

CONDITION PROBLEMS RELATED TO
ZINC OXIDE UNDERLAYERS: EXAMINATION OF
SELECTED ABSTRACT EXPRESSIONIST PAINTINGS FROM
THE COLLECTION OF THE HIRSHHORN MUSEUM AND
SCULPTURE GARDEN, SMITHSONIAN INSTITUTION

DAWN ROGALA, SUSAN LAKE, CHRISTOPHER MAINES,
AND MARION MECKLENBURG

ABSTRACT—The presence of zinc oxide oil paint and the condition problems observed in a group of paintings from the collection of the Hirshhorn Museum and Sculpture Garden prompted analytical examination of the museum's mid-20th century holdings. Results reveal a link between upper layer deterioration and underlying zinc oxide paint layers, and suggest that certain visible signs of deterioration may signal the presence of more serious and widespread condition problems. The popularity of zinc oxide house paint among mid-century artists creates a higher probability of this type of deterioration in works from this period.

TITRE—Les problèmes de conservation reliés aux couches sous-jacentes d'oxyde de zinc: l'examen de peintures expressionnistes abstraites sélectionnées parmi la collection du Hirshhorn Museum and Sculpture Garden, Smithsonian Institution. RÉSUMÉ—La présence de peinture à l'huile contenant de l'oxyde de zinc et des problèmes de conservation observés sur un groupe de peintures du *Hirshhorn Museum and Sculpture Garden* (musée Hirshhorn et jardin de sculptures) ont incité l'étude technique de peintures de la collection datant du milieu du XXe siècle. Les résultats révèlent un lien entre la détérioration des couches supérieures et la présence de couches sous-jacentes de peinture contenant de l'oxyde de zinc, et suggèrent que certains signes avant-coureurs de détérioration peuvent signaler la présence de problèmes de conservation plus sérieux et étendus. La popularité de la peinture domestique à base d'oxyde de zinc chez les artistes du milieu du XXe siècle crée une forte probabilité que ce type de détérioration soit présent sur les œuvres de cette période.

TÍTULO—Problemas de conservación relacionados con capas subyacentes de óxido de zinc: examen de una selección de cuadros de expresionismo abstracto de la colección del Hirshhorn Museum and Sculpture Garden, Smithsonian Institution. RESUMEN—La presencia de pintura al óleo de óxido de zinc y los problemas de conservación detectados en un grupo de cuadros de la colección del *Hirshhorn Museum and Sculpture Garden* (Museo Hirshhorn y Jardín de Esculturas), motivó el examen analítico del acervo del museo de mitades del S XX. Los resultados revelaron que hay una correlación entre el deterioro de la capa superficial y las capas subyacentes de pintura de óxido de zinc, y sugieren que ciertos signos visibles de deterioro pueden ser indicativos de la presencia de problemas de conservación más serios y extendidos. La popularidad del uso de pintura casera de óxido de zinc entre los artistas de mitades del S XX, aumenta la probabilidad de este tipo de deterioro en obras de este periodo.

TÍTULO—Problemas de conservação relacionados às camadas subjacentes de óxido de zinco: exame de pinturas expressionistas abstratas selecionadas da coleção do Hirshhorn Museum and Sculpture Garden, Smithsonian Institution. RESUMO—A presença de tintas a óleo com óxido de zinco e os problemas de conservação observados em um grupo de pinturas da coleção do *Hirshhorn Museum and Sculpture Garden* (Museu e Jardim de Esculturas Hirshhorn) ocasionaram o exame analítico do acervo de meados do século 20 pertencente ao museu. Os resultados revelam uma relação entre a deterioração da camada superior e as camadas subjacentes pintadas com óxido de zinco e sugerem que alguns sinais visíveis de deterioração podem indicar a existência de problemas de conservação mais sérios e abrangentes. A popularidade, entre artistas de meados do século, da pintura de casas com óxido de zinco, gera uma maior probabilidade deste tipo de deterioração em trabalhos desta época.

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Table 1.
List of paintings from the HMSG collection selected for examination

Artist	Title	Acc. No.	Date	Dimensions (cm)
Karel Appel	<i>Birds: Storm over the Sea</i>	80.68	1957	191.5 x 241.3
	<i>The Donkey</i>	66.64	1957	129.5 x 161.6
	<i>Magic Doll with Pig's Head</i>	66.65	1961	130.0 x 161.8
	<i>Commencement du printemps</i>	66.67	1963	129.5 x 96.5
	<i>Issa</i>	66.68	1963	162.0 x 130.5
Hans Hofmann	<i>Trophy/Verso: Untitled</i>	66.2483	1951	138.4 x 102.9
	<i>Radiance</i>	66.2477	1956	63.5 x 76.2
	<i>Flowering Swamp</i>	66.2482	1957	122.0 x 91.5
	<i>Prelude of Spring</i>	66.2479	1958	127.7 x 214.0
	<i>Oceanic</i>	66.2480	1958	152.7 x 121.9
	<i>To J.F.K.: A Thousand Roots Did Die with Thee</i>	76.125	1963	151.5 x 182.5
Franz Kline	<i>Delaware Gap</i>	66.2751	1958	198.6 x 269.5
	<i>Palladio</i>	66.2754	1961	266.1 x 193.7
Jackson Pollock	<i>Composition with Pouring II</i>	66.4082	1943	63.9 x 56.3
	<i>Water Figure</i>	66.4087	1945	182.7 x 73.7
Antonio Saura	<i>Sija (Gina Lollobrigida)</i>	66.4461	1959	195.4 x 97.4
	<i>Crucifixion (Triptych)</i>	66.4455	1959–60	195.6 x 325.7
	<i>Self-Portrait</i>	66.4459	1960	73.7 x 81.3

1. INTRODUCTION

In 2007, the Hirshhorn Museum and Sculpture Garden (HMSG), Smithsonian Institution, completed an inventory and condition survey of their paintings collection. During the review of approximately 4,600 paintings, evidence of a particular type of deterioration was observed in some of the mid-20th century holdings. Conservation records and the registration database were used to select a group of paintings from the same period and from a particular geographical region. After visual inspection and in situ analysis of these paintings, the study group was limited to a selection of 18 works by five artists. Detailed analysis of this study group, performed in collaboration with the Museum Conservation Institute (MCI),

Smithsonian Institution, reveals a link between upper layer deterioration and underlying zinc oxide oil paint layers, and suggests that certain visible signs of deterioration may signal the presence of more serious and widespread condition problems.

2. STUDY GROUP SELECTION

The museum's conservation records and registration database were used to create an inclusive initial selection of works from the same period as the paintings with deterioration identified during the 2007 inventory. Regional and material considerations helped determine the final study group of 18 paintings.

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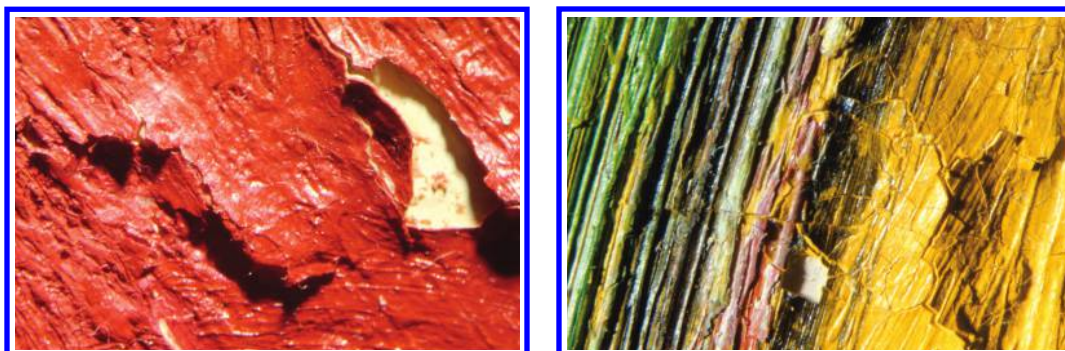


Fig. 1 Examples of blind cleavage in cadmium colors painted over zinc oxide oil paint. Use of cadmium yellow in the study group paintings, although limited, shows cleavage similar to that found in cadmium red layers. Hans Hofmann, *Trophy/Verso: Untitled*, 1951 (HSMG acc. no. 66.2483). Left: cleaving ground/red layer, T 2.5 x L 3.4 cm., photomicrograph, 6x focus, 10x lens, 0.32x factor. Right: cleaving ground/mixed layers, T 9.5 x R 25.0 cm., photomicrograph, 6x focus, 10x lens, 0.32x factor

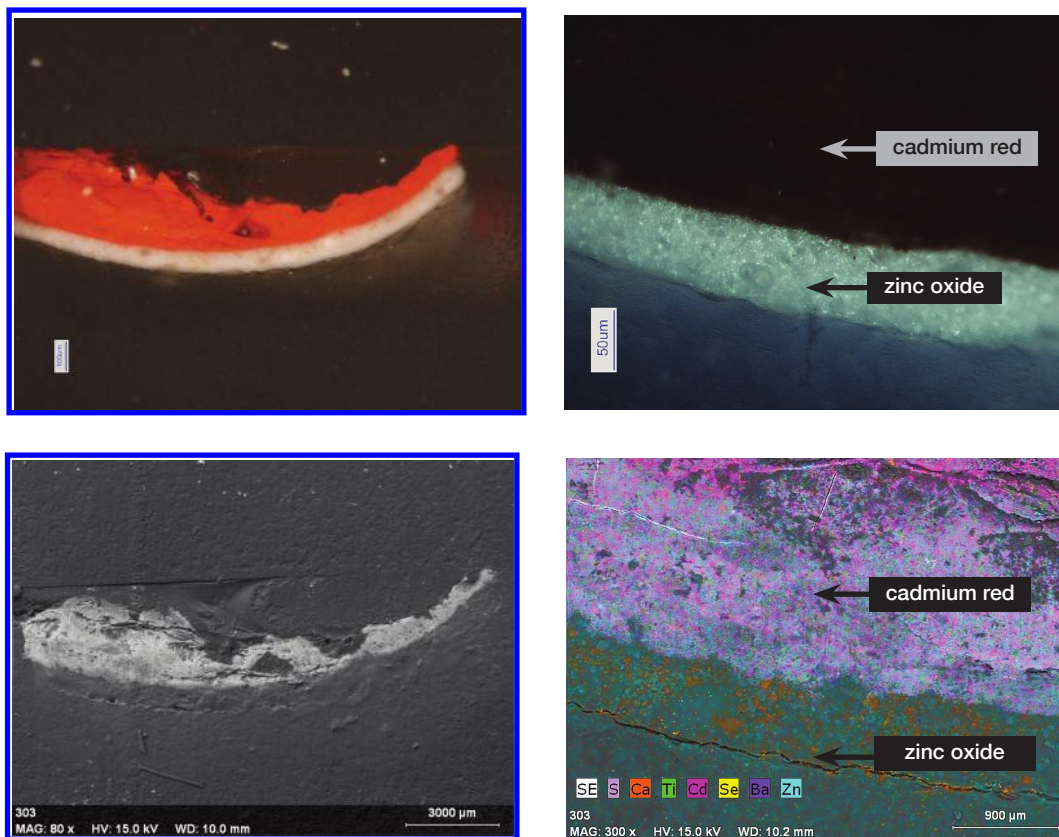


Fig. 2 Paint sample from area of lifting cadmium red shown in figure 1. White underlayer has split, resulting in white paint attached to both the support and the lifting red paint. Confirmation of zinc oxide in white underlayer obtained by microscopy and SEM-EDS. Hans Hofmann, *Trophy/Verso: Untitled*, 1951 (HSMG acc. no. 66.2483). HMSG cross section no. 303, sample of cleaving ground/red layer, T 2.5 x L 3.4 cm. Top left (TL): 50x magnification, dark field with cover slip; Bottom left (BL): Back-scattered electron (BSE) image. Top right (TR): 200x magnification, ultraviolet illumination (Leitz filter A, 340–380 nm excitation range). Bottom right (BR): Elemental map

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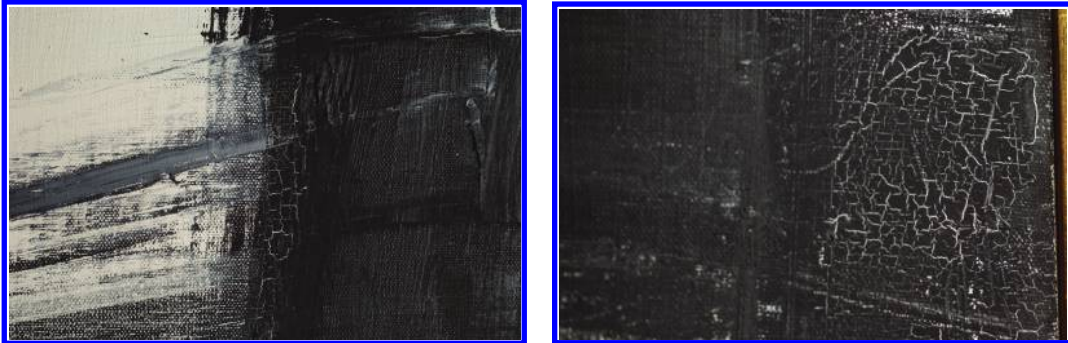


Fig. 3 Examples of selective cracking of carbon blacks painted over zinc oxide oil paint. Two white underpaints are used; the black paint remains intact where it is painted over lead white, but exhibits widespread cracking over zinc white. Franz Kline, *Palladio*, 1961 (HSMG acc. no. 66.2754). Left: cracking black layer, digital photograph, Nikon D70, macro lens. Right: cracking black layer, digital photograph, Nikon D70, macro lens

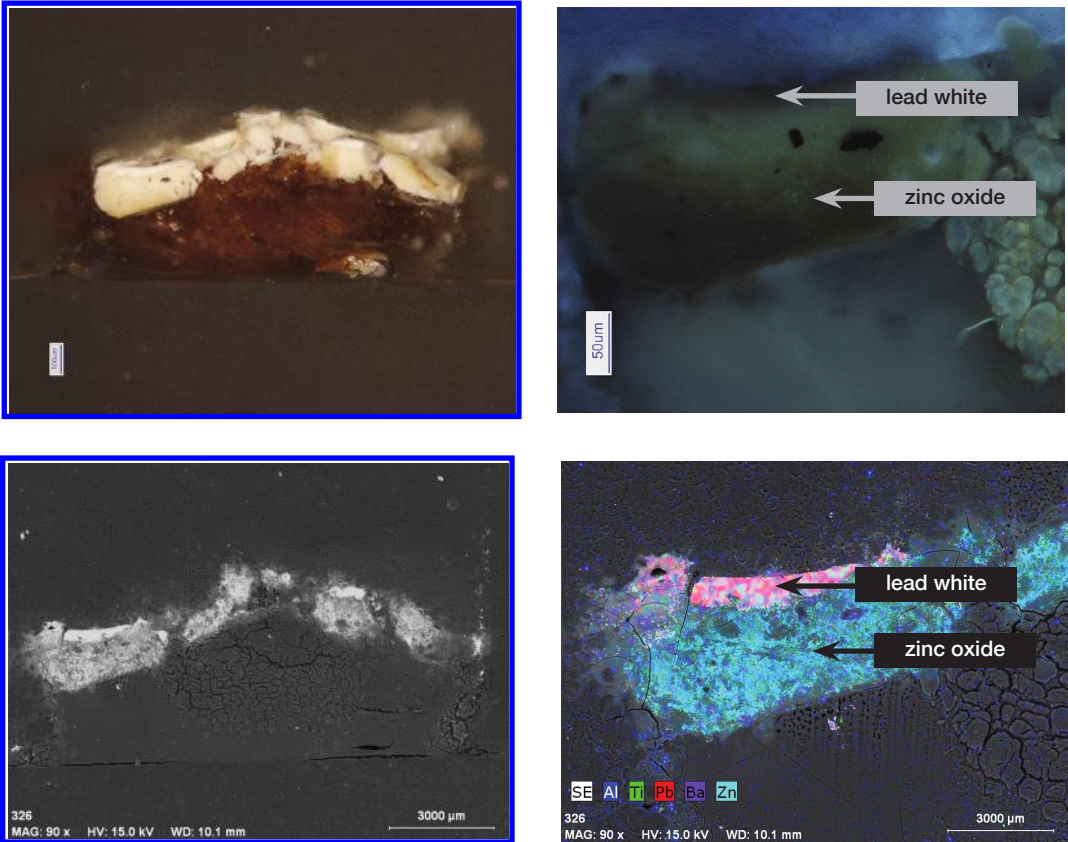


Fig. 4 Paint sample from a tacking margin with matching underlayer stratigraphy to that shown in figure 3. Confirmation of zinc oxide underlayer by microscopy and SEM-EDS. The lead and zinc whites on the painting are distinguishable with the unaided eye. The lead white layer is sporadic, resulting in areas of both lead white and zinc white in contact with the carbon black paint. Franz Kline, *Palladio*, 1961 (HSMG acc. no. 66.2754). HMSG cross section no. 326, sample of PL tacking edge, B 132.8 cm. Top left: 50x, dark field, with cover slip. Bottom left: Back-scattered electron (BSE) image. Top right: 200x, ultraviolet illumination (Leitz filter A, 340-380 nm excitation range). Bottom right: Elemental map

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Table 2.

List of study group paintings showing relationship between zinc oxide oil paint and upper layer deterioration

Artist	Acc. no.	Zinc underlayer	Cadmium	Cadmium cleavage	Carbon black	Black cracking
Karel Appel	80.68	X				
	66.64	X	X	X		
	66.65	X	X	X		
	66.67	X				
	66.68	X				
Hans Hofmann	66.2483	X	X	X		
	66.2477	X	X	X		
	66.2482	X	X	X		
	66.2479		X			
	66.2480		X			
	76.125	X	X	X		
Franz Kline	66.2751	X			X	X
	66.2754	X			X	X
Jackson Pollock	66.4082				X	
	66.4087	X	X	X	X*	
Antonio Saura	66.4461	X			X**	
	66.4455	X***			X	X
	66.4459	X***			X	X

* No zinc oxide underlayer beneath carbon black

** Could not determine whether the artist-applied zinc underlayer continues beneath the carbon black

*** An artist-applied overall zinc priming layer over a non-zinc ground layer

Note: Relationship between zinc oxide oil paint and upper layer deterioration, incorporating previous analysis of Pollock works performed by Lake (2004, among others).

2.1 SELECTION OF ARTISTS

In order to study the effect of similar materials under similar conditions, study group selection was limited to artists known to associate with the mid-century Abstract Expressionist community. Five artists were selected for the final study group to limit the influence of individual techniques. Considerations guiding artist selection included similarities in paint application (gestural, thick brushwork, as opposed to stain painting, for example), and the regional potential amongst the New York City community of artists for shared information regarding materials. Paintings

in the study group include works by New York-based artists Hans Hofmann (1880–1966), Franz Kline (1920–1962), and Jackson Pollock (1912–1956), as well as works by CoBRA founder Karel Appel (1921–2006), who encountered these painters' techniques through his close friendships with Kline and Sam Francis, and El Paso group founder Antonio Saura (1930–1998), friend of CoBRA members, and international advocate for the works of the New York painters. The final selection of artworks included those with condition problems noted during the previous collection inventory.

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Table 3.
Number of analysis sites for each study group painting

Artist	Acc. no.	XRF	Sampling	Microscopy	SEM-EDS*	Py-GC-MS	FTIR
Karel Appel	80.68	7	9	3	3		
	66.64		6	3	1		
	66.65	8	11	5	4	2	1**
	66.67	4					
	66.68	4					
Hans Hofmann	66.2483		5	3	6	2	1**
	66.2477	8	8	2	3	2	
	66.2482	4					
	66.2479	2					
	66.2480	7	6	3	2		
	76.125		5	2	2		
Franz Kline	66.2751	4					
	66.2754	4	8	6	9	4	1**
Jackson Pollock	66.4082		3***	2***	1***		
	66.4087		8***	5***	1***		
Antonio Saura	66.4461	3	5	1			
	66.4455	5	5	4	5		
	66.4459	3	8	3	4		

* Four data sets obtained at each analysis site

** Indicates FTIR elemental mapping of one cross section

*** Previous analysis of Pollock works performed by Lake (2004, among others) not included in tally

Note: Number of analysis sites, not including XRF of works excluded from the final study group, Py-GC-MS of samples from SI Materials Study Collection.

2.2 SELECTION OF PAINTINGS

Visual inspection and on-site analysis by x-ray fluorescence spectroscopy (XRF) were used to ascertain similarities in technique and palette, informing the choice of 18 oil paintings for further examination (table 1). Paintings by each of the selected artists with and without visible deterioration comprised the final study group, including works by Hofmann from the period when his New York school was active, two early '60s paintings by Kline, early work by Pollock, paintings by Appel that highlight the shift in palette inspired by his first trip to New York, and

three late '50s paintings by Saura, one each from his female portraits, crucifixion, and self-portrait series.

During selection, attention was paid to repeating condition problems. A review of conservation records, as well as visual inspection of potential study group paintings, revealed two distinct types of deterioration: widespread blind cleavage of the cadmium colors (fig. 1), and a selective cracking of upper layer carbon blacks (fig. 3).

Blind cleavage and lifting of the cadmium colors is found in works by Appel (66.64, 66.65), Hofmann (66.2483, 66.2477, 66.2482, 76.125), and Pollock

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Table 4.
List of white paint samples from the Materials Study Collection (MCI) selected for comparative analysis

Manufacturer	Description	Medium	Cast date
Control (Gamblin)	Lead white (no zinc)	Cold-pressed linseed oil	2/7/1990
		Cold-pressed safflower oil	3/7/1990
	Titanium white (no zinc)	Cold-pressed linseed oil	11/8/1999
		Cold-pressed safflower oil	11/8/1999
		Cold-pressed linseed oil	4/11/1990
		Cold-pressed linseed oil	1/6/1999
Zinc white	Cold-pressed linseed oil	4/23/1990	
Gamblin (Artist's Oil label)	Titanium white (contains zinc)	Alkali refined linseed oil	3/22/1999
Grumbacher	Flake white (contains zinc)	Alkali refined linseed oil	3/19/1999
	Titanium white (contains zinc)	Linseed oil	3/23/1978
		Alkali refined linseed oil	3/19/1999
Zinc white	Linseed oil	3/25/1978	
Speedball (XIX label)	Zinc white	Linseed oil	11/23/1998
		Safflower oil	11/23/1998
Winsor & Newton	Flake white (contains zinc)	Safflower oil	12/26/1980
	Titanium white (contains zinc)	Safflower oil	3/25/1978
		Safflower oil	12/26/1980
		Safflower oil	3/18/1999
	Zinc white	Safflower oil	3/25/1978

Note: Control samples prepared for the Materials Study Collection by Gamblin Artists Colors Co.

(66.4087). This deterioration appears to be associated with an intralayer failure of the underlying paint layer. Examination of cleavage sites shows this failure to be lamellar in appearance, resulting in white underlayer material remaining attached to both the support and the lifting paint (figs. 1, 2).

Selective cracking of carbon blacks is visible in study group works by Kline (66.2751, 66.2754) and Saura (66.4455, 66.4459). The use of two white underlayers is visible to the unaided eye. One white underlayer was applied overall, then partially covered with a sporadically applied second white underlayer. The black remains intact where it is painted over the sporadically applied white, but exhibits widespread cracking where it is in direct contact with the white underlayer that was applied overall (figs. 3, 4).

Although this crack pattern is likely related to the slow drying mechanism of carbon black oil paints, its underlayer-related preferential cracking, as well as its appearance in paintings by more than one artist, recommend its inclusion in this study.

Analysis of the white underlayers in these paintings shows that cleavage of the cadmium colors and cracking of the carbon blacks only occurred above areas of zinc oxide oil paint (table 2). No cracking or cleavage of other colors applied over the zinc oxide underlayer was observed; however, conservation treatment of these works revealed widespread deterioration of the zinc oxide oil paint. This suggests that deterioration of cadmium colors and carbon blacks may be used as a "red flag" to signal the presence of more serious underlayer condition problems.

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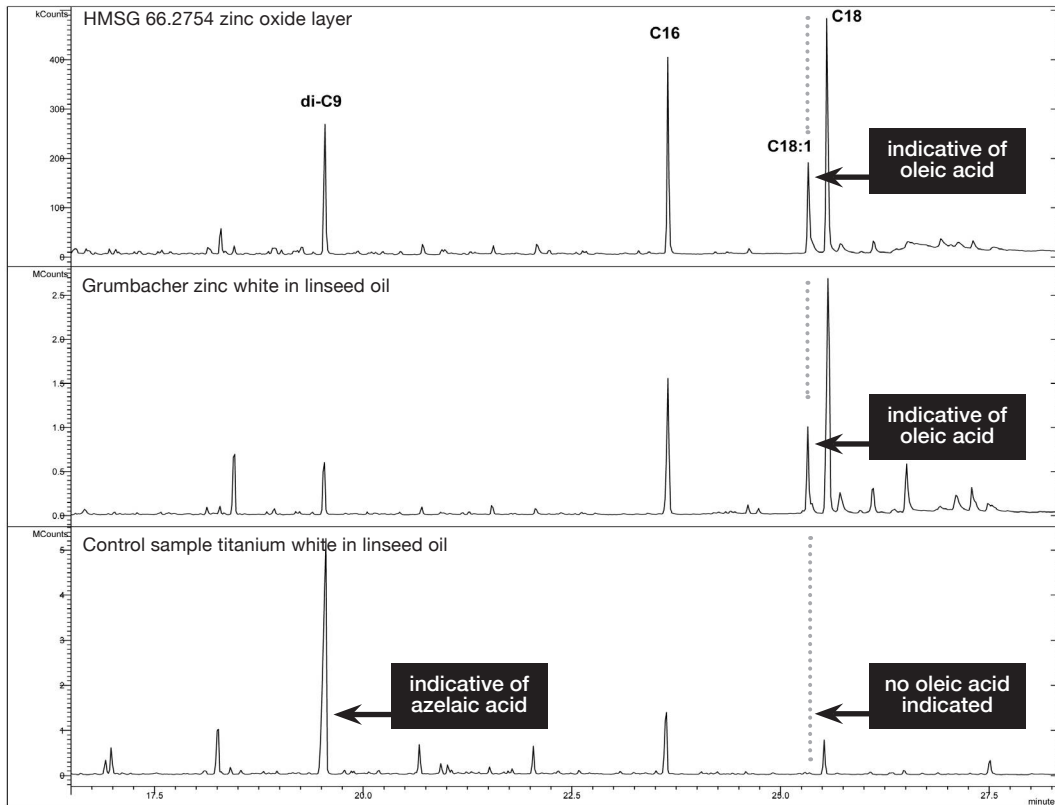


Fig. 5 Gas chromatogram of methylated free fatty acids from study group ground layers. di-C9 = dimethyl azelate, C16 = methyl palmitate, C18:1 = methyl oleate, C18 = methyl stearate. Py-GC-MS analysis reveals unexpected amounts of oleic acid, seen here as methyl oleate (C18:1), in aged zinc oxide oil paint films. Fully cured drying oil films typically contain little, if any, oleic acid (a monounsaturated free fatty acid) as it is converted through oxidation to azelaic acid. In comparison, analysis of the titanium white control sample shows a comparatively large amount of azelaic acid, seen here as methyl azelate (di-C9), and no oleic acid. The extant oleic acid is the result of the formation of plate-like structures consisting of alternating layers of close-packed fatty acid chains and zinc oxide, which limits oxidation and thereby cross-linking of drying oils within zinc oxide oil paints contributing to their brittle nature.

Top: Franz Kline, *Palladio*, 1961 (HMSG acc. No. 66.2754, same sample area as cross section no. 326). Large C18:1 peak seen in zinc oxide-containing ground layer.

Center: Grumbacher zinc white in linseed oil from SI Materials Study Collection, cast 3/25/1978. Large C18:1 peak seen in zinc oxide-containing ground layer.

Bottom: Control sample titanium white in linseed oil from SI Materials Study Collection, cast 4/11/1990.

3. ANALYSIS

The link between visible deterioration and the presence of zinc oxide was initially indicated by XRF analysis. Further analysis of works with and without visible deterioration was undertaken using a variety of analytical techniques (table 3). The presence of zinc oxide underlayers in deteriorated works was

confirmed through microscopy and scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS). Pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS) and Fourier transform infrared (FTIR) imaging subsequently revealed unexpected amounts of unsaturated oleic acid in the aged zinc oxide oil paint layers. Details for all analytical methods are given in the appendix.

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3.1 X-RAY FLUORESCENCE

Using XRF analysis, 16 paintings in storage and on display at HMSG were examined. XRF spectra for areas of cleaving cadmium colors exhibited high zinc peaks, suggesting the presence of zinc oxide in the white underlayer. XRF spectra for undamaged cadmiums contained no zinc peaks. XRF spectra for cracking blacks also exhibited high zinc peaks, while spectra for undamaged blacks showed no zinc peaks. The final selection of 18 study group paintings included works with and without XRF evidence of zinc oxide.

3.2 MICROSCOPY AND SCANNING ELECTRON MICROSCOPY-ENERGY DISPERSIVE SPECTROSCOPY

Material samples were taken from 13 of the 18 study group paintings (table 3). A total of 87 samples were obtained, including samples of support materials, underlayers, and upper paint layers. Samples of paint stratigraphy were taken at or near areas of deterioration, avoiding obvious areas of previous treatment. In some cases, samples with similar stratigraphy were taken from exposed tacking margins. Where possible, samples from undamaged areas were also obtained.

Bright field and dark field illumination, with two filters to view ultraviolet fluorescence, were used to examine 42 embedded samples. In cross sections from damaged areas, a distinctive auto-fluorescence visible under UV radiation indicates the presence of zinc particles in the white layer beneath the damaged cadmium and black paints. SEM-EDS of samples from the same areas confirmed the presence of a zinc-containing underlayer beneath a layer of either cadmium red or carbon black (figs. 2, 4). In a work by Kline (66.2754), microscopy and SEM-EDS revealed a continuous zinc-containing paint layer beneath a sporadic lead-containing layer (figs. 3, 4); XRF analysis of underlayers was inconclusive in areas containing lead paint.

3.2.1 SUPPLEMENTARY MATERIALS

No documentation has been found to identify the specific brand of paints used in the study group paintings. Because of the necessarily limited number of samples available from the original artworks, useful information was obtained by repeating several analyses on samples from the Materials Study Collection

at MCI (table 4). This collection contains aged draw-downs (samples painted out under industry-accepted and consistent conditions) of hundreds of brand name paint samples, as well as a limited number of control samples with simple formulations prepared specifically for the MCI collection. Analytical similarities between known Study Collection paints and unknown paints from HMSG artwork allow the structure and behavior of the known materials to be compared to the structure and composition of the unknown study group paints.

3.2.2 SUPPORT MATERIALS

Samples of canvas fibers were obtained from works by Appel (66.65, 80.68), Hofmann (66.2477, 66.2480, 76.125), Kline (66.2754), Pollock (66.4082, 66.4087), and Saura (66.4455, 66.4459, 66.4461). When discernable, both warp and weft fibers were sampled. Cross-polar microscopy of twelve mounted samples indicated the sole presence of bast (likely linen) fibers on all sampled fabric supports. On-site examination and a review of conservation department records identified plywood on one Hofmann solid support (66.2483). Although the response of support materials to environmental conditions will vary, they appear to exert no noticeable effect on the zinc-related condition patterns.

3.3 PYROLYSIS-GAS CHROMATOGRAPHY-MASS SPECTROMETRY

Paint layer samples from works by Appel (66.65), Hofmann (66.2483, 66.2477), and Kline (66.2754) were analyzed using Py-GC-MS; all samples were portions of samples obtained earlier at or near areas of deterioration, as described in section 3.2. Py-GC-MS analysis of ten samples indicated an oil-based binder for all samples. No alkyds were detected.¹ The same chromatogram plots exhibited unusually high concentrations of oleic acid. For comparison, Py-GC-MS analysis was performed on a range of oil-based white paint samples from the Materials Study Collection. The zinc oxide comparison samples from the Materials Study Collection exhibited the same high concentrations of oleic acid found in the zinc oxide-containing study group paints. Zinc-oxide-free comparison samples showed little to no oleic acid (fig. 5). The amounts of oleic acid found in the study collection paints were independent of the type of drying oil.

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3.4 FOURIER TRANSFORM INFRARED REFLECTOGRAPHY

FTIR false color imaging (results not displayed here) confirmed widespread distribution of unoxidized oleic acid in zinc oxide underlayers in works by Appel (66.65), Hofmann (66.2483), and Kline (66.2754), as well as samples of 20-year-old zinc oxide drawdowns from the Materials Study Collection.

3.5 DISCUSSION OF ANALYSES

The discovery of significant amounts of unsaturated oleic acid is unexpected; in aged oil paint films oleic acid is expected to have been converted through oxidation to azelaic acid. The extant oleic acid in samples of zinc oxide paint films from the study group paintings and the Materials Study Collection is the result of the unique drying process of zinc oxide oil paint.

In an oil medium, zinc oxide forms a tightly packed crystalline structure with a highly ordered lamellar distribution that orients layers of fatty acid chains between layers of the fatty acid carboxyl groups and the zinc matrix. This layered structure makes the paint very stiff, and the highly ordered packing of the hydrocarbon chains makes the unsaturated bonds more difficult to oxidize. Consequently, unsaturated fatty acids in zinc oxide paint remain trapped within the paint layer years after oxidation of the paint should have been completed.² It is as if the paint was prematurely “frozen” in position without the structural stability afforded by the cross-linking that normally accompanies the natural drying process. Shortly after application, zinc oxide has formed an unusually stiff and brittle oil paint.

Recent research has hypothesized that anomalous bond formation within the zinc matrix further disrupts the structure (Vasudevan and Barman 2006). This weakness, along with the zinc–hydrocarbon–zinc layering, may explain the plate-like intralayer cleavage visible on the paintings in areas of deterioration. Studies comparing opposing faces of failure sites in zinc oxide paints (Funke 1967, Eissler and Princen 1972) appear to support the idea of plate formation on a macro scale within the paint layer. Rather than a well-formed paint layer consisting of a uniform cross-linked network, a paint containing zinc oxide consists of a collection of plate-like layered “islands” held together by only a few cross-links.

Analysis of zinc oxide paints from the HMSG

study group and the Materials Study Collection reveal brittle and poorly formed paint films. Zinc oxide oil paint underlayers, such as ground or priming layers, may compromise overall structural stability.

4. INDUSTRIAL LITERATURE

Despite the lack of documentation identifying the specific paints used the study group paintings, there is generous documentary and anecdotal evidence of the Abstract Expressionists’ use of house paints in their work (de Kooning 1950, Goodnough 1952, Lake et al. 2004, among others). Cross-linking and the loss of volatile components during drying result in a comparable loss in the identifiable markers of industrial formulation required for analyzing samples of dried mid-20th century paints. Although particle size and grind consistency may be used as a guide, in the absence of alkyds, there is little that can be done analytically to determine if a mid-century artist was using an oil-based house paint or a similar, commercially prepared artists’ paint (Croll 2009). Ultimately, these similarities in formulation recommend a review of the industrial literature for clues to the behavior of both types of zinc oxide paints.

Until the introduction of titanium dioxide substitutes, the commercial paint industry struggled to fashion a zinc oxide paint that could replace lead white to fill the need for an opaque and durable white coating. Industrial literature from the period directly addresses the behavior and characteristics of zinc oxide oil paint.

4.1 EARLY LITERATURE

Frustrations from within the industry can be found as early as a 1907 treatise on the merits and defects of zinc white that begins: “Zinc white covers poorly. It dries poorly. It stands the weather badly” (Petit and Grant, 84). From the earliest articles, zinc oxide oil paint is acknowledged as reactive, brittle, and difficult to use. It is worthwhile to remember that much early industrial literature was written with an eye towards convincing a wary post-lead market. As with all published research, references to varied sources is preferable. Making note of language usage is also informative—the term “common knowledge” often precedes passing references to important and accepted zinc oxide behavior and failure patterns.

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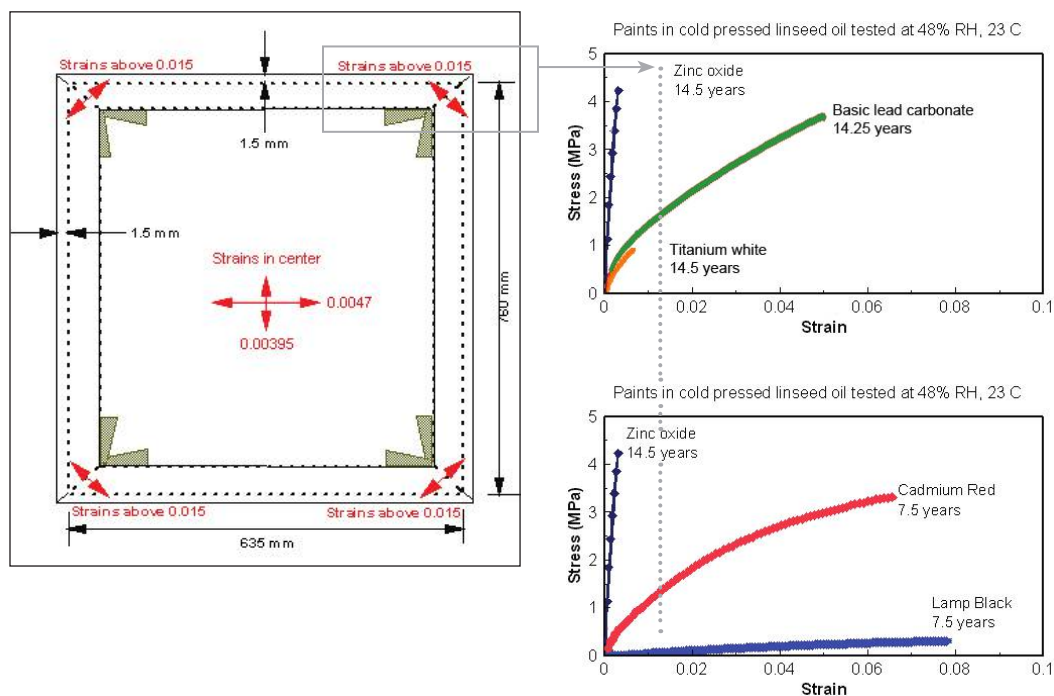


Fig. 6 Strain on a painting, and the corresponding stress response in painting materials. Using paints from the SI Materials Study Collection as examples, tensile testing data can be used to make theoretical comparisons between the materials found in the HMSG study group paintings. In the plot at the upper right, zinc oxide oil paint films are shown to be significantly more stiff and brittle than other typical underlayer materials. The lower right plot indicates a similar disparity in the stress behavior of the materials found in areas of deterioration in the study group paintings. The limited strain imposed at the corners of a painting is indicated by the vertical dotted line; note that the zinc oxide paint film fails before reaching this strain level. Left: Illustration of strain imposed on a theoretical 760 x 635 mm painting. Overall keying out (corner expansion) by 1.5 mm creates 0.015 strain at corners, 0.00395–0.0047 strain at center. Top right: Tensile test results for Materials Study Collection aged control samples of zinc oxide, lead carbonate, and titanium white. Bottom right: Similar tensile test results for zinc oxide, cadmium red, and lamp black (carbon) black (Mecklenburg 2007).

4.2 MID-CENTURY LITERATURE

There is a wealth of industrial literature from the same period as the study group paintings, including a 1949 Oil & Colour Chemists' Association symposium devoted entirely to the discussion of zinc oxide. One presenter at the symposium noted that "zinc fails by checking and cracking with flaking...and erosion which seems fairly severe," but added the optimistic comment that "paints containing zinc pigments have, however, a natural useful life of at least three and a half years" (Bailey and Pass 1953, 183, 171). Anticipating regular repainting, it is clear that the industry definition of "useful" differs significantly from that of the artist and conservator considering the long-term stability of a work of art.

Analysis of the HMSG study group paintings reveals a relationship between the failure of upper layers of colored oil paint with the presence of a zinc oxide oil paint underlayer. For this reason, the industrial literature is particularly useful when it addresses the use of zinc oxide oil-based house paint as a priming layer. The author of a 1935 article from the *Official Digest of the Federation of Paint & Varnish Production Clubs* (Schmutz 1935, 356) states:

It is possible that too little thought has been given as to how a primer might work under different paints.... In some cases there is a marked increase in checking and cracking of the finishing coats and in others an actual decrease in adherence of the

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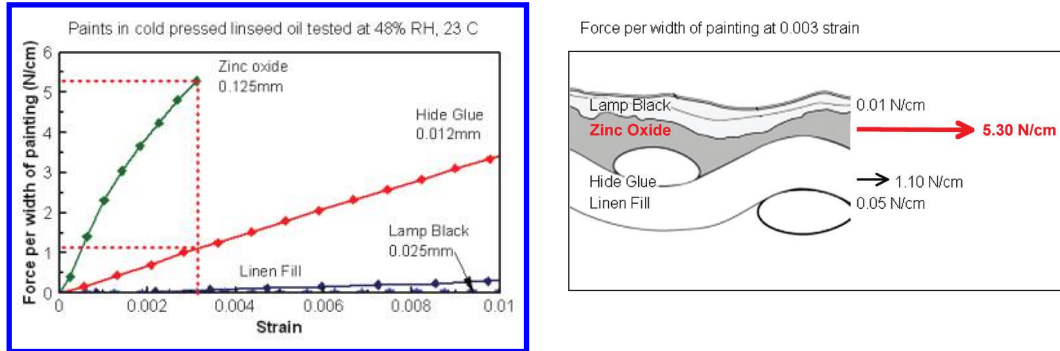


Fig. 7 Theoretical force-strain projection for the layered structure of Kline's *Palladio* (66.2754), using stress-strain data for comparable paint films from the SI Materials Study Collection (Mecklenburg 2007). The plot on the left charts the force imposed on the painting materials; the illustration on the right highlights that imposed force at the point at which the zinc oxide paint film fails, at one fifth of the strain imposed by corner expansion. The highest force is found in the zinc oxide layer until its failure. The forces imposed by uneven artist-applied grounds should also be considered; regions with thicker zinc oxide underlayers would show increased force in those layers. Average layer thickness calculated by sample microscopy; illustrations are based on a drawing by Lopez (Mecklenburg 2007). Left: Force-strain projection based on average thickness of materials in HSMG acc. no. 66.2754. Right: Projected amount of force on materials at the point of zinc oxide layer failure.

whole system... for satisfactory durability, adjacent applications must not differ too greatly from one another in distensibility or hardness.

A 1941 article in *Industrial and Engineering Chemistry* (Browne 1941, 901) notes:

Complete elimination of zinc oxide from primers is recommended by one school of thought on the subject and is opposed by another... Conclusions about the use of zinc oxide in primers must be subordinated to the more fundamental problem of compatibility between primer and finish paint... Leaders of both schools of thought concerning the place of zinc oxide in primers agree that compatibility is a dominant consideration.

The authors of a 1936 study in the same journal conclude that "there is a direct relationship, in terms of performance, between relative hardness of undercoat and top coat, and that certain combinations are incompatible" (Robertson and Jacobsen 1936, 403).

5. MECHANICS

One way to predict the behavior of a layered painting structure is to compare the mechanical properties of each individual layer. For this portion of the project, representative data were culled from previous mechanics research performed over a period of thirty

years on control samples from the Materials Study Collection. Control samples from the collection were chosen based on similarity in pigment composition and analytical testing profile to materials in the HMSG study group paintings. Using paints from the Materials Study Collection as examples, tensile testing data was used to make theoretical comparisons between materials (fig. 6).

5.1 STRESS-STRAIN

Stress is the distribution of force through a material; stress units are generally expressed in pounds per square inch (psi), newtons per square mm (N/mm^2) or megapascals (MPa). Stress-strain plots from a series of tensile tests (Mecklenburg 2007) highlight the stiffness and brittleness of zinc oxide oil paint. This is especially significant when the behavior of the zinc oxide paint film is compared to similar tensile test results for other types of underlayer materials, or to the stress responses of other materials found in areas of visible deterioration in the study group paintings.

To place these plots in context, consider the limited amount of strain imposed by the keying out (corner expansion) of a medium-sized painting. It is worth noting that in both stress-strain comparisons, the zinc oxide paint film fails before reaching this strain level, and before any significant strain is placed on the other pigmented layers (fig. 6).

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5.2 FORCE-STRAIN

Force is defined as any action that causes a body to either deform, accelerate, or both. In a static system (such as a painting) the action of a force is limited to deformation. To better visualize the potential behavior of the HMSG study group paintings, customized force-strain projections were created for the layered structures of works by Kline (66.2754) and Pollock (66.4087), using the stress-strain data for comparable paint films from the Materials Study Collection (Mecklenburg 2007).

Figure 7 is a theoretical force-strain projection for a work by Kline (66.2754). The plot on the left charts the force imposed on the painting materials; the illustration on the right highlights that imposed force on the layered structure at the point at which the zinc oxide paint film fails, at one fifth of the strain imposed by corner expansion (as noted in fig. 6). The highest force is found in the zinc oxide layer until its failure. The forces imposed by uneven artist-applied grounds should also be considered: For this projection, the average layer thickness was calculated by sample microscopy; regions with thicker zinc oxide underlayers would show increased force in those layers.

5.3 HUMIDITY AND TEMPERATURE

The presence of hygroscopic supports should be considered when evaluating the study group paintings. Traditional painting structures contain glue (size) and canvas, which both respond to changes in relative humidity. The zinc oxide oil paint layer, however, is practically non-responsive to changes in humidity, and cannot adapt to dimensional changes in the other materials. (Erlebacher et al. 1992, Mecklenburg 2007)

5.4 DISCUSSION OF MECHANICAL ANALYSES

The stiff and brittle qualities of zinc oxide oil paint are not suited to the flexible and textural requirements of a successful ground or priming layer for Abstract Expressionist compositions. Mid-20th century paintings have the potential for additional problems, as ineffective industrial formulations of zinc oxide were marketed during a period when many artists experimented with house paint. Mechanical stresses and forces on composite structures containing zinc oxide underlayers can result in the widespread

failure of the zinc oxide paint film. Resultant fissures within this paint layer are susceptible to subsequent damage from smaller amounts of stress.

6. RECOMMENDATIONS

Analysis of the HMSG study group paintings suggests that deterioration of paint integrity in regions containing cadmium colors and carbon blacks is a strong indicator of the presence of zinc oxide underlayers. Brittle zinc oxide oil paint underlayers, such as ground or priming layers, pose a serious threat to the overall structural stability of an artwork. The artists in the HMSG study group did not use zinc oxide oil paint as a ground layer for all of their paintings; it is therefore recommended that mid-20th century painting collections be reviewed to determine which paintings might contain zinc oxide oil ground or priming layers. Works with zinc oxide oil underlayers should be carefully observed, particularly during periods of mechanical stress or changes in the environment. Areas of deterioration in cadmium colors or carbon blacks may indicate a compromised ground layer, and should be closely examined. The possibility of blind cleavage and delamination should be considered.

7. CONCLUSION

The examination of paintings from HMSG's mid-20th century collection reveals a link between upper layer deterioration and underlying zinc oxide oil paint layers, and suggests that certain visible signs of deterioration may signal the presence of more serious and widespread condition problems.

A variety of analytical techniques were used to examine the final study group of 18 oil paintings by five artists with ties to the New York City Abstract Expressionist community. Two distinct types of deterioration were observed: widespread blind cleavage of cadmium colors, and a selective cracking of upper paint layers containing carbon blacks. Analysis indicates this deterioration only occurred in contact with zinc oxide oil paint underlayers. Conservation treatment of these works revealed widespread deterioration of the zinc oxide oil paint. This suggests that deterioration of cadmium colors and carbon blacks may be an indicator of underlayer condition problems.

Through analysis of samples from the HMSG study group, as well as comparison samples from the

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SI Materials Study Collection, significant amounts of unsaturated oleic acid in the aged zinc oxide oil paint layers were discovered. The unique drying process of zinc oxide oil paint traps fatty acid chains in a lamellar matrix, creating a brittle and poorly formed paint film that is not suited to the requirements of a ground or priming layer. There is therefore an inherent vulnerability in paintings with zinc oxide oil underlayers. Mid-20th century paintings have the potential for additional problems, as ineffective industrial formulations of zinc oxide were marketed during this period, in which many artists experimented with house paint.

Mid-20th century painting collections should be reviewed to determine which paintings might contain zinc oxide oil ground or priming layers. Such works should be carefully observed, particularly during periods of mechanical stress or changes in the environment.

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APPENDIX:

EXPERIMENTAL CONDITIONS

X-ray Fluorescence

Paintings were examined using a Bruker Tracer III-V handheld energy dispersive x-ray fluorescence spectrometer (ED-XRF). The instrument has a miniature x-ray tube with a rhodium anode, a Si PiN diode detector, and was operated at 35 keV and 15 μ Amps, using a Cu-Al-Ti filter without vacuum. The instrument beam spot size is approximately 3 mm in diameter. Resolution is 149 eV FWHM at 5.9 keV. For these analyses, the portable XRF unit was used in a stationary mode on a tripod. In this configuration, good contact with the object is optimized as much as possible with the instrument window. Exposures were set for 120 seconds in order to maximize the signal-to-noise ratio.

Microscopy

Paint cross sections were mounted for microscopic examination according to the procedure described in Wachowiak (2004). Samples were positioned on precast epoxy half-tablets and adhered using a small droplet of cyanoacrylate adhesive. The sample and half-tablet were then transferred into a silicon rubber mold for embedding. The epoxy resin, Tra-bond 2113 two-part system, was mixed and poured over the adhered sample and half-tablet. The liquid resin was held at room temperature for 1 hour, then cured at approximately 45°C for 3 hours. Once demolded, the tablets were trimmed with a mill to expose the sample. The cut tablet face exposing the paint section was polished with aluminum oxide abrasive, using an aliphatic hydrocarbon as a lubricant. (Some samples were dry polished later for FTIR imaging.)

Samples were viewed on Leica models DMLM and DMRX research microscopes. Both share some components such as objectives and filters. The range of magnification for the reflected light techniques is 50–500x, measured at the eyepieces. Objectives are 5, 10, 20, 40, and 50x. Through the use of a prism system, brightfield and darkfield illumination conditions can be alternated. The tungsten halogen lamp is the primary source of reflected light and it is corrected for daylight color temperature using a dichroic mirror. The fluorescence illuminator is

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Table 5.
Leitz filters for fluorescence microscopy (DMR and DMLM)

Filter system	Excitation	Excitation filter	Split mirror	Suppression filter	Part no.
A	UV	BP 340-380	RKP 400	LP 430	513 804
D	Violet	BP 355-425	RKP 455	LP 460	513 805

BP = band pass filter; RKP = reflection short pass filter; LP = long pass filter

either a 100W mercury or a 150W xenon lamp. The filters span the range from ultraviolet to the violet. The Leitz (Leica) filter sets characteristics are provided in table 5.

A purpose-built digital camera was used for image acquisition (camera chip dimensions, as well as pixel count, determine ultimate magnification of image). The camera system is a Nikon DMX 1200 24-bit color system. The camera uses the Sony ICX085AK color CCD and Nikon's proprietary Inter Pixel Stepping (IPS) high-density imaging technology. Images are normally captured as 24-bit 1.4 Mp tif images, approximately 3.8 Mp each.

Scanning Electron Microscopy-Energy Dispersive Spectroscopy

Samples were imaged and analyzed using a Hitachi S3700-N scanning electron microscope and a Bruker XFlash energy dispersive spectrometer with Quantax 400 software. The samples were received after they had been prepared for and imaged by polarized light microscopy. They were carbon coated before analysis. The SEM was operated at 20 to 25 kV, at full vacuum. Elemental maps were generated over 300 seconds real time (with 0-18% dead time). Spot and area analyses were conducted for 200 seconds live time. Analyses were conducted at a working distance of 9.8-10.2 mm.

Pyrolysis-Gas Chromatography-Mass Spectrometry

Samples were analyzed using a Varian Saturn 2000 GC/MS equipped with a CDS Pyroprobe 2000. Each sample was derivatized using 2 μ L tetramethylammonium hydroxide (TMAH) put onto the sample in a quartz boat. The boat was placed into the coiled platinum probe of a CDS Pyroprobe 2000 filament

pyrolysis unit, and the probe was then placed into a helium-purged CDS 1500 Valved Interface attached to the Varian GC. The interface was held at a constant 310°C and purged with helium for 10 seconds before opening the valve to the GC column. The sample was then heated with the pyroprobe to a temperature of approximately 600°C for 10 seconds. The pyrolysis products were transferred directly to a capillary column (ZB-5ms; 30 m x 0.25 mm i.d.; 0.25 micron film thickness; He flow of 1.2 ml/min; splitless) in a Varian 3800 gas chromatograph (GC) equipped with electronic flow control. The GC oven was programmed with an initial temperature of 30°C, which was held for 5 minutes. The temperature was increased at a rate of 10°C per minute to 300°C and held for 10 minutes. The Varian 3800 GC was interfaced to a Varian Saturn 2000 ion trap, the transfer line being held at 270°C. Operating conditions for the trap were: trap 150°C, manifold at 80°C; electron multiplier 1500 V; scan range 45-650 amu; scan time 1 second; data analysis Saturn GC/MS Workstation 6.5 software and the NIST 2005 spectral libraries.

Mechanics

Paints were cast directly from the tube containers onto polyester films using black vinyl electrical tape as draw-down guides. After an initial drying period of 1-2 days, the electrical tape was removed and the paints were allowed to dry naturally indoors in fairly controlled (24°C and 35-55%RH) environments at the Museum Support Center, Smithsonian Institution. The paint drawdowns are quite uniform. Paint film thickness ranges between 0.125 mm and 0.250 mm. The control paints were manufactured using only specified pigments and oils. As noted, samples were cast in either the 1978-1980 or 1990-1999 time periods. The polyester film was removed prior to testing. All of the

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tests were conducted on free, unsupported paint films, at the same strain rate, and after the paints had fully equilibrated to the environmental conditions. Each test continued until failure of the specimen occurred. Multiple tests of each specimen were conducted to ensure consistency.

The equipment used for conducting the tensile stress-strain tests of the materials was designed and constructed at the Conservation Analytical Laboratory (now MCI) and can be generally described as miniature screw-driven tensile machines. The testing procedure and methodology is described in detail in Mecklenburg and Tumosa (1991).

NOTES

1. This result is expected for the zinc oxide pigment, which, according to documents in the Materials Study Collections Archives, is not used in alkyd formulations due to stability issues.
2. In a similar manner, the matrix restricts the movement of the zinc soaps, which have remained distributed throughout the paint layer in the study group paintings. While fatty acids will typically saponify in the presence of metal salts, it is unusual to find such high concentrations of unreacted unsaturated fatty acids, which is directly related to the unique failure characteristics of zinc oxide paints.

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