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# Surfactants & Solvents: Potential for MCP - Designed Nanostructured Fluids for the Removal of Polymer Films

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For conservators few things are as frustrating as designing a solution for a complex cleaning problem and ending up with undesirable, inconsistent, or negligible results. It's detrimental to the object, our time, and our already restricted budgets. The Modular Cleaning Program (MCP) developed by Chris Stavroudis and its widely accepted underlying methodology go a long way in providing a systematic approach to solving difficult cleaning problems, giving conservators a variety of tools to manipulate in a controlled manner.

However, an underutilized and often misunderstood cleaning tool within the MCP (with untapped potential in solving our cleaning problems) is the surfactant. In order to effectively utilize surfactants we need to understand as many of their properties and interactions as possible.

While their activity at the solid-liquid interface is widely reported in the relevant literature, it is not sufficient to consider this alone. We need to understand the interactions between the surfactant and other components within the solution itself in order to effectively control and fine-tune what we're doing. As emulsions and microemulsions become more prevalent we especially need to consider surfactant interactions within systems that contain both water and organic solvents.

In this article, I am going to delve into surfactant and solvent properties, their interactions, and how we can manipulate them to give us our desired results: safer, more effective, and more controlled removal of soiling materials and polymer films.

We'll explore how polar solvents increase detergency, the advantages of nonionic surfactants in comparison to ionic surfactants, the dewetting of polymer films, and how to practically apply these concepts using the MCP.

## Out of the Clouds and into the Studio

A significant property of a surfactant to consider is the cloud point temperature ( $T_c$ ). The  $T_c$  of a surfactant solution is the temperature at which phase separation occurs, resulting in two distinct phases: surfactant rich & surfactant poor. Cloud point temperature is generally measured by dissolving 1% (w/v) surfactant in water, then warming the water until it transitions from clear to cloudy (phase separation). A surfactant's  $T_c$  is often higher than ambient room conditions, so normally a solution will be clear.

So, the cloud point temperature can be reached by raising the temperature of a surfactant solution. It can also be reached if the  $T_c$  is lowered by something that interacts with the surfactant, such as increasing salt concentration or adding polar hydrogen bond accepting (HBA) solvents.

An example of a lowered  $T_c$  caused by a polar HBA solvent would be the addition of a solvent like MEK (butanone) to a nonionic polyoxyethylene surfactant like Ecosurf EH-6. Here, clouding results from decreasing the hydration of oxyethylene oxygens in the surfactant's hydrophilic head group(s).

Solvent-induced lowering of  $T_c$  occurs by increasing the ratio of the dispersed polar solvent phase (MEK) to the aqueous continuous phase. Essentially, MEK is such a strong hydrogen bond acceptor<sup>1</sup> that it interferes with the interaction between water and the polar head of the surfactant, resulting in dehydration of the polyoxyethylene (POE) chains, thus bringing the system's  $T_c$  closer to room temperature. Understanding this interaction enables us to use it to our advantage.

## Detergency and You

So why would we want to move the  $T_c$ ?

Because detergency, a surfactant's relative effectiveness in separating a soiling material from a substrate as a function of concentration, increases as the surfactant gets closer to its  $T_c$ . Maximal detergency occurs just before phase separation at  $T_c$ .<sup>2</sup>

In this instance detergency consists of rolling-up, emulsification, and solubilization of oily soil layers or components of polymer films. By manipulating the cloud point temperature of a surfactant, for example through the addition of a polar organic solvent, increased detergency can be achieved while utilizing less surfactant.

For some surfactants, like those in the MCP, 2-5x critical micelle concentration (CMC) can generate the same cleaning effect as the 10x CMC generally recommended for aqueous cleaning systems. This significant reduction of surfactant concentration results in easier clearance and less potential residue on the surface being cleaned.

Further, some surfactants nearing their  $T_c$  experience an elongation of their spherical micelle shape, which results in a prolate spheroid. Size reduction of micelles has also been consistently observed near  $T_c$ . Both behaviors improve cleaning through an increase in the soil-micelle interfacial contact zone (Fig. 1).<sup>3</sup>

The disadvantage of a reduced cloud point temperature is that exceeding it will result in phase separation, a potential disaster in conservation treatment context. This is a very real concern when working in environments where uncontrolled temperatures may influence a surfactant's  $T_c$  (outdoor sculptures, wall paintings, archaeological sites, etc.).

## Microemulsions vs. Micellar Solutions

Before going any further, a brief clarification on the difference between oil-in-water (o/w) microemulsions and the swollen o/w micellar solutions will be discussed here.

O/w microemulsions contain organic solvents that are not soluble in the aqueous continuous phase. The dispersed solvent is contained completely within the micelles, resulting in a more thermodynamically stable system.

This also enables you to tune the size of the micelles by increasing or reducing the amount of solvent in the microemulsion. Less solvent results in smaller, but still

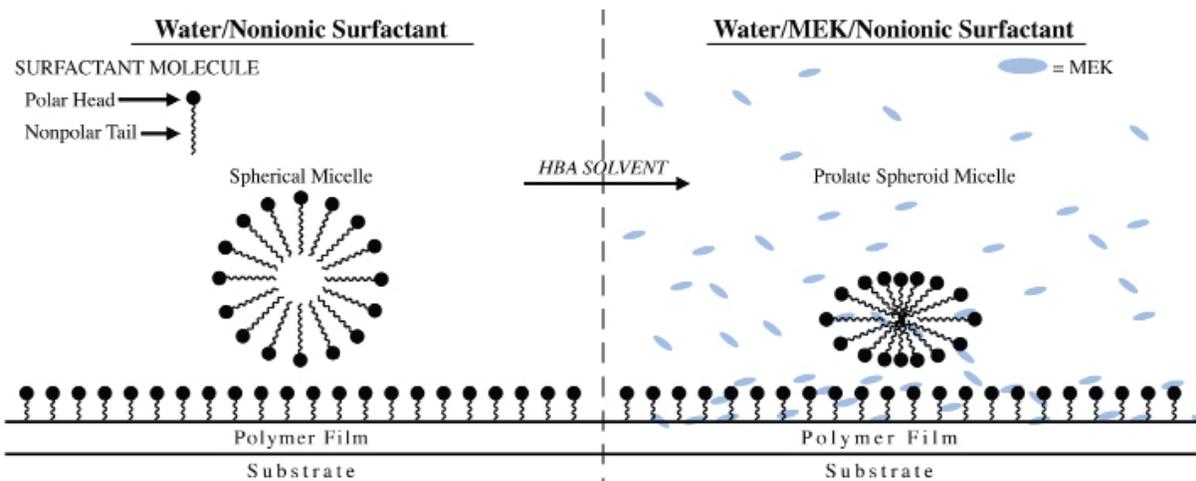


Figure 1. Left: Spherical micelle in water with surfactant layer adsorbed on polymer film. Right: Addition of HBA solvent resulting in prolate spheroid micelle and interaction with polymer film. Illustration by Joe Brown.

strained micelles. A micelle size of less than 100nm in diameter is generally favorable for microemulsions and often results in an optically translucent solution.

In a swollen micellar solution the dispersed organic HBA solvent (like MEK or propylene carbonate) is partially soluble in the aqueous continuous phase. As a result the solvent is not only dispersed throughout the aqueous continuous phase, but also inhabits the interface between the micelle shell and water (Fig. 2).<sup>4</sup>

For water-in-oil (w/o) microemulsions, water is the dispersed phase contained inside the micelles while the organic solvent (Shellsol D38, xylenes, etc.) is the continuous phase. All of the above can be generally classified as nanostructured fluids (NSFs) due to the self-assembly properties of the surfactant.

### Nonionic vs. Ionic Surfactants

The ternary water/polar solvent/nonionic surfactant systems investigated by Baglioni et al.<sup>5</sup> are excellent examples of swollen o/w micellar solutions.

A typical formulation would be 80mL water, 20g MEK, and Triton X-100 (2-5x CMC) at room temperature 25°C.. These newer systems have multiple advantages in comparison to previously reported ionic surfactant systems consisting of the strongly anionic surfactant sodium lauryl sulfate (SLS), 1-pentanol (cosurfactant), propylene carbonate, and ethyl acetate.<sup>6</sup>

Anionic surfactants such as SLS are particularly sensitive to ionic materials, whether present in the cleaning system or the surface undergoing treatment. Consider  $Ca^{+2}$  ions that may leach from a gesso layer on a panel painting, polychrome wooden object, or fresco. The resulting complex of  $Ca^{+2}$  and anionic surfactant would yield a difficult to clear metal soap with very low water solubility.

Three major advantages of nonionic surfactants are that they are not as affected by ionic materials (salts), are more easily compatible with the buffers and chelators already in the MCP, and more readily self-assemble into micelles and at interfaces.

Another advantage is that they much more effectively solubilize triglycerides, the backbones of oil & alkyl coatings, near their  $T_c$  in comparison to ionic surfactants.<sup>7</sup>

Further, these newer systems utilize MEK, which is generally available throughout the U.S. while ethyl acetate and propylene carbonate are more boutique solvents that are less available and familiar. While propylene carbonate is an excellent material that outperforms MEK in certain applications, MEK still gets the job done.

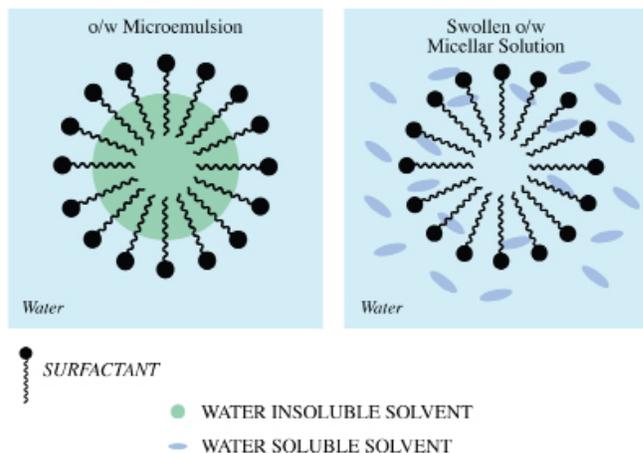


Figure 2. Left: Oil-in-water microemulsion droplet. Right: Swollen o/w micellar solution with a water soluble solvent. Illustration by Joe Brown.

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## Surfactants & Solvents: Potential for MCP - Designed Nanostructured Fluids for the Removal of Polymer Films, continued

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One final advantage of these newer systems is the solubility of MEK in water: 21.1% w/v at 25°C, making a max 20% w/v solution of MEK/water and surfactant straightforward in preparation. No cosurfactants or additional cosolvents are required.

Baglioni reports the cleaning process (removal of a soil or polymer coating) likely consists of two processes:

- 1) swelling and partial detachment of the coating by the solvent/NSF, then
- 2) removal of the coating by a detergency-like and/or dewetting mechanism.<sup>8</sup>

With detergency contributing to these processes it should be clear why working closer to the  $T_c$  is advantageous (closer to  $T_c$  = stronger detergency). The closer the surfactant is to its  $T_c$  the more quickly and efficiently both steps should proceed – an important consideration when dwell times can be upwards of 1.5-3 hours.

### Dewetting & Detergency

It is important to note that detergency and dewetting are separate, distinct mechanisms.

Dewetting is a complex phenomenon, but for our purposes it can be practically understood as the spontaneous separation of a polymer film from a substrate. It can be induced by application of any class of appropriately designed NSF.

Detergency contributes to the dewetting process. Film components like free fatty acid chains, synthetic polymer chains, intermixed grime, etc. can be extracted during NSF-induced swelling of the film, thus accelerating dewetting.

Further, as the hydrophobic polymer film dewets or detaches from the substrate the surfactant aids in desorption and solubilization of the film itself. The dewetted state of a hydrophobic polymer film is actually more thermodynamically stable and energetically favored in comparison to its metastable swollen state.

Essentially, we are taking advantage of the fact that the polymer film has a greater affinity for itself than for the substrate under the conditions we subject it to. We coax it into pulling away from the surface and into itself to form more stable polymer droplets.

Think of a spherical droplet of water on a waxed car. If you press the droplet down with your finger you force it to spread over the hydrophobic wax surface (wetting). When you remove your finger the water droplet spontaneously reforms in a spherical shape (dewetting).

The water molecules require less energy to be in contact with each other than with the waxed surface due to the surface energy difference between the materials. It requires energy, or a surfactant, to overcome this difference. So, the lowest-energy configuration for that water droplet is a sphere because as few water molecules as possible are in contact with the hydrophobic surface in that configuration.

By applying a nanostructured fluid to the polymer film and swelling or rehydrating it, you are removing the finger holding the droplet down and enabling it to reform as a droplet (dewetting).

If you're having flashbacks to physics and surface tension equations, hang on. It's only going to get a little worse before it gets better.

The NSF-induced swelling of a polymer film lowers the film's glass transition temperature ( $T_g$ ) below room temperature. This effectively weakens intra- and interchain polymer interactions and enables the mobility of the polymer chains.

Think of the swollen film as a fluid instead of a solid, or cooked vs. uncooked spaghetti.<sup>9</sup> After swelling, to induce dewetting, the surfactant plays an important role in reducing the interfacial energy difference between the involved phases. It also decreases the activation energy barrier that prevents the spontaneous dewetting of the film. At this point dewetting occurs due to wave-like oscillations within the polymer film. These are caused by thermal energy, motion, and vibration of polymer molecules (Fig. 3).<sup>10</sup>

With appropriate surfactant concentration (2x CMC or greater), partial or complete dewetting can occur.<sup>11</sup> Without the surfactant, the solvent/water system will swell the film and create pockets of detachment at the film-substrate interface, but dewetting will not occur. This is a simplified version of the role surfactants play in dewetting, and it is important to mention they perform other functions during this process.

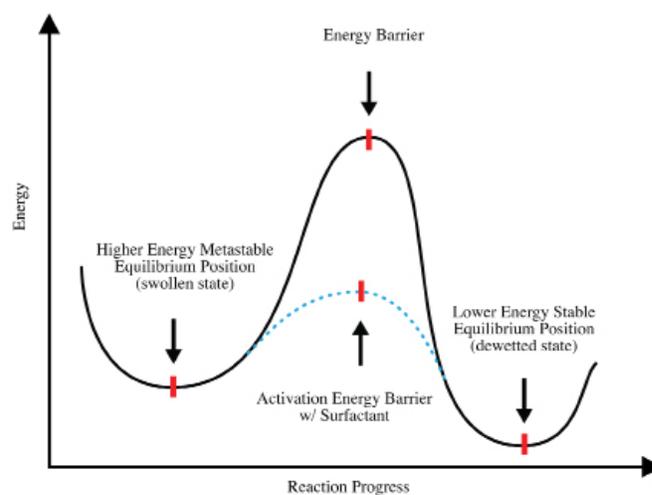


Figure 3. Higher energy metastable equilibrium position (swollen film) → oscillations (due to thermal energy, motion, vibration of polymer molecules) overcome activation energy barrier → lower energy stable equilibrium position (dewetted film) results. Illustration by Joe Brown.

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## Surfactants & Solvents: Potential for MCP - Designed Nanostructured Fluids for the Removal of Polymer Films, continued

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### Practical Applications

In the studio we have used a ternary water/MEK/nonionic surfactant system with 20% (w/v) MEK and Triton X-100 (5x CMC) to partially dewet a thick (>60 $\mu$ m) catalyzed nitrocellulose lacquer coating on a nineteenth-century marquetry table.<sup>12</sup>

The swollen micellar solution was uploaded into Evolon CR micro-filament textile and allowed to dwell on the surface, covered with polyethylene, for approximately one hour. We achieved partial dewetting and lifted the coating off the surface with a plastic business card.

Triton X-100 contains nine ethoxy groups that undergo a similar dehydration process as the surfactants tested by Baglioni. They exhibit a comparable drop in  $T_c$  due to the similar length of their POE chains. In fact, all nonionic ethoxylated and/or propoxylated surfactants should function in a similar manner in conjunction with water and a polar HBA organic solvent.

As a result the Brij, Ecosurf, Ethofat, Pluronic, Surfonic, and Triton surfactant families in the MCP should exhibit some degree of reduced  $T_c$ . In combination with strong HBA solvents a related increase in detergency, reduction in micelle size, and elongation of micelle shape can be reasonably expected.

Fortunately, this is an instance where practice supports theory. At the Arizona State Museum conservation laboratory we successfully tested a swollen micellar solution that swelled and detached a significantly cross-linked, non-original, shellac coating from the delicate paint layer of a basket.

We dispersed kaolin nanoparticles into an MCP-designed pH 8.5 triethanolamine/HCl, Ecosurf EH-6 (5x CMC), 20% (w/v) MEK, and 3% (w/v) benzyl alcohol solution to form an emulsion. It was covered with polyethylene and allowed to dwell on the surface for approximately 1.5 hours. We achieved partial dewetting which resulted in the coating swelling and detaching from the surface (Fig.4).

If you're wondering what the fuss about these systems is and why someone would go through all this trouble, consider that here we are swelling and detaching the film, not dissolving it. The normal associated issues of redeposition and absorption of soiling materials by the substrate (and the frankly sticky mess) are completely sidestepped.

More importantly, significantly cross-linked coatings that may not be possible to dissolve can be relatively safely removed through swelling and partial dewetting.

Finally, much less solvent is used compared to the neat solvent blends typical for this kind of treatment, resulting in safer and greener working conditions for the conservator and the environment.

### MCP Implications

So, how does this affect the MCP, its ability to add cosolvents to aqueous solutions, and its new microemulsion phase diagrams? How can we use the MCP more effectively in designing NSF's?

From a safety and phase separation standpoint, most surfactants in the MCP with a listed cloud point temperature have a 1% (w/v)  $T_c$  above 50°C, so most surfactants should still be safe to use in lab or studio conditions with a polar HBA solvent. Be aware that some viscosity modifiers do affect  $T_c$ . This is why relatively inert cellulose pulp poultices and chemical gels are often utilized in cleaning with NSF's.

Using the broad range nonionic POE surfactants Baglioni investigated as analogs, we can reasonably expect the drop of surfactant  $T_c$  by addition of 20% MEK to the aqueous solutions we develop using the MCP to be around 10°C, and cautiously 15°C.

For example, Surfonic JL-80X has a  $T_c$  of 59°C at 1% (w/v). A 10°C drop in cloud point temperature would result in a new  $T_c$  at 49°C. A drop of 15°C would result in a  $T_c$  at 44°C. Ecosurf EH-9 has a  $T_c$  of 61°C. A 10-15°C drop would result in new  $T_c$ s at 51°C and 46°C respectively. Thus both surfactants would be reasonable choices for certain outdoor applications.

However, if planning to use either on a bronze sculpture or at an archaeological site in the middle of summer you may want to reconsider. A biodegradable surfactant like N,N-Dimethyldodecan-1-amine oxide (DDAO) that does not exhibit a cloud point temperature when maintained at a neutral pH would be a safer choice.<sup>13</sup>



Figure 4.

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## Surfactants & Solvents: Potential for MCP - Designed Nanostructured Fluids for the Removal of Polymer Films, continued

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Ecosurf EH-6 has a  $T_c$  of 40°C at 1% (w/v). A solvent-induced  $T_c$  drop of 10-15°C would make it a poor choice for outdoor applications. However, it is a very interesting option for working close to  $T_c$  in climate-controlled lab or studio conditions. Addition of 20% (w/v) MEK would result in nearly room temperature  $T_c$ s at 30°C or 25°C. This makes it an excellent choice for removing soiling materials and polymer films from surfaces via increased detergency.

This is another instance where practice and theory align. A piece of pottery in the Arizona State Museum collection accumulated a layer of practically insoluble, chemically complex soot during a house fire. We designed and tested a relatively high pH aqueous solution in the MCP consisting of sodium bicarbonate buffer, EDTA & citric acid chelators, triethanolamine, and Surfonic JL-80X (10x CMC) 20% (w/v) propylene carbonate, and 2% (w/v) benzyl alcohol.

This solution had almost no effect on the soot. By substituting the Surfonic JL-80X with Ecosurf EH-6 (10x CMC), there was a dramatic shift in the cleaning effect in agreement with the predicted properties of a solution close to its cloud point temperature. (Fig. 5)



Figure 5.                      cleaning test                      soot layer

### Conclusion

Going forward it may be worthwhile to investigate new surfactants, considering hydrophilic-lipophilic balance (HLB), CMC, and  $T_c$  together to more finely tune the result we are trying to achieve. If the goal is film removal and not simply cleaning of soiling materials, more deliberate and informed choices of surfactant and solvent are required.

Practically, new formulations can be tested for  $T_c$  after addition of surfactant and solvent by gently heating on a magnetic stirrer with a thermometer and observing at what temperature clouding occurs. A formulation with optimal detergency at room temperature should not take much heating to reach  $T_c$ .

If using an MCP-designed NSF, regardless of surfactant or solvent choice, the conservator should at least consider:

- 1) if the solvent is a hydrogen bond acceptor that may interfere with the interaction between water and the polar head of the surfactant,
- 2) the surfactant's listed and predicted resultant  $T_c$ , and
- 3) how these factors could affect the solution's cleaning properties. While difficult to predict, at least being aware of the possible effects and knowing to watch for phase separation will go a long way.

### End Notes

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<sup>2</sup>Kralchevsky, P. A., et al. "Kinetics of Triglyceride Solubilization by Micellar Solutions of Nonionic Surfactant and Triblock Copolymer. 1. Empty and Swollen Micelles." 2002.

<sup>3</sup>Kralchevsky, P. A., et al. "Kinetics of Triglyceride Solubilization by Micellar Solutions of Nonionic Surfactant and Triblock Copolymer. 2. Theoretical Model." 2002.

<sup>4</sup>Baglioni, Piero and David Chelazzi. *Nanoscience for the Conservation of Works of Art*. 2013. pp. 189-190.

<sup>5</sup>Baglioni, Piero, et al. "Nanostructured Fluids from Degradable Nonionic Surfactants for the Cleaning of Works of Art from Polymer Contaminants." 2014.

<sup>6</sup>Stavroudis, Chris and Soraya Alcalá. "Nano-Lime, Emulsions, Gels, and Nanostructured Materials." 2018.

<sup>7</sup>Kralchevsky, P. A., et al. "Kinetics of Triglyceride Solubilization. 2. Theoretical Model." 2002. P. 7888.

<sup>8</sup>Baglioni, Piero, et al. "Surfactants Mediate the Dewetting of Acrylic Polymer Films Commonly Applied to Works of Art." 2019.

<sup>9</sup>Baglioni, Piero, et al. "The Use of Surfactants in the Cleaning of Works of Art." 2020.

<sup>10</sup>Ibid, p. 115.

<sup>11</sup>Baglioni, Piero, et al. "Dewetting Acrylic Polymer Films with Water/Propylene Carbonate/Surfactant Mixtures – Implications for Cultural Heritage Conservation." 2017.

<sup>12</sup>Baglioni, Piero, et al. "Polymer Films Removed from Solid Surfaces by Nanostructured Fluids: Microscopic Mechanism and Implications for the Conservation of Cultural Heritage." 2015.

<sup>13</sup>Baglioni, Piero, et al. “An Amine-Oxide Surfactant-Based Microemulsion for the Cleaning of Works of Art.” 2015.

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“**Light Relief: Could New Lighting Technology Avert the Need for Restoration?**,” *The Art Newspaper*, 01/31/2020

Lighting affects our perception of a work of art. Just ask Robert van Langh, the head of conservation and science at the Rijksmuseum, where the Netherlands’s most famous painting, Rembrandt’s *The Night Watch*, is undergoing the most ambitious conservation effort in its 378-year history.

A chance observation made when the 1642 painting was temporarily relocated to a side gallery alerted the museum staff to details that had been previously thought lost, such as the architectural background.

Changes in technology over the past decade have transformed art lighting from a presentational aid to a tuneable precision tool that can function as a non-invasive means of limiting the need for traditional restoration techniques. The steady replacement of traditional halogen lamps with energy-efficient LEDs has resulted in significant savings for museums and galleries, while reduced heat emissions and little to no ultraviolet or infrared radiation have lowered (though not eliminated) the risks posed to works of art from light sources, thereby granting greater freedom to conservators and curators.

The treatment plan for *The Night Watch* has yet to be determined, Van Langh emphasises, and while he was not prepared to say whether lighting might offer a substitute for other conservation steps, he makes it clear that non-invasive interventions are always preferable.

Van Langh insists that observations must be substantiated by science, with one likely source of data a map of the painting’s chemical constituents currently being compiled through macro X-ray fluorescence (XRF) scans. For now, Van Langh is focused on establishing “which wavelengths of light to use so that we see as much as possible of *The Night Watch*”.

“**‘The Scream’ is Fading. New Research Reveals Why.**” *The New York Times*, 02/07/2020

“The Scream” is fading. Tiny samples of paint from the 1910 version of Edvard Munch’s famous image of angst have been analysed as scientists have used cutting-edge technology to try to figure out why portions of the canvas that were a brilliant orangeish-yellow are now an ivory white.

The research also provides insight into Munch and how he worked, laying out a map for conservators to prevent further change, and helping viewers and art historians understand how one of the world’s most widely recognized paintings might have originally looked.

Jennifer Mass, the president of the Scientific Analysis of Fine Art lab in Harlem, whose team is on “The Scream” research, explained the science. Nanocrystals are growing on the painting — stark evidence of the degradation near the central figure’s mouth, in the sky and in the water. Dr. Mass’s team was able to narrow down Munch’s paint choices using his paint tubes, some 1,400 of which are held by the Munch Museum.

Over time, with exposure, the yellow cadmium sulfide has oxidized into two white chemical compounds, cadmium sulfate and cadmium carbonate. The analysis, Dr. Mass said, has implications for Impressionist through Expressionist paintings made between the 1880s and the 1920s painted with cadmium yellow, 20 percent of which she estimates are experiencing