

# **IAEA Workshop on Innovative Approaches of Accelerator Science and Technology for Sustainable Heritage Management**

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## **Book of Abstracts**



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## Opening session / 32

## Advances, problems and potential for innovative accelerator science and technology approaches to sustainable Heritage Management in Cyprus, the Eastern Mediterranean and the Middle East (EMME)

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Sustainable Heritage Management requires complex knowledge, transdisciplinary approaches, and diverse competencies to meet the multiple challenges of heritage management, policy, and research. Understanding and appreciating the links between humans, the environment, and anthropogenic, tangible cultural heritage are critical in this endeavour. Innovative accelerator science and technology approaches, and advances within these domains, including synchrotron radiation enabled approaches, are key components in this process. These approaches provide critical data not only on structure, composition, age, manufacturing technology and techniques, the source of materials used, alterations, and conservation treatments of material culture, but also multiple types of data on ancient human remains and culturally relevant environmental samples.

This paper scrutinizes the advances and problems in, and potential for, innovative accelerator science and technology approaches to sustainable Heritage Management in Cyprus in particular, as well as draws comparisons within the Eastern Mediterranean and the Middle Eastern (EMME) region. This is done through (1) showcasing the application of accelerator science and technology for characterization and treatment of heritage objects, archaeological materials and remains, using specific case studies from Cyprus and the EMME region; (2) case studies on interpretation, presentation and dissemination of the scientific results; and (3) consideration of Cyprus' access, and regional access to research infrastructures, and international as well as regional collaborations and networks. On the latter point, the importance of feeder facilities, such as the BioMERA Platform (<https://biomera.cyi.ac.cy>), initiated through a 1-million-Euro grant from the Research and Innovation Foundation of Cyprus, are discussed in the context of synchrotron use, and SESAME synchrotron (Synchrotron-light for experimental science and applications in the Middle East) in particular. Further, the potential impact of scientific service companies in the context of Cyprus, and the EMME region is discussed.

The paper also reviews Sustainable Heritage Management policies and modalities in the context of Cyprus, Open Science and dissemination of knowledge in the Cypriot context, as well as research resources and funding structures relevant to accelerator science and technology approaches to sustainable Heritage Management in Cyprus.

The paper concludes with case studies and success stories from Cyprus and the EMME region (Eastern Mediterranean and the Middle East) on how to make science tangible, and how to promote public awareness of the transdisciplinary combinations of science and heritage studies leading to new holistic approaches. Novel methods and approaches in education and outreach, as undertaken within the framework of the FF-MAC project (Face to Face: Meet an Ancient Cypriot; a 1-million-Euro integrated project including enterprises and societal stakeholders as partners, together with research and governmental organisations; <https://face2face.cyi.ac.cy>), and their potential impact in the context of Cyprus will be explored.

## Opening session / 70

## AMS 14C dating of artifacts - prospects and challenges

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The radiocarbon dating method, which was developed in the early 1950's, is an essential chronometer for studying human history. An advent of the accelerator mass spectrometry AMS and a substantial downscaling of sample sizes from grams to milligrams of the material opened the doors for dating precious objects of cultural heritage.

The material studied typically is parchment, textiles, paper, and wood. Developments are made in analyzing less common materials such as iron, mortar, binding media, and pigments. Other technical developments lead to an increase in the capacity of existing AMS laboratories. Detection of forgeries can be supported by  $^{14}\text{C}$  analysis, which allows detecting material dating to other than declared time. Application of 'bomb peak'  $^{14}\text{C}$  allows successfully to see material formed in the last 60 years (post-nuclear test era). Successful applications of bomb peak resulted in an increased interest in applications of the  $^{14}\text{C}$  dating of art and cultural heritage objects. However, the conservation applied to Heritage samples is an obstacle that accurate  $^{14}\text{C}$  analysis must overcome. Understanding the nature of contamination is an essential part of the process.

This paper will present an overview of the methods applied in  $^{14}\text{C}$  dating heritage samples and discuss potential and limitations.

## Opening session / 1

# Neutron techniques employed to study objects from the Wallace Collection

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As well as being a picture gallery, this museum holds the largest collection of European armour in London, and one of the best collections of Indo-Persian swords and armour outside the subcontinent. While it is possible to analyse plate armour by microscopic examination of a cut edge, or a fragment detached from a hidden interior damaged surface, such as a sample from the helmet of King Henry V. Such examination is not possible with

swords, unless they are already broken, which rules out museum objects, although archaeological specimens may be available. So we have for some years been using neutron techniques to study swords and helmets from this museum, as well as from Canterbury Cathedral, at ISIS the neutron source in the Rutherford-Appleton Laboratory, Harwell, and other neutron sources. Neutron diffraction can tell us the carbon content of the steel, the phases present, and whether any heat treatment to harden the steel has been carried out.

We have examined a number of swords and daggers from the Oriental collection by neutron diffraction (since 2015 up to now 75). Many Oriental blades were made from crucible steel, a hypereutectoid cast steel, and a proportion of those were forged in such a way as to retain a distinctive surface pattern (the so-called "Damascus steel"). The high value of this steel led to many attempts to simulate its presence by etching, by differential welding, and even by welding thin layers of such a steel onto an iron body. Neutron diffraction can tell us the quantity of iron carbides present, any anisotropy in their distribution (viz. whether there is a pattern below the surface that has been hidden by overzealous 19th century polishing) and, possibly, even information about its thermal history.

Phase-contrast neutron imaging, developed at the Helmholtz Zentrum, Berlin, has proven to be an invaluable technique for rendering partly obliterated armourers' marks visible again. If these marks were struck cold, then the strained metal will

change the path of neutrons, and be detectable, even when covered over by later polishing. On the other hand, marks struck before the final heat-treatment and assembly, will not show any signs of microstrain. While revisualization of such marks will then be disappointing, new unexpected insights into workshop practice may arise.

## Opening session / 2

## **Ionizing Radiation for Preservation: Uses of Electron-Beam Technology for Conservation of Photographic and Cinematographic Films**

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The Nuclear and Energy Research Institute – IPEN through the Multipurpose Gamma Irradiation Facility and the Electron Beam Irradiation Facilities has disinfected several tangible cultural collections from the University of São Paulo – USP. Brazilian weather conditions added to the actions of insects and fungi promote biodegradation especially in cellulose based materials. In this sense, ionizing radiation is an excellent alternative to the traditional preservation process mainly because of the biocidal action. Electron beam irradiation also presents new possibilities for processing materials with greater speed, despite having limited penetration. Adequate storage of photographic and cinematographic materials is a challenge for experts from preservation institutions. Contamination by fungi is one of the leading causes of problems in this kind of collections. In addition, another common physicochemical degradation affecting cellulose triacetate films causing deacetylation of polymer chain is called “vinegar syndrome”. In this work are presented results of the effect of the electron beam irradiation on photographic and cinematographic films using an electron beam accelerator with energy of 1.5 MeV and beam power of 37.5 kW. Selected film samples were characterized by FTIR-ATR spectroscopy and FEGSEM-EDS microscopy. Samples were irradiated with absorbed dose between 2 kGy and 200 kGy. Irradiated samples were analyzed by UV-Vis spectrophotometry, FEGSEM, thermogravimetric analysis (TG) and differential scanning calorimetry (DSC). Results showed that disinfection by electron beam radiation can be achieved safely applying radiation absorbed doses between 6 kGy to 10 kGy with no significant change or modification of main properties of the constitutive polymeric materials. Electron beam irradiation, due to the effect of crosslinking is presented as an alternative to treat films affected by “vinegar syndrome” applying absorbed dose of 80 kGy in order to increase shelf life of cultural heritage materials.

**Interpretation, presentation and dissemination of the scientific results. Case studies and success stories / 94**

## **Success stories on the characterization of Cultural Heritage in Latin America: dating of the Mayan Codex of Mexico and determination of arsenic in Chilean mummies**

**Author:** Cecilia Fuertes<sup>1</sup>

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Two success stories on the characterization of cultural heritage in Mexico and Chile using accelerator-based techniques will be presented and discussed.

The first case is the determination of the authenticity of the Mayan Codex of Mexico (MCM). The Mesoamerican codices are documents describing pre-Hispanic history and cosmogony. The MCM was unveiled in 1971 during an exhibition at the Grolier Club of New York. This codex is a folding-screen book, with 11 pages painted on bark paper covered with a fine layer of stucco. It illustrates astronomical events related to Venus cycles. Since the discovery of the Mayan Codex, its authenticity has been subject of controversy and discussion due its scarce iconographic content and its unknown origin. Some studies have been conducted to verify if the materials in the MCM match with those found for other pre-Hispanic documents. Among them, the group led by Dr. Ruvalcaba presented a comprehensive study of the whole document using a set of non-destructive techniques (1). They concluded that no modern inorganic materials are present in the codex. Besides, it was also

determined that it contains materials used in pre-Hispanic times. More recently, the group led by Dr. Solís revised the dating of the MCM using AMS 14C (2). Results obtained placed the age of the bark paper between 1159 and 1261 cal AD, indicating that the vegetal support of the MCM belongs to Postclassical Mayan period.

The second case is the study of arsenic poisoning in mummies from Chile. Arsenic (As) is an abundant trace element in Andean groundwater, and arsenicism has been detected in ancient and modern populations. However, discriminating endogenous arsenic by ingestion from external contamination as a result of exposure to geogenic toxic contaminants remains an analytical challenge. To evaluate the origin of As in pre-Hispanic populations, Dr. Kakoulli and collaborators from University of Chile determined the distribution and chemical speciation of As in ancient human hair using synchrotron radiation based techniques (3). In addition, the group led by Dr. Echeverría measured the hair As concentrations of ninety-two pre-Hispanic naturally mummified individuals from northern Chile using INNA (4). These mummies belonged to communities with diverse traditions and had access to different water sources. The work points to the analysis of arsenic in hair as a useful tool in the study of living conditions and patterns of diet and mobility in ancient populations.

#### References:

- (1) The Grolier Codex: A Non Destructive Study of a Possible Maya Document using Imaging and Ion Beam Techniques, Jose Luis Ruvalcaba *et al*, Mater. Res. Soc. Symp. Proc. Vol. 1047, (607) 2007.
- (2) AMS 14C dating of the Mayan Codex of Mexico revisited, Corina Solís *et al*, Radiocarbon 62 (6), 2020.
- (3) Distribution and Chemical Speciation of Arsenic in Ancient Human Hair Using Synchrotron Radiation, I. Kakoulli *et al*, Anal. Chem. 86, 2014.
- (4) Arsenic in the hair of mummies from agro-ceramic times of Northern Chile (500 BCE - 1200 CE), J. Echeverría *et al*, Journal of Archaeological Science: Reports 21, 2018.

### Interpretation, presentation and dissemination of the scientific results. Case studies and success stories / 39

## A non-invasive protocol based on Ion Beam Analysis for the study of lapis lazuli provenance in antiquity

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Lapis lazuli is a semi-precious blue stone, used since the VII millennium BCE for the manufacturing of small carved artifacts (e.g. jewels, seals, amulets) in the Ancient Middle East and in part of Central and Southern Asia. The Badakhshan mines in Afghanistan are generally considered the most plausible hypothesis for the origin of the lapis lazuli used in antiquity[1]; however, the limited number of reference samples from well-documented geological sources, the records in ancient written evidence and the results of scientific provenance studies conducted since the 1960s have shown that the question of the provenance is still unsolved.

Despite the useful and interesting results obtained, most of these previous studies exploited invasive or destructive techniques, as for example IRMS[2,3] or AAS[1,4]. This approach is not always applicable in the case of precious archeological artifacts, where micro- or no sampling for the analysis is required. Driven by the growing interest in adopting conservative methods when analyzing such valuable objects and by the lack of a systematic and exhaustive study of the origin of the raw material of lapis lazuli artworks, the Solid State Physics group at the University of Torino started in 2008 a project involving different departments and institutions, still ongoing, with the aim of creating a provenance protocol based on Ion Beam Analysis[5]. In fact, the use of  $\mu$ -PIXE and  $\mu$ -IBIL has proved effective in finding markers within reference geological rocks able to distinguish among five provenances (Afghanistan, Tajikistan, Siberia, Chile and Myanmar, this last one recently added and

under study[6]. The markers can be then exploited to relate the raw material used for artifacts production to a specific geological source. The possibility of applying these techniques both in vacuum and in air (in this case without any sample preparation) ensures at the same time a non-invasive and high-sensitive approach, also in the case of archeological samples. Due to the lapis lazuli heterogeneity, minero-chemical markers are searched inside single mineral phases, exploiting the IBA capability to investigate matter down to the single crystal scale with a microprobe. The sample provenance can be investigated by scanning micrometric areas of its surface, and the target crystals are usually pre-identified with cold-Cathode Luminescence maps (when allowed by the sample dimensions) and SEM-EDX analysis, avoiding unnecessary irradiation of the sample and minimizing the analysis time.

The realized protocol has been so far applied to different archeological artifacts of various museum collection[5-7], successfully obtaining results regarding the provenance of the lapis lazuli material.  $\mu$ -PIXE and  $\mu$ -IBIL have been performed at the in-vacuum microbeam line of the National Laboratories of Legnaro of the Italian National Institute of Nuclear Physics and at the external microbeam lines at INFN-LABEC (Florence) and also, within the CHARISMA, IPERION CH and IPERION HS European programs, at the NewAGLAE facility (C2RMF, Paris).

An overview of the non-invasive methodologies selected and adopted in the protocol for approaching the provenance problem of lapis lazuli will be presented and discussed, together with some examples of case studies involving archaeological artifacts and future prospects.

**Interpretation, presentation and dissemination of the scientific results. Case studies and success stories / 41**

## Unravelling the past of glazed beads using accelerator-based techniques

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A set of glazed beads was discovered in Lisbon during an archaeological survey performed on an ancient vessel. The archaeological interpretations allow a chronological framework in the period between the last quarter of the 18th century and the beginning of the 19th century. Questions arise regarding the provenance of the accounts, as these can provide clues about the route traced by the vessel. Beads have different colours, from pale orange to dark blue/black and five of them (figure 1) were studied by using non-destructive techniques, in order to achieve the chemical and mineralogical composition.

X-ray diffraction results revealed quartz as the main constituent of the analysed beads. Iron oxides and manganese compounds may also occur, particularly on the orangish and dark beads, respectively.

The presence of Si was confirmed by means of PIXE technique, where it was also possible to identify Fe, K, Ca, S and Cl elements in all the beads (figure 2). The beads also emit light of different colours when irradiated with 2MeV proton beam. It is planned to perform ionoluminescence to identify impurities responsible for the light emission and results will be also presented.

**Interpretation, presentation and dissemination of the scientific results. Case studies and success stories / 48**

## Neutron tomography and gamma spectroscopy applied to bronze sculptures: a non-destructive and safe analysis

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**Co-authors:** Ineke Joosten ; Marlies Goorden ; Sara Creange ; Lambert van Eijck

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Neutron imaging is one of the advanced tomography techniques similar to X-ray scanning with an outstanding ability to visualize the internal structure, in a non-destructive way. Contrary to X-rays, which interact with the electrons of the atom, neutrons dominantly interact with the nucleus of the atom since neutrons are electrically neutral. Therefore, neutrons have a remarkable penetrating ability for metallic materials, while neutrons are also quite sensitive to light elements which have higher interaction probabilities with neutrons than X-ray. Neutron imaging, although only developed since approximately two decades, has emerged as one of the most powerful neutron technologies, complementing X-ray scanning, notably in the research fields of material science and cultural heritages.

Since a few years, Rijksmuseum Amsterdam, the national Heritage Agency (RCE) and NPM2, a group of TU Delft, are collaborating in a project called “Beeldvorming”. In this project, we combine neutron tomography (using the FISH neutron imaging facility located in Reactor Institute of Delft) with gamma spectroscopy, to inspect the interior of heritage objects, including information not only on the internal geometric structure but on the elemental compositions as well. In addition, this technology allows visualization of the corrosion state and the internal porosity, from which the preservation strategies can be optimized and the fabrication technique can be inferred. In the “Beeldvorming” project, we focus mainly on bronze objects, such as bronze statues, tools, decorations, etc.

Here we will present our research on the bronze statue of Kuvera (11.5 cm x 6.6 cm x 5.9 cm x 488 gr, AK-MAK-311, Rijksmuseum, Amsterdam, Netherlands), see Fig. 1. It represents the god of prosperity Kuvera or Jambhala within Hindu and Buddhism culture; the statue is probably dated to the 9th or 10th century in Java, Indonesia[1]. The hollow pedestal is partially filled with lead, cast after the fabrication of the statues and a portion of a yellow foil can be observed sticking out a bit underneath the lead.

We will demonstrate how neutron tomography allows us to reveal such precious “mantra’s”, hidden inside the lead, and how we can identify those objects and composition of the solid cast bronze, non-invasively. The information that we collect in this way serves to gain insight into the craftsmanship and culture of the Chola dynasty and, at the same time, assist curators and conservators to make decisions on such museum collection objects, and strategies in preservation, conservation and restoration.

[1] <http://hdl.handle.net/10934/RM0001.COLLECT.1979>

**Interpretation, presentation and dissemination of the scientific results. Case studies and success stories / 45**

## Neutron imaging - an alternative tool for the study of cultural heritage objects

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Neutron imaging is a non-destructive testing method working along similar principles as the more common X-ray imaging methods. Due to different interaction behaviour with matter it provides different and partially complementary results. Contrary to X-rays, neutrons can penetrate many

metals and heavy elements relatively easy, while they show at the same time high sensitivity for some light elements such as hydrogen. This complementarity makes it a very valuable tool for investigations of metallic or metal containing cultural heritage objects.

This presentation will give an overview of the variety of cultural heritage related topics, which can be investigated using neutron imaging techniques. We will show up the method's possibilities and limitations by presenting examples of investigations carried out at the neutron imaging facilities of the Paul Scherrer Institut (PSI), Villigen (CH). These exemplary studies comprise a broad variety of topics such as the virtual unfolding of ancient Norwegian lead amulets or the study of swords from medieval time.

The presented examples should make potential users aware of the possibilities of neutron imaging methods and encourage them to get in touch with the responsible people at the large scale facilities.

**Interpretation, presentation and dissemination of the scientific results. Case studies and success stories / 18**

## **Thin layers characterization with negative muons: a case study of gilded bronzes**

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Muonic X-ray Emission Spectroscopy ( $\mu$ XES) is a rather novel technique based upon the interaction of a negative muon beam with matter. This process leads to the production of high energy x-rays (from  $\sim 20$  keV of Lithium up to  $\sim 6500$  keV of Uranium), that are characteristic of the emitting atom and can be used for elemental characterization. Over the last few decades, the technique has been applied for a wide range of studies, with special attention to cultural heritage artefacts [1], [2]. Thanks to the multi-elemental range, a negligible self-absorption effect of the x-rays and very low residual activity left in the sample after irradiation,  $\mu$ XES has proved to be a very powerful probe for material characterization. In this work we report preliminary results of the analysis on two gilded surfaces. Along with the common elemental analysis, the technique offers the possibility of investigating the variation of the layers within a sample. By varying the energy (momentum) of the incident muon beam it is possible to perform depth profile studies. Here, the thickness of the gold layers is addressed by using Monte Carlo based simulations software [3]. The investigated sample were two small pieces of a copper alloy with a gilded surface, made as a replica of the gates of the Baptistery in Florence. The gilding was made with the mercury "amalgam" technique, and the gold layer was supposed to have a thickness between 10 to 20 microns. During the experiment, a momentum scan was performed by varying the incident muon beam energy. Each of the resulting x-ray spectra was then analysed with a data analysis software and the characteristic peaks of the different elements present in the sample were fitted with a gaussian function. Then, the normalised area values were plotted against the momentum to obtain a profile describing the variation of the elements as the penetration depth of the beam increased. The profiles, in this case for gold, were then compared with the output of the simulations, and, as reported in the figure below, a remarkably good agreement was reached. This non-destructive approach to the study of a material and the consequent data analysis shows the potential of the technique. It can be applied to many different types of samples, like the one coming from the Baptistery gate in Florence (that have been recently analysed) or freshly excavated samples coming from archaeological sites, that can be analysed without removing patinas or corrosion layers.

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## Comparison of ATR-FTIR and O-PTIR techniques for characterization of a degraded oil paint cross-section

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ATR-FTIR (Attenuated Total Reflection Fourier-Transform InfraRed) spectroscopy is widely used to characterize the complex composition of layers in cross-sections extracted from painted works of art. However, some limitations, such as poor contact of ATR crystal with sample or sample area of interest below the IR diffraction limit, prevail.

Recently, a novel O-PTIR (Optical- Photothermal InfraRed) spectromicroscopy claims to open a new avenue for non-destructive, efficient and reliable analysis at sub-micron resolution without the need to touch the sample. O-PTIR produces transmission-like FTIR spectra for interpretation.

This work reports a comparison of O-PTIR and ATR-FTIR techniques applied to a thin cross-section (about 15 µm thickness) delaminated from a late 19th - early 20th century "Portrait of Mr Tan Beng Wan", a zinc white oil-based painting belonging to the National Heritage Board, which depicts a crucial Peranakan figure in the shipping industry of early Singapore.

Zinc soap (palmitate / stearate) is a common degradation product in zinc white oil paints, of which the crystalline form is known to cause paint delamination and flaking problem. In the ageing of oil paints, fatty acids derived from the hydrolysis of oil components are mobile and tend to move through layers and form aggregates. It was postulated that the haze developed on the painting's surface was brought about by the migration of zinc soaps from the ground to the paint layers, which subsequently interact with atmospheric pollutants to form the haze (gordaite in this sample). Zinc lactate, another degradation product that is less reported in oil paints, is soluble in water and its detection suggests for a water-sensitive paint that can lead to implications in conservation treatments. During O-PTIR analysis, various degradation products that have formed in the cross-section, including zinc palmitate / stearate (both crystalline and amorphous), zinc lactate and gordaite ( $\text{NaZn}_4\text{Cl}(\text{OH})_6\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ), were identified with better signal to noise ratio and resolved at the sub-micron resolution. These compounds, though detected with ATR-FTIR imaging, could not be well resolved. Knowing the spatial locations of the degradation compounds characterized at the haze-paint interface is beneficial in understanding the paint's condition and behaviour during conservation.

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## Synchrotron-based techniques to study calcite crystals covering ornated cave walls

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Paleolithic ornated cave walls can be affected by the development of a calcitic coating called moonmilk, that can lead to conservation issues when located at the vicinity of painted or engraved areas. Moonmilk is made of calcite needles called NFC (Needle Fiber Calcite) around one micron large and tens to hundreds of microns long and presenting different morphologies. It is still being discussed whether a purely physico-chemical or a biological process is involved regarding the formation and growth mechanisms of moonmilk. In this study, moonmilk samples coming from different non ornated caves located in the Vézère Valley (Dordogne), and representative of the ornated caves of this area part of the UNESCO World Heritage List (Lacanette *et al.* 2013), were analyzed at the SOLEIL synchrotron light source (Université Paris-Saclay). The objective was to look for potential tracers of the growth mechanisms involved, both from a chemical and crystallographic point of view. Experiments were carried out on three different beamlines combining X-ray diffraction analyses on single crystals (PROXIMA2 beamline), micro-spectrofluorimetry analyses to assess the presence of organic matter (DISCO beamline) and X-ray fluorescence mapping with a resolution of 150 × 150 nm in order to determine local chemical variations (Nanoscopium beamline). This work presents a synthesis of the results obtained, giving new insights regarding the discussion on the origin of moonmilk.

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## Archaeometallurgical Study for a Set of Ancient Coins Excavated from Mediterranean Sea North of Egypt using Synchrotron Radiation Based XRD.

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### Introduction

Archaeometallurgy is an important field of study which allows us to assess the preservation state and value of ancient metal artifacts and better understand the ancient cultures that made them. Metallic artifacts, bronze in particular have specific characteristics that differs them to the other classes of materials. Electrochemical transformations that occurs to them mainly cause a process called corrosion to takes place that starting at the surface of the object changing the metals in to oxides, sulphates, carbonates or other compounds that cause a detectable physical degradation characterized by the presence of corrosion pits and cracks and result in substantial modifications in their morphology

or the total mineralization. Therefore, scientific investigation of such ancient metal artifacts is necessary. One important requirement of analytical techniques is that they be non-destructive methods, since many of these artifacts are unique and irreplaceable. Thus, synchrotron radiation (SR) techniques highly recommended for such studies in last two decades. Therefore, We could achieve valuable information only using Synchrotron Radiation which is mandatory and it would guarantee the signal to noise ratio and the lateral resolution needed for such studies.

#### **Abstract**

The major goal of our research project was to study a set of 8 ancient bronze coins that excavated from the Egyptian Mediterranean Sea, Eastern Port of Alexandria in particular and are dated back to the Ptolemaic era. The study served to evaluate their recent conservation states as well as the effects of marine environment on their degree of alteration and helped to set the suitable strategy to preserve those coins using synchrotron based X- ray diffraction and X- ray fluorescence techniques. The measurements were performed in order to study their present chemical structure, corrosion phases and their respective crystallinity and grain size, in both the bulk metal and the surface/corrosion layers.

In addition, the ancient manufacturing methods in mining revealed too by examining the depth profile of the corrosion products and the metallic bulk core using the diffraction patterns behavior from the outer corrosion layers to the bulk metal where in cooling rates the diffraction phases changed along various depths. The effect of marine environment on the deterioration aspects of the coins have been studied too. Due to the manufacturing of ancient metallic coins in a quite primitive manner; they are inhomogeneous on a micrometer scale, containing remains of imperfect smelting and inclusions. Therefore, studying their elemental compositions and ratios (trace and minor element presence) could be used as a specific fingerprint to know the sources of the ore materials which have been used in manufacturing coins and could provide important clues about the metal provenance, leading to conclusions regarding the commercial, cultural and religious exchanges between the old populations and their way of life. Synchrotron radiation based X- ray fluorescence measurements have been carried out in order to verify this objective.

**These measurements have been carried out at Elettra Synchrotron under ICTP - Elettra User Program**

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## **Eswatini's Shift Towards Sustainable Cultural and Natural Heritage Management**

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Eswatini's rich natural and cultural heritage have received little attention from researchers over the years until in the late 1960 when excavations began in the Old Ngwenya Iron-ore Mine. Although more than 100 archeological sites have been identified through work done by researchers like Beaumont which demonstrated deep history of human occupancy spanning as far as the Early Stone Age (ESA), little had been done to sustainably manage such rich cultural and natural heritage sites [1]. This is despite the fact that the country is known for its rich culture in the Southern African region. The current paper identifies sustainable public policy gaps in the administration of archeological sites amid a growing economy that exerts enormous pressure on archeological sites. As a middle income country, the main goal is economic development which when coupled with lack of knowledge on the importance and benefits of natural and cultural heritage has led to the distraction of valuable heritage sites including their information. This highlight the need for a sustainable and comprehensive cultural and natural heritage management plan in line with the National Trust Commission Act of 1972 as amended which provides the foundation for heritage management in the country. The comprehensive plan covers key aspects of cultural heritage management including identification of heritage sites, proclamation, management (preservation and conservation), public awareness and community engagement, financing and marketing. This enables eco-tourism development, community-based tourism and nature-based tourism across the spectrum of art centers, museums and monuments. Key success factors for the implementation of the plan include a clear

science-policy interface, strengthening of cultural and natural heritage management institutions, budget allocation and the implementation of sustainable management practices.

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## **Accelerator IBA Techniques for the study of ancient pottery from some Ghanaian archaeological sites**

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Ghana has a very rich cultural heritage comprising of several monuments, artefacts, historic and archaeological sites which can be studied, restored and developed for several purposes including Heritage preservation, education as well as Archaeo-tourism.

However, lack of scientific data in this regard tends to encourage unrestrained speculations, which largely accounts for some insupportable hypotheses put forward by many pioneer scholars concerning the nature of culture change in Ghana.

The aim of this work seeks to bring out the viability and potentialities of using integrated and simultaneous IBA techniques to investigate potshards recovered from some Archaeological sites to showcase the contributions that Accelerator IBA applications can be made in cultural heritage studies and Archaeo-Tourism development in Ghana.

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## **The application of accelerator-based analytical techniques to assess heavy metal contamination from legacy ASM and historic formal mine sites to produce contaminant status map and provide remediation options and for ASM environmental management in Ghana**

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Artisanal and small-scale mining (ASM) in Ghana is a time-tested poverty driven livelihood activity that predates formal mechanised mining. Currently ASM contributes, on the average 35% of total gold output of Ghana. The sector's gold output outstrips the combined production of the top three of the nine large scale mines in Ghana. In the last decade, however, ASM's economic significance has been overshadowed by its devastating environmental impacts including widespread destruction and pollution of critical water bodies and farmlands, posing existential threat to populations in rural Ghana. Recent upsurge of highly mechanised illegal mining involving foreigners has exasperated the environmental crisis. Vast areas of legacy or ancient mining sites are currently being aggressively reworked by ASM and large-scale mining companies spurred by the buoying gold prices. These ancient mine sites and mine spoil constitute reservoirs of mercury which was the reagent of choice for gold processing by ASM practitioners and formal miners at the time. These sites constantly supply

toxic heavy metals to the surrounding environment. There has, unfortunately, not been any systematic study to assess the extent of Hg and other heavy metal contamination in mining areas in Ghana, both historic and present, apart from isolated patches of studies targeting gold and a few associated heavy metals. Two exploratory studies focused on ASM sites and historic large scale mining sites include Akabzaa and Yidana (2012) and Ahmed et al (2004). The first assessed spatial distribution and sources of anthropogenic mercury and other heavy metal contamination in the Ankobra River Basin using cold vapour Atomic Fluorescence Spectrometry (CV-AAS) while the second studied a set of rock and soil samples from Dome Beposo in the Ashanti Region, suspected to contain gold. The media were analyzed using instrumental neutron activation analysis (INAA) coupled with conventional counting techniques for gold, arsenic, mercury, and antimony. These and other exploratory studies of spatial distribution of historic mine sites have shown high concentration of these contaminants in rocks, sediments, soils, and water bodies proximal to these sites. Given the obvious advantages of the use of accelerator-based ion beam analytical techniques, quick, highly sensitive and accurate with low detection limit, this paper explores the potential for the application these techniques to conduct broader assessment of the spatial distribution of the widespread historic mine sites across the country and their toxic potential. The results could provide guide for the design of comprehensive remediation measures, reclamation options and general ASM environmental management. The research will commence with examination of satellite images of landforms across the gold belts of Ghana to identify ASM workings and legacy mine sites, followed by systematic sampling of these sites, and analysis of various environmental media, rocks, sediments, soils, historic mine tailings and other mine spoil using accelerator-based analytical techniques to provide a contaminant status map. The design of the suggested assessment campaign will be guided by the results of the stated limited works.

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## **Radiation processing for cultural heritage preservation – Tunisian experience**

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Tunisian National Centre for Nuclear Science and Technology-CNSTN (Tunisia) has two irradiation facilities : a 10MeV 5kW LINAC electron beam accelerator (since 2010) and Cobalt-60 gamma irradiator de 100 kCi (since 1999) used for radiation sterilization of medical devices, food irradiation and preservation of art objects.

Since ionizing radiation has been considered an efficient technique for decontamination of biodeteriorated cultural heritage, wooden and textile items as well as mummified animals, from national museums, are irradiated by the CNSTN's facilities.

This paper presents some examples of cultural heritage treated by irradiation in Tunisia.

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## **Characterisation of clay figurines from Koma Land of Ghana**

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The clay statuettes left behind by settlers in the Mamprugu Moagduri District of northern Ghana over 1000 years ago raise more questions than they answer. The folks who created the figurines are unknown.

Some have ascribed the development of the various mounds of buried figurines to Komaland being the point of gathering of slaves who brought these sculptures from their homes, while others have related it to trade with the other towns north of ancient Ghana.

This ongoing collaborative research project on characterization of some ancient clay figurines from Yikpabongo, in Koma Land of Ghana seeks, among other things, to answer the question of “who manufactured these figurines?”. Different analytical methods, including ion beam analysis approach, are being employed for the study of the figurines.

Chemical signatures of soil samples, primarily clay samples, dug from Koma Land, where the majority of Ghana’s figurines were discovered, are compared to those of the figurines with the aim of establishing their provenance.

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## **Characterization of Mineral Constituents of Archaeologically Recovered Ancient Pottery from Dixcove, Ghana using Ion Beam Analysis**

**Author:** Fritz Biveridge<sup>1</sup>

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**Abstract**

Dixcove is settled by the Ahanta ethnic group and is located about 42 kilometers west of Takoradi, the Western Regional Capital in Ghana. It was one of the earliest states on the Guinea Coast to have encountered and commercially interacted with Europeans during the early phase of the Atlantic contact era (circa, 1485 – 1620). The main objective of the study was to establish the principal mineral constituents of the over 55,000 potsherds recovered from archaeological excavations using Ion Beam Analysis (IBA), to enable identification of the source areas of the clays used to make them and possibly the potters who made them. The study revealed that the bulk of the potsherds ( $\geq 90\%$ ) contained minerals not unique to Dixcove and its immediate environs but from neighbourhoods approximately 6 - 11 kilometers north of it, suggesting they may have been procured via exchanges. The study also indicated that they were produced by different potter groups from those areas.

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## **Endangerments surrounding the tangible heritage in Sudan and the desired solutions through modern technologies**

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The tangible heritage in Sudan is exposed to a number of problems that have strongly affected its sustainability. There are many sites that have been lost and destroyed due to these problems surrounding this heritage. These problems can be summarized in some points:

The biggest threat of tangible heritage is the natural disasters. In Sudan, they represented in floods

and dredger torrents during the rainy season. There are a number of archaeological sites have influenced by strong waves of floods, which affected them greatly and created a moisture that would be affected in the long term. Hence, there is a necessity to use modern technologies to confront such a danger after treating their current situation by stopping the effect of the humidity.

The second big problem facing some archaeological sites in Sudan is the height of groundwater that became an obstacle even during the excavations and study. Here, the importance of using modern technologies is embodied in finding radical solutions to making strong barriers that could be able to stop the water leakage.

The third danger is the desertification that has swallowed a number of large archaeological sites. It is necessary to show the ingenuity of science and scientists in confronting this great hazard, because there are some sites would be ingested absolutely.

There are human problems that led to the destruction of several archaeological sites, they represented in development projects such as dams, agricultural schemes, residential projects, in addition to classic gold mining, and this last danger is the major catastrophe that would be led to the disappearing of the archaeological sites. Because of the lack of adequate oversight, we need an authentic smart monitoring system that plays a strong role in protecting these archaeological sites.

Finally, we can say that we need tight technical systems that can stand up to this rapid deterioration of the tangible heritage in Sudan.

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## **Employing technology in the protection of archaeological sites in Marib Governorate, Republic of Yemen**

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**Abstract.**

Marib Governorate is one of the Yemeni governorates that contains many archaeological sites, including the ancient city of Marib, the capital of the ancient Kingdom of Saba, whose historical roots go back to the beginning of the first millennium BC. There are other sites, including the Awam Temple, which is the most important ancient temple in the ancient Arabian Peninsula, and the Baran Temple, as well as the ancient Marib Dam, which is the oldest ancient irrigation facility that the Kingdom of Saba relied on in the formation of its civilization in the cultivation of the ancient Marib Oasis, which depended on regulated irrigation by the ancient Marib Dam.

The study aims to employ modern technology in the protection of archaeological sites in terms of the natural factors that alternate with the four seasons of the year, and thus it is possible to protect the archaeological sites by using modern devices to monitor the extent to which the building materials of these sites are affected, and work to remedy and treat them in appropriate ways based on the impact.

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## **Application of Electron Beam Accelerator Technology for Treatment of Bio-threatened Cellulosic Artefacts**

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As organic materials, artefacts like papyri are attacked by biological threats from different organisms specially fungi and mold. Insects have been also known to damage rolled papyri. Recently preservation of artefacts could be achieved by controlling biological damage, insect eradication and disinfection in different heritage materials using ionizing irradiation treatments. However, these technologies suffer from some limits as total dose and dose rate which can influence the efficient

elimination of bio-burden, with an increase in deterioration and changes in the material during radiation treatments.

The purpose of this study is to assess influence of the irradiation dose rate on organisms cause infection to cellulose as well as on bio-threatened cellulosic material changes, i.e. degradation. The possibility of using the electron beam accelerator for treating the heritages, especially artefacts made from cellulosic materials was discussed. One of the major tasks in the electron beam radiation conservation of cellulosic cultural heritage artefacts is to reduce material deterioration caused by exposure to ionizing radiation. Electron beam is the advantage of high dose rate which in practice can reduce material deterioration processes. Papyrus paper was selected as example of natural occurring materials from which artefacts were made. The samples were exposed to electron beam irradiation using a 3 MeV linear electron accelerator. The influence of electron beam irradiation on cellulosic -based objects was evaluated in order to determine safe effective dose for cellulosic materials. To avoid over-exposure, a wide range of doses from 3 kGy up to 20 kGy were studied and many analytical techniques were used to determine possible changes of mechanical, chemical and physical properties of treated cellulosic -based objects. The effect of irradiation on post-irradiation effects and appropriate irradiation procedures for wider use of the technique was evaluated. A comparative study on the effect of gamma, and e-beam, on papyri properties was also investigated. The results revealed that the Electron beam is the advantage of high dose rate which in practice can reduce material deterioration processes if compared with gamma rays. Electron beam in doses not more than 15 kGy does not cause significant deterioration in the mechanical properties of cellulosic material. The radicals trapped in EB- irradiated papyrus papers were decayed after short time.

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## The effect of electron beam radiation on simulated fabrics used as a funeral shroud in Antiquity

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With the development of radiation techniques, attempts were made to use both gamma radiation and electron beam radiation for the disinfection of various types of historical objects that were inhabited by microorganisms (e.g., fungi and bacteria) or insects, which ended up causing the objects' destruction. Natural fibres of plant and animal origin constitute the material of many valuable antique objects, works of art, and ethnographic exhibits. The use of ionizing radiation can be an effective and useful method for the disinfection of these objects. However, one should bear in mind that ionizing radiation has a destructive effect on many natural polymeric materials and may lead to deterioration in their mechanical, physical, and chemical properties. In this work Scoured, unbleached fabric was used as a simulation for the studied ancient shroud. The fabric was aged by heating at 100 °C for 72 h. The textile fabric was cut into 10 × 3 cm (length × width) warp test specimens. The experimental samples were infected with the selected species within the used media through a direct incubation period for 20 days in order to be infected with the species that may affect the ancient shroud. The effect of radiation on the mechanical properties and surface structure of woven fabrics made of fibres was investigated. It was found that small radiation doses (up to 15 kGy), sufficient for effective disinfection of textiles, caused only insignificant changes in the fibre parameters tested. However, it was found that doses increased to 50 kGy bring about a considerable weakening of the tested fabrics. Surface morphology of treated and untreated samples was investigated using a scanning electron microscope. Infrared spectra of irradiated samples before and after ageing by heat method were recorded. The results showed that there were slightly changes in the IR spectra of all irradiated samples compared with spectra of untreated sample. ESR studies showed that the radical formed by radiation decayed after short time and as the radiation dose increases, the amount of radical increases. In conclusion it is possible to use the electron beam for disinfection and preservation of texture used as funeral shroud in ancient Egypt with slight effects on their properties

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## Opening word TC Asia-Pacific

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### Ion Beam analysis of Phoenician silver coins

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In this paper we will present the elemental analysis results on silver coins and artefacts using Ion Beam Analysis. We will explore the challenges of applying non-destructive methods on the surfaces of archaeological metals. We will compare three methods of analysis, namely PIXE, SEM-EDS and XRF and present how we resolved problems of surface alterations. The results obtained have a great impact on the archaeological records and we will explore how these contributed to our knowledge on ancient coin systems.

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### Characterization of pottery at Oc Eo, Vietnam by multivariate statistical and neutron activation analysis

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Neutron activation analysis was used to evaluate the composition of 98 pottery samples excavated from the Oc Eo site in An Giang province. The possibility of provenance and technology of ancient pottery was investigated using Agglomerative Hierarchical Clustering (AHC) and Principal Component Analysis (PCA) in combination with archaeological data. The AHC dendrogram identified two primary groups: local and imported pottery. Local potteries are classified into two subgroups based on their compositions: fine-grained pottery and coarse-grained pottery. The fine-grained potteries are mostly made from fine clay, and some are made from fine clay mixed with fine sand while coarse-grained potteries are made mainly of fine clay mixed with coarse sand. Imported potteries were classified into three sub-groups including Chinese pottery (Han, Tang, Song Dynasty), Indian pottery, and unknown sources. The results of PCA analysis show the difference in composition between the elements in the pottery samples. For the rare earth elements: China > Oc Eo > unknown sources > India. For the major elements: India > unknown sources > Oc Eo > China.

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### Application of low energy X-rays for conservation and restoration of Nguyen dynasty woodblocks in Vietnam



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The woodblock of the Nguyen dynasty of Vietnam is one of the World Documentary Heritage recognized by UNESCO. Fungi and termites play a considerable role in the deterioration of these woodblocks. Both chemical and physical methods have been developed for treatment and restoration, and their results are not as expected. Based on the merit of low-energy X-rays, such as their low shielding requirements, cost-effectiveness, easy transport to storage places like museums and libraries, as well as the demand for reducing the negative effect of fungi and understanding the inner deterioration of woodblocks from the restorer; low-energy X-rays seem appropriate for conserving treasured wooden cultural heritage objects. In this study, the effects of low-energy X-rays irradiation were investigated for disinfection of fungi-contaminated woodblock and inspection of the internal damage level of woodblock by termites.

Decontamination of woodblocks by X-ray irradiation was studied at 160 kV, 18.6 mA (3 kW). Fungi were isolated from these woodblocks, and *Cladosporium* sp. was identified as the most radiation-resistant strain in the woodblock. The dose rates of the 1-mm-aluminum filter that was used to cut the low energy part of the X-rays and non-filtered X-rays at the woodblock's surface were 1.14 and 4.64 kGy/h, respectively. At the woodblock middle (8.5 mm from the surface), the doses of the filtered and non-filtered X-rays decreased to 76% and 20% of the surface doses, respectively. The concentration of the fungi in the middle decreased by more than 4 log fractions at 6.2 kGy, and the fungi were eliminated with a surface dose 8.3 kGy. Furthermore, the contaminated fungi in the woodblock were disinfected by both-side irradiation with filtered X-rays delivering a dose of 10 kGy at a dose uniformity of 1.04. The low-energy X-rays showed their effectiveness in disinfecting fungi, and a cut-off filter (1-mm-aluminum) could be recommended to avoid high surface doses of the X-rays.

X-Ray Radiography can provide morphological and physical information on the inner structure of woodblock. X-ray Computed Tomography (CT) was used for the whole 3D inspection of a woodblock. The results 3D obtained were very satisfactory, but it took a long time and was expensive. Whereas X-ray images from the surface can detect the hole and tunnels within the woodblocks caused by termites. Therefore, in this study, the simple method, Radiographic testing for the woodblock in the two directions with an X-ray generator (MG165: COMMET group) at 50kV and 4mA, was carried out to detect the deterioration inside the woodblocks by termites. Based on this data, adequate conservation and restoration procedures will be proposed.

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## Scientific approaches in characterization of Ayutthaya gold and gemstones artifacts

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More recently, ancient gold sites have been discovered in many European countries and are not limited to other parts of the world. The earliest gold artifacts were dated to approximately 4,600 BP. Apart from date determination; a number of ancient gold artifacts were analyzed using available technology to address variety of questions. In general, the best practice on ancient artifact analysis involves delicate and complicated strategies that need strong collaboration between scientists, archaeologists, and curators. Like elsewhere, due to the precious appearance of gold, some original artifacts are disappeared beforehand of authority body. Therefore, the quantity of registered gold artifacts is only a small part of them. Besides, the scientific approach such as nuclear and radiation techniques are difficult to consider as an excellent tool for cultural heritage study.

In Thailand and Southeast Asia countries are well recognized in the past as "Suvarnabhumi" which its

meaning is “the golden land”. Unfortunately, the scientific approach on ancient gold artifacts in Thailand is rare. Due to MOU between Thailand Institute of Nuclear Technology (TINT) and Fine Arts Department (FAD) signed in 2013, a number of research projects have been conducted continuously. TINT provides available analytical techniques for cultural heritage study including conventional radiocarbon dating, TL/OSL dating, elemental analysis by NAA, XRF, and ICPMS, XRD, autoradiography, radiography, tomography with X-ray, gamma ray and neutron. Majority of research projects emphasize on the establishment of elemental composition database of tangible Thai cultural heritage. The dataset is then integrated with other relevant information such as historical records and oral traditions. The prioritized artifacts in the past couple of years include ancient gold ornaments at Chao Sam Phraya National Museum in Ayutthaya province, bronze sculptures from different locations displayed at Bangkok National Museum and temples throughout Thailand. Nowadays, TINT provides consultation on authentication of valuable objects such as gold artifacts, Buddha sculptures, paintings, stone inscriptions, and glass beads.

The analysis of ancient gold ornament project was initiated under fair agreements between TINT and FAD in 2016. Until now, total of 389 gold ornaments which 73% are samples from Wat Ratchaburana located in Ayutthaya historical park, a UNESCO World Heritage Site, were analyzed onsite by portable XRF. Besides, gemstone and decoration materials were studied in complementary with Raman spectrometry, polariscope and microscope by gemstone experts at TINT laboratory. The results indicate unique character of gold ornaments manufactured 600 years ago. The majority of Ayutthaya gold artifacts made of 75-95% of Au and the remaining elements are silver, copper, tin, lead and iron. Some interesting elements such as iridium may be used to indicate imported raw materials. This study employed a principal component analysis software (PCA) to interpret the elemental profiles of ancient gold samples. The linear correlation between gold and silver concentrations suggests electrum of gold that probably used in Ayutthaya gold manufacturing. The results reveal variety of gemstones decorated on gold ornaments and can be further studied to understand provenance of those cultural artifacts as well as the inter connection among the regions.

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## PIXE Contribution for an Elemental Composition Database of Phoenician Pottery from Kharayeb Site

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The study the ancient Phoenician cult place of Kharayeb, in the rural hinterland of Tyre, southern of Lebanon, dated to the Iron Age and Hellenistic periods is particularly helpful in evaluating the complexity and variability of the so called “Hellenism” and of “Greek cultural influences” in the Phoenician world. PIXE analytical technique was used to characterize the elemental composition of several artefacts from this archaeological site, mainly figurines and some artisanal objects, as well as from two another archaeological sites, Jemjim and Tyre which is the prestigious city of antiquity, located at the eastern Mediterranean coast. Furthermore, the resulted PIXE information helped to understand if the figurines were locally produced or imported from the coast and how was the process of production connected to the sanctuary.

The elemental composition of the archeological ceramics was determined by using proton beam of 3 MeV and 13  $\mu\text{C}$  of integrated charge on the samples placed in a conventional in-vacuum PIXE chamber of the 1.7 MV tandem Pelletron accelerator of the Lebanese Atomic Energy Commission. Thus, it was possible to determine more than 20 elements in one spectrum (i) Na, Mg, Al, Si, S, P, Ca, K, Ti, Mn and Fe as major and minor elements, and (ii) V, Cr, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Nb, Ba and Pb as trace elements. Then, a hierarchical cluster analysis was performed, using a multivariate statistical software code, to group together objects having comparable elemental compositions, using the concentration of the the most abundant chemical elements determined by PIXE (12 to 15 elements). The resulted data showed a compositional similarity between the samples of the Hellenistic period with the samples from Tyre, the coastal site. In fact, Tyre is a historical site, known for its long time prosperity as an important center of ceramics production and maritime trade for several centuries (IV B.C. till XIII A.D.). This suggests that most of the figurine samples from Kharayeb are produced in Tyre and the other samples could be imported from other areas in the country. However, the main characteristic of most of the samples is the unusual high calcium content which characterizes the geological area surrounding the 3 archeological sites (Tyre, Jemjim and Kharayeb). Finally, the existing database on excavated pottery form coastal sites in Lebanon (mainly Roman and Byzantine)

is now consolidated with Phoenician artefacts.

PIXE analyses with multivariate statistical methods were used to compare and to reveal characteristic groups. PIXE technique contributed to establish a first database of chemical composition of Phoenician pottery, which will enlarge the existing database that is currently only related to archaeological pottery from coastal sites in Lebanon. Therefore, this work will be used for any future archaeological study on Phoenician pottery in the Phoenician colonies in the Mediterranean basin, highlighting the trade routes at that time.

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## The Use of Nuclear Techniques for the Analysis of Archaeological Artefacts from The Early Bronze Age (3rd mill BC) Dahwa Site in Oman

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Dahwa is an Early Bronze Age (EBA) (ca. 2600-2000BC) site located in the northern Al-Batinah region in Northern Oman. It has since 2014 been excavated by the Archaeology Department at Sultan Qaboos University, Oman. Five relatively large sites concentrated in one area around the modern village of Dahwa were uncovered, marked as DH1 and DH5-DH8. Two of these sites have been subject to excavations which exposed a settlement with well-preserved buildings including domestic structures, large building/warehouse and a small ritual building. Different types of artefacts, including metal and pottery, were found during the excavations in which the most important is a pair of circular copper-alloy cymbals with a diameter of 13.8cm. Analysis of two C14 charcoal samples collected from the building suggest a date falling in the third quarter of the third millennium BC.

This study discusses the results of radiation techniques employed for the analysis of artefacts from the site, including XRF, XRD, X-ray, CT-scan, Isotope, GPR and C14.

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## Accelerator Development Plan to Support National Research and Innovation Program

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Indonesia's Nuclear Energy Agency (BATAN) was established in 1958 in accordance with Law No. 10/1997 regarding Nuclear Energy with the main duties in conducting research, development and utilization of nuclear energy base on safety for peace and welfare. Thus, BATAN is a key player in research and innovation related to nuclear energy which covers five main areas as shown in Figure 1, that area 1) Energy, 2) Industry, 3) Health, 4) Food and Agriculture, and 5) Environment. To achieve the goals, BATAN has been operating various nuclear facilities which are spread out in four nuclear area, that are Research Reactors, Irradiator, as well as Accelerator. The most important achievements in each field are, the research reactor's fuel element which has been and is being used in G.A. Siwabessy Research Reactor, various mutation plant breeding which is spread nationwide, various radio pharmaceutical, and the development of accelerators. As long as Indonesia became member

state in IAEA, these achievements are inseparable from the support of the Agency (IAEA) through various projects (Technical Cooperation). Drawing on its national widely acknowledged expertise, BATAN actively participates in collaborative projects with a large number of other government institution, academic and industrial partners. Collaborative Projects with Ministry of Education and culture, BATAN have been involved in archeological activities especially in artefacts dating and elements analysis using nuclear techniques.

On April 2021, the New Agency called National Research and Innovation Agency (BRIN) was established by Presidential Regulation No. 78. This agency is formed by integrating all of previous Research and Development Agencies and from all of Research and Development Department in Ministry. Consequently, BATAN was also integrated in this Agency. The main objectives of this integration are, 1) to improve efficiency and effectiveness of resources, and 2) to create critical mass in Research and Innovation in Indonesia. As shown in Figure 2, BRIN's organization structure covers all research field, including science and technology and called as "Research Organization". According to this structure, collaboration between Research Organization could go effectively.

As a new agency, BRIN established some program to create critical mass on Research Innovation by Developing an Advance Research Infrastructure and Human Resources. In Infrastructure Development, some Nuclear Facilities are consider to be built, especially accelerator. The development of accelerator is considering by building advance accelerator and conducting research and innovation in order to acquire accelerator technology. In Human Resources Development, Study Assignment Program in Graduate degree, apprenticeship, training by providing scholarship and through IAEA projects. Besides that, BRIN launched post graduate program by inviting doctoral graduate (national and foreigner), for conducting research and innovation in BRIN. To accelerate global research achievement, BRIN also launched Invited Professor Program for conducting research and innovation in BRIN in collaboration with its researchers.

As BRIN Policy's that all of research and facilities are open platform, thus all of nuclear facilities are being planned could be used by another researcher within BRIN or external (National or International). Therefore, the various nuclear techniques could contribute and support in Heritage Management intensively.

Poster session / 5

## Identifying key players in Heritage management in Bangladesh: Strategies for Capacity Buildup

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Bangladesh has a rich and diversified legacy, a heritage to be proud of. With its rich history, heritage tourism in Bangladesh should have been booming. As reported by UNESCO 2020, Bangladesh has three (3) heritage sites inscribed on the World Heritage List, five (5) properties on the Tentative World Heritage List, as well as more than 509 heritage sites across the country that are under protection of the Department of Archaeology under the Ministry of Cultural Affairs. In order to establish a comprehensive understanding on the heritage management in Bangladesh, the baseline situation must be understood at first. A desk-based study on the type, number, and distribution of heritage resources, as well as additional data acquisition methods such as surface surveys, archaeological samplings, built condition assessments, local interviews, community-based cultural mappings, or consultations with relevant academics, experts, practitioners, communities, and other stakeholders need to be undertaken. Moreover, the documentation of vulnerabilities e.g., (a) deterioration of archived paper materials, built fabric, (b) agricultural disturbance of archaeological site, (c) Intense visitation pressures, (d) modern intrusions in historic areas, or (e) environmental degradation/erosion etc. will provide strategic guidelines in Heritage conservation. This report compiles the state of the art information on heritage management in Bangladesh and attempts to identify the key players involved. Recommendations for establishing linkages among the players to adopt nuclear approaches in safeguarding and protecting heritage sites of Bangladesh; have also been discussed.

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## The future of accelerator technology application for archaeology in Indonesia

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### History

In 1978, R.P. Soejono led the National Centre for Archaeological Research (Puslit Arkenas) to open laboratory of Paleocology and Radiometry (Palrad) in Bandung. The founding of Palrad has marked the first usage of nuclear technology for archaeological researches in Indonesia. The research activities were ended on July 1, 1992 when their function was transformed into Bandung Archaeological Center.

After the disbanding of Palrad, the National Center for Archaeological Research established cooperation with the Nuclear Research Centre of Yogyakarta (BATAN). They have conducted various studies of radiocarbon dating and elemental analysis on various artifacts, both with X-Ray analysis and neutron activation analysis using the Kartini 250 kW research reactor, in Yogyakarta.

### Recent progress

After the political changes of 1998, most nuclear-based archaeological research was carried out in cooperation with institutions abroad. Carbon dating and stable isotope analysis have been done in the Waikato University, National Museum of Natural History Japan, and Arizona University. Recently uranium series dating was also carried out in collaboration with MNHN France and Griffith University, while luminescence dating was cooperated with Naturalis Biodiversity the Netherland. In addition, research based on X-Ray microtomography was also active in collaboration with AST-RX UMS 2700 CNRS (France) and CENIEH, Burgos (Spain).

### The future

The reorganizing of all research institutions in Indonesia has brought the Puslit Arkenas under the umbrella of National Research and Innovation Agency (NRIA), named as Research Organization of Archaeology, Language, and Literature (RO ALL). NRIA has promoted many collaboration opportunities, and currently RO ALL is planning strategic steps with Research Organization of Nuclear Energy to develop the implementation of accelerator technology for archaeological research and heritage conservation.

- Accelerator Mass Spectrometry

AMS dating laboratory is very important in archaeological research, as they can cover the range of events from hundreds of years up to three million years ago. Laboratories equipped with the AMS method has a very sensitive detection quality, able to detect using a very small sample quantity (less destructive), and the analysis process is very fast (minutes to hours).

- Uranium series laboratory

The U/Th laboratory can analyse dating samples such as fossils, speleothems and other carbonate materials. The laboratory can also perform elemental detection and quantification of material samples, determination of elemental composition in artefact and paleontological raw materials, palaeodietary investigations through Strontium analysis, also traces of human migration by identification on marker elements.

- Archaeo-Chemico and Mineralogical Laboratory

The main purpose of this laboratory is for the characterization of materials, based on the study of their chemical composition and mineralogy, as well as their vibrational and thermal analysis. This type of laboratory is supporting research in geology, archaeology, heritage, chemistry and pharmaceuticals, construction, civil engineering, and nanomaterials.

- Luminescence laboratory

Luminescence dating is based on the ability of quartz and feldspar minerals for determining the age of sites, the age of rock formations and sedimentation environments, determination of sedimentation

rates and their continuity, measurement of Beta and Gamma dose rates in sediments, as well as of Gamma's natural dose level at archaeological site.

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## LAEC investment in the use of IBA techniques to valorize cultural heritage

Author: Mohamad Roumie<sup>1</sup>

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Since 1990, large scale campaigns of archeological excavations were undertaken in Lebanon, showing the richness and diversity of the Lebanese archaeological patrimony. Due to the geographical and historical place of Lebanon, the related findings revealed unearthed vestiges dating up to thousands of years, as old as the prehistoric age, and belonging to many ancient civilizations: Phoenician, Babylonian, Assyrian, Hellenistic, Roman, Persian, Byzantine, Omayyad, Abbasside, Mamluk and Ottoman.

Nuclear analytical techniques, accelerators based, have proven to be an effective tools for the studies of archeological artifacts, in particular for provenance, authenticity, fabrication techniques and dating. As early as its installation, the LAEC particle accelerator invested in this direction. IBA techniques such as PIXE and RBS contributed to characterize ceramics, figurines, coins and other artefacts from different excavated archaeological sites. This was expressed by many collaborative projects with archeologists where IBA techniques contributed to solve some of the issues related to the characterization of workshop production and authenticity. In this work, relevant case studies will be highlighted as to show the usefulness and capabilities of accelerator based techniques. Furthermore, many technical updates of the accelerator machine were undertaken, such as the new external micro-beam, as to accommodate more advanced analysis of archeological objects.

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## Low energy S- Band Linear Accelerator(s) for Radiography and Cargo Scanning as a tool for Cultural Heritage Management

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The design & development of low energy electron LINAC(s) for non-destructive radiography and cargo scanning applications is reported. The industrial radiography and cargo scanning play a vital role in cultural heritage management and preservation with potentials of non-destructive testing, identification and restoration of works of art and other monuments of cultural heritage for a country having rich cultural sites and commodities like Pakistan.

In the first phase of development, a 6 MeV standing wave side coupled,  $\pi/2$  mode LINAC has been designed and fabricated and converted to a Non-destructive testing (NDT) Radiography system which will be discussed in detail with the focus on characteristics that dictate the ability to resolve small artefacts in objects including cultural art and other monuments. These X-ray imaging characteristics are also required for Cargo scanner and will be discussed. The indigenously developed accelerator-based cargo scanners can fulfil the country's need of safe trade and control any illegal movement of cultural goods. Pakistan's accelerator program not only focuses on industrial application but medical and food irradiations as well. An in-house accelerator test facility has been established, which comprises of 10 MW klystron powered by solid state modulator and the compatible RF transmission system. The research and development on various aspects of accelerator technology including

electron gun, HV pulsed modulators, accelerator cavity design & manufacturing, e- beam dynamics and diagnostics, X-rays target, RF conditioning, accelerator operation and control systems will be presented. The progress on transformation of the developed accelerators into radiography, medical, cargo scanning and food irradiation systems shall also be presented.

The characterization of radiography LINAC will be discussed in detail including the development of exposure chart of the machine. The effective energy is estimated through half value layer method and beam quality index is measured by Tissue Phantom Ratio (TPR<sub>20/10</sub>). Other radiography characterizations include penetration measurement as a check of energy of x-rays, spatial resolution and contrast sensitivity measurement. Simulations in Monte-Carlo based software GEANT4 have been carried out to design and develop collimators and filters for radiography, cargo and medical applications and will also be presented.

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## **Opening word TC Europe**

**TC Europe / 50**

## **Analysis of gold-leaf glass tesserae using micro-PIXE/PIGE**

**Author:** Polytimi Loukopoulou<sup>1</sup>

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The use of gold-leaf glass tesserae begun in roman times along with the development of wall mosaics and established during the byzantine period. Gold-leaf tessera is a unique type of glass tessera due to its sophisticated manufacture technique - a metal leaf is enclosed between two layers of normally transparent glass. Published data on gold-leaf glass tesserae are limited and mainly focused on the composition of the glass and gold.

The research aimed to establish a non-destructive investigation of nature and decay of gold-leaf glass tesserae. Systematic examination of a large number of tesserae was followed by analysis of selected tesserae. Tesserae were analysed as received, without sample preparation, using Scanning Electron Microscopy coupled with Energy Dispersive x-ray Spectroscopy (SEM/EDX) and supplementary micro-Proton Induced X-ray and  $\gamma$ -ray Emission Spectrometry ( $\mu$ -PIXE/ PIGE).  $\mu$ -PIXE/PIGE analysis was carried out using the scanning nuclear microprobe installed at the 5 MV Van de Graaff electrostatic accelerator of the Institute of Nuclear Research of the Hungarian Academy of Sciences (ATOMKI-HAS) in Debrecen, Hungary, via the Financial support by the Transnational Access to Research Infrastructures activity in the 7th Framework Programme of the European Union (CHARISMA Grant Agreement no. 228330).

In the context of this presentation results of  $\mu$ -PIXE/ PIGE analysis of gold-leaf tesserae would be provided, demonstrating the efficiency of the selected methodology.

**TC Europe / 28**

## **Study and restoration of icon "Dormition of the Mother of God"**

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This presentation focuses on restorative intervention and non-destructive diagnostic analyses conducted in two icons part of the Medieval Art Pavilion at the National Historical Museum in Tirana. The recognition of the materials used in their realization by carrying out these diagnostic tests helped us to discover their author. The two icons are the "Dormition of the Mother of God" (dated 1762) and the other icon "St. Mary with Christ" (dated 1701) are taken by the iconostas of the church of St. Mary's Monastery, located near the village of Pojan of Fieri district on the territory of the ancient city of Apollonia. In collaboration with the Institute of Nuclear Physics of Albania, Prof. Nikolla Civici, were performed several analyses. These tests were carried out with ED-XRF show that some colors vary and some others do not. For example, the red color used in the 1762 repainting of the icon "Dormition of the Mother of God" has more Mercury (Hg) composition and less Lead (Pb) while the red used in the icon "St. Mary with Christ" in 1701 has more Lead (Pb) composition and less Mercury (Hg) composition. So do brown, yellow and white pigments, while green, blue and okra pigments result in the same.

The icon "Dormition of the Mother of God", was radiographed part-by-part with X-rays through 15 radiographic plates Kodak, which served for the coverage of its entire surface. From the study of these plates were recorded the areas of wood with knots, the part where the two pieces of the icon join, with two crosses fixed with nails, as well as their shape and position. Also, the surface of the wire used under the grunt, the state of the preparatory layer, as well as the cracks and wood degradations of wood insects were also accurate. Quite interesting was the recording of the initial layer of the painting, with the preparatory discs, as well as the highlighting of two parallelogram-shaped connectors in the interior of the wood. From this diagnosis we understood the way in which the author made the ascent and reinforcement of the two parts of the wood.

The types of metal, earth, vernique and organic binding oxides were examined by ED-XRF analysis. The archeometric measurements targeted the characterization of pigments used in the icon, during which measuring systems are optimized for detecting elements with average atomic numbers (Ca-Zr). During these measurements the instrument worked with high x-tube voltage of 30 kV, current 20 µA. Spectral analyses of the preparatory layer for the verification of stratigraphic layers observed that the first layer is wires related to the support of wood with organic animal connectors. After this layer lies an organic connecting layer and the preparatory layer of the gipsy grund and after this layer come gradually the layers of colors.

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## E-beam simulation for Cultural Heritage cellulosic materials preservation at IRASM

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Romania has an experience of over 20 years of Cultural Heritage preservation by gamma irradiation treatment at IRASM Radiation Processing Centre, Department of IFIN-HH. The cultural heritage artefacts include archives, books, documents, wood artefacts (furniture from the Theodor Aman Museum in Bucharest, orthodox churches), wood icons, and other artefacts made from natural biopolymeric materials. IRASM IFIN-HH has Conservation Treatment and Investigation Laboratory Licenses issued by Romanian Ministry of Culture since 2014.

The field of radiation preservation of cultural heritage artefacts is well established within the European Region, important contributions being made by France (ARC-NUCLEART, Grenoble), Romania (IRASM IFIN-HH), Poland (ICHTJ), Croatia (Ruđer Bošković Institute), Hungary (Institute of Isotopes), Italy, Portugal, Serbia and others.

The use of accelerators for Cultural Heritage preservation and disinfestation was done successfully in Poland by ICHTJ for wood and fabric. In Romania there are two old research LINAC facilities



at INFLPR. In 2015 was made a Feasibility study for an “Electron Accelerator for Radiation Technology Applications” at IRASM IFIN-HH. This work aims to simulate the best irradiation conditions for treatment of Cultural Heritage cellulosic materials, using a compact e-beam.

Monte Carlo Simulations were made with RAYXPRT, ver 1.8.5 (TRAD Tests & Radiations).

Starting from an e-beam irradiation room model, made after a feasibility study of an “Inhouse-inline” irradiator (MEVEX, Canada, 2015), the matrix dose mappings resulted from different irradiation geometries simulating conveyor movement were merged. The simulation was made for a stack of books L x l x h 42 cm x 45 cm x 9 cm and a maximum target absorbed dose of 10 kGy, obtained with 5 MeV electrons and 1 mA beam current. Usual conveyor speeds (cm/s) and 2 passes (one irradiation on top and one on the bottom) were used for studying the dose distribution for the selected artefact model. The simulation results showed the total irradiation time at around minutes per stack of books, resulting in a productivity of a few cubic meters per day, which is comparable with the productivity of existing SVST Co60 existing industrial irradiator. The maximum dose rate on the surface of the experimental model was found to be on the order of magnitude of MGy/h.

The findings are encouraging for the large volume treatment of book collections, archives and paper cultural heritage items.

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## Contribution to the Roman glass study: analyses performed by PIXE/PIGE, INAA and XRF

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As with ceramics and metallurgy, glass production is a high-temperature technology invented by ancient civilizations. The material quality and manner of manufacture of glass artifacts provide insight into the technological and social progress of the societies who developed them.

Despite the abundance of Roman glass discoveries in Bulgaria, only three archaeometric studies have been undertaken to date. They were conducted between 1999 and 2021 and mostly involve samples from southern Bulgaria and the country’s Black Sea coast. Different instrumental methods were employed to determine the elemental composition: INAA for Roman glass from Odessos (modern-day Varna, Northeastern Bulgaria)(1), PIXE/PIGE for Southeastern Bulgaria samples(2), and XRF for Southwestern Bulgaria samples examined in 2021. The IAEA financed the examination of the largest collection of specimens from Southeast Bulgaria in 2010, which was conducted using PIXE/PIGE technique at “Jožef Stefan” Institute in Ljubljana (Slovenia).

Glass is a supercooled liquid (amorphous solid solution) of alkaline silicate containing sodium or potassium ions, with a divalent alkaline earth metal serving as the curing agent. Throughout the Roman Empire, the primary raw materials for glass manufacture came from natural sources of quartz sand, soda (trona), and limestone. Later, natural soda and limestone were phased out in favor of plant ash as a flux since it contains potassium and magnesium ions. Numerous mineral additions containing iron, manganese, antimony, and copper have been added to the product to color or clear it. Thus, the results of the elemental composition analyses of Bulgarian Roman glass can be summarized as follows:

1. During the Roman Empire, glass production was based on the sodium-calcium-(aluminum)-silicon type of glass.
2. The majority (almost 77 percent) of glass samples analyzed exhibit traits common to European Roman glass, such as the use of antimony and manganese as decolorizing agents and an increased level of impurities later in production. The increased concentrations of oxides of certain basic elements (Al, Mg) in some samples, as well as the observed scattering, indicate a change in raw material sources around the end of the third and beginning of the fourth centuries AD, and/or the expansion of glass production with new production centers, which in practice means less controlled production.
3. The majority of the glass objects discovered at Southwestern Bulgaria were made according to the so-called Middle Eastern formula, but with different raw materials and in a different location than the glass artifacts from Southeastern and Northeastern Bulgaria from the same period.

4. To gain a better understanding of the origins of the Bulgarian Roman glass, it is important to undertake future analyses on a larger number of glass finds from Bulgaria and to compare the results to data from likely raw material sources.

(1) Kuleff, I., R. Djingova (1999) Archaeometric investigation of Roman glass finds from Bulgaria. *Berliner Beiträge zur Archäometrie* 16, 183-198

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## Development of Latvia Cultural Heritage research. Recent trends and impact achieved.

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Application for a poster

Latvian Cultural Heritage is definitely not so recognizable and still remains mostly undiscovered for the rest of the world. Only some of our most well known painters, like Mark Rothko or Vija Celmins, who exhibited in the MoMa (LA, USA) and many other places, as well as our archaeological collection is more or less known to the World's public. But, despite this, we have a remarkable number of unique objects and monuments that characterize our nation, culture and history. The conservation and preservation of Latvian Cultural Heritage starts 90 years ago, when National History Museum of Latvia started conservation and research of historical objects. However, serious research on the cultural heritage materials started only in the eighties of the 20th century, when National History Museum of Latvia founded the Department of Research and Imitation. Until the department received more advanced equipment, investigation process of the historical objects was based on the analytical chemistry and historical prejudices and views on the composition of materials.

With the development of technologies and new opportunities, it was possible to supply the laboratory with Raman spectrometer InVia (Renishaw) and portable  $\mu$ -XRF spectrometer ARTAX (Bruker Nano GmbH), as well as Leica microscopes, which allowed us to study historical objects much more detailed.

As a result of this we were able to change some of the prejudices of cultural heritage professionals about the materials used in cultural heritage objects. For example, the long-held belief that all grey archaeological metal objects are made of tin has proved to be misleading. XRF spectrometry provided us more detailed information about the compositions of archaeological items and lead to interesting, new and unexpected discoveries about the composition of the objects. At the same time the Raman spectroscopy, equipped with a confocal microscope, has made it possible to identify traces of colour layers in objects that were not considered polychrome at all. Detailed and deeper view into historical objects opened a new level of possibilities to see that Latvia cultural heritage has been much richer, colourful and more interesting than we have thought for a long time.

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## Opening word TC Latin-America

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## **Applications of X-ray fluorescence in the research of the Chilean archaeological material culture. Case studies of the National Center for Conservation and Restoration.**

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Conservation Science has emerged as a scientific discipline widely applied to the research of Cultural Heritage, which has a direct impact on the work of conservators and restorers, both in the understanding and documentation of cultural goods, as well as supporting the decisions about conservation, museography, and collection management of Cultural Heritage.

On the other hand, due to the great social relevance that defines Cultural Heritage, its study directly impacts the assessment of cultural goods by their communities and the society, leading to the necessity reconfigure the working teams towards an interdisciplinary and multifactorial form.

In the studies usually carried out at the “Centro Nacional de Conservación y Restauración - CNCR” (National Center for Conservation and Restoration), the use of portable X-ray fluorescence (p-XRF) takes on a significant value because it is a non-destructive mobile technique that allows the preliminary characterization of the material composition of cultural goods at an elementary level. In addition, it allows the direct analysis of areas of interest in objects of various formats. These aspects permitted the study of four different pigment categories commonly found in the local archaeological material.

The case studies presented correspond to the analysis of Diaguita ceramics belonging to the Semiárid North of Chile. The study’s objective corresponds to the first approach to the characterization of imitative restorative interventions.

The results indicated different elemental composition behavior of the intervened ceramics, which could be associated with imitative actions, both aesthetic and morphological. Additionally, the results showed the presence of common elements for the original and the intervened areas. However, differences were observed between the intervention groups; it was impossible to differentiate them statistically. Nevertheless, it was possible to associate the presence of certain elements exclusively in the intervention groups, such as lead (Pb) and titanium (Ti). Meanwhile the absence of these elements does not necessarily indicate detection of an original pigment in the ceramic.

As a projection of the work, we expect to increase the measurements in a wider ceramic universe to perform chemometrics analyses and be able to recognize a pattern to distinguish between the different imitative actions, and finally associate elemental composition with chronology and places of intervention.

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## **Analytical methodology for the study and diagnosis of the conservation status of archaeological objects**

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In archaeology, materials characterization techniques can provide information about the origin of objects, temporal assignment, manufacturing aspects, use and deterioration processes after discarding.

In the framework of the Project for Conservation and Restoration of the Gaii collection of the Museum of Patagonia (PNNH-APN), we developed a working methodology for the study of cultural assets. This was done by implementing analytical techniques for the characterization of materials with minimum impact on the objects, prioritizing non-destructive studies. Here we present, as a case study, the metallurgy of a silver earring from the colonial period belonging to the aforementioned collection (Fig. 1). The information provided led us to approximate answers to archaeological questions and to move forward in the diagnosis of the conservation status.

All the experiments that we will describe were carried out in different dependencies of the Atomic Energy Commission of the Argentine Republic (CNEA).

These experiments were approached methodologically through interdisciplinary interaction to re-elaborate the archaeological and conservation questions into chemical and morphological fingerprints. Once these were established, it was possible to select the appropriate analytical techniques to be applied.

It is possible to differentiate locally manufactured objects, objects of European origin, and objects that were manufactured with local techniques reusing European raw materials by knowing the composition of the alloy. We applied SEM/EDS technique using the FEI Inspect S50 Scanning Electron Microscope equipped with an energy-dispersive X-ray spectroscopy (EDS) detector. The identified alloy composition, was, on average 75% silver, while also identifying the presence of copper, aluminum, iron and sulfur. The latter is associated with silver degradation products, such as silver sulfide. Particle induced X-ray emission (PIXE) technique in the 1.7 MeV Tandem Accelerator will be applied to determine trace elements that could provide information about the origin of the raw material.

We performed X-ray diffraction measurements in order to identify the alloy phases and the corrosion products and to determine the preferential orientation of the crystalline grains, that is, the crystallographic texture. These measurements were carried out on a PANalytical Empyrean diffractometer with an Eulerian cradle using Cu K $\alpha$  radiation. We observed that the earring possesses a face-centered cubic phase (FCC) with a lattice constant ( $a$ ) of  $\sim 4.064 \text{ \AA}$ , which is close to pure silver ( $4.0860 \text{ \AA}$ ). No other phases were detected. We found that the observed texture components aligns the  $\{110\}$  planes toward the  $\langle 112 \rangle$  direction, this component is called "Brass". Brass components are observed in metals with FCC structure subjected to cold rolling processes (Kocks et al. 1998) (Liu et al. 1998). There is no evidence of texture components associated with annealing/recrystallization processes, giving us insight that the fabrication procedure does not involve temperatures higher than  $150 \text{ }^\circ\text{C}$  (Liu et al. 1998).

Kocks, U. F., C. N. Tomé y H. R. Wenk (1998). Texture and anisotropy: preferred orientations in polycrystals and their effect on materials properties. Reino Unido: Cambridge University Press.

Liu, W. X., Y. Liu, H. Suo y H. J. Bunge (1998). Texture in silver. Materials science forum, 273:503-510. Trans Tech Publications, Ltd.

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## First gamma irradiation in the colombian cultural heritage: the case of the artwork Mutantes of the National Museum of Colombia

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My name is Ana Paula Gómez Uribe a cultural heritage conservator and restorer. I work in the Museum Strengthening Program of the National Museum of Colombia - Ministry of Culture, and I am currently the main counterpart of the ARCAL RLA1019 project in Colombia and I coordinate the collaboration between the country's museums and the services of the Directorate of Nuclear Affairs of the Colombian Geological Service (SGC) to make peaceful and correct use of nuclear techniques in favor of the conservation of the national cultural heritage. An agreement is currently being drawn up between the National Museum of Colombia and the Directorate of Nuclear Affairs of the Colombian Geological Service so that more cultural entities benefit from the application of nuclear techniques for the treatment of their heritage affected by a fair or poor state of conservation.

Last year, within the framework of this project, gamma-ray irradiation was carried out for the first time on an artistic heritage asset of the Nation: the work "Mutantes" by Carlos Rojas, which is a work of contemporary art from the National Museum of Colombia and whose mixed technique with wood presented a biological attack of xylophagous insects. The selection of this artwork to be irradiated was based on the fact that it was chosen by the Museum's curators to be part of the permanent exhibition for room 15. The artwork had to be free of any biological infestation to avoid the risk of contaminating other objects in the exhibition room. Its large format, made up of 13 vertical wooden modules, each approximately 1 meter high, made the conservation area consider methods for treating biological attack that could cover the entire surface of the work. These modules were built by master Rojas from recycled wood that he found on the street and never had a previous preparation treatment. Already part of the Museum's collection, they were temporarily stored in the reserves of the General Archive of the Nation until 2021, when 4 of the 13 modules with an active biological attack by xylophagous insects were inspected and identified.

From the nuclear affairs directorate of the Colombian geological service, radiation with gamma rays of 8 kGy was experimented with, which can reduce microbial load and insects according to the literature. Given that other food or cosmetic products with similar densities and geometries are irradiated in the SGC, routine doses were used. This process was accompanied by a microbiologist who made a micro-culture of the affected area before and after the process. However, due to several factors, the results were not conclusive, but under macroscopic inspection, it was found that the treatment was effective.

The ideal is that this process can be repeated with other heritage objects that present different techniques and materials in small, medium, and large formats and that belong to collections of Colombian museums, as is the case of a large-format tapestry by the Colombian artist Olga de Amaral. of the Museum of Antioquia in the city of Medellin.

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## **Preservation and conservation of information contained in glassy materials from cultural collections with processing by ionizing radiation**

**Authors:** Maria Luiza Nagai<sup>None</sup>; Pablo Antonio Vasquez Salvador<sup>1</sup>

<sup>1</sup> *Nuclear and Energy Research Institute – IPEN/CNEN/SP, Brazil*

Glass-based photographic materials are commonly found in historical cultural heritage collections. Inadequate storage conditions for these materials lead to problems of biodeterioration, mainly by fungi.

Processing by ionizing radiation with electron beam has a biocidal effect to combat fungal contamination. However, a known undesired effect on glassy materials is increased opacity which affects the readability of images on photographic glass negatives.

In this way, the study proposes a methodology to minimize the darkening effects of the glasses that are subjected to irradiation.

For this work, glass samples were subjected to irradiation with electron beams at doses of up to 25 kGy, under different controlled temperature conditions, and the effects analyzed by UV-visible spectrophotometry and colorimetry analysis.

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## Peruvian cultural heritage material characterization, achievements during the last twenty years

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Peru is a country worldwide recognized for its rich cultural tradition and heritage, spanning from monumental architecture as ancient as several thousand years to historical building, objects and documents corresponding to the colony and republican periods (XV to XIX centuries).

In this context archeologists, historians and conservators face a hard work dealing with a large variety of materials, manufacturing techniques and degradation agents that must be identified and characterized to rescue and adequately conserve this invaluable cultural heritage, which includes materials as raw earth, straw, textiles, human and animal mummies, pottery, metals and jewelry, wood, paintings and documents.

Approximately twenty years ago, with the help of IAEA, we started an ambitious plan to develop nuclear analytical techniques to give support to other characterization techniques generally used in the field and in the conservation workspace, giving emphasis to capacity building in students and professionals working in this marvelous field.

In this presentation we summarize the work, achievements and difficulties faced in the last two decades, relevant to the themes proposed to the workshop, and the most outstanding necessities to manage with the huge work to properly characterize and identify materials and techniques used by ancient crafters as well as to conserve our cultural heritage.

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## Moving toward a sustainable conservation – Experience of the Museum of Archeology and Ethnology (MAE-USP)

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Most of the ethnographic collections present in traditional museums in Brazil were formed by collectors, purchases, donations and exchanges by large encyclopedic, naturalist museums, typical of the 19th century. It is in this context, the Museum of Archeology and Ethnology (MAE-USP) collection were constituted.

The MAE has been made a big effort to guarantee their conservation. Preservation of tangible objects as well thousands of organic objects, composed of plant fibers, wood, skins, feathers, seeds and various materials, has always been challenging as they are prone to deterioration by biological agents such as insects, mold, bacteria and rodents.

Chemical treatments are traditionally examples of actions to preserve many museum collections around the world. Since the 19th century, collectors and museum professionals have applied a variety of toxic substances through fumigations and direct applications trying to enhance the conservation.

Although a well-intentioned practice, the application of pesticides to protect ethnographic objects could not predict the negative impact on the safety of those who would handle these objects in the future and by restricting the possibilities of using these collections by the descendants of their creators. Today, these contaminated objects cannot be touched without gloves or experienced by for example, indigenous groups.

The current insertion of native communities in curatorial actions at museums has made it possible to renew the way in which these institutions work. At this moment, it is no longer plausible that a museum institution continues to carry out toxic treatments on funerary, sacred objects, human remains, among others. The possession and use of these objects transcend the museum's borders

and the possibilities of use must be considered in the perspective of the future.

Due to the renewal of the theoretical parameters of the conservation discipline, the Integrated Pest Management policy is more suitable for museological institutions. Efforts to prevent damage have been more effective than just thinking about curative conservation. In addition, the need to develop a more sustainable present and future has led institutions to develop greener prevention policies, without the use of toxic products, respecting the environment and the user.

In this scenario, since 2010, MAE-USP has abolished the use of pesticides to treat the collection. Since then, the institution has been dedicated to building a protocol to reduce risks related to infestations. This protocol encompasses, among other actions, the treatment of objects affected by biological agents and the preventive treatment of new objects through ionizing radiation.

The use of ionizing radiation for the disinfestation of museum objects is a very safe process and has proved to be a great alternative to traditional methods of disinfestation that involve pesticides of high persistence and toxicity. For this reason, we have also worked to disseminate the technique among conservators.

This work intends to share the actions carried out by MAE-USP in partnership with the Nuclear and Energy Research Institute (IPEN) to facilitate the treatment of ethnographic objects, as well as an important collaborator to make the conservation process at MAE more sustainable.

## Poster session / 12

### **Diagnosis and proposal of treatment by gamma rays for documents with fungi in the National Archive of Chile**

**Author:** Natalia Ríos<sup>1</sup>

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The National Archive of Chile is a public institution that safeguards 40 linear kilometers of documents from all over the country, from the year 1541 to the present. Its documents are made on different types of paper depending on the time of production. From the 16th century to the 19th century, it is mainly cotton fiber paper and from the 19th century onwards, it is characterized by different papers of industrial production. Paper and its high cellulose component is an attractive medium for attack by fungi, which degrade the material to the point of destroying it and thereby losing the information it contains.

Because there is not a systematic procedure to determine the state of conservation of the documents (this corresponds to the evaluation of the physical condition and the characteristics of the supports), was developed a diagnostic methodology, using documentary collection of the Real Audiencia and its 3,272 books produced between the 16th and 19th centuries. The methodology used showed that the most worrying risk detected was that of fungi, whose percentage is 5.1% of the total fund, which, although proportionally scarce, threatens the physical disappearance of the documentation.

The methodology proposed in this article and the results obtained based on it allow to identify the physical conditions of the supports and their deterioration, all information has been stored in a database that currently gives the possibility of having controlled the characteristics of each unit of documents individually, a fact that was not known before the application of this methodology.

The technique that conservation uses for the treatment of fungi, consists of applying an alcohol solution on the documents and cleaning with cotton, this technique is slow and expensive, a conservator only manages to disinfect 10 books per month, therefore, apply this method of disinfection in the 5.1% affected, it would take several years. Another solution is the application of ultraviolet light, but due to its photo-damaging characteristic, it is highly invasive for the paper fibers. A proposed solution that appears to be the most suitable for treating fungi on paper is the application of gamma rays, due to its high effectiveness, because it is non-invasive and because it can be applied en masse to a set of objects, which makes it a highly efficient and effective solution for dealing with documents. Providing general solutions that massively satisfy the need for the archive is a great investment.

The diagnostic experience, as well as the gamma ray treatment proposal, is an experience that can be disseminated and replicated in other archives with similar institutional characteristics and in other countries of the region. All the countries of the former Spanish colonies have this same type of documents with similar deterioration. In addition, this technique would allow saving time and prolonging its useful life, to remain as sources of live information for today and the future.

## Poster session / 6

**A sustainable approach to archaeometrical and conservation studies using nuclear techniques****Author:** Ariadna Mendoza Cuevas<sup>1</sup><sup>1</sup> *Colegio Universitario San Geronimo de la Habana, Oficina del Historiador de la ciudad de la Habana, Cuba*

3D modeling and printing technology was sustainably used in the design of the portable X-ray based multi-technique equipment and for the sample holder used for the archaeometric studies performed with such a system, as well as in the design of the multi-level sample holder for irradiation, where other recyclable materials were also used.

Irradiation treatments in a gamma irradiation plant combined with previous exposition to essential oil atmosphere was evaluated as a sustainable treatment due to their economic and environmental advantages over traditional chemical biocide treatments. Viability methods, independent or dependent on culture medium, are experimented in the search for a quantitative, sensitive and sustainable method to evaluate the efficiency of massive antifungal treatments on documentary material. A limited dilution method, applied in a multiple inoculum with a reduced amount of culture medium, is evaluated for the estimation of spore viability. A probabilistic model, developed for this purpose, was programed in a computer software. This viability method, sensible to the individual spore level, shown to be a reproducible. A general methodology is conceived and applied, which includes sampling procedures and efficient extraction of spores by means of mini-probes containing radiosensitive silica glass pearls, designed for reproducible growth of inoculated material, allowing the determination of incubation conditions and the optimal treatment dose. The miniprobes were inoculated with fungal spores, isolated from documentary material, in culture medium and on paper, leather and synthetic binding materials, which made it possible to determine the doses of irradiation that produced inhibition/mitigation, decontamination or sterilization. Gamma irradiation was also used as a sustainable procedure for sterilization of plastic materials (24- or 96-well plates) used in the proposed viability methods. It is necessary to record the doses and dose rates received by the materials in the irradiation treatment of documents and books, for which sensitive dosimeters in the application range are required. A miniprobe with 0.5 mm silica glass beads (recyclable) is proposed as a routine on-site dosimeter. Finally, the proposed dosimeter was found to be sensitive in the useful dose range for document and book irradiation treatments.

Key words: biodeterioration, fungi, viability, preservation, irradiation.

## Poster session / 61

**Irradiation of Cultural Heritage for Conservation: presentation of the Bolivian Multipurpose Irradiation Centre.****Authors:** Danny José Ortiz Condori<sup>1</sup>; Sara Fernandez<sup>2</sup><sup>1</sup> *Agencia Boliviana de Energía Nuclear, Bolivia*<sup>2</sup> *Agencia Boliviana de Energía Nuclear*

The aim of the Bolivian Nuclear Energy Agency in Bolivia is to develop, supply and commercialise nuclear technology goods and services for peaceful purposes, for which it is responsible for the construction and operation of the Nuclear Technology Research and Development Centre - CIDTN. The Centre for Research and Development in Nuclear Technology - CIDTN has the following components: Nuclear research reactor, Cyclotron - Radiopharmacy - Preclinic and Multipurpose Irradiation Centre.

One of the applications of the Multipurpose Irradiation Centre is the conservation of cultural heritage using Gamma Irradiation technology from cobalt-60 sources.

Bolivia has historical, artistic, palaeontological, archaeological, ethnographic, documentary, bibliographical, scientific and technical assets of great value. Bolivia's cultural heritage is distributed



in different repositories in the country, and for its conservation, Bolivian conservators use conventional techniques, delaying the conservation process for several days.

The Multipurpose Irradiation Centre plans to start its operation with the irradiation of cultural heritage for conservation with the following institutions and focal points (end-users) of the RLA 1019 project:

1. The Cultural Foundation of the Central Bank of Bolivia. - Which has in its custody historical documentation considered the country's cultural heritage.
2. The Historical Archive of the Mining Corporation of Bolivia. - It holds important documentation on the protection of the country's mining industry.

Given the importance of the documentation of the Historical Archive for the country and the fact that it is difficult to treat by the preservationists due to the danger of diseases that could be caused by its handling, it is intended to inactivate the fungi and microorganisms present in the documentation by means of irradiation in the Multipurpose Irradiation Centre. After irradiation, the documents will be digitised and kept in appropriate environments for their preservation. The Multipurpose Irradiation Centre, which is already in place, is expected to start operating this year. Given that the technology is new in the country, there is a need for the experience of experts and countries in Latin America with experience in the use of technology in the application of nuclear techniques for the conservation and characterisation of cultural heritage.

One of the areas that needs to be strengthened in the country and which is of great interest to the RLA 1019 focal points, end-users, is the use of nuclear techniques for the characterisation of cultural heritage

#### Poster session / 9

### 3D Printing and Photogrammetry in The Restauration and Conservation for an Historical Artefact

**Authors:** Camilo Blanco Torres<sup>1</sup>; José Vega<sup>1</sup>; Rodrigo Mora<sup>2</sup>

<sup>1</sup> LANOTEC

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Industry 4.0 is a new phase that includes new technologies aiming to solve problems and bring effective solutions to complicated problems in short time. Using tools of this industry like photogrammetry technique and 3D printing as methods for detailed fabrication, it was possible to create a prothesis that helped to the restauration of an artefact found by the National Musseum of Costa Rica. Through an interation of modeling, designing and fabrication a final model for the conservation of an historic piece was created. The process was considered as a success and it was exhibited in a national event by the National Musseum of Costa Rica.

#### Poster session / 10

### Proposal for the application of neutron activation analysis of micro-samples of pigments in archaeological ceramics from the Argentine Northwest

**Author:** Valeria Palamarczuk<sup>1</sup>

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The general objective of this project is to make comparisons between archaeological ceramics of different styles, locations of discovery and chronologies, from the Argentine Northwest. The observation of similarities and differences in the chemical composition of ceramic materials has been useful to investigate the standardization in ceramic production, the identification of sources of raw materials, and the circulation of vessels and materials in space in past times.

Given their status as cultural heritage objects, the characterization of archaeological ceramics requires non-destructive and minimally invasive experimental approaches. Our group has experience in the use of micro-destructive techniques on flakes or powders obtained from fragments of non-museumable archaeological ceramics: SEM coupled with EDS, Raman micro-spectroscopy and micro-XRD. These techniques showed great potential for characterizing slips and paints in red, black and cream tones, formed by heterogeneous compounds, generally with low crystallinity, and the presence of solid solutions. However, it is necessary to use other analytical techniques, such as neutron activation analysis (NAA), to quantify the presence of traces and reliably determine the chemical composition of the paints.

The advantages of this nuclear characterization technique are the use of small amounts of material, the short time necessary to prepare the sample, the possibility of analyzing multiple samples simultaneously and determining, in a single measurement, major and minor elements and traces. Since NAA is a multielemental technique that requires processing a very small mass for its implementation, it has been applied to the study of archaeological ceramic fragments considered non-museumable. In previous works, this technique was used to study archaeological ceramic pastes and clay sources from the Argentine Northwest. However, its utilization to evaluate slips and paints, applied in thin layers (a few microns) on ceramic surfaces, still needs to be developed, since the minimum sample size (usually 2 to 3 g) according to standard sample preparation protocols is not suitable for these cases. Micro-sampling techniques was used and irradiation with high neutron flux was applied to small samples (10 mg), belonging to objects declared as cultural heritage (Landsberger and Yellin, 2018); micrograms of meteorites were also evaluated with this technique (Sekimoto et al., 2016). These antecedents provide an interesting perspective for the study of slips and paintings in the archaeological ceramics of the Argentine Northwest, whose analysis, combining different analytical techniques, we are looking to develop according to a multidisciplinary and collaborative approach (Palamarczuk et al., 2020; Tomasini et al., 2020).

**Detecting intellectual property crime as forgeries and fighting illicit trade of art objects with nuclear techniques / 87**

## **Introduction to the session by the Panel Members**

**Detecting intellectual property crime as forgeries and fighting illicit trade of art objects with nuclear techniques / 88**

## **Panel discussion**

**Access to research infrastructure, and international as well as regional collaborations and networks / 21**

## **The Cultural Heritage Project at ANSTO**

**Author:** Filomena Salvemini<sup>1</sup>

<sup>1</sup> ANSTO, Australia

A strategic scientific research project on Cultural Heritage has been formally endorsed at the Australian Nuclear Science and Technology Organisation (ANSTO) since 2015. The project aims to promote the access to the suite of nuclear methods available across the organisation, and the use of a non-invasive analytical approach in the field of cultural-heritage, archaeology, and conservation science. The latest scientific analytical tools, which are available under the operation of ANSTO, including neutron-, synchrotron- and accelerator-based techniques, have been increasingly demanded for a wide range of applications to heritage materials.

In this paper, the most relevant case studies conducted at ANSTO and undertaken in collaboration with Australian research institutions, universities, and international stakeholders will be showcased.

**Access to research infrastructure, and international as well as regional collaborations and networks / 96**

## **NCRRT as Collaborating Centre in Industrial Radiation Processing and Cultural Heritage Preservation**

**Author:** Hassan Abdelrehim<sup>1</sup>

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The National Centre for Radiation Research and Technology (NCRRT) is one of the four centres affiliated to the Egyptian Atomic Energy Authority (EAEA). It was established in 1972 to enable the nation to exploit the radiation processing technology for peaceful purposes in medical, industrial, environmental, basic sciences, engineering and agricultural fields and to keep up with scientific progress in these fields.

The centre contributes to providing irradiation services to various parties by sterilizing medical products, biological tissues, and preserving food by radiation, in addition to conducting experiments at the semi-industrial level to improve the properties of polymeric materials, textiles, rubber, wood and paints, which improve their economic value added. The irradiation facilities at the NCRRT are Mega Gamma 1 Irradiator (MISR I) at Cairo, Gamma Irradiation Facility (MISR II) at Alexandria, Research Gamma Sources and Electron Beam Accelerator Facility 3 MeV with X-ray unit.

NCRRT participates in several regional activities under AFRA, e.g. providing advice to other AFRA Member states, hosting topical meetings, training courses and workshops. It has been selected as Regional Designated Centre (RDC) in order to play a leading role in AFRA and TC-IAEA projects as well as in the field of Irradiation Processing. NCRRT is a permanent partner in performing inter-comparison exercise in the field of dosimetry and has a Practical Arrangements with IAEA on "Enhancing Technical Cooperation Among Developing Countries (TCDC)". The NCRRT Central Laboratory Facility (NCRRT-CL) provides access to state-of-the-art instrumentation for both research and training purposes. The facility offers scientific analysis, technical advice, tailored hands-on training programs for researchers, faculty staff and students, and graduate students as well as for industry from all over Egypt. The provided instrumentation covers both basic and applied sciences. This core facility is considered one of the pillars to achieve excellent science and as a venue to enhance capacity building at NCRRT, it is expected to contribute directly to the quality and quantity of international publications and collaborative research projects. NCRRT The center was selected as a collaborating center by the IAEA for 4years (2021-2025) in the field of Industrial Radiation Processing and Cultural Heritage Preservation.

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## **The IAEA Collaborating Center Atoms for Heritage at Université Paris-Saclay**

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<sup>5</sup> CEA

<sup>6</sup> CNRS/IRAMAT

The International Atomic Energy Agency has designated Université Paris-Saclay as the first IAEA Atoms for Heritage Collaborating Centre in heritage sciences on Friday 17 September 2021. The new Centre will focus on five key themes linked to heritage preservation: characterising and dating materials; developing safe analysis methods; analysing and sharing data in accordance with Open Science strategies; educating and raising awareness among the public and future generations about heritage issues; and combatting the illicit trafficking of heritage objects.

The Collaborating Centre builds on a unique ecosystem of networks and infrastructure. The Collaborating Centre works closely with three of the University's Graduate Schools (Humanities – Heritage Sciences, Physics and Chemistry), and in coordination with two interdisciplinary programmes (The Interdisciplinary Institute of Materials and Palabre).

Through the Atoms for Heritage Collaborating Centre, the IAEA and Université Paris-Saclay, alongside its partner organisations, aim to play a key role in the development and application of physical, chemical and digital techniques to study and improve the preservation of heritage objects, whether they are cultural artefacts such as monuments and paintings, or natural objects such as fossils.

The support of the IAEA will facilitate the hosting of international scientists, curators and technical personnel who will all come to Paris-Saclay for training, creating opportunities to establish and intensify international research collaborations through the arrival of experts from around the world as part of the University's programmes.

Peaceful applications of nuclear science for development and cooperation

Two years after Université Paris-Saclay co-signed the Paris Declaration on “Heritage, Sciences and Technologies: An Opportunity for our Societies and the Global Economy” at the Institut de France, the launch of the Collaborating Centre will play a key role in the sustainable use of nuclear methods for the benefit of society.

**Access to research infrastructure, and international as well as regional collaborations and networks / 4**

## “Open SESAME for cultural heritage research and beyond”

**Author:** Gihan Kamel<sup>1</sup>

<sup>1</sup> SESAME Light Source, Jordan

During the last two decades, a huge increase in the use of accelerators-based techniques is witnessed in a wide range of scientific applications such as physics, chemistry, biology, geology, environment, materials science, among others. Exhibiting a strong interest in the fields of archaeology and cultural heritage compared to laboratory-based source, they are commonly implemented to shed light on the methodologies of manufacturing, conservation, and/or restoration of different forms of heritage materials and objects. This presentation will highlight the accelerators' importance in heritage studies, with a focus on different examples of related research in the field conducted at the recently operational SESAME synchrotron facility. SESAME is the only synchrotron light facility in the Middle East. It aims at promoting advanced research capabilities and technology within its Members. Current Members are Cyprus, Egypt, Iran, Israel, Jordan, Pakistan, Palestine, and Turkey. A few examples of SESAME Users' first experiments on heritage science such as archaeological remains, historical parchments, and ancient mummies will be demonstrated together with the future opportunities and perceptions in the field of cultural heritage at SESAME.

**Access to research infrastructure, and international as well as regional collaborations and networks / 79**

## Status of the MACHINA project, the Movable Accelerator for Cultural Heritage In-situ Non-destructive Analysis

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**Co-authors:** Massimo Chiari <sup>1</sup>; Lisa Castelli <sup>1</sup>; Caroline Czelusniak <sup>1</sup>; Pier Andrea Mando <sup>1</sup>; Giulia Calzolari <sup>1</sup>; Serge Mathot <sup>3</sup>; Giovanni Anelli <sup>3</sup>; Alexej Grudiev <sup>3</sup>; Alessandra Lombardi <sup>3</sup>; Eric Montesinos <sup>3</sup>; Maurizio Vretenar <sup>3</sup>

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<sup>3</sup> *CERN-European Organization for Nuclear Research*

Over the years, there has been an ever growing interest for in-situ compositional analyses in the field of Cultural Heritage (CH). A good example of this trend is provided by the noticeable increase of studies employing mobile XRF scanners. However, XRF systems have some limitations that do not affect Ion Beam Analysis (IBA) techniques. Unfortunately, at present, IBA analysis are possible only in laboratory, as no transportable accelerator has been developed so far.

To allow for IBA in-situ measurements, the Italian National Institute for Nuclear Physics (INFN) and the European Organization for Nuclear Research (CERN), both with a long and significant experience in the development, use and application of particle accelerators, have jointly started the MACHINA project for the development of a transportable accelerator system. The pillars of such a project are the competencies developed both at INFN-Labec, for out-of-vacuum IBA studies in CH, and at CERN, concerning high-frequency radiofrequency quadrupole cavity (HF-RFQ).

The current status of the MACHINA project after four years from the beginning will be here shortly presented, together with expected future activities.

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## IAEA activities to facilitate access to accelerators

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<sup>1</sup> *International Atomic Energy Agency, Austria*

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Ion beam accelerators and synchrotrons serve as hubs for networking and R&D at national and international level. Being medium to large-scale facilities with significant human and research infrastructure, accelerators serve to conduct fundamental and applied research, as well as to render services contributing to the achievement of several Sustainable Development Goals (SDGs).

The IAEA has supported the development and applications of accelerators for a very broad range of scientific applications, including the characterization and preservation of objects of the tangible cultural heritage. The support is implemented via the Technical Cooperation Programme (TC) and by implementing different activities within the Division of Nuclear Applications in Physics and Chemistry.

While several TC projects in the past have supported the installation of ion beam accelerators as well as to expand their infrastructure by adding new end stations allowing to diversify their use by the international community, especially by researchers from Developing Countries is still limited. One feasible mechanism would be to foster the access to both ion beam accelerators and synchrotrons via the implementation within the TC Projects of Scientific Visits focused onto conducting experiments that could expand the analytical tools used in the project.

This contribution presents the activities implemented in the Physics Section to foster the application of accelerator-based studies and to facilitate the access to accelerator facilities, including:

- Introductory e-learning courses available at the IAEA Open Learning Management System
- Fostering information exchange through the Accelerator Knowledge Portal , the Nuclear Science and Instrumentation Portal and the NSI Newsletter.
- Organizing Technical Meetings and Training Workshops

- Providing Technical Assistance Missions to improve the work of accelerator facilities upon request from the IAEA Member States.
- Conducting Coordinated Research Projects to facilitate the access in the past to Elettra Sincrotrone (G42005) and currently to a network of particle accelerators (G42008).

As part of the CRP G42005 (“Experiments with Synchrotron Radiation for Modern Environmental and Industrial Applications”, 2014-2018) participants from seventeen countries obtained research results of high-quality tackling with challenging interdisciplinary applications, including cultural heritage and preventive conservation.

The ongoing CRP G42008 (“Facilitating Experiments with Ion Beam Accelerators”) relies in the access to 11 ion beam accelerator centres distributed in different areas of the world, and 14 research teams scheduled or already performed their experiments, including one on Archaeology.

Some examples of results obtained within these projects are provided for illustration.

**Access to research infrastructure, and international as well as regional collaborations and networks / 65**

## **The CARNAÚBA beamline of Sirius synchrotron light source - a platform for nanofocus studies**

**Author:** Douglas Galante<sup>1</sup>

<sup>1</sup> *Brazilian Center for Research in Energy and Materials, Brazil*

The CARNAÚBA beamline of the Brazilian synchrotron light source, Sirius, is an X-ray nanoprobe for simultaneous multi-analytical and coherent X-ray imaging techniques, with spectroscopic capabilities in the 2.05 to 15 keV energy range. It includes X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS), X-ray fluorescence (XRF), X-ray excited optical luminescence (XEOL) and ptychographic coherent diffractive imaging (ptycho-CDI). The sample is raster-scanned through the nanoprobe to provide two-dimensional maps, which can then be combined with a rotation for computed tomography. Two end-stations are under development: an all-in-vacuum and cryo genic nanoprobe (SAPOTI) and an environmental in-air nanoprobe (TARUMÁ).

The TARUMÁ station, the first entering in operation, has a large working distance to accommodate more flexible sample environments, and to facilitate alignment and installation of ancillary instruments. Its mechanical design is heavily based on precision engineering concepts and predictive modeling. As an environmental nanoprobe, TARUMÁ will cover a large variety of scientific areas, from cultural heritage, agro-environmental, geophysical, and biological research to energy and other condensed matter-related areas. These areas are tackled using the innovative instrumentation solutions for in situ, operando, cryogenic, and in vivo sample environments developed for this station.

**Access to research infrastructure, and international as well as regional collaborations and networks / 77**

## **Heritage Science Austria. An Austrian national hub of infrastructures and resources for the tangible cultural heritage**

**Author:** Maria Bianca D’Anna<sup>1</sup>

<sup>1</sup> *Austrian Academy of Sciences, Austrian Archaeological Institute, Austria*

Heritage Science is the transdisciplinary scientific research into cultural heritage, which provides a comprehensive access to art, cultural properties, natural assets and intangible heritage through their documentation, interpretation, preservation, and management. Heritage Science infrastructures and expertise are particularly crucial in our understanding, conservation, restauration and management of the tangible cultural heritage.

In this talk we present the Heritage Science Austria initiative, its network of institutions and resources, as well as its efforts in building an Austrian National Node of Heritage Science infrastructures.

**Safe analysis of heritage objects; Best practice and Irradiation Passport for Art / 90**

## **To be announced**

**Safe analysis of heritage objects; Best practice and Irradiation Passport for Art / 95**

## **X-ray Microscale Spectroscopic Characterization in Cultural Heritage – Implications for the Monitoring and Mitigation of Radiation Effects**

**Author:** Samuel M Webb<sup>1</sup>

<sup>1</sup> *SLAC National Accelerator Laboratory*

Synchrotron based techniques are widely used for the analysis of complex materials, in part due to the high brilliance of the x-ray source and the ability to tune the x-ray energy. While these characteristics often provide superior performance over traditional lab-based sources, it may also create problems with respect to the high photon flux and potential modification of the sample, which presents unique challenges to the stakeholders of cultural heritage experiments.

Paintings and paint pigments have been well studied by synchrotron-based techniques. While a wealth of information can be obtained through x-ray analysis, one must also be aware of radiation damage. Of particular interest in this work is the chemistry of S moieties in lapis based ultramarine pigments, and As in emerald green pigments. Both of these pigments have been used extensively throughout history and the elements at the center of their color have shown the ability to undergo beam-induced reactions in many samples of biological and environmental origin.

Results from this work show that the x-ray beam can alter these compounds under some experimental conditions. Calculation of the doses applied and absorbed allows a kinetic analysis of the reactions, allowing an assessment on the acceptable amount of radiation exposure. Further work continues to characterize the reactivity of increasingly complex matrices found in real-life Cultural Heritage materials in order to define optimal acquisition parameters for safer analyses.

**Safe analysis of heritage objects; Best practice and Irradiation Passport for Art / 76**

## **Irradiation Passport for Art (IPA)**

**Author:** Ineke Joosten<sup>1</sup>

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This talk zooms in on a project that has recently been executed in the Netherlands, and that now continues developing in an international context (within a European project, IPERION HS). As is well known, exposure to radiation is of a cumulative nature. This fact is important for everyone involved in cultural heritage research and treatment, as this means that previous exposure of objects or environments may change the sensitivity of objects or research samples in future examinations, or may even alter the material characteristics if certain thresholds are surpassed. This makes it important for the cultural heritage field to record and document the exposure of art objects as well as research samples to irradiation. This idea is the starting point of the project presented in this talk, which introduces what we call the Irradiation Passport for Art (IPA), a database in which can be recorded the location, total exposure and circumstances of use of radiation. With such a passport, the field can work towards safer and more controlled use of radiation for the examination and treatment of cultural heritage. The passport can be used for research into the long-term effects of irradiation of objects of art. The information on earlier exposures is crucial for the selection of future areas of investigation and prevents double exposure. Finally, when a restoration treatment is planned it is of importance to know if and where an object was irradiated. The IPA project is led by conservation scientists, a physical chemist and a conservator and has the support of an international team with different irradiation expertise, in which important synchrotron-institutes and research institutes involved in research in cultural heritage are represented. The IPA database has been developed as a password-protected website that institutes can register for, which holds information on the exposure, all the relevant parameters that were used during the measurement and information on changes if they occurred. We hope that putting the information in the website will become part of the regular documentation process. The website is meant to be used by conservation scientists and conservators. It will not hold the actual data, this stays with the partner institutes that have uploaded the parameters. Partners can contact each other if they wish to have detailed information and would like to request examination results to be shared.

**Open Science and dissemination of knowledge / 73**

## **Beyond data FAIRification: converting New AGLAE data into digital heritage objects**

**Author:** Claire Pacheco<sup>1</sup>

<sup>1</sup> *C2RMF and New AGLAE, Palais du Louvre, France*

On April 15th 2019, Notre-Dame de Paris was burning. The glass-stained windows have fortunately been preserved but were polluted by lead. These colorful iconic objects are deposited now and some of them will be studied in 2022 at the New AGLAE facility. Once restored, the precious artifacts will go back to the cathedral for centuries, hardly accessible for further physico-chemical analysis.

Integrating and sharing scientific data within the frameworks, norms and processes of Open Science should be considered as the alpha and the omega of any study of Cultural Heritage entity. As a matter of fact, if the property of a Cultural Heritage object is not always institutional, its cultural and spiritual dimension is intrinsically universal and belongs to anyone. According to the UNESCO World Heritage Convention dating from 1972 and now signed by 194 states, it is the duty [of France] of ensuring the identification, protection, conservation, presentation and transmission to future generations of the cultural heritage.

So, how should we consider the IBA data acquired on such specific targets? Are they part of the cultural heritage object which will then be digitally augmented? Are they part of a digital twin of the piece of art?

Hence, the mission of the New AGLAE team is to preserve and transfer the IBA data sets acquired on precious objects made of stones, glass, ceramics, metals, etc. and dating from Paleolithic to 21st c. to future generations as a piece of digital cultural heritage, starting by making the data respect the FAIR principles (Findable, Accessible, Interoperable, Reusable). The FAIR process is optimized along the IBA data life cycle, from the application for New AGLAE beam time to the perennial storage of data sets and the publication of data, through their processing and current storage.

Following this logic and also in order to improve the various uses of IBA data within the communities built around the New AGLAE, IBA for Cultural Heritage and more widely Heritage Science, the Euphrosyne project was conceived. Euphrosyne is not only Aglae's sister in mythology, but it is also the name of the digital platform that is being developed with the Digital Workshop of the French Ministry of Culture to make the New AGLAE data FAIR.

Euphrosyne first version was deployed in March 2022 and enables New AGLAE users first to prepare



their experimental run which is an essential step for high quality measurements and the FAIRification of the future dataset. The digital tool also permits to safely reach their data. The team is now working on the next version of Euphrosyne, which should present more functions such as giving remote access to some New AGLAE processing software. Finally, the digital tool should enable interrogating, accessing and sharing IBA data respecting the FAIR principles. The issues, methodology and challenges will be presented as well as the progress status of the Euphrosyne platform and a potential demonstration of the digital platform.

**Open Science and dissemination of knowledge / 25**

## **Infrared characterisation of cultural heritage objects: standard approach and going beyond diffraction limit**

**Author:** Krzysztof Banas<sup>1</sup>

**Co-author:** Agnieszka Banas<sup>2</sup>

<sup>1</sup> *Singapore Synchrotron Light Source, Singapore*

<sup>2</sup> *Singapore Synchrotron Light Source*

Infrared spectroscopy has been used for analysis of the numerous objects that may be attributed to cultural heritage. Verifying the authenticity of documents, paintings, or musical instruments are just a few examples of this type of analysis. Another vital aspect is evaluating the status of the cultural heritage artefacts, namely checking how environmental conditions of the storage or display influence the safety and integrity of the objects. This issue is critical in the case of paintings, as various ingredients used in the creation process (in canvass, ground, paint, varnish and other layers) may go into interaction and produce potentially irreversible changes.

Innovative techniques based on photothermal effect were recently added to the well-established in laboratories methods as FTIR (Fourier Transform Infrared) spectroscopy and microscopy. They allow to break the diffraction limit of the classic infrared spectroscopy, improve the measurements' spatial resolution, and access the information unavailable before.

In contemporary science, the key to understanding obtained experimental results is proper data processing and evaluation regardless of method that was selected for the samples characterization.

In this contribution detailed data analysis of huge hyperspectral objects collected during experiments (FTIR and novel photothermal effect based spectroscopies) on cultural heritage samples will be presented. Special attention will be paid to proper pre-processing (baseline correction, experimental artifacts removal, normalization) and the selection of adequate statistical methods. Any spectroscopic results of the experiments (spectra and hyperspectral objects) are highly correlated data-sets with many redundant variables. In order to visualise the results and extract the essential information, dimension reduction methods are required. To find the similarities and differences between spectra it is necessary to evaluate the spectral distance and to cluster the results into groups.

All examples presented here are prepared in open-source R Environment and are freely available at the Github repository. R is recommended by many data scientists as a versatile platform for spectral data evaluation and it is available for all modern operating systems. It promotes code-driven analysis that enables reproducible and repeatable research.

**How to make science tangible – Public awareness of how science and heritage work together in a tangible way – novel methods in education and outreach / 16**

## **Scientific analysis of artworks as an ally for teaching science in current times**

**Author:** Annette Ortiz Miranda<sup>1</sup>

<sup>1</sup> *National Gallery of Denmark*

In ancient times, it was common for artists to do chemistry tasks to prepare pigments, solvents, varnishes, and even their tools. Nowadays it is highly unlikely for an artist to prepare his own materials, although the relationship between art and science remains obvious for them. During the last decades, science has made its way into art through fields such as conservation and it was precisely through this that it found its ally as a diagnostic tool, since the chemical industry is largely responsible of the evolution of artistic materials used today. However, the contribution of science to art is not known in the same way worldwide, as, for example, in regions of the Caribbean. The international scientific and technology evolution have given rise to new fields in the sciences that have generated greater interest and have left the pure sciences aside, which is why teachers and professors look for more motivating teaching methods. The concerns for better education and interest in scientific fields is an international issue. One of the goals is to make science courses more interesting for students. In some cases, the relationship between chemistry, physics and art has been used as an interdisciplinary field of study, highlighting the use of this scientific career as a diagnostic tool. This paper shows how the scientific analysis of artworks by great artists worldwide was used as headline in dissemination, education and mentoring programs. Proving that the relationship between these two fields, especially with painting, can be an efficient and motivational teaching method for scientific careers.

**How to make science tangible – Public awareness of how science and heritage work together in a tangible way – novel methods in education and outreach / 43**

## **Human Skeleton Conservation at Choeng-Ek Genocidal Center in Phnom Penh, Cambodia**

**Author:** Sovannarith Ry<sup>1</sup>

<sup>1</sup> *Ministry of Culture and Fine Arts, Cambodia*

The Choeng-Ek Genocidal Center in Phnom Penh, Cambodia, is one of the 19,733 mass grave sites that have been located (DC-Cam 2004). Choeng Ek was originally a Chinese burial site where Chinese tombs still exist. It was used by the Khmer Rouge as a “killing field” and mass burial site between 1975 and 1979. The site is located about 17 kilometres (11 mi) south of Phnom Penh, Cambodia. Many of the dead were former political prisoners who were kept by the Khmer Rouge in their Tuol Sleng detention center. Exhumations took place in 1980s, with 89 mass graves disinterred of the estimated 129 graves in the vicinity. The remains of 8,985 individuals were exhumed and are now housed within a memorial stupa (Buddhist shrine).

In 2013, the Ministry of Culture and Fine Arts (MoCFA), Municipality of Phnom Penh and Choeng-Ek Genocidal Center proposed to the government for preserving all skeletal remains. In result, a Laboratory of Conservation of Human Skeleton to be installed at Choeng-Ek. The laboratory aims were to preserve any remaining evidences from the recent conflict after four decades.

There are 3 types condition of skeleton decay on this preservation project. The skeleton selected on basis of 40% to 50%, 25% to 40%, and 15% to 25% preservation. It means that the badly preserved bones are consolidated first, then the next preserved, then the best of the preservation. The process of conservation, first of all the bones moved from Stupa and created new Choeng Ek Identification number. So each cranium had a plastic tag with its new “Choeng Ek project number” attached. And the next preservation started with measurement, violent trauma assessment after the bones taken to the Laboratory. This step an “Inventory List” was made for each cranium and everything on cranium were recorded such as the color of bone, the mud on the bone, and the trauma etc. After that the cleaning and consolidation started. The bones were cleaned by spraying with reverse osmosis (purified) water in a pressure spray and consolidating mixture by Acryloid B-72 and Acryloid B-48 mixed with acetone, with added Don Thorn Phong and Ponal chemicals. The last step it was documentation work, the inventory sheets transferred to the Access Database. The information on the Inventory sheet was entered into the Access database for that Choeng Ek Identification number and data sheets were printed as inventory book. Access Database is backed up in 2 ways, one in CD and two in external Hard-drive back-up.

This is the first time in the history of the exhumation of the Choeng Ek gravesites in Cambodia; scientific program of data collection was completed on the group of human remains in the Memorial Stupa. It is a significant achievement and is an example of the capacity of Khmer researchers. The results of this work indicate that 6426 crania and 63,112 other skeletal elements including long bones, vertebrae, pelvis, were consolidated.

**Application of accelerator science and technology for characterization and treatment of heritage objects / 75****Accelerators, X-rays and cultural heritage: combining micro and macro level investigations with different purposes in mind****Author:** Koen Janssens<sup>1</sup>**Co-author:** Collaborators<sup>2</sup><sup>1</sup> *AXIS Research group, NANOlaboratory Centre of Excellence, University of Antwerp, Belgium*<sup>2</sup> *AXIS Research group, NANOlaboratory Centre of Excellence, University of Antwerp*

Cultural heritage artefacts such as paintings, stained glass windows and (illuminated) manuscripts are examples of complex macroscopic objects consisting of a multitude of different materials, in close proximity to or in intimate contact with one another. Given sufficient time and a number of external stimuli (such as impinging UV and visual light, variable relative levels of humidity or of reactive volatile species from the ambient atmosphere) these react with one another and form secondary products. When such spontaneous chemical reactions negatively influence the mechanical properties of the artefact at the macro-level or significantly change one of its relevant properties (color, surface texture, ...), they become noticeable. Understanding the mechanisms and principal factors that trigger or determine the speed of these alteration reactions is very relevant for both preventive and corrective art conservation.

To gain such insights, employing a combination of analytical methods that allow to extract information on the chemical constituents of the degraded surface of CH artefacts at different length scales, has been proved to be very effective. Preferably (but not all) such methods are non-invasive, i.e. do not cause damage to the CH artefacts being studied.

At the macro-scale, various forms of hyperspectral imaging can be used. Macroscopic X-ray fluorescence (MA-XRF) and diffraction (MA-XRPD) are non-invasive imaging methods with intermediate to high chemical specificity that are very suitable for examining the surface of CH artefacts, either to obtain information on the original materials used to construct the artefacts or on any surface alteration that took place during the artefact's history. MA-XRF and MA-XRPD scanners can be constructed using X-ray tube sources.

Large scale particle accelerators such as synchrotron facilities are able to generate X-ray beams of sub-micrometer dimensions. It is thus possible to study heritage materials at the nanometer to micrometer level. Methods such as (sub)microscopic X-ray fluorescence, X-ray absorption spectroscopy and X-ray diffraction, either used sequentially or simultaneously, allow to study minute samples of CH artefacts, complementing the information obtained by macro-level investigations and allowing to formulate hypotheses on the manufacturing technology the artefacts and/or degradation mechanisms that have modified their surface.

As a first example of the combined use of micro- and macro-level chemical imaging methods, the study of metal-based inks used to write Egyptian papyri will be discussed. Red and black inks inscribed on 12 ancient Egyptian papyrus fragments belonging to the Papyrus Carlsberg Collection (Copenhagen), deriving from the Tebtunis temple library" in Fayum, Egypt were analyzed using synchrotron-based 2D elemental and phase mapping.

Another example of a macro/micro combination that will be discussed involves the examination by MA-XRF and scanning electron microscopy coupled to energy-dispersive X-ray analysis of a medieval stained-glass panel formerly from Notre Dame, Paris revealing its material history, including the degradation of the substrate glass and some of the glass paints employed to create it.

Last example involves the use of micro- and macro-level XRF and XRPD for the study of the pigments and their degradation behavior in Rembrandt's masterpiece 'The Nightwatch', a large oil on canvas from the 17th C.

**Application of accelerator science and technology for characterization and treatment of heritage objects / 7****Future Possibilities in Portable Accelerators for Cultural Heritage****Authors:** Alejandro Castilla<sup>None</sup>; Tessa Charles<sup>1</sup>

<sup>1</sup> *University of Liverpool, United Kingdom*

Particle accelerators conducting Ion Beam Analysis (IBA) have proven to be a powerful tool for gaining insight into cultural heritage. For the most part, these IBA techniques are non-destructive and can be applied in a non-invasive manner. However this does require that the objects being studied are removed from their setting and taken to an accelerator facility. It is not always feasible to remove an object from the field or museum and transport it to the laboratory, and in these situations a compact accelerator would be greatly beneficial. We will present the possibility of a compact 2 MeV proton accelerator that could be taken into the field to perform PIXE measurements. We detail some of the main challenges and considerations for such a device. Such a device could be used across an array of cultural heritage fields. In this talk, we will focus on the application of IBA to rock art – one of the more challenging environments for a portable accelerator.

**Application of accelerator science and technology for characterization and treatment of heritage objects / 33**

## **ACCELERATOR MASS SPECTROMETRY TECHNIQUES FOR CULTURAL HERITAGE STUDIES**

**Author:** Vladimir Levchenko<sup>1</sup>

**Co-author:** Andrew Smith<sup>2</sup>

<sup>1</sup> *ANSTO, Australia*

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ANSTO operates four tandem particle accelerators in the Centre for Accelerator Science (CAS) at its Lucas Heights campus. All these machines are used for accelerator mass spectrometry (AMS) and together deliver a suite of radioisotope dating capabilities (including radiocarbon and cosmogenic in-situ) that cover ages from 100's years old to millions years old. Three are used for ion beam analysis (IBA), as described elsewhere. Since its establishment, CAS has strived to develop and refine AMS radiocarbon measurements, improving the precision and reliability and reducing the required sample size by a factor of 1000 from a few milligrams (mg) of carbon to just a few micrograms ( $\mu\text{g}$ ). This 'micro-sample' capability has allowed the application of AMS radiocarbon studies to an ever-widening range of objects and artefacts. To illustrate this, a few of the interesting archaeological and cultural studies undertaken on our 10-million volt Australian National Tandem for Applied Research (ANTARES) accelerator are presented. These include dating of such unique objects as the Venafrò chessmen, discovered in 1932 in Venafrò, a Roman necropolis in Southern Italy, and the 'Corona Ferrea' (Iron Crown) of Monza (Italy). CAS AMS was heavily involved in establishing reliable dating for the objects from the Egyptian collection from the Nicholson museum in Australia, and for the mediaeval Torah scroll from the rare book collection of Sydney University.

The sophistication of the technique allowing sample sizes in the microgram range, and so the dating of individual chemical constituents of the item, enabled a whole new group of objects to be studied, many of which were not considered suitable previously. This CAS capacity was utilised in several Rock Art studies from Africa to Central Europe, and indeed closer to home for establishing chronology for Rock Art styles in Australian Kimberley and Arnhem Land regions and their Pleistocene antiquity. The 'micro-radiocarbon' capability allowed determining the age of the oldest dated Australian rock motif – a naturalistic depiction of kangaroo – to between 17,500 and 17,100 years ago. Some work has also been undertaken on dating wear and use residues on lithic objects, where the amount of carbon-containing material is very limited.

The AMS facility at CAS is capable of measuring other rare isotopes of intermediate half-life besides carbon, and some of these find application in nuclear heritage studies. As an example, we present measurements of cosmogenic in-situ  $^{10}\text{Be}$  and  $^{26}\text{Al}$  were used to investigate the evolution of the rock shelters in the Kimberley, where the most rock art in the region is found. Exposure dating of collapsed overhang blocks provided minimum and maximum age limits for the art on the fallen blocks and on freshly exposed surfaces. These results are important not only for establishing the time frame of the motifs, but also for assessments of art preservation and survival over time.

**Application of accelerator science and technology for characterization and treatment of heritage objects / 80****The Determination of the Age of Creation of Metal Artefacts Using Radiogenic Helium****Author:** Roger Webb<sup>1</sup>**Co-authors:** Fin Stuart<sup>2</sup>; Valerie Olive<sup>2</sup><sup>1</sup> *Surrey Ion Beam Centre, United Kingdom*<sup>2</sup> *Scottish Universities Environmental research Centre*

Metal artefacts are difficult to date from the time of their manufacture. It is not the age of the material that requires dating, but the time at which the material was worked into its final shape. This often involves melting and casting the metal to produce the final shape. We are interested here, in the production of some lead books, originating in and around Jordan and often referred to as the "Jordan Codices". These items have been very controversial in their claimed origins. Some historians claiming them to be modern items of "tourist tat" and others claiming them to be around 2,000 years old and providing keys to the gospels. The writings found on them have proven almost impossible to translate. Until now there has been no way to demonstrate the true nature of these items and much mystery and controversy surrounds them. We have previously managed to show that the lead that these books are made from is deficient in <sup>210</sup>Pb and is therefore at least 200 years old, but this does not prevent someone using old lead to manufacture "modern artefacts" nor does it show the items to be truly old.

We have now investigated a technique, used to date gold artefacts, to show demonstrate the true age of this items. The technique measures the small amount of He released and captured in the metal by the gradual decay of U and Th contamination in the original metal. The beauty of this technique is that when the metal is melted the He is released, effectively starting the clock for the technique, so that we are able to measure the time at which the metal was last melted, ie manufactured, not the age of the metal itself. We will give details of this method and the preliminary results that we have found in analysing these artefacts.

**Application of accelerator science and technology for characterization and treatment of heritage objects / 26****Capabilities of MeV SIMS for identification of traditional and modern paint materials – building a database of binders and pigments****Author:** Matea Krmpotić<sup>1</sup>**Co-authors:** Iva Bogdanović Radović<sup>2</sup>; Zdravko Siketić<sup>2</sup>; Marko Brajković<sup>2</sup>; Marko Barac<sup>2</sup>; Dubravka Jembrih-Simbürger<sup>3</sup>; Mirta Pavić<sup>4</sup><sup>1</sup> *Ruder Bošković Institute, Croatia*<sup>2</sup> *Ruder Bošković Institute*<sup>3</sup> *Institute for Natural Sciences and Technology in the Arts, Academy of Fine Arts Vienna*<sup>4</sup> *Museum of Contemporary Art Zagreb*

The preservation of works of art for cultural heritage entails the need for sensitive analytical techniques that can aid in the identification of the materials that were originally used by the artist. In the case of paintings, accurate determination of major ingredients of the paint provides invaluable information for restorers and curators and most often encompasses the use of multiple, complementary analytical techniques.

Secondary ion mass spectrometry with MeV primary ions (MeV SIMS) is an accelerator-based nuclear analytical technique that has a high potential in the study of cultural heritage objects due to its

high surface sensitivity, the ability to desorb and detect molecular ion species as well as the ability for chemical imaging [1,2]. The benefit of MeV SIMS compared to conventional (keV) SIMS, which is gaining an increasing interest in cultural heritage studies, lies in the sputtering process. By irradiating the sample with an ion beam in the MeV energy range, desorption of intact molecules from the sample surface takes place through electronic sputtering, which leads to significantly higher secondary molecular ion yields, and consequently facilitates the identification of organic components. By coupling it to other ion beam analysis techniques, such as particle-induced x-ray emission (PIXE), imaging and identification of inorganic components is also possible.

The utilization of MeV SIMS for the study of artists' paints is only starting to be recognized and data on this technique is very scarce or unavailable. The need for collecting information on traditional and modern paint materials and investigation of MeV SIMS capabilities in the study of paints is important to benefit from this technique's high potential as well as to couple it with the increasingly popular keV SIMS. In this work an overview of the current studies conducted at Ruđer Bošković Institute is presented, with the focus being on the identification of a wide selection of pigments and binders from commercially available artists' paints, falling into categories of both traditional and modern materials. The aim is to facilitate the accurate identification of major components of the paint mixtures in various case studies, for which several examples are also presented and discussed.

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[2] M. Krmpotić et al., Identification of Synthetic Organic Pigments (SOPs) Used in Modern Artist's Paints with Secondary Ion Mass Spectrometry with MeV Ions, *Anal. Chem.* 92 (2020) 9287–9294.

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### Angle Dependent XRF for the analysis of Cultural Heritage Samples

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Due to their complex structure, cultural heritage (CH) related samples are often very challenging to analyze; besides, due to their rarity and their value these samples require to be studied by means of non-invasive or non-destructive techniques. The other great challenge in the CH artifacts analysis is the plethora of materials they are composed of, from metal to glass, to ceramic, paper, or textile; these material present different conditions in which they can be analyzed. Finally, the size or the nature of the sample does not always allows moving the samples, or putting them in vacuum chambers. Thus, in many occasions, in-situ analysis are preferred as the instrument can be brought to the museum or the archaeological site to perform directly the measurement.

XRF is a very useful tool to get elemental information on the sample in a non-invasive way, it is usually affordable by many laboratories, and compact spectrometers can perform in-situ analysis. XRF can give information on the first micrometers of the sample, in this sense, it is defined as bulk technique. In the case of layered samples, the fluorescence line intensity of an element depends on the layers' composition, and the position of the element inside the sample. Moreover, attenuation and self-attenuation of the fluorescence lines, depend on the geometry of analysis. In particular the angle of irradiation and detection influence the probed depth of the technique. Thus, angle dependent XRF techniques (Grazing Emission (GE), Grazing Incidence (GI), Angle Resolved (AR) XRF) can give information on the layers' structure and composition.

These techniques consist of a scansion of the sample, collecting XRF spectra at different angles. In the case of GE -XRF the detection angle is scanned around the critical angle of reflection of the fluorescence radiation; for GI -XRF the irradiation angle is scanned around the critical angle of

reflection of the source radiation; while in AR-XRF the angle is greater than the critical angle, so no effects of reflection or interference are observed. As Grazing techniques require a small divergence in the solid angle of irradiation (for GI-XRF) or detection (for GE-XRF) they are usually applied in synchrotron facilities, where the high flux of the source compensates for the collimation, allowing to collect data with a good statistics in reasonable time.

The aim of my PhD work is to evaluate the capability of angle dependent techniques to analyze CH sample, starting with instrument that can ensure a high intensity (synchrotron or sources coupled with capillary lenses). The ultimate goal is to bring this application to laboratory instruments, to be more accessible and to perform in-situ analysis, more suitable conditions to analyze the great variety of samples that compose the CH family.

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### The Neutron Resonance Transmission Imaging technique for elemental characterization of inhomogeneous samples

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We present the implementation and applications of the Neutron Resonance Transmission Imaging (NRTI) technique, performed at the INES (Italian Neutron Experimental Station)[1] beamline of the ISIS spallation neutron source [2].

This technique relies on the measurement of the neutron beam attenuation due to the resonance absorption of epithermal neutrons ( $0.3 \text{ eV} < E_n < 100 \text{ eV}$ ) by the nuclei of a material. Since resonance structures appear at specific energy for each nuclide's neutron-induced reaction cross-sections [3], they can be used to identify and quantify elements in materials and objects. Moreover, it is possible to distinguish between isotopes of the same element.

A time and spatial-resolved nGEM (neutron Gas Electron Multiplier)[4] detector is employed for Time-of-Flight (ToF) measurements of the neutron beam transmitted through the object, providing resonance-selective imaging of the isotopic and elemental composition of the samples. Therefore, NRTI allows the localization of isotopes and elements distribution within 2D (and potentially 3D) maps of the bulk of the analyzed object.

The peculiarities of NRTI make it suitable for the characterization of inhomogeneous samples [5,6] and it can be applied to archaeological objects as it is a non-destructive technique. In particular, NRTI can be suitable for metallic artifacts analysis as neutrons can penetrate them up to a few centimeters.

Within this context, several metallic standards have been measured to study the transmission response in function of composition and thickness. The NRTI capability for isotopic imaging will be presented.

Moreover, in order to deepen the feasibility of NRTI in Cultural Heritage applications, a set of crucible fragments related to bronze and brass objects production in Roman Italy (I-II AD)[7] has been investigated through a combination of NRTI and other well-consolidated non-destructive techniques. These crucibles consist of mass-produced terracotta pots, coated with a thick layer of refractory clay. Inside, metallic inclusion related to copper and zinc alloys production can be present. In fact, some fragments show metallic depositions on their surfaces, while others could contain traces inside their volume.

At present, NRTI analysis returned the qualitative elemental composition of the fragments, revealing the presence of brass and bronze. In addition, arsenic, antimony, silver, and lead were detected in their bulk.

These kinds of archaeological samples are an example of inhomogeneous objects that can be interesting to be investigated with the NRTI technique, exploiting its possibility of visualizing the elements (and isotopes) distribution within the bulk.

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## Probing Beneath the Surface without a Scratch: Using Muonic X-ray for Depth Sensitive Elemental Analysis on Roman Coinage

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Non-destructive compositional analyses are extremely important in many cultural heritage fields. The use of negative muons (an electron analogue) has seen a resurgence in recent times, with developments occurring at several muon sources. After implanting negative muons into a sample muonic x-rays and gammas are released – these can then be detected to determine the composition of the sample. While similar in principle to X-ray fluorescence, the negative muon technique can analyse deep beneath the surface of the sample. By controlling the muons' momentum (or energy) the implantation depth of the muons can be controlled, ranging from 10s of  $\mu\text{m}$  to 10s of mm. This means the composition at different depths within the sample can be determined non-destructively. Here we review the technique and its recent applications to cultural heritage, and present case studies on Roman gold and silver coinage: the former showing no evidence of surface enrichment, the latter unambiguous evidence of it, as well as the discovery of fake coins and corroded materials.

Part of the 'Rome and the Coinages of the Mediterranean' (RACOM) project (funded by the European Research Council under the Horizon 2020 programme, grant agreement 835180) is to use muonic X-rays to determine the core composition of silver coins of Mediterranean states from c. 150 BCE to the mid-first century CE. The quality of ancient silver coinage is often seen as a comment on the fiscal health of the issuing states. Two processes are detectable: a reduction in the fineness of the alloys; and a reduction in weight standards. However, the hypothesis here is that the two processes are interlinked and that the second is largely an illusion caused by the first. These Roman silver coins were, often, produced from an alloy of copper and silver. This enabled mints to disguise debasements from the general public by exploiting the dissimilarity between the electrode potentials of the two metals. The blanks for the coins were cast and, after solidifying, they were probably kept at red heat, or reheated to red heat, for a period of time, to oxidise the copper at the surfaces of the blanks. Once oxidised, the copper oxide could then be stripped out of the alloy by soaking in an organic acid or other solution. This left a honeycomb structure of nearly pure material at the surface of the blanks,



which would then be consolidated as a rich layer when the blanks were struck between two dies to make the coins. The technique could even be made to work on alloys that contained more than 80% copper, with the result that coins left the mint looking as if they were pure.

However, Roman gold coins were thought to be pure – except for brief periods of crisis when they were debased. Due to the high art market value of the coins, aggressively destructive techniques are simply not viable. Negative muons, then, give us a unique opportunity to determine whether the surface composition of Roman gold is actually representative of the bulk composition.

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## Welcome remarks

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## Highly-specific mapping of oil paintings to detect anachronistic pigments

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*The Ghent Altarpiece* (1420s-1432) is the masterpiece of the south-Netherlandish painters Jan and Hubert van Eyck. This polyptych has been subject to an extensive restoration by the Royal Institute of Cultural Heritage (KIK-IRP, Brussels) during the last decade, a process that is not completely finished. On the panels of the polyptych that were already conserved [the outer wing panels during phase I of the restoration) and the lower register of the inner panels during phase II)] a very high degree of overpainting covering the original 15th c. paint of the *van Eyck* brothers was observed. Most of this overpaint is assumed to date from the 16th c.

The panels under treatment were examined with a combination of Macroscopic X-ray fluorescence (MA-XRF, phase I and II) and X-ray powder diffraction (MA-XRPD, phase II) with the aim of distinguishing the original and non-original (overpainted) parts of the painting. For comparison purposes, we also examined two other works by J. Van Eyck in Flemish collections: *Madonna & Child with Canon van der Paele* (1436, Musea Brugge) and *Madonna at the Fountain* (1439, Royal Museum of Fine Arts, Antwerp).

In the presentation, attention will be given to the different subtypes of three inorganic pigments that were encountered in *the Ghent Altarpiece*: lead white, copper greens and ultramarine blue.

Lead white (LW) is an omnipresent pigment in most historical paintings and is mainly composed of two components: hydrocerussite and cerussite. However, the mass ratio between these two components is not always the same and has the potential of making a distinction between the LW as employed by (the) van Eyck (brothers) and LW added in later restorations.

Prior to 2012, in the central Adoration panel, in the green meadow surrounding the altar of the Mystic Lamb, several darkened green areas were evident that corresponded to ancient retouching areas. MA-XRPD mapping revealed that in these areas, the rare copper sulfate Posnjakite ( $\text{Cu}_4(\text{SO}_4)(\text{OH})_6 \cdot \text{H}_2\text{O}$ ) was present, while this pigment is never encountered in the original green areas of *The Ghent Altarpiece* panels. Posnjakite is a mineral blue-green pigment that originates from a mining area in (what is now) the Czech Republic/Moravia.

A comparison of the pattern accessory minerals (such as albite, diopside, ...) that accompany the intensely blue mineral lazurite among different van Eyck paintings suggests that the Van Eyck workshop may have used a fairly specific type of natural ultramarine (NU). This could have potential to distinguish early 15th c. from those of later periods and also allows to differentiate original from add-on ultramarine paint in the Ghent Altarpiece.

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## Material aging in the environment

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The Institute of Physics and Materials Science is specialized in characterization of biological and bio-based material including a variety of techniques and fields of application. One of them is the determination of age.

Brief description/core competencies

Chemical changes over time can be used to describe various processes. Provided these changes are slow and uniform, it is possible to use the taphonomic behavior as a chronometric clock to predict age.

Wood: Dating models have been created for spruce, fir, larch and oak. Different storage conditions, different species/genera and the extension of the existing models in time and spatial validity are the focus of current research.

Charcoal: delineation of the factors of pyrolysis conditions and aging processes is critical to long-term stability. The study of traditional charcoal processes provides the means to separate the two effects. Degree of pyrolysis, as well as elemental composition, can be predicted from infrared spectra. Aging processes lead to specific chemical changes over time. Therefore, different epochs can be separated from each other. Further investigations aim at the improved description of pyrolysis processes, as well as the development of dating models.

Straw: dating of straw as an aggregate in clay bricks, clay plasters and similar construction methods; investigation of various influencing factors, such as storage and construction method.

Hair, bone, tooth: aging studies.

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## MeV-SIMS for analysis of modern and contemporary paints - an accelerator-based method for analysis of synthetic organic components

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Secondary Ion Mass Spectrometry with primary ions in the MeV range (MeV-SIMS) was employed as a new accelerator technique for the analysis of synthetic organic pigments (SOPs) and binders in modern and contemporary paints. Due to the so-called "soft ionization" of organic molecules caused by MeV primary ion beam, large intact organic molecules or larger molecular fragments are desorbed from the outermost surface of the sample. This results in MeV-SIMS spectra with molecular peaks of the SOPs and only low number of larger molecular fragments which can be easily used for their identification.

We could show that MeV-SIMS is very suitable, easy and straightforward analytical method especially for exact identification of synthetic organic pigments (SOPs) of different pigment classes in artists paints. Materials tested were pure pigment powders, unaged and artificially aged paint mock-ups as well as several naturally aged samples from painted outdoor sculptures and murals dating from 1970 to 2000. Compared to well-established mass spectrometry and spectroscopic methods

used in this field<sup>1</sup>, MeV-SIMS offers many advantages: information on molecular level is obtained without sample preparation prior to analysis, identification of SOPs and binder results from same mass spectrum, very small amount of sample is needed for the analysis, the sample is not consumed during measurement, thus still remaining for complementary measurements. Another benefit of the MeV SIMS is that we deal with a surface sensitive method capable for organic materials which is important concerning that material degradation occur mostly in the uppermost layers and this area is usually not accessed by chromatographic methods coupled to MS.

The investigations were performed with the 5 MeV Si<sup>4+</sup> primary ion beam from 1 MV Tandem<sup>2</sup> at Rudjer Bosković Institute, Croatia. Secondary ions were detected with linear Time-of-Flight (ToF) spectrometer. Results gained on paint muck-ups and real object samples including molecular imaging will be presented and discussed.

Due to this first application in the area of cultural heritage objects, the MeV-SIMS can be introduced as an additional accelerator-based technique giving access to organic (and inorganic) components on molecular level thus broadening the accelerator application field in sense of supplement the well-established methods in this field such as PIXE, PIGE, and RBS which give only elemental information.

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## **To be announced**

**Access to research infrastructure, and international as well as regional collaborations and networks / 89**

## **To be announced**

**How to make science tangible – Public awareness of how science and heritage work together in a tangible way – novel methods in education and outreach / 91**

## **To be announced**