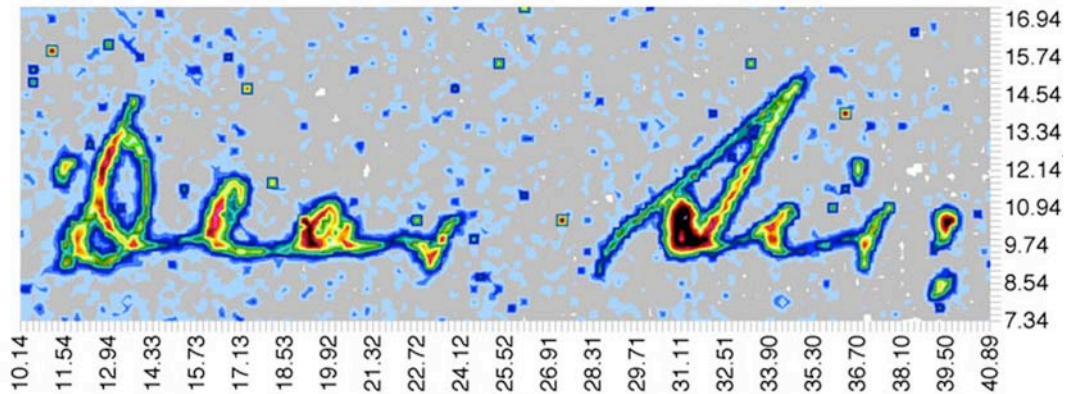


The Conservation of Letterpress Copying Books: A Study of the Baird Collection

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1. Abstract

This study investigates the conservation treatment options to preserve the treasured Smithsonian collection of letterpress copying books handwritten by Spencer Fullerton Baird (1823-1887), the second Secretary of the Smithsonian Institution. This study employs analytical techniques to investigate the complex nature of the materials, explores and evaluates treatment options with experimental procedures using artificially aged samples, and investigates best practices for the digitization of the materials. Technical analysis showed that iron II ion migration is a particularly severe problem in copying books and confirms a correlation between iron migration and the severity of ink corrosion. Several conservation treatments were conducted including anti-oxidant treatments, sizing, de-acidification, and paper splitting. One of the most promising treatments conducted is a non-aqueous anti-oxidant treatment using Tetrabutyl Ammonium Bromide [TBAB] in ethanol followed by non-aqueous de-acidification with magnesium oxide using the Bookkeeper spray and sizing with Klucel G in ethanol. Several imaging techniques were explored, and a simple and inexpensive set-up and procedure was found to give excellent results.

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3. Introduction and Literature Review

3.1 Project Overview

This study investigates the conservation treatment options to preserve the treasured Smithsonian collection of letterpress copying books handwritten by Spencer Fullerton Baird (1823-1887), the second Secretary of the Smithsonian Institution. Housed in the Smithsonian Institution Archives, the Baird copying books comprise seventy-nine volumes of outgoing correspondence (1850-1877) including approximately 44,000 leaves



Figure 1. Spencer Fullerton Baird. Photo credit: SI Archives.

bound in quarter-leather cloth bindings. The letters document, not only the history of the Smithsonian Institution, but also the growing fields of museology and natural history in the mid-nineteenth century. This unique primary source provides an irreplaceable window into a formative period of the history of the nation. The copying books are currently unavailable to researchers due to their severely deteriorated condition. The fragile paper exhibits conditions such as fading inks, offsetting, feathering, embrittlement, and letter drop-out or lacing, which are symptomatic of advanced iron gall ink corrosion and are commonly found in copying books. Much of the microfilm of the Baird books is illegible, so large portions of the information they contain are completely inaccessible.

This study employs analytical techniques to investigate the complex nature of the materials, explores and evaluates treatment options with experimental procedures using artificially aged samples, and investigates best practices for the digitization of the materials. The use of analytical techniques compliments previous historical research by verifying as well as challenging the claims of marketing materials and patents, and by further differentiating the composition of the inks and papers from traditional writing materials. The experimental phase has resulted in an evaluation of treatment options, weighing their benefits and disadvantages, which may be used as a guide for conservators and archivists in treatment planning and prioritization. The digitization exploration has resulted in a best practice procedure, which employs inexpensive and accessible equipment to gain high quality images requiring minimal processing time. The knowledge gained through the study of the Baird copying books is directly applicable to the conservation and preservation of millions of artifacts in thousands of archival collections worldwide.

3.2 History, Use, and Composition of Copying Books

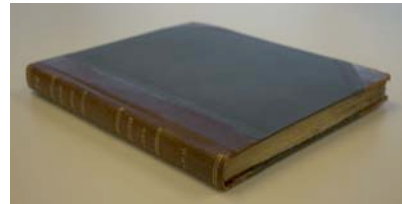
Copying books are an early type of document copying process. They were commonly used in office environments throughout the 19th century and even as late as the 1950's (Rhodes 1999, 113). The historic copy press process involved the transfer of ink on a freshly written document to a moistened sheet of copying paper through the use of direct contact and pressure. The books were manufactured specifically for this purpose, and



Figure 4. Copying press. Photo credit: Early Office Museum.

would have been purchased blank with between 300 and 800 pages of extremely thin tissue paper. The user would have written a letter on a regular sheet of writing paper but with specially formulated copying ink, which would have stayed wet longer than a standard writing ink. A sheet of copying paper would have been dampened and the original

document laid onto the damp sheet. The book would be closed and put into a copy press, transferring the ink onto the copying book page. Because the soluble copying ink was transferred directly, it left a mirror image print to be read from the verso of the thin paper (Rhodes 1999, 9).



Figures 2-3. Baird copying books.

Most copying inks are iron gall inks with additional ingredients to improve their copying capability. It was necessary for the ink to remain wet for an extended period in order to achieve multiple high quality copies without causing unacceptable damage to the original. Copying inks and papers were impregnated with hygroscopic ingredients and colorants with high tinctorial power to improve the clarity of the copy and the number of copies that could be produced. The copying paper had to be thin enough to read through the verso of the sheet, but also had to be strong enough to withstand the strains of being handled wet and pressed repeatedly. These parameters led to the experimentation and development of many different formulations of ink and papers (Rhodes 1999). Some papers and inks were manufactured to be used together to achieve a particular result, but in practice they may have been used separately with other products.

This experimentation and development of ink properties created not only a great variety of ink formulations, but also a general decrease in the quality and permanence of copying inks (Cleveland 2000, 24). As manufacturers were under pressure to make inks more convenient to use, more effective at copying, and less expensive, they resorted to methods that compromised quality. These methods included the reduction in the amount of iron used and the addition (often in excess) of several unstable substances, including (1) colorants, such as aniline and logwood dyes; (2) acids, such as vinegar, hydrochloric, carbolic and sulfuric acids; (3) alcohol, usually in the form of beer or wine; (4) preservatives such as alum and clove; and (5) countless hygroscopic ingredients including sugar, honey, molasses, mucilage, glycerin, deliquescent salts, and marshmallow root.

Copying paper quality also suffered from the development of optimal copying properties. The papers were often pre-conditioned with aqueous solutions in order to physically encourage the ink to transfer, chemically alter the ink upon contact, or to “fix” the ink to the paper fibers (Cleveland and Rhodes 1999, 40). By the 1860’s some copying books

were advertised to be “perpetually moist at room temperature” (Cleveland and Rhodes 1999, 43). Additives included hygroscopic components, such as glycerin, sugar, honey, deliquescent salts, and gelatin; and metal salts and mordants, such as ferrous sulphate (vitriol or copperas), copper acetate (verdigris), aluminum acetate, potassium chromate, potassium aluminum sulphate (alum), sodium arsenite, and tannic acid. These solutions often also contained acids, such as vinegar; alkaline components such as borax and calcium carbonate; and adhesives such as albumen, gum arabic, and gluten. The complexity of these prepared papers and their interactions with various ink formulations is unpredictable and often disastrous. See appendix A for a complete list of possible ink ingredients and paper additives.

3.3 The Conservation Challenges of Copying Books

There are several factors that make the conservation of letterpress copying books a particularly difficult problem. First of all, the volume of the materials is quite daunting. It is a widespread and serious problem affecting nearly every archive in the Western world. Archival collections often contain hundreds or even thousands of volumes of copying books, and a single volume contains between 300 and 800 leaves. This large quantity of materials necessitates a practical solution that can streamline the treatment and/or digitization process.

Secondly, the nature of the materials is complex and poorly understood. To date, little literature has been produced on the conservation of letterpress copying books, and only very limited scientific analysis of the materials has previously been conducted. Most copying books contain many different inks because copying ink recipes vary widely in composition, people tend to use whatever they have on hand, and the materials used in ink production were often impure. Even with extensive testing, it is impossible to differentiate them all. This means that there are many variables to consider in the treatment of any single copying book, as the various inks and paper preparations may respond differently to treatment. With this great variation in material composition, it may not be possible to develop a single treatment method that is effective and safe to conduct on all copying books.

Thirdly, the severity of the deterioration of copying books is distressing. A survey conducted in 2008 illustrated the severity of the problem, as 74% of questionnaire respondents reported that copying books in their collections were currently losing information (Antoine 2009). Copying books often contain the only extant version of a record as the originals were routinely discarded or lost (Ubbink and Partridge 2003, 39). In archival collections, copying books are commonly restricted from access to researchers due to their extremely fragile condition, and many have either not yet been digitized or were done so poorly.

And finally, these unusual papers and inks create special challenges for conservation treatment because they are extremely fragile, they are sensitive to aqueous treatment, and they are in a bound format. Copying papers with iron gall ink deterioration are

particularly sensitive to water because the papers and inks are very hygroscopic, the paper is very thin, and the deterioration is severe. Although the fibers used to make copying papers were typically of high quality, some of the manufacturing techniques cause them to be vulnerable in aqueous treatment. In order to make the paper transparent enough to read through the sheet, the fibers were heavily beaten and the freshly formed sheets were calendered, or fed through heated rollers while drying (Rhodes 1999, 50). These techniques eliminate air pockets and voids by breaking down the fiber structure and reducing porosity, making the paper appear translucent. Papers made with heavily beaten fibers are extremely hygroscopic and expand significantly in humid conditions, causing them to be especially vulnerable to planar distortion and dimensional changes during aqueous treatment (van der Reyden 1993, 201). The ink is likely to crack during aqueous treatment from the stress caused by uneven wetting at the borders between the hydrophilic areas of the un-sized, impregnated paper and the hydrophobic areas of ink deterioration (Reissland 2000, "Side Effects," 112). The inks also become quite sticky when remoistened with water, likely due to the added humectants, which causes problems when drying the materials on a support after treatment (Antoine 2009, 122). The inked areas are likely to stick to any support used in conservation, causing the loss of letters when removing the support. Currently, the most effective and promising conservation treatment for iron gall ink deterioration is an aqueous anti-oxidant treatment using calcium phytate followed by de-acidification. An effective non-aqueous version of this treatment has not yet been developed. Since aqueous treatment is not appropriate for the treatment of copying books, no treatment has been shown to safely chemically stabilize the rampant iron gall ink corrosion in copying books. Currently, the most effective treatment for copying books is mechanical stabilization by mending or lining with a solvent-set or heat-set method (Ubbink and Partridge 2003; Antoine 2009; Titus 2009). The bound format of copying books is also problematic for treatment. Dis-binding the books for treatment is an option, but is time-consuming and dramatically changes the nature of the objects. These materials are not simply carriers of information. They are examples of an historic copying process, and their value as artifacts should be weighed in any treatment decision.

This study builds upon a research project conducted by the author in 2008, which illustrated a great need for further research on the topic (Antoine 2009). The previous study investigated preservation needs in archival copying book collections through a questionnaire sent to professional listservs; developed an assessment tool for consistent survey and examination of letterpress copying book collections; and explored treatment options including lining, mending, humidification and flattening, and washing. Although the previous study did develop an effective lining technique for copying book paper, lining is not always a desirable treatment solution and it does not address the widespread ink corrosion problem.

In an effort to develop an appropriate conservation treatment for these complex and fragile materials, this study investigates the composition of the materials through scientific analysis, examines the current research for the treatment of iron gall ink corrosion, and experiments with and evaluates treatment protocols adapted to fit the needs of the materials.

3.4 Iron Gall Ink and Copying Books

3.4.1 History, Composition, and Degradation Mechanisms of Iron Gall Inks

Commonly used since the late Middle Ages and well into the twentieth century, iron gall ink may be the most important ink in Western history (Eusman and Karnes). The use of iron gall ink has several advantages over other early writing inks, such as carbon black ink. It is easy and inexpensive to make; it does not clog the quill; it stays in suspension without constant stirring; and, most importantly, it is indelible (Krekel 1999, 54).

The four basic components of iron gall ink are tannic- or gallic acid (usually extracted from galls), an iron salt (usually iron II sulphate, a.k.a. vitriol or copperas), a binder (usually gum Arabic), and a liquid (usually water, wine, or vinegar); but recipes vary widely in procedures, ingredients, and proportions. During use, the colorless ink reacts with the air to form a blue-black iron III complex, which is insoluble in water. During this reaction, sulphuric acid is also formed, causing the pH of the ink to lower to 1.5-3 (Krekel 1999, 54).

Iron gall ink degrades the paper substrate by two forms of decomposing reactions; namely, acid catalyzed hydrolysis and oxidation catalyzed by transition metal ions. An acidic environment accelerates those reactions (Banik 1997, 23). Ink corrosion progresses through four stages of degradation, which can be monitored by the following conditions: (1) ultraviolet fluorescence around the edges of the ink lines, (2) brown discoloration surrounding the ink line (haloing), (3) migration of the discoloration throughout the substrate with common offsetting to adjacent pages, and (4) loss of material in the ink lines (lacing) and overall embrittlement of the substrate (Reissland 2000, "Progress," 116).

Acid hydrolysis of cellulose is caused by the sulphuric acid inherently present in the ink. It is water-soluble and migrates out of the ink line and throughout the paper substrate with age, especially in humid or fluctuating RH conditions (Neevel and Mensch 1999, 531). Acid hydrolysis causes the depolymerisation of the cellulose molecule, resulting in the loss of mechanical strength of the substrate (Reissland and Hofenk de Graaff, 2000).

Most historic inks were made with excess iron (II) sulphate, causing them to be "unbalanced" and to degrade the paper substrate more quickly than those with the stable iron to tannin molar ratio of 3.6:1 (Neevel and Mensch 1999, 528). This excess iron does not react with the tannins in the ink during ink production, and therefore remains in the material as free iron II ions, which are water soluble and catalyze the oxidation of cellulose through the "Fenton reaction" (Neevel and Mensch 1999, 528). This excess of iron increases with age as the tannic acid degrades causing the ratio of iron to tannin to continually increase (Krekel 1999, 56). Oxidation causes the depolymerisation and cross-linking of the cellulose, resulting in fluorescence, browning, embrittlement, and loss of mechanical strength of the substrate (Reissland and Hofenk de Graaff, 2000).

Transition metals other than iron are common in historic iron gall inks, because in addition to iron sulphate, copper and zinc sulphates were often called for in historic recipes, and many ingredients were commonly contaminated with copper, zinc, manganese, and aluminum (Krekel 1999, 55). All of these metals are destructive to cellulose and aggravate the problem of iron gall ink corrosion. Like iron II ions, copper II ions catalyze the oxidation of cellulose. Zinc sulphate can be hydrolyzed to zinc oxide, becoming a photo-catalyst for the radical oxidation of cellulose. And, aluminum sulphate and potassium aluminum sulphate (alum) can cause the acid hydrolysis of cellulose (Neevel and Mensch 1999, 531).

The severity of iron gall ink corrosion on a particular object depends not only on the composition of the ink and the object's storage conditions, but also the depth of the penetration of the ink into the paper (Reissland 2000, "Progress," 114). Deeper penetration encourages corrosion to spread through to the verso of the substrate, causing ink drop-out or lacing. The thinness of the paper, heaviness of ink application, and amount of sizing all contribute to the depth of penetration.

The hygroscopicity of copying books due to additives in the papers and inks likely contributes to the severity of the problem by enabling the migration of soluble and destructive ink components. Because copying paper is thin, un-sized, and particularly hygroscopic due to additives, as well as its composition of heavily beaten fibers, it is reasonable to extrapolate that copying books are especially vulnerable to iron gall ink corrosion.

3.4.2 Conservation of Iron Gall Ink

The search for a solution for the preservation of paper containing iron gall ink has been underway for over one hundred years. In 1898, P. Ehrle, the head of the Vatican Library held a conference addressing the problem of iron gall ink corrosion for the first time (Banik 1997, 21).

Current research dictates that an appropriate treatment for iron gall ink corrosion should follow a three-pronged approach (Schafer 2004). It must arrest the acid catalyzed hydrolysis by neutralizing the sulphuric acid in the ink, deactivate or remove the transition metal ions that catalyze oxidation, and strengthen the weakened substrate mechanically when necessary. The most accepted treatment protocol currently employs an aqueous anti-oxidant treatment using calcium phytate, followed by de-acidification with calcium bi-carbonate and re-sizing with gelatin. A detailed work standard has been developed for this treatment (Huhsmann and Hahner 2008), and it has been proven unambiguously to stabilize the paper on a molecular level (Henniges and Potthast 2008, 231).

Anti-oxidants inhibit the oxidation of cellulose catalyzed by transition metal ions by interfering with the pathways of the Fenton reaction. By forming a complex with the metal ions, they eliminate coordination sites for oxidation (the Fenton reaction) to take

place, thereby blocking the decomposing reaction (Neevel 2002, 76). Kolar et al. 2005 includes a detailed description of the three types of anti-oxidants: peroxide decomposers, chelating agents, and radical scavengers; and the mechanisms employed by each of them.

De-acidification prevents further acid hydrolysis by neutralizing the sulphuric acid in the ink and any other acids present in the ink and paper. De-acidification has been shown to greatly improve the results of anti-oxidant treatments (Havlinova et al. 2007, 126), but treatment with a strong alkali solution is not recommended because the blue-black colorant in iron gall ink is dissolved and turns reddish-brown in pH higher than 8.0 (Krekel 1999, 57).

Gelatin has been shown to be the most effective sizing agent when re-sizing iron gall ink on paper after aqueous treatment. It seems to have the ability to fix free iron II ions present in unbalanced iron gall inks by binding them in an elastic film, thus making them inert (Kolbe 2004, 35).

Although calcium phytate is the most proven and accepted anti-oxidant treatment currently in use, it does have a few disadvantages. Phytate complexes with iron II ions but no other transition metal, leaving other metal ions free to catalyze the oxidation of cellulose. It also relies heavily on washing in aqueous solution to remove free iron II ions from the paper (Neevel 2002, 84).

In the last decade, a few projects have been undertaken to develop a treatment for water-sensitive objects that contain iron gall ink. These studies have built upon research into the use of halides, such as bromides and chlorides, as effective anti-oxidant alternatives to calcium phytate (Kolar et al. 2003). Halides show great potential because they are not iron-specific. They have the capacity to deactivate other transition metal ions, most notably copper ions, which are believed to be as destructive as iron II ions during ink corrosion (Kolar et al. 2003, 765). Another benefit of halides is their solubility in organic solvents. Halides not only have the ability to effectively stabilize iron gall inks regardless of their composition (Malesic et al. 2005, 14), but they also can be used in non-aqueous solution.

Researchers in the European Commission (EC) co-funded project, InkCor, compared several halides, and found that Tetrabutyl Ammonium Bromide (TBAB) had a stabilizing effect on ink corroded model paper (Malesic et al. 2005, 15). The InkCor project investigated non-aqueous treatment for water-sensitive materials with iron gall ink corrosion by testing several halides in combination with Magnesium ethoxide in various non-aqueous solvents. Unfortunately, the prototype treatment solutions, known as *inksave*, that were found to be effective are proprietary, so the formulations will not be made public. The solutions were evaluated primarily based on visual changes caused by treatment, but samples were also subjected to bursting strength testing. The results were very positive, but the data was subject to error due to a small sample size. The solutions have not been widely adopted by the conservation community, and members of this group continue to test these and other treatment solutions.

Maitland built on InkCor's research by testing the use of TBAB in both aqueous and non-aqueous solution, for the stabilization of corroded copper watercolor pigments and iron gall inks (Maitland 2007). The study showed promising results for non-aqueous ink corrosion treatments with an increase in fold endurance and lower concentrations of iron II ions in samples treated with TBAB in ethanol.

These recent studies lead the way for experimentation with non-aqueous TBAB solutions in combination with de-acidification and sizing on copying book materials. Treatment procedures other than immersion remain untested in the previous studies. Because dis-binding is an undesirable requirement for immersion treatment, exploring in-situ treatment procedures could be highly beneficial for the treatment of copying books.

4. Materials Characterization of the Baird Books

4.1 Overview

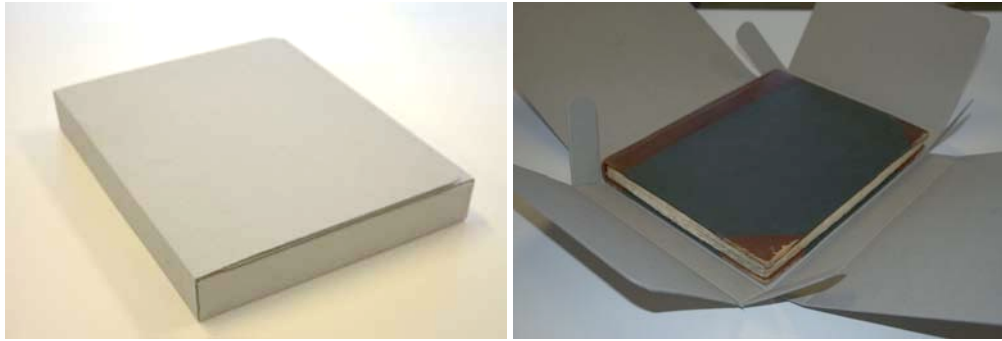
With the help of the scientists at the Museum Conservation Institute of the Smithsonian Institution, visual examination, solubility testing, and technical analysis were conducted in order to characterize the materials that comprise the Baird letterpress copying books. After conducting a thorough survey of the collection, analysis was performed to determine the components of both the paper and the inks on a molecular and elemental level in an effort to gain a deeper understanding of the degradation processes of these materials, and to discover clues for their preservation.

4.2 Survey

A visual examination of each volume in the collection was conducted, and the data was entered into a File Maker Pro database. The collection consists of 79 volumes of copying books of Spencer Fullerton Baird's correspondence from September 1850 to November 1877. Each volume spans between 3 and 6 months of material. There is some overlap in dates, presumably because he had more than one office (Henson 2009). The collection is housed in 24 document boxes, stored spine down in archival folders, 3-4 volumes per box with spacers inserted as needed. During the course of this project, the books were re-housed in custom e-flute corrugated board phase boxes (see figures 6-7). The collection is permanently stored off-site at Iron Mountain, a climate controlled space (55F, 35% RH). Volume numbers run from 1 to 81; volumes 61 and 64 are missing.



Figure 5. Survey in progress in the Smithsonian Center for Archives Conservation Lab.



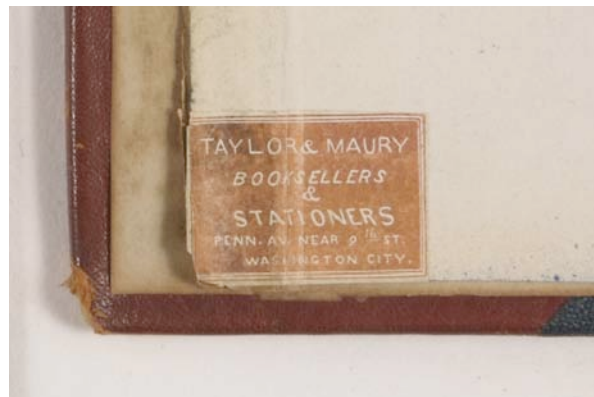
Figures 6-7. Baird books stored in custom e-flute corrugated phase boxes.

4.2.1 Collection Description

The volumes are covered in matching half brown calf and dark green cloth with the initials “SFB,” the volume number, and dates of use stamped in gold on the spine. Although the covering materials and finishing of the bindings match, their construction varies slightly, and only two volumes contain a binder’s ticket. None contain any of the commercial marketing labels common to copying books. These details indicate that the collection was not

bound at the same time or by the same binder. It is likely that the books were re-bound over time as they were filled and ready to be filed or that the textblocks were custom bound at the time of purchase and finished later to reflect the dates of use. The two binder’s tickets are in volumes 10 and 25, and are from Taylor & Maury Booksellers & Stationers Penn. Av. Near 9th St. Washington City and Metropolitan Bookstore Philp & Solomons 332 Pennsylvania Av. Washington, D.C.,

respectively. Most of the bindings are faux tight-back cases and are sewn on parchment slips or linen tapes. Some textblocks include edge treatments including green, blue, or red sprinkles. All volumes include an index at the front of the textblock, but they vary in color and format. The dimensions of the bindings vary between 28.2H x 23.2W cm and 31.5H x 27W cm. The textblocks vary in size, dimension, color, and paper thickness. Each volume includes between 300 and 800



Figures 8-9. Binder’s tickets found in the Baird books.

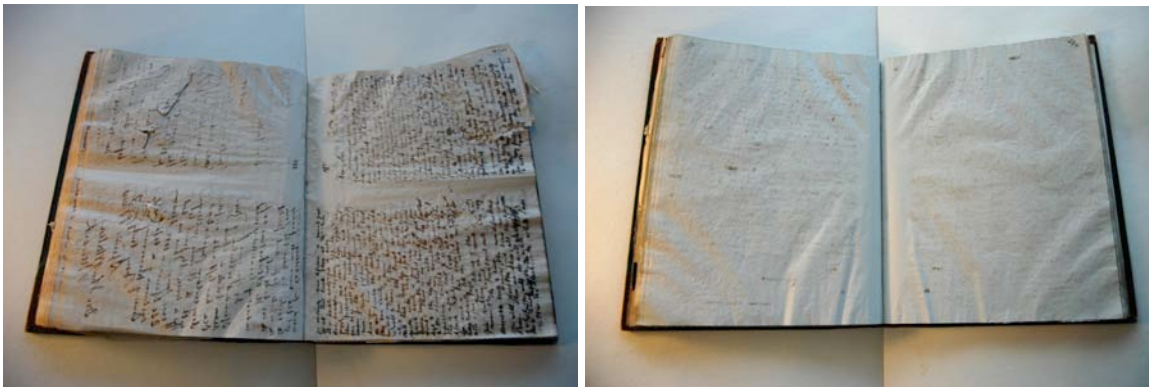


Figure 10. Smithsonian Property stamps found in the Baird books.

leaves, totaling 44,395 leaves in the collection. Paper color ranges from Cream 2 to Beige 2 with a few blued white examples (Lunning and Perkinson 1996), and paper thickness ranges from 0.025mm to 0.054mm with the average being 0.036mm. The predominant medium is iron gall ink. Other media include aniline ink, graphite, black and red stamp pad ink, blue and red crayon, and blue ballpoint pen. All volumes include many different inks. It is impossible to know how many inks are present. Except for the first few volumes, most page numbers are in black stamp pad ink. Many volumes include a red Smithsonian Institution property stamp. There are a few original letters that correspond with copied letters within the collection. These letters are housed in archival folders in the document boxes along with the copying books that they correspond to.

4.2.2 Collection Condition

While no volumes were found to be in excellent condition, 37 were in good condition, 27 in fair condition, and 15 in poor condition. Most volumes include a range of condition levels, indicating that the media plays a major role in the severity of the deterioration of the paper. 70 of 79 volumes, or 89% of the collection is currently losing information. All volumes exhibit some symptoms of iron gall ink corrosion, including brown haloing, letter drop-out, and embrittlement (see figure 11). A sweet, syrup smell appears to correlate with severe iron gall ink corrosion. It was noted that the most severely corroded volumes smell the strongest, while many of the volumes in the best condition do not smell at all (see figure 13). Few volumes exhibit water damage beyond the cockling associated with the copying process, but all volumes exhibit some feathering of the ink. This may be due to the hygroscopic nature of the paper and inks. 26 of 79 volumes, or 33% of the collection, exhibit severe fading of iron gall ink (see figure 12).



Figures 11-12. Examples of Baird books in poor condition. The volume on the left exhibits severe ink corrosion, while the one on the right is severely faded.

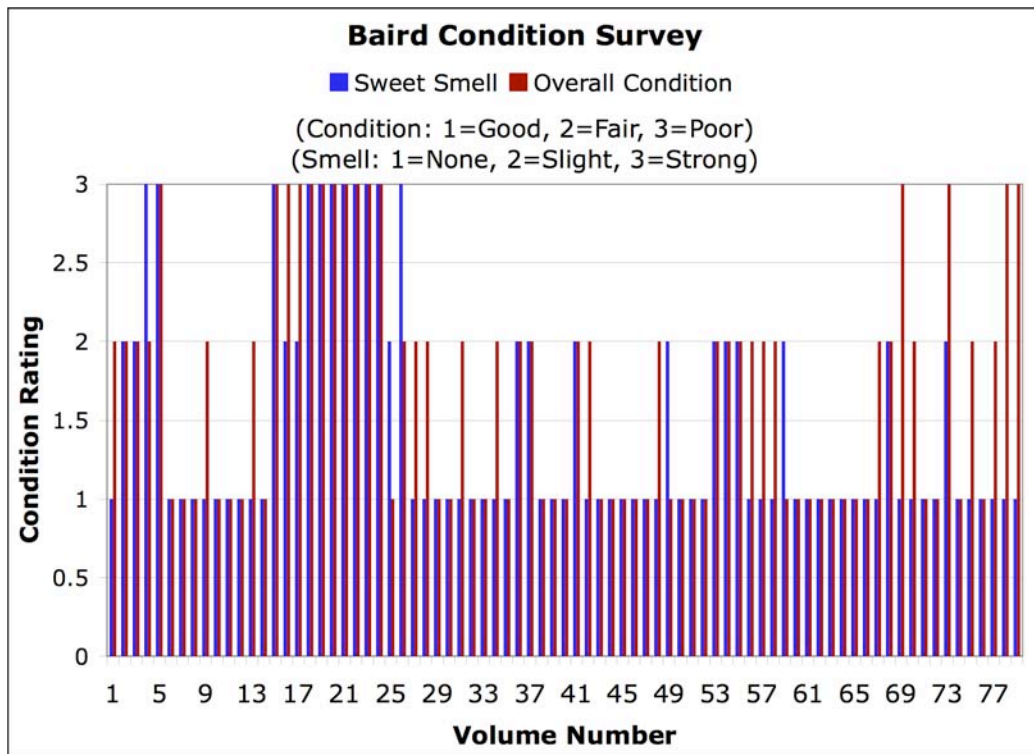


Figure 13. Correlation between sweet smell of Baird books and overall condition.

4.3 Solubility Testing

Seventeen samples were tested for solubility in de-ionized water and ethanol, both with light pressure from a damp blotter for ten seconds and with five drops from a small brush. Samples were observed under magnification.

Only one sample was soluble in ethanol (it was an aniline ink). None of the samples in good condition were soluble. Three out of five severely corroded samples were found to be slightly or moderately soluble in water, but none were soluble in ethanol. Some cracking of the ink line occurred while testing with water. Three of five severely faded samples were found to be slightly soluble in water, but none were soluble in ethanol. One of two aniline ink samples was found to be slightly soluble in water, and one was found to be slightly soluble in ethanol.

4.4 Fiber Analysis

Twenty-one copying paper samples were prepared using Isenberg's fiber analysis procedure (Isenberg 1967) and were analyzed using a polarizing light microscope in order to identify the fibers present in the samples. All samples were found to include a mix of fibers, most containing flax and cotton, some containing mechanical and/or chemical wood, and some possibly containing jute and/or hemp. Most papers appear to be fairly good quality, using strong fibers, such as flax and cotton, with only some including weak, acidic fibers like mechanical wood mixed in. This finding is consistent with historical research (Rhodes 1999, 51). The fact that the paper fibers are consistently

fairly high quality suggests that factors such as media composition, paper impregnation solutions, and storage conditions have more impact on the condition of copying books than paper quality.

4.5 XRF

X-ray fluorescence spectrometry (XRF) was conducted to characterize the Baird materials on an elemental level using a Bruker ARTAX 800 micro-XRF spectrometer with x-y mapping capability. XRF spectrometry is among the most widely used and versatile of all instrumental analytical techniques. An XRF spectrometer uses primary radiation from an X-ray tube to excite secondary emission from a sample. The radiation emerging from the sample includes the characteristic X-ray peaks of elements present in the sample. Dispersion of these secondary X-rays into a spectrum permits identification of the elements present. The height of each characteristic X-ray peak relates to the concentration of the corresponding element in the sample, allowing quantitative analysis of samples for many elements in the concentration range from low parts-per-million to 100 %. XRF spectrometers can simultaneously measure elements that occur in the range from Na (11) to U (92). Depending on the sample matrix, XRF generally produces compositional data for approximately 15 major and minor elements with good precision and accuracy and is 100% nondestructive. The instrument used for this study is equipped with a molybdenum target polycapillary lens X-ray tube that has ca. 80 μm spatial resolution. The X-ray detector is a Si drift detector with a 10 mm^2 active area and energy resolution of ca. 143eV for the Mn Ka at 100kcps. Although the sample size is too small to be definitive, results can confirm or disprove previous theories and can help develop new ones.



Figures 14-15. Bruker ARTAX 800 micro-XRF analyzing Baird books at MCI.

Forty-one samples were selected for spot analysis. Samples were selected from fifteen volumes: five in the best condition, five with the most severe ink corrosion and embrittlement, and five with the most severe iron gall ink fading. Each volume was analyzed in an area with ink as well as an area without ink for a total of thirty samples.

Eleven additional samples were included to examine aniline inks, blank paper, and different inks in a single volume. Each area was analyzed at 50 kV and 600 μ A for a live-time count of 60 seconds. The goal of this analysis was to differentiate the components of the inks from those of the papers, to compare various inks and papers, and to determine if there is a correlation between any components and conditions.

In addition to the spot analyses, five areas of the Baird books were analyzed using the x-y mapping capability of the micro-XRF. The data from these area scans were manipulated in Microsoft Excel to create visualizations of the distribution of the elements over the samples. These area maps show differences in how the components of the inks and papers have migrated and/or interacted over time. Baird's signature on three copied letters were analyzed: one in good condition, one severely corroded and embrittled, and one severely faded. These samples were chosen to investigate the behavior of the components in materials in varying conditions. The "Dear Sir" of a severely corroded copied letter as well its corresponding original were also mapped. These samples were chosen to explore how the behavior of copying paper compares to that of Baird's standard writing paper. To produce the XRF maps, an area was selected (ca. 8 x 30 mm) on the document and thousands of points within that area were analyzed at 50 kV and 600 μ A for a live-time count of 20 seconds; spacing between analyses was 0.2 mm.

The results of the spot analysis show that nearly all of the copying inks and papers in the Baird collection contain multiple transition metals in addition to iron, including copper, zinc, manganese, aluminum, chromium, and lead. The area maps confirm that many of the papers are impregnated with these metals, as copper, zinc, manganese, chromium, lead, tin, antimony, and aluminum were all detected throughout the paper, showing no trend with the ink line. It is also interesting to note that arsenic was detected in the ink and paper of one sample, likely added as sodium arsenite.

The area maps show several possible correlations between components of the materials and their conditions. The maps of iron show greater blurring of the ink lines was on severely corroded samples than samples with less corrosion, indicating that there is likely a correlation between the severity of iron migration and the severity of ink corrosion. It is interesting to note that the maps show that sulphur and potassium tend to migrate more than iron, but there are no apparent correlations with condition associated with the migration of these elements. There is also a likely correlation between the overall quantity of iron, sulphur, and potassium present in ink and the severity of fading, as the maps show faded samples exhibit much lower quantities of these three elements than all other samples. A possible correlation was also found between the presence of calcium in the ink and the good condition of the sample, as calcium exhibits a strong trend with the ink in the sample in good condition, while all others exhibit only a slight trend with the ink. Analysis shows that chlorine is usually present in the paper, but not in the ink. Spot analysis shows that with the exception of one sample, all samples that do not contain ink, contain chlorine. In the area maps, chlorine exhibits a negative trend with the ink lines in 4 of 5 samples and an even distribution throughout the fifth sample. In the inked areas the higher Z iron blocks the emission of lower energy chlorine x-rays from the paper, which results in a negative image.

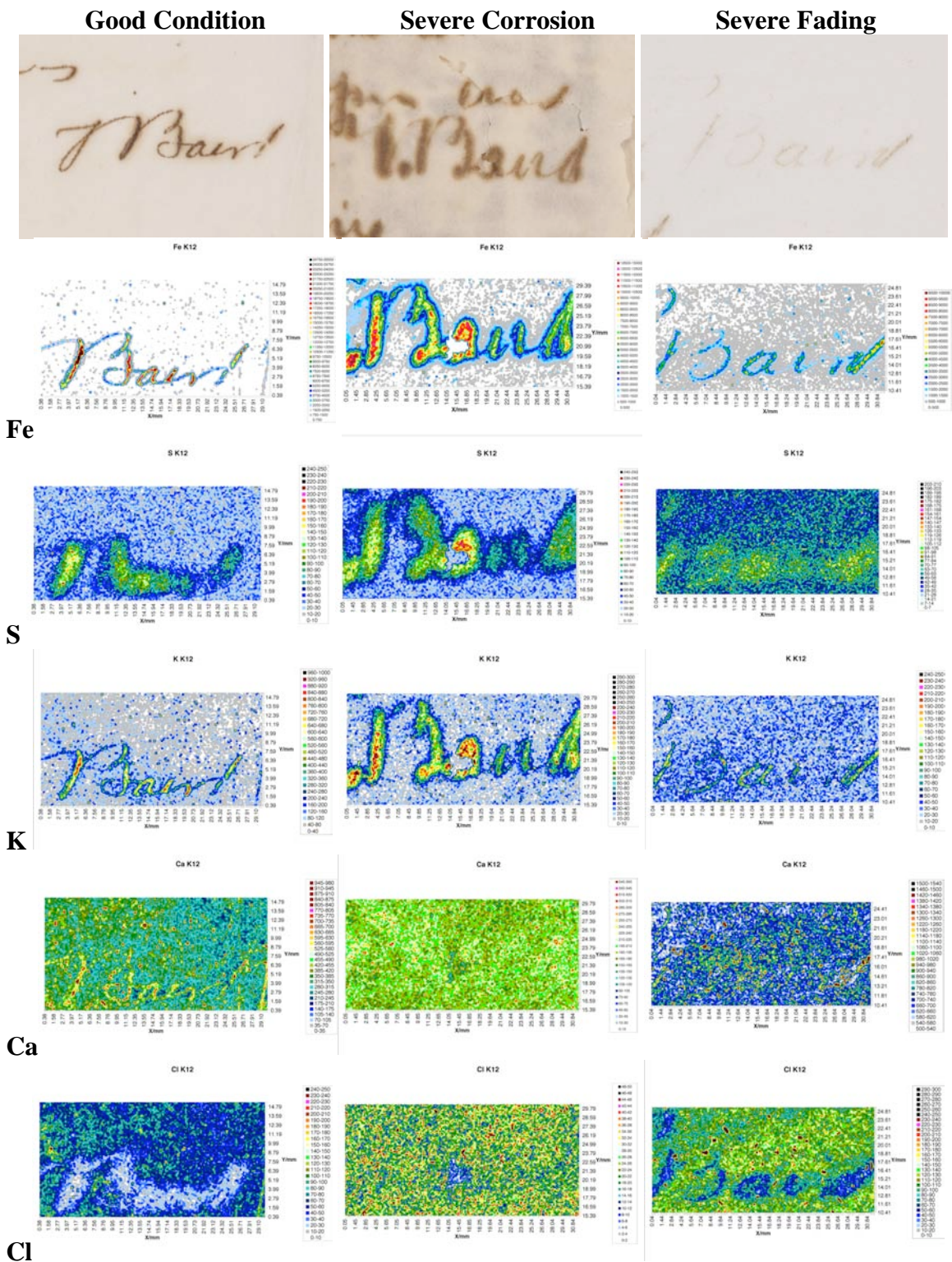


Figure 16. Comparison by XRF x-y mapping of elements present in three samples of varying condition. Left column: sample in good condition, middle: sample severely corroded, right: sample severely faded. Rows: 1. Photograph of sample, 2. Iron, 3. Sulphur, 4. Potassium, 5. Calcium, 6. Chlorine. Warmer colors signify higher concentration; cooler colors signify lower concentration.

By comparing the area maps of an original letter and its copied counterpart (figure below), it is clear that the copy exhibits greater iron migration than the original. Because the inks are the same, but the papers vary, it can be speculated that the copying paper encourages iron migration.

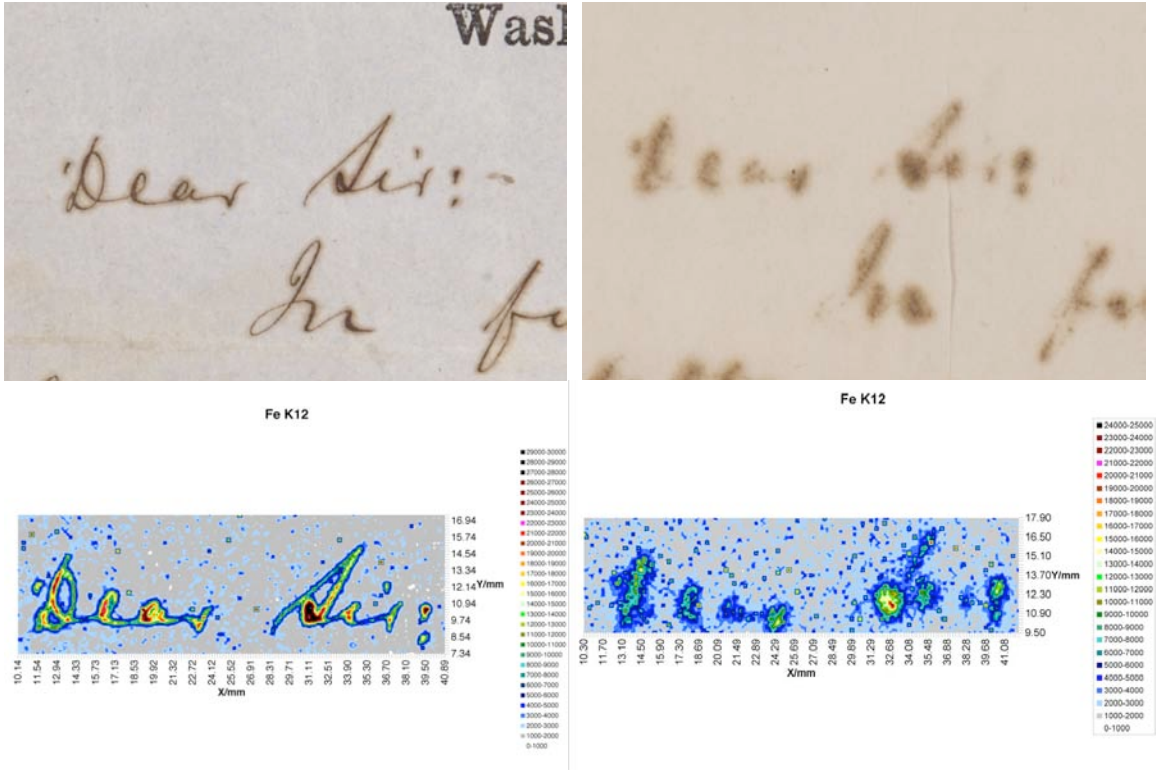


Figure 17. Comparison by XRF x-y mapping of iron present in two samples, an original letter on the left and its copied counterpart on the right. Warmer colors signify higher concentration; cooler colors signify lower concentration.

The following notes from the spot analysis may also be of interest. All samples contain manganese, potassium, and calcium. All samples except those on blank pages contain iron. All samples on blank pages contain manganese, potassium, calcium, sulphur, and chlorine. All samples that contain ink, contain: iron, manganese, potassium, calcium, and sulphur.

4.6 Summary and Discussion of Findings

Survey:

- There is not only a great quantity of materials, but there is also great variety. The papers vary in color, thickness, texture and transparency. There are an untold number of inks present with multiple inks present in all volumes. The bindings are similar in appearance, but vary slightly in construction and materials.

- Overall conditions vary from good to poor, but many volumes are very severely damaged. 89% of volumes are currently losing information. Ink corrosion is the predominant problem, as all volumes exhibit at least some symptoms.
- A sweet syrup smell associated with the volumes seems to correlate with severe ink corrosion, but the source of this smell was not identified due to time restrictions. It would be interesting to conduct Solid Phase Micro-extraction (SPME) and/or complex and simple carbohydrate spot tests to identify the material. Based on historic copying ink recipes, it is likely sugar, molasses, or honey.
- Fading is also a serious problem; 33% of the volumes exhibiting severe fading. This problem highlights the importance of digitization and the proper storage of the materials.

Fiber analysis:

- Samples contained a mix of fibers, most containing flax and cotton, some containing mechanical and/or chemical wood, and some possibly containing jute and/or hemp.
- Consistent with historical research, copying papers appear to be fairly good quality.
- The paper fibers present are not likely a major factor in determining the condition of the materials.

Solubility:

- Solubility testing showed surprisingly low solubility in ethanol for all inks except aniline.
- Corroded inks cracked upon the application of water, but not ethanol.
- Ethanol seems to be a more promising treatment solution than water for both ink cracking and solubility concerns. Perhaps items in very good condition could be treated with water, but items in poor condition (both corrosion and fading) should not be treated with water.
- All inks, especially aniline inks, should be tested extensively before treatment.

XRF Analysis:

- XRF confirms the presence of several transition metals other than iron present in copying inks and papers, illustrating the need for a treatment for ink corrosion that is not iron-specific.
- It has been hypothesized in previous research using the iron indicator test that the migration of iron II ions from the ink into un-inked areas of the paper furthers the

progress of ink corrosion (Eusman 2002). XRF analysis further confirms this theory with a positive correlation between iron migration and the severity of corrosion.

- Iron migration is shown to be particularly problematic for copying books by comparing area maps of an original document and its copied counterpart. This could be due to the fact that the copying paper is un-sized and therefore more absorbent than the writing paper of the original. It is also hypothesized that because copying ink is more hygroscopic than standard writing ink, it is more likely to solubilize and move into the surrounding absorbent paper in high or fluctuating RH conditions.
- A possible correlation between calcium in the ink and good condition suggests that de-acidification is likely a beneficial treatment for copying books.

A list of possible ingredients in the inks and paper impregnation solutions compiled from historic recipes is included in the appendix of this report.

5. Treatment Experimental

5.1 Sample Preparation

Because copying book papers and inks are quite complex and varied, an attempt was made to include all of the major variations in the materials while also controlling variables in the experimental design. Copying paper would be very difficult if not impossible to replicate, so samples were made using blank pages of two copying books from the Baird collection, in addition to a known modern Gampi tissue paper with properties similar to copying paper. An effort was made to select ink recipes that would isolate certain components of the inks in order to distinguish how these various components respond to treatments. For the purposes of this experiment, copying ink was defined as a writing ink that includes an additional hygroscopic ingredient, such as sugar or glycerin. The iron gall ink recipe used for research at the Library of Congress (LC) was used as a control. Several variants of copying inks were made by adding ingredients to this control ink. Additional components included sugar, glycerin, deliquescent salt, copper sulphate, alum, potassium chromate, Prussian blue, aniline dye, and logwood dye. Two complex inks from historic recipes were also used in order to more accurately mimic an ink and paper combination that would be found in collections materials. A total of eight inks were



Figures 18-20. Sample preparation. Ink ingredients, above; sample inks, middle; applying inks to sample papers, bottom.

applied in two lines of varying width using a Pilot Parallel Pen, which gives a constant line thickness no matter at what angle the pen is held. The main components of the sample inks are listed below; full recipes can be found in the appendix. The samples were aged prior to treatment in an aging oven for four days at 70 degrees Celsius and 50% RH. After the treatments were completed, the samples were aged for nine days in the same conditions and tested for strength, pH, and iron migration. The treatments were also evaluated for solubility problems, color changes, risk of treatment, practicality, and health and safety concerns.



Figures 21-23. Aging samples in lab oven. Samples before (left) and after aging (right.)

Sample inks:

1. Control: LC iron gall ink
2. LC iron gall ink plus sugar
3. LC iron gall ink plus glycerine
4. LC iron gall ink plus sugar and Prussian blue
5. LC iron gall ink plus glycerine and aniline dye
6. Complex historic iron gall copying ink with copper
7. LC iron gall ink plus deliquescent salt
8. Historic logwood bi-chromate ink with ferrous sulphate but no gall nuts

5.2 Treatment Procedures

Because copying papers and inks have been found to be highly sensitive to moisture (Eusman 2002, 125) and to react poorly in aqueous treatment, (Antoine 2007, 122) this study focuses on non-aqueous treatment of copying books with Tetrabutyl Ammonium

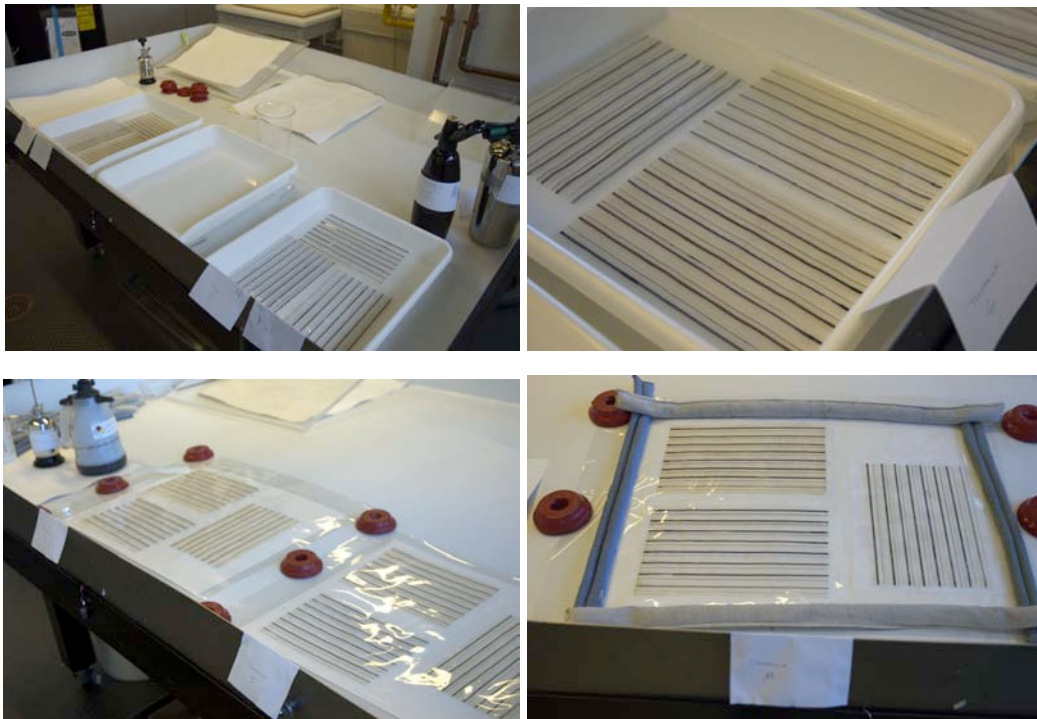
Bromide (TBAB) and its comparison to the more standard aqueous phytate treatment for iron gall ink corrosion. There are several concerns regarding the non-aqueous treatment of iron gall ink corrosion that must be considered (Reissland, 1999, 175). It requires the use of magnesium for de-acidification as opposed to calcium for solubility reasons. The high alkalinity of magnesium can be a concern for iron gall ink, because it can degrade the black colorant in the ink. Non-aqueous treatment does not remove degradation products, iron II ions, or excess anti-oxidant. The phytate treatment relies heavily on the removal of iron II ions, and the inability to accomplish this in non-aqueous treatment is likely to reduce the effectiveness of treatment. The excess anti-oxidant is a concern because no one knows how much is used up, how much is left over, and how it reacts in the paper over time (Maitland 2010). Some treatment chemicals, such as EDTA have been found to be destructive if left in the paper (Neevel 2002, 77). Suction table and capillary washing set-ups were proposed to attempt to mitigate this problem. Non-aqueous treatment lacks the benefit of an increase in paper flexibility due to the re-activation of fiber-to-fiber bonding (Reissland 1999, 169). And finally, the possible solubility of the inks in organic solvents is a concern. Both ethanol (TBAB in ethanol) and methanol (Bookkeeper) were used in the experimental treatments. Copying inks that include aniline dyes and deliquescent salts may be particularly vulnerable (Cleveland 2009).

Treatment #	Procedure
Treatment A	Control: No Aging / No treatment
Treatment B	Control: Aging / No treatment
Treatment C	Control: Aging / Washing
Treatment D	Control: Aging / EtOH
Treatment E	Phytate / CaB
Treatment F	Phytate / CaB / Gelatin
Treatment G	TBAB in EtOH – immerse
Treatment H	TBAB in EtOH – immerse / MgO
Treatment I	TBAB in EtOH – spray
Treatment J	TBAB in EtOH – spray / Size
Treatment K	TBAB in EtOH – suction table
Treatment L	TBAB in EtOH – capillary washing
Treatment M	TBAB in EtOH – spray / MgO / Size
Treatment N	TBAB in EtOH – spray / MgO / Size / Line
Treatment O	Non-aqueous Paper Splitting

Figure 24. Experimental treatments performed. Aborted treatments K & O are addressed in sections 6.2 & 6.3.4.

The great quantity of copying books in archival collections necessitates a practical approach. A fast, inexpensive, and streamlined procedure is highly desirable. Immersion treatment is the only procedure that has been studied previously for iron gall ink corrosion treatment. In this study, in an effort to preserve the bindings of copying books and to streamline the process, in-situ procedures were explored.

The procedure used for the phytate treatment was a slightly modified version of the standardized procedure published on the Ink Corrosion Website. Because TBAB is a new treatment, there is not much precedent for procedure details such as concentrations and dwell times. These procedures are based on the few previous studies that used TBAB in ethanol at a concentration of 0.3mol/L with a dwell time of 20 minutes (Kolar et al. 2005; Maitland 2009). All spray treatments were conducted on Hollytex and blotter supports enclosed in a silicone release Mylar sandwich and were kept saturated for 20 minutes. Procedure details can be found in the appendix of this report. The suction table treatment was not completed due to mechanical equipment failure.



Figures 25-28. Experimental treatments in progress. In clockwise order from upper left: Phytate treatments; TBAB immersion; TBAB spray treatment during dwell time; TBAB spray set-up.

5.3 Treatment Evaluation

The treatments have been evaluated based on several criteria. The samples were tested for iron II ion migration, pH, and tensile strength before and after treatment.

Considerations were also be made for the risks of treatment, visual changes, ease and practicality of treatment, and health and safety concerns for the conservator.

Evaluation of treatment effectiveness and side-effects are considered for archival materials, not works of art. These materials are valued primarily for the information they contain, so the importance of legibility and stability is paramount, while minor color shifts and other visual changes may be tolerated.

5.3.1 Iron Migration Testing

All eight inks were monitored for iron II ion migration with an iron indicator test before and after treatments and again after post-treatment aging. The indicator paper is filter paper impregnated with bathophenanthroline, which reacts with iron II ions to form a pink complex. The paper was dampened and lightly pressed to the samples for 30 seconds, creating a pink stain where it was in contact with iron II ions. It is not a quantitative test, but relative concentrations can be determined by comparing the darkness of the stain, as long as variables such as surface sizing and length of exposure are controlled. This test illustrates the effects of aging and treatment on the movement and relative quantity of iron II ions in a sample. Logwood is known to interfere with the results of the test, so results for ink #8 are likely invalid. Results are listed in the table below.



Figures 29-30. Iron II migration testing.

Treatment	Result	Implications
A – Control – No treatment, No Aging	<ul style="list-style-type: none"> • Most inks transferred dark pink very tight to the ink lines. • Inks 1, 2, 6, 8 appear darkest. • Ink 4 appears light pink. • Inks 3, 5, 7 appear very light. 	A lot of Iron II ions are present, but no migration has occurred.
B – Control – No treatment, with Aging	<ul style="list-style-type: none"> • All inks appear lighter than A, but much more migrated. • Inks 6 and 8 are darkest and most migrated. 	<ul style="list-style-type: none"> • Age caused iron II migration in all inks. • The LC recipe is a relatively stable iron gall ink, while 6 and 8 are less stable.
C – Washing Control	Almost no pink appears on all samples.	Washing is effective at removing iron II ions and inhibiting their formation over time.
D – Ethanol Control	Appears similar to B (no	Ethanol alone does not

	treatment).	remove iron II ions.
E – Phytate/CaB and F – Phytate/CaB/Gelatin	Almost no pink appears in all samples.	<ul style="list-style-type: none"> Phytate treatments are effective at removing iron II ions and inhibiting their formation over time. Result expected because treatments include washing.
G – TBAB in EtOH immersion	All inks appear slightly lighter after treatment, but darken after aging.	TBAB alone is somewhat effective at reducing iron II ions, but does not inhibit their formation over time.
H – TBAB in EtOH immersion / MgO	All inks appear moderately lighter after treatment, but darken slightly after aging.	MgO improves the performance of TBAB, but not nearly as effective as washing or phytate.
I – TBAB in EtOH spray	Some inks improved slightly, others had no change.	TBAB spray is not as effective as TBAB immersion.
J – TBAB in EtOH spray / Klucel G	Some inks improved very slightly, other had no change.	Klucel G had little effect on Iron II ions.
L – TBAB in EtOH capillary washing	Not very effective.	Not very effective.
M – TBAB in EtOH spray/MgO/Klucel G	<ul style="list-style-type: none"> All inks appear moderately or significantly lighter after treatment, and darken only slightly after aging. Ink 6 quite dark after aging. 	MgO and Klucel G together significantly improve the performance of TBAB, but not as effective as washing or phytate.
N – TBAB in EtOH spray / MgO / Klucel G / Lined	<ul style="list-style-type: none"> All inks appear significantly lighter after treatment, and darken only slightly after aging. Ink 6 appears light pink. 	<ul style="list-style-type: none"> Lining improves the performance of TBAB, but not as effective as washing or phytate. The most effective TBAB treatment tested.

Figure 31. Iron II migration testing results.

- All of the aqueous treatments were extremely effective at removing iron II ions from the samples and at inhibiting their return over time. The effectiveness of the non-aqueous treatments varied, but none were as effective as the aqueous treatments.
- TBAB alone is somewhat effective at reducing iron II ions, but does not inhibit their formation over time. Although Klucel G alone had little effect on iron II ions, MgO and Klucel G together significantly improved the performance of TBAB.

- Both Treatment M (TBAB spray/MgO/Klucel G) and Treatment N (TBAB spray/MgO/Klucel G/Line) show potential. Although they are not as effective at removing iron II ions as the washing or phytate treatments, for non-aqueous applications, they may be useful.
- The two complex inks (6 and 8) made from historic recipes performed the worst in all treatments. The LC recipe creates a relatively stable iron gall ink, while inks #6 and #8 are likely the worst case scenarios.

5.3.2 PH Testing

Cold extraction pH testing was performed for treatments on all three sample papers according to ASTM designation: D778-97 (re-approved 2002). The samples were macerated in distilled water with a glass stirring rod for one minute, then covered and allowed to stand at 25 degrees C (+/- 5) for one hour. Measurements were taken to 0.1pH unit twice with a pH meter calibrated with 4, 7, and 10 buffer solutions. Because sample material was limited, 0.1g of paper was tested in 7.0 ml of distilled water in keeping with the ratio from the standard. The samples were cut to include the same amount of all eight inks as opposed to measuring each individual ink and paper combination. This method measures the effect of the treatments on the papers as opposed to the differences among the inks.

Findings:

- Washing in water alone had little immediate effect on pH, but may have inhibited the rise in acidity after aging.
- Ethanol alone is not effective at increasing pH.
- Both phytate treatments had a slight improvement in pH after treatment and held fairly steady after aging.
- TBAB alone resulted in a slight increase or same pH after treatment, and a slight or moderate decline after aging.
- Both types of sizing, Klucel G and gelatin, improved the performance of both TBAB and phytate, respectively.

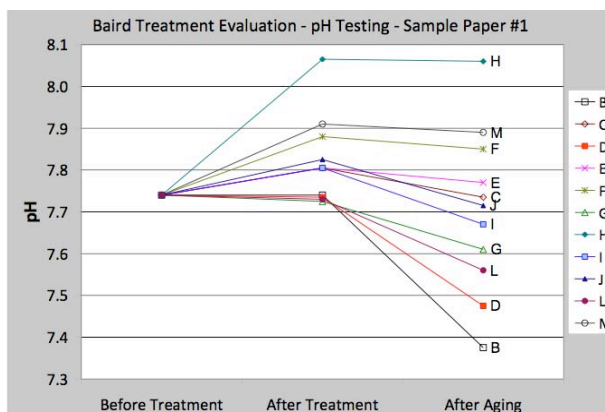


Figure 32. pH testing results, before treatment, after all treatments, and after post-treatment aging on paper sample #1.

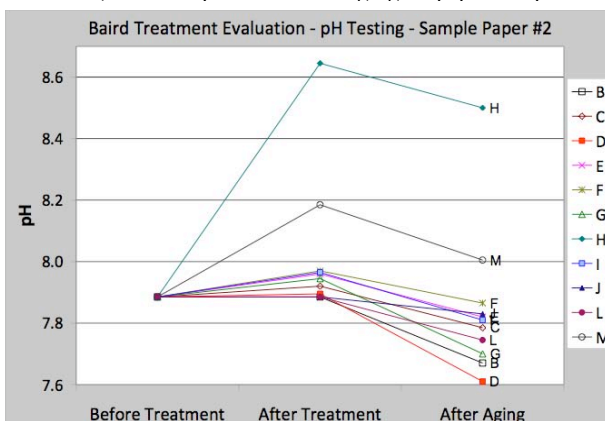


Figure 33. pH testing results, before treatment, after all treatments, and after post-treatment aging on paper sample #2.

- MgO significantly improved the performance of TBAB, but the immersion treatment in combination with MgO caused the pH to rise higher than the recommended limit of 8.0 for alkaline-sensitive iron gall ink. The spray treatment with MgO rose close to or slightly above this limit as well. It should be noted that these samples had a relatively high pH (between 7.7 and 7.9) before treatment. Most collections materials needing treatment would be much more acidic before treatment, so the increase in pH would likely be lower as well.

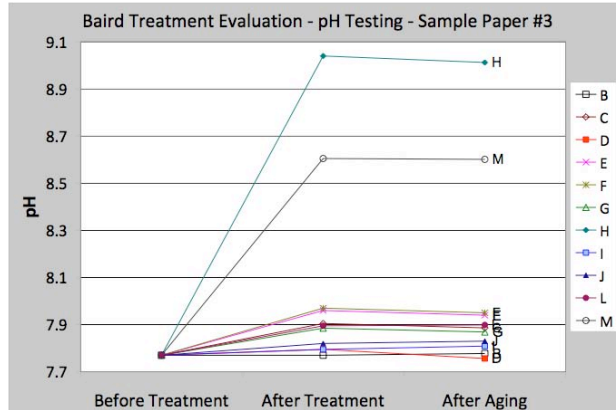


Figure 34. pH testing results, before treatment, after all treatments, and after post-treatment aging on paper sample #3.

- Combining both MgO and Klucel G with TBAB significantly improved the performance of the TBAB spray treatment. Treatment M (TBAB in EtOH spray / MgO / Klucel G) showed potential with a significant increase in pH after treatment that remained steady after aging, and did not reach dangerously high levels.

5.3.3 Mechanical Testing

Samples were tested for tensile strength before treatment and after post-treatment aging. Using tensiometers in climate-controlled cases, samples were subjected to increasing tension until their breaking point. Samples were pre-conditioned for twenty-four hours prior to testing. Only sample paper #1 was tested due to time limitations. Results are listed in the table below, and can be read as follows:

- An increase in strain (elongation) and an increase in force at breaking indicates an overall improvement in the properties of the samples when compared to the control groups, Treatments A or B.
- A loss of strain and a loss of force indicates ineffective reinforcement of the samples when compared to group A or a detrimental effect when compared to group B.
- An increase in strain and a loss of force indicates an increase in flexibility.



Figures 35-36. Tensiometers in climate controlled cases used for mechanical testing of paper samples.

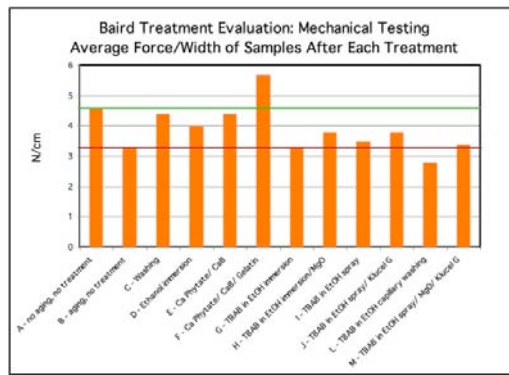
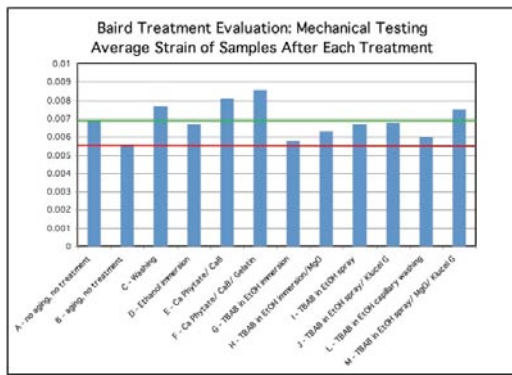
Treatment	Average Strain	Average Force/Width
A – Control - no aging, no treatment	0.0069	4.6
B – Control - aging, no treatment	0.0056	3.3
C - Washing	0.0077	4.4
D - Ethanol immersion	0.0067	4.0
E - Ca Phytate/ CaB	0.0081	4.4
F - Ca Phytate/ CaB/ Gelatin	0.0086	5.7
G - TBAB in EtOH immersion	0.0058	3.3
H - TBAB in EtOH immersion/MgO	0.0063	3.8
I - TBAB in EtOH spray	0.0067	3.5
J - TBAB in EtOH spray/ Klucel G	0.0068	3.8
L - TBAB in EtOH capillary washing	0.0060	2.8
M - TBAB in EtOH spray/ MgO/ Klucel G	0.0075	3.4

Figure 37. Results of mechanical testing. Average strain and force/width of paper samples after each treatment after discarding outliers and errors.

As expected, all aqueous treatments were beneficial, and Treatment F (Ca Phytate / CaB / Gelatin) was clearly the most beneficial. The gelatin had a significant enhancement of the properties of the samples, and it was the only treatment that had an increase in Force/W as compared to the un-aged control.

TBAB treatment alone was ineffective at strengthening the samples, but was somewhat beneficial when combined with de-acidification and re-sizing (Treatment M). Treatment M shows a significant increase in strain and slight increase over B in force/w, but does not restore the samples to their condition before aging. It is possible that TBAB has a detrimental effect when left in the paper (non-aqueous treatment does not wash it out), but that the addition of MgO and Klucel G mitigate those effects.

It should also be noted that Ink #6, one of the complex inks from an historic recipe, was significantly weaker than all other samples, before and after all treatments.



Figures 38-39. Results of mechanical testing. Average strain and average force/width of paper samples after each treatment after discarding outliers and errors. The green and red horizontal lines represent the control groups A and B.

5.3.4 Risks of Treatment

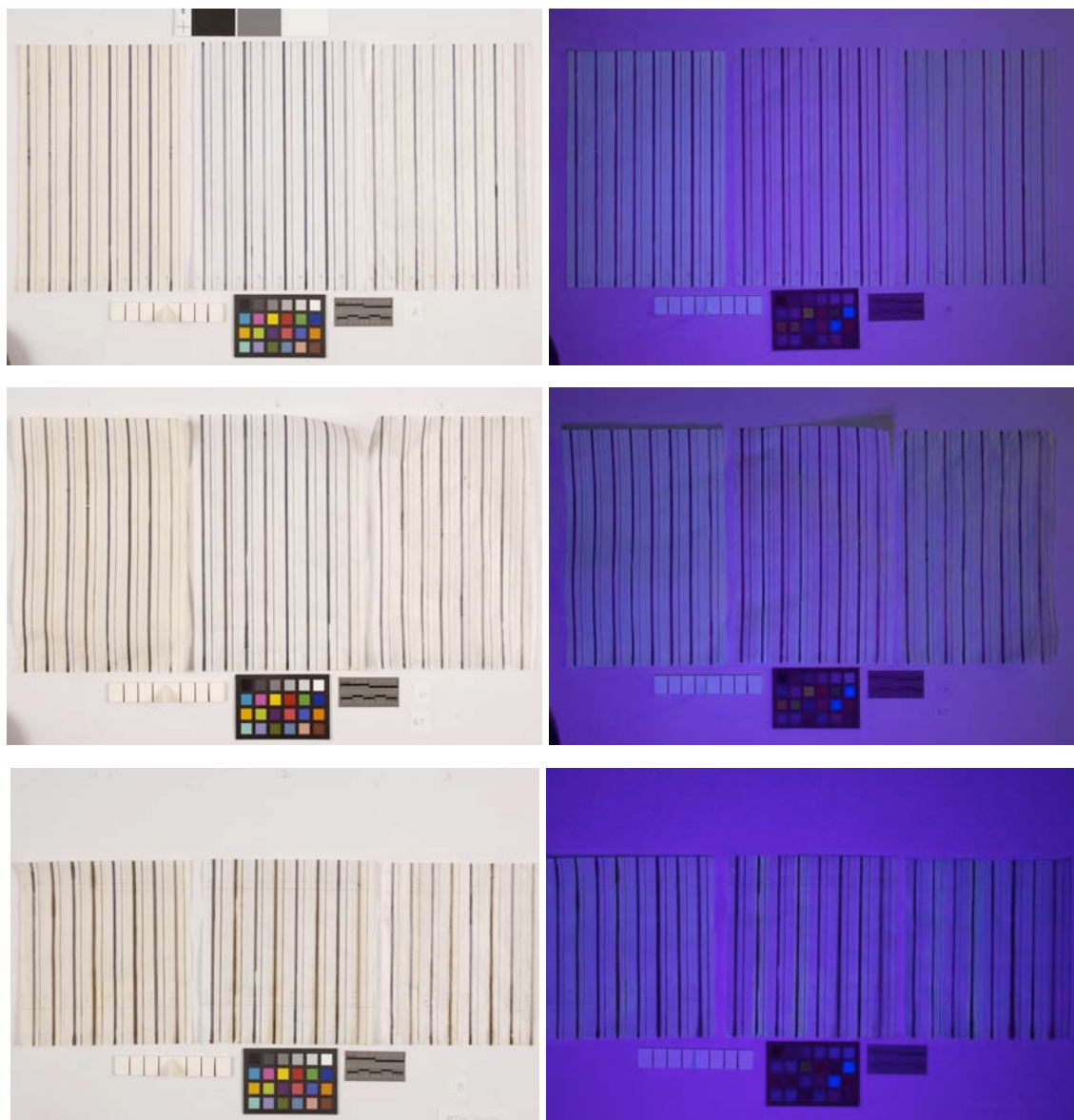
There were no noticeable solubility problems during any of the aqueous treatments, though there may have been some minor fading caused by the phytate baths. Immersion in ethanol caused many of the inks to solubilize slightly, and particles of ink lifted off. With the exception of the aniline ink, solubility problems were much less severe in the spray ethanol treatments than immersion. As expected, the aniline ink ran badly when covered with Mylar after being sprayed with ethanol. The solubility of the aniline ink was not as problematic during Treatment N, presumably because it was sprayed from the verso. Although solubility remains a concern for treatment with ethanol, even the most severe fading was very minor and would likely not have caused illegibility unless the text was extremely faint before treatment. It is also interesting to note that the only ink that did not exhibit any solubility problems was the control ink, the only ink in the study that is not a copying ink.

Although the samples are not noticeably sticky after treatment, stickiness during treatment and during drying is a significant concern. After all aqueous treatments, the samples stuck somewhat to the Hollytex drying supports. The application of Klucel G caused some samples to stick very slightly to the Hollytex supports. No loss occurred due to stickiness during the experimental treatments, as the samples were not extremely fragile, but it should be assumed that corroded ink in collections materials will be lost if it sticks to the drying support after treatment. Stickiness from the use of Klucel G in Treatment N can be avoided by spraying the verso only, because it is protected by the lining.

Non-aqueous paper splitting proved to be excessively risky for the object. It is extremely difficult to get the splitting started without causing unacceptable damage, because the copying paper is so thin and fragile. It was decided not to continue treatment because it would be time-consuming, and it is not practical to attempt by hand. Mechanized paper-splitting is no longer available commercially, so it is no longer a viable solution for the conservation of copying books.

5.3.5 Visual Changes and Observations

During the post-treatment aging of the samples, the inks that contain sugar developed much darker and larger brown halos around them than the others. All aqueous treatments completely removed this discoloration. TBAB alone was less effective at removing it, but in combination with Klucel G and MgO, it was slightly more effective. Ultraviolet examination of florescent haloing is consistent with visual examination of the brown haloing.



Figures 40-45. Samples before and after aging in normal and UV light. From left to right: before aging in normal light, before aging in UV light, after pre-treatment aging in normal light, after pre-treatment aging in UV light, after post-treatment aging with no treatment in normal light, and after post-treatment aging with no treatment in UV light.

All treatments except for the ethanol control prevented the aniline ink from discoloring from purplish brown to dark brown or nearly black after aging.

As expected, bath solutions in all aqueous treatments turned yellowish brown indicating the removal of dirt and/or degradation products, while non-aqueous treatment solutions did not.

The samples become much flatter and less cockled after aqueous treatments than ethanol treatments. While flatter is usually more desirable in paper conservation, the cockled samples actually appear more similar to collections materials after treatment, because

copying papers were distorted when originally copied, due to the damp copying process. This physical change caused by aqueous treatments is also indicative of dimensional stress, which should be avoided for corroded and embrittled papers.

5.3.6 Practicality and Health & Safety Considerations

In situ procedures proved to be efficient, fast, required less exposure to chemicals, and avoided dis-binding. The capillary washing treatment with ethanol led to the most exposure to solvents because it required nearly continuous spraying to avoid differential washing. TBAB spray treatments caused much less exposure than immersion or capillary washing. When the treatments are covered in the silicone-release Mylar sandwich during the spray treatment procedure, they remain saturated, so there is no need to spray repeatedly. Treatments M (TBAB in EtOH spray / MgO / Klucel G) and N (TBAB in EtOH spray / MgO / Klucel G / line) are particularly safe and efficient, as all solutions are sprayed at once, and they do not require multiple exposures.

5.4 Summary and Discussion of Findings

- The two complex inks (6 and 8) made from historic recipes performed the worst in all treatments. The LC recipe creates a relatively stable iron gall ink, while inks #6 and #8 are likely the worst case scenarios.
- During the post-treatment aging of the samples, it became clear that the inks that contain sugar developed much darker and larger brown halos around them than the others. This could indicate that the most severe deterioration in copying books is caused by the inclusion of sugar in the inks and/or papers. This observation is consistent with the correlation between poor condition and the sweet smell noted during the survey.
- As expected, aqueous treatments were effective at removing iron II ions, increasing pH, and restoring strength to the samples. The effectiveness of the non-aqueous treatments varied, but none were as effective as the aqueous treatments.
- Treatment with gelatin significantly improved the strength and pH of the samples.
- Although comparison with the ethanol control treatment shows that TBAB treatments do have a positive effect beyond the ethanol, TBAB alone did not cause significant improvement in the properties of the samples.
- It is possible that left over TBAB in the paper after treatment causes a detrimental effect that is counteracting the positive effect of the de-activation of the transition metal ions. There was concern for this problem before the project began because non-aqueous treatment does not rinse out excess treatment solution.

- MgO and Klucel G together significantly improve the performance of TBAB. Both Treatment M (TBAB spray / MgO / Klucel G) and Treatment N (TBAB spray / MgO / Klucel G / Line) show potential. Although they are not as effective as the phytate treatment, for non-aqueous applications, they may be useful.
- Although both types of sizing improved the performance of treatment, they also caused the papers to become stickier and may cause problems when drying. Adjusting the treatment procedure by spraying from the verso and drying by laying the object recto side up on silicone-release Mylar and covering with Hollytex and blotter or felt, may reduce or eliminate the problem.
- Solubility is a concern for both aqueous and non-aqueous treatment, so thorough testing should be an important component of any treatment protocol. Based on observation during treatment, it appeared that the only ink that had severe solubility problems in ethanol was the one containing aniline dye. Most others fared quite well. The spray procedure reduced solubility problems, especially when sprayed from the back.
- The spray procedure has several benefits. It allows in situ treatment to avoid dis-binding; it increases the safety of treatment by allowing the controlled application of solutions to one side of the object; and it is fast, efficient, and requires less exposure to chemicals than immersion treatment.
- MgO should be used cautiously, as it can increase the pH beyond the recommended limit of 8.0 for alkaline-sensitive iron gall ink.
- Paper-splitting is not a practical or safe option for the treatment of copying books. The thinness of the paper and its sensitivity to moisture make paper splitting risky. Paper-splitting is more commonly used for printed materials like brittle newspapers, which are not unique. If the damage is caused to copying book material by the splitting process, the replacement is not possible. The process is also too time-consuming to be considered a viable option unless it can be done by a mechanized process, and currently, this service is not offered commercially anywhere internationally.
- It is important to note that these treatments have been performed on prepared experimental samples only. Unpredicted side-effects are possible when treating collections materials. Cautious testing and treatment of collections materials should precede any large-scale conservation project.

6. Imaging and Digitization Exploration

6.1 Goals and Considerations

Copying books present quite a few challenges for digitization, stemming from the translucence of the paper, the bound format, faint and feathered ink, the fragile substrate, cockling and creasing, and the overwhelmingly large volume of material. The ultimate goal of this portion of the project was to develop a protocol that archives around the world can follow to gain excellent results using inexpensive and accessible equipment. Several imaging techniques were explored with the help of the Museum Conservation Institute, and a simple and practical set-up and procedure was found to give excellent results.

6.2 Results and Discussion

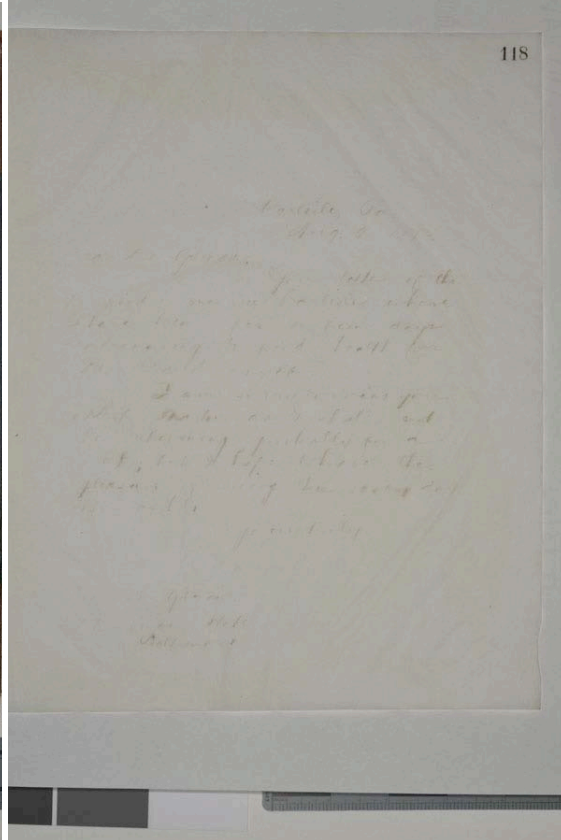
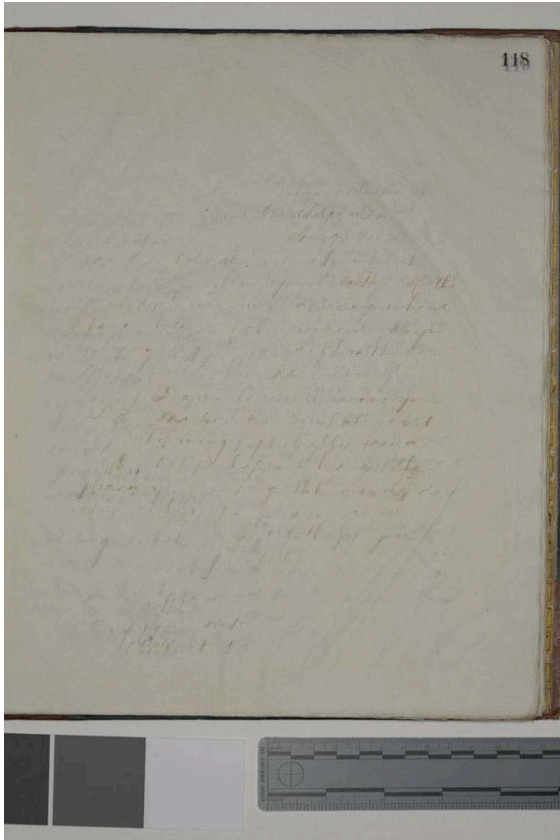
Experimentation was conducted using a Canon 5D Mark II digital camera on a copy stand with a Canon 100mm macro 2.8 lens, a Canon 50mm 1.4 lens, two Canon Speedlite 580 EX with diffusers, a Micro RingLite MR-14EX, and two Tiger UV lights.

After experimenting with several light sources and set-ups, it was found that most light sources provided unsatisfactory images. Transmitted light and traditional reflected light set-ups with one and two light sources exacerbated the problem by intensifying the distracting elements of the materials. Transmitted light highlighted discoloration from water staining and ink offset, while traditional set-ups enhanced the distracting shadows from surface texture and cockling. For non-faded text, ultraviolet (UV) light caused the writing to appear darker and blotchier, which reduced legibility. Infrared light produced a blank image.

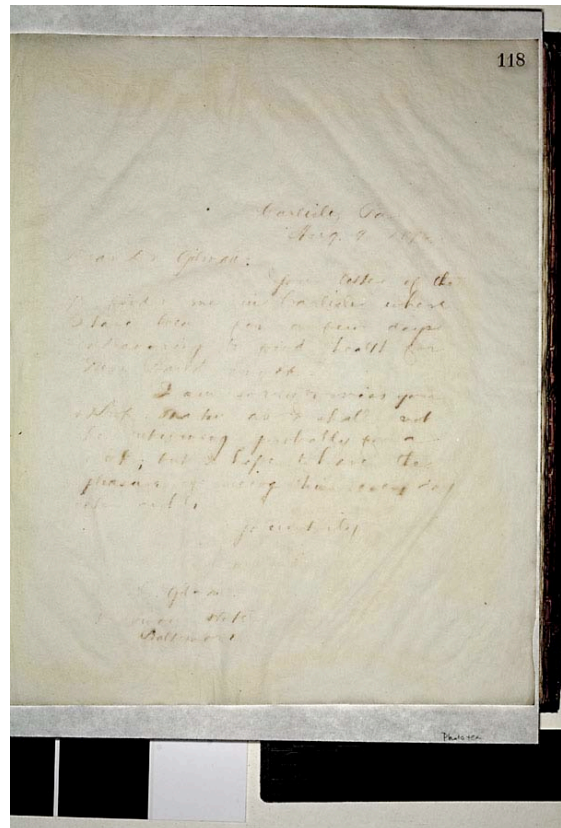
The best results were achieved using the following procedure. The book was placed on a copy stand on a book cradle that allowed one side to remain flat while elevating the facing side. An interleaving sheet of Phototex paper was placed directly under the leaf to be imaged in order to counteract the translucence of the sheet. Phototex was chosen because it contains no optical brighteners that would fluoresce under UV light and because it is very soft and safe to use with the fragile paper. Many archival papers would be appropriate to use, but distracting watermarks and optical brighteners should be avoided. A ring flash was attached to the lens of a digital camera. A ring flash is literally a ring of light that provides a very even light and projects directly down onto the object at a perpendicular angle. This light source eliminates the distraction in the image from surface textures and cockling (See figures 47, 48, and 49). For faint text, UV light was used instead of the ring flash.



Figure 46. Digitization set-up on a copy stand with a digital camera, ring flash, and interleaving paper.

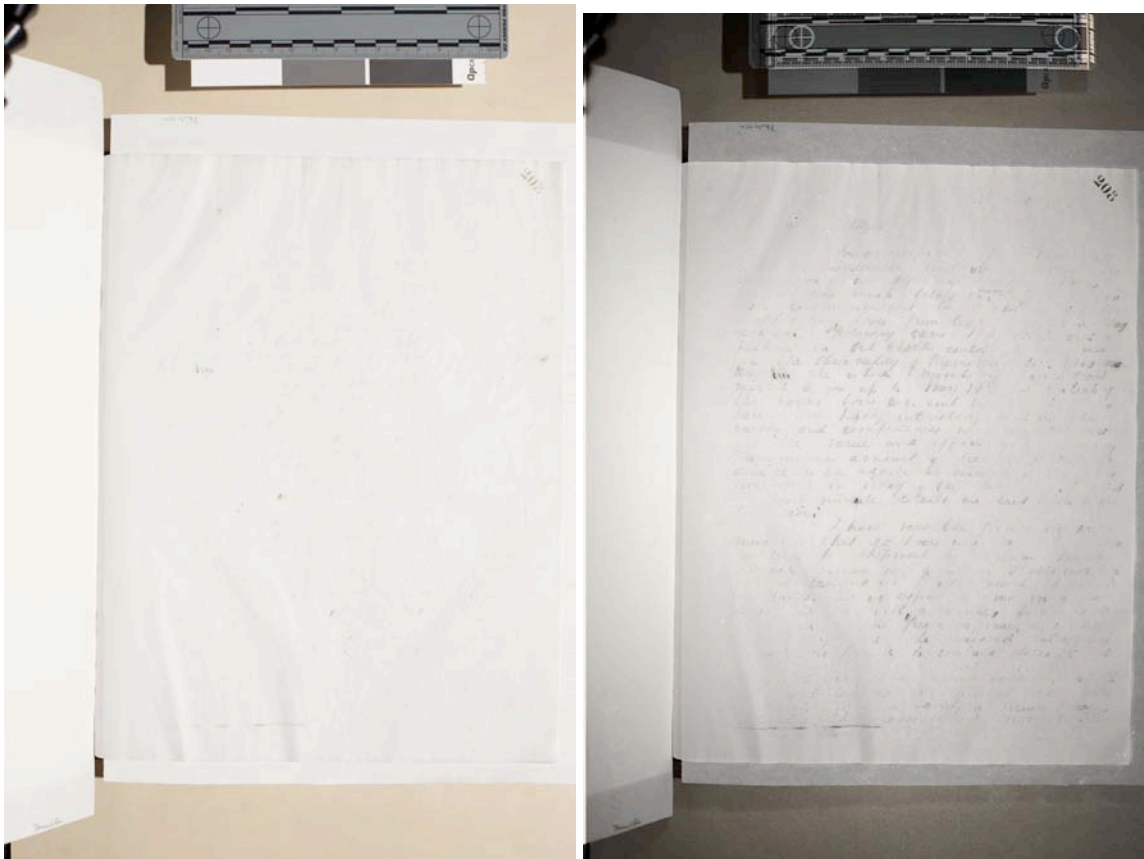


Figures 47-49. Baird copying book, volume 71, page 118. Example of Baird copying book digitization using interleaving, a ring flash, and image processing to increase legibility and decrease distraction from translucency, distortion, and surface details. Upper left, page photographed in reflected light with a ring flash and no interleaving and no image processing. Upper right, page photographed in reflected light using a ring flash with an interleaving sheet and no image processing. Lower right, page photographed in reflected light using a ring flash with an interleaving sheet and minimal image processing using Digital Photo Professional.

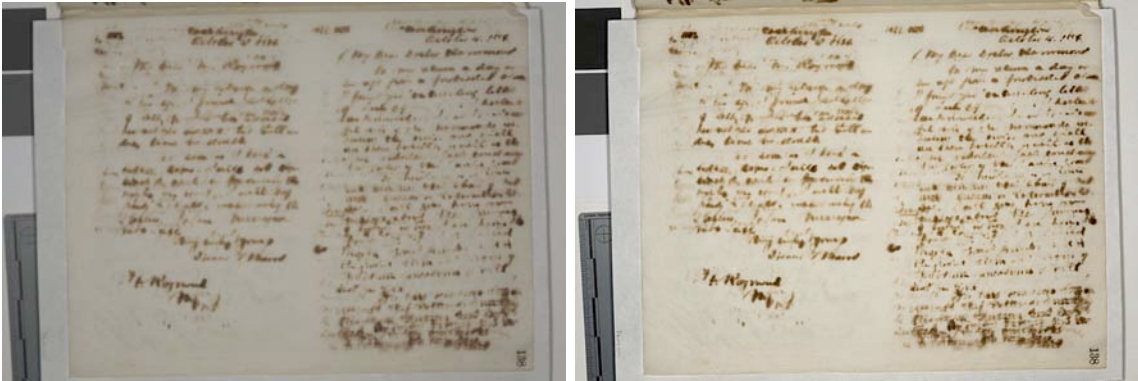


Most of the faint iron gall ink that is virtually invisible in normal light, became legible under UV (See figures 50 and 51). Simple image processing techniques such as compressing the histogram to enhance the brightness and contrast and un-sharp masking improved most of the images dramatically. Blurred and feathered text proved to be the most challenging problem, and only limited success was found using the image processing techniques (See figures 52 and 53). The most effective method of image processing involved the conversion of the images from RGB to CIE Lab color space (This can be done in Photoshop by selecting the Image menu and changing the Mode to “Lab color.”) By working only with the lightness, all of the information is retained, but the image is cleaner without the distraction of color (The lightness appears black and white, but actually contains more information than a black and white version of an image.) The contrast and brightness was increased in Curves by adjusting the mid-tones only, leaving the lightest lights and the darkest darks alone. This non-destructive technique offers the best result without losing information in the image.

This set-up is quite affordable and quick to use. Most ring flashes cost between \$400 and \$600, but some models may be available for as little as \$100. Although it was not attempted, it is possible that a blue LED light would give the same results as UV, but without the potential harmful effects on the photographer.



Figures 50-51. Baird copying book, volume 9, page 208. Example of Baird copying book digitization using UV light and image processing to enhance faint text. Left, page imaged in normal light with no image processing. Right, page imaged in UV light with minimal processing using Digital Photo Professional, manufactured by Canon.



Figures 52 and 53. Baird copying book volume 15, page 138. Example of Baird copying book digitization using image processing to increase the legibility of blurred and feathered text.

7. Conclusions

The complex composition of copying book papers and inks complicate the problem of iron gall ink corrosion by enabling the rapid migration of sulfuric acid and transition metal ions. The large quantity of materials and their moisture sensitivity limit conservation treatment options to moderately effective non-aqueous anti-oxidant treatments or mechanical stabilization. Considering the complexity and fragility of the materials and the difficulties of conservation, digitization and proper storage should be very high priorities in any preservation plan concerning copying books.

Although the TBAB treatments were found to be less effective at reinforcing the samples as the aqueous phytate treatments, especially the one with gelatin, most do show some improvement over the untreated, aged samples. All forms of evaluation agree that the best TBAB treatment is Treatment M, the spray procedure with MgO and Klucel G, and that the TBAB treatment needs the help of de-acidification and resizing to have a significantly positive effect. The possibility that the leftover TBAB in the paper has a detrimental effect is cause for concern, but the inclusion of MgO and Klucel G seems to mitigate any negative effect.

Experimentation with in situ procedures was fruitful, as the spray procedure was found to have several benefits. It allows in situ treatment to avoid dis-binding; it increases the safety of treatment by allowing the controlled application of solutions to one side of the object; and it is fast, efficient, and requires less exposure to chemicals than immersion treatment.

Overall, the experimentation of non-aqueous treatment with TBAB led to a step in the right direction, but not a solution to the problem of the conservation of letterpress copying books. As often occurs in research, it also raises many more questions. The clear benefits of aqueous treatment and gelatin pique interest in the testing of anti-oxidant solutions with gelatin in various mixtures of ethanol and water. Perhaps an 80:20

ethanol/water solution would provide the benefits of aqueous treatment while avoiding its problems. The questions would be:

- Would a small percentage of water in the treatment solution provide the benefits of washing:
 - Effectively wash out the water-soluble transition metal ions?¹
 - Rinse out excess anti-oxidant?
 - Wash out dirt and degradation products?
 - Enable the strengthening of paper fibers through hydrogen bonding?
- Would contact with the water in the treatment solution cause the ink to crack?
- Would the water in the treatment solution cause the paper to stick to drying supports after treatment?
- Can gelatin be effectively delivered to the paper fibers and ink through a mixed solution?

During the course of this study, two alkyylimidazolium bromides have been shown to be more effective than TBAB for the non-aqueous treatment of ink corrosion, 1-ethyl-3-methylimidazolium bromide [EMIMBr] and 1-butyl-2,3-dimethyl-imidazolium bromide [BDMIMBr] (Kolar et al. 2008). It would be very useful to test these anti-oxidants on copying book samples using in-situ procedures and as described above.

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¹ Eusman found that even a small amount of water in an ethanol/water washing solution resulted in the diffusion of some iron II ions into the bath solution, but less than with 100% water bath (Eusman 2002, 124.)

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9. Appendix

9.1 Appendix A: List of possible components in copying papers and inks.

Note: This list is not comprehensive. It was compiled from Rhodes 1999, Cleveland 2000, Dorning 2000, James 2000, the Ink Corrosion Website, and from recipes found in various scientific journals and encyclopedia.

Ink ingredients:

Tannins

Galls (crushed, fermented, oak galls, gall nuts, oak apples, Aleppo galls, Japanese galls, Chinese galls, Turkey galls, Levant galls, oak marble galls, blue galls, green galls, white galls, acorn galls)

Tannic acid

Gallic acid

Gallotannic acid

Pyrogallic acid

Pomegranate bark

Pomegranate peel

Sumach plant

Oak-bark

Tormentilli root

Chestnut wood or bark

Walnut husks

Hemlock

Pine bark

Hawthorn bark

Astringent vegetable material

Aqueous myrobalous

Mirobalan extract

Iron sources: metal salts and contaminants

Ferrous sulphate (green vitriol, iron II sulphate, copperas, cyprate vitriol, sal martis, sulphate of iron, vitrolum hungaricum, atramentum)

Perchloride of iron (ferric chloride)

Iron filings

Copper sulphate (blue vitriol, Roman vitriol, chalcantum)

Zinc sulphate

Aluminum sulphate (rock alum, Cyprus alum)

Potassium chromate (neutral chromate of potash)

Potassium dichromate

Potassium aluminum sulphate (alum, roach alum)

Potassium hydroxide (caustic potassa)

Sodium hydroxide (caustic soda, lye)

Crystallized carbonate of soda (crystal soda, sodium carbonate)

Ammonium oxalate
Ammonia alum
Ferric chloride
Ammonium chloride (sal ammoniac)
Uranium acetate

Hygroscopics and Binders

Potassium carbonate (potash, subcarbonate of potash, pearlash)

Soluble gum

Gum Arabic

Gum Tragacanth

Gum Kino

Gum Senegal

Glycerine

Glucose

Sugar

Brown sugar

Syrup

Honey

Molasses

Funori

Treacle

Varnish

Isinglass

Glue

Animal glue

Rock candy

Marshmallow root

Mucilage

Spanish licorice

Solazza juice

Deliquescent salt

Ammonium nitrate

Ammonium chloride

Magnesium chloride

Colorants

Potassium permanganate

Logwood extract (Campeachy wood)

Plus alum to produce purple colorant

Plus potassium chromate or dichromate to produce black colorant

Silver nitrate

Lamp black

Indigo

Indigo sulphate (chemic blue, soluble indigo)

Indigo carmine

Prussian blue (soluble blue, ferric ferrocyanide)

Aniline dyes

 methyl violet

 nigrosine black

 eosine red

 aniline green

Iodine green

Malachite green

Verdigris

Bengal green

Bismarck brown

Magenta

Mercuric chloride (perchloride of mercury)

Acids (to prevent early oxidation of ferrous ion)

 Vinegar (Acetic acid)

 Wood vinegar

 Hydrochloric acid (Muriatic acid)

 Sulphuric acid

 Salicylic acid

 Oxalic acid

 Citric acid

 Picric acid

 Tartaric acid

 Sulphindigotic acid

Alkaline agents (to retard the degradation from ink corrosion)

 Gypsum (calcium sulphate)

 Calcium carbonate

 Lime water (water and calcium hydroxide)

 Calcium chloride

 Calcined oyster shells

 French chalk

 Borax

Preservatives (to prevent mold, etc.)

 Carbolic acid (creosote, phenol)

 Cloves

 Cinnamon

 Anise

Liquids

 Any standard writing ink

 Water (distilled, rain, spring)

 Rose water

Beer
Wine
Brandy
Sherry

***Paper Additives:** materials used to “fix,” mordant,” or “attract” soluble colorants or to retard degradation from ink corrosion*

Iron sources: metal salts and contaminants

Ferrous sulphate
Copper acetate
Aluminum acetate
Ammonium nitrate
Sodium arsenite
Potassium bromide
Potassium iodide
Potassium carbonate
Calcium chloride
Magnesium silicate
Sodium chloride
Potassium acetate

Mordants

Tannins
Potassium chromate (mordant for logwood ink)
Potassium dichromate (mordant for logwood ink)
Prussiate of potash (potassium ferrocyanide) (Prussian blue if iron gall applied)
Prussic acid (hydrogen cyanide) (Prussian blue if iron gall applied)
Sulpho-cyanide of potassium

Hygroscopics and Binders

Glycerin
Sugar
Honey
Soluble gums
Gelatin
Ammonium chloride (deliquescent salt)
Magnesium chloride (deliquescent salt)
Varnish
Wax
Starch
Dextrin

Preservatives and Alkaline materials to retard the degradation from ink corrosion

Clay
French chalk

Oyster shells
Borax
Formalin
Chloride of Lime

9.2 Appendix B: Experimental Ink Recipes

1. Control ink: traditional writing iron gall ink

- Tannin (Tannic acid), 4.92g
- Ferrous sulfate, 4.2g
- Gum Arabic, 3.14g
- Water, 100ml

Recipe from Library of Congress conservation department.

2. Iron gall copying ink with sugar

- Common Iron Gall Ink (LC recipe), 40ml
- Sugar, 20ml syrup, or "until slightly glossy when dry."

Recipe modified from "Mackenzie's Copy Ink, 1829," in Cleveland 2000, p. 25.

3. Iron gall copying ink with glycerine

- Evaporate 10 volumes of ordinary ink (LC Recipe) to 6 volumes, 100ml
- Add 4 volumes of glycerine, 40ml

Recipe and procedure modified from "Allfield's Copying Ink," In Lehner's, *The Manufacture of Ink*, 1892.

4. Iron gall copying ink with Prussian blue

- Common Iron Gall Ink (LC recipe), 40ml
- Sugar, 20 ml syrup
- Prussian Blue 1g

Recipe modified from "Mackenzie's Copy Ink, 1829," in Cleveland 2000, p. 25.

5. Iron gall copying ink with Aniline dye

- Evaporate 10 volumes of ordinary ink (LC Recipe) to 6 volumes, 100ml
- Add 4 volumes of glycerine, 40ml
- Add 0.33 volumes of Aniline, 3.3ml (1.3 g)

Recipe and procedure modified from "Allfield's Copying Ink," in Lehner's, *The Manufacture of Ink*, 1892.

6. Complex Iron gall copying ink with copper and other contaminants

- Gall nuts, 10g in 60ml water
- Logwood, 2.5g in 20ml water
- Ferrous Sulfate, 10g in 10ml water
- Copper Sulfate, 0.46g in 10ml water
- Glycerin, 20 ml (Substituted for Marsh Mallow Root)
- Gum Arabic, 5g in 10ml water
- Sugar, 5.1g in 10ml water

- Heat all ingredients except gum Arabic and sugar for one hour without boiling.
- Mix well and let sit 6 hours.
- Add gum Arabic and sugar and let sit 6 hours.
- Stir and decant into bottles.

Recipe and procedure from: "Monsieur Bowy de Praegny's Copy Ink," *Manuels Complet du Marchand Papetier*, Paris, 1854 in Cleveland 2000, p. 26

7. Iron gall copying ink with deliquescent salt

- LC iron gall ink 40 ml
- Ammonium Chloride 4g in a few drops H₂O
- Sugar 3g in a few drops H₂O
- Glycerine 10 ml

Mix all ingredients.

8. Logwood bi-chromate

- Logwood, coarsely broken extract 14.175 g
- Alum 4 g
- Distilled water 118.3 ml
- Glycerine 14.77 ml
- Potassium Chromate 0.4875 g
- Gum Arabic, pulverized 4 g
- Ferrous Sulfate 8 g

Heat water, logwood, and soda (sub alum) until deep red and all dissolved. Stir in glycerine, chromate of potash (dissolved in a little water), and gum Arabic (dissolved in a little hot water).

Recipe and procedure modified from "A New Copying Ink," *Scientific American*, Vol. 20, p. 308, 5/15/1869 and "Adolph Ott's Copy Ink," in Cleveland 2000, p.26.

9.3 Appendix C: Experimental Treatment Procedures

Treatment #A: Control – No Aging – No Treatment

Procedure: None

Treatment #B: Control – Aging – No Treatment

Procedure: None

Treatment #C: Control – Aging – Washing

Procedure:

- Use Hollytex support.
- Spray out with re-calcified water.
- Immerse in 3 consecutive 20 minute re-calcified water baths.
- Agitate bath every 5 minutes.
- Let drain on Hollytex and blotter 1 minute.
- Dry between felts and Hollytex under weight.

Treatment #D: Control – Aging – EtOH immersion

Procedure:

- Use PeCap washing screen support.
- Spray out with 100% ethanol.
- Immerse in ethanol bath 20 minutes.
- Let drain on Hollytex and blotter 1 minute.
- Dry between felts and Hollytex under weight.

Treatment #E: Phytate / CaB

Procedure: Modified Ink Corrosion Website procedure:

- Use Hollytex support.
- Spray out with re-calcified water,
- 5 minute initial re-calcified water bath,
- 5 minute second re-calcified water bath,
- 20 minute Ca Phytate bath,
- 10 minute final re-calcified water bath,
- 20 minute CaB bath.
- No sizing.
- Let drain on Hollytex and blotter 1 minute.
- Dry between felts and Hollytex under weight.

Treatment #F: Phytate / CaB / Gelatin

Procedure: Modified Ink Corrosion Website procedure:

- Use Hollytex support.
- Spray out with re-calcified water,
- 5 minute initial re-calcified water bath,
- 5 minute second re-calcified water bath,
- 20 minute Ca Phytate bath,
- 10 minute final re-calcified water bath,

- 20 minute CaB bath,
- Pre-dry between felts until damp, not dry,
- Spray gelatin sizing (spray out to slightly dampen with water if needed),
- Drain on blotter until damp,
- Dry between felts and Hollytex under weight.

Treatment #G: TBAB in EtOH immersion

- Procedure:*
- Use Hollytex and washing screen.
 - Spray out with EtOH.
 - Immerse 20 minutes in 0.3 mol/L TBAB in EtOH.
 - Let drain on Hollytex and blotter 1 minute.
 - Dry between felts and Hollytex under weight.

Treatment #H: TBAB in EtOH immersion / MgO

- Procedure:*
- Use Hollytex and washing screen support.
 - Spray out with EtOH
 - Immerse 20 minutes in 0.3 mol/L TBAB in EtOH
 - Let drain on Hollytex and blotter 1 minute.
 - Spray with Bookkeeper – one even pass.
 - Dry between felts and Hollytex under weight.

Treatment #I: TBAB in EtOH spray

- Procedure:*
- Spray with 0.3 mol/L TBAB in EtOH on hollytex and blotter in silicone-release Mylar sandwich, keep saturated 20 minutes.
 - Peel back sil-rel Mylar
 - Spray, cover, and surround with snake weights.
 - Dry between felts and Hollytex under weight.

Treatment #J: TBAB in EtOH / Size / spray

- Procedure:*
- Spray with 0.3 mol/L TBAB in EtOH on hollytex and blotter in silicone release Mylar sandwich, keep saturated 20 minutes.
 - Peel back sil-rel Mylar
 - Spray, cover, and surround with snake weights.
 - Spray on size: Klucel G 1% in EtOH enough to saturate.
 - Let air dry few minutes.
 - Dry between felts and Hollytex under weight.

Treatment #K: TBAB in EtOH / MgO / suction table – Treatment cancelled because table not working

- Procedure:*
- Spray with 0.3 mol/L TBAB in EtOH on hollytex and thin blotter on suction table, keep saturated 20 minutes.
 - Spray with Bookkeeper – one even pass.
 - Dry between felts and Hollytex under weight.

Treatment #L: TBAB in EtOH / capillary washing

- Procedure:*
- Wash on slant board with 0.3 mol/L TBAB in EtOH on Hollytex and

Tek-wipe with reservoir of solution at top, 20 minutes. Cover area with polypropylene sheeting to keep surface from drying during washing.

- Dry between felts and Hollytex under weight.
- See notes for procedure problems and variations.

Treatment #M: TBAB in EtOH / MgO / Size / spray

- Procedure:*
- Spray with 0.3 mol/L TBAB in EtOH on hollytex and blotter in silicone release Mylar sandwich, keep saturated 20 minutes.
 - Peel back sil-rel Mylar
 - Spray TBAB in EtOH
 - Spray with Bookkeeper – one even pass.
 - Spray on size: Klucel G 1% in EtOH.
 - Cover, and surround with snake weights.
 - Let air dry few minutes.
 - Dry between felts and Hollytex under weight.

Treatment #N: TBAB in EtOH / MgO / Size / Line / spray

- Procedure:*
- Spray with 0.3 mol/L TBAB in EtOH on hollytex and blotter in silicone-release Mylar sandwich, keep saturated 20 minutes.
 - Set up Mylar, blotter, Hollytex, and lining tissue
 - Spray out objects with TBAB in EtOH on verso on Hollytex
 - Spray out lining tissue with Klucel G 2% in EtOH.
 - Place object verso down onto lining tissue, lift off Hollytex
 - Spray TBAB in EtOH again
 - Spray with Bookkeeper – one even pass.
 - Cover with sil-rel Mylar
 - Bone down, dwell 20 minutes.
 - Lift object off of Hollytex on silicone release Mylar, and place onto fresh Hollytex and felt.
 - Dry between felts and Hollytex under weight.

Treatment #O: Non-aqueous Paper Splitting – Treatment abandoned because time-consuming and not practical by hand.

- Procedure:*
- Face with heat-set tissue.
 - Split.
 - Adhere core with Filmoplast Gudy-O.
 - Remove facings with ethanol spray on Hollytex and blotter.
 - Dry between felts and Hollytex under weight.