J. SUZUKI[1]
R.J. KOESTLER[2]

VISUAL ASSESSMENT OF BIOCIDE EFFECTS ON JAPANESE PAINT MATERIALS

1 Museum of Fine Arts, Boston, MA 2 The Metropolitan Museum of Art, New York

PAINT MATERIALS

ABSTRACT This paper reports on the results of experiments to test for visual changes to paint systems using a statistical binary procedure after biocidal treatments, and a comparison of the visual data with those from a spectrophotometer. Four biocides were tested: two gases—argon (Ar, suffocant for fungi and insects) and nitrogen (N_2, suffocant for insects); and two liquid fungicides—the architectural antimicrobial D/2 (Prosoco, Inc., Lawrence, KS), and an experimental pine emulsion (Tampere University of Technology, Finland). Four paint sets were subjected to the biocides: watercolor, acrylic color, egg tempera, and Japanese paint, making for a total of 52 sample sets. The visual assessment procedure provided information on tone and hue change, gloss change, and formation of precipitate. The results indicated that argon and nitrogen gas had no visual effect on any pigments; D/2 changed parts of each sample set; and the experimental pine resin caused such a major change that it was dropped from testing. The spectrophotometric results had better than 92% agreement with the visual technique.

INTRODUCTION Virtually all art materials are susceptible to microbial attack. When it occurs, an immediate response is needed to minimize the damage to the material. This response may include application of a biocidal treatment. In order to

make an informed selection of what biocidal treatment to use, it is important to have some idea of the side effects of any treatment. A technique to assess for visual change in paint samples was presented in Koestler et al. (1993). This technique has been used to assess the changes to four water-based paints: watercolor, acrylic color, egg tempera, and Japanese paint, after treatment with four different biocides —two gases—argon and nitrogen; and two liquids—an architectural biocide, D/2, and an experimental pine resin extract.

MATERIALS AND METHODS The watercolor and acrylic color used were proprietary brands (for watercolor, Winsor and Newton, England; and for acrylic color Liquitex, USA). Egg tempera was made with dry pigments (Holbein, Japan), then ground into the medium of 1:1 egg yolk and water. The dry pigments for Japanese paint were ground into the solution of Japanese animal skin glue, Sanzenbon-nikawa (all pigments were Japanese products). Each paint had 12 colors, making for a total of 52 samples sets. [TABLE 1]

Supports Watercolor and acrylic color were applied on watercolor paper (Fabrian, cellulose 100%, Cartiere Miliani Fabriano, Italy). Egg tempera was applied on a poplar plywood panel of 3-mm thickness. The panel was sized with 7% rabbit skin glue solution and prepared with gesso ground (for the lower six layers, gesso grosso; for the upper four layers, gesso sottile with 7% rabbit skin glue solution). Japanese paint was applied on Japanese paper (Kumohada-mashi, a hemp and Kozo fiber 100%, sized with animal skin glue, Uematsu Co., Japan).

Paint Application and Dryness Each paint was applied in 2.5-cm-wide strips on each support. And each paint was diluted with water in the usual painting method, and applied in about six layers with a soft-flat brush as evenly as possible. Triplicate strips were prepared for controls and each experimental sample. Control and experimental samples were stored under identical light conditions before and after treatment. Both were hung on a wall, in dim light, where they dried for about 10 months.

Biocides and Application Four biocides were tested: two gases—argon (Ar, suffocant for fungi and insects) and nitrogen (N_2, suffocant for insects); and two liquid fungicides—the architectural antimicrobial D/2 (Prosoco, Inc., Lawrence, KS), and an experimental pine emulsion (Tampere University of Technology, Finland). Argon and nitrogen gas have been used as insect suffocants while argon has recently been

[TABLE 1] Paints tested

WATERCOLOR	ACRYLIC COLOR[1]	EGG TEMPERA[2]	JAPANESE PAINT[3]	
			English Name	Japanese Name
Chinese White	Zinc White	Zinc White	Chalk	*Gofun*
Titanium White	Titanium White	Titanium White	White Lead	*Enpaku*
Alizarin Crimson	Alizarin Crimson	Rose Madder	Synthetic White	*Iwashiro*
Cadmium Red	Cadmium Red	Cadmium Red	Synthetic Pink	*Iwamomo*
Terre Verte	Chromium Oxide Green	Terre Verte	Light Red	*Taisha*
Winsor Emerald	Emerald Green	Oriental Green	Vermilion	*Shu*
Prussian Blue	Prussian Blue	Prussian Blue	Red Lead	*Entan*
Cobalt Blue	Cobalt Blue	Cobalt Blue / Deep	Verdigris	*Rokusho*
Cadmium Yellow	Cadmium Yellow	Cadmium Yellow	Synthetic Verdigris	*Shin Iwarokusho*
Raw Sienna	Raw Sienna	Raw Sienna	Azurite	*Iwagunjo*
Burnt Umber	Burnt Umber	Burnt Umber	Gold	*Ao-Kindei*
Ivory Black	Ivory Black	Ivory Black	Silver	*Jun-Gindei*

[1] Support paper was also tested. [2] Support wood board and gesso were also tested. [3] Japanese paper was also assessed.

shown to be effective on some fungi (Tavzes et al., this volume).

The argon and nitrogen gases were commercial grade (minimum 99.995% pure, actual measured value of oxygen about 10 ppm). Samples treated with Ar or N_2 gas were enclosed in Marvel Seal 360 (a heat-sealable aluminized Mylar) and brought down to an oxygen level under 100 ppm. Packets of oxygen scavenger (Freshpax D-2000 cc, Multisorb Technologies, Buffalo, NY) were placed inside each bag to maintain the low oxygen level. The humidity level was about 55%. Triplicate samples were kept in this environment for 1 month.

Triplicate sample strips of each paint sample were sprayed with the D/2, from a distance of about 3 inches, until the complete surface was covered (two to three spurts of the applicator bottle). The pine emulsion was tested on canvas and found to severely stain the canvas brown, so further testing with this product was not conducted.

Evaluation Methods Visual color change was assessed by two trained observers with the unaided eye and with a light microscope using a statistical binary procedure (as per Koestler et al., 1993). A 2.5 × 3 cm grid was superimposed on each sample, and divided into a 5 × 6 matrix, and five sites were selected by a random number technique and examined separately by the two observers. Samples were compared to corresponding control areas. Each observer looked at 5 of 30 boxes for each of the three paint samples per test. A total of 15 out of 90 boxes were looked at by each observer, percentages were calculated separately for each paint sample/observer, and then the two percentages were averaged to yield the final estimate for the area affected (see Koestler et al., 1993, for details). Changes were assessed in the following categories: visual color change (broken into tone and hue), change in gloss, and presence of any precipitate. Along with each set of binary statistical data is an impression of overall percentage change of the surface.

Instrumental evaluation of changes resulting from treatment was carried out with a Minolta CM-2002 spectrophotometer. The color values using CIE 1976 L*a*b* of control and experimental samples were measured before and after treatment. The same random field design as was used in the visual method described above was used for the instrumental method, with minor modification. A total of three areas, measured three times each, for each of the three experimental paints (27 measurements/test paint) were averaged to yield the raw data for the color calculations. The diameter of the measuring beam was 0.3 mm. Tests were performed first on samples of the paint to assess for structural color differences and orientation of the sample. These were done by predefining one direction of the samples

as 0°, measuring, as above, then rotating the sample 45° at a time so that a total of eight directions (the four cardinal and four sub-cardinal directions) were measured. Final tests were run at 0° and 90°; however, only the 0° results are presented here. The visual tone results for black or white samples were compared to ΔE^*_{ab} values, and the visual hues of color samples were compared to ΔH^*_{ab}.

RESULTS AND DISCUSSION Argon and nitrogen gas had no visual effect on the pigments. The spectrophotometric readings were also negative. The experimental pine sol resin was tested on one sample and the results were so immediately disfiguring that no further testing was carried out with this material.

The D/2 results were the most interesting; full results from the visual assessment are summarized in TABLES 2–5. Normally, it is not recommended that a water-based biocide be used on the test materials used in this study. But there is really no choice in the U.S. market, as D/2 is the only registered biocide available for use on anything related to art (in this case masonry), and since this study was interested in testing and developing the assessment methodology, its inclusion is warranted.

The visual color change has been divided into tone and hue. This methodology is a refinement of that used in Koestler et al. (1993), which had just referred to color change. Assessing tone yields information on the lightness or darkness of the pigment, while hue gives information on the similarity of color. There are many ways to describe color differences; Berns (2000, pp. 73–74) recommends using ΔL^*, Δa^*, and Δb^* for neutral tones such as blacks, whites, and grays. For chromatic samples he recommends the use of ΔH^*_{ab}, ΔL^*, and ΔC^*_{ab}. A summary of the spectrophotometric data is given in TABLES 6–9. The CIELAB formulae used in this paper are:

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}, \text{ for black, white, and gray samples;}$$

and

$$\Delta H^*ab = [(\Delta E^*_{ab})^2 - (\Delta L^*)^2 - (\Delta C^*_{ab})^2]^{1/2}, \text{ for chromatic samples.}$$

In general, D/2 caused significant changes in tone and hue in three out of four color sets (acrylic being the exception). Gloss changes occurred in all four paint sets. Sometimes the change in gloss was accompanied by an apparent precipitation on the surface. On some samples white particles (visible with light microscopy) remained on the surface, and on others there seemed to be a dried emulsion.

The acrylic sample set had the least change of the four with D/2. All of these changes were alterations in the gloss (an increase in all four cases). It is possible that the change in gloss on acrylic samples could have been countered by rinsing.

[TABLE 2] Summary of visual observations on acrylic samples treated with D/2

	VISUAL COLOR CHANGE				CHANGE IN GLOSS		PRECIPITATION	
	TONE		HUE					
	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION
Support Paper	0%	0%	0%	0%	0%	0%	0%	0%
Zinc White	0%	0%	0%	0%	0%	0%	0%	0%
Titanium White	0%	0%	0%	0%	7%	67% G	0%	0%
Alizarin Crimson	0%	0%	0%	0%	0%	0%	0%	0%
Cadmium Red	0%	0%	0%	0%	0%	0%	0%	0%
Chromium Oxide Green	0%	0%	0%	0%	0%	0%	0%	0%
Emerald Green	0%	0%	0%	0%	30%	50% G	0%	0%
Prussian Blue	0%	0%	0%	0%	3%	17% G	0%	0%
Cobalt Blue	0%	0%	0%	0%	0%	0%	0%	0%
Cadmium Yellow	0%	0%	0%	0%	0%	0%	0%	0%
Raw Sienna	0%	0%	0%	0%	0%	0%	0%	0%
Burnt Umber	0%	0%	0%	0%	0%	0%	0%	0%
Ivory Black	0%	0%	0%	0%	15%	100% G	0%	0%
Total % change	0%		0%		4%		0%	

G: glossy

G: glossy

[TABLE 3] Summary of visual observations on Japanese paint samples treated with D/2

	VISUAL COLOR CHANGE				CHANGE IN GLOSS		PRECIPITATION	
	TONE		HUE					
	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION
Japanese Paper	0%	0%	0%	0%	0%	0%	0%	0%
Chalk (*Gofun*)	13%	17% D	3%	83% Y	0%	0%	0%	0%
White Lead (*Enpaku*)	0%	0%	0%	0%	0%	0%	0%	0%
Synthetic White (*Iwa-shiro*)	0%	0%	0%	83% Y	0%	0%	0%	0%
Synthetic Pink (*Iwa-momo*)	20%	67% L	23%	17% Y	0%	0%	0%	0%
Light Red (*Taisha*)	0%	0% L	0%	0%	0%	17% M	0%	0%
Vermilion (*Shu*)	0%	0%	0%	0%	0%	0%	0%	0%
Red Lead (*Entan*)	20%	67% L	7%	50% Y	3%	100% UE	7%	17%
Verdigris (*Rokusho*)	7%	33% L	7%	17% Y	0%	17% M	0%	0%
Synthetic Verdigris (*Shin Iwa-rokusho*)	37%	83% L	27%	67% Y	0%	0%	0%	0%
Azurite (*Iwa-gunjo*)	3%	17% D	0%	0%	0%	0%	0%	0%
Gold (*Ao-Kindei*)	0%	0%	0%	0%	0%	0%	0%	0%
Silver (*Jun-Gindei*)	3%	67% L	3%	67% Y	0%	0%	0%	0%
Total % change	8%		5%		<1%		<1%	

L: light, D: dark, Y: yellowed, R: reddish, M: matte, UE: uneven

[TABLE 4] Summary of visual observations on watercolor samples treated with D/2

	VISUAL COLOR CHANGE				CHANGE IN GLOSS		PRECIPITATION	
	TONE		HUE					
	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION
Chinese White	0%	0%	0%	0%	0%	0%	0%	0%
Titanium White	0%	0%	0%	0%	0%	0%	0%	0%
Alizarin Crimson	13%	33% L	0%	0%	0%	0%	0%	0%
Cadmium Red	0%	17% L	0%	0%	0%	0%	0%	0%
Terre Verte	0%	17% L	0%	0%	0%	0%	0%	0%
Winsor Emerald	17%	50% L	3%	0% Y	0%	0%	17%	100%
Prussian Blue	10%	33% D	23%	67% Y	0%	0%	0%	0%
Cobalt Blue	40%	67% D	0%	0%	17%	100% UE	0%	0%
Cadmium Yellow	0%	0%	0%	0%	0%	0%	0%	0%
Raw Sienna	37%	67% L	0%	0%	0%	0%	13%	50%
Burnt Umber	13%	67% L	3%	0%	0%	0%	0%	0%
Ivory Black	33%	33% D	0%	0%	7%	33% UE	0%	0%
Total % change	14%		2%		2%		3%	

L: light, D: dark, Y: yellowed, UE: uneven

[TABLE 5] Summary of visual observations on egg tempera samples treated with D/2

	VISUAL COLOR CHANGE				CHANGE IN GLOSS		PRECIPITATION	
	TONE		HUE					
	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION	RANDOM FIELD	OVERALL IMPRESSION
Support Wood Board	0%	0%	0%	0%	0%	0%	0%	0%
Gesso Ground	20%	50% D	17%	33% Y	0%	0%	0%	0%
Zinc White	50%	100% D	53%	100% Y	0%	0%	0%	0%
Titanium White	37%	33% D	30%	83% Y	0%	17% UE	0%	0%
Rose Madder	0%	0%	0%	0%	0%	0%	7%	50%
Cadmium Red	0%	0%	0%	0%	0%	0%	3%	33%
Terre Verte	57%	100% L	3%	17% Y	3%	0% G	0%	0%
Oriental Green	0%	0%	0%	0%	7%	83% UE	0%	0%
Prussian Blue	37%	67% D	0%	0%	13%	100% UE	23%	67%
Cobalt Blue / Deep	63%	100% D	0%	0%	23%	83% UE	27%	67%
Cadmium Yellow	0%	0% D	0%	0%	0%	100% UE	0%	33%
Raw Sienna	90%	100% D	37%	50% R	0%	67% UE	0%	67%
Burnt Umber	60%	100% D	0%	0%	3%	50% UE	30%	83%
Ivory Black	17%	83% L	0%	0%	20%	100% UE	53%	100%
Total % change	**31%**		**10%**		**5%**		**10.0%**	

L: light, D: dark, Y: yellowed, R: reddish, G: glossy, UE: uneven

	DE*Lab FOR B&W	DH*ab FOR COLOR SAMPLES
Support Paper		
Zinc White	0.28	
Titanium White	0.20	
Alizarin Crimson		0.08
Cadmium Red		0.30
Chromium Oxide Green		0.08
Emerald Green		0.31
Prussian Blue		2.87
Cobalt Blue		0.02
Cadmium Yellow		0.43
Raw Sienna		0.17
Burnt Umber		0.18
Ivory Black	0.66	

[TABLE 6] Summary of spectrophotometry data for acrylic samples treated with D/2

		DE*Lab FOR B&W	DH*ab FOR COLOR SAMPLES
Japanese Paper			
Chalk	*Gofun*	1.00	
White Lead	*Enpaku*	0.06	
Synthetic White	*Iwa-shiro*	0.70	
Synthetic Pink	*Iwa-momo*		0.20
Light Red	*Taisha*		0.08
Vermilion	*Shu*		0.30
Red Lead	*Entan*		0.20
Verdigris	*Rokusho*		0.10
Synthetic Verdigris	*Shin Iwa-rokusho*		0.11
Azurite	*Iwa-gunjo*		0.36
Gold	*Ao-Kindei*	0.90	
Silver	*Jun-Gindei*	0.69	

[TABLE 7] Summary of spectrophotometry data for Japanese paint samples treated with D/2

	DE*Lab FOR B&W	DH*ab FOR COLOR SAMPLES
Chinese White	0.90	
Titanium White	0.62	
Alizarin Crimson		1.30
Cadmium Red		0.05
Terre Verte		0.72
Winsor Emerald		0.82
Prussian Blue		2.31
Cobalt Blue		1.06
Cadmium Yellow		1.65
Raw Sienna		0.32
Burnt Umber		0.13
Ivory Black	1.24	

[TABLE 8] Summary of spectrophotometry data for watercolor samples treated with D/2

	DE*Lab FOR B&W	DH*ab FOR COLOR SAMPLES
Support Wood Board		
Gesso Ground	1.13	
Zinc White	0.72	
Titanium White	0.70	
Rose Madder		0.21
Cadmium Red		0.02
Terre Verte		0.70
Oriental Green		0.34
Prussian Blue		0.80
Cobalt Blue / Deep		1.32
Cadmium Yellow		0.74
Raw Sienna		3.26
Burnt Umber		0.92
Ivory Black	1.52	

[TABLE 9] Summary of spectrophotometry data for egg tempera samples treated with D/2

This is not something that is usually recommended with paper, and it was not done in this study. It should be noted that D/2 is meant for use on masonry surfaces. It is possible that if it were used on acrylic painted masonry surfaces, if rinsed, it would cause little or no change to the paint.

Egg tempera has a ground, so it is believed that the D/2 soaked into it and browned it slightly. Nine samples had their gloss changed, but not in a consistent direction as was the case with the acrylic.

Japanese paint has a coating on the surface, but it is not enough to inhibit the D/2 from soaking in and changing the appearance of the paint. The effect was mostly a lightening and yellowing of the samples, and only three had their gloss changed.

The visual observations overall showed that D/2 was detrimental to all four paint types. To rank effect from least to most damaging: The acrylic was damaged the least, with a 4% average gloss change; then Japanese paint (8% average tone change and 5% average hue change); then watercolor (14% average tone change and 2% average hue change); and finally egg tempera was the most damaged by the D/2 with a 31% average tone change and a 10% average hue change.

Comparison of the visual observations with the spectrophotometric results yields agreement in as many as 48 out of 52 (92%) sample sets. It all depends upon the value for ΔE or ΔH that is accepted as the cutoff point for measuring a real difference. There is no absolute cutoff point for when a real color difference (ΔE or ΔH) is measured. It depends upon the surface roughness. Tiano et al. (this volume) on rough stone surfaces uses a ΔE of 3, while Koestler et al. (1993) on oil paint surfaces had discussed a ΔE of 1. On smooth painted surfaces it may be that a lower value is correct, but on uneven surfaces (or if uneven distribution of pigment occurs) a higher value may be more meaningful. If we assume a ΔE (or ΔH) of 1 as signifying a real difference, four samples gave an instrumental reading greater than that *and* these did not agree with the visual observations. If we move the cut-off point up to a ΔE or ΔH of 3, then *no instrumental reading said there was a change when the visual said there wasn't.* There are still four visual changes that the instrument did not see that have to be explained.

In the four remaining cases that did not agree, it was the visual observations that measured a change and the spectrophotometer that did not. These differences may be explained as follows: Japanese paint samples synthetic pink and synthetic verdigris are made of small particles of pigment, and it is possible that the spectrophotometer's probe size (0.3 mm) relative to the pigment particle sizes gave a higher variability in readings between control and treatment. In the case of the egg tempera, two pigments, both white (zinc white and titanium white) were clearly

changed in tone and hue; these samples also had darkened, yet the spectrophotometer did not measure a change. This may be due to the fact that a small precipitate from the D/2 was on the surface. Visual examination probably compensated for the presence of the white particles and noted the overall change in the surface. The spectrophotometer may have "seen" the white particles and averaged them in with the darkened surface to give an unchanged reading.

In general, it was felt that the trained conservators' eyes, when coupled with the statistical binary assessment method, yielded truer results than did the instrumental technique, but they were close. (This assumes, of course, that both techniques didn't produce an error in the same direction.) The binary statistical assessment procedure permits a relatively rapid low-tech procedure to assess the effects of a treatment on a painted surface. The refinement of Koestler et al. (1993) of dividing color change into tone and hue permitted the tone of black and white samples to be compared to color calculation values that reflected just a lightening and darkening effect, and the hue of color samples to be compared to the color calculations that reflected mostly color changes. This seems to give a greater degree of agreement between visual and instrumental data. (Had DH^*_{ab} rather than the ΔE^*_{ab} been used for the black and white samples, three data sets would have fallen out of agreement with the visual observations.)

One other point worth noting concerns the meaning of the ΔE, or color difference value and visual observations. Billmeyer and Saltzman (1981, p. 109), in their influential book on principles of color chemistry (2nd edition) state:

> "It is our belief that color measurement is the same whether the eye alone is used, or the eye and other instruments are used in combination. The instruments are aids to the eye. Their function...is to express the color of materials or color differences in terms of what the eye perceives.... It does not supercede the trained eye...in many cases it may become a substitute for years of experience in the training period."

Berns (2000, p. 130), in the 3rd edition to the Billmeyer and Saltzman classic, states:

> "We recognize that all existing color-difference calculations do not correlate perfectly with visual judgments, even under the most carefully controlled experimental conditions. Thus, built into any instrumental color control system is visual validation. In cases where the instrumental measurement is close to a color tolerance limit, visual observations are required. Ideally, they should be performed by a number of observers."

Despite the power and utility of the visual binary statistical procedure, it does not eliminate the need for an instrumental approach to color assessment. Not many art objects have an appropriate color control samples that can be used to assess color change in the object over time. Indeed, the control is usually the previous state of the same object under consideration. This necessitates having some instrumental technique that can record the current color state of an object's surface that can then be compared to the object at some later time to assess for change. The essential aspect of the color measurement is that the conditions of measurement be clearly defined to make future comparisons meaningful. In future testing we will try to examine the error bar of ΔE's or ΔH's associated with unevenly distributed paint surfaces and determine how to relate this to real art painting surfaces when a control sample doesn't exist.

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EDITED BY
ROBERT J. KOESTLER
VICTORIA H. KOESTLER
A. ELENA CHAROLA
FERNANDO E. NIETO-FERNANDEZ

ART, BIOLOGY, AND CONSERVATION: BIODETERIORATION OF WORKS OF ART

The Metropolitan Museum of Art, New York