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In Search of a Remedy: History of Treating Iron-Gall Ink at the Library of Congress

ABSTRACT

As a prominent player in the early field of preservation, the Library of Congress has been on the forefront of many major trends aimed at stabilizing paper documents, as well as those ravaged by corrosive iron-gall ink. At first the focus was on physical support for damaged documents. By the mid-nineteenth century, a diversity of transparent materials were used in the United States and Europe to reinforce iron-gall ink manuscripts without significantly obscuring the text. Tissue paper appears to have been the first material applied as an overall support and was adapted at the Library in early 1899. However, later that same year the Library transitioned into the silking process, a new technology, to arrest the increasing deterioration of its eighteenth- and nineteenth-century manuscripts.

By 1940 William Barrow convinced Library officials that cellulose acetate lamination was the method of choice for supporting weak documents, including iron-gall ink manuscripts. Also around this time Barrow shifted his focus to the acidity which undermines iron-gall ink and paper alike. He was responsible for an innovation that involved deacidifying documents prior to cellulose acetate lamination, with consecutive immersions in calcium hydroxide followed by calcium bicarbonate. This method was practiced by the Library of Congress.

In compensation for the risks of this highly alkaline treatment and in the interest of efficiency, Barrow promoted the use of single immersion in magnesium bicarbonate in the 1960s. While the scientific community extolled the benefits of deacidification in general, Library conservators experienced an array of deleterious side effects from treatment with saturated magnesium bicarbonate. Consequently, for the past three decades they have modified magnesium bicarbonate with various dilutions of deionized water and/or ethanol. Non-aqueous deacidification methods, such as methylmagnesium carbonate and Bookkeeper, have been selectively applied to treat especially water-soluble iron-gall ink, as well.

The Conservation Division staff became greatly interested in the promise of the anti-oxidant calcium phytate treatment proposed by Han Neevel and Birgit Reissland in the late 1990s. Currently the Library is testing and comparing the two-step calcium phytate/calcium bicarbonate technique to iron-gall ink treatments practiced by staff conservators in recent times. Meanwhile, the Library is interested in coordinating its resources with relevant iron-gall ink research underway in other laboratories. This paper reviews the institution's research initiatives and treatment approaches from the perspective of the Library of Congress' two-hundred-year history. The study does not include research done by other institutions that nonetheless has influenced Library staff, or address the complex problems of retreatment of iron-gall ink materials.

From the vantage of the Library of Congress's two-hundred-year record, we are afforded a window on the changing aesthetic, technical, and philosophical approaches to manuscript restoration and conservation. As a prominent player in the early field of preservation, the Library has been on the forefront of many major trends aimed at stabilizing paper documents, as well as those ravaged by corrosive iron-gall ink.

The Library did not staff its own "restorers" until 1967. Instead, in 1897, restoration technicians from the Government Printing Office were detailed on site to mend and repair Library collections. At first the focus was on physical support for iron-gall ink damaged materials. Repairs and patches with hand-made and machine-made papers, of medium to tissue weight, were a common early

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remedy for the Library's manuscript collections. To remove cockles, manuscripts were simply moistened and flattened in a screw press.

By the mid-nineteenth century, a diversity of transparent materials were used in the United States and Europe to reinforce iron-gall ink manuscripts without significantly obscuring the text. Transparent paper appears to have been the first material applied as an overall, sandwich-style, support and was adapted by the Library in early 1899 (Marwick 1964).

However, later that same year, the Library of Congress transitioned to the silking process, a new technology publicized by the Vatican to arrest the increasing deterioration of its eighteenth- and nineteenth-century manuscript collections. Silk's greater transparency likely stimulated this transition. Unfortunately, some silked documents were observed to show deterioration within seventeen to thirty years. The presence of alum in the Library's silking paste recipe, recorded in 1913, probably was a contributing factor in the degradation of silked collection materials (Fitzpatrick 1913). It is widely accepted that, at certain concentrations, alum catalyzes the deterioration of iron-gall ink, paper, and silk, potentially rendering manuscripts extremely acidic, brittle, and unusually discolored over time while contributing to the occurrence of ink "strike through."[1]

Descriptions from the same 1913 source tell us that in preparation for silking, manuscripts would have been flattened by dampening between moist sheets of newspaper, then placed between smooth pulp boards in a letterpress. Those which were stained may have been washed by immersion in warm water, blotted between towels, and flattened as previously described. Based on conservators' observations, the consequences of these treatments for iron-gall ink manuscripts include excessive overall flatness, often accompanied by embossing from the silk texture. The results of aqueous-based treatment, in some but not all cases, has been seen to include bleeding, sinking, and changed color or intensity of the ink.

As the deficiencies of silking became evident, the Library continued to search for solutions that might be more cost effective and durable. In the context of the period, silking of manuscripts was time consuming and required highly skilled labor. In 1928 the Library of Congress and the National Bureau of Standards (NBS) experimented with cellulose acetate applied as a dip coating or spray, and with cellophane as a laminating film (Gear 1965).

The modern age of plastics had arrived. By 1946, these experiments and the persuasive entreaties of William Barrow convinced Library officials that cellulose acetate lamination was the method of choice for supporting weak documents, including iron-gall ink manuscripts. Perhaps the most treasured Library document treated in this manner is the Rough Draft of the Declaration of Independence, written in iron-gall ink in Thomas Jefferson's hand. It previously had been silked in the early twentieth century. Degradation of the silking had progressed to the point that, while displayed on the *Freedom Train*, the silking split along a horizontal crease, probably due to stress on the embrittled silk from fluctuating environmental conditions. The *Freedom Train* was a seven-car locomotive which toured the country from 1947 to 1950, carrying a patriotic exhibit featuring a number of important American documents such as the Mayflower Compact, Lincoln's Gettysburg Address, and the Bill of Rights (U.S. National Archives 1950). As a consequence of damage occurring on the *Freedom Train*, the Rough Draft was "desilked" in a water bath, immersed in calcium bicarbonate, cellulose acetate laminated, and then returned to the *Freedom Train* in 1947 (Stiber 1997).

Typically, items were laminated by sandwiching a document between two sheets of cellulose acetate film, and this package was subsequently sandwiched between two sheets of tissue; the five-sheet composite was passed through a roller press, which was heated to 315ºF–320ºF (180ºC–190ºC) and exerted pressures of over seven hundred pounds per square inch, according to Barrow's own estimate (Barrow 1955; Stiber 1998). Predictably, Library-laminated iron-gall ink documents are uncharacteristically flat and inflexible compared to eighteenth- and nineteenth-century paper. Details of handwriting are veiled by the laminating tissue.

Around this time, Barrow shifted his focus to the acidity which undermines iron-gall ink, and paper generally. In the early 1940s, he and NBS scientist, B. Scribner, were responsible for an innovation that involved the "alkalization" of documents prior to cellulose acetate lamination. "Alkalization" had the dual effect of lessening the yellowing of documents when heated during lamination, Barrow's principle concern, and the more important effect of slowing the rate of deterioration of the paper documents. Although the relationship between the acid content of the cellulose acetate and the destabilizing effects of acid on the film did not appear to be understood by Barrow at the time, his "alkalization" step did mitigate acid-induced deterioration from the laminating film as well.

"Alkalization" involved an aqueous treatment (known as the "Barrow Two-Step" or "Two-Shot") with consecutive, twenty-minute immersions in saturated calcium hydroxide,[2] followed by calcium bicarbonate (Kelly 1972).[3] The treated manuscripts were air dried. For subsequent flattening, every tenth or fifteenth sheet was dampened with a sponge and gently weighted in a stack for one or more hours. About six sheets were removed from the stack at a time, and placed between blotters or chip boards in a screw press. Use of saturated calcium hydroxide often caused deposits of calcium carbonate on the surface of treated manuscripts and exposed treated materials to pH values as high as 12.5. As a result of the Barrow protocol, which was employed at the Library, some iron-gall inks inevitably changed color or intensity (Waters 1973). In compensation for the potential risks of this treatment and to minimize the time required for "alkalization," Barrow promoted the use of saturated magnesium bicarbonate in the mid-1960s, known as the "Barrow One-Step" or "One-Shot" (W. J. Barrow Research Laboratory 1964).[4]

The sixties marked a turning point for the Library, as well. In 1967, the Preservation Office was established with a research and testing laboratory and its own staff of paper and book "restorers." Conservation theory and practice underwent close scrutiny by its own preservation team. Peter Waters was appointed Preservation Officer in 1971. Notably, immersion in "super saturated magnesium bicarbonate" is common practice in conservation treatment reports from this time.[5] In a January 1973 memo, Waters explains to the Research Officer, Dr. John C. Williams: "Calcium hydroxide in combination with calcium bicarbonate which has been used in this Library for a number of years and as general practice in other libraries and archives where the Barrow lamination technique is practiced, has been observed by us to often change the color density of some writing inks from a deep tone to a lighter one or from brown to orange. No doubt this is due to the high pH which is achieved by the hydroxide solution. Because we must lessen the possibility of tonal, color, or other changes likely to occur as a result of a treatment we have replaced the hydroxide solution with a warm water wash. Of course, water washing is followed by calcium or magnesium bicarbonate immersion to buffer the paper" (Waters 1973). His description of the writing ink that changes from brown to orange as a result of highly alkaline water-based treatment is entirely consistent with conservators' observations of some iron-gall ink.

While the scientific community extolled the benefits of deacidification in general, a number of Library conservators experienced an array of deleterious side effects from treatment with saturated magnesium bicarbonate,[6] including some of the same alterations to iron-gall ink observed with the "Barrow Two-Step." Similar to the surface precipitation of calcium carbonate resulting from the use of calcium hydroxide, deposit of magnesium carbonate crystals on manuscripts was a problem. Regarding this phenomena, which staff conservators referred to as "gritting," Waters speculated that one cause was the concentration of the magnesium bicarbonate. He suggested that diluting the solution to "half saturated strength" might be appropriate for some deacidification treatments, but cautioned that: "We do not have specific guidelines for selection of an aqueous technique where one is indicated. I have taken the view that we need to find out a great deal more about the requirements of and reactions to treatment of the LC material before we formalize standards" (Waters 1974). Deacidification treatments using even "half saturated strength" magnesium bicarbonate, such as that given select pages of Thomas Jefferson's 1776, multi-page manuscript, Notes on the Proceedings of Congress, Containing the Text of the Declaration of Independence, are testament to the degree of change iron-gall ink could potentially undergo.[7] The iron-gall ink on the pages treated in this manner is orangish brown and diminished in intensity when compared to untreated portions of the manuscript (figs. 1–2).

Options for treating especially water-soluble iron-gall ink were expanded when methylmagnesium carbonate, a non-aqueous deacidification system, was patented by Library chemist George Kelly in the mid-1970s. In 1974 the Library's Research Office began to confront some of the many practical issues and theoretical questions arising from the myriad deacidification treatments available. In support of this program, Waters organized a Restoration Office Deacidification Committee, "to insure that the right questions are asked" with respect to conservation practices (Waters 1974). Conservators were concerned about how much alkaline reserve was desirable for various materials and how to evaluate adequate alkaline reserve after treatment. Further queries revolved around whether aqueous versus non-aqueous deacidification techniques were ideal, and how best to apply them. Appropriate treatments for iron-gall ink entered into the discussion. The Deacidification Committee chair, Norvell Jones, succinctly summarized the sentiments of the conservators: "We are at a stage where every advance in knowledge raises more questions. The water will probably seem much muddier before it clears" (Jones 1976).

One outcome of this collaborative period between the Library's scientific and conservation staff was a set of guidelines from Waters to deacidify iron-gall ink manuscripts with magnesium bicarbonate only, not calcium hydroxide,[8] unless recommended by Waters himself. Conservators were advised to reduce the concentration of magnesium bicarbonate so that no precipitation occurred. The strength of the solution might have to vary from one paper to another. The prevention of "gritting" would override concern for the degree of alkaline reserve achieved (Waters 1977).

It was also in the early seventies that Waters persuaded the Library to abandon lamination in favor of polyester film encapsulation to provide physical support for weak and damaged manuscripts. He developed a non-aqueous adhesive formula for "heat-set mending tissue" using Rhoplex ethyl acrylate/methyl acrylate copolymer, as well. This was especially helpful in repairing water-soluble iron-gall ink manuscripts.

In 1976, the Library set out in earnest to tackle the treatment of iron-gall ink and contracted Margaret Hey to research deacidification and stabilization of this medium.

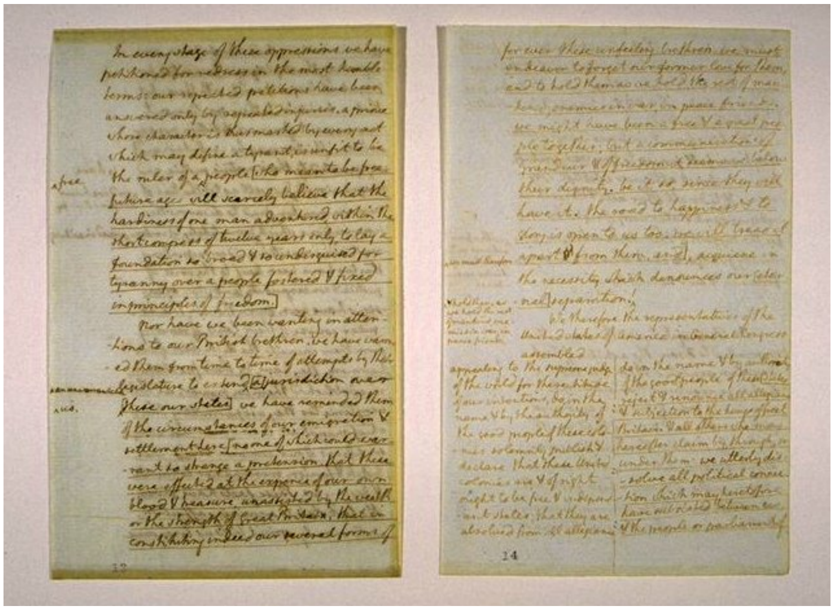

Fig. 1. Notes on the Proceedings of Congress, containing the text of the Declaration of Independence, written in iron-gall ink by Thomas Jefferson, 1776. P.11 untreated (on left); p.12 immersed in deionized water followed by saturated magnesium bicarbonate diluted 50% with water (on right).

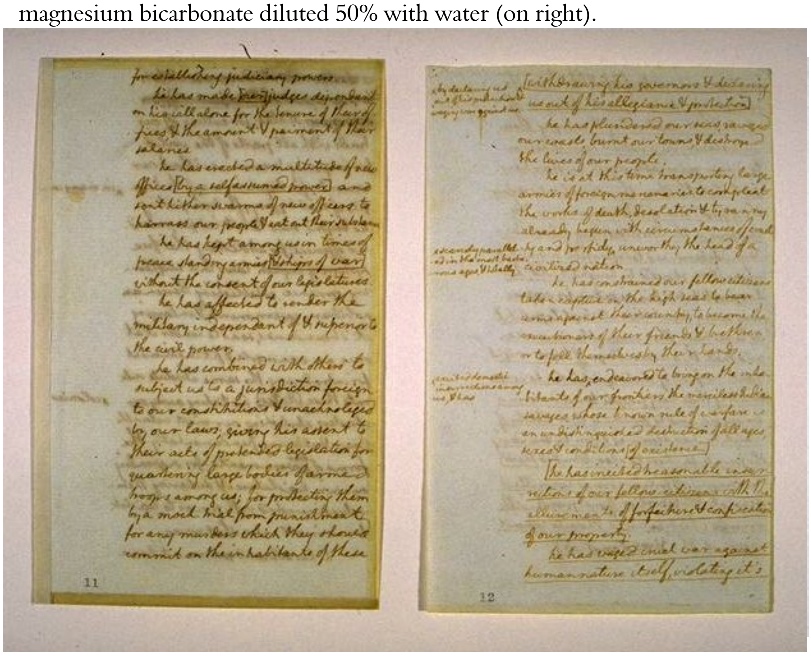

Fig. 2. Notes on the Proceedings of Congress, containing the text of the Declaration of Independence, written in iron-gall ink by Thomas Jefferson, 1776. P.13 untreated (on left); p.14 immersed in deionized water followed by saturated magnesium bicarbonate diluted 50% with water (on right).

Her experiment was designed to investigate the effect of alkaline compounds on sulfuric acid and ferric oxides in paper, and not the consequences for iron-gallotannate. The study samples were made of cellulose pulp containing different concentrations of iron oxides.[9] They were immersed in 4% sodium borate; "1/2 saturated calcium hydroxide;" magnesium bicarbonate (concentration not specified but presumed to be saturated); and methylmagnesium carbonate "dissolved in Freon plus methanol." Hey concluded that the higher the ratio of calcium or magnesium carbonate to iron, the greater the protection conferred on the cellulose. Understandably, the sodium borate performed poorly. She recommended washing well in water prior to deacidification (Hey 1981–82).

By 1979, Library chemist Lucia Tang and conservator Norvell Jones demonstrated that washing with distilled or deionized water, which was retreated by passing it through calcium carbonate chips, was more beneficial to paper longevity than washing with non-calcinated distilled or deionized water (Tang and Jones 1979). Immersion in "calcinated" deionized water has since become a standard washing procedure and pre-deacidification treatment at the Library of Congress, and indeed, in the United States and elsewhere.[10]

Library interns Lois Price and Diane van der Reyden studied the effectiveness of various application techniques for methylmagnesium carbonate, also in 1979. They discovered that an approximately 1% alkaline reserve, by then considered an adequate amount, was deposited by both spraying and immersion methods (van der Reyden and Price 1979). Because methylmagnesium carbonate is mixed with methanol and Freon, both fast-evaporating solvents, it is possible to inadvertently deposit uneven quantities of alkaline reserve in treated paper. This potential has continued to raise concern about resulting differential aging, though it has not been demonstrated experimentally.

Treatment records of the 1980s reveal that "1/2 saturated magnesium bicarbonate" diluted with water was the aqueous deacidification method most commonly employed for non-water-soluble iron-gall ink manuscripts. By the middle of the decade washing alone, or pre-washing prior to deacidification, with ethanol-water mixtures was introduced as well. In a 1991 presentation to an American Institute for Conservation/Book and Paper Group deacidification panel, conservator Terry Boone summarized the alkalization treatments in use at the Library of Congress then. Calcium hydroxide was added to wash water to modify its pH, saturated magnesium bicarbonate was diluted with 75% to 85% water, and methylmagnesium carbonate was spray or brush applied when an aqueous treatment was not indicated. She noted that magnesium bicarbonate was observed to cause a "warmer, more reddish-orange color to some iron-gall ink after treatment."

In the early 1990s, Dr. Chandru Shahani and Frank Hengemihle, of the Library's Research and Testing Division, investigated the relative efficiency of aqueous versus non-aqueous deacidification methods to extend the life of paper. The research concluded that even low alkaline deposits delivered in an aqueous solution were more effective than eleven times as much alkaline reserve delivered non-aqueously, based on fold endurance performance after humid oven aging (Shahani and Hengemihle 1991. These data would inspire conservator Heather Wanser's 1996 study of various treatments for iron-gall ink, including magnesium bicarbonate diluted with ethanol (Wanser 1996). The research indicated that, with respect to total alkaline reserve achieved, papers[11] immersed in a fully aqueous solution containing 25% saturated magnesium bicarbonate performed similarly to ethanol-diluted solutions also containing 25% saturated magnesium bicarbonate (these mixtures were comprised of 50% and 65% ethanol, respectively). On average, the fully aqueous magnesium bicarbonate treatment resulted in depositing approximately 0.6% alkaline reserve, while the 50% and 65% ethanol-diluted solutions deposited approximately 0.7% and 0.55%, respectively. A second phase of the study compared the effect of fully aqueous and ethanol-diluted solutions of magnesium bicarbonate on six iron-gall ink documents dating from the eighteenth and nineteenth centuries. A 70% ethanol-diluted magnesium bicarbonate solution performed best, causing no visible alteration to any of the six iron-gall ink manuscripts. It is noteworthy that two fully aqueous treatments of 100% and 25% saturated magnesium bicarbonate caused loss of intensity and color change in the ink of four of the six documents. Though not based on a statistically valid sampling, the test results suggested that the addition of ethanol to magnesium bicarbonate could precipitate an adequate alkaline reserve and preserve the visual appearance of aged iron-gall ink.

Bookkeeper non-aqueous deacidification technology was introduced at the Library in 1995 and since 1997 has been applied to selected iron-gall ink manuscripts, among other materials. Bookkeeper contains sub-micron sized particles of magnesium oxide dispersed in perfluoroalkane and, at the time of its introduction, a surfactant of perfluoropolyether derivative. When sprayed, the sub-micron particles of magnesium oxide become lodged in paper. The theory is that ambient moisture reacts with the particles to form magnesium hydroxide. In a 1998 study of this non-aqueous system, conservators Terry Boone, Lynn Kidder, and Susan Russick found that spray application achieved uniform and adequate alkaline reserve (Boon, Kidder, and Russick 1998).

Over time, the concept of acceptable change and appreciation for the consequences of treatment have evolved, influenced by scientific research, empirical observation, and aesthetic taste. Consequently, for the past three decades, Library conservators have modified wash water with ethanol, magnesium bicarbonate with various dilutions of deionized water and/or ethanol, or simply employed non-aqueous deacidification methods to abate the corrosion of those iron-gall inks which were unacceptably altered by even ethanol-diluted magnesium bicarbonate.

Yet questions concerning the stabilization of iron-gall ink still exist. Conservation Division staff became greatly interested in the promise of the antioxidant calcium phytate treatment proposed by Han Neevel and Birgit Reissland, of the Netherlands Institute for Cultural Heritage (ICN), in the late 1990s (Neevel 1995; Reissland and De Groot 1999). Library and visiting conservators collaborated with staff scientists in discussion groups to debate the recent technological advances in understanding and treating iron-gall ink, and also to establish a research plan to better evaluate new treatments. Practical experience was gained through in-house workshops in the application of the two-step calcium phytate/calcium bicarbonate protocol.

Currently a team of Library conservators, including the author and Cindy Ryan, Elmer Eusman, Heather Wanser, and Holly Krueger, are testing and comparing the calcium phytate technique to iron-gall ink treatments practiced by staff in recent times. Performance of the calcium phytate/calcium bicarbonate protocol will be evaluated against treatments such as 25% saturated magnesium bicarbonate diluted in 75% deionized water, and 25% saturated magnesium bicarbonate diluted in 65% ethanol and 10% deionized water, among others. A further variation on the ICN's experiment includes accelerated aging at conditions of 90ºC and 50% relative humidity, which are broadly accepted by the conservation science community, instead of the extreme conditions chosen by Neevel and Reissland, of cycling between 80% and 35% relative humidity every three hours. The Library's storage conditions generally do not manifest extremes of cycling relative humidity. In addition, the Library's study will include gelatin-sized experimental samples, as well as unsized sheets similar to those used in the ICN research. Use of gelatin-sized sheets is thought to approximate more closely the characteristics of original iron-gall ink manuscripts or drawings. The results of these experiments will lead to the eventual testing of expendable antique iron-gall ink materials before possibly assimilating the calcium phytate treatment into common conservation practice. Meanwhile, the Library is interested in coordinating its resources with relevant iron-gall ink research initiatives underway in other laboratories.

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NOTES

1. "Strike through" is a term used to describe corrosive penetration of iron-gall ink through a support, such that the ink can be seen on the support verso.

2. Calcium hydroxide made with calcium oxide.

3. 0.2% calcium bicarbonate made with calcium carbonate.

4. The 1964 Barrow Research Laboratory article makes the first suggestion known to the author of adding alcohol to magnesium bicarbonate, in this case to reduce cockling of spray-deacidified bound book pages.

5. Author's survey of treatment reports dated 1971 through 1985 in the Conservation Division, Library of Congress. In the Preservation Office, supersaturated magnesium bicarbonate was made to achieve a titration of 25ml EDTA to end point.

6. Made with magnesium carbonate.

7. Four pages from the Notes of the Proceedings of Congress were immersed in deionized water prior to immersion in saturated magnesium bicarbonate diluted 50% with water. Library of Congress Preservation Office treatment report number 001998, April 1, 1975.

8. Preservation Office treatment records of this period for a wide range of materials indicate that immersion in saturated calcium hydroxide diluted 50% with water was common.

9. The cellulose pulp contained 16 ppm, 399 ppm, and 525 ppm iron respectively.

10. Tang's further research of 1979 provided evidence that paper immersed in deionized water with even small quantities of calcium, (9.2 and 11.4 ppm calcium respectively) substantially increased fold endurance compared with unwashed paper (Tang 1981).

11. Three types of papers were tested: rag waterleaf made by the Library of Congress Preservation Research and Testing Division; antique rag from a 1768 publication; and ten-year-old newsprint. The alkaline reserve data represents an average of the uptake of all three types of papers with respect to each immersion protocol and is expressed in percent calcium carbonate equivalents.

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