

## DIFFERENCES IN COMPOSITION OF NAPLES YELLOW AND SYNTHESIS OF ITS MAIN PHASES

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Naples yellow is a historical pigment of chemical composition  $\text{Pb}_2\text{Sb}_2\text{O}_7$ , but its phase composition and variability are much more complicated. In these days, Naples yellow is not used due to the ecological and toxicological restrictions. On the other hand, there are several reasons why to study the historical pigments again even if they contain risk elements. One is the identification and assignment of the likely date of creation of artworks. To identify the specific type of pigment used in a work of art, it is important to know its main characteristics.

Synthesis of Naples yellow was studied by many authors who followed the original recipes, but these recipes do not contain complete information regarding the conditions of the calcination process. This leads to formation of multiphase products ( $\text{Pb}_2\text{Sb}_2\text{O}_7$ ,  $\text{PbSb}_2\text{O}_6$ ,  $\text{Pb}_{3+x}\text{Sb}_2\text{O}_{8+x}$ ,  $\text{Pb}_x\text{O}_y$ ,  $\text{Sb}_x\text{O}_y$ , ...). Therefore, the presented work is focused on the synthesis of phases which were the most often detected in Naples yellow, i.e.  $\text{Pb}_2\text{Sb}_2\text{O}_7$  and  $\text{PbSb}_2\text{O}_6$ . The pigments were prepared by solid state reaction in temperature range of 600–1000 °C. Besides the composition of initial reaction mixtures and heating temperature, the phase composition of the products is strongly influenced by the type of furnace used, heating duration and rate at a given temperature. Optimal conditions of calcination process of each pigment were evaluated with help of XRD analysis. Colour parameters  $L^*$ ,  $a^*$ ,  $b^*$  and particle size distribution of the pigments was analysed. X-ray microanalysis and molecular analysis by means of micro-Raman spectroscopy were performed for full characterisation of the best samples.

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