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The contractile tail of bacteriophage T4 undergoes major structural transitions when the virus attaches to the host cell surface. The baseplate at the distal end of the tail changes from a hexagonal to a star shape. This causes the sheath around the tail tube to contract and the tail tube to protrude from the baseplate and pierce the outer cell membrane and the cell wall, before reaching the inner cell membrane for subsequent viral DNA injection. Analogously, the T4 tail can be contracted by treatment with 3 M urea. The structure of the T4 contracted tail, including the head-tail joining region, has been determined by cryo-electron microscopy to 17 Å resolution. This 1200 Å-long, 20 MDa structure has been interpreted in terms of multiple copies of its approximately 20 component proteins. A comparison with the metastable hexagonal baseplate of the mature virus shows that the baseplate proteins move as rigid bodies relative to each other during the structural change.

**Keywords:** bacteriophage T4, infection, conformational changes

#### MS58 CRYSTALLOGRAPHY AND UNDERSTANDING OF CULTURAL HERITAGE

**Chairpersons:** Salvatore Siano, Henk Schenk

##### MS58.28.1

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#### An Archaeometric Study of Lead-White Pigment and its Production using Neutron Diffraction

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In antiquity up to very recent times lead-white pigment was widely used. However the structures of its phases as well as the interaction of the lead-white pigment with oil are not well known. In this paper we look at the different production processes of the lead-white through the ages by means of historically based reconstructions (the Dutch stack method, the German and French production processes). The products involved in the different stages of the lead white production process are characterised by means of Rietveld phase analysis of neutron diffraction data. The interaction of linseed oil, produced according to historical recipes, with the lead-white pigment as well as with the hardener materials is studied using paint reconstructions under varying external conditions. Neutron diffraction can also be used in this case to obtain information on the reacted products.

**Keywords:** archaeometry, phase analysis, neutron diffraction

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#### Powder Diffraction in Art and Archaeology

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Powder diffraction is one of the most common techniques used as a tool for non-destructive investigations of archaeological objects. It enables both the study of small specimen (extracted from easel paintings, cosmetics recipients, walls of prehistoric caves...) and the direct analysis of painted fragments. Part of our activity is to apply relevant crystallographic techniques on ancient powdered materials, particularly using synchrotron X-ray and neutron radiation, supplemented by spectroscopic and microscopic methods: identification of the crystalline phases, quantitative and microstructural analysis, structure determination... Some difficulties, due to the specificity of the samples (rare, precious, multiphased,

poorly crystallised, spread on a support...) have to be controlled. We will give some relevant examples showing the applicability of powder diffraction to the study of ancient pigments[1-3]. Recent developments about data analysis routines will be presented.

[1] Walter P., et al., *Nature*, 1999, **397**, 483-484. [2] Martinetto P., et al., *Acta Crystallographica*, 2002, **C58**, i82-i84. [3] Sanchez del Rio M., et al., *Archaeometry*, in press.

**Keywords:** powder diffraction, archaeology, non-destructive analysis

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#### X-ray Powder Microdiffraction: Useful Tool in Investigation of Wall Painting Layers

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Identification of inorganic and organic compounds in colour layers helps in dating and restoring of historical paintings. X-ray powder microdiffraction (micro-XPRD) extends the possibilities of traditional optical microscopy and SEM/EDX used in microanalysis of mineral pigments in fragments of paints. Laboratory diffractometers with monocapillary primary optics and x,y,z-stage are now available for a routine analysis of 0.1 mm large samples, heterogeneities in fragments and colour layers in cross-sections with a roughly flat surface. Resulting diffractograms usually need no pre-treatment before their search-match analysis.

As an example, measurements of the top green layer in a sequence of colour layers in wall paints from the Plasy Monastery, Czech Republic, are shown. The sample was measured both in Bragg-Brentano geometry and micro-XPRD, and quartz, albite, celadonite, chlorite, calcite, and gypsum were found. Micro-XPRD showed that chlorite and quartz are a contamination from the sand used in plaster. Micro-XPRD also revealed that gypsum is a part of the salt efflorescence on the outer surface. Micro-XPRD identified celadonite as a green pigment in colour layer that would not be possible using spectral methods.

**Keywords:** micro-XPRD, mineral pigments, paintings

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#### Colouring and Opacifying Agents in Roman Glass: a Multi-technique Analytical Approach

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Many samples of opaque Roman glass of different typology (mosaic tesserae, game counters, vessels and artefacts) and colours (red, green, yellow, white and blue) coming from different Italian archaeological sites have been analysed to identify and characterize the colouring and opacifying agents.

The chemical analyses were carried out using wavelength dispersive electron microprobe (WDS-EMPA), the crystalline phases used as opacifiers were identified using both an automatic powder diffractometer (XRPD) and a Gandolfi camera. Secondary electron (SEM) and backscattered electron (BSE) images were performed to study the distribution and morphology of the opacifier particles, whereas their qualitative chemical analysis was obtained with an energy dispersive system (EDS). Moreover X-ray absorption spectroscopy (XAFS) and XPS studies have been performed on green and red samples to investigate the influence of coordination and oxidation state of copper atoms on colour and opacity of the glass. These analyses allowed to conclude that, in the most of the red opaque samples, copper is mainly present as metallic nano-clusters, accompanied by monovalent copper coordinated to the oxygen atoms of the glass framework, whereas in few ones, it is present as less stable cuprite crystals, leading to a more weathered glass. In green