dating from *ca.* 1550 BC onwards; of particular interest is the site at Armana which was occupied for a very short period of time (1350-1325 BC). Amarna is located in middle Egypt and was the seat of reigning God-King Akhenaten and Queen Nefertiti. Despite the prevalence of faience, the chemical makeup and manufacturing techniques used to produce it remain somewhat enigmatic.

The work presented herein utilized X-ray absorption spectroscopy (XAS) and synchrotron powder diffraction (PD) techniques to examine a range of faience objects obtained from collections acquired in Amarna, in addition to a selection of faiences prepared in the laboratory. The artifacts for this study were provided by the Aegyptisches Museum, Berlin. Faience for comparison was synthesized using a base mix of 80 wt-% SiO_2, 14 wt-% $NaHCO_3$ and 3 wt-% $CaCO_3$. Before firing at 900°C, Cu colorant was added to the equivalent of 3 wt-% Cu. Copper sources included bronze, corroded bronze, various natural minerals (malachite, azurite, turquoise, dioptase, chrysocolla, atacamite, etc), and laboratory chemicals (CuO, Cu_2O, $CuCO_3$, $CuSO_4$, etc).

XAS data were acquired at Beamline C, DORIS, HASYLAB. These data showed that all spectra acquired from the faiences were very similar, regardless of color, Cu concentration, or Cu source. However, analysis of the subtle differences between spectra reveal Cu coordination can be described as, either, i) a distorted Cu-O octahedron with some Si present in a fairly weak second coordination sphere, or ii) exhibiting a Cu-Cu second sphere of coordination. The former is consistent with data from glasses or related materials and the later suggests incomplete transformation to a glassy state. Such results may open avenues to use XAS to assess firing temperatures of ancient Egyptian faiences and to provide context on levels of control in ancient faience making.

PD data were acquired using the powder diffraction beamline at the Australian Synchrotron which allowed the non-destructive examination of these precious artifacts. Data from the Egyptian faience show that the dominant crystalline phases present are quartz and tetragonal cristobalite. These results are suggestive of different maximum firing temperatures. Data acquired from samples synthesized in the laboratory, to different temperatures, affords additional information as to the mineral chemistry and thermodynamics of faience formation.

This presentation will discuss the use of synchrotron techniques for the characterization of Egyptian faience and the subsequent outcomes.

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Keywords: XAS, powder_diffraction, archeometry

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Evidence of the role of Zn AND Fe cations as dopants in lead antimonate yellow by x-ray absorption spectroscopy (XAS)

F. Dacapito,[a] L. Cartechini,[b] C. Miliani,[b] F. Rosi,[c] V. Manuali,[c] B.G. Brunetti,,[c] A. Sgamellotti,[c] *[a]CNR-IOM-OGG, c/o ESRF, GILDA CRG, 6 Rue Jules Horowitz, 38043 Grenoble, (France), [b]Centro SMAArt and ISTM-CNR, via Elce di Sotto 8, 06123 Perugia, (Italy). [c]Centro SMAArt, c/o Dipartimento di Chimica, Università di Perugia, via Elce di Sotto 8, 06123 Perugia, (Italy).*

Naples yellow ($Pb_2Sb_2O_7$) is one of the oldest known synthetic pigments. It shows a cubic pyrochlore structure, obtained by roasting mixtures of Pb and Sb oxides. Recent studies demonstrate that Naples yellow may exist also in a modified form obtained from a ternary mixture of Pb, Sb and Sn oxides ($Pb_2Sb_{2-x}Sn_xO_{7-x/2}$).

X-ray diffraction and Raman spectroscopy investigations of standard doped yellow pyroantimonates provided evidence that, in general, doping cations (such as Sn^{4+}, Zn^{2+}, Fe^{3+} or exceeding Pb^{4+}) induce significant structural modifications of the pyrochlore lattice of the pigment, suggesting that ternary cations enter the octahedral sites replacing Sb^{5+} ions.

Here we report on the results obtained by XAS investigations carried out at the GILDA beam-line of ESRF (Grenoble, FR) on the role played by Zn and Fe cations in modified Naples yellow, characterising the ions' local properties (interatomic distances, coordination number and oxidation state). The XAS study has been non-destructively carried out on different standard yellow pyroantimonates, as well as on Renaissance ceramic shards from the collection of the *Musei Civici di Pesaro* (Italy). XAS measurements at the Zn-K and Fe-K absorption edges evidenced that Zn and Fe enter the antimonate structure. A*b-initio* structural simulations based on Density Functional Theory were used to simulate both the structure around the metal (via a conventional structural relaxationl) and the EXAFS spectra via a DFT Molecular Dynamics. The latter represents a novel and promising method for the analysis of point defects in crystals by EXAFS. The comparison with theory permitted to establish that in both cases the metal occupies the site of Sb. The same structure has been also observed for Zn cations in the yellow pigment of a Renaissance ceramic shard.

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Keywords: archeometry, EXAFS, defect

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Practical experience with powder XRD microdiffraction in forensic science field

Marek Kotrly, *Institute of Criminalistics Prague (Czech Republic).* E-mail: kup321@mvcr.cz

X-ray powder microdiffraction is a very valuable method for exact phase analysis. The size of the area being analyzed – approx. 100 μm is roughly comparable to areas that are studied by methods of optical and electron microscopy.

Most of the examinations in forensic science deal with determination, description and comparison of practically any substances that can come into contact with persons or objects. In this context the potential of X-ray methods is critical. Of course, not even XRD methods are a panacea and are routinely used in combinations with other methods (namely SEM-EDS/WDS, Raman microspectrometry, optical microscopy, XRF, FTIR etc.).

A recent trend in the forensic sphere is determination of the phase by at least two independent methods. The outcomes of the examinations are the grounds for bodies responsible for penal proceedings; i.e. for deciding the issues of guilt and punishment, and therefore they have to possess maximum credibility. The role of XRD methods is irreplaceable here because they allow phase analysis on a physically different basis than the majority of standard analytical methods for inorganic and organic bases.

Thorough testing of the potential and comparison with other possible diffraction arrangements was carried out - classic reflection (including collimators), rotary capillary, and transmission arrangement - during the introduction of the X-ray powder microdiffraction into the analytical routine use. Tests have been carried out for the arrangement with rotary capillary in order to determine the optimal capillary diameter with respect to the signal strength and FWHM value. Zero-background silicon plates are commonly used for imaging in reflection geometry. Standard plates are non-conductive and this fact in some