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Tip: A Preliminary Investigation Into the Use of Diethylenetriaminepentaacetic Acid and Ethylenediaminetetraacetic Acid to Treat Iron Induced Foxing in Paper Objects

INTRODUCTION

The purpose of this research was to understand the extent to which diethylenetriaminepentaacetic acid (DTPA) and ethylenediaminetetraacetic acid (EDTA) can reduce the iron content in iron-induced stains on paper. The tests were developed based on common treatment methods used to apply chelating agents to stains. The tests were more aggressive than what would be exercised in conservation practice in order to gain a preliminary understanding of the risks and benefits of chelation treatments.

Iron contaminants may be incorporated into paper from metal machinery and unfiltered water sources during manufacture, water damage, pollutants and dust, or ancillary materials in contact with the paper object (Ardelean and Melniciuc-Puică 2013; Choi 2007; Daniels 1996). Localized staining may be a result of iron contaminants and is sometimes called foxing.¹ As a transition metal, iron is highly reactive existing in the form of colored iron(III) corrosion salts or colorless but highly reactive iron(II) salts which catalyze oxidative cellulose degradation (Bicchieri and Pepa 1996; Daniels 2002; Giorgi 2013; Kolar 2001; Neevel 1995; Sheldon and Kochi 1981).

CHELATING AGENTS IN PAPER CONSERVATION

Conservators may address localized staining with routine stain reduction methods such as washing, alkalization, or bleaching. Washing and alkalization are appropriate for removing soluble degradation, however, iron salts are very difficult to solubilize, particularly the colored iron(III) salts. Bleaching addresses the effects of staining by changing the colored molecules into non-colored molecules rather than the cause of the staining. Also, reversion of staining has been recorded in objects after bleaching treatments, especially in the case of iron induced foxing (Gallo and Hey 1988; Owen 1994).

Alternatively, chelating agents have been used in paper conservation to improve stain reduction treatments, allowing metallic components of stains to be solubilized and removed from the paper. The properties of iron as a transition metal allow the heavy metal to react with chelating agents forming coordination bonds (Cramer, 1973). Chelating agents are a class of organic molecules that can form coordination bonds with heavy metal ions under the following two criteria: (a) the chelate must have two or more functional groups that can donate a pair of electrons to a positively charged metal ion forming a coordination bond, and (b) the stereochemistry of the chelate allows formation of rings during complexing where the metal ion acts as the closing member of the rings at the center of the complex (Burgess 1991; Dwyer and Mellor 1964). Successful chelation of metal ions will occur when the chelate formation ($\log K_f$) constant is higher than the solubility product (pK_{sp}) of the targeted metal salt (Rivers and Umney 2003; Wolbers 2000).^{2,3,4,5} Some chelates effectively used for stain reduction on works of art on paper include iron(II) chelators like ammonium citrates, DTPA, and EDTA; or iron(III) chelators like di(ortho-hydroxybenzyl)-edthylenediamine diacetic acid (HBED). A survey of conservation literature shows the use of a reducing agent such as sodium dithionite (SDT) or sodium metabisulfite⁶ to reduce iron(III) into iron(II), an essential step for enhancing chelation treatment outcomes when using iron(II) specific chelates. Treatment methods include immersion, local application on the suction table with a brush or spray, or topical application with hydrogels (Baker 1987; Blank and Dobrusina 1984; Burgess 1991; Gent and Rees 1994; Hashimoto 2015; Irwin 2011; Owen 1994; Selwyn and Tse 2008; Sullivan et al. 2014; Suryawanshi and Bisaria 2005).

Some empirical studies have analyzed chelation treatment of paper staining, testing parameters that would only be appropriate in a laboratory setting. Baker (1987) found that EDTA and DTPA sodium salts had very little effect on stain reduction and observed reversion of treated staining after three years of natural aging. Blank and Dobrusina's (1984)

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test showed that the DTPA calcium salt improved the paper stability of the samples tested. Results from Suryawanshi and Bisaria (2005) revealed high concentrations of EDTA with unadjusted pH levels had adverse effects on paper strength after artificial aging. Immersing samples in alkaline EDTA and 2% weight per volume (w/v) solution of SDT effectively reduced staining and had the least damaging effect to the paper support after aging. Hashimoto (2015) found that overall discoloration was reduced with triammonium citrate immersion baths, however, samples that were not alkalinized after chelation treatment increased in discoloration after aging. While these studies have found chelating agents to successfully reduce staining, they do not express effectiveness of treatment in terms of the amount of iron ions removed from the paper support, nor do they compare treatment methods commonly used for chelation treatments.

EXPERIMENTAL

The treatment methods and variables tested were chosen to reflect what has been commonly used in published case studies and analysis of chelation treatments in paper conservation. The following variables were tested to understand what impact they have on chelation treatment effectiveness: chelating agents (DTPA and EDTA), use of a reducing agent (SDT), different treatment application (immersion and local application on the suction table), and different solution pH levels (pH 9 and pH 10).

SAMPLE PREPARATION

Deionized water was sprayed onto Whatman No.1 ashless filter papers until they were evenly dampened in a plastic photographic tray. Iron filings and Saxa table salt were sprinkled onto the filter papers. The tray was covered with a sheet of acrylic and kept in an uncontrolled environment with elevated temperatures for a week. The papers were thoroughly foxed with no signs of mold bloom visible. Twelve samples and one control were cut from the foxed papers in 35mm diameter circles for the Spectro X-Lab 2000 x-ray fluorescence (XRF) small sample die.

TECHNICAL ANALYSIS

Samples were analyzed before treatment, treated with chelation treatments, dried, analyzed during treatment, immersion washed in 20ml of reverse osmosis (RO) water, dried, and analyzed after treatment. Samples were examined and photographed in visible and ultraviolet (UV) light. Details of photographic equipment used may be found in the Appendix. The $L^*a^*b^*$ values of stains in the samples before and after treatment were taken with a CM-2600d spectrophotometer using the small aperture view and SpectraMagic TM NX software. Quantitative XRF readings were taken of the samples using a Spectro X-Lab 2000 with a 400 W Pd end window

x-ray tube High resolution Si(Li) semiconductor detector. Scanning electron microscopy (SEM) was conducted on select samples after treatment using an FEI Quanta 200 SEM with INCAEnergy Software.

SAMPLE TREATMENT

There were three treatment sets with four samples tested per set. The free acid of the chelates were dissolved in 20ml of diluted 20% ammonium hydroxide at pH 11.5 until the desired pH for the chelate solution was achieved. 0.4g of SDT was added to 20ml of RO water to make the 2% w/v solutions of SDT. Below is a breakdown of the samples and corresponding procedures tested:

Set 1. Immersion in 20ml of chelating solutions for six hours.

- sample 1—20ml of DTPA at pH9
- sample 2—20ml of DTPA at pH10
- sample 3—20ml of EDTA at pH9
- sample 4—20ml of EDTA at pH10

Set 2. Immersion in 20ml of 2% w/v SDT reducing agent for two hours followed by immersion in 20ml of chelating solutions for six hours.

- sample 5—20ml of DTPA at pH9
- sample 6—20ml of DTPA at pH10
- sample 7—20ml of EDTA at pH9
- sample 8—20ml of EDTA at pH10

Set 3. Local application with a brush on the suction table of 20ml of 2% w/v SDT followed by local application of 20ml of chelating solutions.

- sample 9—20ml of DTPA at pH9
- sample 10—20ml of DTPA at pH10
- sample 11—20ml of EDTA at pH9
- sample 12—20ml of EDTA at pH10

RESULTS

NORMAL LIGHT

For samples immersed in SDT and chelate solutions (samples 5–8), noticeable changes in staining plateaued after forty-five minutes to an hour of treatment. Visible staining was significantly reduced. Samples treated locally on the suction table experienced lateral movement of the staining; no visible reduction in staining was observed. Dark gray tidelines surround the treated areas. No visual change was apparent in samples treated by immersion in chelating solutions. Distinction between the types of chelate used or the different solution pH levels could not be made visually. Immersion treated samples appeared slightly swollen and samples treated locally had displaced paper fibers in the treated areas. Table 1 shows all samples in normal light before and after treatment.

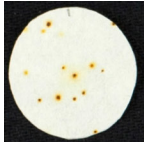
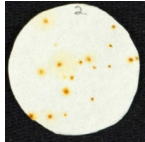
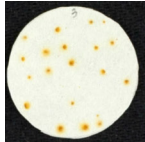
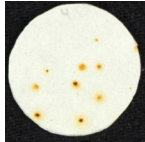
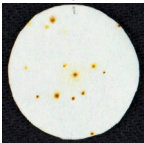
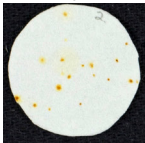


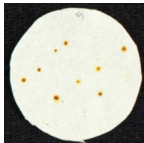
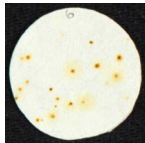
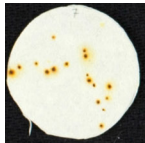
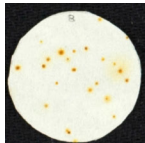






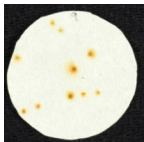
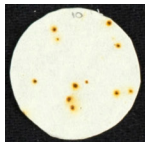
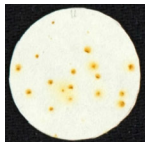
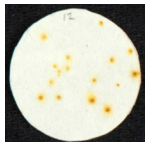




		DTPA pH 9	DTPA pH 10	EDTA pH 9	EDTA pH 10	
6 hour immersion in 20ml of chelate solution	Before Treatment					
	After Treatment					
2 hour immersion in 20ml 2% w/v SDT followed by 6 hour immersion in 20ml of chelate solution	Before Treatment					
	After Treatment					
2 hour immersion in 20ml 2% w/v SDT followed by 6 hour immersion in 20ml of chelate solution	Before Treatment					
	After Treatment					

Table 1. Images of all sample rectos in normal light before and after treatment.

ULTRAVIOLET FLUORESCENCE

Table 2 displays all samples in UV light before and after treatment. Samples 8 to 11 immersed in SDT then chelate solutions showed little UV absorbance after treatment. All samples treated locally had fluorescent tidelines. Samples treated solely by immersion in chelate solutions show no change under UV light after treatment. No distinctions between solution pHs was possible through UV fluorescence. Finally, the support of all samples treated with EDTA had an overall pale yellow fluorescence (fig. 1)

SPECTROPHOTOMETRY

Spectrophotometer readings were taken of one stain in each sample using a template for consistency. Three readings of the

same stain were measured before treatment and again after treatment. Chart 1 shows the change in a* readings (Δa^*) recording the shift in the measured stains from red to green. A strong shift from red to green occurred in samples treated by immersion in SDT prior to chelating solutions (samples 5 to 8) correlating with the decrease in visible staining formerly mentioned.

X-RAY FLUORESCENCE

A general decrease in iron content of all samples treated with chelating agents was seen with XRF analysis regardless of any other variables tested (see chart 2). The most significant drop in iron concentrations was found in samples treated by immersion in sodium dithionite followed by immersion in

		DTPA pH 9	DTPA pH 10	EDTA pH 9	EDTA pH 10		
6 hour immersion in 20ml of chelate solution	Before Treatment						
	After Treatment						
2 hour immersion in 20ml 2% w/v SDT followed by 6 hour immersion in 20ml of chelate solution	Before Treatment						Control
	After Treatment						
2 hour immersion in 20ml 2% w/v SDT followed by 6 hour immersion in 20ml of chelate solution	Before Treatment						
	After Treatment						

Table 2. Images of all sample rectos in ultraviolet light before and after treatment.

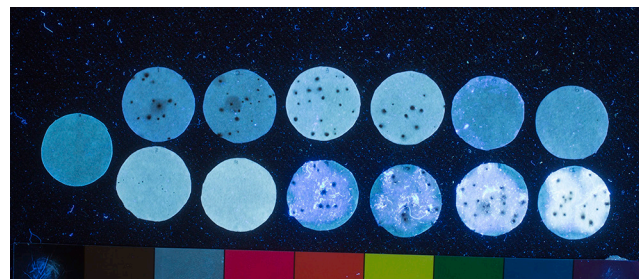


Fig. 1. Ultraviolet fluorescence of all samples after treatment, arranged in numerical order from left to right, top to bottom. All samples treated with EDTA chelate solutions show a pale yellow fluorescence after treatment.

chelating solutions. Solutions at pH 9 were generally more effective in reducing iron content than pH 10 solutions. Interestingly, samples treated with EDTA showed the greatest reduction in iron content compared to samples treated with DTPA. Refer to chart 2 for XRF results.

SCANNING ELECTRON MICROSCOPY

The control and samples 2, 4, 6, 8, 10 and 12 were selected for analysis with SEM after treatment to assess physical changes in the paper fibers and iron deposits in stained areas. Readings were taken at three levels of magnification, approximately 100xM, 500xM, and 2500xM. Images were taken of areas of paper fibers at 100xM and 500xM followed by at least two different locations of iron deposits at 500xM and 2500xM.

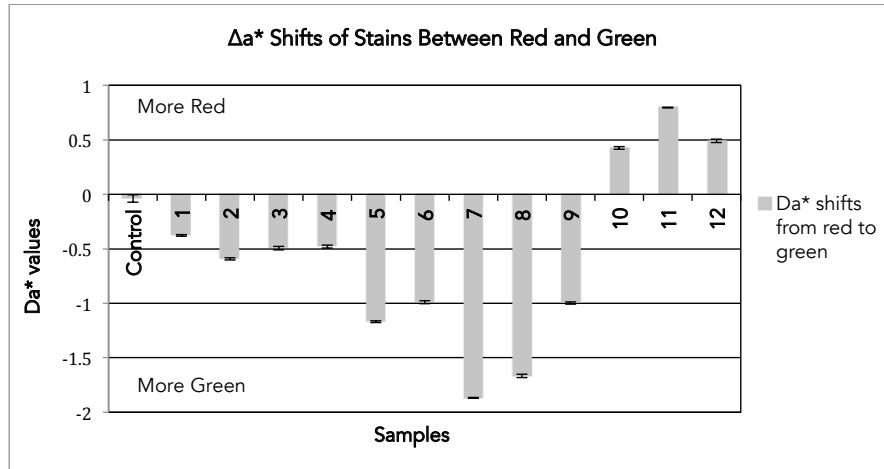


Chart 1. The Δa* values showing shifts from green to red.

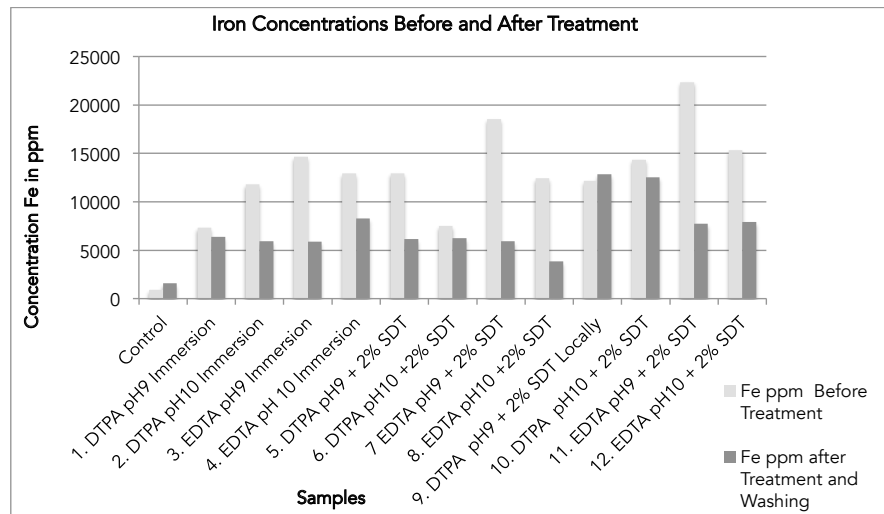


Chart 2. The iron concentration in parts per million (ppm) of all samples before and after treatment. The XRF was operated Gordon Forest by Life Sciences staff.

As shown in fig. 2, the paper fibers in the control were compacted and dehydrated. The paper fibers appear to be more swollen and less compacted in samples treated by immersion (fig 3). Samples treated on the suction table showed a drastic physical change in the paper fibers apparent at the 100xM range. Some fibers were swollen, disturbed, and raised out of the surface plane; while other areas of the same sample remained compacted and looked similar to the fibers in the control (fig. 4).

Iron deposits examined in samples treated by immersion in chelating solutions completely coat the paper fibers and significant corrosion is apparent at the 2500xM range (fig. 5). Samples treated by immersion in SDT followed by chelating solutions showed very fine, dense, iron deposits with no corrosion coating the surrounding paper fibers (fig. 6). Examination of samples treated with local application of SDT

followed by chelating solutions on the suction table had iron deposits that were slightly less corroded (fig. 7). Interestingly, many small deposits are seen extending from large deposits into the support at the 100xM range indicating lateral movement of the iron deposits as a result of local treatment (fig. 8).

DISCUSSION

Treatment by immersion in SDT followed by immersion in chelation tested on samples 5 to 8 were the most successful in reducing visible staining and decreasing the iron ions because the treated surface area was maximized and longer open working times were possible. Chelation treatment appeared ineffective in samples after forty-five minutes to an hour of treatment. Absorption of UV light was significantly reduced and spectrophotometer readings recorded a significant shift



Fig. 2. Untreated, desiccated and compacted fibers: control at 522xM, working distance 10.3mm, area 1. Image taken by Kayleigh Fuller, Conservation Program staff.



Fig. 3. Slightly hydrated and less compacted fibers: sample 2 immersion treatment in DTPA at 534xM, working distance 10.5mm, area 3. Image taken by Kayleigh Fuller, Conservation Program staff.

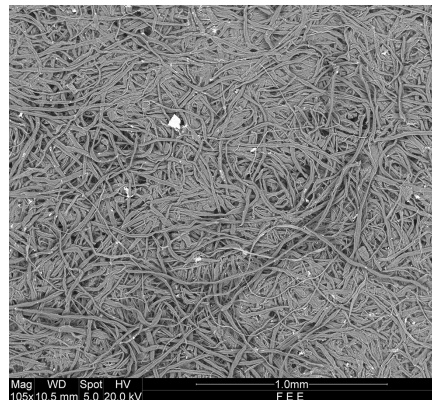


Fig. 4. Disturbed paper fibers in some areas and compacted fibers in others: sample 10 localized treatment with 2% SDT followed by DTPA at 105xM, working distance 10.5mm, area 3. Image taken by Kayleigh Fuller, Conservation Program staff.

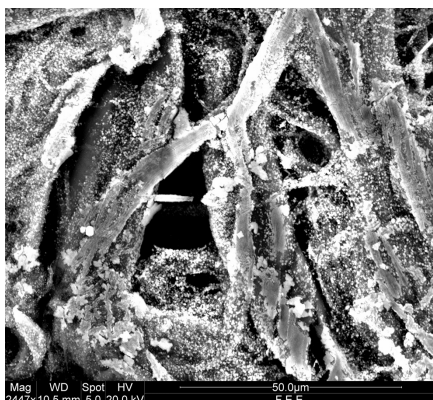


Fig. 5. Corroded iron deposit with fibers coated with corrosion: sample 2, immersion treatment with DTPA at 2447xM at 10.5mm working distance, area 2. Image taken by Kayleigh Fuller, Conservation Program staff.

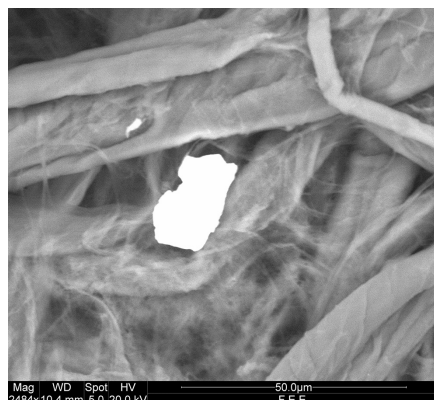


Fig. 6. Very dense, small iron particles with no corrosion coating surrounding fibers: sample 8, immersion treatment with 2% SDT followed by EDTA: 3484xM 10.4mm working distance, area 6. Image taken by Kayleigh Fuller, Conservation Program staff.

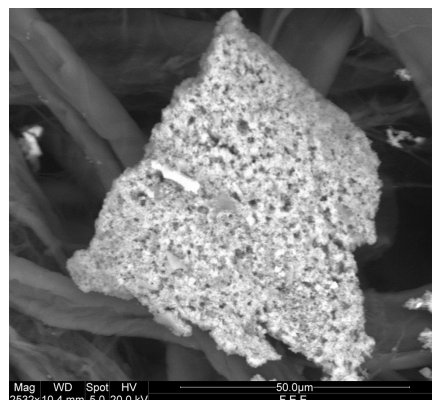


Fig. 7. Moderate corrosion of large iron deposits with very little corrosion on surround fibers: sample 10, localized treatment with 2% SDT and DTPA at 2532xM, working distance 10.4mm, sample 10 area 4. Image taken by Kayleigh Fuller, Conservation Program staff.

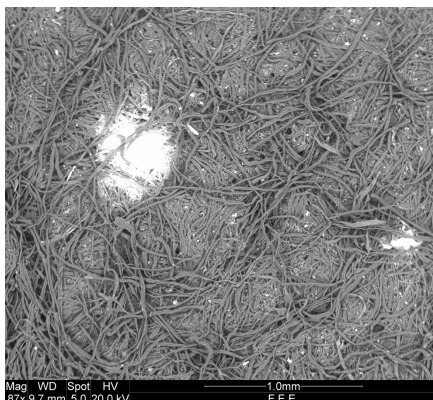


Fig. 8. Small iron particles scattered laterally around large iron deposits: sample 10 localized treatment with 2% SDT and DTPA at 87xM, working distance 9.7mm, area 5. Image taken by Kayleigh Fuller, Conservation Program staff.

from red to green. XRF analysis showed a slight increase in the amount of iron ions removed from the paper supports compared to samples treated by chelation without SDT. Physical differences in the fibers and iron deposits in select samples after treatment related to treatment application methods as well as using SDT in combination with chelates were observed with SEM.

Visual analysis in normal light, spectrophotometer readings and SEM did not allow for differentiations between either the performance of the two chelates tested or the effects of solution pH on treatment outcomes. However, XRF revealed that all samples treated with EDTA had greater decreases in iron concentrations than samples treated with DTPA. This was unexpected because EDTA has two fewer complexing sites than DTPA and its formation constant is lower than the iron(II) solubility product. Also, samples treated with EDTA showed a pale yellow fluorescence. XRF analysis indicated that pH 9 chelate solutions generally gave better treatment results than pH 10 solutions. This was surprising because a higher pH would allow for more of the complexing sites in the chelate molecule theoretically improving chelation.

This preliminary investigation has shown that chelating agents DTPA and EDTA were noticeably effective in reducing iron concentrations and visible staining in samples. It is important to continue investigating chelation treatments because of its ability to successfully treat aggressive iron degradation in paper objects. Future research opportunities include the investigation of aging characteristics of chelation treatments as well as refining treatment methods to improve open working times, control the surface area treated, and reduce physical changes in the paper supports.

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NOTES

1. It is important to note that foxing may also refer to localized discoloration caused by microbiological attack, localized moisture condensation, and irregular accelerated aging. Choi, S. 2007. Foxing on paper: a literature review. *Journal of the American Institute for Conservation*. 46 (2): 137-152.

2. The solubility product for iron(II) hydroxide is 15.1 and for iron(III) hydroxide is 37.4. These numbers show that iron(III) salts are very insoluble and therefore difficult to remove from paper with aqueous treatments.

3. DTPA is an octadentate chelate with eight complexing sites giving it the potential to form stable bonds with metal ions. The DTPA formation constant for iron(II) is 16.0 and for iron(III) is 27.5. Rivers, S. and Umney, N. 2003. *Conservation of Furniture*. Oxford: Butterworth Heinemann.

4. EDTA is a hexadentate chelate with six potential complexing sites. The formation constant of EDTA for iron(II) is 14.3 and for iron(III) is 24.2. Rivers, S. and Umney, N. 2003. *Conservation of Furniture*. Oxford: Butterworth Heinemann.

5. DTPA will successfully complex iron(II) but iron(III) salts will not be effected. EDTA has a slightly lower formation constant compared to the iron(II) solubility product yet it is the chelate most commonly used in paper conservation.

6. Sodium metabisulfite is used as an alternative for SDT to decrease health and safety risks. However, SDT is more commonly used in combination with chelation treatments because sodium metabisulfite is not as effective. Irwin, S. 2011. A comparison of the use of sodium metabisulfite and sodium dithionite for removing rust stains from paper. In *The Book and Paper Group Annual, Vol. 2*. Washington, DC.: AIC. 37-46.

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APPENDIX

Photographic Equipment

Visible Light Photography:

- Camera-Cannon EOS 6D, 50mm lens
- Light Source- 'Cold' studio white lights

Ultraviolet Fluorescence Photography:

- Camera-Cannon EOS 6D, 50mm lens
- Light Source- Large UV BLB Strip Lights from CLE Design Ltd 280-440nm short wave UV
- Filter- Camera- 2E Kodak Filter

Suppliers for Materials

- *Whatman No. 1 (ashless) filter papers*: Sigma-Aldrich Company Ltd.
- *Ammonia 20%*: Sigma-Aldrich Company Ltd. # UN2672
- *DTPA for complexometry ≥ 99.0%*: Sigma-Aldrich Company Ltd. #32319-100G-F
- *EDTA ≥ 98.0%*: Fluka Analytical through Sigma-Aldrich Company Ltd. #03620-250G
- *Sodium Dithionite*: BDH Laboratory Supplies #GPR UN1384

BROOK PRESTOWITZ

Kress Conservation Fellow
 Conservation Center for Art and Historic Artifacts
 Philadelphia, PA
 brookprestowitz@gmail.com

DR. CHARIS THEODORAKOPOULOS, PHD.

Senior Lecturer in Conservation Science
 Northumbria University
 Newcastle upon Tyne, UK
 charis.theodorakopoulos@northumbria.ac.uk

DR. JANE F. COLBOURNE

Program Leader and Senior Lecturer for Conservation of Fine Arts
 Northumbria University
 Newcastle upon Tyne, UK
 jane.colbourne@northumbria.ac.uk