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Magnesium Revisited

ABSTRACT

A recent project undertaken at the Library of Congress (LC) and reported at the AIC conference in Washington in 2003 was interesting in more ways than originally intended. Inspired by the conference theme of "Treatment Revisited," we embarked on a project focused on examination of objects executed in iron-gall ink and treated with various deacidification solutions over the years. It is surprisingly uncommon to examine objects treated in the past as part of our mechanism of evaluation and we wondered what we might glean from such an exercise. Magnesium bicarbonate as a deacidification agent has been used at the Library for decades and its use would seem to have been challenged by the results of recent research. Additionally, iron-gall ink has been the subject of intense study in the past few years. Although a multi-year research project designed to gather objective data on various treatments options used in treating iron-gall ink is currently underway at the Library, we wondered what might be revealed by a concurrent, more subjective project looking at objects which had undergone deacidification with magnesium bicarbonate. Acknowledging that the judgments resulting from visual examination of objects treated in the past would be subjective, it was generally felt that given the pervasive use of subjective knowledge in our profession, the exercise had some validity.

While various conclusions and opinions could be drawn from this exercise, some of the tangential issues that arose are equally worth noting. A brief history of the evolution of the Library's standard treatment of objects executed in iron-gall ink will be articulated. This process was particularly interesting since the exercise led to a greater appreciation of the procedures that are currently employed and put them in a context that is not always apparent if one is unfamiliar with how they were arrived at. In order to fairly assess our present methods it is necessary to understand their evolution out of decades of common experiences, exhaustive discussions, and scientific inquiry. The complexity and small size of our field makes definitive and/or objective data hard to come by and fuller understanding of any given issue can only be gained through familiarity with a broad cross-section of the literature and related practical experience. Secondly, the results of our examination of the objects previously treated with magnesium bicarbonate will be presented. Finally, some of the unexpected results that arose during the course of the study and what they reveal about how we evaluate our work will be discussed.

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HISTORY

The treatment of iron-gall ink has posed complicated challenges for conservators and conservation scientists. The variety of iron-containing ink formulas and the dramatic impact those formulations can have on properties, the ink's self-destructive nature which compels us to "do something," and the complexity of its chemistry, making scientific research that is applicable to treatment difficult to design, has left us with an impressive amount of conflicting information. The Library of Congress, with millions of pages penned in iron-gall ink and a strong mandate for public access, has been interested and participatory in the development of appropriate preservation strategies. Three distinct eras of treatment during the twentieth century can be defined at the Library.

The first era from 1900 into the 1940s was dominated by the practice of silking. Throughout this period, thousands of documents were silked every year although treatment details on individual objects do not exist. The practice of silking as described by the chief practitioner at the Library, William Berwick, began with immersion into a warm water bath to clean the manuscript of grime and pressing between newspapers to flatten. In a second, sep-

arate step, both sides of the document were pasted out with cooked flour paste and the silk was placed on top and smoothed out. The object was then placed between two sheets of paraffin paper and pressed to flatten between smooth, white, unglazed pulp board.

It is difficult to match the variations in the silking materials and techniques with an object's condition today as there are no individual records. Paste recipes varied, as did the silk itself in quality and gauge. Silked objects today are in various states of preservation. Silked documents general display a low surface pH, between 3.5 and 4. Some early recipes for the starch paste include alum and some silked items do test positive for alum. Most silked documents have darkened significantly and become brittle. Sometimes the silk itself is in an advanced state of deterioration with the document holding the silk together rather than the other way around! In other cases, although yellowed, stiff, and unnaturally flat, the document remains relatively intact. While the technique represented the best knowledge of the day and required considerable skill to execute, the alum and embrittling nature of the silk proved to be its undoing. Additionally, many of the inks reacted badly to the treatment—sinking, smearing, and bleeding—and the paste acted as a poultice, pulling elements of the ink into the paste and silk. The silk texture was impressed into the paper due to the excess pressure exerted. The silk interferes with the legibility of the writing, a quality that becomes more noticeable with time as the paste and paper yellow. These factors, along with better understanding of the nature of paper aging and new ideas about how to treat them, led to the decline of silking.

The second era of treatment of iron-gall ink documents is dominated by the practice of lamination. Lamination involved sandwiching an artifact between cellulose acetate film and semi-transparent paper and running it though a hot press to melt the layers together. In 1948, the annual report from the Manuscript Division states that over thirty-five thousand documents were laminated. The era of mass treatment had arrived at the Library. It is easy to see the managerial appeal of this type of operation. Lamination was seen as far superior to silking and a method that received considerable attention from the leading figures in the manuscript restoration field at the time. Its superiority was demonstrated by its speed, reduced specialization of labor, and cost.

The early laminations did not include a deacidification step, and the objects frequently turned brown after the process. This browning was thought to be a result of accelerated aging due to the tremendous heat involved with the process. The browning phenomena led William Barrow to incorporate a deacidification step. Although he was not the "discoverer" of the role acid plays in paper deterioration, he was unparalleled in disseminating this information through the promotion of his laminating technique. The first method he recommended was the "Barrow Two-Step" that involved successive twenty-minute baths in concentrated calcium hyroxide, followed by concentrated calcium bicarbonate. This treatment was replaced by the "Barrow One-Step," immersion into saturated magnesium bicarbonate. The quantity of buffering salts introduced into the papers by these methods is unknown but certainly adverse reactions of iron-gall ink to what is now seen as extremely high pH is something we have all encountered in our work. The high pH of the baths and the resultant high pH of the papers are almost certainly to blame for the familiar changes to iron-gall inks such as reddening, fading, and/or sinking. Despite the managerial appeal of the laminating practice, it became increasingly acknowledged that the difficulties associated with this approach outweighed the benefits. Unfavorable reactions of the media to the concentrated deacidification solutions were noted and began to be taken more seriously. The concept of "individual treatment" began to gain stature as conservation took its early steps towards professionalization in the 1950s and '60s.

The third era in treatment of iron-gall ink began with the establishment of the Conservation Office in 1967 and the appointment of Peter Waters as the first "Restoration Officer" in 1971. One of his early accomplishments was convincing the Library to halt the practice of lamination. Additionally, deacidification and the treatment of iron-gall ink were issues that received considerable early attention. Many conservators working in the Library at the time reported problems with deacidification in general and aqueous treatment of iron-gall ink, such as sinking and physical breakage of ink-corroded areas. Use of the concentrated deacidification solutions frequently caused a change in color and/or intensity of the inks and left a gritty deposit on the surface of the paper. Mr. Waters formed a deacidification committee of staff conservators and scientists. One of the first actions he took as head of the committee was to direct Norvel Jones to conduct a survey of conservators at the Library on practices they used. He hoped to record the various methods employed, define areas of needed research, and establish standard practices within the laboratory. The survey revealed that many conservators were diluting the deacidification solutions by at least half to alleviate the gritting problem. It was also noted that some did not use magnesium or calcium salts at all in their treatment of iron-gall ink due to experiences with unwanted effects. As a result of the work of the deacidification committee and Margaret Hey's research as a visiting scientist at LC studying deacidification and iron-gall ink treatments, Mr. Waters developed and distributed standard guidelines to the staff. These guidelines acknowledge and support individual experience and judgment but direct that if a book or other item is washed, it should be followed by deacidification. Magnesium bicarbonate is mentioned as

the agent of choice and conservators were directed to dilute it as necessary to avoid unwanted effects. Two important principles are implicit in these guidelines that remain relevant today. The first is that whenever possible, deacidification should follow washing—a concept which, while it has fallen from favor in practice, remains firmly supported by science. The second important principle spelled out in these guidelines is that the visual integrity of the object is paramount. This principle, coupled with the abandonment of lamination, articulated a shift in thinking away from one whose primary focus was preserving information to the current focus which places far greater emphasis on preserving what we normally think of as aesthetic or original qualities of the material.

After the issuing of the laboratory standards, treatment of iron-gall ink objects underwent many ingenious modifications in the late seventies and throughout the eighties. While washing and deacidification proceeded along the directives of Margaret Hey's recommendations, many objects could not be treated in this manner. Work with various non-aqueous methods of deacidification continued to be an active area of research but is beyond the scope of this paper. What is perhaps most prevalent and effective has been the development of the technique of treating water-sensitive inks through the "dilution" of the aqueous washing and deacidification solutions with ethanol, which forms the basis of how we treat iron-gall ink today. While acknowledging that the efficacy of the cleansing and deacidification operations could be proportionally decreased with the increase of ethanol percentage, conservators still felt that the improvement resulting from the intervention validated these techniques. On the other hand, a generally accepted opinion that the addition of alcohol to a washing bath would actually increase the efficacy of bathing was supported by Margaret Hey's research. Additionally, work was published which supported the superiority of aqueous deacidification over non-aqueous methods. Empirically, conservators arrived at methods of washing and deacidification that would accommodate the medium's unique sensitivities while attaining maximum cleansing and deposition of alkaline material into the paper. Typically, conservators would test an object with 25%, 50% , 65%, and 100% water and deacidification solutions while arriving at a treatment recommendation. These alcohol modifications were in use from at least the 1980s and firmly established by the early nineties. In 1995, Heather Wanser studied these various modifications and demonstrated that the effectiveness of cleansing and the deposition of salts by even a 65% ethanol bath were significant. The study results revealed that up to 0.5% alkaline salts are deposited into the paper by this treatment. While this amount of alkaline salt deposition was short of the 2% alkaline reserve recommended by earlier publications as the level required to ensure "permanence," the trade-off in media stability was seen as well worth it.

If what grew out of this evolution of techniques for treating deteriorated iron-gall ink documents from the establishment of the Office in 1967 until very recently could be summarized as a standard approach at the Library, perhaps it may be articulated as follows:

1. As part of the standard examination process, the need for treatment is assessed. The level of ink deterioration is considered, as is the paper's state of preservation. At the Library, we are faced with a multitude of objects which have been treated with materials now known to contribute to their deterioration, and thus the impetus to retreat may be greater in this case. Objects that have not been previously treated and appear in a good state of preservation are unlikely to undergo any treatment other than housing improvement. Additionally, a great deal of information pertaining to a document's reactivity to aqueous treatment may be obtained by assessing its reaction to either the silking or the deacidification process which was part of most of the lamination treatments. For example, if the inks on a silked document appear to be relatively intact, it is likely that they will hold up well to aqueous treatment. Conservators at the Library of Congress have developed a testing protocol that we feel is reasonably predictive of an object's behavior during treatment.
2. Ideally, if the object will permit it, iron-gall ink documents are treated with a twenty-minute recalcified deionized water bath followed by a twenty-minute magnesium bicarbonate bath. The magnesium bicarbonate is diluted from a saturated solution to a 25% solution by volume. There are many variations, including length and number of baths and whether or not the object is dried between baths.
3. If testing or experience has indicated that the inks may change with this treatment, the washing and deacidification baths may be diluted up to 65% ethanol/35% water or deacidification component.
4. After aqueous treatment, resizing with gelatin or methyl cellulose is commonly performed and is seen as beneficial and protective on a number of levels.
5. If testing or experience indicates that the inks should not undergo aqueous treatment, a non-aqueous deacidification treatment is considered. Bookkeeper is most commonly used today.
6. If the object cannot be chemically stabilized, support is obtained through housing solutions. When an object is Mylar-encapsulated, a buffered sheet is inserted into the package.

The subject of treating iron-gall ink has garnered considerable renewed attention in recent years. Since the mid-to-late nineties no less that four international conferences have been held on the subject. New information about the

degradation of iron-gall ink has been published indicating the role of the iron 2+ compound (FE II) in prompting the oxidation process.

This work inspired the development of two decidedly new approaches to treatment. Washing objects in water near the boiling point removes most of the FE II and has been reported to have been used successfully. More promising, perhaps, is a treatment proposed by Han Neevel at ICN (Netherlands Institute for Cultural Heritage) which would sequester any free FE II permanently through the use of phytates. Conservators at the Library were both excited by this second approach and alarmed by some of the results that were reported in the study. Neevel's experimental results included papers deacidified with saturated solutions of magnesium bicarbonate that yellowed significantly upon aging, though effectively protected from acid hydrolysis and oxidation. Yellowing of paper associated with magnesium compounds has been previously reported but is countered by other research, and these conflicting results can often be attributed to the study's protocols. Studies using concentrated solutions tend to produce negative results and conflicting information is produced by differing aging methods. Neevel used saturated solutions in his experiments and employed a cycling temperature and relative humidity aging protocol. Additionally, the ink recipe employed by Neevel exaggerated the proportion of FE II in historic recipes in order to study the effects of the phytate on highly corrosive ink. While acknowledging the limitations of our current practices using magnesium compounds to treat iron-gall ink, in that they do nothing to prevent FE II from continuing to form, we were nevertheless reluctant to abandon our practices and the thirty years of accumulated experience for something that is unproven in practice. As a result, the Library of Congress is currently engaged in a multi-year study aimed at gathering data that would allow us to assess ICN's work within the context of our current and past practices.

Among the changes in experimental protocol adopted are three important ones. Because Library conservators have not used saturated solutions for thirty years, the more diluted solutions are being substituted. In addition, recently adopted ASTM (American Society for Testing and Materials) standards for artificial aging are being used instead of the cycling method. Finally, real ink samples will be tested along with the exaggerated ink formula for comparison. Early results of the Library's study tend to support the results obtained by Neevel, but as is typical of studies involving iron-gall ink, raise more questions than they answer. While a full report is still a year or two off, we were inspired by this year's AIC annual meeting theme of "Treatment Revisited" to perform a parallel study that would look at actual objects treated in the past.

RESULTS OF THE PROJECT

While well versed in the dangers associated with aqueous treatment of iron-gall ink, many conservators past and present had been able to perform safely the "standard" Library of Congress treatments previously described. I personally have treated scores of iron-gall ink documents through washing and deacidification with magnesium bicarbonate and felt that with proper pre-testing and consideration the problems with ink changes could be avoided. On several occasions there was the opportunity to "test" the treatment on part of the document, compare it with the untreated part, and allow for more confidence in proceeding. The significant body of positive experiences in treating iron-gall ink was difficult to reconcile with the accumulating evidence of possible harm that might result. After all, if paper yellowing or dramatic ink changes were readily apparent, development of the Library's standard treatment would have developed in a different direction. Perhaps these changes occur only upon aging. To begin our reexamination of treatments performed in the past, all of the treatment, photographic, and some administrative records accumulated since the establishment of the Office were combed, and iron-gall ink objects that were treated with magnesium compounds were noted. The records that had treatment details and photographic information associated with the treatment that reasonably could be used to make some type of assessment were brought to the lab. All of the conservators were invited to visually examine the objects and discuss their perceptions. Unfortunately, documentation such as colorimetry or spectrophotometry that would provide a mechanism for an objective assessment does not exist. Nevertheless it was decided to proceed with the available information, reasoning that while fallible and notoriously variant, a conservator's visual perception and aesthetic sense are a valuable part of the preservation discipline and will remain so.

What was found through this exercise was surprising on a number of levels. The first surprise was that relatively few objects penned in iron-gall ink received aqueous treatment in the thirty years since the establishment of the Office. Perhaps the reason for this is that an iron-gall ink document has to pass a gauntlet of tests before it is considered for aqueous treatment and some conservators have felt that treatment with magnesium bicarbonate is too risky to be considered. The second surprising result was that those objects that did pass the various tests and were treated through the standard approach described previously have held up well, mechanically as well as visually. Examination of the available documentation that includes written information, color slides, and color transparencies supported our visual perceptions. Of the several hundred pages examined, we could find no obvious paper color shift associated with the magnesium treatments. Any alteration

to inks that had been noted in the reports were subtle and it was difficult to determine if the inks had actually changed in color or our perception was influenced by the color balance shift in the paper tone. While acknowledging the limitations of this exercise, the results obtained supported the notion that the careful testing and open exchange of information amongst ourselves had been effective in avoiding many of the negative effects associated with aqueous and magnesium bicarbonate treatment of iron-gall ink documents.

UNEXPECTED RESULTS

While definitive and objective answers to the questions that prompted this study were not found, some of the tangential details revealed warrant discussion. If conservators are to have meaningful discussions about previous treatments, the following factors need to be taken into consideration.

Conservators remember every excruciating detail of a treatment they judged went wrong but do not remember the ones that went well. While "self-flagellation" is a well-known, and possibly beneficial, character trait of conservators, the weight given to negative experiences was disproportionate to the far more common positive experiences. Does this built-in "prejudice" serve us well? Could it, in part, explain the reluctance of conservators today to incorporate deacidification into their practice despite their admission that it is beneficial to the paper? After all, if done correctly, we do not see any change. What we do remember are objects that have behaved badly or reports of others who have had negative experiences. While "negative experiences" is obviously an enormously important factor to consider when designing a treatment protocol, is it the only one? Certainly this prejudice impedes the formation of a balanced program as well as colors the sometimes decidedly emotional aspect of our discussions.

The notion of "acceptable change" in manuscript conservation is true in theory only. Much is made of the differing standards between fine art and manuscript conservation. It was found that among LC conservators past and present that the only acceptable change in the visual appearance of iron-gall ink is "no change." This concept has driven development of the standard treatment protocol principle, overriding the desire to address deteriorating components by invasive treatments, and has been in direct conflict with information coming from the scientific community about the long-term benefits of treatments that deposit alkaline salts into the paper.

We need to find some practical way to incorporate objective standards in our measurements and more fully understand and acknowledge that our visual perceptions are subjective. Conservators are critical and have a highly developed visual sense. This quality is obviously one of the things that sets us apart from much of the rest of the world and is a vital component in our work. Unfortunately, what any given individual conservator "sees" and makes judgments about can be quite at odds with his or her colleagues. Without objective standards, we are forced to accommodate these differences in our practice.

It became obvious during the course of this project that these qualities may be seen as a source of both strength and weakness. The following two examples encountered during this study highlight this point.

When looking for objects for this project, several people mentioned one of the Library's "Top Treasures," James Madison's "Notes on the Continental Congress" as an example of a deacidification treatment of iron-gall ink gone awry. There are two volumes; the first had been washed and deacidified. Lab legend had it that the inks had changed so dramatically in response to the magnesium bicarbonate deacidification that the treatment was halted halfway. In fact, it was generally felt that one could tell just where treatment had stopped by looking through the papers. We started by looking at the volume that had been treated. We immediately began ascribing qualities that we normally associate with washing and deacidification such as blurred, reddened, and sinking inks. Then we turned to the volume that had not been treated. To our surprise, the inks looked very similar. There were inks that were blurred and some that were just as reddened in the untreated volume as there were in the treated one. In short, the range of ink appearance in the treated volume looked very similar to the ink appearance in the volumes that had not been treated. There were no details about the treatments other than that the papers were "washed and buffered." Very curious about exactly what had been done during the treatment, we had the volumes tested by scanning electron microscope (SEM) analysis. The manuscripts had indeed been washed, displaying the typical spectra of water-washed objects. To our complete surprise, however, no magnesium or other alkaline salt could be detected by this method. Although the results are not definitive—more research would have to be performed to clear the mystery of exactly how the first volume of the treasure was treated—it is most interesting to note the mythology that had surrounded this object and the weight given to this "myth" when conservators were considering treatment options for other objects.

The second example illustrating the difficulty conservators encounter when assessing their work may be found with Heather Wanser's research. In this study, one expendable iron-gall ink document was cut up and each piece subjected to various alcohol-modified washing and magnesium bicarbonate deacidification treatments. Twelve senior conservators were then asked to visually evaluate the results. The judgments of the highly-trained, visually-

acute conservators could be charitably described as varied. The ink in one sample was judged as having undergone no visible change by some and displaying the most drastic visual change by others. Interestingly, the iron-gall ink in the control sample was also judged as having been treated by some. This experience is not uncommon. We aim at objectivity but our visual perceptions are imbued with subjectivity.

To conclude, this study veered in a different direction than the one in which we originally set out. It was evident from this exercise that support for both the magnesium and the anti-magnesium arguments could be found by selective examination of the published literature and practical experience on the subject. Another benefit of the research was a renewed appreciation of the dramatic evolution in treatment over the past one-hundred years. Most interesting was being able to trace the emergence of "original" appearance as an ultimate goal in document conservation. Scientific evaluations of chemical stability and how to achieve them have been fully integrated into a conservator's planning and decision-making but these considerations do not override aesthetic judgments. Many successful modifications of the standard washing and deacidification treatments at LC have been developed to try to get the best of both worlds. Effective treatments have been carried out, and to the extent that we can judge, have held up well. New research informed by a conservator's real life experiences impact research design with the promise of more targeted results. The dynamics of how we assess our work deserves wider acknowledgement to ensure that better preservation standards evolve. Finally, a conservator must balance the information available at any given time with the mission of the institution and make the best decision; it will inevitably involve a compromise.

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