



Joint ICTP-IAEA Advanced Workshop on Accelerator Mass Spectrometry Radiocarbon Dating for Heritage and Forensic Sciences | (SMR 3839)

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Reconstructing the Chronology of the Foundation of the Southwest Church (Umm el-Jimal, East Jordan) by Accelerator Mass Spectrometer Radiocarbon Dating.

Khaled Al-Bashaireh¹

¹(Dept. of Archaeology, Yarmouk University): khaledsm@email.arizona.edu)

The Umm el-Jimal archaeological site is located in the eastern desert of Jordan. It is distinguished by its monuments, water system and continuous occupation from the Nabataean till the early Muslim periods (about 800 years). The site's structures were destroyed by earthquakes, wars, agricultural expansion, etc., some structures were rebuilt and used for new functions, while stones' reuse was common even for off-site construction in neighbouring villages. The site comprises sixteen churches, but only three of them were dated by inscriptions found out of their original contexts and lately by radiocarbon dating technique. The proposed presentation introduces the aims of an ongoing research to reconstruct the chronological sequence of three churches of unknown or uncertain dates at the site (the West church, the Southwest church, the Julianos church), and better understand the production technology of their mortar and plaster cement materials. It will present primary results about one of the churches – the southwest church- including radiocarbon dates for the foundations and other parts of the building and primary results of the recipes used for its mortar and plaster. The research, when complete, will contribute to a larger comparative study of the three churches with the goal of refining the history of Christian church construction, use and style during the Late Antiquity. The results will assist archaeological conservators, heritage architects, planners and site managers to develop scientific-based plans for a long-term protection and conservation of the site and will help repair mortars with solutions that are adapted to local raw materials.

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[2] A. Author, B. Coauthor, J. Sci. Res. **17**, 7531 (2013).

Developing approaches to determine the age of “really ancient trees” beyond tree-ring counting

Jonathan Barichivich¹

*¹Laboratoire des Sciences du Climat et de l'Environnement (LSCE), IPSL,
CRNS/CEA/UVSQ, France*

Old and large trees have a singular appeal to inspire us to appreciate and protect the natural world. Many of such exceptional trees around the world are considered as monumental or remarkable trees because of their unique size and historic, aesthetic and cultural values. These trees are also living time capsules of the natural history of the Earth, which are threatened by climate change, logging and fires. Among the three trillions of trees on Earth, only few are ancient and are still alive. The oldest non-clonal living trees on Earth are Bristlecone pines in the western US and Alerces in the Valdivian rainforest of southern Chile. Living trees of these species can reach maximum ages of around 5000 years as determined by counting their annual growth rings. Ring counting and dating is considered the “gold standard” for determining the age of trees in dendrochronology – the discipline of the study of tree rings. However, this gold standard often falls short for aging very ancient living trees. The technique is limited when the trees have a large size that precludes complete sampling with the longest boring tool, lack distinct annual growth rings or the wood in the inner part of the trunk is rotten or missing. In such cases, at best only a minimum age can be obtained from ring counting. The outstanding scientific challenge is to find non-invasive ways to estimate the age of these living trees that we know that are ancient but do not have all their tree rings. A new, reproducible science-based framework to date ancient trees with uncertainty quantification is needed. It should be generalizable and integrate multiple complementary lines of evidence, such as tree-ring counting, growth modelling and accurate radiocarbon dating. I will present a study case of an Alerce tree in southern Chile known as Alerce Milenario or Lañilawal. We used a combination of tree-ring counting, modelling and radiocarbon dating of dead root wood to estimate that there is an 80% of chance that the tree is older than 5000 years [1]. This means that with certainty it is one of the oldest living trees in the world. Whether it could be the oldest or not so far reported has raised a debate in the tree-ring community, which tends to be dogmatic with their accurate albeit limited “gold standard”. Improved techniques of radiocarbon dating can help to quantify and reduce uncertainties in estimating the age of very ancient trees to raise awareness of their global value and ensure their strict protection.

[1] G. Pokin, Science, ScienceInsider: <https://www.science.org/content/article/world-s-oldest-tree-growing-ravine-chile> (2012)

State of art of micro-samples measurements at INFN-LABEC, Florence

Serena Barone^{1,2}, Mariaelena Fedi², and Lucia Liccioli²

¹*Department of Physics and Astronomy, University of Florence*

²*National Institute of Nuclear Physics, Florence*

In the ¹⁴C-AMS field, minimising the invasiveness of the analysis has been one the most relevant research topics, especially when applied to Cultural Heritage.

As a matter of fact, there are applications in which the mass required for the analysis may become problematic: for example, when a central portion of the object should be collected, thus risking to alter the integrity and spoil the legibility of the object to be dated; in case of highly degraded materials or samples for which a particular selective pre-treatment is mandatory, hence leaving us with very small amount of mass for the analysis.

At INFN-LABEC in Florence, we built up a new experimental set-up, aiming at reducing the necessary mass of graphite for the ¹⁴C concentration measurement. Indeed, we are now able to deal with samples of few tens of μg of carbon, while the typical masses collected at the end of the “large” graphitization process are about 700 μg .

We installed new graphitization reactors, reducing their volumes to favour the graphitization reaction by increasing the collected pressure. New reactors were equipped with a small quartz tube used as the “hot” part and a silver cold finger. We also designed and assembled dedicated ovens and small Peltier-based cooling devices, used to reach the temperature needed to trigger the reaction and to trap the unwanted water produced during the graphitization reaction, respectively. Moreover, we installed new miniature pressure gauges, sensitive to low pressures, and we assembled a home-made data acquisition system based on an Arduino board.

As far as the optimization of the AMS measurements in the Tandem accelerator, we performed “Lilliput” beam runs using different operating conditions, especially considering the sputtering ion source and the injection timings of the three carbon isotope masses into the accelerator tube.

The preliminary tests on the new “Lilliput” experimental set-up were successful and nowadays micro-samples are routinely measured, studying different applications in the field of Cultural Heritage.

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Radiocarbon Dating of Lead White Colouring Materials from Archaeological Sites along the Adriatic Coast

Katarina Batur¹, Lucile Beck², Cyrielle Messenger², Adelphine Bonneau³, Martina Čelhar¹, Irena Radić Rossi¹

¹*University of Zadar, Department of archaeology, Zadar, Croatia*

²*Laboratoire de Mesure du Carbone 14 (LMC14), LSCE/IPSL, CEA-CNRS-UVSQ, Université Paris Saclay, 91191 Gif-sur-Yvette, France*

³*Département de chimie et Département d'histoire, Université de Sherbrooke, Sherbrooke, Canada*

Lead white, composed of both lead carbonate or cerussite (PbCO_3) and basic lead carbonate or hydrocerussite ($\text{Pb}(\text{CO}_3)_2\text{OH}$), has been used as a coloring material - pigment or cosmetic – from Antiquity to the 20th Century. The synthesis of lead white was known as early as the 4th - 3rd century BC. Lead white became widely accessible in Europe during the Early Modern period, especially in 16th and 17th century when its manufacture reached a high peak. Since the ancient production process of lead white involves a fermentation phase, where atmospheric carbon dioxide is incorporated into lead carbonates, researchers have successfully performed ^{14}C dating of samples from cultural heritage sites [1, 2, 3]. Moreover, recent studies have demonstrated that the combination of ^{14}C radiocarbon analyses and $\delta^{13}\text{C}$ can help to determine production processes, providing significant potential to understand archaeological sites [4]. This poster presents the results of experiments performed on lead white materials sampled at archaeological sites in present-day Croatia, taking into consideration their well-recorded contexts, including the environmental impact of the type of site (underwater or land) and the fact they have not been altered with binding medium. Experimental studies were performed on samples from archaeological sites located along the Adriatic coast: Liburnian settlement Nadin (2nd century BC – 20 AD) [5], a Roman shipwreck in the cove Glavat, on Mljet island (end of 1st century AD) and the Gnalić shipwreck site (precisely dated to 1583). The poster discusses the viability of dating certain sequences of an archaeological site through radiocarbon dating of lead white, including an analysis of favourable preservation conditions to obtain relevant results.

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[2] L. Hendriks, L., *Anal. Chem.* **92** (**11**), 7674–7682 (2020).

[3] G. Quarta, M. D'Elia, S. Paparella, A. Serra, L. Calcagnile, *J. Cult. Heritage* **46**, 102–107 (2020).

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Radiocarbon dating of iron reinforcements of Notre-Dame de Paris cathedral

M. Bernard¹, L. Beck², E. Delqué-Kolic², M. L'Héritier³, A. Azéma⁴, P. Dillmann¹

¹*Laboratoire Archéomatériaux et Prévision de l'Altération (LAPA), IRAMAT UMR7065, NIMBE UMR3685, Université Paris-Saclay, CEA/CNRS, France*

²*Laboratoire de Mesure du Carbone 14 (LMC 14), LSCE-IPSL, CNRS/CEA/UVSQ, France*

³*Université Paris 8, ArScAn UMR7041, CNRS, France*

⁴*Laboratoire de Recherche des Monuments Historiques (LRMH), CRC UAR3224, Muséum National d'Histoire Naturelle (MNHN), France*

Iron reinforcements of Notre-Dame de Paris cathedral are a source of data for the interpretation of gothic architecture. The radiocarbon dating of these metals can provide information on the chronology of the different construction periods of the building as well as revealing potential recycling during restoration phases. In addition, this method coupled with archaeometallurgical analyses can illuminate the roles played by iron in the strategy of the erection of the monument.

Among iron artefacts in the cathedral Notre-Dame de Paris are found staples in the masonry of the tribunes, nave, aisles and upper walls. The restoration works also reveal various tie-rods, dowels, ridge caps and framing irons such as nails, bolted rods or keyed dowels. M. L'Héritier et al. [1] performed the radiocarbon dating of six staples: two of them come from the tribunes and four from the upper walls. These first results revealed that staples from the tribunes are older than those from the upper walls, and it coincides with the cathedral's construction phases (middle of the 12th century and beginning of the 13th century). Following the experimental procedure described by S. Leroy et al. [2] for Bourges and Beauvais cathedrals' iron reinforcements, two ancient supporting tie-rods coming from the clerestory windows were also dated. According to the first data obtained, it can be confirmed that these crossbars are not from the same period, which confirms the metallographic data. This could be an example of restauration in the monument. However, for better and consistent results, other samples of these tie-rods should be analyzed in the future.

In this way, radiocarbon analyses, coupled with metallographic and archaeometric studies, will help for our understanding of this gothic monument and more precisely of the buildings techniques through the use of iron reinforcements.

[1] M. L'Héritier, A. Azéma, D. Sylvilay, E. Delqué-Kolic, B. Lucile, I. Guillot, M. Bernard, P. Dillmann, PLOS ONE, 18(3) (2023).

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Brazilian 14C-AMS facility after 10 years: What is new and what is to come?

Carla Carvalho^{1,2}, Kita Macario^{1,3}, and Fabiana Oliveira^{1,4}

¹*LAC-UFF - Radiocarbon Laboratory, Fluminense Federal University, Brazil.*

²*Geochemistry Department, Fluminense Federal University, Brazil.*

³*Physics Department, Fluminense Federal University, Brazil.*

⁴*Physics-Chemistry Department, Fluminense Federal University, Brazil.*

The first 14C-AMS facility in Latin America has completed one decade in 2022. The sample preparation of the Radiocarbon Laboratory of the Fluminense Federal University (LAC-UFF) was installed in 2009, and its NEC Single Stage Accelerator Mass Spectrometry system has been operational since 2012, and since then LAC-UFF became a reference center for radiocarbon dating in Brazil receiving several kinds of samples from many research groups around the country. Over these years we have implemented pretreatment protocols for a variety of materials received on a national research scale aiming at the scientific development and training of human resources. We will present some current protocols for the sample preparation of some kinds of samples, such as cellulose, soil, bone, parchment, algae and biofuels. Moreover, after 10 years of operation, with the aim of expanding the range of materials to be analyzed, we will report some tests we have made in order to improve accuracy and precision, background levels, and data handling results using chronological models applied to different contexts [1]. Additionally, we will present future perspectives on the implementation of recently established protocols [2, 3] of rock art dating.

[1] F. Oliveira, K. Macario, C. Carvalho, V. Moreira, E. Alves, I. Chanca, M. Diaz, R. Jou, I. Hammerschlag, B. Netto, M.I. Oliveira, A. Assumpção, D. Fernandes, *Radiocarbon*, 63, 1233, (2021).

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^{14}C Scientific Dissemination as a means to improve multidisciplinary research

Kita Macario¹, Eduardo Q Alves^{1,2}, Fabiana Oliveira¹, Carla Carvalho¹, Ingrid Chanca^{1,3}, Renata Jou¹, Vinicius Moreira¹

¹Radiocarbon Laboratory of the Fluminense Federal University, Brazil

²European Geosciences Union, Germany

³Max Plank Institute for Biogeosciences, Germany

Over the last decade the Radiocarbon Laboratory of the Fluminense Federal University (LAC-UFF) has been measuring hundreds of samples per year, among scientific and commercial samples [1]. As the first full equipped ^{14}C Accelerator Mass Spectrometry Facility in Latin America we have had demand for a wide variety of sample materials and, as far as possible, have implemented new protocols so that we could respond to this demand [2]. Several students were trained, new laboratories were installed in other universities with our collaboration and the First Latin American Conference was organized in Niteroi so that we could foster new collaborations and help a new branch of the radiocarbon community to be established [3].

Despite the absence of a AMS facility before 2012, the Radiocarbon technique was widely used for all kinds of applications in Brazil, most of the samples being measured by commercial laboratories over the world. Along the years, the unfamiliarity of ^{14}C users with its basic concepts, limits and data handling called our attention. The lack of ^{14}C researchers in multidisciplinary works contributed to lowering the standards of ^{14}C based research. Although this is most probably a global effect, in this work, we aim to discuss it, from our perspective and experience, claiming our responsibility towards this problem and proposing the scientific dissemination of ^{14}C as a contribution to minimize the damage of misinformation to society.

[1] Macario KD, Gomes PR, Anjos RM, Carvalho C, Linares R, Alves EQ, Oliveira FM, Castro MD, Chanca IS, Silveira MF, Pessenda LC. The Brazilian AMS Radiocarbon Laboratory (LAC-UFF) and the intercomparison of results with CENA and UGAMS. *Radiocarbon*. 55(2):325-30. (2013)

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[3] Macario K, Solis C. CLARa: Proceedings of the 1st Latin American Radiocarbon Conference INTRODUCTION. *RADIOCARBON*. Aug 1;63(4):V-IX. (2021)

What we learned dating mortar

Alexander Cherkinsky

Center for applied Isotope studies, University of Georgia, Athens, Georgia, USA

There was analyzed a single mortar fragment (25x15x13cm) from the Roquefort Castle, France. From this fragment was extracted 3 different charcoal samples, one shell samples and also analyzed different fractions of mortar itself. All three charcoal samples were pretreated with the standards AAA technique and resulted with the three different ages from 830 ± 25 to 1850 ± 25 BP ^{14}C years. The shell sample came out with the age 920 ± 25 BP ^{14}C years. There also was dated fine soft colored unpretreated mortar lump (Prevosti et al. 2016) which has age 2390 ± 25 BP ^{14}C years.

The mortar was then separated into fractions by different techniques including: 1) gentle mechanical crushing by hammer with following ultrasonic dispersing and sieving; 2) gentle cryogenic crushing with following ultrasonic dispersing and sieving (Marzaioli et al. 2012); 3) gentle cryogenic crushing with following sieve separation fractions without ultrasound disintegration.

As it was developed by Lindroos et al (2007) we also used the sequential dissolution. However the condition of the reaction and acid concentration were different. Each mortar fraction was reacted with 100% H_3PO_4 in vacuum at 0°C . There were collected two fraction of CO_2 . The first fraction was collected after 1 minute of reaction and the second after 30 minutes of reaction. Such condition allows significantly reduce the reaction rate and especially the reaction of dissolution of the limestone carbonates, so the 100% H_3PO_4 is significantly weaker that 85% acid and decrease the temperature on each 10°C reduce the reaction rate in 2-3 times.

The oldest dates were obtained for the fine soft mortar lump 2390 ± 25 . The fraction of $45\text{-}250\mu\text{m}$ remaining in precipitant after 30 min of ultrasound treatment in both mechanical and cryogenic crushing is also significantly older then historical data; they are 1630 ± 25 and 1700 ± 25 , correspondently. The fraction $45\text{-}250\mu\text{m}$ which was not treated with ultrasound after 30 minutes reactions also shows older date 1120 ± 25 as a result of the reaction not only with fine calcium carbonate formatted in a process of hardening but also with some residual limestone carbonates. The fine fraction, which has formed the suspension after 30 minute of ultrasound treatment has shown ^{14}C ages which are correspond with he historical data.

The reaction rate could play the significant role in the dating of mortar. The using 100% H_3PO_4 , and decreasing the temperature to 0°C allows recover carbon dioxide only from the CaCO_3 , which was formatted in the hardening process and do not dissolve the limestone carbonates. The first portion of CO_2 should not be used for the dating, so it mostly carbonates which could be formatted or exchanged with precipitation after hardening of the mortar.

Lindroos A, Heinemeier J, Ringbom A, Brasken M, Sveinbjornsdottir AE. Radiocarbon, 49 (1):47-67(2007).

Marzaioli F, Nonni S, et al. NIMB, 294, <http://dx.doi.org/10.1016/j.nimb.2012.09.006>(2012)
Prevosti M, Lindroos A, Heinemeier J, Coll R. Journal of Archaeological Science: Reports 6: 275–283 (2012).

Compound-specific Isotope analysis of lipid residues in Brazilian ceramics

Fabiana M. de Oliveira¹, Jayane M. C. da Silva¹, Kita D. Macario¹, Marcelo C. Muniz², Roberto M. Anjos², and Angela Buarque³

¹ *Laboratório de Radiocarbono, Instituto de Física, Universidade Federal Fluminense, Av. Gal. Milton Tavares de Souza, s/n, Niterói, 24210-346, RJ, Brazil.*

² *Laboratório de Radioecologia e Alterações Ambientais, Instituto de Física, Universidade Federal Fluminense, Av. Gal. Milton Tavares de Souza, s/n, Niterói, 24210-346, RJ, Brazil.*

³ *Departamento de Antropologia, Museu Nacional, Universidade Federal do Rio de Janeiro, Quinta da Boa Vista, s/n, Rio de Janeiro, 20940-40, RJ, Brazil.*

For a long time, the origin of the Tupinambá and Guarani peoples, who intensively occupied the Brazilian coast in the 16th century has been studied and there are few previous results for the chronology of these archaeological sites [1]. In this work isotopic results related to diet of these groups will be presented relating to previous radiocarbon dating [2]. The Fatty Acids Methyl Esters (FAME) used as biomarker for carbon dating was C16:0 and C18:0. The compound-specific isotopic analysis (CSIA) was performed coupling the gas chromatograph (Trace GC 1310) to a Mass spectrometry and (ISQ 7000, Thermo Fisher Scientific) and an isotope ratio mass spectrometry system (Delta v Advantage, Thermo Fisher Scientific). These results will allow relating the existing chronology of ceramics and remaining archaeological sites of the Tupi-Guarani groups that inhabited Brazil thousands of years ago [3,4].

[1] R. Scheel-ybert, K.D. Macario, A. Buarque, R.M. Anjos, M. Beauclair, *Anais da Academia Brasileira de Ciências*. **80**, 763 (2008).

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Ion beam techniques: simple protocols for detecting fraudulent medicines and food supplements

Guilherme M. S. Souza¹, P. Chytry¹, L. Amaral¹ and Johnny F. Dias¹

¹*Ion Implantation Laboratory, Institute of Physics, UFRGS (Porto Alegre, Brazil)*

Ion beam analytical techniques like PIXE (Particle-Induced X-ray Emission) and MeV-SIMS (Secondary Ion Mass Spectrometry) proved to be valuable tools for the differentiation of legal sildenafil-based substances from illegal ones [1] and for establishing patterns for authentication and provenance of coffee [2].

The present work expands the research program developed during *the Coordinated Research Project F11021* sponsored by the International Atomic Energy Agency (IAEA). The primary goal of this research project is to develop simple protocols for sample preparation and analysis of medicines and food supplements using ion-based techniques. To that end, different sildenafil-based medicines like Viagra®, Cialis® and their respective generics were analysed with PIXE and MeV-SIMS and compared to vardenafil-based medicines like Levitra®. The results were summarized into a comprehensive database, leading to the establishment of an easy-to-follow procedure for the analysis of these materials with ion beams. In this way, we were able to discriminate legal medicines from counterfeit ones.

Unlike medicines, food supplements are usually not regulated by local sanitary authorities, thus posing a real threat for potential consumers. Nowadays, vitamins and omega 3 are among the top selling supplements sold over the internet in Brazil and other countries. While vitamins are sold in the form of tablets, omega 3 is usually marketed as fish oil capsules claiming to be a potential source of this fatty acid. Consequently, the analysis of vitamins is relatively easy as far as PIXE and MeV-SIMS are concerned. On the other hand, the development of protocols for target preparation and analysis of omega 3 is challenging for in-vacuum measurements. First results on the measurements of omega-3 will be shown.

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[2] P. Chytry, G. M. S. Souza et al., *Forensic Science International* **335**, 111281 (2022).

¹⁴C preparation protocols for archaeological samples at the LMC14, Saclay, France.

J.-P. Dumoulin¹, I. Caffy¹, E. Delqué-Količ¹, C. Goulas, S. Hain, C. Moreau¹, M. Perron¹, M. Sieudat¹, B. Thellier¹, L. Beck¹

¹ Laboratoire de Mesure du Carbone 14 (LMC14), LSCE/IPSL, CEA-CNRS-UVSQ, Université Paris-Saclay, F-91191 Gif-sur-Yvette, France

jean-pascal.dumoulin@lsce.ipsl.fr

The LMC14 (Laboratoire de Mesure du Carbone 14) and its AMS ARTEMIS is the national facility dedicated to high-precision radiocarbon measurement for five French institutions (CNRS, CEA, IRD, IRSN, Ministère de la Culture). Around 4000 samples of different types of organic matters and carbonates are prepared and measured every year (Dumoulin et al. 2017). Radiocarbon analysis of very small samples (below 0.2 mg of carbon) has also been explored (Delqué-Kolic et al. 2013) and modified graphitization procedures combined with a specific protocol of measurement are now used when the carbon content is low (Moreau et al. 2020). Samples like archaeological iron (Leroy et al. 2015) as well as water (Dumoulin et al. 2013) are also analyzed. Since 2017 and our last status report, we have developed new protocols to expand our range of datable samples and take into account the expectations of new archaeological projects. The specific protocols developed for dating cellulose (wood), oxalates (rock art) or lead white (cosmetics and paintings) will be detailed. The results obtained for artifacts containing various carbon contents as paint, leather, wax or pearl will also be presented.

Some Dating Results of Prehistoric Sites in Kalimantan

Nia Marniati Etie Fajari¹

¹ *Research Center for Archaeometry, National Research and Innovation Agency, Indonesia*

We have been working on prehistoric research in Kalimantan, Indonesia. Our research found that Kalimantan has prehistoric sites from various cultural periods. Determining the absolute dating of those sites relies on radiocarbon dating analysis. This paper discusses some of the results of the radiocarbon dating analysis that we have carried out. These results play an important role in determining the sites' chronology.

We conducted AMS radiocarbon dating on several sites: Nanga Balang, Diang Kaung, and Diang Balu in the tropical forest of West Kalimantan¹; and Liang Bangkai², Gua Batu^{3,4} and Gua Payung⁵ in karst hill of South Kalimantan. The AMS radiocarbon dating from Nanga Balang gave 3.074-2.923 cal BP⁶. This dating confirms that Nanga Balang was a dwelling during the Neolithic period with finds of pottery, stone adzes, bark cloth beaters. Research in Diang Balu found a cultural layer (layer C at 80 cm) with glass beads, bones, stones and shells. The AMS radiocarbon from this gave 149-187 cal BP. This dating is much younger than the dating of embedded charcoal at the flowstone (depth of between 30-80 cm). Analysis of the charcoal samples yielded 14,289-14,992 cal BP, dated back into the Terminal Pleistocene¹. Meanwhile, the results from Diang Kaung dated 11,082-11,214 cal BP (the Pleistocene-Holocene transitional dwellings at DKG-01) and 426-500 cal BP (the campsite at DKG-03)¹.

Dating analysis of limestone rock shelters and caves of the Meratus Mountains in South Kalimantan indicates a dwelling period during the Terminal Pleistocene-Holocen (15.514-940 BP). The result of 15514-14568 BP dating found at Liang Bangkai is the oldest^{2,7}. However, there has been no further report regarding this data. Liang Bangkai's findings are stone adze, lithic, bone tools, pottery, animal bones, animal teeth, molluscs, and rock art². The radiocarbon dating analysis from the upper layer gave 3867-3810 BP, confirming the Neolithic cultural layer at Liang Bangkai. In the middle layer, Liang Bangkai has 6.573--6.424 cal BP^{2,4}. Recent research has dated Gua Batu with 6,065-5,040 cal BP³. Gua Batu is a Pre-Neolithic dwelling with lithics, bone tools, shell tools, animal bones, animal teeth, human teeth, and molluscs. The Gua Batu dwelling is equal to the middle layer in Liang Bangkai³.

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MORPHOLOGICAL AND SPECTROSCOPIC INVESTIGATIONS OF ANCIENT EGYPTIAN GILDED WOODEN STATUE

Mohamed Moustafa¹

¹(Wood Lab- The Grand Egyptian Museum, Giza, Egypt)

The purpose of this study is to identify the materials used in painting and decorating a painted and gilded wooden Bastet statue from the recent excavation in Saqqara for the first time from the cemetery discovery. A wide range of non-invasive analytical techniques has been applied to reveal unexpected information of the studied statue.

The painted layer, inlaid materials, wooden base, and gilded layer were investigated using the optical microscopy (VHX-Microscope), SEM–EDX, and Raman spectroscopy. Also, applying anew improvements on the technical photography methods to detect the traces of filler materials in the micro-scale, and the signs of brushes which were used in painting including multi spectral imaging microscopic technique, and raking microscope technique as well.

The wood species was identified as Sycamore-fig (*Ficus sycamores*). Calcium carbonate was detected in the white painted layer, and the coarse ground layer as well. Whereas, the greenish inlaid eyes were identified as a glass mixture with malachite, and traces of Egyptian green for the first time recorded in the colored inlaid glass. Besides, traces of Egyptian blue was detected as a filler material used to fill gaps around the inlaid eyes. However, the evaluation of gilded layer showed a low concentration of gold alloy in a mixture with copper. According to the results of the study, the improvements of technical photography methods along with non-invasive spectroscopic techniques provides a clear results to identify the manufacturing techniques and reduce the possibility error and using destructive techniques.

There are a big challenges related to this study, that we don't have a clear information about the dating of this object, it was discovered around cemeteries areas from the old kingdom, new kingdom, and the late period, so based on the technical features and manufacturing techniques could help to know more information about the dating of this object, in addition, radiocarbon dating could confirm the results about accurate determination of the dating related to the studied object [1].

Keywords: Bastet Statue, Multispectral imaging microscope, Raking microscope, SEM–EDX, Raman

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A detailed chronology of the sedimentation in the Danube abyssal fan records the major episodes of the late-Holocene Black Sea evolution

Maria Ilie^(1,2), Tiberiu Sava⁽¹⁾, Alfred Vespremeanu-Stroe⁽³⁾, Octavian G. Dului⁽²⁾, Gabriela Cristea⁽⁴⁾, Gabriel Ion⁽⁵⁾, Dan Olteanu^(1,3), Aritina Haliuc⁽⁶⁾, Cristian Manailescu⁽¹⁾ and Gabriela Sava⁽¹⁾

⁽¹⁾*RoAMS Laboratory, Horia Hulubei National Institute for Physics and Nuclear Engineering, 30 Reactorului St., 077125, Magurele-Bucharest, Romania*

⁽²⁾*University of Bucharest, Faculty of Physics, Doctoral School of Physics, 405 Atomistilor St., 077125, Magurele (Ilfov), Romania*

⁽³⁾*GEODAR Research Center for Geomorphology, Geoarchaeology and Paleo-environments, Faculty of Geography, University of Bucharest, 1st N. Balcescu St., Bucharest, Romania*

⁽⁴⁾*Department of Mass Spectrometry, Chromatography and Applied Physics, National Institute for Research and Development of Isotopic and Molecular Technologies, 67- 103 Donath St., 400293, Cluj-Napoca, Romania*

⁽⁵⁾*National Institute of Marine Geology and Geo-Ecology (GeoEcoMar), 23-25 Dimitrie Onciul St., 024053, Bucharest, Romania*

⁽⁶⁾*Romanian Academy, Institute of Speleology, 5 Clinicilor St., 400006, Cluj-Napoca, Romania*

In the poster paper will be presented the construction of a high-resolution Bayesian sedimentation model spanning the last 5500 years based on 25 AMS radiocarbon dated sediments of bulk organic matter (OM) sampled from the NW Black Sea anoxic waters of the continental slope. The corrections for the ¹⁴C ages due to marine reservoir effect (MRE) and detritus organic carbon will be correlated with exogenous information such as ²¹⁰Pb dating, metallurgy pollution and human-induced soil erosion, highlighting the Danube influence on the geochemistry and chronology of the NW Black Sea sediments through the input of terrigenous organic matter.

The results will show excellent agreement with some of the previous studies, supporting a total age offset for the bulk OM of 60 years as MRE and 580 years as detritus organic carbon influence. The revisited chronology will pinpoint the first and second invasion of the coccolithophores *Emiliana huxleyi* at 2524 ± 87 and 625 ± 65 years cal. BP. Sedimentation rate will show an increase of about three times with the starting of the late Medieval, which correspond to the highest observed sediment discharge of the Danube as are considered the last 500-300 years.

This type of high-resolution sedimentation model will be an important step for constructing the carbon budget in bottom waters of variable oxygen concentration.

Age determination of the Rotunda of St. George in Nitrianska Blatnica and the Church of St. Margaret of Antioch in Kopčany

**Ivan Kontul¹, Pavel P. Povinec¹, Alexander Cherkinsky², Jozef Dorica³,
Irka Hajdas⁴, Yao Gu^{4,5}, A. J. Timothy Jull^{6,7,8}, Tomáš Lupták⁹, Mihály Molnár⁷,
Peter Steier¹⁰, Ivo Svetlik¹¹, Eva Maria Wild¹⁰**

¹*Faculty of Mathematics, Physics and Informatics, Comenius University, Bratislava, Slovakia*

²*Center for Applied Isotope Studies, University of Georgia, Athens, GA, USA*

³*Restauro Complet s.r.o., Žilina, Slovakia*

⁴*Laboratory of Ion Beam Physics, ETH Zurich, Switzerland*

⁵*Laboratory of AMS Dating and the Environment, Nanjing University, Nanjing, China*

⁶*Accelerator Mass Spectrometry Laboratory, University of Arizona, Tucson, AZ, USA*

⁷*INTERACT Centre, Institute for Nuclear Research, Debrecen, Hungary*

⁸*Department of Geosciences, University of Arizona, Tucson, AZ, USA*

⁹*Restauro, s.r.o., Bratislava, Slovakia*

¹⁰*VERA Laboratory, Isotope Physics, Faculty of Physics, University of Vienna, Vienna, Austria*

¹¹*Nuclear Physics Institute, Czech Academy of Sciences, Prague, Czech Republic*

The Rotunda of St. George in Nitrianska Blatnica and the Church of St. Margaret of Antioch in Kopčany are two standing sacral buildings in the western part of Slovakia. Both underwent several extensive reconstructions in their past, which complicated determination of the time period of their origin. Over the years, archaeological and architectural research showed the possibility of the origin of these two building being as far as 9th century AD. In recent years, both internal and external restoration works were carried out on these buildings, and this represented unique opportunity to collect samples from the exposed original building materials and use radiocarbon dating to determine their ages. Wood, charcoal, mortar and plaster samples were collected and dated using accelerator mass spectrometry.

An international consortium consisting of radiocarbon laboratories from Debrecen (Hungary), Athens (Georgia, USA), Prague (Czech Republic), Tucson (Arizona, USA), Vienna (Austria), Zurich (Switzerland), and Bratislava (Slovakia) has been organized to determine the age of these samples and by proxy the age of these historically significant buildings. Radiocarbon analysis confirmed that both structures were built in the 9th century, the earliest time of the construction of Rotunda of St. George is 783-880 AD and for Church of St. Margaret of Antioch it is 799-884 AD. The wide age interval has not been influenced only by uncertainties of radiocarbon measurements, but mainly by the specific character of the calibration curve (plateau) during this time period. The obtained radiocarbon age makes the Rotunda of St. George and Church of St. Margaret one of the oldest still-standing Christian churches in the eastern part of Central Europe.

Plans for an AMS installation

A. Lagoyannis¹

¹ *TANDEM Accelerator Laboratory, Institute of Nuclear and Particle Physics, NCSR
“Demokritos”, 153.10 Aghia Paraskevi, Athens, Greece*

In 2021, the School of Archaeometry of The Oxford University donated at NCSR “Demokritos” a fully functional AMS Tandetron accelerator. The AMS is equipped with two ion sources: one for solid and one for gas samples. The analysis of the samples is fully automatized through a computer-controlled system. In late 2021 – early 2022 the accelerator was disassembled and packaged at the University of Oxford premises. Since last September the AMS machine is stored at the ground floor of the Tandem Laboratory. In the next few months, the room that will host the new system will be prepared by properly enforcing the ground plate of the building and by installing the necessary electrical and cooling subsystems.

The plans for installing and operating the AMS will be presented.

NIR Technique for Non-Invasive Study and Dating of Islamic Paper

Hend Mahgoub¹ and Matija Strlič¹

¹*Faculty of Chemistry and Chemical Technology, University of Ljubljana, Slovenia*

This research introduces a new non-destructive spectroscopic method for characterization and surveying of Islamic paper based on NIR spectroscopy. A reference collection of Islamic paper was used to build the calibration models using multivariate data analysis methods in order to correlate analytical techniques results and spectral measurements [1]. Two discriminant models and two regression models were developed to investigate Islamic paper main material properties. 43 books from the Islamic collection of the Wellcome Library [2] were surveyed using this portable and non-destructive method, in order to identify four paper properties: presence of starch and polishing, pH and degree of polymerization (DP). The first two could be used for identification of Islamic paper (those with starch and polishing have most probably been produced in Islamic countries), while pH and DP reflect the condition of paper.

In addition, a dating model was successfully developed using NIR spectral data of the Wellcome Library collection based on multivariate data analysis, which can now be used to date historic Islamic paper from the 17th-19th century (1652 -1880) AD.

The survey results have shown that ~40% of the Wellcome Library Islamic collection has the potential to be of Islamic origin in respect to the two main characteristics of Islamic paper; the presence of starch and polishing. Moreover, the results have reflected that most of the collection is in a good condition with a low rate of deterioration and can be handled safely except few papers based on the estimated pH and DP values of the surveyed papers.

Although the validation and prediction errors of the developed models are considered good for surveys in comparison to the calibration error of the reference method, the results and the method can be refined by expanding the reference database using a wide range of Islamic paper samples covering the different types of papers found in Islamic libraries.

The results show the potential of NIR spectroscopy in the study and surveys of Islamic paper collections. If such study and method applied across Islamic paper collections, it could be used to systematically develop evidence-based preservation policies in Islamic libraries and archives.

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¹⁴C dating for MODIS 2 carbonate mortars – do time and size matter?

Fatima Pawelczyk¹ Yao Gu^{2,3}, Natalia Piotrowska¹, Alicja Ustrzycka¹, Irka Hajdas²

¹Institute of Physics, Silesian University of Technology, Konarskiego 22b, Gliwice 44-100, Poland

²Laboratory of Ion Beam Physics, ETHZ, Otto-Stern-Weg 5, 8093 Zurich, Switzerland.

³Laboratory of AMS Dating and the Environment, School of Geography and Ocean Science, Nanjing University, Nanjing 210023, China

In our work we present a procedure applied for the ¹⁴C dating of three samples of mortars from the project MODIS 2, which is an international laboratory intercomparison. The samples selected for the exercise comprise three different mortars:

- 1) MODIS2.1: mortar from Finnish medieval church
- 2) MODIS2.2: mortar from Swedish medieval church
- 3) MODIS2.3: mortar from Spanish early Christian basilica

The material was sieved and three different size fractions were separated: <45 micrometers, 45-63 micrometers, and >63 micrometers. The composition of each fraction was analyzed under the binocular microscope.

The mortar carbonates were dissolved under vacuum conditions, using H₃PO₄ and the CO₂ coming from following time intervals was collected: 1-3sec, 4-6sec, 7-9sec, 10-12sec, and remaining CO₂. Also, the bulk carbonate from each size fraction was dated. In general, a growing age trend was observed with older age for samples from later time intervals, and the oldest for remaining CO₂ fraction.

The CO₂ was graphitized using AGE system at the ETH or Gliwice, or introduced to gas ion source of the MICADAS at the ETH. Moreover, the charcoal fragments were discovered in a sample MODIS2.2. The age of this charcoal was measured in ETH and Gliwice laboratories to 1090±30 BP and 950±30 BP, respectively. For comparison, the age of carbonate fractions for this mortar ranged from 600 to over 2000 BP.

In addition, the stable isotope IRMS measurements were performed for carbonates from different size fractions and bulk material using the CF-IRMS IsoPrime coupled to Multiflow device.

Correction of isotope fractionation in radiocarbon based determinations of biocomponents

Jacek Pawlyta

AGH University of Science and Technology, Laboratory for Environmental Radioisotopic Analyses (LERA-C14), Kraków, Poland, jpawlyta@agh.edu.pl

Radiocarbon is one of the methods used to determine the proportion of biocarbon. It can be used for solid, liquid and gaseous fuels, packaging and construction materials for consumer and industrial products. There are several standards that allow the use of the radiocarbon method for these determinations. For example, all standards ASTM D6866-22, ISO 16620-2, EN 16785-1:2015, EN 16640:2017 follow the Stuiver and Polach paper [1] and require correction for isotopic fractionation. However, following Stuiver and Polach in the case of fossil/biological carbon mixtures leads to systematic error. I will present the results of modelling the systematic errors, as well as a solution to this problem.

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Possibilities and limitations of materials used for radiocarbon dating using the example of the Cordillera Vilcabamba area (Peru)

Dominika Sieczkowska^{1,2}, **Andrzej Rakowski**¹, **Jacek Pawlyta**³ and **Jose. M. Bastante**⁴

1 - Silesian University of Technology, Institute of Physics -CSE, Krzywoustego 22B, 44-100 Gliwice, Poland

2- Center for Andean Studies, University of Warsaw, Krakowskie Przedmiescie 26/28, 00-927 Warsaw, Poland

3 -AGH University of Science and Technology, Faculty of Geology, Geophysics and Environmental Protection, Mickiewicza 30, 30-059 Kraków, Poland, Warsaw, Poland

4 -Peruvian Ministry of Culture, Dirección Desconcentrada de Cultura del Peru, PParque Arqueológico Nacional de Machupicchu, Peru

Radiocarbon dating is an extremely useful tool for the chronological study of archaeological cultures. However, using it when dealing with very short periods can be problematic, where the accuracy of a few decades can change everything. The proposed case-study for the issue at hand is the Inca culture operating in the Andes in the 14th and 15th centuries in a particularly important area for it, namely the Cordillera Vilcabamba in Peru. The research issue focuses on the possibilities and limitations of the samples of excavation materials used for the analysis. For this reason, distinctions by dated material will very often be based on its ethnographic, historical or political limitations.

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Late Quaternary evolution of piedmont faults in the foreland basin of Darjeeling-Sikkim Himalaya

Atul Kumar Singh¹, Pankaj Kumar¹, Sundeep Chopra¹ and Manoj Jaiswal²

¹ *AMS and Geochronology Group, Inter-University Accelerator Centre, New Delhi, 110067, India*

² *Department of Earth Sciences, Indian Institute of Science Education and Research, Kolkata, 741246, W.B., India*

The collision of the Indian and the Eurasian plates resulted in the formation of the Himalaya. This collision also resulted in the formation of south propagating fold and thrust belts. Main Frontal Thrust (MFT) is considered to be the youngest of the thrusts, but faults younger than the MFT have been reported in the Salt Range, Pakistan, and in the Western Himalaya, India. In this work, we have identified a fault in the foreland of the Darjeeling-Sikkim Himalaya known as the Baradighi scarp. Nakata [1] reported the scarp for the first time but little work was done to understand its evolution. Baradighi scarp is a 30 m high scarp to the south of the MFT, locally known as the Chalsa fault. In this work, we have used Accelerator Mass Spectrometry (AMS) Radiocarbon and Optically Stimulated Luminescence (OSL) dating methods to develop a robust chronology of the geological events. In addition to this various morphometric and geomorphic index have been used to understand the evolution of the drainage basin.

The AMS and OSL dates show that the Baradighi fault was first active around 25 ka. It must have been active in the recent past also as reflected by some soft sediment deformations in recent sediments from the Indong stream. The geomorphic indices used to understand the tectonic activity in the region were the Asymmetry factor (AF), Basin elongation ratio, Hypsometric Index, etc. The AF shows that most river basins are tilted towards the west. Basin elongation ratios show that not only E-W faults are affecting the drainage system but there are some N-S oriented lineaments that are controlling the fluvial system in the region. Hypsometric curve and index on the contrary show that the region is not tectonically active this is because a major portion of the rivers under study is in the unconsolidated Quaternary sediments which are easy to erode and thus the signature of upliftment events get erased. In this work, we propose that in order to understand the active tectonics in a region, smaller rivers should be studied since the chances of preservation of the signature of tectonic events are high as compared to larger rivers. We have also dated the fault using two different dating methods viz. AMS radiocarbon and OSL and these ages are in good agreement with each other.

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AMS radiocarbon dating for the chronological sequence reconstruction of the mural paintings in Shanxi Province

Y.Zhou¹ , YZ.Sun²

¹ *Austrian Archaeological Institute, Department of Historical Archaeology, Austrian Academy of Sciences, Vienna, Austria*

² *Department of Mural Painting Conservation, Chinese Academy of Cultural Heritage, Beijing, China*

Shanxi Province is renowned for its ancient Buddhist and Taoist temples, adorned with a vast collection of mural paintings. The paintings cover a long and uninterrupted periods from the Tang to Qing Dynasty (618-1912CE). Many of them are considered as unique examples of Chinese mural paintings of their periods, contributing to Shanxi's reputation as the "Eastern Mural Painting Museum".

During the 2022 survey of Shanxi's mural paintings in 231 temples, we found a significant number of paintings were repainted with several layers executed in different periods. However knowledge about the execution historical context of these paintings are limited, since the archival documentation specifying the painting periods was lost in most of the cases. By radiocarbon dating the organic binder applied in the mural paintings through Accelerator Mass Spectrometry, this proposed study aims to establish a clear chronological sequence of these mural paintings and the their repaintings. The dating results will enhance our comprehension in the development and transformation process of Chinese mural art. Integrated with art history, paint material composition and execution technology characterization, this research will also contribute to revealing how diverse cultural, political, and economic conditions have influenced the style, technique, and material that applied in mural paintings though time.