XRF AND XRD ANALYSIS OF CONVERTED LEAD PIGMENT ON A GEORGIA O'KEEFE PASTEL DRAWING

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X-ray fluorescence (XRF) spectrometry and micro-X-ray diffraction (μXRD) were used to analyze the composition of pigments on a pastel drawing, *Special No. 32*, by Georgia O'Keefe. The analyses were undertaken to identify the nature of what was suspected to be discoloration in certain red- and orange-hued areas of the drawing, and to determine if the pastel's current appearance is a significant departure from the artist's original intent.

XRF instrumentation consisted of a handheld Innov-X ED-XRF with a silver anode X-ray tube, Al filter, and Si PiN diode detector. The instrument was operated at 35 kV and 13 μA and at 15 kV and 12 μA; resolution was < 0.2 keV. The spectrometer was attached to a tripod arm that extended over the object on a laboratory benchtop (that did not contribute to the elemental signature) and positioned 2–3 mm above the surface so as not to detach any of the extremely friable media. A precise distance was maintained throughout the analyses by repositioning the drawing. Four areas also were examined using a window mask, which limited the spot size to < 5 mm. XRD analyses were performed on a Rigaku D/Max Rapid diffractometer using copper Kα radiation, 50 kV acceleration voltage, 40 mA current, chi axis fixed at 45º, omega axis fixed at 0º, and phi axis spun 360º. For these analyses, two tiny particles, about 30 μm each, were mounted on glass fibers and exposed for 180 seconds using a 0.1 mm collimator.

XRF analyses show that the drawing's masterfully layered and blended red, orange and yellow pigments are associated with the elements Pb, Cr and Ca. This suggests the presence of pigments made from lead chromates, red lead oxides, and/or lead carbonates, plus a pastel filler such as whiting ($CaCO_3$). These results cannot be strictly interpreted in a quantitative manner because of non-ideal examination conditions, as described above. However, it is shown that comparison of Pb:Cr ratios detected in select areas of the drawing, as derived from standard calibration curves, allow the more stable red and orange lead chromate pigments to be differentiated from other likely lead pigments. Red lead oxide pigment is well known to be unstable to light and air, often leading to non-uniform darkening. XRD analysis of a sample removed from a blackened stripe in a red area of the drawing in fact confirms that the darkened appearance and high Pb:Cr ratio in this case is associated with the presence of red lead oxide, minium (Pb_3O_4).

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