EUROPEAN SYNCHROTRON RADIATION FACILITY
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Experiment Report Form

	Experiment title: Metal carboxylates in 15th century paintings	**Experiment number**: EC-805
Beamline: ID21	**Date of experiment**: from: 18-07-2011 to: 23-07-2011	**Date of report**:
Shifts: 12	**Local contact(s)**: Marine Cotte	*Received at ESRF:*

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Report:

A set of 15 samples has been analysed using micro-FTIR technique from altarpiece by Antigó and Borrassà painters (MNAC museum), altarpiece by Jaume Huguet (Sant Agata Chapel) and altarpiece by Martí Bernat (Fundació Gòdia). Also a set of 18 samples has been analysed using SR-XRF technique from altarpiece by Antigó and Borrassà painters (MNAC museum), altarpiece by Bernat Martorell (MNAC museum), altarpiece by Jaume Huguet (Sant Agata Chapel), altarpiece by Lluís Dalmau (MNAC museum) and two altarpieces from author unknown (MNAC museum and Museu Diocesà de Lleida). We obtained a large quantity of experimental data and we are currently working in their analysis.

Thanks to the possibilities of SR-FT-IR microspectroscopy, we could isolate and identify the different compounds from very small samples. The small quantity of material and its heterogeneous nature hamper the effectiveness of conventional methods in determining the presence of certain substances, in particular, the binding medium and reaction compounds.

Measurements for ATR of cross sections of painting samples were made and also measurements for transmission of small fragments of sample dispersed in a diamond cell were made. With regard to the analysis of ATR some difficulties were found which need to be resolved to take measurements in the future. On the one hand some of the samples were embedded in a excessively deformable polyester resin for the pressure exerted by ATR crystal. There was some difficulty to obtain maps due to problems with the system (software problems) which shut down several times.

For the measurements obtained from the cross-sections prepared with epoxy resin (more rigid than polyester resin) it was observed that there are difficulties to determine the correct distribution of compounds since the contact of the crystal along the samples was not homogeneous enough at each point. For this reason, diamond cell measurements in transmission mode were also made.

One of the objectives of the project was to be able to see how the reaction compounds are distributed in the samples, especially the carboxylates. Two examples are shown, one corresponding to a cross section and measurements of ATR (figure 1) and the other of a sample over a diamond cell (figure 2).

The *figure1* correspond to a green sample (from Antigó-Borrassà altarpiece) and the distribution of copper carboxylates (peak area 1585 cm^{-1}) principally in the first green layer where the presence of drying oil is also determined.

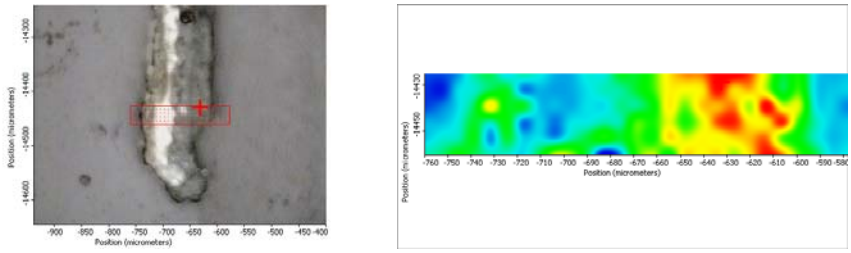


Figure 1.

Figure 2. In this preparation made in a diamond cell, the sample was spread trying to maintain the layers in the original order (gypsum preparation, lead white, pigment proteine red lake and lead white, varnish). Thus the area with lead carboxylates is discernable (peak area 1513 cm^{-1}) corresponding to the area where drying oil as binder is also determined, while in the others layers in which the lead carbonate is also present, the carboxylates have not formed, because the material with which it is in contact is a protein.

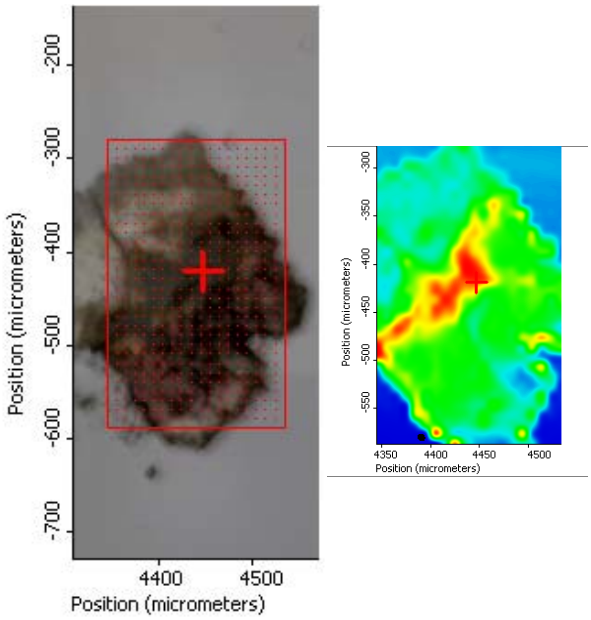
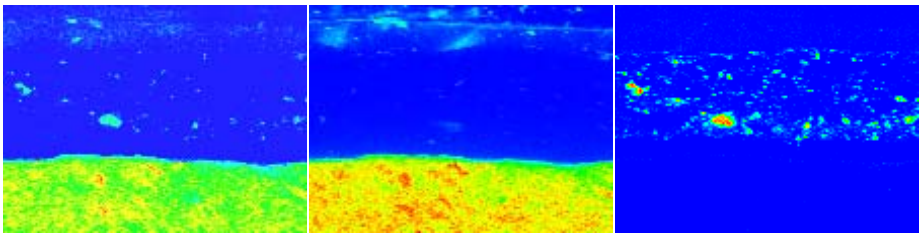


Figure 2.

With X-ray analysis measurements with a beam of 1x1μm^2 we obtained elemental distribution maps. High resolution allowed us to obtain interesting maps to help us understand the distribution of the reaction compounds which can be of great use and help in the interpretation of the formation of substances. An

example of the utility is it allows us to distinguish between environmental origin of elements and origin from the paint materials. Examples are chloride, sulphur or calcium. In the next figure 3 the sulphur distribution from the red sample (altarpiece from Lluís Dalmau) are shown. The top layer is related to calcium sulphate environmental, the middle cinnabar, HgS, red pigment, and the bottom to calcium sulphate from the preparation. The superficial calcium is also related to calcium oxalates and calcium carbonate environmental.



S Ca Hg *Figure 3.*

The datas obtained will be incorporated in several works that we hope to publish presently.