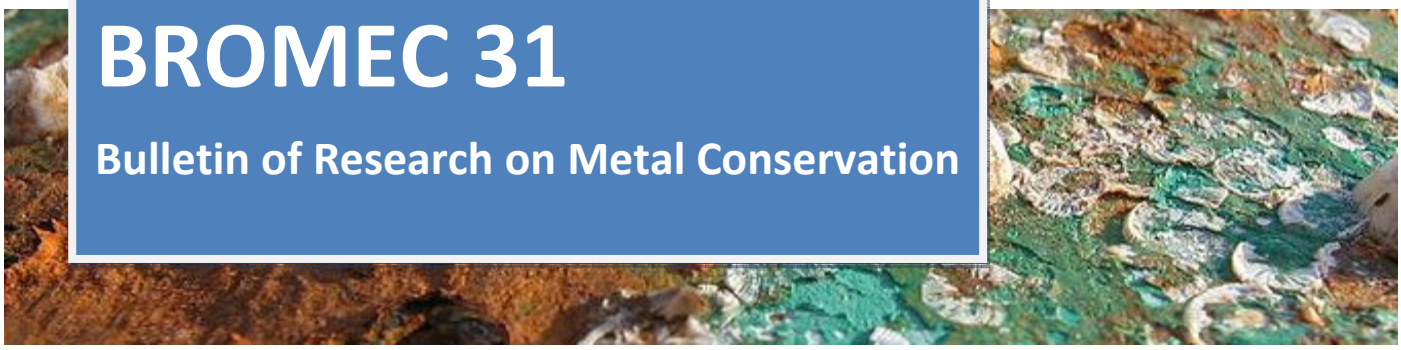
BROMEC 31

Bulletin of Research on Metal Conservation



July 2010

Editorial

This BROMEC provides you with news on various electrochemistry-based research projects for analysis and conservation of metals and their corrosion products. We present a *new research project* involving the development of a portable environmental and electrochemical cell for long-term *in situ* spectro-electrochemical experiments. Two *ongoing research projects* exemplify the increasing heritage application of voltammetry of microparticles for identifying metal corrosion products: firstly copper and its alloys, and secondly, tin and tin alloy coatings on archaeological iron. Another *ongoing research project* describes the use of a cavity microelectrode for various electrochemical techniques when coupled with complementary spectroscopic methods. Lastly, an investigation into the protective efficiency of coatings comprising conducting polymers and self-assembled monolayers is made. These authors presented related investigations at the Lorentz workshop (the Netherlands); the website address for downloading presentations is given in the announcements. Other new announcements include a Masters in Heritage Science at University College London, a book about sculpture conservation and research at the Getty, an online journal for heritage conservation science and restoration (published in English, Spanish and Portuguese) and two meetings: Tecnarte 2010 and the 3rd International Conference in Archaeometallurgy in Europe 2011.

A reminder: Metal2010, the Interim Meeting of the ICOM-CC Metal Working Group, is fast-approaching (Oct 11-15), with the discounted registration closing July 31.

In BROMEC communication news we welcome conservators, Inmaculada Traver and Marc Voisot, as Hispanophone and Francophone translators. They complete the third side of our linguistic triangle, facilitating direct and more accurate French-Spanish and Spanish-French translations: a significant contribution to the Editorial Team, for which we are very appreciative. Also, readers might well have noticed a new look BROMEC! Emilio Cano leads the revised format of BROMEC to improve its legibility and readability, with the help and advice from Manel Alagarda, from the IVC+R (Valencia, Spain). Many thanks to Emilio for this initiative!

In dissemination news, the new, more efficient method of delivering BROMEC publication web links and news directly to your email inbox via the BROMEC ListServ is now in place. Most readers will have received BROMEC 31 via an email from the BROMEC ListServ. If you would like to subscribe, unsubscribe, or update your preferred email address and settings, you can do so at the address given in *websites*. Welcome to archaeological conservator, Pia Klaavu, Finland's new National Contact person. And thanks to Eero Ehanti, for previously undertaking this role and suggesting his replacement. Of note, email addresses of National Contacts are now also listed at the end of BROMEC so the readership can contact them more readily. We trust these initiatives will facilitate collaborations and we look forward to receiving your feedback in order to further improve international and intranational metal conservation research networking.

Enjoyable reading!

James Crawford

Anglophone Editor & Translator:

James Crawford
jamesbcrawford76@ gmail.com

Francophone Coeditor:

Michel Bouchard
mbouchard@caraa.fr

Francophone Translators:

Nathalie Richard
n.richard.elmesti@videotron.ca

Elodie Guilminot
elodie.guilminot @arcantique.org

Hispanophone Coeditor:

Emilio Cano
ecano@cenim.csic.es

Hispanophone Translators:

Diana Lafuente
diana.lafuente @gmail.com

Inmaculada Traver
lacirujanadelarte @gmail.com

Marc Voisot
horloger@pendulerie.com

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General information

Cover image: “Corrosion, atmospheric and biologic; Biologic made by Barnacle” [*sic*], Rafal Konkolewski, http://upload.wikimedia.org/wikipedia/commons/9/97/Corrosion.jpg

Development of a portable environmental and electrochemical cell for heritage metals conservation research *(UW, GU & EH)* [1]

New research project

Contact: James Crawford (UW) (j.b.crawford@warwick.ac.uk), Mark Dowsett (UW), Annemie Adriaens (GU), David Thickett (EH)

Funding: Warwick Postgraduate Research Scholarship and English Heritage

Conservation of society's tangible heritage is a battle against time waged out on minuscule levels: the processes of material degradation, and the means of extended conservation, are fundamentally understandable on microscopic levels and necessarily span (long) periods of time. This Ph.D. project is interdisciplinary between the fields of chemistry, physics and cultural heritage conservation and will contribute to the:

- Development of a portable environmental and electrochemical cell (peCell) for material-environment studies
- Advancement of conservation strategies for metal artefacts from/in marine/maritime environments

Over the past five years the Electrochemistry and Surface Analysis (ESA) and Analytical Science Projects (ASP) groups at Ghent and Warwick universities have developed equipment and methods for time-resolved *in situ* monitoring of (electro)chemical reactions using synchrotron X-rays and other surface analysis techniques. A core invention is the electrochemical/environmental cell, eCell, which can be used for electrochemistry (both analysis and treatment) while simultaneously analysing the metal surface through a thin electrolyte layer using a spectroscopic technique (*spectroelectrochemistry*) [2-5].

To relate measurements made using infrastructural facilities (e.g. synchrotrons) more closely to real long-term behaviour (lessening the need for acceleration via extreme and unrealistic parameters), we intend to design and build derivatives of the eCell so that corrosion processes can be studied on a long term basis (months or years), whilst maintaining an uninterrupted controlled environment around the sample. The objective is to use a single cell design for both gaseous and liquid environments.

1. Original language version; submitted by author in English.

2. Dowsett M., A. Adriaens, Cell for Simultaneous Synchrotron Radiation X-ray & Electrochemical Corrosion Measurements on Cultural Heritage Metals & Other Materials, Analytical Chemistry, 78(10) (2006) 3360-3365.

3. Adriaens A., M. Dowsett, K. Leyssens, B. Van Gasse, Insights into electrolytic stabilization with weak polarization as treatment for archaeological copper objects, Analytical Bioanalytical Chemistry 387(3) (2007) 861-868.

4. Adriaens A., M. Dowsett, Time-resolved spectroelectrochemistry studies for the protection of heritage metals, Surface Engineering 24(2) (2008) 84-89.

5. Adriaens A., M. Dowsett, G.K.C. Jones, K. Leyssens, S. Nikitenko, An in-situ X-ray absorption spectroelectrochemistry study of the response of artificial chloride corrosion layers on copper to remedial treatment, J. Anal. At. Spectrom. 24(1) (2009) 62-68.

Layer-by-layer identification of copper alteration products in works of art and archaeological pieces using voltammetry of microparticles *(UV, UPV, IVC+R)* [1]

New research project

Contact: Antonio Doménech (UV) (antonio.domenech@uv.es), María Teresa Doménech-Carbó (UPV), Isabel Martínez-Lázaro (IVC+R)

Funding: No external funding

A method for identifying alteration products in copper and copper alloys distributed in different corrosion product stratigraphies, using the voltammetry of microparticles approach, has been developed. Measurements were made using a graphite pencil working electrode in contact with the sample, each locally immersed in an aqueous electrolyte. Application of repetitive voltammetry, or sequential performance of constant potential reductive steps and voltammetric potential scanning, yielded voltammetric records corresponding to the stepwise reduction of corrosion products deposited in successive layers on copper surfaces. Multivariate analysis using voltammetric parameters and Tafel ln(current) vs. potential plots in the increasing portion of voltammetric peaks was used for identification of the corrosion products.

1. Original language version; submitted by author in English.

Evidence of tin coatings on archaeological ferrous artefacts by voltammetry of microparticles *(IA, LRMH)* [1]

New research project

Contact: Estelle Ottenwelter (ottenwelter@arup.cas) (IA), Virginia Costa (virginia.costa@gmail.com) (LRMH), Annick Texier (LRMH)

Funding: No external funding

Tinning of iron is a technique used since the 5th century BC by ancient craftsmen. It refers to coatings of tin or its alloys on the surface of ferrous objects. It was performed for various purposes. Owing to its similar appearance to silver, tinning was used as a decorative technique. It provides a corrosion resistant barrier, and was therefore used as a good corrosion protection method. It also provides a suitable surface for soldering. On archaeological/excavated artefacts, the tin layer can be more or less well preserved, but is usually hidden under voluminous iron corrosion products. Its presence can be detected using X-radiography, but a complete characterization is very important from both the curatorial and conservation point of views.

Among the analytical methods satisfying the requirements for investigating cultural heritage, voltammetry of microparticles (VMP) appears to be a very suitable method to evidence the presence of tin coatings: locally and selectively, using extremely small amounts of material. In this work VMP has been used to characterize five medieval objects covered with white coatings in variable states of conservation. They comprise two iron fittings, a buckle, a fragment of a spur and an ear-pendant.

Results evidenced that all the objects were tinned completely with a coating of tin/lead alloy, although the tinning layers showed very variable appearance according to its differential conservation over the same object. Furthermore, results demonstrated the spur was coated firstly with a tin/lead alloy, then silver-plated with a foil of silver and a soft solder. It was also repaired on its extremity by another solder, which contained copper. These analyses have successfully demonstrated that VMP is a suitable method to:

- identify residual tin coatings present in wide states of conservation;
- differentiate it from soft solder and silver plating; and
- highlight heterogeneities of coatings, if present.

1. Original language version; submitted by author in English.

Local investigation of corrosion processes by coupled electrochemical and spectroscopic techniques *(LISE)* [1]

Ongoing research project

Contact: Vincent Viver (vincent.vivier@upmc.fr), Marie-Claude Bernard, Suzanne Joiret (LISE)

Funding: No external funding

The investigation of corrosion of historic and archeological artifacts is a difficult task, especially from an electrochemical point of view due to the lack of available samples for destructive analysis, the wide range of sample compositions, and the poor knowledge of object history and storage conditions. Since the nominal composition of each metallic artifact is usually unique, the synthesis of artificial corrosion products offers a route for the global understanding of corrosion processes. However, one should be able to compare, at least once, the reactivity of the synthesized products to the ones of the artifact.

To overcome these difficulties, we took advantage of the development of the cavity microelectrode (CME) technique, which allows investigation of very small amounts of powder materials (typically nanograms).[2, 3] This technique offers several advantages, including the ability to:

- Take layer-by-layer sampling of the corrosion products in order to separate the electrochemical contribution of the bare metal from that of the corrosion products;
- Locally characterize regions of interest; and
- Couple it with spectroscopic techniques such as Raman spectroscopy; allowing *in situ* and *ex situ* investigations of the reactivity of corrosion products.

The CME technique has been developed for the study of powder materials. In addition, special attention has been paid to the coupling of this local electrochemical technique with Raman spectroscopy. For example, cyclic voltammetry was performed concurrently on patinas, thereby evidencing various redox moieties that depend on the nominal composition of the bronze and the history of the artifact. Another electrochemical technique, electrochemical impedance spectroscopy (EIS) allowed a fine analysis of the corrosion mechanism, while Raman spectroscopy permitted the identification of species.

The “Mission des Ressources et Compétences Technologiques” of CNRS and the UMEC network are gratefully acknowledged for their support.

1. Original language version; submitted by author in English.

2. M. Serghini-Idrissi, M.C. Bernard, F.Z. Harrif, S. Joiret, K. Rahmouni, A. Srhiri, H. Takenouti, V. Vivier, M. Ziani, Electrochim. Acta, 50 (2005) 4699-4709.

3. M.C. Bernard, S. Joiret, Electrochim. Acta, 54 (2009) 5199-5205

New routes for metal protection – combining conducting polymers with SAMs *(UL)* [1]

Ongoing research project

Contact: Luisa Maria Abrantes (lmabrantes@fc.ul.pt) (UL)

Funding: Fundação para a Ciência e Tecnologia

Conducting polymers (CP) can be prepared by an oxidative treatment of the respective monomer in solution. The formed material is doped, i.e. in the conducting state, but it can be reduced to the neutral form, this being a reversible process. The oxidised form of the polymer has metallic conducting properties and is hydrophilic, whereas the reduced state has semiconducting properties and is hydrophobic.

Besides the separation of the metal dissolution and oxygen reduction processes (by covering the metal substrate with a CP film), other unique features of these materials explain the great interest in their use as corrosion protective coatings: the positive value of their redox potentials and the possibility of promoting a catalytic reduction of oxygen with simultaneous formation of a passive metal oxide film on the substrate.

For a large variety of metals where corrosion protection is needed (e.g. Fe, Cu), the anodic or chemical oxidation of the monomer is accompanied by dissolution of the metal. Several efficient ways have been described to overcome this difficulty; among others, oxalic acid and sodium salicylate are commonly used for the formation of an interlayer which suppresses the metal dissolution without preventing the electropolymerization process.[2]

Concerning the protecting ability of the polymer, the behaviour of Cu coated with polypyrrole (PPy) in NaCl 3.5% solution when compared with bare metal, has shown a shift of corrosion potential to more positive values, an accentuated decrease in oxidation currents,[3] as well as a diminishing anodic peak assigned to CuCl formation, indicating that the substrate is isolated from the aggressive anions. Identical properties have been reported for polyaniline coatings on steel.[4]

It is recognized that corrosion rate depends on the coating adhesion strength.[5] It is also known that self-assembled monolayers (SAM) with two reactive groups, one to the metal surface and the other to the protective coating, can act as adhesion promoters.[6] By applying this concept to conducting systems, a covalent grafting of protective layers on metal substrates is feasible.[7] Bi-functional molecules with a polymerizable terminal group have been used to carry out *in situ* surface polymerization or reaction with further monomers in solution. The potentiostatic deposition of PPy on Cu modified with a pyrrolyl lipoic acid SAM exemplifies the promising performance of such new coatings.[8] This novel route for metal protection might be of interest for the preservation of heritage materials exposed to atmospheric or aquatic media. Work is in progress considering silver and copper alloys artefacts.

1. Original language version; submitted by author in English.

2. A.C.Cascalheira, L.M. Abrantes, Electrochim. Acta 49 (2004) 5023

3. A.C.Cascalheira, L.M. Abrantes, Corr. Prot. Mater.,23 (2004) 6

4. D.E.Tallman, Y.Pae, G.P. Bierwagen, Corrosion. 55 (1999) 779

5. J.O. Iroh, Y. Zhu, K. Shah, K. Levine, R. Rajagopalan, T. Uyar, M. Donley, R. Mantz, J. Johnson, N.N. Voevodin, V.N. Balbyshev, A.N. Khramov, Progress in Organic Coatings, 47 (2003) 365

6. I. Maege, E. Jaehne, A. Henke, H-J. P. Adler, C. Bram, C. Jung, M. Stratman, Progress in Organic Coatings,34 (1998) 1

7. E. Jaehne, S. Oberoi, H-J. P. Adler, Progress in Organic Coatings,61 (2008) 211

8. L.M. Abrantes, Workshop "Electrochemistry in Historical and Archaeological Conservation" Jan 11-15, 2010, Leiden, Netherlands

Abbreviations and acronyms

ASP: Analytical Science Projects

CME: cavity microelectrode

CNRS: Centre National de la Recherche Scientifique, France

CP: conducting polymer

EH: English Heritage, United Kingdom

EIS: electrochemical impedance spectroscopy

ESA: Electrochemistry and Surface Analysis

GU: Ghent University, Ghent, Belgium

IA: Institute of Archaeology, Czech Republic

IVC+R: Instituto Valenciano de Conservación y Restauración de Bienes Culturales de la Generalitat Valenciana, Spain

LISE: Laboratoire Interfaces et Systèmes Electrochimiques, France

LRMH: Laboratoire de Recherche des Monuments Historiques, France

SAM: self-assembled monolayer

UL: Universidade de Lisboa, Portugal

UMEC: utilisateurs de la microélectrode à cavité, France

UPV: Universitat Politècnica de València, Spain

UV: Universitat de València, Spain

UW: University of Warwick, Coventry, United Kingdom

VMP: voltammetry of microparticles

General information

Future seminars and conferences

- New **Masters in Heritage Science** – a new course is available at UCL Centre of Sustainable Heritage, London, UK. It caters for interdisciplinary students and for heritage professionals with several years of experience and demonstrable aptitude for research. The one-year research-focussed course is delivered using innovative pedagogy and will be attractive to researchers and practitioners alike. Further information is available at: http://www.ucl.ac.uk/sustainableheritage/mres.html

- New **3rd International Conference, Archaeometallurgy in Europe 2011** (29 June – 1 July 2011, Bochum, Germany). The call for presenters and attendees is open for this interdisciplinary conference hosted by the Deutsches Bergbau-Museum (www.bergbaumuseum.de). For more information contact Andreas Hauptmann: aie3@bergbaumuseum.de

- New **Tecnarte 2010** (September 8-10 2010, Buenos Aires, Argentina): a conference on advances in analytical techniques applied to the study of materials in art and archaeology. The event, organized in thematic workshops, aims to update and discuss the application of analytical techniques to characterize and conserve cultural, artistic, and archaeological heritage. Universidad de Buenos Aires, Faculty of Engineering, Argentina. E-mail: tecnarte@fi.uba.ar

- **Big Stuff 2010** (6-8 October, 2010, Duxford, England). Imperial War Museum is delighted to announce Big Stuff 2010, hosted at IWM Duxford, near Cambridge. The theme of the conference is conflict and avoidance of conflict between the display and conservation requirements of Large Technology Objects within the museum setting. For further information contact: Chris Knapp ACR, Conservation Manager, Imperial War Museum, Duxford (cknapp@iwm.org.uk).

- **ENAMEL 2010** 3rd Experts meeting on Enamel on Metals Conservation (8-9 October, 2010, Frick Collection, New York, United States of America). Organised by ENAMEL, sub-WG of the WGs "Metals" and "Glass & Ceramics". For more information see http://www.icom-cc.org/52/event/?id=68.

- **Metal 2010**: Triennial Metals Conservation Conference (11-15 October, 2010, Charleston, South Carolina, United States of America). Metals Working Group of ICOM Committee for Conservation. Publication timeline: http://www.timetoast.com/timelines/4880 Further information: http://www.icom-cc.org/51/news/?id=22.

Announcements

- New **Ge-Conservacion** is a periodical published by GEIIC (Grupo Español de Conservación/Spanish Conservation Group of the International Institute for Conservation of Historic and Artistic Works: www.ge-iic.com/) in association with the Duques de Soria Foundation. Its purpose is to contribute to the scientific development, dissemination and exchange of cultural heritage conservation and restoration knowledge. The journal aspires to be a critical tool and will give priority to interdisciplinary approaches and reasoned criteria and methodologies. It is open to all persons whose articles are in keeping with the publication's objectives and characteristics: http://ge-iic.com/revista/index.php?lang=en

- New **Conserving Outdoor Sculpture (The Stark Collection at the Getty Center)** by B. Considine, J. Wolfe, K. Posner and M. Bouchard ISBN 978-1-60606-010-0, 2010. When the J. Paul Getty Museum received twenty-eight sculptures created by a who's who of twentieth-century artists, it took on the responsibility for their preservation, interpretation, and long-term stewardship. To honor its responsibility, the Museum embarked on new research into the collection's materials — bronze, lead, ceramic, and painted metal — and construction techniques. This book presents the conservators' comprehensive account of the process and addresses key issues facing anyone charged with caring for works of art displayed outdoors: http://www.getty.edu/bookstore/titles/starkcons.html

Websites

- New **BROMEC ListServ:** For direct email notification of BROMEC publication web links and calls for submission of abstracts and announcements, simply subscribe with your preferred email address: http://listserv.csv.warwick.ac.uk/mailman/listinfo/bromec-bulletin-of-research-on-metal-conservation

- New **Electrochemistry in Historical and Archaeological Conservation** (11-15 January 2010, Leiden, the Netherlands). The majority of presentations from this workshop held at the Lorentz Center (http://www.lorentzcenter.nl/), are available for download: http://tinyurl.com/lorentzpresentations

- **ANDRA**: Agence Nationale pour la Gestion des Déchets RadioActifs. The following documents can be ordered for free from this website: *Analogues archéologiques et corrosion* (French) and *Prediction of Long Term Corrosion Behaviour in Nuclear Waste Systems* (English) (http://www.andra.fr/interne.php3?publi=publication&id_rubrique=82&p=produit&id=5).

- **ARTECH network**: Network facilitating the access of conservation professionals to different investigation techniques for Cultural Heritage artefacts (http://www.eu-artech.org/).

- **BigStuff 2004**: Care of Large Technology Objects (http://www.awm.gov.au/events/conference/bigstuff/index.asp).

- **CAMEO**: Chemical, physical, visual, and analytical information on over 10,000 historic and contemporary materials used in the conservation, preservation, and production of artistic, architectural, and archaeological materials (http://cameo.mfa.org/).

- **Cost Action G7: Artwork conservation by laser**: (http://alpha1.infim.ro/cost).

- **Cost Action G8: Non-destructive analysis and testing of museum objects**: Abstracts and booklets from previous workshops can be downloaded as well as announcements of past activities (Short Term Scientific Mission deadlines, training schools…) (http://srs.dl.ac.uk/arch/cost-g8/).

- **Cost Action D42: ENVIART**: Chemical Interactions between Cultural Artefacts and Indoor Environment. Register (free) to access all information (http://www.echn.net/enviart/).

- **e-Preservation Science**: Online publication of papers in conservation science (http://www.morana-rtd.com/e-preservationscience/).

- **European Cultural Heritage Network**: European network of professionals interested in the conservation of Cultural Heritage (http://www.echn.net/).

- **ICOMAM**: International Committee of Museums and Collections of Arms and Military History: (http://www.klm-mra.be/icomam/).
- **ICOM-CC Metals Working Group**: (http://www.icom-cc.org/31/working-groups/metals/). This site is for all official ICOM-CC Metals WG activities, forums, news, file downloads and information. The co-ordinator can email members from this site once members have registered on-line as a member of the Metals WG. Public access to this site is limited.
- **Incredible Industry**: On behalf of the Nordic Association of Conservators, Denmark, Morten Ryhl-Svendsen, Karen Borchersen and Winnie Odder, the proceedings from the Nordic Association of Conservators 18th Conference, "Incredible Industry, Preserving the Evidence of Industrial Society" (25-27 May 2009, Copenhagen, Denmark) are now freely available online (www.nkf-dk.dk/Bulletin/NKF-Incredible-industry09.pdf). The 243-page (PDF, 9.5 Mb) publication consists of 25 papers from the presentations given during the three-day conference.
- **Industrial artifacts review**: Industrial design and the role of art and photography in promoting cultural heritage (http://industrialartifactsreview.com/).
- **Infrared and Raman for cultural heritage**: (http://www.irug.org/default.asp).
- **Laboratoire Pierre Sue**: LPS PhD thesis related to the alteration of archaeological artefacts can be downloaded in French. Follow the link to "Archéomateriaux et prévision de l'altération" (http://www-drecam.cea.fr/lps/).
- **LabS-TECH network**: (http://www.chm.unipg.it/chimgen/LabS-TECH.html).
- **METALCons-info**: Metals Conservation Information (http://metalsconservationinfomation.wetpaint.com/) is where the old METALCons-info site is being moved and redeveloped. This is a wiki based site, which means it can be grown by contributions from "writers" - i.e. you. Its power depends on how willing you are to use it. Each week it sends a summary of activity to members – so sign up! It is currently publicly visible, but this may change with any unwanted activity.
- **M2ADL**: Microchemistry and Microscopy Art Diagnostic Laboratory (http://www.tecore.unibo.it/html/Lab_Microscopia/M2ADL/).
- **New York Conservation Foundation**: (http://www.nycf.org/).
- **PROMET**: A 3.5 year European 6th Framework funded project (21 partners from 11 countries around the Mediterranean basin) that developed conservation strategies for outstanding metals collections throughout the Mediterranean (http://www.promet.org.gr).
- **Restauración Metal Sur América**: (http://www.restauraciondemetales.cl/).
- **TEL**: PhDs on line (http://tel.ccsd.cnrs.fr/).
- **Yahoo Groups Metals Conservation**: A discussion group for all who are interested in Metals Conservation. Join in and make this a "Metals Cons-Dist List" (http://groups.yahoo.com/group/Metals-Conservation-Discussion-Group).

National Contacts

- **Argentina**: Blanca Rosales (brosales@fibertel.com.ar), researcher, CIDEPINT, La Plata.
- **Australia**: David Hallam (dhallam @ nma.gov.au), senior conservator-restorer of objects, National Museum of Australia, Canberra.
- **Belgium**: Annemie Adriaens (annemie.adriaens@ ugent.be), researcher and lecturer, head of the group "Electrochemistry and Surface Analysis", Ghent University, Ghent and Gilberte Dewanckel (gilberte.dewanckel@ kikirpa.be), conservator-restorer, Royal Institute of Artistic Heritage (Institut Royal du Patrimoine Artistique), Brussels.
- **Bulgaria**: Petia Penkova (petiapenkova @ yahoo.com), conservator-restorer, National Academy of Arts, Department of conservation-restoration, Sofia.
- **Canada**: Judy Logan (judylogan @rogers.com), conservator-restorer (retired), Ottawa.
- **Chile**: Johanna Theile (jtheile@udd.cl), conservator-restorer and lecturer, Faculty of Art, University of Chile The Oaks (Facultad de Arte, Universidad de Chile Las Encinas), Santiago de Chile.
- **Croatia**: Zoran Kirchhoffer (zoran.k @ tehnicki-muzej.htnet.hr), conservator-restorer, Zagreb Technical Museum (Tehnički muzej Zagreb) and Sanja Martinez (smartin@ fkit.hr), electrochemist and lecturer, Faculty of Chemical Engineering and Chemical Technology, University of Zagreb (Sveučilište u Zagrebu), Zagreb.
- **Denmark**: Karen Stemann Petersen (karen.stemann@ natmus.dk), conservator-restorer, The National Museum of Denmark (National Museet), Copenhagen.
- **Egypt**: Wafaa Anwar Mohamed (wafaaanw @yahoo.com), conservator-restorer, Giza.
- **Finland**: Pia Klaavu (pia.klaavu @ nba.fi), conservator-restorer, National Museum of Finland (Suomen kansallismuseo), Helsinki.
- **France**: Elodie Guilminot (elodie.guilminot@arcantique.org), conservation scientist, Arc'Antique, Nantes.
- **Germany**: Britta Schmutzler (britta.schmutzler @ gmx.de), PhD "object conservation" student, State Academy of Art and Design (Staatliche Akademie der Bildenden Künste), Stuttgart.
- **Greece**: Vasilike Argyropoulos (bessie@teiath.gr), assistant professor, Department of Conservation of Works of Art, Technological Educational Institution, Athens.
- **Hungary**: Balazs Lencz (lenczb@gmail.hu), senior conservator-restorer, Conservation Department, Hungarian National Museum (Magyar Nemzeti Muzeum), Budapest.
- **Italy**: Paola Letardi (paola.letardi@ismar.cnr.it), scientist, Institute for Marine Corrosion of Metals (Istituto per la Corrosione Marina dei Metalli), Genova.
- **Morocco**: Hind Hammouch (hamhind @ yahoo.fr), scientist, Laboratory of Electrochemistry, Corrosion and Environment, Faculty of Science, Ibn Tofail University, Kenitra.
- **The Netherlands**: Ineke Joosten (ineke.joosten @ icn.nl), conservation scientist, The Netherlands Institute for Cultural Heritage (Instituut Collectie Nederlan), Amsterdam.
- **Norway**: Douwtje Van der Meulen (d.l.v.d.meulen@iakh.uio.no), conservator-restorer, Conservation Department, University of Oslo (Universitetet i Oslo), Oslo.
- **Portugal**: Isabel Tissot (isabel.tissot@archeofactu.pt), conservator-restorer, Portuguese conservation-restoration Institute (Instituto Português de Conservação e Restauro), Lisbon.

- **Romania**: Dorin Barbu (barbu_dorin_laboratory@ yahoo.com), conservator-restorer, National Brukenthal Museum (Muzeul Naţional Brukenthal), Sibiu.
- **Russian Federation**: Andrey Chulin (andrey_chulin @ yahoo.com), conservator-restorer, the State Hermitage Museum, St Petersburg.
- **South Africa**: Jaco Boshoff (jboshoff @ iziko.org.za), maritime archaeologist, Iziko Museums of Cape Town, South Africa.
- **Spain**: Emilio Cano (ecano @ cenim.csic.es), scientist, National Centre for Metallurgical Research (Centro Nacional de Investigaciones Metalúrgicas), Spanish Council for Scientific Research (Consejo Superior de Investigaciones Científicas), Madrid.
- **Sweden**: Helena Strandberg (helena.st @ comhem.se), conservator-restorer and conservation scientist, freelancer, Göteborg.
- **Switzerland**: Valentin Boissonnas (valentin.boissonnas @ he-arc.ch), conservator-restorer and lecturer, Technical University (Haute école de conservation-restauration Arc), La Chaux-de-Fonds.
- **United Kingdom**: Maickel van Bellegem (Mbellegem @ thebritishmuseum.ac.uk), conservator-restorer, British Museum, London and Mark Dowsett (m.g.dowsett @ warwick.ac.uk), physicist, University of Warwick, Coventry.
- **United States of America**: John Scott (NYConsnFdn @ aol.com), New York Conservation Foundation, New York.