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Speciation and traceology of copper in two selected "Egyptian blue" pigments. A combined μ-XRF and μ-XANES study.

F. Farges (1,2), and M.-P. Etcheverry (3,4)

(1) Laboratoire des Géomatériaux, Université de Marne-la-Vallée, France (farges@univ-mlv.fr/FAX : +331 49 32 91 37), (2) Department of Geological and Environmental Sciences, Stanford University, USA, (3) Centre de Recherche en Physique Appliquée à l'Archéologie, Université de Bordeaux 3, France, (4) Now at Laboratoire de Recherche des Monuments Historiques, Champs-sur-Marne, France

Combined μ-XRF and μ-XAFS experiments were performed on two historical Egyptian blue pigments from Karnak (Egypt, dated 1500 BC) and St Romain-en-Gal (France, dated 1^{st} c. AD) using the 10.3.2 beamline at the Advanced Light Source (Berkeley, USA). A textural analysis of the samples indicates that the oldest pigment was formed by solid-state reaction. This is in contrast to the Gallo-Roman sample, which was formed from the nucleation of a melt, leaving residual glassy domains after cooling (which were subsequently weathered). The samples were previously studied by a combination of microscopic (optical and SEM), chemical (EDS-SEM and neutron activation), physical (cathodo-, and thermoluminescence), Raman-scattering and X-ray diffraction studies. The present analysis of the XRF information confirms that Cu and Ca are correlated within cuprorivaite domains identified based on their Cu K-edge μ-XAFS (both XANES and EXAFS) spectra. Large polarization effects, related to the layered structure of cuprorivaite can led to erroneous conclusions on the redox and speciation of Cu in these phases. In the Gallo-Roman sample (St Romain-en-Gal), large variations in the XAFS spectra were observed, suggesting the presence of another Cu-rich phase, formed by the weathering of a Cu-rich glass. A wavelet analysis of the EXAFS suggests that the phase is related to a defective tenorite (CuO) phase, which forms layers at the rim of the weathered glassy regions of the Gallo-Romain sample. The mapping of the Cu-speciation suggests, however, that these domains are scarce. Thus, the comparison of both samples provides interesting clues on the dura-

bility of two Egyptian blue pigments, prepared under different conditions.

Additional information is provided about the origins of the Cu used in the Karnak sample. The Karnak sample does not show significant amounts of Sn, suggesting that bronze was not used as a source for Cu (in contrast to St Romain-en-Gal). However, significant amounts of Ni and Cr are measured by μ-XRF in this sample, which are highly correlated to Cu. The presence of Ni and Cr suggests an ultrabasic geochemical origin of the copper ore (whereas little Zn and no Pb were detected). Such origins also rules out the use of malachite, which is highly depleted in the weakly mobile Cr, whereas Ni preferentially forms hydrous silicates on its own in ophiolites, such as garnierite. Such outcrops suggest that some hydrothermally remobilized ophiolites must have been the geochemical settings of this geochemical association. Intriguingly, a Cu-Ni metallogenic province is a strong characteristic of the Nubian shield metallogeny (South-East of Egypt), and more specifically the Gebel Gerf ophiolitic massif (Pan-African orogenesis). For instance, the mines of Gabbro Akarem, El Genena El Gharbeya or Abu Swayel are known to associate chalcopyrite with pentlandite, whereas, near the surface, gossans are formed (and therefore exposed to the surface for a long time, making prospection and mining much easier). Such related outcrops could well have been the source of the primary sulfides used to extract the Cu used to prepare the pigment found in Karnak.