



Article: 19th Century Photography in a Modern Chemistry Lab

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19th Century Photography in a Modern Chemistry Lab

Corina E. Rogge and Anikó Bezur

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Abstract: College courses focusing on the interface of chemistry and art are an increasingly popular way to introduce non-majors to scientific subjects, but few courses seek to engage science majors in the art world. We recently developed an upper-level laboratory module for chemistry and chemical engineering majors that introduced students to the chemistry and conservation science of 19th century photographic processes. Students learned to visually identify 19th century photographs; made cyanotypes, van Dyke brown prints, gum bichromate prints, and salted paper prints (using negative/positive processes or cliché-verre), and were introduced to sepia, selenium, and gold toning methods. After learning the importance of non-destructive analytical techniques, the students used attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) to identify organic binders on study collection prints and x-ray fluorescence spectroscopy (XRF) to identify toning or stabilizing materials on salted paper prints and van Dyke brown prints. Students also examined the size and constituents of the image particles using scanning electron microscopy coupled with energy dispersive x-ray spectroscopy (SEM-EDX). The course was well received, because in addition to providing a foundation in new instrumental techniques it also provided students with an introduction to wet-process photography and to the museum and conservation worlds.

Introduction:

Interdisciplinary science courses that seek to attract and engage humanities and art students have become increasingly popular in undergraduate education. Many academic institutions have begun offering courses, mainly through their chemistry departments, that focus on the synthesis of artists' materials and/or the analysis of cultural heritage objects. Dr. Patricia Hill from Millersville University has been a particularly strong proponent of this approach and offers NSF-funded workshops each year for educators interested in integrating art into their science courses (<http://www.millersville.edu/chemistry/cheminart.php>). These 'science of art' classes are usually introductory courses aimed at broadening the education of non-science majors, and few upper-level undergraduate science courses employ a focus on conservation science. To show that this multi-disciplinary approach provides an exciting context for teaching advanced scientific concepts and experimental practices, we developed an upper level laboratory module on conservation science for chemistry and chemical engineering majors.

Collaborations between the MFAH and Rice University, including an introductory course on the chemistry of art, led to a 2007 Andrew W. Mellon Foundation grant to the Museum of Fine Arts Houston (MFAH), The Menil Collection, and Rice University that funded a conservation science initiative, including the hiring of Dr. Anikó Bezur as research scientist to the museums. Dr. Bezur and Dr. Rogge, then the Wiess Lecturer in Chemistry at Rice, sought to develop a course that would meet the requirements for a 300 level (junior undergraduate) Rice Chemistry laboratory module. Such courses should introduce students to new instrumental techniques,

integrate previously learned chemical concepts, and explore new ones, in a ‘capstone’ manner. This course would also engage students in the analysis of museum study collection objects and the reproduction of historic techniques in order to fully understand the materials with which they were working. The chemical complexity of photographs as objects and the important role that scientists played in their development made their study and replication appropriate subject matters. The ready availability of original texts and detailed references that describe the photographic methods simplified the replication process.

The course focused on 19th century photographic processes as the resulting products are prone to degradation reactions and thus their identification is of great importance to conservators. The laboratory module’s schedule – three-hour sessions once a week for eleven weeks – imposed some restrictions on the processes the students would be able to reproduce. Single layer binderless images such as cyanotypes, salted paper prints and van Dyke brown prints were amenable to the time restrictions and, with the inclusion of gum bichromate prints, presented a sufficiently varied selection in terms of chemistry and analytical challenges. Media that were not reproduced in the laboratory such as gelatin, collodion and albumen prints were represented in the study collection materials the students analyzed.

Students learned to utilize both non-destructive and micro-destructive analytical methods that would actually be used in museum conservation science labs for the characterization of photographs. Binders were identified by micro-destructive microchemical tests and non-destructive attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR), a technique often overlooked in chemistry labs in favor of transmission FTIR, but commonly used in museums. X-ray fluorescence spectrometry, another non-destructive analytical technique, was used to determine the image and toning materials in photographs. Although increasingly common in cultural heritage institutions, this is a relatively rare method in academic research labs, and the lab module thus presented students with a novel opportunity to learn how to use this technique. Scanning electron microscopy coupled with energy dispersive x-ray analysis (SEM-EDX) was used for the visualization of the paper support and imaging particles and the elemental analysis of individual particles.

Course Outline:

Week 1 (no assigned reading)

Introductions: The instructors introduced themselves and explained the format of the course. It met three hours a week for 11 weeks and was worth 1 credit hour. Prerequisites included general chemistry and organic chemistry, while inorganic chemistry was strongly recommended. Lectures on analytical techniques and photographic methods would be interspersed as appropriate and individual laboratory sessions would focus on analytical techniques, creation of photographs or both. Readings from appropriate texts would be provided as resources, as would a series of handouts describing the chemistry behind each photograph process.

Construction of darkroom boxes: The laboratory took place in a chemistry department that did not have darkroom facilities. In order to prevent fogging of light-sensitive papers each student constructed a darkroom box based on a design suggested by Mark Osterman from the George

Eastman House. A large cardboard box had a window cut out on one side leaving a 2-inch lip (Fig. 1). The bottom of the box was lined with duct tape to make it easier to clean. To provide working light the top of the box had a small window cut into it and a 5x7 inch amber safelight filter taped over it. A length of black polyester fleece was used for the dark cloth.



Fig. 1. A simple cardboard darkroom box.

Introductory lecture: This lecture introduced the students to the history of photography, beginning with Johann Heinrich Schulze's discovery that silver chloride and silver nitrate salts darken upon exposure to light and covering the Daguerreotype, salted paper, calotype, ambrotype, and tintype processes. The differences between printing out and developing out processes were also discussed.

Week 2 (assigned reading: chapters 1-4 of Reilly (1986), handout on history and chemistry of photographic processes)

Visit to the MFAH photograph conservation laboratory: Mr. Toshiaki Koseki, the Carol Crow Conservator of Photographs at the MFAH, provided an introduction to the use of a stereomicroscope for identification of photographic processes. He provided a series of 'known' photographs from his study collection and taught the students how to apply the identification flow chart in *Care and Identification of 19th Century Photographic Prints* (Fig. 2). The students were then challenged to identify 'unknown' photographs using this method. Surprisingly, the students were quite anxious about this task as it was far outside the realm of their normal scientific experiences, but quickly realized that they could bring their analytical skills to bear and were able to identify many photograph types by the end of the class period.



Fig. 2. Toshiaki Koseki helping students to identify their unknown prints. Photograph courtesy of Karen Willis.

Week 3 (assigned reading: an article on the use of XRF for the analysis of cultural heritage objects (Deming Glinsman 2005))

Introduction to XRF and its application to photograph identification: As none of the students had previously encountered XRF spectrometry, an introductory lecture was given on the theory behind this technique. This was followed by a practical introduction to the Bruker Tracer III-V instrument. The students then used this method to characterize their unknown photographs from week 2 in order to determine if their visual identifications were correct.

Week 4 (assigned reading: article on the chemistry of platinum and palladium prints (Gottlieb 1995), and handouts on cyanotypes and van Dyke brown processes)

Lecture on iron-based photographic processes: This lecture introduced the students to the chemistry behind cyanotype, van Dyke brown, and platinum/palladium processes.

Assembly of contact printing frames: Commercial contact printing frames are prohibitively expensive so we constructed simple ones from wooden frames, latex tubing, Volara and foam-core (Barnier 2000; Rogge 2012).

Cyanotypes: The students made cyanotypes using the traditional iron(III) citrate and potassium ferricyanide recipe (Ware 1996-2004a) and compared them to those created by Dr. Mike Ware's modification of the method that uses iron(III) oxalate and potassium ferricyanide (Ware 1996-2004b). They determined proper exposure time by performing trial exposures with transmission step wedges. They were also introduced to toning processes and used trisodium phosphate or tea to create yellow or brown prints, respectively (Fig. 3).

This provided the first opportunity for the students to witness photochemistry in action. It is well known that exposure to light causes citrate to break down, forming carbon dioxide and acetone: $C_5H_5O_7^{2-} + \text{light} \rightarrow 3CO_2(g) + C_3OH_6(l) + H^+ + e^-$. The electrons produced reduce potassium ferricyanide to ferrocyanide, which in the presence of excess iron(III) forms ferric ferrocyanide, or Prussian blue. The blue color derives from intervalence electron transfer between the two different oxidation states of iron, Fe^{2+} and Fe^{3+} , linked by cyanide bridges. However, exposure to light can also cause the temporary photobleaching of Prussian blue to Prussian white, ferrous ferrocyanide. Therefore the initial print density appears to be weak, and only upon reoxidation does the true print density become apparent. These concomitant processes provided an apt illustration of importance of doing test exposures using transmission step wedges in order to optimize the appearance of the final image.

The toning experiments also raised the concept of preventive conservation and the importance of housing materials. Exposure of cyanotypes to weak bases causes the formation of hydrated iron oxides, which are yellow. Some archival storage materials are buffered and contain a base reserve that can cause the same reaction and upon exposing their cyanotypes to base the students immediately understood why buffered materials should not be used for cyanotype housings.



Fig. 3. From left to right: a traditional cyanotype, a Ware method cyanotype and a tea toned traditional cyanotype.

Paper sizing: To investigate the role paper sizing plays in tonality of salted paper prints each student sized a series of papers with arrowroot starch, gelatin, acrylic matte medium or gum Arabic with potassium dichromate.

Week 5 (assigned reading: handout on salted paper prints)

Halide-stabilized and thiosulfate-fixed salted paper prints: The students created halide-stabilized and thiosulfate-fixed salted paper prints using their sized papers. Talbot initially formed light sensitive papers by washing paper in a solution of sodium chloride and then brushing a solution of silver nitrate onto the surface (Schaaf 1996). We chose to use a slightly later adaptation invented by Alfred S. Taylor (and eventually adopted by Talbot), where the ‘ammonio-nitrate of silver’ (diamine silver(I), $[\text{Ag}(\text{NH}_3)_2]^+$) is used instead (Ware 1994). This species is a complex ion formed by titration of a silver nitrate solution with ammonium hydroxide. As ammonium hydroxide is added an initial precipitate of neutral Ag_2O forms but as more ammonia is added the equilibrium shifts to favor the formation of the diamine complex. Because this complex ion is charged it is soluble in water and the precipitate re-dissolves. This provided a graphic example of relatively simple chemistry taught in general chemistry classes but applied in a new and novel setting. Another example of the solubility of charged complex ions occurs during the ‘fixing’ of prints with sodium thiosulfate: $2 \text{Na}_2\text{S}_2\text{O}_3^{2-} (\text{aq}) + \text{AgCl} (\text{s}) \rightarrow [\text{Ag}(\text{S}_2\text{O}_3)_2]^{3-} (\text{aq}) + 2 \text{NaCl} (\text{aq})$.

Salted paper prints also provided an ideal format to discuss the importance of equilibrium constants in photography. It is rather surprising to most people that formation of silver halides from metallic silver and halogen gas is strongly thermodynamically favored. Therefore, in the absence of scavenging reactions that remove the halogen formed during exposure of the sensitized paper to light no image would be created. Luckily for photographers, water is capable of reacting with halogens via disproportionation to a hypohalous acid and halide ion: $\text{X}_2 (\text{g}) + \text{H}_2\text{O} (\text{l}) \rightarrow \text{X}^- (\text{aq}) + \text{HOX} (\text{aq}) + \text{H}^+ (\text{aq})$, where X is a halogen (Ware 1994). The equilibrium constant K for this reaction is 500, 0.01 and 10^{-9} for Cl_2 , Br_2 and I_2 (respectively), suggesting that in the absence of other reactions recombination of iodine with Ag to reform AgI is favored. The fact that this reaction is not seen by the students leads to a discussion of what this implies, namely that another reaction removes the HOI or HOBr formed and thus pulls the reaction forward; the reaction is: $3 \text{HOX} (\text{aq}) \rightarrow 2 \text{X}^- (\text{aq}) + \text{XO}^{3-} (\text{aq}) + 3 \text{H}^+ (\text{aq})$.

The production and scavenging of halogens is necessary for image formation, but continued exposure to light after the image has been formed can cause overexposure and loss of image quality. Talbot initially halted exposure by soaking his exposed prints in concentrated solutions of halides. This causes the silver halide crystals to sorb excess halide ions onto their surface. The negatively charged halide ions repel photoelectrons produced upon exposure, preventing reduction of Ag^+ and formation of metallic silver. Halogens can also be scavenged by excess Ag^+ : $\text{X}_2 (\text{g}) + \text{H}_2\text{O} (\text{l}) + \text{Ag}^+ (\text{s}) \rightarrow \text{AgX} (\text{s}) + \text{HOX} (\text{aq}) + \text{H}^+ (\text{aq})$ but in the presence of excess halide the reverse of this reaction is favored and a more photostable material results. However, these images are still very vulnerable and can visibly deteriorate over a very short period of time, particularly if the concentration of halide is incorrect. Too little stabilizing halide results in an image that will continue to darken whereas too much iodide can result in loss of the image via the reaction of silver with iodide and oxygen: $4\text{Ag} (\text{s}) + 4\text{I}^- (\text{s}) + \text{O}_2 (\text{g}) + 4 \text{H}^+ (\text{aq}) \rightarrow 4 \text{AgI} (\text{s})$

+ 2 H₂O (l). These reactions were observed in some student-made prints, providing a dramatic analog of the deterioration seen in a Talbot print by Rheingold (1993).

Week 6 (no assigned reading)

XRF analysis of salted paper prints: In addition to being able to detect the presence of halide stabilizers as shown in (Rogge 2012), the stabilized prints can also be distinguished from thiosulfate fixed prints due to equal levels of silver being present in the light and dark areas of the halide-stabilized prints (Fig.4).

Week 7 (assigned reading: handout on toning processes)

Toning of thiosulfate-fixed salted paper prints: The students toned their prints using Kodak Professional Sepia II Warm Toner, Kodak Rapid Selenium Toner, Clerc's thiourea gold toner or gold borax toner (James 2008).

Toning provided an excellent opportunity to discuss common degradation reactions, the importance of the electrochemical series, and relative reduction potentials. Toning may be an aesthetic choice by the artists, but also has a decided impact upon the stability of the final print. Silver is a reactive material and is prone to the formation of silver sulfide, Ag₂S, in polluted environments or when the print has been insufficiently washed and thiosulfate remains in the support. Silver sulfide has a lower extinction coefficient than pure silver (Ware 1994) and therefore 'sulfided' prints have reduced image density. However, silver sulfide itself is a very inert material and this stability can be exploited by sepia toning the print, where the silver image material is deliberately converted into silver sulfide. The Kodak Professional Sepia II warm toner used by the students has interesting chemistry where silver is first reduced by potassium ferricyanide (previously encountered in cyanotypes), then converted into silver bromide, and finally reacted with sodium sulfide:

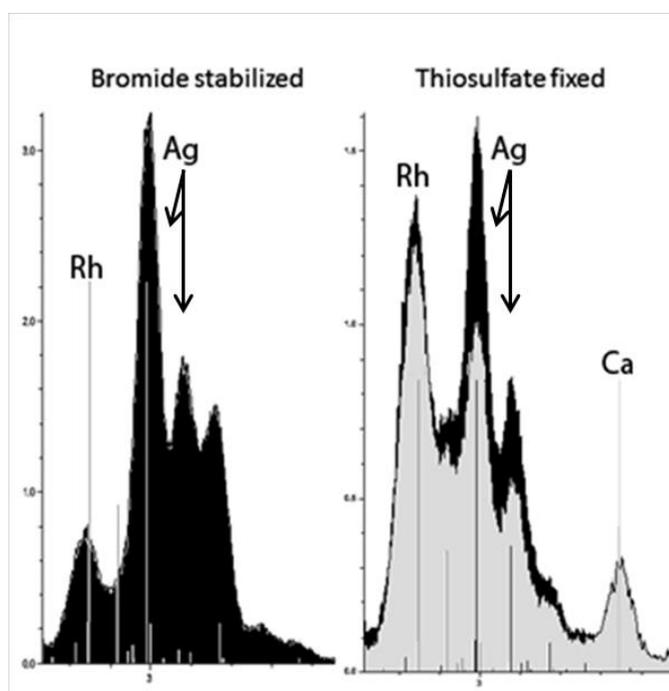
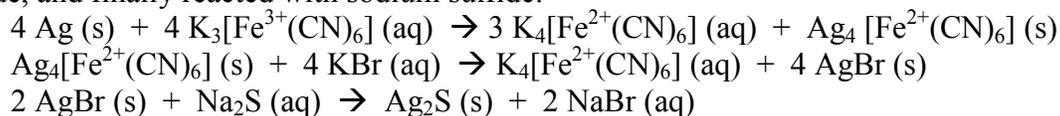


Fig. 4. Comparison of the intensity of the silver peaks in the light (grey) and dark (black) areas of bromide stabilized and thiosulfate fixed prints. The light and dark areas of the halide stabilized prints cannot be distinguished by XRF, whereas there is clearly less silver in the light areas of thiosulfate fixed prints.

In gold toning, silver image particles are exposed to a solution containing gold(III) ions. Because of the difference in reduction potentials, an electrochemical replacement occurs and some of the silver atoms in the image particles are replaced by gold: $3 \text{ Ag (s)} + [\text{AuCl}_4]^- \rightarrow \text{Au (s)} + 3 \text{ AgCl(s)}$. Because gold has a more positive reduction potential than silver, it is less prone to oxidation reactions and a more stable image results.

XRF analysis of toning materials: As described by Rogge and Bezur (2012), XRF can be used to determine if a print has been toned with selenium or gold. The issue of sulfur (sepia) toning is more complex because the detection of sulfur by XRF could also indicate incomplete removal of the thiosulfate fixer or subsequent exposure to sulfur-containing pollutants and undesired formation of silver sulfide.

Week 8 (assigned reading: Chapter 5 from Derrick, Stulik, and Landry (2000), relevant microchemical tests from Odegaard, Carroll and Zimmt (2005) and a handout on paper sizing)

Identification of binders and paper sizes by ATR FTIR: The students learned how to use the ATR interface of a bench-top FTIR instrument for non-destructive analysis of photographs. Albumen, silver-gelatin and salted paper prints (binderless) were used as examples. The students then analyzed the papers they had sized using the references provided.

Identification of binders and paper sizes by microchemical tests: Utilizing the tests outlined in Odegaard, Carroll and Zimmt (2005) as a reference the students analyzed the papers they had sized as well as the ‘unknown’ photographs from week 2.

Week 9 (no assigned reading)

van Dyke brown prints: Coming back to iron-based photography the students made van Dyke brown prints and toned them using the same techniques they had learned for salted paper prints (Fig. 5).

Cliché-verre: The students smoked a glass plate over a candle flame, lightly sprayed it with Krylon workable fixative, and



Fig. 5. Gold borax toned van Dyke brown print made from an antique glass plate negative



Fig. 6. Cliché-verre plate (left) and van Dyke brown print made from the plate (right).

then drew on the plate (Fig. 6). Students printed their plates and toned the resulting image with a method of their choosing.

Week 10 (no assigned reading)

Gum-bichromate prints: The students made single color gum bichromate prints using a variety of inorganic pigments from Kremer Pigmente mixed with gum Arabic and potassium dichromate. To determine proper exposure times, they performed test exposures for each pigment mixture (using transmission step wedges) because different pigments accelerate or retard the crosslinking reaction.

This was the only two layer print the students made, and it also introduced the concepts of oxidative crosslinking, metal crosslinking, and the dependence of polymer solubility on molecular weight.

Week 11 (no assigned reading)

Analysis of photographs by SEM-EDX: Sections of cyanotypes (both traditional and Ware method) and thiosulfate-fixed salted paper prints were taken from dark areas and carbon coated. Image particles were located and their sizes measured. This provided graphic evidence for the nanoscale size of the particles and the fibrous nature of the paper.

Conclusions:

The instructors felt that this course successfully introduced the applicability of science to the study of photographs and the museum world while drawing on previous chemical knowledge. The students learned several new techniques that they would not otherwise have been exposed to, and a great deal about photography, a subject that none of them had considered in terms of chemistry before taking the course. The students appreciated the history behind the subject, and the critical role played by scientists in the creation of this type of art gave them a sense of pride and resulted in more investment in the lab. Because the students produced their own images, there was a surprisingly strong drive not only towards understanding and experimentation but also to create more perfect prints. As none of the students had visual arts backgrounds or had done any photography other than simple digital ‘snaps’, this was unanticipated, and sometimes required the instructors to limit the number of prints created in the interest of time. Student morale was high throughout and one of the students later said that this laboratory module had been his favorite class at Rice.

Based upon these experiences, we feel that the integration of conservation science into chemistry and materials sciences programs should not be limited to the introductory level classes, and that science majors can benefit from the opportunity to learn about more complicated materials and techniques in a more advanced course. 19th century photographic methods proved a convenient topic for these purposes given local museum expertise and instrumentation, and the interest in nanoscale chemistry at Rice, but we expect that other conservation science topics could also be productively explored in advanced laboratory modules. We encourage our colleagues to explore

these options and to this end will be happy to make any of our course materials available upon request (email C. Rogge: crogge@mfah.org).

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