ratio elements such as strontium and zirconium are all now possible in the field with these new advancements and analytical capabilities. Examples and explanations of these handheld XRF advancements and analytical capabilities for archaeological applications, including metals, ceramics, soils, and site delineation will be demonstrated. Additionally, guidelines for the selection of optimum portable XRF configurations for various archaeological applications will be presented.

**G9 Magnetic Gradiometry at Oğlanqala, a 1<sup>st</sup> Millennium Iron Age Fortress Site, Naxçıvan, Azerbaijan**

**Rob Sternberg<sup>1</sup>, Stella Dee<sup>2</sup>, Travis Johnson<sup>3</sup>, Veli Bakhshaliyev<sup>4</sup> and Lauren Ristvet<sup>5</sup>**

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Oğlanqala is a first-millennium Iron Age site in the Naxçıvan Autonomous Region, Azerbaijan. The site is on top of a 140-m high limestone hill, adjacent to the Arpaçay river and just south of one of the major passes through the Zangezur Mountains, one of the major north-south passes through the Lesser Caucasus. A fortress encloses an area of about 15 ha, with another 5 ha of possible outlying occupation located on the 50-ha hill. The site includes both Achaemenid and pre-Achaemenid levels. The architecture utilizes both limestone and imported basalt rock.

A cesium vapor vertical gradiometer survey was carried out during the summer of 2010. Seven areas at Oğlanqala were surveyed, covering a total area of about 0.9 ha, and two areas covering about 0.2 ha at the nearby tell of Maxta were surveyed. Unidirectional profiles, measurements every 0.2 s, marks every 5 m along lines, and line spacings of 0.5 m were used. Grid corners were surveyed in; tapes were used within grids. Areas surveyed included: the citadel, the area of the ancient palace; an area of private houses in the southeast of the site; the southwest plateau; a possible temple area on a basalt-strewn slope; the lower plateau; a kurgan in the valley beneath the site; and the nearby Early Bronze Age tell of Maxta.

Geologic signals of 200 nT/m were commonly seen in areas of outcrop. Anomalies with amplitudes of less than 10 nT/m and rectangular patterns consistent with room outlines having dimensions of about 10 m x 10 m are suggested for the “temple area,” the southwest plateau, and the lower plateau. Geometric signals in the kurgan with anomalies of <10 nT/m with some isolated larger values are also consistent with the presence of graves.

Magnetic gradiometry appears to be a suitable complement to archaeological survey and excavation to indicate habitation and activity areas at Oğlanqala.

## **G10 Long Distance Import of Polished Stone Artefacts: HP Metamorphites in Hungary**

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Polished stone tools made of HP metamorphites constitute one of the most spectacular items on the list of prehistoric long-distance trade goods in Europe. As the rocks are attractive, and of high quality, they were distributed over large distances mainly to W-Europe and scarce to E-Europe. In Hungary only 3 pieces of these tools known until now. It is, however, not easy to identify them unambiguously among “greenstones”. The first suspect pieces were located in the course of macroscopic and petrographic investigation of large collections on polished stone tools in the Veszprém Museum and the Hungarian National Museum. Recently, with the possibility to use non-destructive analytical techniques for chemical composition (PGAA), mineralogical composition (parallel-beam X-ray Diffraction) and crystal chemistry (EPMA) a series of “greenstone” axes were analysed and the first authentic pieces were demonstrated. Our results show that the raw materials of analysed “greenstones” are various metamorphites: greenschist-metabasite, serpentinite as well HP-rocks, nephrite and jadeitite/omphacite.

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## **G11 What is Left on the Floor? An Interdisciplinary Archaeological and Archaeometric Study of a Hellenistic House Floor at Düzen Tepe (SW Turkey)**

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In 2008, excavations of a presumed multi-room building were initiated at the Classical-Hellenistic settlement of Düzen Tepe (SW Turkey). The excavation activities were executed in a 5x5m Wheeler box-grid (Wheeler, 1960) and small finds – mainly hand-collected – were subsequently studied by find specialists. The building was soon identified as a complex consisting of a series of interconnected rooms around an L-shaped courtyard, constructed with fieldstone socles and wattle-and-daub walls, and with simple beaten earth floors. Based on an identification of the archaeological features – mainly plastered hearths, fire remains and refuse pits – and of the excavated objects, an interpretation of the complex as a “house” was suggested. Faced, however, with a lack of information on the functional interpretation of the individual rooms offered by a study of the architectural remains and archaeological features, with the uncertainty regarding the preservation status of the archaeological contexts and, consequently, with the question whether a contextual analysis was at all possible, the archaeological research was introduced to a new level of detail. In 2009 and 2010, the floor sediments of two of the rooms were sampled and analyzed according to the methodology for multi-elemental characterization of a mild acid extract of anthropogenic sediments, developed by Middleton (2004) for prehistoric house floors. A principal component analysis of the analysis results, with a clear reflection of the sample site’s geological background characteristics, allowed to identify chemical residues within the sediment of both rooms. A comparison with residues identified through ethnoarchaeological studies allowed link part of the residues to ancient activity zones (Vyncke et al., 2011). The study of the floor sediments thus not only offered a tool for the interpretation of functions within the individual rooms, but also allowed to positively evaluate the preservation state of the floor level itself. During the 2009 sampling all remaining floor sediment was collected according to the sampling grid and wet-sieved. Following the positive evaluation of the floor level’s preservation state, the spread of artefacts and ecofacts could be compared to the identification of the residue zones. This comparison, finally, allowed to evaluate the value of each of these studies for the functional interpretation of the individual rooms. The evaluation of the different studies performed is currently being executed and the results will be presented at ISA 2012.

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### **H1 Site Catchment Analysis and Non-Invasive Approaches in Romanian Chalcolithic. Case study: Cucuteni Settlements from Valea Oii river Basin**

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Archaeological site catchment analysis produces valuable information regarding prehistoric subsistence strategies and social organization. Despite a long tradition of studies on the Romanian Chalcolithic cultures, the analysis of human communities territorial behaviour remains underexploited. This work combines concepts used in landscape archaeology with the potentiality of a Geographic Information System (GIS) in order to mobilize archaeological artefacts in a large-scale setting and multiple thematic scopes.

This paper presents spatial and temporal distribution of Cucuteni settlements from the Valea Oii River basin, area well-known for its fortified settlements density and its extremely suitable soils for agriculture. Applying both integrated approaches through GIS analysis and geophysical prospections, its purpose aims to explore the natural, economic and social phenomena involved in territorial population dynamics during the Chalcolithic. In the chronological framework of the Cucuteni culture (5000-3500 BC), different kinds of viewshed analysis are computed in order to highlight the strategic nature of the area.

An exhaustive archaeological database was built for each area investigated, using both earlier and most recent published repertories. The main goal was to obtain, for every prehistoric settlement, different qualitative indicators regarding the spatial precision of the topographic mapping, the nature of settlement and its chronological framework, the quality and period of the archaeological information. Another goal was to focus on the natural indicators and on their relative attractiveness in order to define their relation with economic development by chronological periods. The general scope was to evaluate how prehistorical territories were constituted and how natural resources (water, salt and land) were the driving factors for these farming groups from eastern Romania. Visual analysis and spatial patterning allowed us to describe the territorial models which explain the original organization of these territories.



## **H2 From Heating Marks to Intensity of Fires: Thermal Characterization by Thermoluminescence of Reddened Wall in the Chauvet Cave and Fire Experimentations**

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Numerous rubefactions and desquamations are visible on walls of the Chauvet cave (Ardèche, France). We firstly wanted to know if these marks resulted from heating or chemical alteration. Thus, four fragments of reddened limestone were sampled from different locations (Galerie des Mégaceros, Diverticule des Ours, Secteur de l'Entrée). They were studied by thermoluminescence (TL) to characterize their possible heating state. In a first approach, it is possible to know whether a sample was heated or not by measuring the state of saturation of its natural TL signal (if the TL signal does not grow after a consequent laboratory irradiation, we can assume that it was not heated in the past). Secondly, thermal references samples were processed by heating for one hour at various temperatures samples of the same Urgonian limestone (a large piece of unheated material was sampled in the debris cone that plug the cave entrance) as the ornamented Chauvet walls. The comparison of shape and intensity of TL curves of samples and those of thermal references allowed us to estimate the equivalent paleotemperature (equivalent temperature depending on the thermal cycle characteristics chosen to make the thermal references). We showed that the reddened samples were heated in the past at more than 300°C.

The marks observed in the Chauvet cave were then caused by a quite intense heating and yet are sometimes stretched over some m<sup>2</sup>. To tackle the question of the hearth function that could be at the origin of the heat marks, fire experimentations in an underground environment (limestone quarry) have been carried out. We want to find what kind of fires can produce temperature over 300°C on walls and create marks comparable to those of the Chauvet cave (and what could be their function).

### **H3 Hearths in the Cave of Les Fraux (Dordogne, France): Thermal Characterization (Thermoluminescence and Magnetic Susceptibility) and Experimentation**

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Accidentally discovered in 1989, the cave of Fraux (France) is a rare example of a site of the Bronze Age (1300 BC) with archaeological vestiges (ceramics, bones, hearths) and cave art. This exceptional site, untouched since its occupation by Man 3300 years ago, shows traces of fires with reddened areas, ashes and charcoal.

In order to characterize the human occupation and its impact on the natural site, we decided to study some of the remarkably numerous ancient fires observed in galleries. A new methodology was implemented for two hearths in the cave of Les Fraux using thermoluminescence (TL) of quartz and magnetic susceptibility of sediment to determine past temperature attained by the surface sediment (palaeotemperature) and to draw map of temperature of hearth. The magnetic susceptibility was used to build a map of the hearth, border the heated zones and estimate the relative intensity of heating of the sediment. Then, the natural TL of fired quartz (extracted from sediment) was compared to that of thermal references. The latter were made by heating cave's sediment (not fired) from room temperature to 650°C in the oven of the laboratory. By comparison of anciently heated quartz to the reference ones, we were able to determine paleotemperatures. Finally, we showed that a relation between temperatures established by TL and values of magnetic susceptibility exists and permits us to obtain a map of temperature of the studied hearths. Two hearths in the cave had been studied by this methodology.

To confront the heating intensity found for archaeological fires, experimental fires were set up. We used the sediment of the cave of Les Fraux as substratum and we imbedded thermocouples at different locations and depths from the surface. These experimentations showed temperatures of the same order of magnitude to those found for archaeological fires and they informed precisely about heat propagation into the substratum (the drying of sediment is an important process), the quantity of consumed wood, the size of the rubefactions produced...These informations and reference data can be used to characterize intensity of ancient fires and to give some clues about their function.

#### H4 Trace Metal Levels in Terrestrial Ecosystem: What Relation to Ancient Mining Activities in the Morvan (Burgundy, France)?

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Considerable amounts of metals have been incorporated in soils over centuries deriving from ore-processing and metal-working (Chopin & Alloway, 2007). These pollutants persist in the modern environment and may affect flora, fauna and human being (Pyatt & Granttan, 2001). In contrast to involvement in the modern consequences of industrial activity, there is a lack of multi-variate studies focusing on the continuing impacts of ancient industrial activity (Fritsch et al., 2010; Mighall & Chambers, 1993).

Monna *et al.* (2011) has shown that aquatic ecosystem is affected by historical mining in the Cevennes (Southern France) till nowadays. This study is now enlarged to the Morvan (Burgundy, France) which is a pristine-like area but has experienced intensive metallurgical and mining activity from Antiquity to Modern Times; like the Cevennes. Not only the aquatic ecosystem but also the terrestrial environment is taken into account in order to have a transdisciplinary approach. In this communication the present terrestrial ecosystem in the Morvan (Burgundy, France) will be examined by combining chemical and morphological analyses in order to understand long-term effects of metals in soil. It is now admitted that pollution emitted in ancient times may persist in the environment (Pyatt et al., 2000). Contamination records of ancient industrial activities are commonly deduced from the analyses of ice cores, lake sediments or peat bogs (Hong et al., 2009; Forel et al., 2010). In order to establish a general scheme on how pollutants behave, a total of 221 topsoils and 466 small mammals were sampled at three locations affected to different extents by historical mining and smelting activities. This study investigates the spatial distribution of trace metal in soil and evaluates the leaching through soil profiles and metal uptake in wood mice (*Apodemus sylvaticus*). Isotopic compositions of lead contained in wood mice kidneys may help to identify the origin of pollutants (Komarek et al., 2008). The instability of morphological development, assessed through fluctuating asymmetry, is another parameter potentially sensitive to metallic contamination (Palmer & Strobeck, 2003). First ICP-AES analyses of topsoil confirm the high contents of metals in the Morvan mining soils. Local metal levels can reach twenty times the regional mean. These results, correlated with those obtained on wood mice kidneys, will contribute to understand bioavailability of leached trace

metals. The mining heritage should not be neglected when establishing strategies for long-term environmental management.

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## H5 Geological Conditions of Development of the Settlement of Pyrgos- Mavrorachi (Cyprus)

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The development of Pyrgos- Mavrorachi settlement in Cyprus, dating to the Early and Middle Bronze Age (Belgiorno, 1999) was closely associated with environmental conditions. This work was undertaken for the purpose of showing the influence on the formation and development of the settlement of Pyrgos- Mavrorachi by geological structure.

The village was founded at a strategic point of elevation between the sea and the area rich in copper deposits. The nearest river allowed access to the sea. It was the perfect solution for the metallurgical industry and trading activities in the second millennium BC (Belgiorno, 1995).

The access to the raw rocks materials, which were the basic building materials, was the very important factor. The commonly building materials from this site were used to take rock samples. Conducted research contained a series of petrographic studies included observation on thin section and SEM-EDS morphological and microchemical analyses. Investigations showed that the fossiliferous porous limestone was basic building material. In the lower parts of buildings were also used crystalline rocks, mainly basalts.

Comparative material also collected from surroundings of the site, the south of Cyprus area (Amathus, Kourion, Kouklia, Paphos) and the Troodos massif (Trimiklini, Mandria and Omodos). Analysis of the samples presented structural similarity with buildings materials of Pyrgos. Particular similarity revealed rocks from Amathus (about 10 km SW from Pyrgos). This indicates that building material came from the local area which belongs to the zone Circum Troodos Sedimentary Succession.

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## **H6 A Summary of Strontium and Oxygen Isotope Variation in Human Tooth Enamel Excavated from Britain over the Last 6000 Years**

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This talk presents a compilation of strontium and oxygen isotope data from human tooth enamel that has been produced at NERC Isotope Geosciences Laboratory over the last c15 years. These many and often small studies are here combined to provide an overview of data from Britain. The strontium isotope composition ranges between 0.7078 and 0.7165 (excluding individuals deemed to be of non British origin). The median Sr concentration is 84 ppm but there is a vector of increasing Sr concentrations related to seawater isotope composition that is seen in individuals predominantly from west coast of Scotland attributed to the use of kelp as a fertilizer. The oxygen isotope data is normally distributed with a mean value of  $17.7\text{‰} \pm 1.4\text{‰}$  (2SD n=615). Two sub-populations of local individuals have been identified that provide control groups for human enamel value from the east and west coast of Britain: east coast =  $17.2\text{‰} \pm 1.26\text{‰}$ , (2SD, n=83) and west coast =  $18.2\text{‰} \pm 0.98\text{‰}$ , (2SD, n=41). These data make it possible to make direct comparisons of population means between burial populations and the control dataset to assess commonality of origin.

## **H7 Discriminating the Fine Alluvia from the Rhône and the Saône Rivers Inundation Layers in Lyon in Archaeological Context**

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The Presqu'île of Lyon, located at the confluence of the Rhône and Saône rivers, is a very rich place as far as Gallo-Roman archaeology is concerned, and thus has been extensively studied. However, one of the major obstacles of these studies is related to uncertainties about the geomorphology of this particular geographical area. The path of both rivers evolved since ancient times, and several channels bisecting the Presqu'île are now vanished. Thus, when inundation levels are encountered when excavating archaeological sites of the Presqu'île, knowing if these flood sediments originate from the Rhône or the Saône is difficult, despite being an essential geoarchaeological data. The difficulty increases even more when faced with silt-only layer, since no easy way of discriminating Rhône fine alluvia from their Saône counterparts.

Our study focuses on this topic. In collaboration with the Longchambon laboratory from University Lyon 1, the Archaeological Service of Lyon has established a protocol to address this issue. Based on 20 XRay diffractometry measurements, we were able to analyze the elementary chemical composition of both rivers' fine alluvia, comparing modern samples with samples dating back to the Antiquity and coming from well-known areas (places where it has been proved that either the Rhône or the Saône was present). We identified patterns that allowed us to establish a chemical profile for the flood deposits of both rivers, thus permitting their discrimination.

We then applied this protocol to two archeological sites of the Presqu'île where the origin of the fine flood levels was still unknown. The 8 tested samples positioned themselves clearly within our model, further validating it, and allowing us to refine our knowledge of these sites. We especially observed that the same site could be flooded at different periods by both rivers, a fact that was suspected but not proven, further highlighting the fact that the Presqu'île was a rapidly evolving area, an essential data as far as anthropization is concerned.

## **H8 Land Use and Subsistence in Middle Neolithic Swifterbant (NL) Revealed by Soil Micromorphology**

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The Middle Neolithic Swifterbant sites (c. 4300 to 4000 cal BP) are located on levees of a former freshwater tidal system. Typical sites consist of a thick layer of black material that may be up to c. 1 m thick, intercalated between fluvial or fresh-water tidal deposits. These layers are commonly rich in fragments of pottery, bone and flint. They have traditionally been interpreted to be occupation sites for seasonal herding, hunting and gathering in a more or less pristine landscape. Recently, however, archaeologists started to contemplate the possibility that some limited cultivation was also practiced in the area.

Micromorphological study at one site (S4) showed that the black groundmass consists almost exclusively of carbonized plant remains and phytoliths, with remarkable similarities to midden deposits from early urban sites like Catalhöyük. The black layers may therefore be better interpreted as midden-like deposits, instead of occupation layers.

Moreover, micromorphologically identified anthropogenic disturbances in the sediment structure lead to the recognition of a layer with a regular pattern of tool marks, indicating tillage immediately below the midden-deposits. A detailed follow-up micromorphological study indicated that at least six phases of tillage alternated with phases of flooding and of midden formation on the same location.

These results have given a new image of the land use and subsistence in the Swifterbant area during the Middle Neolithic: Rather than extensive seasonal activities related to herding, hunting and gathering, the area was probably used intensively for a mixed subsistence in which tillage and crops played a major role.



## **H9 Multi-Isotope Analysis of the Population of a Lost Medieval Village, East Lothian, Scotland**

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The Auldham burial site was discovered during ploughing in 2005 and is located on Auldham Farm, near Tantallon Castle on the East Lothian coast of Scotland, UK. The foundations of a medieval church and a graveyard containing 242 burials have been excavated. The large number of well-preserved human skeletons provides an excellent opportunity to study the population origin and behaviour of a substantial group of individuals across the medieval and early post-medieval periods. The medieval period in Britain was a time of population influx and raids by groups including the Vikings are well established in the historical literature for this time, including in Lothian (Webster, 1997). This study aims to utilise a suite of isotopes to firstly investigate the population composition of the village (strontium and oxygen isotopes) and secondly to examine their dietary behaviour (carbon, nitrogen and sulphur isotopes).

The strontium and oxygen isotope analysis suggests that the group was predominantly comprised of a local, static population and thus allows the examination of the dietary habits of a remote coastal community. The combination of relatively high nitrogen isotope signatures with relatively low carbon isotope signatures suggests little marine protein in the diet. The clear evidence for a history of manuring at the site, which combined with relatively low sulphur isotope ratios, suggests the consumption of cereals was key to the diet and can explain the combination of dietary isotope values. This combination has previously been suggested to be unusual for the medieval period, but we propose it is perhaps more common than originally thought and can be explained by manuring practises. As there are few previous multi-isotope studies from Scottish medieval assemblages on this scale, the study provides an opportunity to build up a comprehensive picture of medieval and early post- medieval life in rural Scotland.

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## **H10 A Second Harvest? The Potential for Meta-Analysis of Stable Isotope Data ( $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ ) to Examine Large-Scale Climatic, Environmental and Palaeodietary Trends**

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Since 1977 stable isotope results ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) from over 19,000 individuals of archaeological interest have been published. This previously published data can provide valuable information about ancient diet, climate and agricultural practices through large-scale, inter-regional comparisons. Many studies use previously-published isotopic data for comparison, but there is little information about the validity of such comparisons across different locations and time periods. The purpose of this study was to provide a framework for future large-scale comparisons by performing a meta-analysis of previously-published data.

This study examined continental-scale trends in European human and faunal  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  over a 1500-year period. A data-set of 4021 individuals (human and fauna) from 115 Roman and Medieval sites was compiled from 40 separate studies published since 1998. This data-set also includes 435 new analyses from Roman and Medieval Italy undertaken as part of this study. Based on historical evidence, it was hypothesized that a decreasing population and economic collapse increased the availability of meat in the early Middle Ages. In order to examine this dietary trend, the meta-analysis investigated the effect of geographic variables (latitude, longitude, altitude etc.) on stable isotope ratios in humans and fauna.

Contrary to expectations, there was no evidence for an increase in human  $\delta^{15}\text{N}$  consistent with an increase in animal protein consumption in the early Middle Ages. The meta-analysis revealed large-scale isotopic trends which had not been apparent from previous small-scale studies. Some of the variation is due to non-dietary factors affecting the isotopic baseline. Future studies must take consider these variations when making inter-site comparisons. This study provides an example of how previously published data can be used to provide multi-scale, interregional dietary reconstructions. This paper concludes by highlighting the importance of creating standard publication criteria, including secondary mass spectrometry data, to enable accurate inter-laboratory comparisons in the future.

## **H11 Development of an Anthropogenic Landscape in the Belgian Loess Belt after 5000 Years of Agriculture**

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This study concerns the human influence on the landscape and sediment dynamics for the Dijle catchment (780 km<sup>2</sup>), located in the Belgian Loess Belt, where agriculture practices were introduced ca 5000 a ago, and peaked during the last 1000 a. We combine a field study with a modeling study to extract qualitative and quantitative information, and compare the human influence with the influence of natural variations like climate variability. The field study includes the establishment of a time differentiated sediment budget, while sedimentation rates are compared with variations in anthropogenic land use and climate. Palynology is used to make a detailed reconstruction of anthropogenic land use changes. A spatially distributed soil erosion and sediment redistribution model was used to model the hillslope sediment dynamics for different scenarios covering the Holocene.

Results of the field study show the overwhelming impact of anthropogenic land use on sediment dynamics, but remain inconclusive on the influence of climatic events. The combination of the palynological and geomorphologic data shows how human land use in the catchment is responsible for changes in the landscape. After the introduction of agriculture, a first peak of soil erosion starts, of which the major part is deposited as colluvium. The largest amount of soil erosion and sediment distribution occurs during the last 1000 a, resulting in a major increase in floodplain and colluvial deposition. As a result, the floodplain morphology changes from a marsh system which lacks a clear river channel and where an Alder forest is dominant, towards a sediment infilled valley with a clear channel and levees, with a more open vegetation dominated by *typha angustifolia*.

Modeling results are in agreement with field study results, providing insight in the increased sensitivity of the landscape for erosion events due to human land use. Land use changes result in an increase in soil erosion by more than 6000%, while climate changes contribute for ca. 6%. The recent increase in built up area is responsible for decrease by ca 50% in soil erosion. The combination of the modeling and field approach allows to understand the importance of the changing connectivity of the different parts of the sediment pathway, which has major results on both the hillslope and the alluvial landscape.

## **H12 Late Pleistocene Climate Seasonality: Impacts on North African Palaeolithic Populations**

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Climate records spanning the late Pleistocene-Holocene transition in North Africa show large-scale variations in climate, particularly in moisture availability, linked to both rainfall amount and temperature. At the same time the archaeological record indicates considerable changes in human behaviour in this region. In order to investigate if climate variability was a driving force behind these cultural changes we need climatological and archaeological records that operate at the same spatial and temporal scales.

Haua Fteah, a large cave site in the Gebel Akhdar of northeast Libya, provides the means to investigate this. The cave, and wider region, shows widespread human occupation during the last 20,000 years. The large faunal assemblage found within the cave allows annual to sub-annual climate reconstructions to be produced through the isotopic analysis of tooth enamel.

Presented here are oxygen isotope results from the analysis of Barbary sheep tooth enamel. Bulk analysis, which represents an annual average signal shows the late Pleistocene was significantly more arid than the Holocene. Intra analysis, which represents a sub-annual signal, shows that climate seasonality was much greater during the late Pleistocene than the Holocene. Human populations living within the region would therefore likely have had to develop adaptive strategies to deal with these climatic changes.

### **H13 Site Formation Processes and Mining Operations at the Prehistoric Salt Mine of Hallstatt, Austria: A Geoarchaeological Multi-Method Approach**

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During the late Bronze and Iron Age, the area of Hallstatt developed into one of Europe's earliest large-scale industrial sites and became part of a continental trading network, based on the extraction of subterranean salt deposits. The prehistoric salt mine of Hallstatt presents a unique interface between social, economical and environmental agencies and patterns and is key to understanding social structures and human-environment interactions of European prehistoric societies. Around 1000 BC, a large collapse sealed in former worked tunnels in the *Christian von Tusch* mining complex and preserved many archaeological features (Kern et al., 2008).

In order to understand the exceptional and complex formation processes of the structures and activities underground, which are crucial to interpretation of the mine's operation and collapse, this research investigated the recovered main deposit types on a series of scales. Micromorphology has been chosen as principal technique to examine the microstratigraphy of selected sedimentary sequences. The study of composition, internal structure and transition zones of each stratigraphical unit provided data on the mine's formation processes, operation, organisation and spatial concepts prior to the collapse. The cause, sequence and chronology of the catastrophic event has been specifically investigated by geochemical (XRF, XRD, pH) and sedimentological (particle size distribution) characterisation and comparison between collapse deposits from under, and potential sources above ground.

Results indicated a more complex micro-stratification than previously anticipated and a relatively short period of time between the cessation of mining activities and the sealing of the underground cavities in the course of the catastrophic event. This observation favours environmental over social or economic reasons for the shut-down of the late Bronze Age mining complex *Christian von Tusch*, possibly forcing people to relocate and altering the economic system of Bronze Age residents of Hallstatt. The analysis of the collapse deposit recovered in the *Christian von Tusch* mining complex speaks for a slow, creeping depositional process triggered by an earth/debris flow on the valley surface. A pollen assessment of selected sediments in the mine emphasized the importance for further palaeoenvironmental investigations to complement the picture of the Hallstatt salt industry and its impact on the surrounding environment.

KERN, A., KOWARIK, K., RAUSCH, A.W. AND RESCHREITER, J., 2008. *Salz-Reich, 7000 Jahre Hallstatt, Veröffentlichung der Prähistorischen Abteilung (VPA)2*. Verlag des Naturhistorischen Museums Wien, Wien.

#### **H14 A Geoarchaeological Study for the Localization of the Prehistoric Harbour at Akrotiri, Thera**

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Large-scale excavations conducted over the last 45 years at Akrotiri on Thera, the southernmost island of the Cycladic archipelago in the Aegean, have brought to light one of the most important Bronze Age settlements in the region, buried under a thick mantle of volcanic ash. Although the extent of the settlement is still difficult to estimate, the investigated area of about one-hectare seems to be its central part. When the settlement was buried at about the end of the 17th century BC, it was a well-organized city with a developed urban plan and an efficient drainage system under the paved streets. Connected with the sewage system were the sanitary facilities of the imposing two- and three-storey houses, which were furnished with exquisite pieces of furniture and decorated with unique wall-paintings.

This high standard of living, as revealed by both the architecture and the movable finds, indicate that the wealth of the city's inhabitants was largely the result of maritime trade with Eastern Mediterranean lands, as it is reflected in the wall-painting of the 'Flotilla' from the 'West House'. There are reasons to believe that the boom in the Theran economy, evident in the first half of the second millennium BC, was due to the trade of copper from Cyprus to Crete in response to the great demand for this commodity by the emerging palatial system on the latter island.

Obviously, such maritime operations and overseas trade required adequate harbour facilities, which are difficult to locate because of the thick deposits of volcanic material. By using geoarchaeological techniques we aim mainly at locating the city's harbour(s), as well as investigating the existence of any possible tsunami deposits from the 'Late Minoan' volcanic eruption.

Study of the LBA palaeo-topography of Akrotiri involves 'stripping off' the post-eruption deposits. The elaboration of a subsurface contour map will facilitate the identification of the palaeo-coastal landforms that might have favoured the development of the prehistoric harbour (or harbours).

The cartographic outline of the palaeo-relief under investigation will require a combination of approaches, including insitu geological and geomorphological research employing also GIS techniques; sampling of sediments from new trenches for further analyses, such as sedimentological assessment by means of grain-size analysis for the identification of the palaeo-coastal depositional environment; mineralogical and chemical analysis of samples by XRD and SEM-EDX and geochronology of sediments by OSL technique.

It is anticipated that the synthesis of the results obtained from archaeological, geological and analytical data will facilitate the determination of the 'possible' location(s) of the prehistoric harbour of the site.

Preliminary results from this recently initiated project will be presented in this paper.

## H15 Sediments and Settlements – Geoarchaeology in the Eastern Nile Delta

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Comparing to the other regions of Egypt the archaeology of the Nile Delta is still underrepresented. In the common opinion the deltaic archaeological sites either don't exist or are eroded, or covered with thick sediments (Hassan, 1997). Only last 20 years brought some new surveys and excavations in the Delta, especially in its eastern part (e.g. van den Brink et al., 1987; Andres & Wunderlich, 1991, 1992; Ciałowicz, 2008).

The Nile Delta confronts archaeologists with several specific problems. Its complicated geomorphology and paleogeomorphology together with composed sedimentation phases request the close cooperation of archaeologists and geoarchaeologists or geologists (Butzer, 2002). Various drillings and geophysical studies as well as tectonic research, not only in the Delta itself, helped to decipher some evidence from the Nile past (e.g. Coutellier & Stanley, 1987; Chen & Stanley, 1993; de Wit, 1993). It is the base for interpretation of recently discovered Predynastic/Early Dynastic sites in the Eastern Nile Delta (Pawlikowski & Wasilewski, 2007; Ciałowicz, 2008), their occupation phases and spatial distribution.

This study is based on the geoarchaeological program (auger drillings, field surveys, petrographical analysis) and archaeological excavations on the Tell el-Farkha and Tell el-Murrah archaeological sites as well as on archaeological surveys and spatial computer analysis. Both excavated sites are located in the eastern part of the Nile Delta and are studied by Polish Archaeological Expedition from 1998 and 2009 respectively. The research is based on the extended series of auger drillings, interpretation of archaeological profiles, petrographical analyses of sediments and comparisons to other studies.

The reconstruction of paleogeomorphology ("turtle backs", the Nile channels' beds etc.), sedimentation rates and sequences, together with archaeological interpretations makes complex environmental and settlement analysis possible.

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CIAŁOWICZ, K.M., 2008. The nature of the relation between Lower and Upper Egypt in the Protodynastic Period. A view from Tell el-Farkha. In: MIDANT-REYNES, B. AND TRISTANT Y. (Eds.) *Egypt at its Origins* **2**, 501-513.

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**C1 Direct Analysis in Real Time Mass Spectrometry for Identification of Organic Dyes**

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Direct analysis in real time (DART) is an ambient ionization method for mass spectrometric identification of small molecules (less than 1000 Daltons) that requires little or no sample preparation. This nondestructive technique has been widely used for forensic science and pharmaceutical applications since its introduction in 2005. In the study of art and archaeological materials, few relevant applications have been reported to date. We report here on our ongoing work towards identifying natural organic dyes in textiles by use of DART with high resolution mass spectrometry.

Madder is one dye of archaeological significance; it has been identified in 4000 year old Egyptian textiles by use of surface enhanced resonance Raman spectroscopy (Leona, 2009), a method that requires extensive sample preparation. High performance liquid chromatography also utilizes destructive extraction procedures for identification of organic dyes. With DART-MS, alizarin and purpurin, the colorants from madder, can be identified by exact mass directly in textile fibers without sample preparation in less than one minute. Because DART-MS under the conditions used produces M+H ions without fragmentation, the high resolution ( $\pm <10$  milliDaltons) mass spectra are specific for all but structural isomers. The primary colorant compounds in indigo, bloodroot, sandalwood, turmeric, and some quercetin-containing botanical sources have also been studied with DART-MS (Selvius DeRoo & Armitage, 2011). The use of simple, minimal solvent extractions and in situ derivatization has the potential to expand the applicability of the method, and orifice switching, which induces fragmentation, should allow differentiating structural isomers like morin and quercetin. We are currently applying DART-MS to archaeological textiles from the collections of the Detroit Institute of Arts, focusing on South American textiles from the Huari culture.

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## **C2 Scientific Examination of Quran Fragments on Parchment**

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Parchment produced from the dermis of animal skin, were widely used after the 2<sup>nd</sup> century AD to produce ancient manuscripts especially for Quran manuscripts in Islamic countries. This study has relied on non-destructive analysis techniques because of the limited and precious nature of the objects under study. In this work authenticity, the material of the document, identification of black, red and green ink of nine Quran fragments parchment belong to 10<sup>th</sup> AD was studied. The authenticity of documents has been established by the examination under UV light and the nature of them was determined by Fourier transform infrared spectroscopy (FTIR). Scanning electron microscopy in combination with energy dispersive X-ray microanalysis (SEM-EDX) was used for the elemental analysis of inks (Stuart, 2007).

The results show that these parchments are severely damaged and brittle. They display marks of environmental and water damages as well as of harm by mildew and mold. The parchments are authentic with no faded, broken, or augmented lines. The ink used in these fragments is not terribly damaged and does not show much scattering or loss. Elemental analysis of ink shows that red ink used in some samples was made from vermilion and some samples include iron oxide. The green ink contains of a considerable amount of copper; therefore it should include verdigris. The results showed also that black ink, must be iron gall ink. Some byproducts from tanning and parchment preparation have also entered the analysis of inks of these fragments.

STUART, B., 2007. *Analytical Techniques in Materials Conservation*. John Wiley & Sons, Ltd.

### **C3 What Did it Look Like? Reconstructing the Original Appearance of Decorative Metalwork**

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Many archaeological publications are illustrated with line drawings of artefacts which show details of their form, but not their colour. More recently, colour photographs have become commonplace but even they usually fail to convey the original appearance of the objects. This paper aims to demonstrate how the polychrome effects achieved by metalworkers over the last two millennia can be reconstructed, based on the careful scientific examination of archaeological finds.

When considering decorative metal artefacts, there are a number of factors that have to be taken into consideration if their original appearance is to be reconstructed. Usually the major change is the corrosion the object has undergone during the time it has been buried. The nature of the corrosion depends on the soil conditions as sometimes these are relatively benign but in other cases very aggressive. In most cases, corrosion processes change the colour of the metal, and may also affect any additional applied materials.

Other factors that have an effect on the appearance of an object are the composition of the bulk metal(s) or alloy(s) of which it was made, and any deliberate patination that it may have undergone in antiquity. Many metal objects also originally had surface additions which changed their appearance. These could be metal inlays, overlays or platings, or applied non-metallic materials such as gemstones, glass, enamel, bone or other animal-derived materials. Organic materials such as *vernīs brun* also changed the original appearance of the artefact, and sometimes traces of this can survive.

The range of materials available to craftsmen in the past will be briefly described, and the effects that were produced by using them will be illustrated. The ways in which the original appearance of objects can be re-created, and examples of the bright and striking polychromy that was achieved, will be demonstrated.

#### **C4 Of Some Blue and Bluish Grey Pigments in Medieval Mural Paintings in the South West of France**

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According to some historians, blue pigments are hardly used before 11<sup>th</sup> century in medieval mural paintings (Pastoureau, 2000). One of the reasons is the lack of availability of raw materials. Consequently, the palette consists of pigments more accessible as black (calcination of organic materials), white (lime), and various nuances of yellow to red obtained with ochre. When blue pigments are used, they must be imported (azurite, lapis lazuli), what makes the coloring materials expensive, reserved for important zones of the painting. In the 11<sup>th</sup> century, the status of blue colour changes. It settles, i.e., in the iconography, as the color of the mantle of the Virgin.

From the 12<sup>th</sup> century, the increase of the use of blue colors obliged, sometimes, to look for cheaper materials or to find technical solutions to obtain, at lesser expenses, the needed colored effect. In the first case, the recourse to particular pigments as aerinite, sources of which, very punctual, are situated in the limit between Aragon and Catalonia. This pigment was abundantly used in the North of Spain and examples of which we found in medieval mural paintings in the south west of France (Danielet al., 2008). On material and stylistic arguments, we can suppose that these pigments were imported by itinerant workshops. It remains an isolated case, although there is a source of aerinite in France (Saint Pandelon, Landes) of which we ignore if it was exploited - or even known - in the medieval period. Another means consisted in employing a mixture of pigments reproducing a colored grey bluish effect. These "false blue" were observed in some paintings in the south of France.

The analysis of all these types of blue pigments in seven sites dated between the 12<sup>th</sup> and the 14<sup>th</sup> century, is presented and discussed.

This work was financed by a program of the working community of Pyrenees (Communauté de Travail des Pyrenees - CTP) "Natural resources in the architectonic heritage in zones of mountain. Exploitation, use, analysis and development."

DANIEL, F., LABORDE, B., MOUNIER, A. AND COULON, E., 2008. Le pigment d'aérinite dans deux peintures murales romanes du sud-ouest de la France. *Archéosciences – Revue d'archéométrie***32**, 83-91.

PASTOUREAU, M., 2000. *Bleu. Histoire d'une couleur*. Le Seuil, Paris.

## **C5 Sources of Natural Organic Colorants from the Andes - A Multi-Disciplinary Study at the British Museum**

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The British Museum houses one of the largest collections of Andean textiles outside Peru comprising pieces ranging from the Early Horizon through the Colonial Period (Dransart & Wolfe, 2011). A detailed study of the natural organic colorants and dyeing technologies is currently being undertaken to complement the “traditional” study of iconography and weaving techniques. Investigation of the organic colorants at molecular level is vital to fully characterise the diverse range of biological sources and manufacturing technologies employed. The focus is therefore on the use of high performance liquid chromatography (HPLC) in this research. An analytical approach based on “soft” extraction techniques has been developed in order to preserve glycosidic components indicative of the biological source and the most sensitive classes of colorants such as flavonoids and carotenoids that are usually lost with traditional extractions by strong acids. The method has also been optimised to ensure extraction of all colorants from a single sample and for the small sample sizes typically available from museum objects. This optimised analytical protocol has been tested on a large range of samples of fibres dyed with plant-derived colorants recently collected in South America, providing a comparative dataset for the study of the archaeological textiles. These reference samples have also been produced to investigate the stability, sensitivity and degradation paths for these fugitive colorants in order to more fully understand the results obtained from the archaeological material. This paper will present the results of a literature review of Andean natural organic colorant sources and the creation of the reference collection of colorants representative of the Highland and coastal dyeing traditions in Peru. The collection of the plants, the dyeing of the fibres and the chemical characterisation of the resulting samples will be described. The results obtained for a number of archaeological samples will also be presented and compared to the modern materials. While addressing key questions about Andean textile production, this paper will demonstrate the wider potential of colorant analysis to explore art historical, archaeological, anthropological and cultural questions. This research will additionally provide new analytical methodologies, better understanding of deterioration processes and improve conservation and display strategies.

DRANSART P. AND WOLFE H., 2011. *Textiles from the Andes (Fabric Folios)*. British Museum press, London.

## C6 Cinnabar in Archaeological Funerary Contexts in the South of Portugal

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The importance of the funerary rituals during the Neolithic period is evident from the numerous megalithic monuments disperse throughout the Western Europe, suggesting that large resources were spent in the veneration of their deceased. Red pigments are often found in archaeological contexts, where they are used to cover the bodies, but their ritual significance is not consensual among the archaeologists.

The red pigments recovered from the dolmens have been mainly identified as red ochers, based solely on visual inspection. Chemical analysis performed on pigments recovered in sites from the south of Spain, surprisingly, identified cinnabar alone or together with ocher (Fernández, 1999; Martin-Gil, 1999; Ortiz et al. 2009; Lazarich et al. 2009; Borja et al. 2006; Hunt-Ortiz, 2011).

Cinnabar, H<sub>2</sub>S, is a rare occurring mineral, available in a very few places worldwide and, within the Iberian Peninsula, it occurs only in Las Alpujarras (Granada), Sierra de los Filabres (Almeria), Usagre (Badajoz) and Almadén (Ciudad Real).

The south of Portugal is one of the richest places in megalithic monuments of the world, and reports often indicate the presence of ochre red pigments in the numerous dolmens excavated since the XIX century (Oliveira, 1997).

In this work we describe analysis performed on red pigments, previously identified as ochers, and recovered from the Zambujeiro Dolmen (Évora), Dolmens of Bola of Cera and Cabeçuda (Marvão) and Horta Dolmen (Alter do Chão). The samples comprised red powder material mixed with soil and red powder dust recovered from stone plaques (Lillios, 2008). The samples were analyzed by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), micro X-ray diffraction (XRD) and Raman micro-spectroscopy.

Most of the samples analyzed proved to be cinnabar but, in the Zambujeiro Dolmen, both ochre and cinnabar were detected in the surface of two stone plaques, indicating the use of both materials in the same funerary context. This is the first time that cinnabar is found in these megalithic region, suggesting the likelihood of commerce routes due to its distance to the cinnabar mining sites.

The number of cinnabar samples identified suggests that some red powders, recovered from dolmens in the south of Portugal, were previously misidentified as ochers. The identification of archaeological red pigments cannot be done based solely in visual inspection and unequivocal identification can only be achieved by chemical analyses.

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## **C7 Characterization of Archaeological Soils and Sediments Using VIS-Spectroscopy**

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Prehistoric pits are filled with ancient topsoil material, which has been preserved there over millennia. The colours of pit fillings in the loess area of NW-Germany are specific for the time the soil material was relocated. Early Neolithic pit fillings, e.g., have a black to dark brown colour, while Iron Age pit fillings have a lighter grey colour.

Soil colours reflect soil characteristics, and the correlations between soil colour and soil components like charred organic matter or iron hydroxides can be measured using spectrophotometrical data. The development of a quantitative spectroscopic method based on soil colour spectra would enable the measurement of large amounts of samples, e.g. to investigate environmental or archaeological research questions on a regional scale.

The aim of our study is the assessment of soil profile information using VIS-spectroscopy, with a main focus on archaeological soil features, and to establish spectrophotometry as a rapid and reliable tool for the analysis of soils and sediments. Furthermore, we want to evaluate the colour differences of pit fillings that are typical for a prehistoric period.

Soil colour spectra were obtained in the 360 to 740nm range, in 10nm steps, using a field-spectrophotometer (CM-700d) and a lab spectrophotometer (CM-5). The spectrophotometric data was calibrated against soil sample sets that have been analyzed on iron, carbonates, organic carbon, and pyrogenic carbon (BPCA). The set of archaeological samples that covers material from the Early Neolithic to the Roman period was collected at excavations in the loess-covered regions of NW- and E-Germany. Models were build based on partial least squares regression. Additionally, we evaluated different colour index values (e.g. Redness Rating), as well as the effects of soil moisture and texture.



## C8 Colour Specification of Terracotta *Calatina* Specimens

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The site of Caltagirone is one of the major production center of pottery in Sicily and southern Italy, and its activity is attested since Neolithic period and is still a vivid tradition.

From the specific requirements of master craftsman to individuate the relation between the cooking process and the color of their artifacts, a project based on the study of this two parameters correlations is developed.

In order to create ceramic specimens with a composition typical of the area of interest, clay were sampled in the quarry of Monte San Giorgio and in the area of Lazzaretto (*Conadomini* furnace). The sand used as inert was sampled from the Monte Stagno quarry; all the sites were chooses following the indications of the major ceramists of Caltagirone.

In this work we present the results from the color specification of two types of mixture that characterize the *Calatina* production used by ceramicists to obtain, respectively, ceramics commonly used and most valuable artifacts.

The specimens were subjected to cooking varying the maximum temperature from 400 °C to 1200°C and then to color measurements with spectrophotometric method recording the trend of the spectral reflectance factor and the values of CIELAB colorimetric space.

The color differences were calculated in reference to the sample before cooking, settled "raw".

The results obtained represent a set of objective data on color changes of ceramic specimens due to the different cooking conditions and they gave precise information for the continuation of research.

The study also provides to cross the colorimetric results with those obtained by X-ray diffraction techniques. This is useful to identify, for each thermal processes, the mineralogical phases that may be responsible of the color differences measured.

## C9 Colours of Nuzi

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The colour palette of a range of materials from Nuzi, including glass, glazes, and pigments, have been studied in detail to determine the key factors that influenced the colour choice for different objects types. The factors considered include technical constraints such as availability of resources and available technology, as well as aesthetic choices and the role of theological and secular symbolism. Certain colours and properties of materials such as shine and translucence were important in Mesopotamian religion and were directly associated with particular deities.

The site of Nuzi, near Kirkuk in modern Iraq, was excavated between 1925 and 1931 and approximately half of the finds went to museums within the USA under the contemporary excavation legislation, predominantly to the Semitic Museum at Harvard University. The majority of finds came from a destruction layer due to the city being sacked and destroyed around 1340 BCE and many artefacts were found in situ. A wide range of materials with vivid and diverse colours were recovered, including polychrome glass, glazed and painted ceramics, wall paintings and some raw pigments. In many cases the original colours of the objects are now hard to determine, either due to alteration during burial or occasionally as a result of post-excavation deterioration. Scientific analysis revealed the original colours employed on these artefacts and enabled the original colour schemes to be recreated. This study of the colour palette used for each class of object revealed the colour choices on a material by material basis. The provenance of the various glass and ceramics were refined to ascertain how this relates to the colours employed, highlighting differences in the palette of Late Bronze Age and Parthian period artefacts from later occupation on the site. The pigments found were compared to those used in wall paintings. At this site, a small regional centre of the Late Bronze Age Near East, it is suggested that the choice of colour appears to be related equally to the colours and their symbolism as well as to the availability of materials over a wide range of objects.

## **C10 Colour Pigments and the Colour Concept of Iberian Iron Age Stone Sculpture**

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In the second half of the 1<sup>st</sup> millennium BC, the Iberians created a great corpus of stone sculpture which were connected with sepulchral and cultic contexts. Like most of the pre-modern statuary, also the Iberian images were originally painted. Today, only on a few sculptures traces of former painting are preserved. In this respect the few existing archaeometric studies of pigment traces are the load-bearing pillar of any archaeological research about the original cultural and historical significance of the colours. Unfortunately, it must be noted that especially archaeometrical studies deprived from any historical or archaeological formulated question produce considerable research problems. Nevertheless a first approach to the Iberian concept of colour can be formulated and it becomes clear that polychromy was an important element of the original context of the sculptures. To a certain extent, the painting of the sculptures reflected the ancient Iberians' intellectual engagement with the world. The colour concept used in Iberian sculpture had a strong symbolic function closely connected with notions of social prestige in real life and afterlife. Firstly the lecture will present a critical overview over the archaeometrical work done so far and secondly on this basis an attempt to decipher the cultural concept of colour in Iberian stone sculpture will be made.

MIELKE, D.P., 2011. Die Polychromie iberischer Skulpturen. *Madridrer Mitteilungen* **52**, 306-332.

## **C11 Colour Perception in Historic Vitreous Artefacts Containing Transition Metal Ions: Optical Spectroscopy and Ligand Field Theory Reviewed in Connection with Chemical Analysis**

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The dissolution of transition metal ions in glasses often results in colored complexes of varying hues and intensity. The color depends foremost on the kind of ion, its valence and coordination as well as on the bonding characteristics, which in turn depend on compositional variations of the glass matrix. The color of transition metal doped glasses arises from transitions between the valence electrons, or d-d electrons of the dopants. The energy of these transitions can be obtained from the optical absorption spectra and application of ligand field theory can in turn be used to establish the site environment. Of much higher intensities are charge transfer (CT) transitions, which are often hidden in the absorption edge in the UV-VIS region. However some ions show strong CT transitions reaching into the visible and thus color a glass yellow, or at higher concentrations, brown. All d-d transitions are parity forbidden by the Laporte rule and the molar extinction coefficients  $\epsilon$  of d-d transitions are therefore several magnitudes lower in their intensities than Laporte allowed CT transitions. The Laporte rule can be relaxed by mixing of metal 3d with metal 4p or ligand p orbitals for non-centrosymmetric sites, such as tetrahedral coordination where  $\epsilon$  can be 100 times higher compared to a centrosymmetric, e.i. octahedral coordination environments. Other transition metal ions show even weaker absorption, as some d-d transitions are not only parity forbidden, but also spin forbidden by the equal multiplicity selection rule. So is the molar extinction coefficient  $\epsilon$  of the doubly forbidden  $\text{Mn}^{2+}$  transitions around  $0.04\text{--}0.4 \text{ Lmol}^{-1} \text{ cm}^{-1}$  but of the spin allowed  $\text{Mn}^{3+}$  transitions around  $20 \text{ to } 180 \text{ Lmol}^{-1} \text{ cm}^{-1}$ . This explains also why 0.1 wt% of CoO in tetrahedral coordination may cause a deep blue, but 0.1% CuO only a light sky-blue color.

Most archaeometric studies rely solely on the chemical composition when deciding on a certain colorant. However some ions color glasses already in the ppm range while others need to be added in several %. The aim of this paper is to show a correlation between the perceived color and its definition in the CIE 1931 color space chromaticity diagram, the analyzed concentration of coloring agents, using SEM or XRF, and the optical transitions from UV-Vis spectroscopy and their interpretation by ligand field theory. The samples studied include historic artifacts and soda-lime-silicate model glasses.

## C12 Orpiment, the “Yellow King” of the Colours, and its Diverse Properties

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Orpiment, the yellow sulphide of arsenic ( $\text{As}_2\text{S}_3$ ), has been used as pigment in painted decorations and manuscripts since ancient times. Early known uses of orpiment as pigment arise in ancient Egypt. In terms of coloured inks, yellow was prepared using orpiment or pigments of various botanicals. In addition, arsenic has always been used as a therapeutic agent and poison. Yellow arsenic was also one of the most common remedies in Arabic pharmacology and medicine. Medieval Arabic alchemical texts describe orpiment and realgar as the 'two Kings'.

The original contents recovered from a small glazed ceramic pot, found during archaeological excavations from the ancient wall of Zaragoza (Spain), were investigated in order to reconstruct its function. The archaeological context suggests it belonged to the Islamic period, dating to the 11<sup>th</sup> century AD or Taifa period. An analytical study of the contents of the ceramic pot revealed an ancient “chemical” preparation. The use of several analytical techniques (Optical microscopy, Scanning Electron Microscopy, X-ray Energy Dispersive Spectrometry, X-ray Diffraction and Pyrolysis-Gas Chromatography/Mass Spectrometry) enabled us to identify mineral, organic and vegetal components: orpiment, a fig, and some grape seeds, together with a small amount of gypsum.

The purpose of this mixture may have been for colouring: a pigment with a possible yellow, orange or “golden” tint. However, due to the lack of knowledge available of medieval compound drugs, the components we identified may have been part of a medical treatment.

### **C13 The Polychrome Synopia of Roman Mosaic at Lod (Israel): Pigments Characterization and Microstratigraphic Study**

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The Roman mosaic from Lod (formerly Lydda, Israel) is one of the most beautiful and famous pavements of the Roman Empire. It was fortuitously discovered in 1996 and excavated by the Israel Antiquities Authority. The mosaic, representing both real and mythological animals, is constituted by 3 very well preserved floors for a total of 150 m<sup>2</sup>. It is dated to about the AD 300 and probably belongs to a large and important Roman villa. After the detachment performed in the 2009, a extraordinary polychrome synopia was discovered under one of the most evocative scene of the mosaic, the nautical representation of fishes and vessels. It worth noting that this finding is absolutely unique in the history of art, being the adoption of a synopia during the production of a mosaic, typical of the Greek word and not of the Roman one. Besides, the Greek artisans used to draw the sketch on which apply the tesserae with only one pigment (red ochre, carbon black), and polychrome synopiae have never been attested before.

The main aims of this investigation were defining of the palette adopted to produce the synopia under the fish panel. Moreover, the microstratigraphy of the paint was studied to define if fresco technique was used to paint the sketch, as macroscopically suggested by the presence of joints in the preparation layer (*sovrannucleus*) indicating daily applications of mortar (*giornate*). The study was performed on 8 fragments of *sovrannucleus* bearing four colours (red, yellow, green, black). The adopted analytical methods are PLM (polarised light microscopy on pigment dispersions), RLM (reflected light microscopy) and SEM-EDS (on transversal polished section), and XRPD (on pigment powders).

The combination of these analyses distinguished the following traditional pigments: red and yellow ochre, cinnabar, green earth, carbon black. In particular, the recognition of micro-foraminifera in the backscattered images (BEI) indicated that green earth from seashore deposits, formed by glauconite, was used. As for the black colour, carbon particles preserving traces of the cellular structure indicated that this colour was obtained combusting wood or straw.

RLM and SEM analysis showed that the microstratigraphy in all the samples is composed by two layers, one constituting the *sovrannucleus* (white mortar) and one the painting layer. Moreover, BEI images highlighted, on the top of the samples, the presence of a carbonation layer including the pigment particles and indicating that the pigments were laid on a fresh mortar.

#### **C14 Application of Near-IR Hylogger™ Technology for Colour and Mineral Analysis of Aboriginal Australian Mineral Pigments**

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Natural mineral pigments on Aboriginal Australian artefacts present a challenging case for mineralogical analysis. Mineral pigments include iron oxides such as hematite and goethite (red, yellow and brown pigments), and clays such as kaolinite (white pigments) and others. In many cases, the pigment colour has a specific cultural meaning when applied to the object, and individuals sought materials from particular quarries. Many of these pigments are naturally composed of other mineral phases, which also complicates pigment characterisation. These mineral pigments are primarily applied to wood, fiber, bark, resin or other organic substrates, making non-destructive in-situ scientific analyses challenging.

Research studies focused on the composition and technology of pigments require methods to identify and differentiate types of mineral pigments. High-resolution, automatic infrared and visible light spectroscopic analyses and imaging of minerals and geological materials for the mining industry have been routinely accomplished by the use of the CSIRO HyLogger™ in Australia. This presentation presents the novel application of the HyLogger™ technology to Australian Aboriginal artefacts for characterization and high-resolution spectroscopy of applied pigments. The Hylogger™ technology allows high-resolution, high-throughput non-destructive characterisation of pigments in less than 1x1 cm spot size (spectral pixel) on Indigenous Australian objects, along with visual imaging of the spot analysis. A case study of 12 artefacts was analysed and evaluated using the Hylogger™ instrument and The Spectral Geologist (TSG™) software analysis package. The spectral range of the instrument allows identification of multiple minerals in the pigment. In addition, the software quantifies colour for each spectral pixel in Munsell and L\*a\*b colour. While many identified pigments such as hematite and goethite were expected for the red and orange and yellow pigments, other white minerals such as pyrophyllite were identified along with kaolinite. These results challenge and add to prior knowledge about the mineralogical composition of Aboriginal Australian mineral pigments. The use of the Hylogger™ technique leads to a more complete understanding of cultural uses and technology of natural mineral pigments in Australia and worldwide.

## C15 Colouring Materials in Western Mediterranean Middle Neolithic Sites: From Procurement Strategies, to Preparations and Uses

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Colouring materials are a privileged medium to approach prehistoric symbolic systems, via the study of technical systems. However, the knowledge on the *chaîne opératoire* of these highly-varied geo-materials remains limited, particularly for the end of prehistorical times. In the franco-italian area, within “liguro-provençal” bow, as well as Baux-de-Provence and Roussillon, many archaeological sites provided colouring materials; their place remains still undetermined in the complex middle Neolithic economy and trade networks.

In order to determine the raw materials exploited, their provenance and the know-how related to their transformation and uses, a set of Neolithic pigments was investigated following a multi-scalar approach, from field surveys to microscale using a combination of complementary imaging, elementary and structural techniques (petrography, SEM-EDS, Raman spectroscopy). Among the sites of the concerned area, we start focusing on the station of Giribaldi (Nice, France) excavated by one of us (Binder, 2004). Belonging to formative stages of Southern French Chassey culture (4700-4050 cal. BC) (Binder et al., 2008), it provided an exceptional corpus of colouring materials. First operations of observation and classification pointed a corpus including a lot of the sequences of the colouring materials exploitation: raw pieces, semi-modified fragments, coloured tools with workshop and utilization traces, red-coloured ceramics, etc. It gives evidence for the presence of craft activities related to transformation and use of colouring substances. Although bauxite is often cited in archaeological literature as the main pigment used in the studied region, the geological and analytical data obtained allowed the determination of two main colouring geo-materials: red and orange bauxites (aluminium oxides with hematite) and red and yellow sandstones containing iron oxides. Information from geological maps combined with field surveys tend to show a provenance higher than 80 km for bauxites and 30 km for sandstones. These results reveal the importance of colouring materials in a complex trade network, which has now to be compared with the data already known for other productions. Concerning the degree of pigments transformation, we could evidence different spraying modes that depend on the nature and the physical properties of the materials (coherence): sandstones were ground, whereas bauxites were also abraded. Heat treatment may have been used to dehydrate goethite  $\alpha\text{-FeO(OH)}$  in hematite  $\alpha\text{-Fe}_2\text{O}_3$ , or diasporite  $\alpha\text{-AlO(OH)}$  in corundum  $\alpha\text{-Al}_2\text{O}_3$  (still under study). Last, the modalities of use seem to cover a



broad spectrum of activities, from technical uses as colouring and/or abrasive agent, to symbolic functions (engraved decors in ceramics).

BINDER, D. (Ed)., 2004. *Un chantier archéologique à la loupe. Giribaldi*. Nice Musée, Nice.

BINDER, D., LEPÈRE, C. AND MAGGI, R., 2008. Épipaléolithique et Néolithique dans l'arc liguro-provençal: bilan et perspectives de recherche. *Bulletin du Musée d'anthropologie préhistorique de Monaco* **Suppl.1**, 49-62.

## C16 Tainted Ores: Colour Preferences at the Dawn of Metallurgy, c. 5000 BC

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Scholarly discussions on the emergence of metallurgy are dominated by the pursuit for the earliest evidence of copper extraction in western Eurasia, which largely shaped the debate on single vs. multiple origins of this once precious and unique skill (most recently Roberts et al., 2009; contra Radivojević et al., 2010). The recent discovery of the world-wide earliest copper smelting in Belovode, a Vinča culture site in eastern Serbia, dated at c. 5000 BC has changed our understanding of *when* and *where* metallurgy possibly first occurred. However, the origins of metallurgy have usually been studied in isolation and detached from their technological, social and environmental context, and there has been little research specifically addressing *how* and *why* metallurgy emerged and evolved.

Here we present results of an interdisciplinary approach using assemblages of high archaeological and archaeometallurgical resolution in order to address the initial stage of the pyrometallurgy in three Vinča culture sites in Serbia and Bosnia. We identified and sampled well-contextualised archaeometallurgical materials coming from the sites of Belovode, Vinča and Gornja Tuzla, all dated to the first half of the 5<sup>th</sup> millennium BC. Microstructural, chemical and isotope analyses of slags, slagged sherds and copper ores gave unprecedented insight into which ore minerals were chosen for metal making. Our results indicated that early copper smiths were indiscriminately sourcing different kinds of weathered minerals, mostly carbonates, sulphates, arsenates and phosphates, apparently basing their decision primarily on distinctive colours in the shades of green and black.

The smelting of these distinctively coloured minerals for the first time demonstrates the long-postulated experimental approach to the diversity of choices available and applied at this very early stage in metal extraction. Significantly, this included smelting unsuitable minerals as well as relatively complex ores, shedding new light on our understanding of the role of experimentation in the emergence and development of metallurgy.

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ROBERTS, B.W., THORNTON, C.P. AND PIGOTT, V.C. 2009. Development of metallurgy in Eurasia. *Antiquity* **83**, 1012-1022.

## **C17 The Short Life of Tannins: Chemical Investigations on Ageing Processes in Tannin Dyed Textiles**

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The large number of historic textiles still preserved today represents an important field of our cultural heritage. The state of conservation of these examples of our historic handicraft may differ in dependence of numerous factors. Different kind of degradation effects can be observed, like fading or loss of material, occurring in the fibres unevenly. Particularly, it is possible to notice that tannin dyed textiles usually show more extended degradation phenomena than textiles dyed with other colouring materials, even in the same condition of preservation. In order to face the problems related to the occurrence of tannins in dyed textiles, the project The Short Life of Tannins has been founded by the Regione Toscana (FAS grants 2008-2013). The project aims at creating a model describing the ageing processes taking place in textile fibres dyed with tannins. The final goal of the research is to appoint a conservation protocol to arrest or to slow degradation processes, on the basis of the developed predictive model.

In the framework of VAT project, a research line is dedicated to the morphological and molecular characterization of tannin-based dyes and proteic fibres such as wool. In particular, the main results obtained in the characterisation of raw colorant materials and textile reference specimens (even submitted to artificially aging protocols) by optical and scanning electron microscopy will be presented. Moreover, such data will be discussed and compared with the ones obtained by analytical procedure base on chromatography and mass spectrometry. In fact, techniques such as HPLC/MS and GC/MS have been used, not only for the characterisation of lipids, proteins and tannic dyes, but also for the identification and quantification of degradation products.

**C18 Specialized « ochre » procurement strategies in the Transition context : the red pigments from the Châtelperronian of the Grotte du Renne, Arcy-sur-Cure (France).**

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In many reports of prehistoric pigment studies, these artefacts are considered as the testimony of past symbolic activities. The first step of the proceeding sequence, that is to say the acquisition of raw colouring material, is not well described and understood. Physico-chemical (SEM-EDS, XRD, TEM-EDX,  $\mu$ PIXE- $\mu$ PIGE) and petrological analysis were carried out on the colouring materials excavated in the châtelperronian layers (40000-35000 B.P.) of the French site the Grotte du Renne in Arcy-sur-Cure. The Châtelperronian is one of the transitional techno-complexes, basically one of the last cultures made by Neanderthals in Europe. The physico-chemical data were related to the location of the colouring materials on the site, in association with exceptionally well preserved “hut” structures. It was thus possible to demonstrate that none of these materials, either red or black, was heated before being used, contrary to what had been assumed so far. The supply in colouring materials was as carefully organized as for mineral materials such as flint, for example; they were collected in different geological formations occasionally showing on the surface, close to the cave and at more than 30 km from the cave. The exploitation of these geological sites did not vary during the whole Châtelperronian period, and privileged materials which can easily be ground to powder. The set of colouring minerals from the Grotte du Renne reveals Neanderthals' in-depth knowledge of mineral materials; they understood perfectly well their properties and qualities, and used them extensively, so that the raw colouring material was part of the livelihood and the Châtelperronian site must have been a literally dazzling sight, all red and black.

## **C19 The Science and Symbolism of Color: Mapping Pigment Use in Archaeological Contexts**

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This research proposes an innovative theoretical framework within which to conduct further studies of color and pigment use. Drawing upon an interdisciplinary, anthropological model, pigment use from the Paleolithic through to the 2<sup>nd</sup> century B. C. is tracked. The results of the literature survey presented here are the mapping of pigments as they occur in Africa, the Near East, Anatolia and Europe. With a view to meta-analysis which surpasses micromorphology of particles, this thesis expands into a vantage of temporal and spatial trends.

Questions of central importance here involve the variables of the selection of colors and pigments as they are used in naturally occurring or human made architecture. Investigations of artworks inherently contain questions beyond the functional aspects of materials. To move forward from technological questions asked of the material such as, “what is it?”, or “was it local or imported from elsewhere?”. The goal is to build upon discrete analysis of specific sites to reach a more full understanding of the evolution of the use of colour and investigate what the selection of materials teach us about intangible variables such as cognition, spirituality and the process of individuation.

## **C20 Raman Spectroscopic Investigation of Prehistoric Pigments: First Results from Hungary**

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In the frame of a recent infrastructural development at the Faculty of Science, Eötvös L. University, Budapest, a Raman spectrometer has been installed. Various applications in the Cultural Heritage field are foreseen. As a pilot project, investigation of prehistoric pigments was started, partly on recently excavated finds (sherds and pigment lumps from Kup-Egyes neolithic site) and partly on classical museum pieces from Aszód, Bodrogkeresztúr and Jászládány localities (containers for prepared mineral pigments). We are planning to include more prehistoric pigments and painted objects. Archeological objects were examined using microscope objectives (powder samples and pigment lumps), microscope objectives with a 90° mirror mount (ceramic pots) and a fibre optic coupled probe head (pigments inside containers).

Pigments identified in the Kup-Egyes finds were hematite, hematite + anatase (supposedly originating from karst bauxite outcrops of the Transdanubian Range), calcite, calcite + apatite (calcinated bone) and graphite. The material in the pigment containers (small ceramic pots) was identified as hematite. It was found that surface treatment by Paraloid B-72 of the painted vessels essentially hinder the Raman spectroscopic identification of the pigments due to a very strongly elevated luminescence background.

By the continuation of the Raman studies of prehistoric pigments we wish to establish the range of mineral and organic substances used for the decoration of Prehistoric vessels.

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## C21 Analysis of Window Glass from the Basilica of Tongeren

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During last ten years, important excavations have been carried out under the basilica of Tongeren. Started in 2011 to shed a new light on the centre of the earliest city of Belgium, a project funded by the Flemish government has been devoted to the study of the archaeological materials and remains excavated there. The first phase of research leads to assess that, roman buildings, namely a domus and a late roman basilica were initially standing at the location of the actual church. These constructions were then successively replaced, during Merovingian and Carolingian periods, by three consecutive religious buildings and then, later on, by different churches, a Romanesque and then two Gothics ones. Therefore, the entire historical period and its continuity from Antiquity to the end of Middle Ages, is preserved and can be monitored on this site. Among excavated artefacts, only 890 glass fragments were discovered comprising namely window glass. About 50 samples of these were selected and analysed by PIXE-PIGE elemental techniques at the CEA-IPNAS laboratory of Liège University. The chemical compositions obtained have allowed both discrimination of different compositional groups and dating of some of the fragments. Additionally, it has also provided indications on glass colorations recipes. A first set of glass fragments can be dated from the Middle Ages with compositions typical of woodash glass (Wedephol et al., 2011). Despite of their poor state of conservation some samples can be dated from the 11<sup>th</sup> to the 13<sup>th</sup> century and others are obviously more recent. Besides of potassium-glass, sodium-glass with typical composition of glass made with natron, was also evidenced. Among these samples, some can be compared to the blue-green glass found everywhere in the Roman Empire between the 1<sup>st</sup> and the 3<sup>rd</sup> century (Foster & Jackson, 2009). Latest samples have the same composition as the HIMT and Levantine I glass dated from the end of the Antiquity (Freestone et al., 2000, 2002; Freestone, 2003). Finally, some turquoise-blue and green fragments can be linked to the early medieval churches. Their composition seem typical from this period (Freestone et al., 2008) and if part of the coloration processes and techniques used are already known (Mitri et al., 2000) few others are more surprising for this period.

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## **C22 Characterizing Organic Colorants in a 15<sup>th</sup> Century Iranian Timurid Qur'an by Direct Analysis in Real Time-Time of Flight Mass Spectrometry**

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The Islamic Department at the Detroit Institute of Arts has in its collection a unique 15<sup>th</sup> century Timurid Qur'an. The calligraphy was applied to brilliantly-colored painted and polished paper. The Qur'an is currently undergoing a multidisciplinary study to identify the inks, pigments, binders, dyes, gold alloys, and fibers used to construct the manuscript. X-ray fluorescence has shown that the colored pages contain primarily lead, suggesting that most of the colors are likely organically derived rather than due to the presence of mineral pigments. Raman microscopy indicated the presence of indigo in the blue pages, but fluorescence from organics, thought to be binder, obscured the spectra for the other colors. FTIR and visible microscopy show that the binder is most likely proteinaceous, and that the paper is a bast fiber.

We report here preliminary results from utilizing direct analysis in real time mass spectrometry (DART-MS) to identify organic dyes in a matrix that mimics that of the DIA Qur'an. While DART-MS has been widely used for identification of small (<1000 Da) molecules on surfaces in forensic and pharmaceutical sciences, its use for identification of organic colorants in cultural heritage materials has only recently been demonstrated (Selvius DeRoo & Armitage, 2011). Samples approximating the construction of the original painted paper have been prepared based on the historical context of both the manuscript and its possible Chinese artistic influences. Relevant organic colorants were mixed into a lead white base with various organic binders and applied to paper. These simulations are being used to study temperature and sample handling for identification of organic colorants in this complex matrix. To date, both indigo and madder have been readily identified based on the exact masses of the primary colorant compounds (e.g., indigotin and alizarin/purpurin). Future studies will explore the nature of yellow colorants as well as mixtures of colors including purple and green. These preliminary studies will provide a basis for future studies of precious samples removed from the original manuscript.

SELVIUS DEROO, C. AND ARMITAGE, R.A., 2011. Direct Identification of Dyes in Textiles by Direct Analysis in Real Time-Time of Flight Mass Spectrometry. *Analytical Chemistry* **83**, 6924-6928.



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