

Section 29 – References

Cross-section Preparation Procedures:

The samples were cast into mini-cubes of polyester resin (Excel Technologies, Inc., Enfield, CT). The resin was allowed to cure for 24 hours at room temperature and under ambient light. The cubes were then ground to expose the cross-sections, and dry polished with 400 and 600 grit wet-dry papers and Micro-Mesh polishing cloths, with grits from 1500 to 12,000.

Cross-section microscopy analysis was conducted with a Nikon Eclipse 80i epi-fluorescence microscope equipped with an EXFO X-Cite 120 Fluorescence Illumination System fiberoptic halogen light source and a polarizing light base using SPOT Advanced software (v. 4.6) for digital image capture and Adobe Photoshop CS for digital image management. Photographs and digital images of the best representative cross-sections are included in this report. UV photographs were taken with the UV-2A filter in place (330-380 nanometers excitation with a 400 nm dichroic mirror and a 420 nm. barrier filter), a B-2A filter (excitation 450-490 nm and barrier filter at 520 nm.), or a BV-2A filter (excitation 380-420 nm and barrier filter at 450 nm.) Please note that the colors in the printed photomicrographs may not accurately reflect the actual color of the samples because the colors in the digital images are affected by the variability of color printing.

The following fluorescent stains were used for examination of the samples:

Triphenyl tetrazolium chloride (TTC) 4.0% in ethanol to identify the presence of carbohydrates (starches, gums, sugars). Positive reaction color is dark red or brown.

Eosin isothiocyanate (EITC) 0.2% in anhydrous acetone to identify the presence of proteins. A yellow or yellowish-green color indicates a positive reaction.

Fluorescein isothiocyanate (FITC) 0.2% in anhydrous acetone to identify the presence of proteins. A yellow or yellowish-green color indicates a positive reaction.

Alexa Fluor 488 (Alexa) 0.02% in water, pH at 9, 0.05M borate and 5% DMF. Positive reaction color is greenish or yellowish-green with a B-2A filter (excitation 450-490 nm and barrier filter at 520 nm.)

2, 7 Dichlorofluorescein (DCF) 0.2% in ethanol to identify the presence of saturated and unsaturated lipids (oils). Positive reaction for saturated lipids is pink and unsaturated lipids is yellow.

Rhodamine B (RHOB) 0.06% in ethanol to identify the presence of oils. Positive reaction color is bright orange.

N-(6-methoxy-8-quinolyl)-p-toluenesulfonamide (TSQ) 0.2% in ethanol to mark the presence of Zn in the cast cross-section. Positive reaction color is bright blue-white.

Potassium iodide (KI) 0.2% in water to mark the presence of lead. Positive reaction color in reflected visible light is bright yellow.

Photographs were taken at 40X, 100X, 200X, 400X, and 1000X (with a 100X oil immersion objective) magnifications.

Information Provided by Ultraviolet Light Microscopy:

When viewed under visible light, cross-sections which contain ground, paint and varnish may often be difficult to interpret, particularly because clear finish layers look uniformly brown or tan. It may be impossible using only visible light to distinguish between multiple varnish layers. Illumination with ultraviolet light provides considerably more information about the layers present in a sample because different organic, and some inorganic, materials autofluoresce (or glow) with characteristic colors.

There are certain fluorescence colors which indicate the presence of specific types of materials. For example: shellac fluoresces orange (or yellow-orange) when exposed to ultraviolet light, while plant resin varnishes (typically amber, copal, sandarac and mastic) fluoresce bright white. Wax does not usually fluoresce; in fact, in the ultraviolet it tends to appear almost the same color as the polyester casting resin. In visible light wax appears as a somewhat translucent white layer. Paints and glaze layers which contain resins as part of the binding medium will also fluoresce under ultraviolet light at high magnifications. Other materials such as lead white, titanium white and hide glue also have a whitish autofluorescence.

There are other indicators which show that a surface has aged, such as cracks which extend through finish layers, accumulations of dirt between layers, and sometimes diminished fluorescence intensity, especially along the top edge of a surface which has been exposed to light and air for a long period of time.

Pigment and Fiber Preparation:

Discrete samples of pigments from individual paint layers were crushed onto microscope slides with a clean scalpel and were permanently mounted under cover slips with Cargille MeltMount with a refractive index of 1.66. Fibers were separated from of the original paper substrates and were cast onto microscope slides in the same manner as the pigments. The samples were examined with transmitted plane polarized light and crossed polars (darkfield) at 400X and 1000X (with a 100X oil immersion objective) to observe the color, morphology and optical properties of the unknown pigments and fibers. These pigments and fibers were also compared to standard pigment and fiber samples to confirm the polarized light microscopy identifications.

Color Measurement Procedures:

Uncast portions of the samples with intact areas of Period III woodwork and distemper paints were used for matching with the Minolta Chroma Meter CR-241, a tristimulus color analyzer with color measurement area of 0.3mm. This instrument has an internal, 360-degree pulsed xenon arc lamp and provides an accurate color measurement in a choice of five different three-coordinate color systems.

The exposed layers were measured three times in three different areas of the exposed target layers to establish the color coordinates for the layers established as the Period I and Period III coatings through cross-section microscopy analysis. The measurements were first generated in the Munsell color system (a color standard used in the Architectural Preservation field), and after the measurements were taken the closest Munsell color swatches from a standard Munsell Book of Color (gloss paint standards) was compared under 30X magnification to the actual samples. The measurements were also generated in the CIE L*a*b* color space system, which is currently one of the most widely accepted industry color space measuring systems.

Color swatches from the Benjamin Moore Paint Color Preview collection, the Pittsburgh Paints Voice of Color collection, the Williamsburg Color Collection and the Sherwin Williams ColorAnswers Collection were generated using comparative Munsell and CIE L*a*b* color measurements from the actual samples compared to the commercial matches to determine the best numeric and visual matches.

The Minolta Chroma Meter CR-241 (a colorimeter/30X microscope) measures in the following color systems:¹

Chroma Meter CR-241 offers five different color systems for measuring absolute chromaticity: CIE Yxy (1931), L*a*b* (1976), and L*C*H* (1976) colorimetric densities Dx Dy Dz; Munsell notation and four systems for measuring color differences.

For two colors to match, three quantities defining color must be identical. These three quantities are called tristimulus values X, Y, and Z as determined by CIE (Commission Internationale de l'Eclairage) in 1931.

Color as perceived has three dimensions: hue, chroma and lightness. Chromaticity includes hue and chroma (saturation), specified by two chromaticity coordinates. Since these two coordinates cannot describe a color completely, a lightness factor must also be included to identify a specimen color precisely.

Munsell Color System: The Munsell color system consists of a series of color charts which are intended to be used for visual comparison with the specimen. Colors are defined in terms of the Munsell Hues (H; indicates hue), Munsell Value (V; indicates lightness), and Munsell Chroma (C; indicates saturation) and written as H V/C.

¹ Derived from the Minolta CR-241 instruction manual and the Minolta "Precise Color Communication" brochure.

CIE Yxy (CIE 1931): In the Yxy (CIE 1931) color system, Y is a lightness factor expressed as a percentage based on a perfect reflectance of 100%, x and y are the chromaticity coordinates of the CIE x, y Chromaticity Diagram.

CIE L*a*b*: Equal distances in the CIE x,y Chromaticity Diagram do not represent equal differences in color as perceived. The CIE L*a*b* color system, however, more closely represents human sensitivity to color...Equal distances in this system approximately equal perceived color differences. L* is the lightness variable; a* and b* are the chromaticity coordinates

ΔE : ΔE (Delta E) is the industry measure used to determine how closely two colors match in the CIE L*a*b*. The symbol Δ means “the change in”. It is based on calculating the sum of the differences between each measure. The calculation is: $\Delta E = \sqrt{(L - L')^2 + (a - a')^2 + (b - b')^2}$. or, the color difference equals the square root of the squared sums of the differences between each of the three L* a* b* tristimulus values. Industry color standards indicate a ΔE of 1 is barely perceptible to the human eye, and ΔE of 6 to 7 is acceptable for color matches in the printing industry.