Analysis of paint layers by light microscopy, scanning electron microscopy and synchrotron induced X-Ray micro-diffraction

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The most common way to analyze paint layers is the preparation of a cross-section of properly sampled specimens, where the sequence of all layers is preserved. The examination of the cross-section by light microscopy and UV-fluorescence microscopy provides frequently sufficient information about the structure of the paint layers, grain size and grain size distribution of the various pigments as well as varnish layers or organic binding media. However, for the identification of individual pigments present in the various paint layers additional investigations of the cross-section are necessary. Scanning electron microscopy (SEM) combined with energy dispersive x-ray microanalysis (EDX) has been used widely to obtain information about the elements present in the pigments as well as their distribution in the different layers. Single pigments can be identified by comparing their colour and elemental composition with standard materials known to be used in the present or past for painting artefacts.

However, many inorganic materials and some of the most interesting pigments can occur in different crystalline structures. CaCO3 – chalk, which has been often used as filler in the ground layer, e. g. can occur in the modification of calcite as well as aragonite. Therefore, x-ray diffraction analysis has been proved to be a valuable tool for the examination of the paint layers too. With common XRD it is rather difficult to carry out those investigations of specific pigments, as the thickness of the paint layers is in the range of several tens micrometers or even below. Therefore, synchrotron induced x-ray micro-diffraction at ID22 of the ESRF could be used in the present work, where the step scan resolution can be much smaller than the thickness of the paint layers.

The sample investigated consisted of 7 layers in total, whereby a thin layer of pure gold was suspected to be the uppermost layer. The sequence of the various paint layers as well as the distribution of the elements present in the pigments could be obtained from the cross-sectioned specimen. Additionally, synchrotron induced x-ray micro-diffraction analysis (XRD) enabled the identification of the crystalline structure of the pigments used for the painting. Traversing the sectioned sample through a focused x-ray beam with a size of 2 µm allows microscopic resolved analysis of the crystalline constituents within the diverse paint layers. By this, it is possible to attribute the usage of various pigment minerals within the paint layers, even including a 2 µm thick gold layer at the surface.