

EAA 2025 Session #119

Recent developments in HH-pXRF studies and the Global p-XRF Network (GopXRF.net)

Abstracts for 10-Minute Theme Pitches

Introduction to the session topics and to the network: "GopXRF.net" Michaela Schauer (Vienna Institute for Archaeological Science - VIAS), Luisa Dallai (University of Siena) (<u>michaela.schauer@univie.ac.at</u>; <u>luisa.dallai@unisi.it</u>)

The Global p-XRF Network (GopXRF.net) emerged after a number of meetings in 2024, including a session at the 30th EAA meeting in Rome and at the University of Vienna conference, Methodological Innovations in P-XRF Studies, where it was formalised. The network is a reaction to the increasing maturity among the user community and in light of the ever-increasing popularity and application of pXRF across the discipline of archaeology, including in academic, museum and professional contexts. The network provides an opportunity for users who are keen to examine their own practices and engage in related discussions on a number of theoretical and practical issues. It brings together a number of expert practitioners from across the working in conservation to field practitioners, and across all classes of material culture and geological materials. The aim of GopXRF.net is to share good practice, disseminate newly emerging techniques and innovative methods, provide resources and training to support new users and to develop guidelines.

Portable XRF: A Powerful Tool in Archaeometry – When You Know the Rules of Physics in X-Ray Analysis

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Ceramics are the most common artifacts in archaeological excavations and represent a precious cultural heritage that spans over thousands of years. The findings range from everyday utensils used for cooking to transportation, storage, and even writing substrates. Hence, ceramics record human travel and cultural exchange like no other material. Provenance analysis has typically been done using invasive analytical methods that are time-consuming, expensive, and limit the number and choice of samples that can be analyzed, as unique cultural heritage objects are often too precious and therefore not available for invasive analysis. Non-invasive, portable XRF-based elemental analysis offers a solution to this dilemma by allowing the instrument to be transported to the sample, reducing the risk of sample degradation.

However, generating robust data sets using portable XRF requires an understanding of the interaction of X-rays with the sample, proper sample preparation, identification of pitfalls, and



an awareness of the limitations of the technique. We will show examples and explain (1) why light element readings from portable XRF instruments are often unreliable, (2) why sample preparation is so important to obtaining reproducible data, and (3) how to type-standardize the factory calibration of any portable XRF instrument using certified reference materials to account for systematic shifts in data accuracy. With a proper approach to data collection and data interpretation based on primarily heavy elements with energy lines above 8 keV, portable XRF can provide reliable results in the laboratory and in the field.

Good practices and recommendations on geochemical soil mapping with pXRF (in the field and in the lab)

Sabina Save (Amélie études environnementales et archéologiques) (save@ameliefrance.com).

Geochemical soil mapping using pXRF is a method of increasing interest to investigate space function and spatial organisation at archaeological sites from all periods and contexts. To produce reliable and useful data, and also to shut the door to still numerous fears and critics from pXRF detractors, good practices need to be in place. PXRF data can be collected directly in-situ, in the field, given some requirements on weather and soil conditions. They can also be collected from sediment samples prepared in the lab if the field conditions are not ideal, if sediment archives are needed or just if it works better for your agenda. Our experience extends across a wide range of site, sediment, and environmental types, including in-the-field and laboratory measurements; assemblage sizes range from a few samples to more than 3000. We will detail good practices from our experience using different case examples. Various aspects can impact data quality and how they can be used in post-excavation. The set-up of the spectrometer is of course very important and ensures precision and accuracy of the pXRF data. Lab preparation techniques may vary and include different steps that are more or less important (drying, crushing, compacting). We will detail our preference from our experience. Weather conditions and soil conditions are crucial for *in-situ* measurement. Soil conditions will also impact the preservation and fixation of the elements in the sediment and thus need to be considered prior to any survey. Geolocalisation of the measurements/samples is a crucial aspect to soil mapping so the data can be integrated into GIS systems. PXRF data can be processed and represented in many different ways to be exploited and interpreted. We will share with the audience our common practices on this subject and will be happy to hear from other researcher's experience.

p-XRF in the Analysis of Anthropogenic Sediments: Field vs. Laboratory Protocols Keelie S. Rix (Pompeu Fabra University, Barcelona) (<u>keelie.rix@upf.edu</u>)

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This paper reports the initial results of an ongoing current investigation within the framework of the project CAMP (ERC CoG 2022, CAMP-101088842) which aims to investigate anthropogenic sediments of archaeological sites using portable X-Ray Fluorescence (p-XRF) that will allow for the generation of provide qualitative and quantitative geochemical data.



The main objective of the implementation of p-XRF is to identify human activities through chemical signatures (anthropic markers) in the anthropogenic deposits from ethnographic settlements – inhabited and abandoned – and archaeological sites.

The sampling strategy followed for this work includes sampling ethnographic and archaeological areas at a 2m grid which can be enlarged or reduced based on a number of influencing factors including the size of the area and time constraints present during sampling. Moreover, constraints on the export of a set of samples may further influence the protocols employed for the processing and analysis of samples. In addition to exploring the factors that may affect the sampling strategies of a settlement, this paper will additionally discuss the different protocols employed for field sample processing when such constraints apply vs practices followed for this project in a laboratory setting.

The analytical setup involves a case study of ethnographic samples from traditional agropastoral inhabited and abandoned BaKalanga sites collected in Maitengwe, (Northern) Botswana using the built-in calibrations of commercial pXRF devices (in this case the GeoExploration calibration implemented in the Bruker CTX portable benchtop analyzer) which were exported and analyzed in the Laboratory of of Environmental Archaeology at Universitat Pompeu Fabra (Barcelona, Spain). This study is compared to practices and protocols used on sediment samples analyzed with the pXRF in different regions.

Analytical Settings, Calibration. Comparing pXRF Data for Obsidian and Other Materials Robert Tycot (University of South Florida) (<u>rtykot@usf.edu</u>)

The now widespread use of pXRF instruments to determine the composition and geological sources of artifacts has raised some issues concerning accuracy and comparisons of published data. A large number of geological obsidian samples from different sources (with variations in their element concentrations) analyzed by the University of Missouri using INAA, LA-ICP-MS, and XRF is available for others to calibrate data from non-destructive analyses by pXRF. Theoretically, this would allow an archaeologist to make a direct comparison of data from analysis of archaeological artifacts with the values for geological samples produced by others using other instruments. Ideally, the combined data could be included in the same graphs and matches correctly made.

I address how reliable this is by evaluating element concentrations, different element ratios, and other statistical values using the data produced on the same obsidian samples from multiple pXRF, INAA, LA-ICP-MS, and ED-XRF instruments. Factors including the time length for analysis, the use of filters, the X-ray amperage, and the specific elements of interest are shown to affect the results obtained, hindering the assignment of obsidian artifacts to specific subsources in the Mediterranean and Near East. Differences between instrumental methods and specific models and detectors affect certain elements much more than others. With pXRF, the user should be careful with major low-Z elements and their secondary X-rays absorbed in air with variation due to flatness and thickness.

Overall, the precision (repeatability) and detection limits of pXRF are excellent for archaeological research. But changes over time in XRF models and detectors, analysis of



certain elements, and use of different calibration programs require great care in direct numeric or graphical comparisons of mixed data. I highly recommend analyzing geological and archaeological samples with the same instrument and settings, and over time repeating analyses of some geological samples to address instrument drift.



Expanding Use of UAV, pXRF and Artificial Intelligence (AI) in Multi-scalar Archeology Applications

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Our contribution to this round-table discussion is an effort to overcome the basic weakness of pXRF analysis – limited spatial applicability – by combining this technique with macro-scale UAV survey guided by trained artificial intelligence (AI) navigator. At Huaca Letrada, Peru, we investigate the spatial and social dimensions of copper procurement and transformation (i.e., smelting and manufacture) using: (1) UAV (drone) mapping which streamlines survey work, (2) analysis of soils, architecture and artifacts with portable X-ray fluorescence spectrometer (pXRF) to elicit high-precision view of chemical interrelationships among activities caried out, and (3) state-of-the-art machine learning/computer vision techniques to improve multi-scalar interpretation. While the innovative, non-destructive approach we advocate allows examination of the interconnectivity between materials source, production spaces and workshops, and habitation areas without compromising site integrity, our work is far from complete. Although pXRF excels in micro-scale compositional analysis it is very restricted for macro-scale spatial analysis that can be augmented using complementary UAV and AI tools that excel in large-scale spatial recording and nuanced interpretation. Can interconnectivity between resources, production loci or living spaces be distinguished using combined multiscalar UAV, pXRF and AI tools? Can pXRF implemented to examine spatial distribution of metalworking areas (and their byproductes) be fine-tuned using AI methods? We present our vision for how such methods can be both strategically implemented and standardized in a wide range of contexts, while at the same time evaluating both the strengths and weaknesses of these combined techniques. As members of the International Archaeological HH-pXRF Users Network, we aim to engage discussions on the use of combined pXRF and artificial intelligence tools, while contributing to the group's broader mission of establishing best practices by sharing our methods with pXRF practitioners across the archaeological community.

Portable XRF in the service of cultural heritage: Some cases studies highlighting its merits and limitations

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Portable XRF is a versatile analytical method that can be deployed for speedy, sensitive, multielemental, non-destructive and non-invasive analysis of diverse cultural heritage materials. These features have also made it suitable for on-site and in-situ investigations of numerous heritage artefacts, monuments and buildings. In this contribution, the application of this technique for investigation of varied materials like archaeological soils, paintings, metals, mortars, lithics and slags would be demonstrated pointing out the multifaceted advantages and drawbacks of the methodology along with the importance of complementary techniques. Taking painting analysis as a case in point, pXRF is routinely applied, mostly for the qualitative identifications. However, the data collected could be utilized even in a more informative manner considering its quantitative aspects. These are often overlooked in the investigation of paintings. They can be used for mapping of the spatial distribution of the chemical



elements of interest from systematically selected spots of paintings. In view of several painting types in different settings and sizes, the benefits of using pXRF for elemental mapping purposes are of paramount importance. Furthermore, the quantitative data acquired can be subjected to multivariate statistical data analyses, extracting very useful information about the correlation between the chemical elements detected and the spots of analyses from the same painting or different ones. These results can be displayed in a graphical manner for simplified presentation of the large multi-elemental data thereby enhancing interpretations and disseminations of the results. Besides being crucial in documentation of the alteration products, painting materials used originally and in later interventions, such approach can shed light on many aspects of the paintings. It facilitates art historical investigations and conservation interventions among other applications. The same hold true when it comes to extracting more valuable information from pXRF as it is employed for studying other materials of cultural heritage.