



IAEA fostering of the development and applications of nuclear analytical techniques for Heritage Science

Léna Bassel¹, Alessandro Migliori², Roman Padilla-Alvarez², Aliz Simon¹

¹ Physics Section, Division of Physical and Chemical Sciences, International Atomic Energy Agency, Vienna International Centre, Wagramer Strasse 5, PO Box 100, 1400 Vienna, Austria

² Nuclear Science and Instrumentation Laboratory, International Atomic Energy Agency Laboratories, A-2444, Seibersdorf, Austria

ABSTRACT

The IAEA Physics Section is strongly involved in the development and utilization of accelerator-based analytical techniques, which are powerful tools for the characterization of cultural and natural heritage objects and materials. Various activities are carried out with the purpose to build capacity, strengthen capabilities, transfer knowledge and foster networking in the field of heritage science. In addition, access to different X-ray fluorescence spectrometers and other analytical techniques is provided at the Nuclear Science and Instrumentation Laboratory (part of the IAEA Physics Section), and access to ion beam accelerators and synchrotron light is facilitated thanks to collaborations with Ruđer Bošković Institute (RBI) in Croatia and the Elettra Sincrotrone facility in Italy, respectively. Member States are also supported on their Research and Development programmes, as well as through the technical cooperation projects. This paper aims to provide a broad overview about how the IAEA Physics Section is engaged in the field of heritage science, promoting the safe, reliable, and effective use of ion beam, X-ray and neutron-based techniques for the characterization and preservation of cultural and natural heritage through its global networks and partners.

Section: RESEARCH PAPER

Keywords: IAEA; nuclear analytical techniques; heritage science

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Corresponding author: Lena Bassel, e-mail: L.Bassel@iaea.org

1. INTRODUCTION

The IAEA Physics Section fosters the utilization of nuclear analytical techniques including ion beam analysis (IBA), accelerator mass spectrometry (AMS), synchrotron and neutron-based techniques, to support fundamental and applied research, as well as to provide capacity building and training world-wide in the field of heritage science.

This paper intends to provide insight into the IAEA activities in support of the characterization of cultural heritage objects, with some emphasis on how the involvement and collaborations with the IAEA could be enhanced.

There are various tools and mechanisms to foster the application of nuclear science and technologies for characterization, dating, authentication, and provenance determination of cultural and natural heritage, such as capacity building, knowledge transfer, and providing support to the IAEA technical cooperation projects; they will be described in the first part of this paper.

Aligned to the same objectives, the activities carried out at the Nuclear Science and Instrumentation Laboratory (NSIL) in Seibersdorf, part of the Physic Section, will be presented in the second part.

2. IAEA TOOLS AND ACTIVITIES FOR CAPACITY BUILDING

2.1. Key topics

Preserving our heritage and hence understanding our own identity, history, and culture is part of our human behaviour. Nuclear analytical techniques have a strong role in it [1].

The Physics Section is strongly engaged in coordinating research on characterization, technological and methodological development for improved elemental sensitivity, as well as imaging in a multi-analytical approach. Improving the understanding and use of nuclear analytical techniques to support authentication and provenance of cultural property is crucial and the IAEA Physics Section is contributing globally to raise awareness, apply nuclear and complementary techniques

and foster collaborations in the field, not only for characterisation but also for forensics purposes [2]. In this regard, through cooperation with the United Nations Interregional Crime and Justice Research Institute (UNICRI) [3], a joint workshop was organized in November 2023 (Vienna, Austria) bringing together scientists and law-enforcement experts, extending the stakeholders network necessary to work together in this field. Such event proved to be efficient working across multiple disciplines to increase communication and knowledge sharing.

Working towards safe and comprehensive analysis of heritage objects and materials is one of the priority areas. When using highly energetic photon or ion beams at relevant currents and reduced beam size, and by scanning the beam several times over the same region of interest; the deposited beam dose during the analysis can result in irradiation side effects which should be considered when planning the analysis. It is especially crucial that such risks are assessed and discussed with the curator before the analysis. After a first Consultancy Meeting in 2013 (Vienna, Austria), the IAEA coordinated a round robin exercise to get new data on possible damage formation and recovery from various techniques. The intercomparison exercise also helped to define thresholds for safe analysis and raised awareness of this field. The results contributed to the development of best practices for scientists and curators when it comes to the investigation of objects with sources of ionizing radiations [4]. A recent review article with 100 references from this area published in the last 10 years confirms the uptake and further development of these efforts [5]. This paper provides additional guidance for radiation damage risk management and proposes both methodological and instrumental developments for in-situ monitoring of possible radiation effects with an opportunity for intervention. Such examples are detailed for Raman spectroscopy or ion beam analysis systems in the same paper. The real challenge in this field is that in several cases, the artifacts under study are precious, therefore it is always a careful trade how to gain the maximum analytical information with minimum intervention. In practice, it is very useful to have a test analysis prior to the investigation.

Real-time monitoring of possible radiation effects is also more and more widespread and part of standard practice. Despite of the recent development of this field and international efforts, as heritage materials are complex material systems, developing a theoretical model for both the description of radiation damage and its possible recovery remains a challenge. It is therefore outmost important to continue with knowledge transfer in this field to reach out the scientific community and humanities.

In addition to the activities on that topic, the first hands-on workshop was organized in November 2023 (Paris, France), including practical exercises and demonstrations. Real-time irradiation experiments and data acquisition were performed, and practical tests using photoluminescence imaging to monitor irradiation induced changes at the synchrotron SOLEIL facility and at the IPANEMA laboratory were demonstrated as well.

2.2. Coordinated Research Projects

The IAEA also supports research by bringing together research institutions from its developing and developed Member States on common projects conceived from assessment of their needs and demands. Such coordinated research projects (CRPs) foster the technical and methodological developments and bring new results to the community. The CRP “Enhancing Nuclear Analytical Techniques to Meet the Needs of Forensic Science”

(2017-2021), in cooperation with 14 Member States (MSs), brought together various experts of nuclear analytical techniques to work closely with forensic institutes and the Police. The key project results are published in a special issue of the Forensic Science International journal dedicated to nuclear analytical techniques, as a tangible contribution to research in this field [6]. As part of this international project, there was a dedicated work package on the authentication of art objects. Neutron Activation Analysis (NAA) and Particle-Induced X-ray Emission (PIXE) analysis were, for instance, performed on fake and genuine coins, with the objective to identify the original ones [7]. A particular focus was put on the authentication of paintings, with samples from a test painting distributed among the participating laboratories. Several accelerator-based analytical techniques applied on the samples contributed to access to numerous information, such as restorations tracking, which allowed to draw conclusions on the history of the test painting [8].

Furthermore, three AMS laboratories participated to an intercomparison study. AMS radiocarbon dating is indeed a powerful method for detecting art forgeries, especially thanks to the so-called bomb peak, leading to the detection of high ^{14}C content, characteristic of modern organic materials. A first set of samples consisted of recent and antique papers as well as textiles. The results evidenced excellent agreements between the laboratories and consistency with the expected ages of the samples, demonstrating the robustness of the AMS radiocarbon dating method [9]. Similarly, the high degree of reproducibility of the results obtained for a second batch composed of bone and ivory samples, highlighted how decisive the use of this method can be in forensic studies [10]. A review paper by describing the strengths and potential application areas of nuclear analytical techniques for forensic cases is also included in the special issue [11]. As one can expect, portable and table-top devices are commonly used in forensic laboratories or in the field. However, nuclear analytical techniques even in small to medium scale facilities have a great potential to provide unique analytical information for forensic cases, not available with other techniques.

In addition, the Nuclear Science and Instrumentation Laboratory coordinated the CRP “Experiments with Synchrotron Radiation for Modern Environmental and Industrial Applications” involving 18 MSs, 3 of which submitted proposals for applications in the field of cultural heritage, representing 10 % of the total beamtime allocated during this project [12]-[13]. More information on the IAEA CRPs can be found at [14], also the ones in planning phase and open for proposals.

2.3. IAEA Collaborating Centres

In support of these programmatic activities related to cultural heritage, the IAEA Physics Section has established since 2021 as Collaborating Centre the University Paris-Saclay, France, and the Australian Nuclear Science Technology Organization (ANSTO), Australia. In 2023, the Centre for Ion Beam Applications (CIBA) at the National University of Singapore, Singapore, also became a collaborating centre and cooperates in the field of cultural heritage including the Singapore Synchrotron Light Source. These partnerships are intended to enhance the use of nuclear technology in the field of characterization and preservation of cultural and natural heritage, and they play an important role in boosting activities related to heritage science, also serving as regional hubs in the subject area. Further information on the IAEA Collaborating Centres can be found at [15].

3. IAEA TOOLS AND ACTIVITIES FOR KNOWLEDGE TRANSFER AND NETWORKING

To exchange ideas and knowledge, share best practice on the application of nuclear analytical techniques for cultural heritage, an inter-disciplinary approach is applied and closely involves also experts from humanities. Their contribution is crucial to the enhancement of scientific-technological knowledge and public acceptance. To this purpose, the IAEA regularly organizes events and develops both outreach materials and e-resources.

3.1. Events

Technical meetings, Training courses, Workshops, Schools, and Conferences are meetings organized, with the main objective to provide a forum for all actors involved in the field of heritage science and enabling participants to acquire specific knowledge, theoretical, practical or both on a given subject. Such events are organized at the IAEA, at the International Centre for Theoretical Physics (ICTP) in Trieste, Italy, or at institutions or laboratories in Member States, with all the relevant stakeholders such as physicists, material scientists, chemists, archaeologists, conservators, or curators.

The IAEA is particularly committed to engaging both scientific community and heritage science stakeholders. Such approach allows to further enhance the nexus between Art and Science; a key aspect to work towards the World Heritage Convention adopted in 1972 which states our obligation to promote the identification, protection, conservation, preservation, and transmission to future generations of the world's cultural and natural heritage.

For the years 2022-2023, five training courses, two international conferences, four workshops and four consultancy meetings gathered about 402 participants among which 48% were women and representing 81 IAEA Member States (Figure 1). During these events, the most recent research works, and their results were presented, as well as innovative technological developments, while at the same time, international collaborations could be initiated or fostered. In particular, the IAEA Workshop on Innovative Approaches of Accelerator Science and Technology for Sustainable Heritage Management held in June 2022 (Vienna, Austria) with the attendance of 125 participants from 55 Member States, where 54 oral presentations and 20 poster presentations were delivered, was an important event.

In addition, a joint ICTP-IAEA School on synchrotron light sources and their applications is organized online since 2020, involving several speakers from synchrotron facilities, especially from the Synchrotron-Light for Experimental Science and Applications in the Middle East (SESAME) facility. For two weeks, three modules are structured around the design, the operation and the research opportunities at a modern synchrotron light source. Cultural heritage is among the topics covered and the recent 2024 edition gathered 286 participants from 51 countries [16].

Cultural heritage was also part of the topics addressed during the International Conference on Accelerators for Research and Sustainable Development: From Good Practices Towards Socioeconomic Impact which took place in May 2022 (Vienna, Austria), with one keynote talk and three parallel sessions [17], [18].

Finally, the IAEA Webinar series on Nuclear Analytical Techniques for World Heritage were launched in autumn 2023, focusing on activities from IAEA Collaborating Centres and other partners in the field. Experts from Singapore and from

Australia have been participating in the first two episodes, highlighting the versatility of nuclear analytical techniques by presenting several case studies [19]. Among others, the colour changes of textiles from the Peranakan culture exhibited in a museum could be understood thanks to the use of XRF. On pink faded areas, the presence of bromine atoms was indeed detected leading to the identification of an eosin dye, known to be fading with time. The use of neutron and X-ray imaging methods to conduct radiographies or tomographies of objects was also illustrated with two specific collaborations of scientific institutes and museums. In particular, the exhibition entitled “The Invisible Revealed” at the Museum of Applied Arts and Sciences in Sydney, Australia, showcased 45 objects examined at ANSTO's facilities, as a result of their partnership.

3.2. Publications and outreach materials

The Physics Section maintains a thematic portal for accelerators, including databases on accelerators facilities available worldwide -324 linear ion beam accelerators from 56 Member States and 60 synchrotron light sources from 26 Member States registered so far- as well as one database on X-Ray Fluorescence laboratories (1314 in 119 countries, out of which at least 172 laboratories in 49 countries carry on analyses of cultural heritage objects and materials) [20]. These databases, offer a broad overview about the availability of such facilities in MSs. A specific page on the IAEA Accelerator Knowledge Portal was designed to serve the scientific community highlighting the use of accelerator-based techniques for cultural and natural heritage [21].

The IAEA Bulletin on Accelerators published in May 2022 includes two articles on cultural heritage applications: one reporting on a bronze statue found in the Adriatic sea and its advanced characterization contributing to draw some conclusions about its origin, and the other on the ability of accelerator based-analytical techniques to detect art forgeries illustrated by a study on potential forged Impressionist and Pointillist paintings, clearly identified as modern thanks to AMS

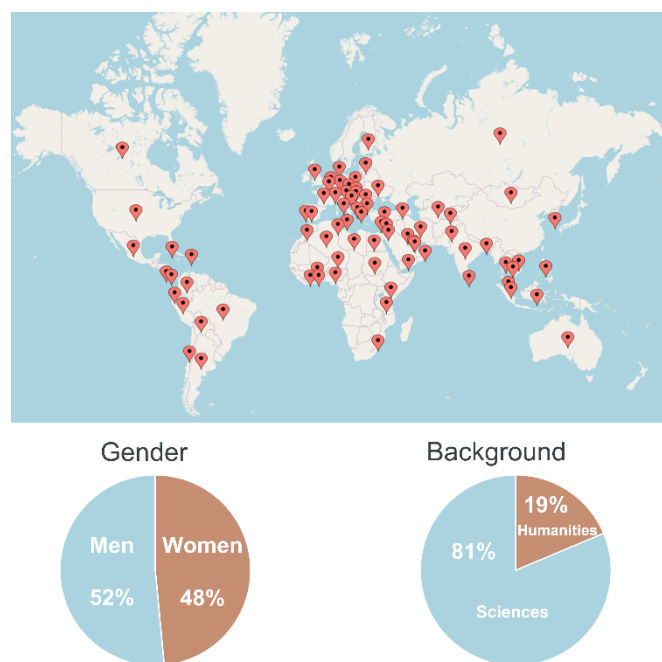


Figure 1. Member states, gender, and background distribution for the total number of participants attending events related to cultural heritage for the years 2022-2023.

radiocarbon dating analyses [22]-[25]. For a wider audience, regular contributions are provided for articles and web stories published by the IAEA Office of Public Information and Communication, shedding light on specific activities or significant achievements [26].

Finally, side events organized within the IAEA's General Conference intend to raise awareness of this important and rich topic.

3.3. Electronic resources

The IAEA learning platform provides free of charge e-learning courses in various fields [27]. In early 2023, a course on accelerator mass spectrometry-based radiocarbon dating for heritage and forensic science was released including six modules [28]. Additional e-learning courses in the field of cultural heritage include Portable X-ray techniques for characterization of valuable archaeological and art objects, Neutron imaging and a package of courses on criminal forensic science. In overall, more than 900 scientists benefited from these e-learning.

The Nuclear Science and Instrumentation Laboratory is also highly involved in the development of electronic resources and has released dedicated lectures, as well as XRF-related practical demonstrations, covering topics like the preparation of solid samples for XRF analysis or assessing the elemental distribution using micro and confocal XRF [29]. Videos introducing two Elettra Sincrotrone beamlines (and related analytical techniques) and IBA methods practical demonstrations about PIXE, Rutherford Backscattering Spectrometry (RBS), Nuclear Reaction Analysis (NRA), Particle-Induced Gamma Emission (PIGE) techniques and microprobe experiments are also available for students, laboratory staff and users of these kind of facilities [30].

3.4. Technical cooperation projects

The IAEA Technical Cooperation Programme is devoted to the transfer of nuclear technology to developing Member States,

addressing the specific priorities defined by the country or by a given region. Within this Programme, projects originating from Member States are implemented at national, regional, or interregional scale. These projects focus on infrastructure and human capacity building and usually give great tool for networking as well. The regional collaborations include two pillars: a) characterisation and b) irradiation treatment and conservation. For the period 2018-2023, 10 TC projects were related to cultural heritage and involved research teams from Latin America and the Caribbean, Asia-Pacific, the Middle East, and Europe (Figure 2). The participating countries have received assistance from the IAEA in a form of nuclear instrumentation, specific reference materials and training on both measurement techniques and interpretation of results. These collaborations allow supporting activities to be conducted aimed at identifying manufacturing technology, trading routes or provenance of materials. As an example, a national project took place during two years in Malta with the objective to upgrade the technical capabilities of Malta's institutions working in the field of cultural heritage. Scientists from the Heritage Malta's Diagnostic Science Laboratory were involved, and this project included a training on X-Ray diffraction (XRD) fundamentals, scientific visits to the University of Ferrara (Italy) and to the Museum of the Opificio delle Pietre Dure in Florence (Italy) and its restoration laboratory, as well as the procurement of one XRD system, one handheld XRF device and one infrared camera [31].

In addition, there is an emphasis to share best practices and provide not only trainings but also analytical services within the regions, as the networking efforts initiated by the regional projects allowed transnational access to medium and large facilities. In the frame of a project in the Asia and the Pacific region gathering 15 countries and entitled "Harnessing nuclear science and technology for the preservation and conservation of cultural heritage", a Memorandum of Understanding was signed between Indonesia's national nuclear energy agency (BATAN) and the national archaeology research centre, to strengthen their

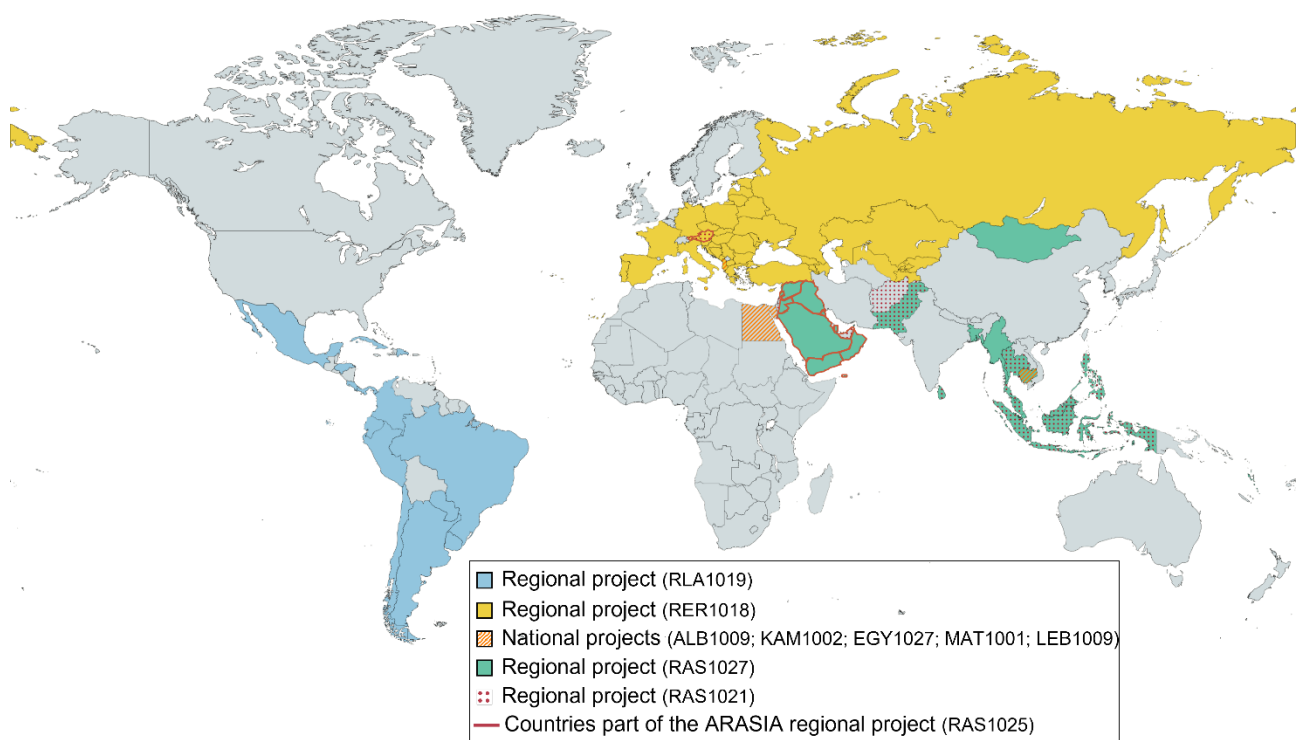


Figure 2. Technical cooperation projects related to cultural heritage for the period of 2018-2023 and involved Member States.

collaboration and providing support to countries across the region [32].

4. NUCLEAR SCIENCE AND INSTRUMENTATION LABORATORY

4.1. Mission

The Nuclear Science and Instrumentation Laboratory (NSIL) located in Seibersdorf, Austria, contributes to the Physics Section's activities by assisting MSs in strengthening their capabilities, introducing, and extending the use of nuclear instrumentation for, among other fields of application, cultural and natural heritage [33].

To this purpose, NSIL provides training and analytical services, and conducts research aimed at improving analytical performance and extending the applicability of the associated techniques. The laboratory is considered a leading training hub on topics related to XRF.

In addition, NSIL facilitates access to ion beam accelerators and synchrotrons thanks to specific collaborations with the Ruđer Bošković Institute (RBI) in Croatia and the Elettra Sincrotrone facility in Italy, respectively. An increasing number of requests from MSs involves cultural heritage projects.

4.2. Access to analytical facilities

The laboratory is equipped with a wide variety of instruments for in-situ inspection of valuable objects and for determining the elemental composition and spatial distribution in samples of diverse origin. Different XRF spectrometers are available at NSIL since more than 20 years, including standard energy dispersive XRF, a micro and a confocal XRF setup, a Full-Field XRF, a handheld XRF, as well as other variants of XRF technique and other analytical techniques such as SEM-EDS and XRD (Figure 3a). The instrumentation is adapting to scientific trends and needs and an in-house developed transportable XRF system is under transformation into a 2D XRF scanner in cooperation with the ICTP.

Recent work involved the characterization of black deposits inside the ornate Palaeolithic Ebbou cave (Ardèche, France) showing the presence of phosphorus, iron and manganese and thus contributing to the discussion on the different assumptions regarding the origin of the coatings. During 2023, in collaboration with the Austrian Archaeological Institute of the Austrian Academy of Sciences, measurements at NSIL were conducted for the characterization of Roman bricks from Noricum exploring further the building materials evolution during the Roman Imperial time [34] and of the ceramic production from Northern Mesopotamia and Arslantepe to complement ongoing research on that topic [35].

NSIL also supports the characterization of valuable objects by providing access to its available facilities and is collaborating with several museums worldwide.

The cooperation with local institutions is important too, and NSIL was involved in the study of a Mexican feather headdress, part of the collections of the Weltmuseum Wien (Vienna, Austria). This object with a long history had raised some issues to the conservators, whether some pesticides were used in the past to preserve the feathers for instance. The determination of the elemental composition of the ornaments was also an essential information to be obtained. In-situ XRF analysis with the handheld equipment on different feathers revealed the presence of elements such as arsenic, bromine and lead, attesting the use of inorganic pesticides and confirming Solid Phase

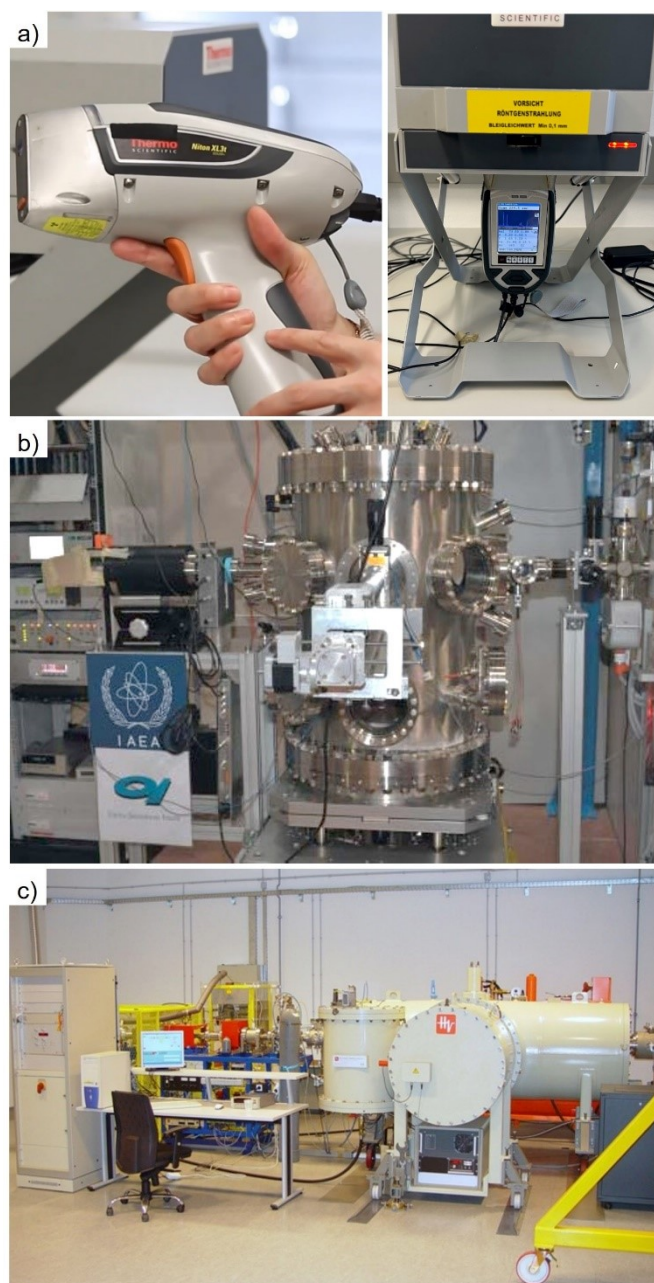


Figure 3. Instrumentation overview. a) Handheld XRF device available in NSIL; b) The IAEA Xspe end station at the XRF beamline of the Elettra Sincrotrone Trieste, Italy; c) 1.0 MV Tandemron accelerator at Ruđer Bošković Institute, Croatia.

Microextraction analysis (SPME) as well as Gas Chromatography Mass Spectrometry (GC-MS) measurements indicating traces of organic pesticides. Regarding the metal ornaments, the handheld XRF measurements allowed to confirm that the original pieces were made by alloyed gold, precisising the elemental concentrations for gold, silver, and copper. As for the replacement pieces, results showed that they were made of gilded brass, and the gold layer thickness could be estimated [36]. Another collaboration with curators from the Kunsthistorisches Museum in Vienna, Austria, led to the analysis of silver coins from the Sasanian Empire. Micro- and confocal-XRF measurements were performed and evidenced a mercury layer together with depletion of silver, whereas the same concentration of copper as in the core material was found. From these results, curators could conclude that the mercury layer was probably

formed due to the application of a mercury containing cream to make the coins shining, as it was a common practice to use mercury containing cream, mostly for medical purposes [37].

A virtual tour of the laboratory allows to have a look at the instruments and find out more information and is available at [38].

4.3. Cooperation with Elettra Sincrotrone, Italy

The IAEA has a cooperation agreement since 2013 with Elettra Sincrotrone Trieste (EST), Italy. Furthermore, EST became an IAEA Collaborating Centre in 2020. This long-standing agreement involves mainly the XRF beamline of EST that offers several complementary X-ray techniques, and for which the IAEA designed and developed the end-station, installed in Autumn 2013 (Figure 3b), and provides beamtime access to interested parties on a competitive basis [39], [40], [13]. During the whole duration of the CRP “Experiments with Synchrotron Radiation for Modern Environmental and Industrial Applications”, 40% of the total beamtime was dedicated to it.

A very recent study involved the combined use of XRF mapping and X-ray Absorption Near Edge Structure (XANES) measurements at the Fe K-edge on gold coins from the Roman Empire. Elemental concentration was obtained for the metallic alloy, and the presence of hematite and magnetite in the dirt accumulated on the coin surface was identified, providing important information for historians studying on this ancient civilization [41].

The IAEA organizes a training in Elettra Sincrotrone every year for young scientists with limited experience with synchrotron light sources and techniques that includes guidance for proposal writing, and hands-on training on three different beamlines. In addition, regarding the XRF beamline, users from developing countries can benefit from IAEA’s technical support for the writing of the proposal, or data acquisition and analysis once beamtime is allocated.

4.4. Cooperation with Ruđer Bošković Institute, Croatia

A long-lasting collaboration with Ruđer Bošković Institute Accelerator Facility in Zagreb, Croatia, also facilitates the access of IAEA collaborating partners to different ion beam analysis methods (Figure 3c). Twenty days of beamtime are available every year for research groups from developing countries. Similarly, two hands-on training courses are organized every year, the last one being held in November 2022 on “Advances in Ion Beam techniques and their applications” including cultural heritage case studies. The IAEA has initiated a five-year CRP “Facilitating Experiments with Ion Beam Accelerators” in 2018, involving 11 ion beam facilities worldwide to provide beamtime and expertise to scientists without access to accelerator facilities to conduct experiments. More than 14 experiments were conducted in total, and one project dealing with archaeology was proposed by a group from Tunisia. A comparative study using PIXE technique was performed for the characterization of archaeological pottery and raw material, looking for information about the provenance (unpublished work).

4.5. Proficiency tests

The coordination and organization of proficiency tests is provided by NSIL to improve both the technical capabilities, and the quality of the analytical results for laboratories using nuclear and related analytical techniques [42]. Participating in proficiency tests is one of the most effective ways for a laboratory to monitor and assess its analytical performance. At the same time, these

proficiency tests serve as a regular forum for discussion and technology transfer. Since 2001, twenty proficiency test exercises were organized on different type of samples, matrices, and concentration levels of given elements, according to the needs in IAEA Member States; for the current one, both soil and plant samples were distributed to 96 participating laboratories from 56 Member States. This activity contributes to guarantee the effective use of nuclear instrumentation and spectrometries, which has a direct impact when it comes to cultural heritage applications.

5. CONCLUSIONS

Characterization of cultural and natural heritage for their conservation is among the key priority areas of accelerator applications at the IAEA. This paper has shown the role played by the IAEA Physics Section in the development and application of accelerator-based analytical techniques, which are a powerful tool to gain better insight into cultural and natural heritage objects and materials. The various activities and diverse mechanisms implemented at the IAEA Physics Section including the NSIL laboratory to enhance capabilities, transfer knowledge and foster networking were highlighted. Finally, a comprehensive overview of the equipment encompassed with data related to scientific publications and case studies from IAEA global networks and collaborators were presented.

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