ABSTRACT

Following the scientific research of the Library of Congress’ Iron-Gall Ink Corrosion Group (2002–2005), the Conservation Division’s Protocols for Iron-Gall Ink Treatment Group (PIT) has worked to incorporate recent findings into treatment practice and to develop a unified approach to the conservation of paper-based collections containing iron-gall ink.

The paper will illustrate how the methodology and tools produced by PIT are used in the treatment of iron-gall ink-inscribed manuscripts. The tools include an examination form for recording the information specific to iron-gall ink that leads directly to treatment choices. Flow charts, called Treatment Trees, guide treatment based on visual, chemical, and solubility characteristics of the ink and address options for washing, alkaline, and complexing treatments. Additional documents on treatment methods work in conjunction with the flow charts to further refine and optimize treatments.

For the past two years Library conservators have used the methodology and tools to guide treatment of collection materials of various formats and containing iron-gall ink at various stages of deterioration. The paper will directly demonstrate the use of the PIT tools in guiding conservation treatments of eighteenth-century American manuscripts undertaken jointly by a paper and a book conservator at the Library of Congress.

INTRODUCTION

The problems associated with historical iron-gall ink are well-known to conservators. The Library of Congress Conservation Division has focused on addressing these problems during the last eight years through two initiatives. The first, the Iron-gall Ink Corrosion Group, scientifically evaluated various papers treated with eight combinations of pH neutral and alkaline solutions following accelerated aging. The second initiative was more practical in focus. The Protocols for Iron-gall Ink Treatment Group, or PIT Group, had a two-year mandate to develop a consistent approach for treatment of iron-gall ink-inscribed materials in the Library’s collections.

Many of the special collections in the Library’s custodial divisions contain iron-gall ink. The Manuscript Division alone currently has over fifty million items in eleven thousand collections, and thus holds the majority of the Library’s iron-gall ink-inscribed materials. Some of the United States’ written historical treasures reside there, including Thomas Jefferson’s rough draft of the Declaration of Independence, Abraham Lincoln’s Gettysburg Address, and the manuscripts presented in this paper.

PROTOCOLS FOR IRON–GALL INK TREATMENT TOOLS

The first part of this paper briefly describes the “toolkit” of documents devised by the PIT Group. The tools are an examination form specific to iron-gall ink with an accompanying Glossary, treatment decision-making flow charts, and a series of explanatory notes to support examination and treatment. PIT developed these tools tailored to practice at the Library of Congress (LC). In LC’s specific, institutional context, several factors were key in shaping the PIT Group protocols: anticipated research or exhibit use for individual items, environmental conditions in storage areas and reading rooms, and the resources available to Division conservators—equipment, materials, and the cumulative conservation experience of the staff. The PIT documents are shown in the final paper of this publication, “Developing Guidelines for Iron-Gall Ink Treatment at the Library of Congress” on p. 129.

As the PIT Group began its work, it recognized that careful examination and testing were the basis of treatment decisions for iron-gall ink-inscribed materials. Although the Conservation Division had developed several examination and treatment forms for various types of paper and book
tests, none of them specifically addressed iron-gall ink. PIT developed a new form, the Record of Examination for Iron-Gall Ink on Paper (See pp. 131–133) to guide close examination, beginning with the appearance of the ink. Characteristics such as the intensity of the ink, the quantity and quality of application, and the degree to which the ink penetrates the paper are noted. The condition of the ink—cracks, losses, and delamination of the inked paper; discoloration surrounding and resulting from contact with the ink; the friability and overall adhesion and cohesion of the ink; the degree of burn-through; and the absorption or fluorescence of an inked area in both visible and long-wave UV light—is also noted.

Some of the Examination terms are familiar, others perhaps are not so well known. The Group developed a Glossary, a common vocabulary, to describe the characteristics and damage associated with iron-gall ink. The examination form provides space to record characteristics of the paper support, but focuses on qualities, such as opacity, surface texture, and degree of sizing, that relate closely to reactions with an iron-gall ink medium.

“Testing” is a large section of the Record of Examination. Here, the conservator can note the rate at which the paper absorbs water, the pH of the paper surface, and the results of the iron (II) ion and solubility tests before, during, and after treatment.

The iron (II) ion test is paper impregnated with bathophenanthrol ine, (4, 7-diphenyl-1, 10-phenanthroline), a chemical indicator used to signal the presence of iron (II) ions (fig. 1). These iron ions are not part of the ink complex, but exist as water-soluble salts. In this chemical form the ions act as catalysts for cellulose oxidation, the most destructive reaction caused by iron-gall ink corrosion. Although the test is qualitative rather than quantitative, the presence of iron (II) ions provides an indication of ink stability and is a valuable tool in assessing the condition of the ink.

The results of examination of the ink and paper lead the conservator to decision-making strategies presented in flow charts. The two types of flow charts or “trees” that PIT includes in its toolkit are Washing Treatment Trees and Alkaline & Complexing Treatment Trees (See pp. 139–140). These Trees are the result of PIT Group member Julie Biggs’s idea for flow charts containing various options for conservators to draw from when treating iron-gall ink-inscribed materials. For the 2006 ICON conference, Julie and fellow PIT Group member Yasmeen Khan wrote Treatment Trees for Iron-Gall Ink on Paper: Using Flow Charts to Develop Treatment Protocols.

At the top of the Washing Trees there are three Guide Boxes with four criteria from examination. The four criteria or “Guiding Factors,” are: a) the condition of the paper, based on evaluation of several factors, including visual examination and surface pH; b) iron (II) ion test results; c) visible presence of ink corrosion; and d) presence of UV fluorescent haloes surrounding the ink. The results of the iron (II) test and corrosion of the ink are the key differences between the two Guide Boxes that branch to treatment steps. For example, in the box on the left the iron (II) test results are positive and the ink is corroded, while in the box at center the iron (II) test is negative or very slightly positive and the ink is not visibly corroded. Below the Guide Boxes the Trees guide the conservator in washing decisions based on the solubility of the ink in water and ethanol. After obtaining results from examination and testing, the conservator can choose the appropriate tree and recommended steps. The steps differ based on the examination and testing results.

In the usual sequence of treatment activities, alkaline treatment follows washing. To use the Alkaline & Complexing Treatment Trees, the conservator considers the criteria appearing in the Guide Boxes. These criteria are best obtained by testing following washing, as an object will have changed in response to the washing treatment. Like the Washing Trees, the branches diverge from the two Guide Boxes, based on the solubility of the ink in water, then divide again, depending on the solubility of the ink in ethanol.

The Alkaline & Complexing Treatment Trees include alkaline treatment via fully aqueous, solvent-modified, and non-aqueous methods, and complexing treatment with calcium phytate and calcium bicarbonate via fully aqueous and solvent-modified methods. The Alkaline & Complexing Trees also recommend re-evaluation of the criteria in the Guide Boxes when phases of treatment are completed. It is important to emphasize that the Trees present a range of treatment possibilities, rather than a prescription for treatment or a substitute for the conservator’s experience.

The Washing and Alkaline & Complexing Trees are summaries of treatment options. Since the primary goal of the PIT Group was to establish a package of examination and treatment guidelines for the LC conservation lab, the Group also created a series of technical notes. The Notes provide a more nuanced view of treatment methods populating the Trees. Subjects of PIT Notes include instructions for examining iron-gall ink using the Library’s imaging equipment;
procedures for spot testing for ink solubility; and procedures for washing, alkaline and complexing treatments, as well as advantages and disadvantages of those procedures.

TREATMENTS DEMONSTRATING THE USE OF PIT TOOLS

[Diary] [Detailed proceedings of the Continental Congress, as observed by Richard Smith, member of the Committee on Claims], (1775 Sept. 12–Oct. 1; 1775 Dec. 12–1776 Mar. 30)

The second part of this paper demonstrates the use of PIT tools for treatment of several late eighteenth-century objects from the Library’s Manuscript Division: the [Diary] of Richard Smith, the Petition of the Continental Congress to the King, and James Madison’s Notes on Debates in the Federal Convention of 1787. These were just a few of the many iron-gall ink-inscribed items selected for the Library’s recently opened exhibit “Creating the United States.” The following conventions apply to all of the treatments that will be described: a) ethanol, with a few drops of deionized water added, was used for conditioning before washing; b) for the washing solutions, the percentage of solvent to water and the pH of the wash bath is stated; c) all of the treatment baths were solutions of deionized water, adjusted with a saturated calcium hydroxide solution to the desired pH, and ethanol; d) ColorpHast (EM Science / Merck) strips were used to indicate the pH of solutions and the surface pH of paper; and e) drying after all phases of treatment included blotting and placing leaves between dry polyester web and lightly weighted felts.

The first treatment concerns a diary kept by New Jersey delegate Richard Smith during the Continental Congress from 1775–1776 (fig. 2). The diary is Smith’s personal record of the proceedings of the Congress, and includes details of property losses and government expenditures during the Revolutionary War. One large pamphlet-sewn gathering of more than 50 bifolia dominated the two-gathering binding structure. The size of that first gathering required the leaves to round around the spine and the paper to flex considerably as pages were turned (fig. 3). Cotton cord wrapped around the gathering held it together. The cord sawed into the edges of the leaves, causing them to break away at the spine folds (figs. 3–4). Some pages had been lost and in some instances only fragments of others remained.

Many pages showed evidence of water damage. The 15 x 9 cm pages contained closely spaced lines of text on both

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**Fig. 2.** Richard Smith, [Diary] [Detailed proceedings of the Continental Congress, as observed by Smith, member of the Committee on Claims], (1775 Sept. 12–Oct. 1; 1775 Dec. 12–1776 Mar. 30), iron-gall ink on paper, approx. 15 x 9 cm, Manuscript Division, The Library of Congress (MMC, Ac. 2877, LC Control No. mm75001790). Before treatment, overall

**Figs. 3–4.** Richard Smith, [Diary]. Before treatment, tail (left) and spine (right)
washing progressed. The first 10-minute bath was 75% ethanol, 25% water, followed by two 10-minute baths of 50% ethanol, 50% water. The last 10-minute bath contained 25% ethanol, 75% water. Leaves that were too fragile for immersion were supported on screens during washing. Bifolia were removed from the last bath, then immersed in 100% ethanol for five minutes to drive out as much water as possible and thereby reduce tensions in the inked paper as it dried. On the Trees, this step is “Pre-dry with ethanol.” The bifolia were removed from the ethanol bath, blotted, and dried.

Returning to the progress of the treatment on the Trees, following washing, test results were: a) slightly positive to positive iron (II); b) paper pH acidic, 5.0; c) ink corroded; d) no haloes. These results guide the treatment to the left side of the Alkaline Treatment Trees (fig. 7). The solubility of the ink was re-tested. It was insoluble in both ethanol and water. With this change in solubility, the treatment was guided along the second branch. Although the ink was not severely corroded, the paper pH and the flexing that will occur as the

sides, thus the ratio of iron-gall ink relative to the surface area of the paper was high (fig. 5).

Essential elements of the information observed and recorded on the examination record are highlighted in the four criteria in the Guide Boxes of the Washing Treatment Trees: a) the iron (II) test was strongly positive; b) the pH of the paper was acidic, between 4.0 and 4.4; c) there was strong visible evidence of ink corrosion—cracks appeared in areas where ink was heavily applied, slight to moderate localized discoloration surrounded the ink, and ink burn-through ranged from moderate to severe—and d) no haloes or fluorescence were observed in UV light. The examination and testing results pertaining to the four criteria guide the treatment to the left of the Washing Treatment Trees. The solubility of the ink in water but not in ethanol directs the user down the first branch where the Trees suggest ethanol-modified washing immediately followed by pre-drying with ethanol, then final drying (fig. 6).

Before washing, each bifolium was conditioned by spraying with ethanol, with special attention given to the spine folds. This additional step was to facilitate more even wetting-out in the wash bath. Most of the bifolia were washed by immersion in batches for 40 minutes total, in four baths at pH 7.5. The goal was to wash the manuscript leaves with as much water in the bath as possible, to remove most of the residual iron. To be cautious with the water-soluble ink, a higher proportion of ethanol to water was used in initial baths; the proportion of water was gradually increased as

Fig. 5. Richard Smith, [Diary]. Before treatment, [f. 53] recto

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Fig. 6. Richard Smith, [Diary]. Sequence of treatment steps on Washing Treatment Trees. Note additional step

WASHING TREATMENT TREES

Fe2 test: strongly +
Paper: suggests treatment and acidic / pH 4.0 to 4.4
Ink corroded
No haloes

Ink water soluble

Ink not ethanol soluble

Conditioning with ethanol

Ethanol-modified washing

Pre-dry with ethanol

Dry

Re-evaluate Guide Boxes.
Results:
Fe2 test: slightly + to +
Paper: acidic / pH 5.0
Ink corroded
No haloes

Continue to Alkaline & Complexing Treatment Trees.
Iron-gall Ink Treatment at the Library of Congress

Dekle and Haude

adhered with wheat starch paste. After brief humidification, the gatherings were re-assembled and sewn onto handmade paper that acts as endleaves and spine support. Loose guards of Japanese tissue were used to separate the sewing thread from original material. A limp paper case was made to protect the text block. With careful handling and proper support, research use of the [Diary] is now possible (figs. 8–10).

Fig. 7. Richard Smith, [Diary]. Sequence of treatment steps on Alkaline & Complexing Treatment Trees

Fig. 8. Richard Smith, [Diary]. After treatment, front cover, spine, and tail

Fig. 9. Richard Smith, [Diary]. After treatment, [f. 1] verso - [f. 2] recto

Fig. 10. Richard Smith, [Diary]. After treatment, [f. 53] recto

book is read were considered carefully. Based on the favorable results for magnesium bicarbonate reported by the Library’s Iron-gall Ink-Corrosion Group, the conservators chose that solution for alkaline treatment of the acidic paper. No color change of the ink was observed during a 20-minute test with 20% magnesium bicarbonate, 80% water. The bifolia were conditioned by spraying with ethanol and then immersed in the magnesium bicarbonate bath for 20 minutes. Fragile leaves were supported on screens. Following alkaline treatment, the iron (II) test was negative and the surface pH of the paper was slightly alkaline at 7.5.

The next consideration for treatment was assessment and application of a sizing agent to the paper. Although some of the original sizing remained in the paper after the washing and alkaline treatment steps, it was not sufficient for anticipated use of the [Diary]. The leaves that had been washed in batches were sized in a vat containing a low percentage gelatin solution, 0.25%. More fragile leaves were sized by brushing the gelatin onto them through polyester web. After treatment, the paper is flexible and the ink appears stable, with no observable changes in color.

Four bifolia were re-established by leaf-casting with the fragments. Pages were repaired with toned kozo tissue
Petition of the Continental Congress to the King, Philadelphia, Pa., Oct. 26, 1774

The next treatment, guided by referring to the Treatment Trees, was performed on the Petition of the Continental Congress ... [to George III of England], Oct. 26, 1774 (figs. 11–13). Through the Petition, representatives of the American colonies sought to negotiate solutions to injustices by laying their “grievances before the throne.” The King’s rejection of this and other diplomatic efforts by the colonies fueled the colonial movement for independence. Although only five leaves comprise the manuscript, numerous hands and inks appear in the document and accompanying letter. The principle hand is that of engrosser Timothy Matlack, who later engrossed the Declaration of Independence. This copy, signed by the delegates to the Congress, was Benjamin Franklin’s. Prior to treatment the manuscript was in a tightback, full leather, presentation binding—the signature binding for the Franklin Collection of Henry Stevens, an American book dealer active in London in the mid-nineteenth century (fig. 14). To produce the highly finished ideal binding of that time, the manuscript was incorporated into a text block and then heavily rounded (fig. 15). The rounding brought the corroded ink text dangerously close to the flexing area near the gutter. Numerous contracted repairs on the pages also threatened the manuscript (fig. 16).

Since so many different inks were used in the document, test results varied widely. The iron (II) tests ranged from faintly to strongly positive. Even uninked paper adjacent to iron-gall ink tested positive for the presence of iron (II). The surface pH of the paper ranged from 4.2 to 4.7. The ink was corroded, as evidenced by cracks, flaking, and losses in areas of heavy application, such as the signatures. Discoloration and moderate burn-through were also present in heavily inked areas, and, in UV light, faint, green-yellow fluorescence appeared on the verso. Using the Trees, a treatment plan was developed to address the worst conditions observed. Solubility testing guided the treatment to “Ethanol-modified washing” (fig. 17).
The leaves were conditioned before immersion in a bath of 80% ethanol, 20% water and then dried completely. The conditioning was to facilitate more even wetting out of the paper; the bath of 80% ethanol, 20% water, followed by complete drying, was to stabilize the varied inks before washing. Prior repairs were removed from the leaves before washing in three 15-minute baths of 50% ethanol, 50% water at pH 8.0. During washing, the baths were agitated by gently rocking the trays every few minutes and the areas of adhesive residue were tamped with soft brushes. Leaves were then immersed in 100% ethanol for five minutes. This step was especially important in areas where the ink had cracked or dropped out. When the leaves were dry, areas of ingrained dirt and adhesive residue were cleaned with dilute methyl cellulose.

After washing, iron (II) tests were faintly to strongly positive; the paper pH was about 4.5, not much improved; the ink was corroded; and no haloes around the ink were visible in UV light. In consideration of the iron (II) and pH test results, the inks were tested for solubility using a higher percentage of water. Then all leaves were spray-conditioned with ethanol; washed in 25% ethanol, 75% water at pH 8 for 15 minutes; pre-dried; blotted; and dried (fig. 18). The additional immersion in a bath with a proportionately larger amount of water removed more discoloration from the paper and improved the flexibility of the leaves. Immersion in a larger amount of water also removed more “free” iron from the iron-gall ink. Tests for most areas yielded faintly positive results for the presence of iron (II). In a few areas of very heavy application, the test results were improved, but still sufficiently colored to indicate iron (II). The paper pH of 5.0, the sensitivity of the inks to water and, unlike the previous treatment, a concern for possible color shift in the warm-toned inks strongly suggested complexing and
alkaline treatment with ethanol-modified calcium phytate/calcium bicarbonate (fig. 19).

Post-complexing and alkaline treatment test results for iron (II) ions were barely perceptible as positive, and the paper pH ranged from 6.5 to 7.0. The paper was slightly lighter in color overall and the contrast between media and support enhanced. As observed for the [Diary] of Richard Smith, the Petition leaves retained some original sizing throughout treatment, but not enough for future handling. Dilute gelatin (0.25%) was brushed onto the leaves through polyester web. Japanese tissues adhered with wheat starch paste were used to repair tears and compensate for losses (fig. 20). After exhibition, Asian tissue and starch paste will also be used to re-unite pages B and H as a folio. Although the Petition will not be returned to the Stevens Collection binding, the binding will be housed with it in an enclosure compatible with the other volumes of Franklin materials collected by Stevens and now held by the Manuscript Division.
Notes on Debates in the Federal Convention of 1787
Sept. 12–13, 1787

The last treatment regards an entry from Virginia delegate James Madison’s journal, in which he recorded the debates at the Federal Convention at Independence Hall in Philadelphia from May 25 to September 17, 1787. It was an important convention—the U.S. Constitution was drafted. Three entries from Madison’s journal were treated so that they could be safely exhibited. The paper was similar for all three entries and thus it was decided to attempt to treat all three consistently. Seen here is the September 12th to 13th entry that records the delegates’ debates about preparing the Bill of Rights (figs. 21–26).

The results obtained during the examination of the Madison document before treatment included: a) strongly positive iron (II) tests; b) acidic paper with a pH of 4.0; c) visible evidence of corrosion in areas of heavily applied ink, with moderate localized discoloration surrounding the ink, and moderate to severe burn-through; and d) UV fluorescent haloes around the ink. As with the earlier example, these criteria guided to the left of the Washing Treatment Trees (fig. 27). Since the heavily applied inks were soluble in water but not in ethanol, the treatment moved toward “Ethanol-modified washing.” The Madison document was conditioned by spraying with ethanol, pre-treated for 10 minutes in 80% ethanol, 20% water, and dried completely. The conditioning spray facilitated even wetting-out of the paper. The high ethanol content pre-treatment was used to stabilize the variety of inks before washing.

After conditioning the document for washing, it was immersed for 30 minutes total in three baths at pH 7.5. As in the Richard Smith treatment, progressively lower proportions of ethanol were used in the baths as the inks demonstrated less solubility in water. The first 10-minute bath was 75% ethanol, 25% water, followed by two 10-minute baths of 50% ethanol, 50% water. Before drying, the water content in the document was reduced by immersing it in 100% ethanol for five minutes. However after washing, the inks still tested strongly positive for iron (II) and the pH rose only slightly to 4.5. Another washing cycle in baths with a larger proportion of water would be necessary to remove more of the residual iron and degradation products in the paper. This experience underscores the importance of testing inks after treatment to determine the efficacy of the treatment.

The second washing cycle proceeded with conditioning by spraying, followed by immersion for 25 minutes (fig. 28). The first bath was 50% ethanol, 50% water for ten minutes. The second bath was 25% ethanol, 75% water for 15 minutes. Again the document was immersed in 100% ethanol for five minutes before drying it. Following the second washing cycle, areas of heavily applied ink tested positive for iron (II) and the pH of the paper was raised to 5.0. While residual
iron remained in some inked areas, it was decided to move to the next phase of treatment rather than to subjecting the document’s inks and paper to further washing.  

Post-washing test results and the solubility of the inks directed treatment to the first branch of the Alkaline & Complexing Treatment Trees (fig. 29). Several entries from Madison’s journal, dated July 16th to 17th, 1787 exhibited severe ink corrosion and cracking in heavily inked areas. To stabilize the ink on those pages, alkaline and complexing treatment with ethanol-modified calcium phytate and calcium bicarbonate was appropriate. The positive iron (II) test and low pH for the September 12th to 13th leaves also pointed to calcium phytate and calcium bicarbonate treatment.

The cracks in the inked areas of the two other leaves required that all three documents be supported on a screen during treatment, and that they be conditioned prior to immersion by spraying with ethanol. The documents were immersed in 50% calcium phytate, 50% ethanol for 20 minutes. After draining and blotting them briefly, they were immersed in a 50% calcium bicarbonate, 50% ethanol solution for 20 minutes. After drying, none of the inks tested positive for iron (II) and the pH of the paper rose to 7.5. The final steps of the treatment included sizing with a 0.5% gelatin solution applied by brushing through polyester web, mending with Korean tissue and wheat starch paste, and humidifying and flattening overall (figs. 30-35).
CONCLUSION

PIT concepts and tools were successfully used to support the examination and decision-making for the three treatments described. Two years after being introduced to Conservation Division staff, PIT protocols have also been applied to many other iron-gall ink-inscribed materials, including nineteenth-century American correspondence, a sixteenth-century letter to the Archbishop of Mexico from Philip II of Spain, and the documents and journals of the founders of the United States. None of the tools is intended to replace the judgment of the conservator. Rather, the tools distill and focus the body of conservation literature and practice related to various aspects of iron-gall ink treatment. In particular, the Washing Trees and the Alkaline & Complexing Treatment Trees display decision-making strategies at different stages of conservation treatment of iron-gall ink-inscribed paper.

The Protocols for Iron-gall Ink Treatment Group has been a successful collaboration of the paper- and book-conservator team members and a successful collaboration of the Conservation Division staff, which provided essential
feedback on the toolkit as it was developed. In a sense, it was, and continues to be, a collaboration among dozens of conservators, charged with preserving the important national collections at The Library of Congress.

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CLAIRE DEKLE
Senior Book Conservator
The Library of Congress
Washington DC
cdek@loc.gov

MARY ELIZABETH HAUDE
Senior Paper Conservator
The Library of Congress
Washington DC
mhaud@loc.gov