µ-XRF at Elettra 2.0: challenges and opportunities

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Elettra-Sincrotrone Trieste

Book of Abstracts

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The XRF beamline at Elettra: current state

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The X-Ray Fluorescence beamline, developed by Elettra Sincrotrone Trieste and operated in partnership with the International Atomic Energy Agency (IAEA), has a high-versatity optical design. Depending on the experimental needs, one can choose between an X-ray beam having high flux or high energy resolution in a broad energy range (2-14 keV) [1].

In the experimental hutch, the beamline hosts a multipurpose X-ray spectrometry endstation [2] which is well suited for any vacuum-compatible samples.

The manipulator and the set of detectors installed in the chamber are routinely exploited to perform XRF, X-ray Absorption Near Edge Spectroscopy (XANES) and X-Ray Reflectivity (XRR).
As far as XRF and XANES are concerned, when the standard geometry (45/45) is not adequate to characterise the samples, other options can be adopted, i.e. grazing incidence, grazing exit, total reflection and - our most recent addition - x-ray standing wave excitation.

The characteristics of this beamline are suitable to a wide variety of fields including, but not limited to: fundamental physics, medicine, biology, cultural heritage, environmental science.

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[1] W. Jark *et al.*, "Optimisation of a compact optical system for the beamtransport at the x-ray fluorescence beamline at Elettra for experiments with small spots", Advances in X-Ray/EUV optics and components IX, **9207**, pp. 100-111 (2014).

[2] A.G. Karydas *et al.*, "An IAEA multi-technique X-ray spectrometry endstation at Elettra Sincrotrone Trieste: benchmarking results and interdisciplinary applications", Journal of Synchrotron Radiation, **25**, pp.189-203 (2018).

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The u-XRF beamline at Elettra 2.0

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The u-XRF beamline is in the portfolio of beamlines of the 4th generation light source Elettra 2.0. In this contribution the general characteristics of u-XRF are described.
The beamline will operate in a large energy range between 2 and 17 keV. The in vacuum undulator source and the optical layout based on a double demagnification stage will feature a micrometric, high brilliant beam at the sample position. The keyword of the future studies at u-XRF is "heterogeneities"with a spatial resolution which is impossible to achieve today at Elettra.

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Welcome and presentation of the Elettra 2.0 project

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PolyX beamline at SOLARIS National Synchrotron Centre - current status

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A new synchrotron multi-purpose beamline PolyX has been constructed at the SOLARIS National Synchrotron Radiation Centre [1] in Krakow. The Solaris 1.5 GeV electron storage ring and has a critical energy of ~2 keV. PolyX is a bending magnet based beamline that provides white or monochromatic X-rays in the 4-15 keV range achieved with a hybrid double multilayer/Si(111) monochromator. The end-station is intended mainly for the spectrometry experiments with X-ray microbeam (μ-XRF, μ-XRF, confocal-XRF, μ-XAS, μ-XRD) as well as for micro-tomography in absorption and phase contrast. It is designed to enable experiments in air/helium atmosphere with scanning area up to 90x170 mm2 and to provide the capability of fast and easy switching between different measurement modes. The beam focusing is achieved by polycapillary lenses (6-100 μm FWHM @ 15 keV) and Achromatic Ellipsoidal
X-ray Mirror Lens (below 5 μm FWHM). Furthermore, the newly established submicron resolution plenoptic X-ray microscopy [2] with 3D imaging capabilities will be accessible for users. The white-beam commissioning has been performed in the third quarter of 2022 [3] and the monochromator has been commissioned in the second quarter of 2023. The beamline will be open for the first users (in the expert commissioning mode) in autumn 2023 and is expected to be fully operational in spring 2024 [4]. During the presentation the beamline design and recent status will be shown and the plan for future developments and upgrades will be discussed.

References
[1] The European Physical Journal Plus (2023) 138:10
[2] Applied Physics Letters 116 (1), 2020, 014103
[3] Nuclear Instruments and Methods in Physics Research B 538 (2023) 131–137
[4] https://synchrotron.uj.edu.pl/en_GB/linie-badawcze/polyx

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Combining soft and hard X-ray Fluorescence

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Low energy X-ray Fluorescence (XRF) microscopy is becoming a more and more widespread analytical tool, especially when combined with other imaging techniques such as Scanning Transmission X-ray microscopy, XANES or even with hard X-ray XRF.
While the first ones can be implemented in the same synchrotron end-station, the latter requires

different optics and design not only for the end-station itself but also for the entire beamline.
The presentation aims at highlighting the advantages but also the challenges of implementing soft and hard XRF microscopes in the same synchrotron facility where XRF expertise can be easily shared. Examples of possible common scientific cases and results will be presented and discussed.

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The end-station of the XRF beamline at EST: advantages and limitations

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The end-station of the XRF beamline of EST is an ultra-high vacuum chamber that hosts a seven-axis manipulator and different detectors. Thanks to this set-up and the features of the beamline, it is possible to perform standard and advanced variants of XRF technique experiments, including GI-XRF, XRR, TXRF and XAS. Some experiments carried out at the XRF beamline will be described, highlighting the advantages offered by the current set-up and its limitations, in view of the new μ-XRF beamline.

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X-Ray detectors @ FBK - current activities and future developments

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Insights into Materials with Hard X-rays: Capabilities of the BAMline

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This contribution provides an overview of the BAMline synchrotron radiation beamline, which specializes in hard X-ray spectroscopy techniques for materials research. The BAMline offers X-ray absorption spectroscopy (XAS), x-ray fluorescence spectroscopy (XRF), and tomography to study materials' electronic structure, chemical composition, and structure. Key capabilities include standard and dispersive XAS for electronic structure, micro-XRF for elemental mapping, coded aperture imaging, and depth-resolved grazing exit XAS. The BAMline enables in situ characterization during materials synthesis and functions for energy, catalysis, corrosion, biology, and cultural heritage applications.
Ongoing developments like the implementation of machine learning techniques for experiment optimization and data analysis will be discussed. For instance, Bayesian optimization is being used to improve beamline alignment and scanning. An outlook to the future, where the BAMline will continue pioneering dynamic and multi-scale characterization, aided by advanced data science methods, to provide unique insights into materials research, will be given.

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The International Atomic Energy Agency (IAEA) and the collaboration with Elettra

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The International Atomic Energy Agency (IAEA) is an intergovernmental organization established in 1957 as part of the United Nations system. Its primary mission is to promote the responsible and peaceful utilization of nuclear energy while preventing its utilization for military purposes, particularly nuclear weapons. While the IAEA operates independently under its founding treaty, it maintains accountability to both the United Nations General Assembly and the Security Council. The IAEA headquarters are located in Vienna, Austria.
In line with the Agency's mission, accelerator applications and nuclear instrumentation are among the thematic areas, where the IAEA supports its member states in strengthening their capacity to adopt and benefit from the use of accelerators. In this context, the IAEA collaborates with designated Member State institutions hosting accelerator facilities under the so-called IAEA Collaborating Centre scheme which helps reach important targets of the United Nations'Sustainable Development Goals. Elettra-Sincrotrone Trieste is one of the IAEA Collaborating Centres. In addition, a cooperation agreement between the Elettra and the IAEA was signed with the aim to facilitate access the Elettra's X-ray fluorescence beamline, which hosts an ultra-high vacuum chamber that was funded and is operated in partnership with the IAEA.
In order to support its Members States in benefiting from accelerator-based technologies, the IAEA applies certain tools and modalities, such as Technical Meetings, Coordinated Research Projects, Training Workshops, Courses, and dedicated Schools. The Agency additional supports national, regional, or interregional Technical Cooperation projects for capacity building via expert missions, training of personnel, and purchase of equipment. Moreover, the IAEA Physics Section implements various activities focusing on assisting Member States in operating and maintaining their accelerator facilities and installing nuclear instrumentation as well as in carrying out feasibility and infrastructure assessment studies aiming at establishing new accelerator facilities.
In response to Member State needs and requests, the Physics Section has recently prepared a feasibility study for an ion beam accelerator facility (IBF) at the IAEA laboratories in Seibersdorf in order to assess the interest of Member States in using this facility. Forty Member States have quantified their needs through replies to a properly designed questionnaire. The analysis of the questionnaires showed high demand in training in accelerator technologies and associated Ion Beam Analysis (IBA) techniques, as well as in analytical services in almost all areas of IBA applications. An appropriate accelerator design, matching the IAEA's programme for capacity building and provision of products and services across many fields of interest for the Member States, was identified.
This presentation aims to introduce the IAEA and share information about its tools and activities that support accelerator-based research and applications, including the longstanding collaboration with Elettra-Sincrotrone Trieste. Furthermore, we will provide details regarding the feasibility study and the instruments and facilities that will be made available through the IBF project.

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Applications of synchrotron based micro-XRF techniques in plant biology

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The development of synchrotron radiation-based spectroscopy and microscopy techniques has opened new possibilities to study the structure and biochemical composition of plant organs, tissues, cells

and biomolecules, which is critical for both basic and applied research.
In this talk, the application of synchrotron-based micro-X-ray fluorescence and X-ray absorption spectrometry in plant biology at different European synchrotron radiation facilities will be presented as tools to reveal the mechanisms of metal uptake, accumulation and metabolism in plants to better understand metal toxicity and tolerance mechanisms, for the purpose of risk assessment and restoration of heavy metal polluted sites.
In addition, food quality and safety present an important aspect, connected to mineral malnutrition on one side and metal pollution on the other. Cereals, as major staple foods, accumulate only low amounts of essential trace elements like iron and zinc, while on the other hand, they can contain elevated amounts of hazardous cadmium. Designing high mineral nutrient and low hazardous element crops present a challenge for modern plant breeders.
Synchrotron-based micro-XRF techniques with focused beam can assist in revealing element accumulation and speciation patterns at tissue and cellular levels and help breeders to choose suitable crop genotypes.

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Highlights of the IAEA coordinated research and beyond - Future Perspectives

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The International Atomic Energy Agency (IAEA), prior to the formal operation of the XRF beamline at Elettra Sincrotrone Trieste, launched a Coordinated Research Project (CRP) entitled: "Experiments with Synchrotron Radiation for Modern Environmental and Industrial Applications" under the Physics Section project "Experiments with Accelerators". The target objective of the CRP was to increase the quality and the competitiveness of Member States research in the field of synchrotron radiation-based X-ray spectrometry methods. This was planned to be achieved by supporting access to synchrotron radiation facilities, fostering the know-how transfer and strengthening the research capacity of MS in various interdisciplinary applications.

This contribution aims to highlight the achievements of the IAEA CRP which have generated high quality results in analytical applications of emerging interest in various scientific domains such as: characterization of structured novel materials for energy storage and conversion technologies, environmental monitoring of contaminants in air, water and biological samples, cultural heritage and preventive conservation, food products security and advances of the current level of knowledge concerning fundamental x-ray interactions and parameters. The follow up activities and engagement of the groups initially participating in the CRP will be also presented and their future perspectives with respect to the Elettra 2.0 upgrade of the XRF beamline will be discussed.

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Developments of mobile X-ray methods at the XRAYLab/ISPC in the Italian node of the European Infrastructure for the Heritage Science (E-RIHS)

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The XRAYlab group of the ISPC-CNR in Catania conducts scientific activity in the Italian node of the European Infrastructure for the Heritage Science (E-RHIS) providing access to advanced mobile X-ray based techniques for non-invasive cultural heritage applications. Recently, in the framework of the infrastructural project SHINE (StrengtHening the Italian Node of E-RIHS), the group upgraded several analytical techniques in operation in the laboratory. A new 3D array of 6 SDDs (total active area equal to 300mm2) operated in parallel was installed in our MA-XRF/micro-XRF scanner based on real-time technology. The novel detection system significantly improved the sensitivity of the device allowing the acquisition of pixel XRF spectra during the scanning sessions by maximizing the measured vs. the input count rate while minimizing the dead time with respect to a single detector setup under a 6-time beam intensity. In addition, a novel mechatronic allowed us to increase the scanning speed up to 150 mm/sec, and to record XRF pixel spectra with a dwell-time down to 5ms in a fast continuous mapping. The lateral resolution achievable by the device in macroscopic contexts (i.e., large dimension paintings) is down to 50um for the MA-XRF and 7um for the micro-XRF (achieved with modular optics installed in the primary X-ray source - i.e., a microfocus Rh-anode tube of 30W power). Finally, the scanner a new software suite was programmed in the scanner with the ability of analyzing XRF pixel spectra by using AI models and providing the elemental distribution images on-the-fly during the scanning. MA-XRF/micro-XRF investigation is now supported by two ancillary mobile techniques developed in the framework of SHINE, namely CXRF and MA-XRPD, providing complementary information on the artworks investigated. A 3D mapping with CXRF is typically used to investigate stratigraphies and MA-XRPD for gaining specific information on the (polycrystalline) nature of materials under study. Some compelling applications are presented and discussed.

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The XAFS/XRF beamline at SESAME: opportunities for emerging countries and complementarity with the Elettra present and future instruments.

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The XAFS/XRF beamline was the first beamline to enter the operational phase at SESAME in 2017. It is a highly demanded and highly productive beamline. It has received in kind support from Germany, UK, Italy and IAEA and allows to perform both x-ray absorption and x-ray fluorescence measurements over a wide photon energy range. Synergies with Elettra beamlines will be highlighted as well as the complementarities between ongoing development projects at the two facilities.

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Understanding the spontaneous degradation of painted works of art: multimodal and multi length scale characterization using X-ray beams

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The AXIS research group of the University of Antwerp (BE) makes use of different synchrotron X-ray microprobe beamlines in Europe: ID21 at ESRF for low energy micro-XRF and -XANES, P06 at PETRA-III for high energy micro-XRF, -XANES and -XRD, the PUMA beamline at SOLEIL for micro-XRD and the micro-XAS beamline at SLS for 2D and 3D micro-XRF. The application area mainly targeted is the cultural heritage/fine art sector, where our focus is on pigment use and pigment degradation in artworks by 15-20th c. artists. These studies usually are done in collaboration with major European or American museums such as the Rijksmuseum (Amsterdam, NL) and the National Gallery of Art in Washington DC (US). Next to that, we regularly participate in synchrotron measurement sessions aimed towards studying the behavior of radionuclides in/their effects on the environment, in particular the transfer of radionuclides from the aqueous medium towards biota. Here we collaborate with the CERAD Centre of Excellence of the University of Aas (near Oslo, NO).
Our studies on the spontaneous degradation of works of art usually comprises two stages/length scales in which we first employ self-built mobile macro-level scanners (doing XRF and XRD measurements) that operate in a non-invasive manner. The elemental and crystal phase maps that result usually provide a wealth of information on the materials used by the artists to create the artworks and on their state of preservation. However, to obtain more complete insights into the chemical transformations that have occurred at and below the surface of painted artworks, sampling of the paint and (sub)microscopic mapping XRF, XANES and/or XRD of the components of the resulting paint stratigraphies is highly relevant in many cases.
Relevant examples of studies involving 13th -20th c. wall, panel and canvas paintings will be presented. These will be used to underline that, next to the elemental maps provided by macro- and micro-level XRF, in the majority of cases, more compound- or oxidation state-specific information is required to answers questions related to artwork degradation. This makes it almost in all cases necessary to employ one or more companion techniques together with macro/micro XRF. Preferably these measurements are all done simultaneously in the same multimodal synchrotron beamline.

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Scanning X-ray Microscopy at the Hard X-ray Micro/Nano-Probe Beamline P06 (DESY) for Bio and Bio-Medical Applications

Author: Gerald Falkenberg[None]

he Microprobe experiment at the PETRA III beamline P06 is a versatile setup for scanning X-ray microscopy with X-ray fluorescence, X-ray absorption spectroscopy and X-ray diffraction / coherent scattering contrasts. It is used for studies in materials sciences, catalysis, cultural heritage, (asteroid-) mineralogy, etc., but the largest share of beamtime is given biological and bio-medical applications. The ability to collect megapixel images in less than an hour facilitates series of 2D images for full 3D fluo-tomography and spectro-microscopy. Samples can be chemically fixed, freeze dried or even fresh (unfixed). The golden standard, however, is shock-freezing and measurement in the frozen-hydrated state in order to avoid beam damage and artefacts of element re-distribution. A cryogenic sample transfer protocol is available both for
measurements under a nitrogen cryo-stream or in a cryogenically cooled UHV chamber.
Examples are presented for various sample types (tissues, bone, teeth, cells), scanning modes (fast 2D, tomographic 3D), and sample preparation techniques (frozen-hydrated, unfixed, chemically fixed).

The figure [see full version of this document in the attachments] summarizes results of a study assessing cellular uptake of exogeneous coenzyme Q10 into human skin [1]. Zinc (cyan) and iodine (magenta) elemental maps for the full scan collected in high flux mode (a,b) and the fine scan of 3 selected cells (c,d), indicated by the blue rectangle in a), collected in high resolution mode. The direct comparison of iodine and zinc emphasizes the homogeneous distribution of iodine among the cytoplasm and agrees with the expected Q10 localization. The large number of individually resolved cells allowed to deduce the correlation between iodine mass per cell and projected cell area. A mean value of 46.5 fg iodine/cell was found with a cell-to-cell uptake variation of 10.7 fg/cell.

[1] T. Staufer, G. Falkenberg, D. Brueckner, et al., Antioxidants, 2022, 11, 1532.

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X-ray microspectroscopy at ESRF beamline ID21: applications in Plant science.

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Trace metal distribution and speciation in plants is an active research field, whether to understand metal biological roles, to improve food quality or to understand and prevent the accumulation of toxic metals in edible crops. This research domain is one of the core activities at beamline ID21 of the European Synchrotron. ID21 is a beamline dedicated to X-ray fluorescence (XRF) mapping and X-ray absorption spectroscopy (XAS) in the tender X-ray range (2-11keV), this energy range allows detecting important nutrient elements (P, S, K, Ca, Mn, Fe, Cu, Zn) as well as rare earths and pollutants (Cd, Ag, Ce, La, Gd). A brand-new X -ray nanoscope is being installed at the beamline to complement the existing microscope, and it will be soon available to users. It will offer enhanced capabilities for nano-XRF mapping, nano XAS and hyperspectral XRF mapping. This new state-of-the-art instrument will offer higher lateral resolution (down to 100 nm) with better XRF detection capacities (sub-ppm), higher acquisition speed, an improved cryogenic sample environment, preserving user-friendless thanks to a new graphical user interface. Cryo-fixed plant samples can better cope with intense X-ray beams and the elemental distributions, chemical states, and sample morphologies are close to the in-vivo state under frozen-hydrated conditions. This presentation will highlight present and future capabilities at ID21 for the plant science user community. Some examples of research done at ID21 will be used to illustrate sample preparation protocols, and data acquisition and analysis strategies.

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Capabilities of XRF at Elettra Sincrotrone Trieste in cultural heritage and paleoclimate research with the focus on elemental imaging and multivariate analysis

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X-Ray Fluorescence beamline [1] is developed by Elettra Sincrotrone Trieste in partnership with the International Atomic Energy Agency. End-station consists of ultra-high vacuum chamber housing an advanced 7-axis motorized sample manipulator stage. It operates in the energy range from 3.5 to 14 keV. Combining the tunable monochromatic X-Ray beam with the manipulator that enables different excitation and detection geometries, XRF beamline enables wide range of analytical techniques in one single facility [2]. XRF elemental characterization is possible in conventional reflection geometry (45°/45°) resulting in elemental maps with spatial resolution of 200 μm x 100 μm or in angle-dependent geometry via grazing incidence/exit XRF (GI/GE XRF) for surface analysis and depth profiling to total reflection XRF (TXRF) mode for detection of ultra-trace concentration levels in liquid samples or particulate matter.

Tunable source with the high resolving power and high flux enables the elemental imaging in broad range of research fields. Its great advantage is seen in the cases when overlapping peaks in spectra restrict the acquisition of elemental maps using conventional X-ray sources or other equivalent techniques like Particle Induced X-ray Emission (PIXE) or Scanning Electron Microscopy (SEM). Advantages of tunable X-ray source will be demonstrated on the XRF imaging of S as a trace element in the presence of Ca as a major element when it is known that Ca-K escape peak coincide with the S-Kα signal. Two case studies where S XRF maps were obtained in the presence of Ca matrix

will be presented: (i) cultural heritage study used to check the efficiency/integrity of the protective surface coating layer of the stone monument treated with ammonium oxalate [3] and (ii) ongoing study of speleothem imaging combined with stable isotope analysis as a proxy records used for the reconstruction of hydrological variably in southern South Africa.

[1] W. Jark, D. Eichert, L. Luehl, A. Gambitta, Optimisation of a compact optical system for the beamtransport at the x-ray fluorescence beamline at Elettra for experiments with small spots, Proc. SPIE, 9207 (2014), 1-12

[2] A. G. Karydas, M. Czyzycki, J.J. Leani, A. Migliori, J. Osan, M. Bogovac, P. Wrobel, N. Vakula, R. Padilla-Alvarez, R.H. Menk, M.G. Gol, M. Antonelli, M. K. Tiwari, C. Caliri, K. Vogel-Mikuš, I. Darby, R. B. Kaiser, An IAEA multi-technique X-ray spectrometry endstation at Elettra Sincrotrone Trieste: benchmarking results and interdisciplinary applications, J. Synchrotron Radiat. 25 (2018), 189-203

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