Identifying paint pigments in ancient masterpieces

In the art of restoring and preserving old paintings, an accurate knowledge of the pigments that were used by the painter is important in order to understand the aging effects in the pigments, and to prevent possible damage to the pigments which could occur when the top varnish is removed chemically. X-ray diffraction is an attractive analysis technique for this problem, as it is the only way to obtain structural information. From a practical point of view however, these samples provide a challenge to the diffractionist, as samples taken from the painting must of course be as small as possible.

For this particular investigation we used a small paint flake which was embedded in polyester resin and machined to expose a cross-section of the layers. The sample was placed on a micro-diffraction stage and rotated during the measurement in order to bring a sufficient number of crystallites into diffracting condition. The system used cobalt radiation, a mono-capillary with an exit beam diameter of 100 µm to define the irradiated spot on the sample, and a scanning RTMS detector, the XCelerator.

In the resulting diffractogram, the phases can be clearly recognized, showing that with this advanced detector you can obtain useful data from extremely small samples. Thanks to the PreFIX system of exchanging optical modules, you can easily exchange between mono-capillaries with different exit diameters, or even transform the system to a standard powder diffractometer in minutes.

Diffraeetogram obtained on sample 2 from an overnight scan. The phases matching the peaks in the diffractogram are shown underneath. Phases identification was done with X\textregistered Pert HighScore. No treatment was applied to the diffraction data; the hump in the beginning of the scan is caused by the polyester resin.

Sample courtesy of Petro Bežička, David Hradil and Tomáš Grygar, Institute of Inorganic Chemistry, Rež, Czech Republic.

The paint flake sample embedded in a polyester resin matrix. The US 1 cent coin is shown for size comparison.