

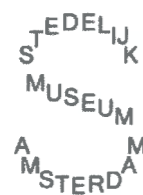


Cultural Heritage Agency
Ministry of Education, Culture and Science

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Issues in Contemporary Oil Paint

Book of Abstracts



The Getty Conservation Institute



Cultural Heritage Agency
Ministry of Education, Culture and Science

Issues in Contemporary Oil Paint

ICOP



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Symposium
28/29 March 2013
Amersfoort
The Netherlands

Symposium *Issues in Contemporary Oil Paint* (ICOP)

Amersfoort, 28/29 March 2013

Book of Abstracts

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The degradation process of chrome yellows: a focus on the reactivity between different forms of chrome yellows and the oil binder

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Chrome yellows represent a class of pigments commonly employed by painters of the late 19th-early 20th century, such as Vincent van Gogh, and that are known for their tendency to become dark. These materials are characterized by different chemical composition [PbCrO_4 , $\text{PbCr}_{1-x}\text{S}_x\text{O}_4$, $0.1 \leq x \leq 0.75$] and crystalline structure (monoclinic and/or orthorhombic), with shades that range from the yellow-orange to the paler yellow hues with increasing sulfate amount.

We demonstrated that the degradation process of chrome yellows is ascribable to a photochemical reduction of the Cr(VI) to Cr(III) when the organic is present and that the darkening behavior of these compounds is critically influenced by their chemical composition and crystalline structure.¹ Synchrotron radiation-based μ -XANES and μ -XRF investigations performed on photochemical aged paints and paint microsamples taken from Van Gogh paintings revealed the presence of several Cr(III)-compounds at the exposed surface.^{1,2} Notably, among the oil model paints prepared by employing historical and in-house lead chromate-based pigments, only those composed of a sulfate-rich orthorhombic $\text{PbCr}_{1-x}\text{S}_x\text{O}_4$ featured a significant darkening after the aging, revealing the formation of up to 60% of Cr(III) at the exposed surface. We also demonstrated that different form of the chrome yellow pigments can be clearly distinguished by means of X-ray diffraction, FTIR and Raman spectroscopy (with benchtop and portable instrumentation).³

The hypothesis whether the oil binder can act or not as redox partner for chromate ions in the paint could be assessed by investigating how the speed of the degradation of the organic binder might be influenced by the chrome yellow types and by characterizing the corresponding alteration products (such as carboxylate compounds). For this purpose a comparison of the analytical results collected from the aforementioned in-house synthesized chrome yellow model paints before and after exposition to UVA-visible light or temperature and high relative humidity conditions and those collected from a series of original paintings and microsamples will be presented.

¹ Monico, L. et al. *Anal. Chem.* 2011, 83, 1214–1223; Monico, L. et al. *Anal. Chem.* 2012, DOI: 10.1021/ac3021592.

² Monico, L. et al. *Anal. Chem.* 2011, 83, 1224–1231.

³ Monico, L. et al. *Anal. Chem.* 2012, DOI: 10.1021/ac302158b.

Derivatisation technique for enhanced FTIR-FPA imaging: localisation of oxidative ageing products in modern oil paint

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The interpretation of standard infrared spectra (FTIR) on oil based paint samples suffers from overlapping signals of the binder, the ageing products such as carboxylates and the pigments. Further it is not possible to distinguish different carbonyl containing functional groups (esters, ketones, carboxylic acids). A sample preparation and derivatisation technique, applicable to microsamples and polished cross sections, was thus developed with the aim to derivatise overlapping components and achieve an enhancement on underlying compounds. The major goal was the judgement of the state of oxidative drying and ageing of modern oil based paint such as oil, alkyd and tempera paint systems. Sulfur tetrafluoride SF_4 , a highly reactive gas, was employed to selectively convert carboxylic acids and carboxylates into acyl fluoride. This conversion product absorbs in a different region while ester groups remain intact. IR spectra after SF_4 treatment of such samples show the characteristic splitting of the carbonyl band, allowing the distinction of carbonyl containing functional groups formed during oxidative ageing of oil paint. Even interference from some pigments such as the very strong ν_2 carbonate signal of lead white can be eliminated. Typical patterns of C-H δ -vibrations of the oil become identifiable. The SF_4 treatment is surficial, homogenous across the surface, stable for several days, and can be removed by repolishing. In combination with FTIR-FPA imaging it is possible to visualise the distribution of chemical ageing products across a paint cross section at a spatial resolution of 1-1.5 microns.

Both artificially aged and real paint samples of 20th century paintings were investigated. Ageing of oil paint is a complex process due to interdependent chemical reactions running either simultaneously or sequentially. Of particular interest are oxidative cross-linking reactions, oxidative fragmentations, hydrolysis, and carboxylate formation. The different reactions are initiated or driven by light, humidity and temperature in different dependencies. The focus was set on the formation of stable oxidation products and the characterisation of the material polarity. Of primary importance are the formation and the decomposition of semi-stable peroxides. The homolytic or catalytic cleavage of these primary reaction products leads to the formation of alkyl radicals followed by different chemical reactions such as the ketone formation or the β -elimination. We found that the chemical pathways are strongly influenced by variable ageing conditions. Another factor of interest was the diffusion of oxygen during natural ageing. The 2D localisation of the different functional groups within a paint layer thus delivers information on the drying process of modern oil paint. Another successful example of SF_4 enhanced FTIR-FPA imaging was the investigation of chemical alteration within very thick oil paint layers in contemporary artwork.