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Metals Working Group

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BROMEC30

Editorial

This BROMEC contains a *call for collaboration* concerning a precipitate formed on excavated marine iron artefacts while undergoing alkaline stabilisation treatments. Also presented are two *ongoing research projects*: the first being from Cuba, assesses the potential use of alkaline waste from industrial paper production for use as atmospheric corrosion inhibitors; and the second is an 18-month project testing sodium carboxylates and carboxylatation [*sic*] solutions for large copper and iron based technical artefacts in uncontrolled indoor environments. Of the two *finalised research projects* described: the first describes how filiform corrosion of atmospherically exposed iron and steel preserves traces of the former metal surface in their corrosion product structures; while the last outlines the use of corrosion potential measurements over time to identify the composition of binary and ternary copper alloys. Between the triennial ICOM-CC Metals Working Group meetings (now less than one year away), we encourage you to make contact with authors for any queries about their research.

Free, online dissemination of conference proceedings is made by the 18th Conference of the Nordic Association of Conservators, "Incredible Industry, Preserving the Evidence of Industrial Society", with several papers specifically discussing appropriate surface finishing and protection of metal cultural heritage. Meeting announcements include: a Daguerreotypes study-day and Big Stuff 2010, the Large Technology Objects meeting, this time with the theme, "On the conflict and avoidance of conflict between the display and conservation requirements of Large Technology Objects within the museum setting."

And finally, welcome to Sanja Martinez, scientist at the University of Zagreb, who joins Zoran Kirchhoffer as Croatia's National Contacts for the ICOM-CC Metals Working Group.

With 30 issues now behind it, BROMEC has further developments in store for the near future. For now, as usual, we wish you informative and interesting reading.

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Contents

Page

Calls for collaboration


The formation process of a white deposit precipitated during the stabilization treatment of iron archaeological objects by reduction with sodium hydroxide 3

Ongoing research projects


Development of atmospheric corrosion inhibitors for ferrous metals 4


POINT: temporary protection of large metal technical and scientific equipment in uncontrolled atmospheres 5

Finalised research projects


Atmospheric corrosion of coated iron and steel, and the effects of corrosion typologies and historical accessibility on the development and survival of modified surfaces in their corrosion products 6


SPAMT Test: qualitative analysis of copper based alloys on scientific, technical and horology objects from E_{corr} versus time plots : feasibility and limits of use 7

Abbreviations and acronyms 8

General information

Future seminars and conferences 8

Announcements 9

Websites 9

National Contacts for the ICOM-CC Metals WG 11

Call for collaboration

 The formation process of a white deposit precipitated during the stabilization treatment of iron archaeological objects by reduction with sodium hydroxide. (***MP-IVC+R-AE***) [1]

Since there is no standard machinery on the market for archaeological iron stabilization by akaganéite removal, each professional has to adapt available resources to serve their needs. From this situation, our desalination treatments – so far with small objects – were made in boro-silicate containers in which 2% NaOH solutions were prepared and objects immersed. These containers were covered with transparent plastic and aluminium foil, all well joined with cellulose adhesive tape, and then placed in an oven; 60º C inside the liquid. They remained there for long periods: as long as they had salts in solution. Every two weeks, a potentiometric titration process was carried out with a sample (50 mL) from each container to plot the corresponding graphs.

After some days or weeks (case dependent) a white *sugary* appearing crust gradually began to form on the bottom of the container, and completely covering the objects in an opaque and uniform way, as shown by SEM photographs. It precipitated even in the finest cracks, pores, etc of any oxidation products surrounding the metal core, as was the case for magnetite. First EDX analysis determined that it was a compound based on silicon, aluminium, potassium and sodium. For the time being, we do not know which process produces this precipitate. It is anticipated to make further analyses on the soil in which the objects were buried, the glass containers in which the desalinations were made and again on the resulting whitish products under question. Once we understand its formation, we will begin to develop mechanisms for its removal. The research continues.

We are not aware of scientific literature previously describing this phenomenon. So, from here, we invite – and appreciate – any researcher who has become aware of the same, or similar phenomenon, to contact the project leaders, to share knowledge and opinions.

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Funding: No external funding

[1] Translated by D. Lafuente, E. Cano & J. Crawford. Original version submitted by author in Spanish; refer to BROMEC 30 Spanish version.

Ongoing research project

 Development of atmospheric corrosion inhibitors for ferrous metals. ***(CENCREM)*** [2]

Atmospheric corrosion is the most widespread type of corrosion since approximately 80% of installations, sculptures, historic buildings and metal objects are exposed to these conditions; causing significant social and economic losses, especially in tropical countries. Since the superficial electrolyte formed by the condensation of unpolluted atmospheric water vapour usually has near neutral pH, selection of atmospheric corrosion inhibitors is limited to those species capable of reducing the aggressive effects of neutral solutions. For low-income countries, an interesting alternative could be the use of national primary resources or industrial waste in order to develop means of corrosion protection. Atmospheric corrosion inhibitors can be of the volatile and contact types. The latter type is applied directly on the metal surface.

This research studies the possibility of using industrial waste to formulate an effective contact inhibitor in Cuba's humid tropical climate. As a contact inhibitor the mixture of sodium nitrite ($NaNO_2$ 25-30%) with sodium carbonate (Na_2CO_3 0.1%) and glycerine (15%) is established. Nitrite ions create a protective, passive film on the metal surface; sodium carbonate ensures an alkaline pH; and glycerine prevents drying out of the surface. GIC-1 is a waste product obtained in the washing step of the bagasse pulp during paper production. Known as *black liquor*, it consists mainly of lignin and has a very alkaline pH, about 12, which prompted our consideration of its potential inhibitive properties for ferrous alloys in neutral media.

GIC-1 was evaluated at differing concentrations (3, 5, 10 g/L) as a corrosion inhibitor for carbon steel (CT-3), in sodium chloride solutions (NaCl 0.001N), and sodium sulphate (Na_2SO_4 0.001N). Evaluation was made by weight loss measurements for 10 days, at room temperature and while in complete immersion. Efficiencies were comparable to potassium dichromate (K_2CrO_4 0.29 g/L); a recognized inhibitor for neutral media. This allowed us to consider its use as a component in temporary means of protection. Formulas with GIC-1 (50%) were prepared, maintaining glycerine at a constant concentration, like the commercial formula (15%). Sodium nitrite, an imported compound, was lowered to 1, 3, 5%, and sodium carbonate was eliminated, due to the alkalinity of the solutions (pH = 9.1-9.9). New formulas were tested with steel samples (20 x 50 x 1 mm) wrapped in wax paper and placed in a salt spray chamber (T = 35 °C, NaCl 3%, t = 96 hours, pH = 7), achieving very satisfactory efficiencies (95.75-98.56%) and protective effects (23.5-69.6%). Two formulas were selected ($NaNO_2$ = 5 and 10%), and tested in a rural corrosion station (sheltered with walls), and in a marine environment (sheltered without walls), for 1 year, placing the wax paper wrapped steel samples (100 x 150 x 1 mm) in polyethylene bags. Corrosion inhibition efficiencies in both cases were higher than the commercial formula:

- Coastal: GIC-1 = 94.09-96.54% versus commercial = 91.6%
- Rural: GIC-1 = 95.88-97.34% versus commercial = 95.55%

Results allow use of GIC-1 waste to formulate atmospheric corrosion inhibitors; reducing the problems of its disposal and damage to the environment.

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Funding: No external funding

[2] Translated by D. Lafuente, E. Cano & J. Crawford. Original version submitted by author in Spanish; refer to BROMEC 30 Spanish version.

Ongoing research project

POINT: temporary protection of large metal technical and scientific equipment in uncontrolled atmospheres. (***HECR Arc***) [3]

The surfaces of large copper and iron based technical artefacts stored or exhibited indoor in uncontrolled environments or outdoor are often protected with opaque paint films initially developed to protect bare metal surfaces. The application of these films requires a very thorough preparation of the metal surface (chemical cleaning, sandblasting). Such a condition is almost impossible to respect on historic metal technical artefacts because of the need to conserve the metal, and potentially any modified original surfaces still present in the corrosion product layers. Therefore it is not surprising to observe on historic artefacts, which have been cleaned of any powdery corrosion products and coated afterwards with paint films, specific corrosion phenomena (filiform corrosion) that develop under the protection system. To help assure long term conservation of the surfaces (and subsurfaces) of these artefacts the complete removal of this paint film is generally carried out, but this cleaning process can create new damage to surfaces that are already highly fragile.

A specific approach needs to be developed for the protection of these technical objects. The proposed protection system must be transparent and compatible with the residual surface (with or without traces of the original surface treatment). It must be reversible or re-treatable to ensure a better long term protection. Protection of several months must be guaranteed. Finally, the personnel in charge of the maintenance of the collections, who are rarely conservation professionals, should be able to apply the protection systems independently with minimal supervision, following training.

The protection systems considered in this project (sodium carboxylates and carboxylatation [*sic*] solutions) have been developed within the European PROMET project[4, 5] for temporary protection of iron and copper based artefacts exposed to uncontrolled atmospheres. These corrosion inhibitors are not patented compounds with restricted production, are non toxic (derived from fatty acids extracted from vegetable oils: colza, sunflower and palm) and seem to be effective over a one year period in uncontrolled indoor environment/s. In this 18-month project, which started six months ago, their formulation and mode of application was optimised on an initial series of artificial metal coupons simulating the alteration of exposed real artefacts, and then exposed to accelerated ageing in a humidity chamber and examined destructively. The best performing protection systems will now be tested in real conditions; both on a second series of artificial metal coupons prepared in a similar way as before, and on artefacts from the Fondation du matériel historique de l'armée suisse (HAM). The coupons and the artefacts will be exposed next to one another so as to assess the efficiency and reversibility of the protection systems tested according to the specific conditions of artefacts' storage[6]. Additional information will be given during Metal 2010.

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Funding: HES SO

[3] Original language version; submitted by author in English.
[4] Hollner, S., Mirambet, F., Texier, A., Rocca, E. and Steinmetz, J., 2007. Development of new non-toxic corrosion inhibitors for cultural property made of iron and copper alloys. Argyropoulos, V., Hein, A. and Harith M. A. (eds), *Strategies for Saving our Cultural Heritage: papers presented at the International Conference on Conservation Strategies for Saving Indoor Metallic Collections 26-28 February 2007*. TEI: Athens. pp. 156-161
[5] Degrigny, C., 2008. The search for new and safe materials for protection of metal objects. Argyropoulos, V. (ed),, *Metals and Museums in the Mediterranean – Protecting, preserving and interpreting*, Athens: TEI. pp. 179-235.
[6] Degrigny, C., Mirambet, F., Tarchini, A., Ramseyer, S., Rapp, G., Jaggi, A., 2009. Le projet POINT – protection temporaire de matériels métalliques techniques et scientifiques de grandes dimensions en atmosphère non contrôlée, in *Conservation préventive – pratique dans le domaine du patrimoine bâti, colloque SCR, Fribourg, 2009*. pp. 121-126

Finalised research project

Atmospheric corrosion of coated iron and steel, and the effects of corrosion typologies and historical accessibility on the development and survival of modified surfaces in their corrosion products. (***HM-UM***) [7]

Corrosion of buried (i.e. *archaeological*) wrought iron, particularly the determination of modified traces of its original surfaces evidenced in its corrosion products (CPs) (i.e. the *limitos*), has attracted much discussion. However, corrosion of coated wrought iron and rolled mild steel in atmospheric (i.e. *historical*) conditions, and how this material-environment affects former surfaces, has not received much attention, if any. These ferrous metals are prevalent in museums and often feature protective organic coatings. Considering documentation of former maintenance and restoration interventions can be scant, or non-existent, it is difficult to confidently determine which period corresponds to current surfaces. But a preceding question remains: "Can corrosion products from atmospherically corroded (coated) wrought iron and low-carbon steel support evidence of the metal's former surface?" The motivation for investigating this issue stemmed from an historically accessible and atmospherically exposed collection: the partly corroded 16-17th century northern Italian-style wrought iron and low-carbon steel armour of the Palace Armoury (PA)[8].

Non-invasive and non-destructive investigations of the PA's largely undecorated munition armour, and its environment, enabled, via observation of the formed CP morphologies, deducing their probable corrosion processes. Investigation techniques included macrophotography, photomicroscopy, metallography, SEM-EDS and μRaman spectroscopy. Information gained from *authentic* armour was used to corrode, in a laboratory, armour *analogues* (artefact simulation material of contemporary rolled low-carbon steel). Corrosion products on the analogues were destructively studied for *markers* above (e.g. applied particles, protective coating), corresponding with (e.g. surface marks) and below (e.g. microstructures) the former metal surface. Analogue results were non-invasively validated with authentic PA armour. Using simple physical techniques, CP removal was performed on the analogues to the level determined by this research to respect modified former surfaces

Filiform corrosion morphologies (most commonly associated with organic polymer coatings) always exhibited evidence of the previous metal surface (in the form of topographical *micro-grooves*) in their raised CP filaments. It is perceived these filaments retained surface evidence due to the limited corrosion beneath. Meanwhile, *local to general corrosion* morphologies demonstrated the former surface was only evidenced with micro-grooves if corrosion was during its earliest phase: afterwards when corrosion in subsurface pits continued, any initially apparent micro-grooves became more displaced and eventually deformed beyond recognition.

The determination of filiform corrosion as an atmospheric ferrous CP morphology that consistently retains certain surface characteristics can assist conservators in readily identifying the location of this modified surface resulting from this specific corrosion typology. Various concerns are presented by CPs; nonetheless, if filiform corrosion filaments are removed, this would now be performed with the knowledge that manufacture finishing, evidence of use and/or maintenance in their surfaces will be irreparably lost.

Electronic copies of this Masters research dissertation are available from the author on request.

Contact: James Crawford (jamesbcrawford76@gmail.com) and Christian Degrigny (HM-UM)
Funding: No external funding

[7] Original language version; submitted by author in English.
[8] See BROMEC 14, 22 and 29

Finalised research project

SPAMT Test: qualitative analysis of copper based alloys on scientific, technical and horology objects from E_{corr} versus time plots : feasibility and limits of use. (***HECR Arc***) [9]

This project was previously introduced in BROMEC 26 and some results are given below. The objective was to study the possibilities of the E_{corr}-Drop Test (EDT) monitoring technique as a *spot test* for the qualitative analysis of metals (BROMEC 11); more particularly with copper-based alloys. The principle is simple since it consists of measuring the potential (E_{corr}) of a metal artefact's surface when a drop of solution (non aggressive towards the material) is deposited on its surface and monitored over time. A clean metal surface, representative of the metal core is required, as well as a reference electrode (RE) with its tip in contact with the drop. Both the metal and the reference electrode are connected to a voltmeter that measures the potential difference over a short period (5 to 15 minutes).

The qualitative determination via the SPAMT Test tool of the composition of an unknown copper based alloy works by comparing the collected electrochemical data with those of reference materials. Using three different solutions (Henniez Swiss mineral water, 1% w/v KNO_3 and 1% w/v sodium sesquicarbonate) we constructed a database of electrochemical behaviour of more than 50 alloys of known composition, and representative of alloys found on historic artefacts. The measurement conditions were optimised (standard polishing of the metal surfaces, insertion of the reference electrode in a junction protection tube (JPT) to prevent any outward diffusion of chlorides from the silver/silver chloride RE, standard position of the metal surface against the RE/JPT system and use of the same amount of electrolyte (volume of the drop = 40µL) to improve reproducibility. All measurements were carried out by conservators to ensure that all ethical and practical aspects would be considered: minimal impact on the artefacts, portability of the instrument, ease of use and low cost. The tool and the relevance of the database were tested on a group of objects representative of the collections of the Fondation du matériel historique de l'armée suisse (HAM, Thun) and the Musée International d'Horlogerie (MIH, La Chaux-de-Fonds).

We found that binary alloys could be identified quite easily. For ternary (or higher) alloys we could determine the presence of the constituents and sometimes with a relative concentration quite similar to the composition of the objects[10]. The SPAMT Test tool and its database appears to be a simple instrument that could be useful to conservators when carrying out diagnosis on site and defining conservation strategies (lead in the alloys makes them more sensitive to volatile organic compounds and the concentration of zinc in brass above 15% (in weight) to dezincification). Since the database is difficult to handle due to the large number of plotted curves, we are currently applying for funds to develop software to identify copper alloys based on their E_{corr} vs time plots. This software would be tested on a larger number of artefacts from the MIH. More information will be given during Metal 2010.

Contact: Christian Degrigny (christian.degrigny@gmail.com) (HECR Arc)
Funding: HES SO

[9] Original language version; submitted by author in English.
[10] Degrigny, C., Guibert, G., Ramseyer, S., Rapp, G. and Tarchini, A., 2009. Use of E_{corr} vs time plots for the qualitative analysis of metallic elements from scientific and technical objects: the SPAMT Test Project, *Journal of Solid State Electrochemistry*, available online: DOI 10.1007/s10008-009-0890-6, 2009

Abbreviations and acronyms

AE : Alcoa Europe, Alicante (Spain)
CENCREM: Centro Nacional de Conservación, Restauración, y Museología, Cuba (Cuba)
CP: corrosion product
EDS/EDX: energy dispersive X-ray spectroscopy
EDT: E_{corr}-Drop Test
GIC: Grupo de Inhibidores de Corrosión
HAM: Matériel historique de l'armée suisse (Switzerland)
HECR Arc: Haute école de conservation-restauration Arc (Switzerland)
HES SO: Haute Ecole Spécialisée de Suisse Occidentale (Switzerland)
HM-UM: Heritage Malta (Institute of Conservation and Management of Cultural Heritage) – University of Malta (Malta)
IVC+R: Departamento Científico, Laboratorio de Materiales, Instituto Valenciano de Conservación de Bienes Culturales, Valencia (Spain)
MIH : Musée International d'Horlogerie (Switzerland)
MP: Museo de Palencia, Junta de Castilla y León (Spain)
JPT: junction protection tube
PA: Palace Armoury (Malta)
POINT: Protection temporaire d'Objets métalliques base fer et cuivre à l'aide d'Inhibiteurs de corrosion Non Toxiques
RE: reference electrode
SEM: scanning electron microscopy
SPAMT: Suivi du Potentiel à l'Abandon de matériaux Métalliques en fonction du Temps

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General information

Future seminars and conferences

New **"Investigación en conservación del Patrimonio Cultural en España" (Cultural Heritage Conservation Research in Spain)** (17-18 March, 2010). Conference organised by the Reina Sofía Museum, the Spanish Ministry of Science and Innovation and the CSIC, within the framework of the NET-HERITAGE Project. The aim of this conference is to present the NET-HERITAGE project and the research work carried out by different groups of universities, research centres, restoration institutions in Spain, etc. For further information: www.investigacionenconservacion.es.

New **Daguerreotypes study-day** (1 April, 2010, Antwerp, Belgium). This one-day meeting on the conservation of daguerreotypes aims to provide a catalyst for uniting conservators who will highlight relevant conservation issues and develop a detailed conservation research framework for these very specific and complex metallic-composite objects. Study-day participants will be invited to join and contribute to this research.
The day is free of charge, and includes coffee and tea. Registration for attendance (or *in absentia* interest) closes March 5, 2010. For more information: patrick.storme@artesis.be.

New **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).

- **Archaeological Iron Conservation Colloquium** (24-26 June, 2010, Stuttgart, Germany). Held at the State Academy of Art and Design Stuttgart, in collaboration with AIAE "Archaeological Iron After Excavation", sub-WG of ICOM-CC Metals. For more information please contact Gerhard Eggert (gerhard.eggert@abk-stuttgart.de).

- **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 **Conference proceedings online – "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.

Websites

- **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.

- **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 America**: (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).

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