Everything Old is New Again: Revisiting a Historical Symposium on Zinc Oxide Paint Films

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ABSTRACT

On June 6, 1949, members of the Victorian Branch of the Oil and Colour Chemists' Association gathered in Melbourne, Australia to share their concerns regarding a sudden increase in problems associated with zinc oxide oil paints, problems which Association members posited were tied to industry-wide changes in processing methods for the zinc oxide pigment. The issues raised during this meeting inform and parallel current studies regarding metal soap behavior. This paper reviews the topics discussed at the 1949 symposium, distilling the historical research and its relationship to modern conservation and scientific inquiry. Topics include existing research literature, pigment processing methods and observed relationships between particle morphology and paint film behavior, and zinc oxide paint film failure patterns. Related metal soap research from the period is also discussed, including investigations of zinc oxide soap formation, the effect of environment on soap formation and film failure in zinc oxide oil paints, and the impact of zinc oxide-specific failure mechanisms on single- and composite-paint systems.

KEYWORDS

Zinc oxide, zinc white, oil paint, zinc soap, metal soaps, paintings conservation

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INTRODUCTION

The Victorian Branch of the Oil and Colour Chemists' Association held a meeting in Melbourne, Australia in the summer of 1949 to discuss a sudden and marked increase in problems associated with house paint formulations containing zinc oxide pigment, and to investigate any possible relationship between this increased failure rate and recent changes in manufacturing methods for the pigment. Ten papers from the meeting were published shortly thereafter in a special issue of *Paint Notes: A Journal of Paint Technology* and are still a valuable resource for modern-day researchers investigating the aging behaviors of oil paint films made with zinc oxide and other metal soap-producing pigments.

The 1949 Symposium on Zinc Oxide highlights how shifts in commercial production can impact the conservation study and treatment of cultural heritage objects.

Modern art objects are often composed of materials produced by industries with manufacturing processes in a state of near-constant flux in response to internal and external market pressures. Production methods play an integral role in material behavior, yet this information is proprietary and therefore rarely accessible to researchers from outside the manufacturing industry. Successful production relies on an in-depth knowledge of material behavior; industrial research literature from this period details competing production methods, links processing techniques to specific material behaviors, and pinpoints shifts in manufacturing method that may help preservation professionals assess the behavior of artwork under their care.

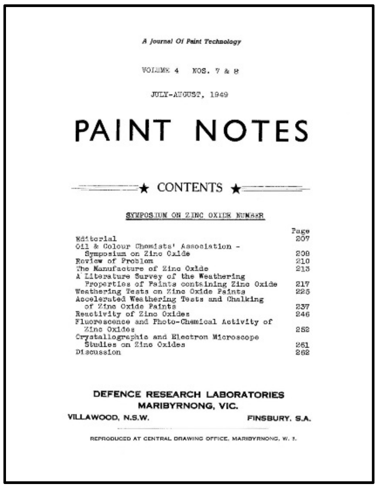
What follows is a review of topics presented at the 1949 symposium. Papers presented at the meeting offer an "insider's" view of the historical research, provide updates on ongoing work, and outline trends in scientific inquiry. Topics include pigment processing methods and observed relationships between particle morphology and paint film behavior. Symposium papers are supplemented here with information from related period research, including examinations of zinc oxide soap formation, the effect of environment on soap formation and film failure patterns in zinc oxide paints, and the impact of zinc oxide-specific failure mechanisms on single- and composite-paint systems. This paper concludes with a summary of the historical zinc oxide research findings of interest to modern conservation and scientific inquiry.

SYMPOSIUM BACKGROUND AND OPENING PRESENTATIONS

The reproduction of papers presented at meetings and symposia is common in research literature, and it is not unusual to see papers in late 19^{th}- and early 20^{th}-century research journals cite one of several regional association meetings at which the paper would be presented. Meetings typically addressed a range of topics, and were attended by a mixed group of material producers, suppliers, and end users. The 1949 Melbourne meeting is unique in its focus. The introduction to the *Postprints from the Zinc Oxide Symposium of the Victorian Branch (Australian Section) of the Oil and Colour Chemists Association* states that while "no zinc oxide problem existed in Australia prior to the war," the introduction of new manufacturing methods designed to meet increased wartime demand for the pigment resulted in paint film problems "noticeable throughout the industry," with a resulting push for further investigation which prompted the meeting (Introduction 1949, 207).[1] Presentations at the symposium were delivered by representatives from paint makers and suppliers; the vice-chairman of the Victoria Branch

[1] A relative definition of "no problem" should be applied to speakers' remarks throughout the symposium papers. The preferred material characteristics of house paint films differ from those of fine art materials. Regular repainting and short life expectancies allowed greater latitude in acceptable behavior for paint films, while some aging behaviors at odds with long-term preservation goals may not appear until after industrial paints reach the end of their anticipated commercial lifespan. Interpreting historical house paint literature from a preservation perspective has been covered previously by this author (Rogala 2011).

"regretted that no paper was to be presented by any of the zinc manufacturers, although they had been invited to do so" (Geary 1949, 208) (Fig. 1).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★ CONTENTS ★

SYMPOSIUM ON ZINC OXIDE NUMBER

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Fig. 1 Contents page from the 1949 Zinc Oxide Symposium postprints, Victorian Branch (Australian Section) of the Oil & Colour Chemists Association. © OCCA. Reprinted with permission.

Manufacturing methods

Coatings manufacturers opened the symposium program with a review of pigment production methods and existing scientific theory regarding the behavior of zinc oxide pigment in oil paint films. The work of pigment suppliers received even-handed treatment; the vagaries of production and market pressures were understood by paint manufacturers and researchers and accepted as a variable to be included in any study of paint film behavior. C.H.Z. Woinarski, then Senior Chemist at Hardie Trading Ltd., outlined the shift in paint production and performance observed during World War II, when new methods of pigment manufacture emerged—at newly built or converted facilities—to meet an increased demand for zinc oxide pigment (Woinarski 1949). Direct Process (also called American Process) methods of pigment production were replaced by Indirect Process (sometimes called French Process) methods, and this manufacturing shift was accompanied by a precipitous rise in failure rates of oil-based house paints containing zinc oxide pigment produced using the new method.[2] These disparate manufacturing processes were further delineated by D.G. Davidson, Works Manager at Goodlass Wall & Co., Pty. Ltd., who summarized the substantive differences as 1) production through simultaneous reduction and oxidation (Direct Process) versus oxidation alone (Indirect Process) and 2) particles whose size and shape are largely inconsistent (Direct Process) or consistent (Indirect Process).

Pigment morphology

Particle shape and size were the focus of a literature review provided by K.R. Bussell, who noted a prevalence of marketing bias in research that "postulated

[2] Indirect Process production was also an economical alternative due to higher pigment purity, lower lead content, lower water soluble content, greater hiding power and tinting strength, and improved storage properties (Schmutz 1935, Morley-Smith 1950a).

theories explaining superior durability of certain types of zinc oxide with little or no experimental evidence to justify them," and were therefore of little value in accurately assessing the role of particle morphology in the observed increase in film failure of oil-based zinc oxide paint (Bussell 1949, 224). This literature does, however, offer useful descriptions of particle shapes and manufacturer expectations. At the time of the symposium, zinc oxide pigment was available in three shapes—amorphous, acicular, and irregular. Amorphous particles are defined in the period literature as crystalline, mostly spherical, and of consistent shape, while acicular particles are needle-shaped, and less prone to clumping during mixing because of their uneven surfaces. Irregular particles (the term nodular is used by some authors) can be a mixture of sizes and shapes, and include both amorphous and acicular forms.[3] Both Bussell and Davidson reported that Direct Process zinc oxide particles tend to be larger and acicular, characteristics believed to be less reactive and cause fewer problems, while zinc oxide produced using the Indirect Process resulted in smaller, more reactive particles with fewer acicular components. Film-forming behaviors attributed to acicular pigments that would be undesirable in artwork include the development of "innumerable microscopic failures" as gaps between binder and large pigment particles created during drying of the paint film (Bussell 1949, 221). Bussell's review included work by authors whose names appear throughout the historical zinc oxide literature, including Kekwick and Pass (1938), Werthan (1941), and Calbeck, Eide, and Easley (1941).

A 1940 *Chemical Industries* article by H.A. Nelson (Fig. 2A) referenced by Bussell provides more information on the pigment structures resulting from various production methods. Zinc oxide is typically a crystalline, close-packed lattice structure of alternating zinc and oxygen atoms. Nelson confirms that Direct Process oxides contained various percentages of acicular variations on the crystalline form, some joined to form "twins" and "threelings" (referred to as "brush-heap" formations by Bussell), while Indirect Process zinc oxides (often marketed under the term "Seal" oxides) were typically irregularly shaped particles of uniform size distribution (Nelson 1940, 509). Nelson suggests that the acicular particles found in Direct Process zinc oxides delay film failure but are more likely to cause localized, longitudinal failures in oil-based house paints, while the film failures related to Indirect Process zinc oxides appear more quickly and are primarily useful, "self-cleansing" failures (surface-layer shedding/sloughing) of even distribution. (Nelson was at this time in charge of the paint research division at the New Jersey Zinc Company, part of the industry using the Indirect Process.)

ZINC OXIDE RESEARCH PRIOR TO THE SYMPOSIUM

The remaining presentations at the Zinc Oxide Symposium were devoted to early-stage and continuing research that examined the relationship between pigment characteristics and zinc oxide paint film behavior, with a particular focus on

[3] Present-day texts simplify zinc oxide particle shapes into two primary categories: nodular and acicular, with the former used in reference to characteristically fine, rounded particles.

differences between oil-based films containing Direct Process and Indirect Process pigments. J.R. Rischbieth and W. Griffiths updated attendees on early results from weathering tests that supported observations of the superior performance of Direct Process over Indirect Process zinc oxide, the slight but not substantive improvement provided by the addition of boiled oils (Bussell), and the acceleration of film failure related to increased exposure to moisture (Woinarski). Just as industrial materials used by artists are formulated for behaviors that may conflict with preservation goals, the needs of the paint industry are complicated by their reliance on materials manufactured for other purposes. Griffiths points out that the war-time increase in zinc oxide production was directly related to an expanded need for rubber, with more than 50 percent of zinc oxide sales supplying the rubber industry (Griffiths 1949, 244). Acceptable behaviors for the newly produced pigment were therefore not the same as those for the zinc oxide primarily manufactured for use in paint films. Both Rischbieth and Griffiths noted the lack of research on the effects of new pigment production techniques on affiliated industries and called for further investigation into particle morphology and zinc oxide reactivity.

Metal soaps

The first Zinc Oxide Symposium paper to address the role played by metal soaps in paint film failure was presented by H.A. Laurie and D.K. Box, who discussed surface area and particle shape in relation to the formation and control of zinc oxide soaps. Their review of existing research revisited problems associated with marketing bias but found useful information on the influence of surface area on metal soap formation, with the reduced surface of acicular particles deemed less reactive. The potential for continued formation of crystalline metal soaps throughout the lifetime of a paint film was noted, but the presenters downplayed the seriousness of this situation as the articles addressing this behavior were primarily written by industry competitors (lead pigment companies). Laurie and Box did alert attendees that their own examination of commercial zinc oxide determined that pigment labeled as Direct Process or Indirect Process often contained particle mixtures and impurities that could skew research results. They concluded that while experimental data was still limited, "the effect of zinc soaps on the properties of paint films . . . [is] of interest and point[s] to a further line of investigation" (Laurie and Box 1949, 251). Familiar authors mentioned by Laurie and Box included Robertson (1942), Dunn (1946), and Dunn and Baier (1948).

In related literature, an early paper by Nelson and G.W. Rundle suggested that paints be formulated with oil binders of low acid number to control soap production, along with limited use of hygroscopic materials such as glycerol to control the strongly moisture-absorbing characteristics of zinc oxide paint films, a behavior "not directly proportional to the relative humidity" and unique among other pigments in the length of time required to return to equilibrium (Nelson and Rundle 1923, 365-367). Work published in the early 1940s by D.G. Nicholson and his team from the University of Illinois found that continued soap production—beyond that necessary for film formation—had a negative impact on film strength and moisture sensitivity of the paint film. Acicular particles were again viewed favorably in terms of strength and durability. Nelson's claim that acicular particles show longitudinal failure patterns was supported by an observation that "acicular films tend to crack in lines which parallel the stroke used in their application" (Nicholson and T.W. Mastin 1942, 999). Other points of interest in this research include reports that 1) zinc oxide pigment can react with moisture prior to paint formulation and become more basic (a reference to Eide and Depew 1936), 2) zinc oxide pigment treated with carbon dioxide gas makes a more durable paint film, and 3) exposure to atmospheric contaminants can vary drying times and behaviors (from Nicholson 1941). Like other researchers, Nicholson and Mastin found incomplete or inaccurate labeling of commercial Direct and Indirect Process pigments.

Performance in a layered system

The compatibility of zinc oxide oil paints and adjacent pigmented paint films arose as a topic of research interest prior to the symposium, with the emphasis placed on layering paints that exhibit well-matched material characteristics. Research from the U.S. Department of Agriculture summarized the argument against zinc oxide oil paint in composite-paint systems by stating that the behavior of zinc oxide paint films differed so greatly from other paint films that it was not recommended as a priming layer for any paint system that did not also contain zinc oxide pigment (Browne 1936 and 1941).[4] Similar research by industry competitors is mentioned at the 1949 meeting by Laurie and Box (a reference to Dunn), and more than a decade before the Zinc Oxide Symposium, a presentation to the Baltimore, Chicago, Northwestern, Philadelphia, Pittsburgh, and Western New York Paint and Varnish Clubs by Titanium Pigment Company representative D.W. Robertson noted that differences in moisture absorption between adjacent oil paint films could also lead to failure through the release of water-soluble compounds between paint layers, resulting in loss of adhesion in the interface between paint layers or between paint and support layers. Interface failure was noted by Robertson in paints that formed zinc oxide soaps, because "metal-oil compounds developed by the interaction of linseed oil and metallic salts have poor adhesive properties compared to the original oil film" (Robertson 1935, 229). A 1941 paper from Titanium Pigment Company representatives A.E. Jacobsen (who also published with Robertson) and W.H. Gardner (Fig. 2B) looked at zinc oleate soaps and reported finding 1) an increase in soap production in the presence of basic pigments and 2) differences in structure between salts formed under normal (stoichiometric) conditions and those produced under conditions with an excess of zinc oxide (sometimes forming complex salts of layered zinc and oleic acid).[5] Twenty-five percent zinc oxide is suggested in the pre-symposium literature (Keckwick and Pass 1938, Nelson 1940) without evidentiary

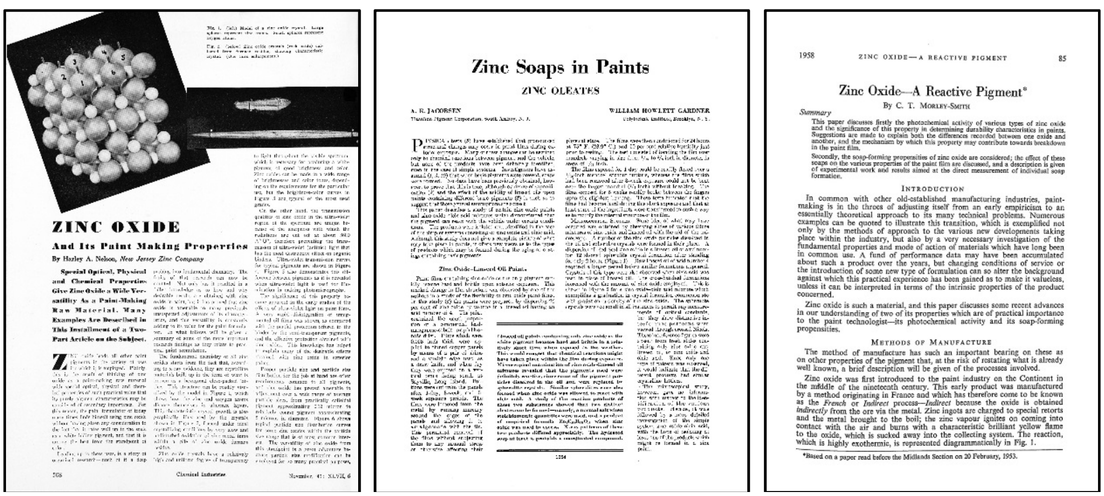
[4] Attendees at a 1949 meeting of London Section of the Oil and Colour Chemists' Association also noted the chalking of zinc whites in simple zinc-and-varnish layering (Morley-Smith 1950b, 268).

[5] Industry research on the poor adhesion of stoichiometric salts in oleic acid appears as early as 1929, in a presentation at the Philadelphia Club by J.T. Baldwin which reported preliminary testing results suggesting that metal soaps appearing in zinc oxide paint remained separate from the body of the paint film under all testing conditions (Baldwin, 866).

support. Pigment volume concentrations for zinc oxide paint films became the subject of further research following the symposium.

Particle fluorescence

The 1949 symposium closed with two papers that reflected increasing interest in developing new analytical techniques for evaluating paint film characteristics and behavior. R.I. Garrod (1949) suggested a possible correlation between ultraviolet-induced visible fluorescence and pigment particle size, and G. Winter and R. N. Whittem (1949) posited a relationship between fluorescence and pigment reactivity. Winter and Whittem noted that research in this area was just beginning, and while fluorescence microscopy was a promising technique, results could be compromised by the impurities and mixed particle types found in commercial pigments, by altered pigment morphology (related to manufacturing), and by disrupted crystalline structures (due to zinc soap formation, for example). Further study was recommended before the usefulness of fluorescence as an analytical technique could be properly assessed. Authors cited by Winter and Whittem include Kekwick (1938) and Robertson (1942).



Figs. 2A-C Examples of period research on zinc oxide pigment in oil paint vehicles (l-r): "Zinc oxide and its paint making properties" (Nelson 1940), "Zinc soaps in paints: Zinc oleates" (Jacobsen and Gardner 1941), and "Zinc oxide—a reactive pigment" (Morley-Smith 1958). © NJ Zinc Co., ACS, OCCA. Reprinted with permission.

POST-SYMPOSIUM RESEARCH

Research following the Symposium on Zinc Oxide built on promising hypotheses from the 1949 meeting. The fluorescence of zinc oxide was further explored as attempts were made by pigment manufacturers to modify Indirect Process pigments to better mimic the stability and aging characteristics observed in Direct Process zinc oxides. In an announcement made to the 1950 meeting of the Oil and Colour

Chemists' Association (New South Wales Branch), a representative of the Zinc Pigment Development Association at Durham Chemicals acknowledged the roles played by moisture and particle size in zinc oxide film failure, but hypothesized the existence of another, more controllable variable with influence over the performance of zinc oxide oil paints (C.T. Morley-Smith 1950a). Fluorescence microscopy was used to differentiate between zinc oxide particles exhibiting green fluorescence (said to be produced in an oxidizing atmosphere) and particles exhibiting purple-blue fluorescence (produced under reducing conditions). The "green" particle was reported to contain stoichiometric proportions of zinc and oxygen, while the "blue" particle showed excess zinc and interstitial zinc atoms capable of initiating photochemical reactions and accelerated film failure (Morley-Smith 1958, 89) (Fig. 2C). Despite the mixed reduction-oxidation conditions of Direct Process production, the Direct Process zinc oxide pigment showed more of the stable, less reactive "green" particles. (Indirect Process particles tended towards dull green-brown and purple-blue fluorescence.) Morley-Smith reported that efforts were underway to reduce the inclusion of ultrafine particles (more surface area) in zinc oxides, and to modify Indirect Process particles to exhibit green fluorescence (and the attendant performance characteristics).

F.L. Browne revisited the high swelling rate of zinc oxide oil paints and confirmed responsiveness of the paint films to high humidity and applied water, with paints incorporating Indirect Process pigments showing greater sensitivity than those made with Direct Process pigments (Browne 1957). Browne's work supported earlier suggestions that stored pigment could become more basic through carbonation, and that paint films containing zinc oxide retained some amount of swelling after drying (Browne 1955, 1957). Additional research from the U.S. Department of Agriculture corroborated reports that even small amounts of zinc oxide pigment "exert a determining influence on [paint film] properties" (Eissler and Princen 1966, 19). Increased swelling was attributed to the basic nature of zinc oxide, as "the weakly acid character of linseed oil is insufficient to overcome excess alkalinity contributed by certain pigments to a film in the presence of water." Equivalent swelling was reported in zinc oxide oil paint films containing 1 percent and 30 percent volume concentration of the pigment (Eissler and Princen 1970, 155). Continued weathering research by Rischbieth showed that problems related to moisture often appeared months after exposure to water (1957). Research from the University of Stuttgart built on rubber industry literature citing a possible link between the oil and resin vehicle and swelling behavior in zinc oxide paint films (Funke 1967), and related tests by Morley-Smith on the influence of fatty acids in zinc oxide reactivity suggested that soap formation slowed with increasing acid chain length, and noted that "a marked difference was apparent between the behavior of the saturated and unsaturated acids, unsaturation reducing the rate of soap formation appreciably" (1958, 94).

Table 1 Comparative zinc oxide pigment characteristics as reported c. 1949 in the paint research literature

Direct/American Process	**Indirect/French Process**
Established production in U.S. mid 19th century; other markets (e.g. Australia) from early 20th century	Industrially produced in France in 1840s; introduced in other markets (e.g. Australia) during WWII
Manufactured from ores via simultaneous reduction and oxidation processes; heavy metal impurities	Manufactured from zinc metal via oxidation; high relative purity
Larger, variable particle size	Fine, relatively uniform particle size
Smaller reactive surface area	Larger reactive surface area
Predominantly acicular particle shape	Predominantly nodular particle shape
Stoichiometric crystal structure	Non-stoichiometric crystal; interstitial zinc atoms
Predominantly green UV fluorescence	Predominantly purple-blue UV fluorescence
Forms relatively durable films	Poor relative durability
Associated with longitudinal cracking in paint film	Associated with widespread/uniform chalking of paint film
Favours localized soap formation in paint film	Favours layered soap formation in paint film
Paint films experience high, prolonged swelling response to water; pigment is less sensitive to RH than Indirect Process	Paint films experience high, prolonged swelling response to water; pigment is sensitive to RH

SUMMARY OF THEMES AND RELATION TO CONTEMPORARY RESEARCH

There has recently been renewed interest in the behavior of metal-based pigments in paint films, and historical literature can be a valuable resource. Papers from, and related to, the 1949 Symposium on Zinc Oxide present substantive research regarding pigment production and the behavior of oil paint films containing zinc oxide pigment. Table 1 outlines the comparative pigment characteristics and behaviors as presented in the period literature. Direct Process zinc oxide is reported to be produced through a combined reduction-oxidation process in stoichiometric conditions that results in larger, acicular particles with reduced surface area. Direct Process zinc oxide is said to be less reactive (on a relative scale) than other zinc oxides. Indirect Process zinc oxide, by comparison, is produced solely through oxidation, resulting in smaller particles with more reactive surface area and the potential for excess zinc and interstitial zinc atoms available for further reaction. Period research associates Direct Process zinc oxide with localized, longitudinal failures in oil paint films, and with the potential for localized soap formation at groupings of pigment particles spaced unevenly throughout the paint film. Indirect Process zinc oxide is reported to show a tendency to form soaps composed of layered pigment and fatty acids, positioned throughout the paint film. The symposium postprints also link the shift by regional manufacturers from Direct to Indirect pigment processing methods with increased war-time demand for zinc oxide, which may be of interest to conservation and material science researchers investigating the appearance of lamellar zinc oxide soap behavior in mid-twentieth century paintings.[6]

Zinc oxide oil paint films were reported to exhibit high swelling behavior. Even small amounts of zinc oxide in a paint film were said to induce swelling. Exposure to moisture was reported to enhance film failure, with some failures appearing long after the exposure period. The research suggested that moisture remained in the paint film and prolonged swelling, thereby extending moisture-related reactivity and alteration of the paint film's physical properties. Water-soluble compounds and metal soaps could also become trapped between paint layers, leading to adhesion loss. Zinc oxide pigment was reported to be sensitive to moisture while in its raw state, prior to paint formulation. Several papers from the 1949 meeting claimed that water exposure prior to mixing made the pigment more basic, and that alkaline pigments could exhibit increased soap production, which in turn made the zinc oxide paint film more moisture sensitive. It is important to note that the historical research related to moisture sensitivity utilized levels of liquid and lengths of exposure unlikely to be replicated in standard conservation treatment; further research is needed to determine what impact moisture exposure (and duration of exposure) may have on the preservation of art materials containing zinc oxide pigment.[7]

A strong grounding in industrial research is central to the preservation of modern artworks. For modern-day researchers of aging behaviors and metal soap formation in zinc oxide oil paints, mining this literature is not as simple as dating a shift from Direct to Indirect pigment production or using fluorescence microscopy. Terminology can be confusing, or vary from author to author.[8] Product labeling can be inaccurate or incomplete. Advancing technology can also confuse physical characteristics, as in the attempts to modify Indirect Process pigments to mimic the behavioral and analytical markers of zinc oxide pigments made using Direct Process methods. The behavior of art materials such as zinc oxide pigment will be affected by the changing needs of other markets, from wartime increases in rubber production to the contemporary call for semiconductor coatings on electronics. The network of influences apparent in historical research relates to era-specific manufacturing trends just as modern-day market pressures direct contemporary

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[6] Previous work on this topic by this author includes Rogala et al 2010, Maines et al 2011.

[7] Weathering tests and lab experiments used to evaluate house paints far exceed the exposure conditions anticipated from a single conservation treatment, but the material response patterns revealed in these studies are relevant to cultural heritage preservation interests, e.g. the cumulative effects of conservation treatment over the lifetime of an artwork and the long-term exposure of art materials to unregulated environments.

[8] Examples of confusing language pairings include Direct/American, Indirect/French, and Irregular/Nodular. In one audience exchange at the 1949 symposium, "Mr. Sutton asked whether the various phenomena of erosion, cracking, chalking, etc. could not perhaps be merely manifestations of one and same thing" to which Mr. Rischbieth "admitted that there probably was . . . some connection between these phenomena" (Discussion 1949, 265).

material science research and innovation. With careful study, historical research can be a valuable resource in the preservation of cultural heritage objects.

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