VISUAL EFFECTS OF SELECTED BIOCIDES ON EASEL PAINTING MATERIALS

R. J. Koestler, E. Parreira, E. D. Santoro and P. Noble

Abstract—This paper reports on the results of experiments to test for visual changes to paint systems after biocidal treatments, using a statistical binary procedure. Four biocides were selected, two of which are fungicides—a quaternary ammonium-organotin mixture (BioMet 66®) in distilled water and an orthophenylphenol (Lysol®) in a spray—and two of which are fumigants—sulfuryl fluoride (SO_2F_6), a gas (Vikane®), and nitrogen gas (N_2) (as an anoxant). The procedure used to assess the effect of the biocidal treatments was a random field visual scoring regimen by two paintings conservators. The tests were conducted on 30 combinations of linen, rabbitskin glue size, lead white oil ground and oil-based paints. The visual assessment procedure provided information on color change, gloss change, blanching, topography change and precipitation. The results indicated that Vikane adversely affected 10 of 11 pigment systems; Lysol adversely affected six of 11 pigment systems; BioMet 66 had a minor effect on four of 11 pigment systems; and nitrogen had no visible effect on any sample. The visual technique provided a quick and broad method for assessment of non-subtile visual changes.

1 Introduction

Conservation of works of art carries with it a unique set of philosophical and material considerations. The philosophical considerations revolve around prescribing treatments that have the potential to alter the original art work. These issues are generally decided by curators, conservators and restorers, and will not be discussed here. The material considerations involve understanding the diverse materials of which artworks may be composed, both the original material and previously introduced conservation or restoration material, and evaluating, as far as possible, what the effect of a new treatment is likely to be on those materials. The present paper focuses on the visual changes that treatments to control microbiological and/or insect infestations can produce on selected easel painting materials.

The components of easel paintings, like virtually all materials, are susceptible to microbiological attack if placed in a favorable environment. Museums, *de facto* in their efforts to protect their collections from such attack, have historically been moderately successful, as evidenced by the large numbers of art collections in the world. Occasionally, in temperate and, more frequently, in tropical and sub-tropical zones, environmental control fails, or is non-existent, and microbial growth may flourish. Mold growth on paintings not only obscures and discolors the painting but may cause permanent color change, decomposition of the medium or structural damage to the pigments or underlying support.

Preventing mold growth on the paint surface is considered easier than removing an infection [1, 2]. The recommended procedure for prevention is to incorporate a biocide into the paint layer itself, before application. This is certainly possible for modern artists' pigments but is not applicable for an historic painting. An apparently successful approach undertaken by Inoue and Koyano [3] in the humid climate of Japan is to incorporate the biocide into a new varnish layer. However, this is not a possibility for those paintings that were never intended to be varnished (e.g., many Impressionist paintings and modern paintings). Even when a biocide can be used, eventually its effectiveness is reduced by continued microbial stress, i.e., growth. Longevity of the biocide is difficult to predict. The biocide Inoue and Koyano [3] used in their tests has lasted seven years, with continuing effectiveness.

Treating a mold infection on an easel painting necessitates cleaning off the infestation and improving the display or storage conditions for the painting. It should be noted that

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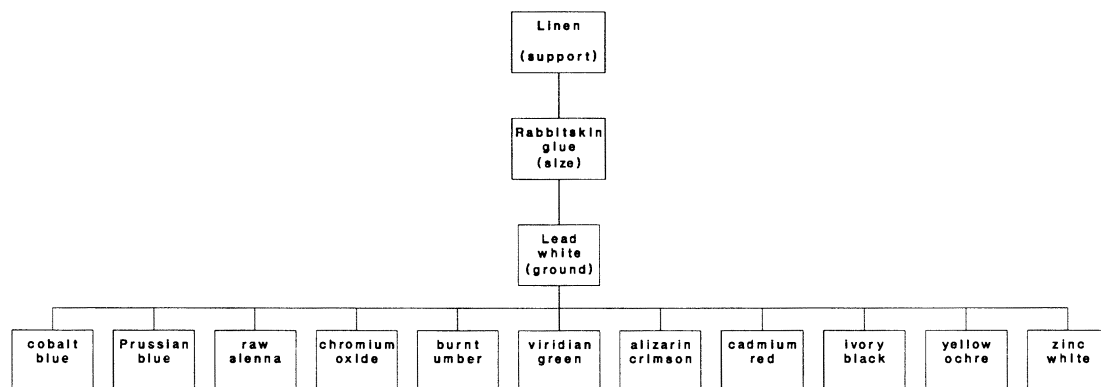
Painting Media
Oil-Based Media Material

Figure 1   Flow chart representing easel painting material used in the current experiment.

if cleaning is performed by wiping the surface, the spores will be spread around the painting surface and, unless the environmental conditions that promoted the original infestation are changed, growth will resume with greater coverage of the painting. Changing the environmental conditions for the painting is not always possible, so the next course of action is to apply a biocide to the surface of the painting.

Selection of an appropriate biocide for direct application to a painting surface requires the same care and testing that is used with a biocide-containing varnish or paint, i.e., non-deleterious interaction of the biocide with the component parts of the painting, non-yellowing or other visible change of the biocide over time, and a long-term fungistatic activity for the biocide.

Testing of the biocide, as alluded to above, requires an understanding of the composition of the easel paintings, which may have been fabricated with a large variety of materials. This is especially true of modern paintings. In addition, there are often adhesives or other restoration materials incorporated into the painting at different times in its history. Often, too, it is not feasible to determine the exact composition of every work before treatment.

The combinations of materials chosen for this series of experiments are common in tra-ditional canvas paintings. They are represented in Figure 1. This figure shows four levels: a support—linen in our tests; a size—rabbit-skin glue; an oil ground—lead white; and the oil paint—11 types reported here. There are, of course, many other possibilities for each level.

The choice of possible biocides for testing is quite large. The term 'biocide' is used to include any treatment that adversely affects living organisms. According to Bravery [4] there are over 150 biocides available in England. Some of the more common biocides and fumigation techniques in the conservation literature were selected. (The reader is referred to Koestler and Vedral [5] which provides an extensive reference listing of these and other articles pertinent to biodeterioration of cultural property, 1721 citations in all.) The biocides chosen were two fungicides—a quaternary ammonium-organotin mixture (BioMet 66®), in distilled water, and an ortho-phenylphenol (Lysol®) in a spray; and two fumigants—sulfuryl fluoride (SO_2F_6), a gas (Vikane®), and nitrogen gas (N_2) (as an anoxant). The testing procedure designed for assessment of the biocides was a visual scoring regimen performed by two paintings conservators (EP and PN). Because of the potential difficulty of visually assessing small changes in color, spectrophotometry was tested on selected samples (see Appendix). The

visual assessment technique was an adaptation of the procedure developed and employed in other studies [6–8].

2 Materials and methods

2.1 Samples
Samples of (1) linen, (2) linen/rabbitskin glue size, (3) linen/rabbitskin glue size/lead white oil ground and (4) linen/rabbitskin glue size/lead white oil ground/oil paint (Figure 1) were prepared and aged by one of the standard procedures in use in the Paintings Conservation Department of the Metropolitan Museum of Art. Aging was carried out in a CI35 Fade-ometer equipped with a Xenon arc lamp filtered with a borosilicate and sodalime filter set to approximate the spectral energy distribution of sunlight through window glass. Irradiance was maintained at 0·90W/m² at 420nm. The black panel temperature was 50°C and wet bulb depression of 10°C gave a dry bulb temperature of 32°C and an RH of 41%. The Fade-ometer operated at approximately 1 × 10⁵ lux. Samples were aged 167·7h and received 1·677 × 10⁷ lux.h of irradiance. If reciprocity holds, this is equivalent to 7·5 years of aging in a gallery illuminated at 500 lux for 12h/day. At intervals during the aging process, paint samples were brushed vigorously and repeatedly with a cotton swab wetted in mineral spirits to test for any sign of solubility. The samples were visually stable after 167·7h.

Ground and oil paints were proprietary brands (Fredix, USA, for lead white ground only; Schmincke, Germany, for Prussian blue; and Winsor & Newton, England, for all other paint). The colors used were lead white ground, zinc white, cobalt blue, Prussian blue, raw sienna, chromium oxide, burnt umber, viridian green, alizarin crimson, cadmium red, ivory black and yellow ochre.

The linen was of medium weight and was sized with Liquitex rabbitskin glue. It was then given three coats of Fredix lead white oil priming, which according to its contents label, contains lead carbonate, calcium carbonate, linseed stand oil and mineral spirits. The paint was then applied with a palette knife in 2·5cm-wide strips. The thickness of the paint layers varied somewhat in order to mimic a

real painting and to permit the assessment of topographical changes. Meyer [9] was consulted for information on the preparation of the paint samples. Completed strips of samples can be seen in Figure 2. Control and experimental samples were stored under identical light conditions (laboratory bench conditions), before and after treatment.
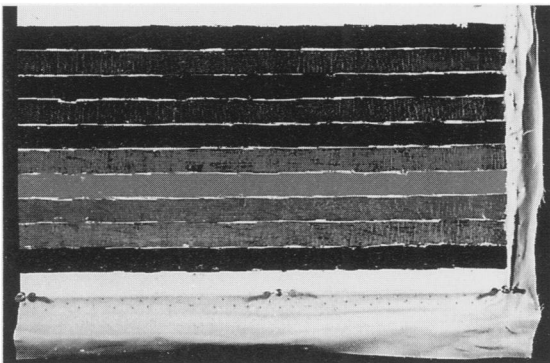

Figure 2   Photograph of easel painting samples prepared for current study.

2.2 Biocides
BioMet 66® represents the class of quaternary ammonium-organotin compounds used effectively in England [4, 10] for control of microbial growths on stone and paint. It is registered for use in the USA for a variety of applications including fungal control in water-cooling systems and on wood. (Recent information from the manufacturer of this product indicates that its use may become restricted due to the presence of the organotin.)

Orthophenylphenol is available as a household commercial product called Lysol® in the USA and Canada. This biocide had been used (in this commercial formulation) for control of fungi in an extensive infestation at the National Museum of Man in the Victoria Memorial Museum, Ottawa [11].

Sulfuryl fluoride, under the trade name Vikane®, is one of the few fumigants for insect control currently in use by museums in the USA. Some studies [12, 13] suggested caution in the use of this product, especially if objects contain metal or glass. The product is currently used by fumigation companies in the USA, particularly in California, for control of

termites in museums and private homes. Several instances were reported in the product manufacturer's literature of museums in California using this fumigant on some of their buildings without removing the works of art within.

The fourth technique, a suffocation treatment using nitrogen gas, while effective against insects, is, at best, a fungistatic treatment. This is under active consideration as a possible replacement treatment for insect eradication by us and others [14–19]. The use of nitrogen as an anoxant is not new; there are reports of its use in the 1860s (and continuing today) for storage of grain in temperate and tropical countries [20–22].

2.3 Biocide application
2.3.1 BioMet 66 (quaternary ammonium-organotin mixture)
Triplicate sample strips had a 2ppm (v/v) solution of BioMet 66 in distilled water brushed on with a new 6mm brush. Brush motion was horizontal, line by line, until the whole surface had been covered; this was followed by vertical motion, again line by line, until the complete surface had been covered again. The brush was dipped in the solution, with the excess tapped off on the side of the jar, between each stroke. Controls were untreated dry samples and distilled-water-only brushed samples. There was no significant difference between wet and dry controls, i.e., no field was different.

2.3.2 Lysol (orthophenylphenol)
Triplicate sample strips were sprayed with the commercially available deodorizer Lysol. Lysol contains 0·1% 2-phenylphenol, 79% ethanol and 20·9% CO_2 propellant and N-alkyl ([18]C 92%, [16]C 8%) N-ethyl morpholinium ethylsulfates. Spraying was from a distance of at least 15cm. The spray density was beneath saturation, i.e., no liquid run-off was observed. Controls were untreated dry samples.

2.3.3 Vikane (sulfuryl fluoride)
Triplicate samples were sent to a commercial fumigation company (Vanguard Pest Control, Inc., Port Newark, NJ). Samples were included in a shipment of other Metropolitan Museum of Art material to be fumigated. The procedure called for a double fumigation technique, with a two-week waiting period between applications. The paint swatches were enclosed in a special package that was not airtight. The package containing the samples was placed, unopened, within the fumigation chamber. This procedure mimicked the normal fumigation procedures for art objects. Controls were untreated dry samples.

2.3.4 Nitrogen gas plus oxygen scavenger
Triplicate sample strips were placed in a nitrogen-filled, thermally-sealed pouch (P-641-B, 3·6mil, 0·6mil is PVDC-coated biaxially oriented nylon which is adhesively laminated to 3·0mil linear low-density polyethylene; oxygen permeability of P-641-B is 1·0cm³ 2 540mm²/day at 0% RH, 23°C). The pouch dimensions were approximately 0·5m × 0·25m. Also placed in the pouch were packets of oxygen scavenger, Ageless[k] Type Z-2000, a humidity/temperature monitor, and an oxygen indicator, Ageless-Eye[k], which starts changing from blue to pink at 0·5% and is completely pink at or below 0·1% O_2. The pouch was sealed except for a small opening to permit flushing with humidified N_2 (bubbled through water to give 50% RH) for 30 minutes at about 14psi (above ambient pressure). At the end of 30 minutes, the Ageless-Eye indicated no oxygen was present, to the 0·1% level. The bag was sealed and placed in a biochamber to maintain a constant 23°C temperature. The samples remained in the N_2 environment for 150 days. The humidity remained about 50% RH, at 23°C, and the Ageless-Eye oxygen indicator remained at or below the 0·1% level.

2.4 Evaluation method
These four treatments were evaluated in an analytical design which provided for a priori selection of sample grids to be viewed, thereby reducing bias. This design has been tested in other projects using analysis of variance techniques [6–8]. Sub-sets of control and treated samples were viewed using a grid system and randomly generated viewing fields.

The procedure used a 5 × 5 grid pattern superimposed over the sample and corre-

sponding control. Grid box selection was accomplished by referring to a random number table for position selection. Five grid boxes per sample were assessed (20% of the total area for each sample), yielding a total of 15 grid boxes viewed for each experiment (i.e., triplicate samples). Changes were assessed in the following five categories: visual color change; change in gloss; appearance of cracks, bubbles, blisters; blanching effect; and topography changes. Any other phenomena, such as precipitation, were noted.

Control and experimental samples were placed side-by-side for observation. The observations were made by two painting conservators (except for nitrogen, when only one was available), first by unaided eye and then by a low-power binocular microscope (50×) with a fiber optics light source at a variety of light angles. Any differences between control and experimental samples were recorded as the number of squares changed per five examined, for each of the five categories. For example, in measuring gloss change, five grid areas in each experiment were examined and compared to the corresponding grid area on the controls. Any change from the controls

would be recorded as the number out of five that was different. Each sample was compared to six controls, one at a time. Results of all observations were averaged. This experimental design tests for effects at each level and combination of material and should mimic actual painting systems.

3 Results and discussion

Table 1 presents a summary of the visual observations for color and gloss change. Changes are presented as a percentage change in color or gloss for 15 fields viewed for each paint system.

Gloss changes were the predominant type of change noted, followed by color change. To a conservator, either type of change is unacceptable and would proscribe the use of a particular treatment. Topographical changes were noted in only three samples—all treated with Vikane. Cracks, bubbles or blisters were not noted for any sample. Blanching effects, residues or salt precipitation were found only with BioMet 66. Figure 3 shows a sample of yellow ochre before and after Vikane treatment.

Table 1 Visual color and gloss changes

Sample type	Vikane		Lysol		BioMet 66**		N_2	
	color	gloss	color	gloss	color	gloss	color	gloss
linen (l)	- - -	- - -	33%	ND	ND	ND	ND	ND
l + size (ls)	- - -	- - -	ND	ND	ND	ND	ND	ND
ls + lead white ground (lsg)	ND	ND	ND	ND	ND	ND	ND	ND
lsg + zinc white	ND	ND	ND	ND	ND***	20%+	ND	ND
lsg + cobalt blue	87%	73%+	87%	87%+	47%	60%+	ND	ND
lsg + Prussian blue	13%	47%+	ND	7%+	ND	ND	ND	ND
lsg + raw sienna	ND	27%-	ND	7%+	ND	ND	ND	ND
lsg + chromium oxide	13%*	87%+	ND	20%-	ND	ND	ND	ND
lsg + burnt umber	47%	40%+	ND	20%+	ND	26%	ND	ND
lsg + viridian green	20%*	7%	ND	7%+	ND	ND	ND	ND
lsg + alizarin crimson	ND	87%+	7%	7%+	ND	ND	ND	ND
lsg + cadmium red	ND	33%-	ND	80%+	ND	40%+	ND	ND
lsg + ivory black	ND	13%+	ND	20%+	ND	ND	ND	ND
lsg + yellow ochre	73%*	53%+	ND	53%+	ND	ND	ND	ND

Notes: numbers are the percent of fields changed as a result of treatment (based on observations of 15 fields each); a value of 7% (one out of 15 fields) may not be significant. ND = no detectable difference between experimentals and controls; - - - = no data available; *topographical change noted; **blanching effects, residues or salt precipitate formation noted; ***tide rings noted; + = glossier; - = more matt.
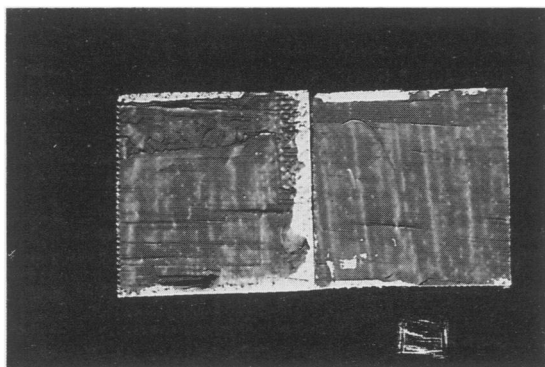
Figure 3 Photograph of yellow ochre before (left) and after (right) Vikane treatment.

3.1 BioMet 66

The quaternary ammonium-organotin biocide had only a minor effect on four pigment systems (cobalt blue, burnt umber, cadmium red and zinc white) and the ground (lead white). Often a residue remained after drying. The residue, however, could be wiped off with a brush dipped in distilled water. Analysis of the residue has not been performed, so it is unclear which component(s) of the biocide were precipitating, or even if some component of the pigment systems was interacting with the biocide. There are several cautions to be attached to this biocide: it is currently not registered for use on/in paints in the USA; organotins may be banned shortly by action of the US Environmental Protection Agency; and even if their use is possible, some testing should be performed on the effects, if any, of residual ammonium or organotin salts on the surface of paintings.

3.2 Lysol

Lysol affected six out of 11 pigment systems in our study, leaving Prussian blue, raw sienna, viridian green and alizarin crimson apparently unaffected. These preliminary results suggest that Lysol should not be used on this type of system. Any contemplated use of this product would require further testing.

3.3 Vikane

Vikane affected 10 out of 11 pigment systems. These results seem to imply that Vikane has more effect on paint systems than currently

thought. This is disturbing as Vikane has been and is being extensively used in the USA for treatment of museum objects, and for historic structures with their contents intact [13]. Research to date has indicated that Vikane has little or no effect on linseed oil and some pigments in a purified form [12, 13]. The only direct comparison that may be drawn between the data of those studies and the results reported here concerns lead white, where no deleterious effect was found.

3.4 Nitrogen

Exposure to the gas N_2 did not cause any visual change in the pigment systems tested. This was to be expected, as N_2 is generally unreactive. Nitrogen and other oxygen-free environments represent a promising direction for development of safe and effective means to eradicate insect infestations within cultural collections. There are drawbacks to these kinds of treatment in that they are slow acting (1–3 weeks to kill common museum pests, [16]), and they are, at best, only fungistatic. Currently, nitrogen and argon are the only gas treatments permitted for insect eradication at the Metropolitan Museum of Art.

3.5 Effectiveness of experimental design

The visual technique provided a fast assessment regimen to screen potential biocides for further study. Differences in viewers' observations were apparent when low levels of change were noted—one out of 15 fields (7%). At this level a change may or may not have occurred. Some spectrophotometric data were obtained to compare with the visual assessment and are discussed in the Appendix.

4 Conclusions

The paint samples chosen were not necessarily the most sensitive to a change in gloss or color. Yet the unaided eye, and low-power binocular microscopy, revealed that Lysol and Vikane adversely affected some or most of the pigment systems tested (six of 11 and 10 of 11, respectively). The effect ranged from minor (13%) to major (87% of the surface appearance altered in the case of Lysol for the cobalt blue pigment system). BioMet 66 had a

minor effect on four of the pigment systems. In general, more gloss changes were noted than color changes. Any of these changes would proscribe the use of the treatment for a painting, in an ideal situation.

N_2 results indicated that no visual effect was apparent after treatment. This does not prove that nitrogen is safe to use but it is probably safer than any of the other treatments in the study. Nitrogen gas continues to show promise as a safe treatment for museum materials.

Further testing is needed to give a more accurate assessment of potential alteration of a pigment system by a given biocide.

Acknowledgements

Appendix

A Perkin-Elmer Model 552A spectrophotometer with integrating sphere was used to collect tristimulus and chromaticity values for two of the samples evaluated visually. Tristimulus and chromaticity values were measured for the visible light range (380–700nm), at a spot size of 10mm. Specular component was excluded—this provides more information on color changes and less on gloss changes. Calculations were for CIE standard source A. CIE 1976 L*,a*,b* were calculated from the spectrophotometric values. Perceptible color-difference calculations (ΔE) utilized the CIE 1976 L*,a*,b* values.

Due to the uneven nature of the sample surfaces, it was decided to investigate the effect of topography on the tristimulus data. One paint system (yellow ochre) was tested extensively. Eight spectra per sample were collected for six controls. The spectra were labeled 1 to 8 with each spectrum representing the same orientation of paint application. With each succeeding spectrum, the sample was rotated 45° in a plane perpendicular to the axis of the incident beam. It was found that a 0° and 90° sample orientation, perpendicular to the incident beam, was adequate to capture the structural color component (0° representing the direction of paint application and 90° at right angles to it). Due to the time-intensive nature of collecting statistical data with spectrophotometry, only two color paint systems were examined—yellow ochre and chromium oxide color systems (at all levels of the experimental design, except linen and linen + size for Vikane)—for BioMet 66, Lysol and Vikane.

Tristimulus data were converted into CIE 1976 L*,a*,b* values. The CIE 1976 L*, a*, b* values were then used to calculate ΔE, the perceptible color-differences. The ΔE or perceptible color-difference formula used was:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

A larger ΔE is indicative of a greater color difference. Spectrophotometric data are summarized in Table 2 for three experiments (Vikane, BioMet 66 or Lysol) and six control sets at two physical orientations of the samples, 0° and 90°.

Spectrophotometric data indicate that yellow ochre changed color after Lysol or Vikane treatment, agreeing with the visual assessment. In the case of BioMet 66, the $\Delta E = 1.1$ may not represent

Table 2   *Color change (ΔE) and means of coordinate changes*

Sample type	0°				90°			
	ΔE	L^*	a^*	b^*	ΔE	L^*	a^*	b^*
Yellow ochre								
Control	—	53·3	27·6	32·8	—	53·6	28·2	32·1
Lysol	0·9	52·5	27·9	32·5	2·4	51·4	27·2	31·7
Vikane	0·9	54·1	27·6	33·1	1·8	54·7	28·1	33·5
BioMet 66	0·5	53·6	28·0	32·9	1·1	53·9	28·0	33·1
Chromium oxide								
Control	—	39·4	−2·2	20·8	—	39·3	−2·6	20·8
Lysol	0·5	39·2	−2·6	20·6	0·2	39·1	−2·6	20·7
Vikane	0·7	39·0	−2·8	20·6	0·2	39·4	−2·4	20·9
BioMet 66	0·5	39·0	−2·5	20·6	0·3	39·6	−2·6	20·9

Note: controls were based on a minimum of six measurements, experimentals on a minimum of three.

a significant perceptual difference, in which case it also agrees with the visual assessment.

Spectrophotometric data for chromium oxide agree with the visual color data from Table 1 for Lysol and BioMet 66, and possibly Vikane if a 13% visual color change is not significant.

More spectrophotometric data would have to be collected to draw a better comparison with the visual assessment.

Materials and suppliers

Ageless and Ageless-Eye: Mitsubishi Gas Chemical Company of America, 520 Avenue of the Americas, New York, NY 10011, USA.

Artists' supplies: Fredix Co., artists' linen; Schmincke & Co., Prussian blue; and Winsor & Newton pigments, available through art supply companies.

BioMet 66: M&T Chemicals, Inc., Rahway, NJ 07065, USA.

Fade-ometer: Atlas Electric Devices Co., 4114 N. Ravenswood Avenue, Chicago, IL 60613, USA.

Lysol: Lehn & Fink Products, Division of Sterling Drug, Inc., Montvale, NJ 07645, USA.

P-641-B: Cryovac Division, W.R. Grace Co., P.O. Box 338, Simpsonville, SC 29681, USA.

Vikane: Dow Corning Chemicals, supplied through Vanguard Pest Control, Port Newark, NJ 07114, USA.

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Résumé—Cet article rapport les résultats d'expériences menées sur des peintures afin de tester les changements perceptibles à l'oeil après traitement contre les microorganismes, en employant un procédé statistique binaire. Quatre biocides ont été sélectionnés, dont deux sont des fongicides—un mélange quaternaire d'ammonium-organotine (BioMet 66) dans l'eau distillée et un orthophénylphénol (Lysol) en pulvérisation—et les deux autres des fumigènes—fluorure de soufre (SO_2F_6), un gaz (Vikane), et de l'azote gazeux. Le procédé a donné des informations sur les changements de couleur, les changements de brillance, le blanchiment et les changements de configuration. Les résultats indiquent que le Vikane affecte gravement 10 des 11 préparations de pigment; le Lysol en affecte six sur les 11; le BioMet 66 a un effet mineur sur quatre des 11 préparations; et l'azote n'a pas d'effet visible sur les échantillons.

Zusammenfassung—Der Beitrag beschreibt, inwieweit Farbaufträge durch Behandlungen mit Bioziden sichtbare Veränderungen erleiden. Für die Versuchsreihen wurden vier Biozide ausgewählt. Zwei waren dabei Fungizide—eine quaternäre Ammonium—Oganozinn Mischung (BioMet 66) in destilliertem Wasser und ein Orthophenylphenol (Lysol) als Spray—und die zwei restlichen Begasungsmittel—Sulfurylfluorid (SO_2F_6, Vikane) und Stickstoff (als Antioxidans). Die Beurteilung zielte auf Farbveränderungen, Veränderungen im Glanz, Vergrauungen ('blanching'), Veränderungen in der Struktur der Oberflächen und auf eine Trennung des Pigment-Bindemittelsystemes ab. Die Experimente lieferten Hinweise darauf, daß Vikane 10 von 11 und Lysol sechs von 11 Pigmentsystemen nachteilig beeinflußte. BioMet wirkte sich schwach negativ auf vier von 11 pigmentsystem aus, während Stickstoff keinen sichtbaren Effekt auf irgendeine der Proben hatte.