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The Use of Simmering Water in the Treatment of a Nineteenth Century Sketchbook of Iron Gall Ink Drawings by James G. Mackay

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This paper describes an approach to the conservation treatment of a sketchbook from the collection of Library and Archives Canada. The J.G. Mackay sketchbook consists of drawings that are severely damaged by iron gall ink corrosion. Two different treatments were considered for the sketchbook: calcium phytate treatment, recently developed at the Netherlands Institute for Cultural Heritage (ICN), and the "boiling", or as the authors prefer to describe it, "simmering" water treatment, based on the experiments of Austrian conservators and treatments carried out in a few European laboratories since the 1970s. Analyses were also carried out to investigate the efficiency of the simmering water treatment by monitoring the wash water's pH, iron concentration and UV absorbance (indicating the presence of soluble salts). This report will describe the difficult process of choosing the optimal treatment and its ethical significance, along with a detailed description of the simmering water treatment.

Cet article décrit l'approche et le raisonnement derrière les décisions prises vis-à-vis du traitement du livre d'esquisses de J.G. Mackay provenant de la collection de la Bibliothèque et des Archives du Canada. Ce livre d'esquisses contient des dessins qui sont très endommagés par les effets corrosifs de l'encre ferro-gallique. Deux différents traitements ont été considérés : le traitement à base de phytate de calcium, récemment mis au point à l'Institut du patrimoine culturel des Pays-Bas (ICN); et le traitement à l'eau chaude, basé sur des expériences faites par des restaurateurs autrichiens et sur des traitements qui ont eu lieu dans quelques laboratoires européens depuis les années 1970. L'efficacité du traitement à l'eau chaude à été vérifié en analysant l'eau de traitement pour vérifier son pH, sa concentration d'ions de fer et son absorbance UV (ce qui est indicatif de la présence de sels solubles). Face à deux options de traitement, chacune avec ses avantages et ses désavantages, les auteures décrivent leur processus décisionnel qui a mené au choix du traitement à l'eau chaude, et ensuite décrivent ce traitement en détails.

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Introduction

Library and Archives Canada houses an important collection of some three hundred sketchbooks and albums containing works by Canadian artists, depictions of Canadian landscapes and the life of this country's past and present inhabitants. The James G. Mackay sketchbook, entitled *The Pilgrim's Progress Colored*, was created around 1875, and was acquired by the National Archives in 1988.[1] Little is known about the author/illustrator, James G. Mackay, who was a wood engraver and illustrator. He was born in 1847 in Hamilton, Ontario, and died there in 1885. His views of that city, as well as his political caricatures were published in the *Canadian Illustrated News* from 1873 to 1885.[2]

The curatorial decision to conserve the James Mackay sketchbook was generated by a concern for its extremely fragile condition due to severe damage caused by iron gall ink corrosion. Stabilization of the sketchbook would allow for consultation and copying.

Description of the Sketchbook

The James Mackay work is a series of illustrations that forms a narrative. The book is typical of blank, commercially available notebooks of the period. It was originally made up of at least six sections with 12 leaves to a section. All 57 drawings contained in the notebook are executed in pen and iron gall ink on calendered, smooth, off-white wove paper. Twenty-seven of the drawings are enhanced with pencil. Several different ink colours, from pale brown to almost black, sometimes on the same page, can be identified in the book. The drawings are inspired by the English literary classic, *The Pilgrim's Progress* by John Bunyan, published first in 1678. The drawings in the MacKay sketchbook are accompanied by captions and/or quotes from Bunyan's original text. Page 28 has a caption only – a drawing was never created. Bunyan's powerful allegory renders the author's personal spiritual experience into the form of a universal myth, where all Christians who seek the truth are embodied within a figure of the solitary man pursuing his pilgrimage. It had a profound impact on English consciousness and the literature that followed its publication. Numerous editions were published and it was translated into many languages. The James Mackay sketchbook is a parody of Bunyan's work, narrating the misadventures of a poor black man, Mr. Christian, within a Canadian context. It includes some finely executed depictions of everyday life and Canadian localities such as Wentworth County and Welland Canal. One of the characters sports the face of Sir John A. Macdonald, Canada's first Prime Minister. The sketchbook gives an interesting view of the position of a black man in the predominantly white society in Canada at the end of the 19th century, and will certainly be a valuable object of study by scholars interested in the history of African-Canadians in Canada (**Figure 1**). A signature located on the front flyleaf of the sketchbook identifies a possible owner.

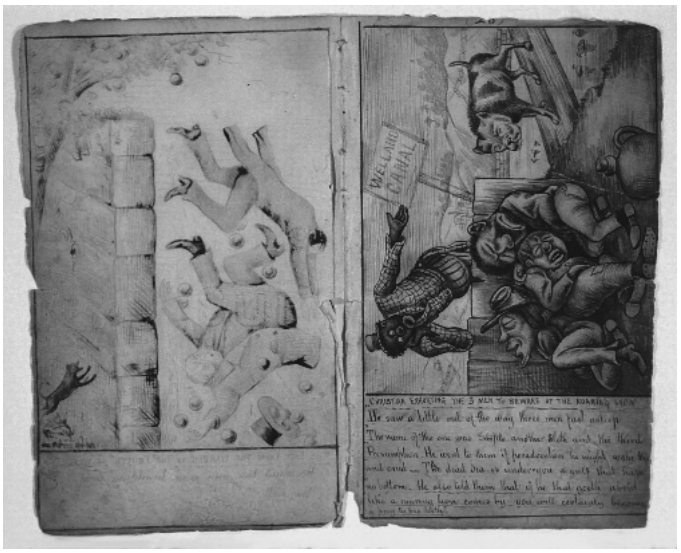


Figure 1. A page from a sketchbook of iron gall ink drawings created ~1875 by James G. Mackay, entitled *The Pilgrim's Progress Colored*. Before conservation treatment. Library and Archives Canada, Accession No. 1988-033.

According to J. Divine, Art Archivist from Library and Archives Canada, it is not clear whether the signature on the sketchbook reads 'James White **M.D.**' or 'James White **M.P.**' From approximately 1876 to 1913, there are entries in the Hamilton directories for a James White **M.D.** who was a medical superintendent and physician at the City Hospital and a coroner as well from 1881 to 1913. Mackay, working in a registry office, might have encountered White. The artist also died in the City Hospital which is where White worked. This is all the information that is available on the possible owner.[3]

Condition

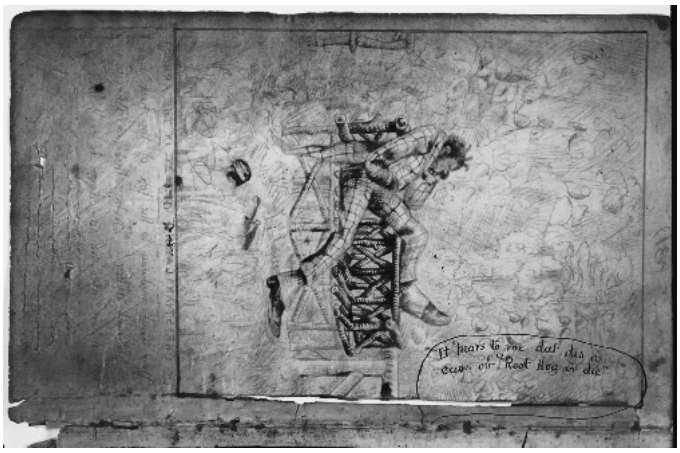
Binding

The binding is half leather with cloth covered boards that measure 21 cm x 13 cm. The dark brown leather is split sheepskin and the greenish grey book cloth has a dotted hexagon grain. The spine leather was adhered with an excessive amount of animal glue directly to the back of the textblock which resulted in stiffness of the spine and damage to the paper. Both boards were detached and the spine leather was fragmented. The sketchbook had been rebound. The only evidence of the previous binding was the sewing holes that showed that the sketchbook had been sewn on two tapes. This had been replaced with recessed cords reinforced and repaired with overcasting. This added to the stiffness of the binding.

Textblock

The paper was brittle and discoloured throughout. Some leaves were very worn and many were heavily stained. Numerous tears and some areas of paper loss had been repaired with paper patches applied to the verso. Most leaves suffered from different stages of iron gall ink corrosion[4] that had resulted in the darkening of the ink, numerous fractures, offsetting of images and small losses of design in heavily inked areas (**Figure 2**). Most fractures took place along the left side of the ink-lined border that framed the drawings, in the area that would have been under stress due to

(a)



(b)

(c)

Figure 2. James G. Mackay, *The Pilgrim's Progress Colored.* Condition before conservation treatment: **a)** title page with extensive and heavy application of iron gall ink that resulted in fractures and losses of design, **b)** verso of page 31 with losses in ink line, and **c)** recto of page 33 with parts of paper split along corroded ink line.

its proximity to the gutter. The title page suffered the worst damage from iron gall ink corrosion, as the ink application on that page was extensive and heavy. Some movement of ink outside ink lines was noticed on stained pages.

Background on Iron Gall Ink and Ink Corrosion

Known from antiquity, iron gall inks were used extensively until the beginning of the 20th century. They belong to the group of metallic inks as their black colour is produced by combining iron with tannins. They consist of four essential components: iron (II) sulphate, gallic or tannic acid, water or wine as a solvent and gum arabic as a binder. The iron compound of the ink is usually ferrous sulphate, a crystalline material soluble in water and slightly corrosive. It is sometimes known as "copperas" or "vitriol". Tannins used in ink-making are organic acids derived from oak galls or other plant material.[5] Hundreds of recipes were used to produce iron gall inks. Records indicate that the ratio in which the four primary ingredients were used could differ greatly. Many different additives were also incorporated in order to achieve specific effects. Their presence in the inks can make conservation treatments very challenging and complex as their long-term effects are still not fully understood.[6] However, the greatest problem that the paper conservator needs to address is iron gall ink corrosion.

Only recently have scientists started to study and understand the complex chemical reactions responsible for the breakdown of iron gall ink and their effect on the paper supports to which they were applied. It is generally agreed that iron gall ink corrosion is caused by two major degradation processes: acid-catalyzed hydrolysis and iron (II)-catalyzed oxidation of cellulose.[7, 8]

- "The sulphuric acid present in the ink catalyzes the acid hydrolysis of cellulose, resulting in chain scission of the cellulose polymer. This process continues until the acid is neutralized, either by paper additives or by conservation treatments.
- Transition metals such as iron or copper can catalyze the oxidation of cellulose, causing chain scission and cross-linking of the cellulose polymer. This results in a reduced water absorption, fluorescence and colour changes in the paper."[9]

Analysis of Paper, Size, Fillers and Inks

Statement of the Analytical Problem

To assist in the choice of treatment to strengthen the brittle paper support, to stabilize fragile areas and to arrest the ink corrosion, an analysis of the paper fibers, fillers, size and inks was undertaken by the Analytical Research Laboratory at the Canadian Conservation Institute. It was important to determine the composition of the inks, in particular to identify if the major deterioration of the paper was related to acid in the ink (sulphuric acid) or to excess iron present in the ink. This was done by analyzing the inked areas to find evidence of the sources of deterioration which can occur as reported in the literature:

1. the migration of sulfur out of the ink into the surrounding paper indicating excess sulphuric acid;[10]
2. accumulation of iron in the middle section of the paper indicating the penetration of the ink deep into the paper;[10]
3. excess iron present in the ink which can act as a catalyst for the oxidative decomposition of cellulose;[10]
4. sulfur/calcium ratios and sulfur/iron ratios in the ink indicating either an excess of iron or sulphur in the ink.[11]

To ensure the integrity of the sketchbook, the analytical methods chosen were non-destructive or required only very small samples. The method used to analyze the inks was based on prior studies of iron gall inks by x-ray microanalysis (SEM/EDS).[12]

Experimental Methods

pH

The surface pH of selected inked and uninked areas from the Mackay sketchbook was determined using an Orion 620 Benchtop pH Meter equipped with an Orion 6165 pHuture Sure Flow® electrode. The electrode was calibrated using pH 4 and pH 7 buffers.

Non-Destructive Analysis of Paper and Ink: X-ray Fluorescence Spectrometry

Several pages from the Mackay sketchbook were examined qualitatively using non-destructive x-ray fluorescence spectrometry (XRF) to detect the elements present in the drawing media. Areas of approximately 3 cm^2 in size of plain paper (no ink present) and of paper coated with ink were analyzed using a Noran Voyager II x-ray microanalysis system, a lithium-drifted silicon x-ray detector and either a cadmium-109 or americium-241 radioisotope as the excitation source. This technique detects elements with an atomic number greater than or equal to that of potassium (K) at a level of 0.1-1 weight % or greater.

Analysis of Paper Fibers, Fillers and Sizes: Microscopy and FTIR

The paper fibers were analyzed by light microscopy (PLM). The fillers and size used in the paper were analyzed by Fourier transform infrared spectroscopy (FTIR).

Analysis of Inks

X-ray microanalysis (SEM/EDS) was carried out on two ink samples from the sketchbook to determine the concentration ratios of the elements iron, sulfur, copper, calcium and potassium in the inks. The samples analyzed were:
1) a paper fragment from the title page which included dark black ink and lighter ink; and
2) ink from a large fragment of the title page.

The analysis was performed using a Hitachi S-530 scanning electron microscope operated at an accelerating voltage of 20 kV equipped with a lithium-drifted silicon x-ray detector and a Noran Voyager II x-ray microanalysis system. SEM/EDS detects elements from sodium (Na) to uranium (U) in the periodic table

at a level of approximately 0.1 -1 weight % or greater, in volumes down to a few cubic micrometers. S/Ca and S/Fe peak area ratios for the ink samples were calculated by dividing the net peak area of the S Kα peak by the net peak area of the Ca Kα or Fe Kα peak. Following Sistach and Espadaler,[11] the S/Ca and S/Fe ratios were used, in combination with information on ink colour and pH, to determine the degradative behavior of the iron gall inks.

To detect variations in the concentrations of iron, sulfur, calcium, potassium and chlorine from an inked to an uninked area, an x-ray line scan, in which the electron beam is scanned across the sample in a preselected path, was performed on sample 1.

To determine whether the ink had penetrated deeply into the paper substrate, a cross-section of an inked area was prepared by embedding the sample (taken from a piece of the inked paper from the title page) in low viscosity epoxy resin under vacuum and then microtomed with a glass knife. SEM/EDS elemental maps of the cross-section were acquired for the elements iron, sulfur and calcium.

Results

pH

The pH readings of the uninked areas of the paper support ranged from 4.5 to 5.2, while those of the inked areas ranged from 4.1 to 4.7.

X-ray Fluorescence Spectrometry of Paper and Ink

Calcium was detected in the paper by XRF. Iron, calcium and traces of other elements were detected in most of the black inked areas, however, the amount of iron varied among different inks. Some inks analyzed contained both copper and iron, suggesting they may have been prepared with both copper sulfate and iron sulfate. The inks containing iron as the major element detected, with no copper, may have been prepared with iron sulfate only.

Paper, Fibers, Fillers and Sizes

The paper fibers were identified as bast fibers by polarized light microscopy. The fillers and size used in the paper were identified by FTIR as a collagen type of glue, starch and calcium sulphate hemihydrate ($CaSO_4 \cdot 0.5H_2O$). Fragments of a resinous material on the paper were identified as glue and starch.

SEM/EDS Analysis of Inks

SEM/EDS analysis of areas of inked paper detected iron, sulfur, calcium, potassium and chlorine. The S/Ca and S/Fe peak area ratios calculated for both the inked and uninked areas of the paper are in **Table I**. The inked areas analysed in sample 1 (1B and 1C) had a S/Fe ratio of <1 which suggests the presence of excess iron was a major contributing factor in the ink's corrosive behavior. The inked areas analyzed in sample 2 (2A and 2B) had a S/Fe ratio >1 (see **Table I**) which, when compared with reported studies,[11] suggests acidity (due to sulphuric acid) was the major contributing factor for corrosion. The high S/Fe ratio in the paper (no ink) samples is a result of the very low concentration of Fe; the S presumably comes from the calcium sulphate hemihydrate used as a filler in the paper.

The results of the x-ray line scans on sample 1 are as follows: the iron and potassium concentrations were highest in the ink and dropped off significantly outside the inked area. The potassium may be a contaminant present in the iron sulphate used to prepare the ink. No evidence of enriched sulphur concentrations at the edge of the ink, which would indicate excess sulphuric acid, was detected in the line scan. This has been observed in studies of other samples of acidic iron gall inks.[10] Most of the calcium and sulfur also detected in the line scans could be accounted for by the presence of particles of calcium sulphate hemihydrate which were present throughout the paper. Chlorine was also detected in both the paper and the inked area of the sample. The chlorine present in the paper is most likely due to bleaching residues.

Table I: S/Ca and S/Fe ratios and properties for ink samples.

Sample Area	S/Ca	S/Fe	Ink Properties based on S, Ca and Fe ratios
Sample 1: Paper only			
1A: paper, no ink	0.9	7.21	
1D: paper, no ink	1.3	9.57	
Sample 1: Paper and Ink (dark black ink)			
1B: ink and paper	1.37	0.42	corrosive (high Fe)
1C: ink and paper	0.629	0.48	corrosive (high Fe)
Sample 2: Paper and Ink (dark black ink)			
2A: black ink and paper	1.56	1.34	dark, acidic ink
2B: black ink and paper	1.586	1.16	dark, acidic ink

The cross-section of the inked paper showed that the ink was present on the surface and that it had penetrated into the upper paper fibers. No accumulation of iron in the interior of the paper, which would indicate that the ink penetrated deep into the paper, was detected.

Conclusion Drawn from the Analyses

The similarity in the initial pH readings (between uninked and inked paper) and the analytical information obtained, i.e. no sulfur halos around the ink, high iron concentrations present in some areas of the ink, and no penetration of the iron into the paper, suggest that the main cause for the degradation of the ink and the support paper in the Mackay sketchbook is oxidation catalyzed by iron (II) ions, and to a lesser extent hydrolysis catalyzed by sulphuric acid.

Discussion of Treatment Options

Disbinding the Sketchbook

The intent of the treatment was to ensure access to the contents of the book. Options were carefully considered and the decision to disbind the sketchbook was based on several factors. Mending the weakened and degraded paper *in situ* was not an option; the binding had to be removed to treat the paper. The binding was not original and if all sewing and spine glue were to be removed, the stiffness would be greatly reduced and full treatment of the text block would be possible.

As the boards were already detached, this entailed one less step in the disbinding treatment. After the fragmented leather was lifted from the spine, the old glue was humidified with methylcellulose paste and then gently scraped off with a bone folder. The sewing was cut and removed and the textblock separated into loose leaves.

Stabilizing Iron Gall Ink Corrosion

An optimal conservation treatment for iron gall ink corrosion should address three main issues:

1. the degradation process caused by sulphuric acid needs to be treated by removing water-soluble acids from the paper and introducing an alkaline reserve;
2. oxidative degradation caused by iron (II) ions should be blocked by removing or complexing excess iron and;
3. the degraded ink and paper support should be strengthened physically.

For the Mackay sketchbook, two different treatments were considered. The first is a new conservation treatment for objects with iron gall ink corrosion, the **calcium phytate** treatment, developed by Han Neevel, conservation scientist at the Netherlands Institute for Cultural Heritage (ICN). Calcium phytate is an aqueous chelation treatment that inactivates iron (II) and is reported to inactivate further production of iron (II) in ink and paper.[13,14] Phytate is a salt of phytic acid, an organic phosphor compound, a naturally abundant iron chelator and a potent antioxidant, often found in nutrients. Its antioxidant action is thought to derive from the fact that it occupies all of the available co-ordination sites of iron, thus preventing oxidation of iron (II) by peroxides. In a recent study carried out in Slovenia, it was shown that in an ideal scenario, in which the phytate chelated all the iron available in the paper, the life span of the paper at pH 8, could be doubled.[15] This treatment, however, introduces a new chemical into the paper, and one that has not yet been tested extensively on originals. Additionally, following this treatment a white precipitate often develops on the surface of the paper. Although the deposits are removable by brushing, it was considered inadvisable as any such mechanical action on the fragile paper might cause damage.

The other method considered for the Mackay sketchbook is the boiling water treatment, or as referred to in this article, the **simmering water** treatment, based on the 1993 experiments of Austrian conservators and scientists.[16] This treatment has been carried out in the Laboratorio di Restauro, Biblioteca Apostolica Vaticana since the 1970s and in a few other European laboratories.[17] It was during a visit to Poland in 1997 that one of the authors (M.T-B.) first heard, through Ewa Wazynska, a paper conservator and professor in the conservation department of the Warsaw Art Academy, that some important Polish manuscripts were being treated using the boiling water treatment.[18] An article by conservation scientist Władysław Sobucki, published in a Polish bulletin for conservators in 1994, described the treatment applied to an eighteenth century bound music manuscript from the collection of the Warsaw University Library.[19] The most compelling testimonial, however, was provided by Julie Biggs in a paper on her "controversial" boiling water treatment of a sketchbook by George Romney from the Folger Shakespeare Library collection, presented during the 1997 Institute of Paper Conservation (IPC) Conference in London.[20] Biggs, in fact, used alkaline water for her boiling treatment. All these sources confirmed that high levels of the destructive iron (II) ions could be removed from the paper into the simmering wash water and that the concentration of possibly redeposited iron (II) ions in other areas of the support was negligible (below the limit of detection by the analytical methodology used).[21,22] These sources reported that the paper supports were much brighter and flexible after the treatment. Contrary to some reports,[23,24] bleeding of inks was not observed by any of the above mentioned authors during the simmering treatment. Sobucki suggests that to avoid bleeding, it is very important to immerse the treated document in truly boiling water.[19] The high temperature of boiling water seems to set the ink compound, making it insoluble. The water temperature can then be immediately lowered to simmering point to prevent mechanical damage to the document that may be caused by the vigorous action of boiling water.

There are considerable risks associated with both the calcium phytate and simmering water treatments. As aqueous treatments, they may affect inks and paper supports irreversibly in many ways.[25] It is virtually impossible to determine the effect of an elevated water temperature on inks and paper supports through spot tests routinely carried out before aqueous treatments. Yet, the fact that the simmering water treatment has been used extensively, and for a long time, and that it did not introduce a new, little known chemical into the artifact were important factors in our decision to further investigate this technique on the Mackay sketchbook.

The "Simmering" Treatment: Description, Tests and Assessment

Treatment Description and Preliminary Trials

The stability of different inks in the sketchbook was spot tested using water, ethanol and mixtures of water/ethanol. A few inks were slightly soluble in water but not in ethanol, nor in a water/ethanol (3:1 v/v) solution. Then a series of simmering water tests were carried out using some original blank pages from the Mackay sketchbook and some already detached pieces of support with traces of inks on them. Since some drawings in the

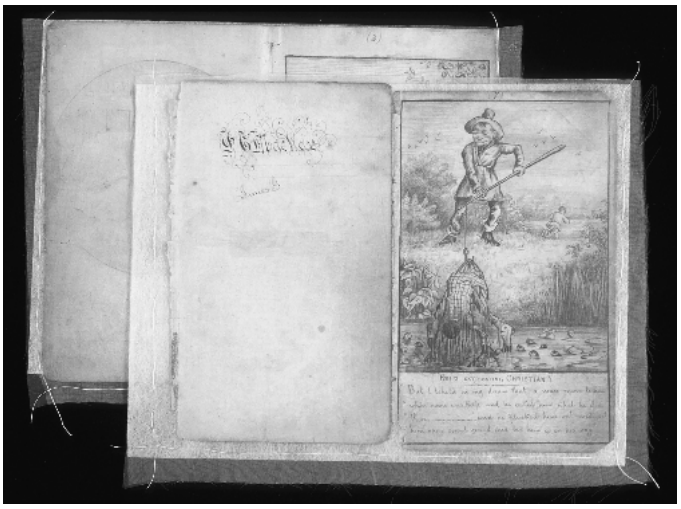
sketchbook were enhanced with pencil lines, it was decided to check the effect of simmering water on a graphite layer as well. Graphite lines were drawn on a sample of smooth, highly calendered paper, similar to the paper in the sketchbook, and immersed in simmering water. After 15 minutes of simmering, there was no visible change in the graphite or the inks when examined under the microscope, and no losses in corroded inked areas in original paper and ink samples were detected. The paper, in all samples tested, appeared much brighter.

Then, a more in-depth investigation of the effects of the treatment on two single pages and one folio from the Mackay sketchbook was carried out at the Canadian Conservation Institute (CCI). The weight of the pages and surface pH of the paper and inked areas were recorded (see **Table II**). Ink and paper were spot tested for fastness to both water and ethanol. The pages were placed on Hollytex supports and sandwiched in envelopes of silk crepeline; the silk crepeline had been pre-washed in boiling water. (When wetted, silk, unlike Hollytex, becomes almost transparent, allowing better viewing and, therefore, control of the condition of the simmered artifact.) The envelopes were stitched along their edges with linen thread. The sandwiched pages were immersed in an ethanol bath for 15 minutes in order to prevent the development of internal stresses caused by the uneven wetting of hydrophobic inked areas and hydrophilic uninked areas upon immersion in water.[25]

Alkaline water was used for simmering, following the procedure described by Biggs.[20] It was prepared by adding saturated calcium hydroxide solution to deionized water, (approximately 2 ml $Ca(OH)_2$ to 2000 ml H_2O) until a pH of 8.5 was reached. Simmering treatments were carried out in stainless steel trays which were heated on burners to 90-95˚C. Two separate treatments were carried out. The folio (3.58 g) was simmered in 1 liter of alkaline water in treatment 1, and the two single pages (3.46 g total weight) were simmered in 2 liters of alkaline water in treatment 2. The sandwiched pages were removed from the ethanol bath and were simmered in one of the two baths for 15 minutes (**Figure 3**).

Assessment of Iron and Other Components Removed During Treatment, and Rate of Removal

The purpose of analyzing the wash water was to determine quantities of acids and other water soluble components that were washed out of the paper, and the rate of removal during simmering. This was achieved using pH measurements, Inductively Coupled Plasma Atomic Emission Spectrometry (ICP/AES) and UV-Visible spectrophotometry (UV-Vis). The pH monitors the acid extraction while the ICP/AES measures dissolved metals in the wash water. Monitoring UV absorbance (at 220nm) is a simple method of determining the relative quantities of soluble compounds in the wash water. Water soluble organic and inorganic compounds absorb UV and visible light, and the amount of absorbance is dependent of the extinction coefficient, unique to each compound, and its concentration. UV-Vis spectrophotometry can be used to determine absolute concentrations of single compounds in solution. Since the wash



(a)

(b)

(c)

Figure 3. The simmering treatment: **a)** page placed on Hollytex support and sandwiched in envelope of silk crepeline stitched along edges with linen thread, **b)** sandwiched pages immersed in ethanol bath, and **c)** pages immersed in a near-boiling water bath and simmered for 15 minutes.

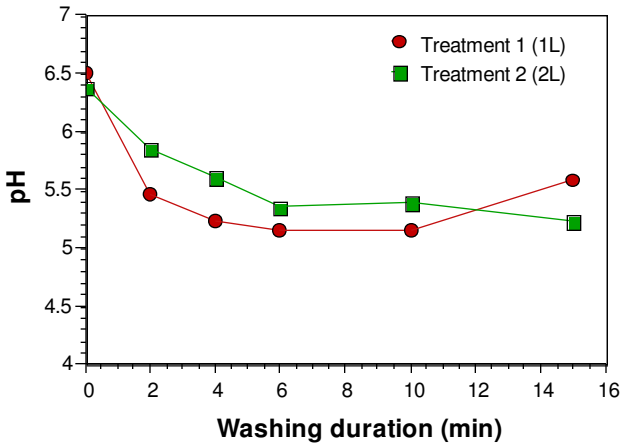


Figure 4. pH Analyses of Water During Simmering Treatment: the rate of acid removal from the papers during simmering.

water contained multiple components at varying concentrations, in this experiment, the technique can only be used to determine relative concentrations, allowing comparison of concentration among wash water samples.

The pH determination of the wash water was done using an Orion Expandable Ion Analyzer EA940 equipped with a ROSS combination pH electrode. The electrode was calibrated using pH 4 and 7 buffers, and the calibration slope was 97.3%. UV/Visible absorbance of the wash water was determined using a Varian Cary3 Spectrophotometer between 190-500nm. Baseline correction was done using deionized water as a reference. The ICP/AES scan for 36 metals was conducted by Caduceon Enterprises, Inc. in Ottawa.

Water samples were collected at 0, 2, 4, 6, 10 and 15 minutes from each of the two treatment baths. The pH, UV-Vis absorbance, and ICP/AES scan results for iron are summarized in **Figures 4, 5 and 6**.[26] These figures show, respectively, the changes in pH, UV/Vis absorbance and iron concentration of the wash water as a function of time. These are indicators of the rate

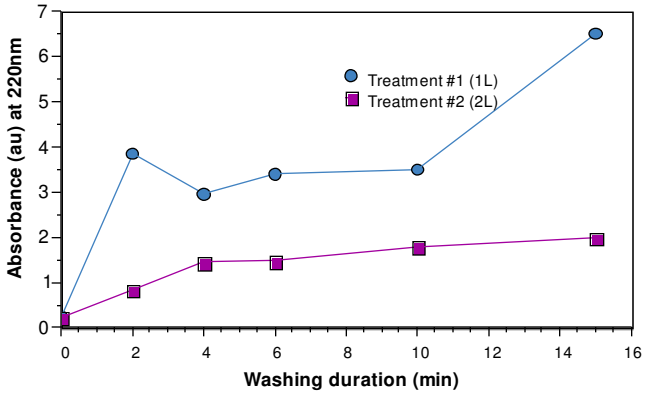


Figure 5. UV-Vis Absorbance (au=absorbance units) of Water During Simmering Treatment: the rate of removal of soluble components from papers during simmering.

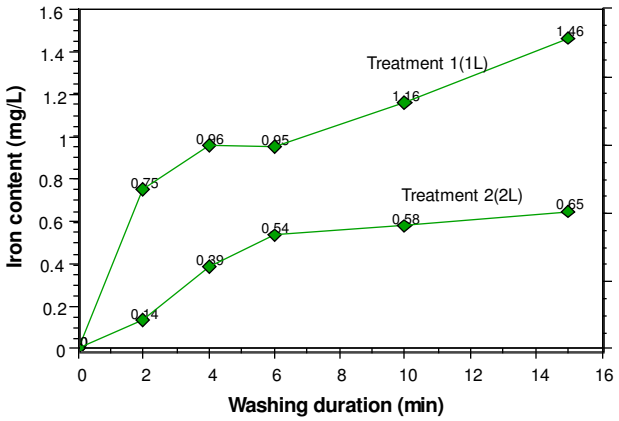


Figure 6. ICP Analyses of Iron in Water During Simmering Treatment - the rate of iron removal from papers during simmering. *Data provided by Caduceon Enterprises, Inc., Ottawa, Ontario.*

of removal of water soluble acids, other soluble compounds and iron salts from the paper during simmering. The differences in concentration between treatments 1 and 2 is due to the difference in paper to water ratio (treatment 1 - 3.58 g/L; treatment 2 - 1.73 g/L). Results from the three techniques show a similar trend: a rapid increase in extraction during the first 6 minutes. For treatment 2, the amount of soluble acids, salts and iron extracted did not increase with time after 6 minutes, and for treatment 1, more was extracted between 6 and 15 minutes.

The differences in the rate of removal between treatments 1 and 2 showed that there may be some variation in the rate of removing dissolved iron from different papers and inks. Fifteen minutes simmering ensures an adequate treatment time with thicker or more heavily sized papers, more hydrophobic or heavily inked areas, or higher concentrations of acids and soluble iron. Results from treatment 2 suggest that if it is considered necessary, a treatment can be terminated earlier. A simmering time of 6 minutes is enough to remove a large amount of the undesirable products and benefit the paper significantly. Overall results from pH, UV-Vis and ICP metal analyses of wash water showed that the simmering treatment was able to remove acids, iron and other soluble salts from the pages.

Testing for the Presence of Iron (II) After Treatment Using Bathophenanthroline Test Papers

Prior to and after the simmering water treatment, the iron (II) content in selected inked areas and in the wash water was checked using a piece of bathophenanthroline iron(II) test paper developed by Han Neveel.[21, 27] In the presence of iron (II) ions, the test paper turns a deep magenta, as it did upon application to untreated inked areas.

Almost the same saturation of colour was obtained when testing the wash water after 15 minutes of simmering. When the same inked areas and some uninked areas of the paper were tested after the treatment, the iron (II) test paper showed no trace of

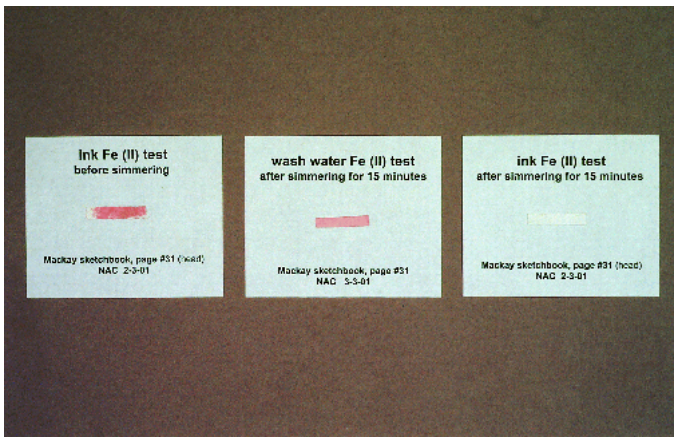


Figure 7. Testing for the presence of iron (II) ions before and after the simmering treatment, using a piece of bathophenanthroline iron(II) test paper developed at the ICN.

Figure 8. James G. Mackay, *The Pilgrim's Progress Colored*, page 5 (left) before simmering and page 22 (right) after a simmering treatment.

colour, indicating an absence of iron (II) ions in both paper and ink (**Figure 7**). When the same areas were re-tested for the iron (II) content a year after the treatment, the iron (II) test paper again showed no trace of colour.

Visual Assessment

After drying, the pages were visually examined. The paper support was much more flexible and appeared much cleaner and lighter in colour (**Figure 8**). The increase in flexibility and brightness may be caused by the removal of calcium filler, as discussed in the next two sections, below. There was no visible bleeding of the ink nor penetration of the ink through the paper fibers onto the back of the page. There was no visible change in the colour of the ink. The dimensions of the treated pages from the MacKay sketchbook were the same when compared to the untreated pages. This was important as one of the concerns associated with the simmering water method is a change of dimensions of treated documents.[23,24] Cracks in the inked areas did not become larger and no additional cracks were found, except for some new small cracks that appeared in the heavily inked areas on the title page.

Calcium Content and Surface pH of Paper and Ink

A high concentration of calcium, determined by ICP/AES, was found in the simmered water (151mg/L in treatment 1 and 54 mg/L in treatment 2). The concentration is above the calcium concentration in the original alkaline water. The removal of calcium from the paper can appear to be alarming as calcium removal has been traditionally associated with destabilization of paper.[28,29] However, it is important to distinguish the effect of different calcium compounds, and not to generalize the benefits provided by simply the calcium cation concentration. Most of the known benefits of calcium carbonate in paper can be attributed to the *carbonate* as a buffer, rather than the calcium cation. In this sketchbook the source of calcium is mainly from the hemi-sulphate filler (see section *Analysis of Paper, Size, Fillers and Inks*), which is not alkaline and does not act as a buffer. This filler is very soluble in hot water. Recent research has found that there is a correlation between the pH of a paper rather than its calcium or magnesium content, and its state of degradation.[30,31] Increasing the pH of the paper will improve the stability—whether this is achieved through simmering or by the addition of a buffering agent such as calcium or magnesium.

To verify that the simmering treatment did increase the pH of the paper, the surface pH of the paper and ink was measured before and after simmering, and again after deacidification in a calcium bicarbonate solution *(see **Treatment** for details on the deacidification)*. The results are shown in **Table II**. Simmering raised the pH of both the paper and the ink from a pH range between 4 and 5 to pH 6. Thus, simmering alone will improve the stability of the paper. Subsequent deacidification in calcium bicarbonate further increased the surface pH to approximately 7. The extracted pH value of the paper is expected to be slightly higher. By neutralizing any remaining acids and adding a small buffer reserve in the paper, deacidification will give further protection against future acid hydrolysis.

Long-Term Stability of Paper Treated with the Simmering Water Method

A follow-up experiment was carried out at the CCI to verify that simmering does not damage paper. The results showed that the treatment did not have a negative impact on the physical and chemical properties of the old and new papers used for testing, but rather benefitted the papers during artificial ageing. Details of this study and analytical results are to be published.[32]

As discussed in the previous section, simmering removes water soluble fillers such as calcium sulphate. This is partly responsible for the increased softness and flexibility of many simmered papers. It is sometimes responsible for an increase in translucency, especially when viewed under transmitted light. The degree of translucency is dependent upon the amount of filler originally in the paper. The difference between simmered and unsimmered papers is even more obvious when viewed under

Table II: Surface pH of Paper and Ink Before and After Simmering and After Deacidification.

	pH Before Simmering	pH After Simmering	pH After Deacidification
Treatment 1: Folio - page#10/19 (3.58g)			
Paper (p.10)	5.1	6.2	7.2
Ink	4.5	6	6.6
Paper (p.19)	4.7	6.1	7
Treatment 2: Page#33 (1.73g)			
Paper	4.8	6.2	7.1
Ink	4.1	6.1	7.3
Treatment 2: Page#34 (1.73g)			
Paper	5	6.3	7.2
Ink	3.9	6	6.8

UV illumination, as UV-absorbing filler such as calcium sulphate is removed by simmering. However, the loss of filler may not be visually noticeable, as was the case for the Mackay sketchbook. The long term physical and chemical impacts of the loss of a filler in paper still need to be fully studied, although results from the follow-up experiment previously mentioned[32] indicate no adverse physical and chemical effects after artificial ageing of an 18th century book paper which also lost calcium filler during simmering.

Treatment

The rest of the pages from the Mackay sketchbook were treated at the National Archives by Maria Trojan-Bedynski, paper conservator, and Frida Kalbfleisch, book conservator, in March 2001, in the same manner as described in the above section, using 2L of alkaline water, and simmering for 15 minutes. After simmering, the pages were transferred onto a suction table, removed from the supporting silk envelopes, and allowed to partially dry and flatten while misting with ethanol. The use of ethanol at this point in the treatment speeds up the drying process, reduces the risk of ink bleeding and prevents the ink-corroded paper from breaking.[20,25] Recent experiments indicated that humidification methods using a cold chamber or Gore-Tex sheets should be avoided to prevent the migration of iron (II) ions across the paper surface.[22] This raised the concern that migration of any remaining iron II ions may occur if after treatment the wet pages were left to air dry. Instead, the pages were placed between layers of Hollytex and blotters under boards and weights for complete drying and flattening (**Figure 9**).

A saturated calcium bicarbonate solution has recently been recommended as a deacidification agent for iron gall ink corroded papers. When treated with this solution, ink and paper never reach values above pH 8.5, which is critical since iron gall inks may fade or change colour when exposed to a pH above 9.[8, 33, 34, 35] The sketchbook pages were deacidified by immersion in a saturated calcium bicarbonate solution for 30 minutes, after pre-soaking in ethanol. The calcium bicarbonate solution, prepared according to instructions obtained from Han Neevel,[36] had a pH value of 6.0. As can be seen in **Table II**, after deacidification, the pH ranged from 6.5 to 7.3 in uninked areas, and 6.4 to 7.1 in heavily inked areas.

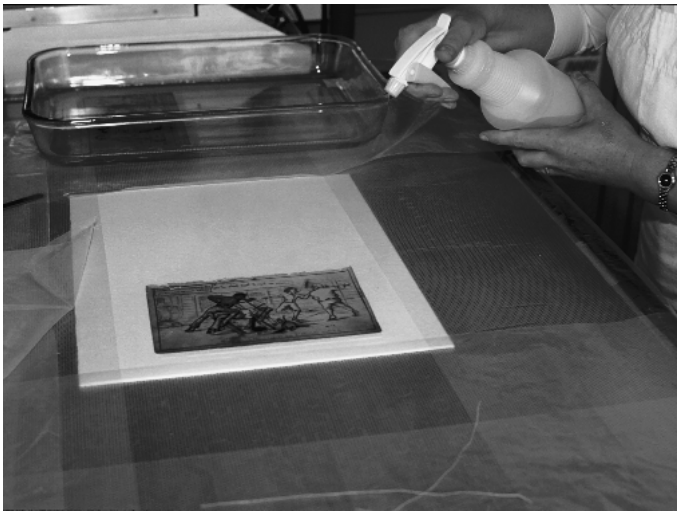
The treated pages were resized with a 0.5% gelatin solution. It has recently been reported that gelatin has beneficial effects on paper corroded by iron gall ink.[37] Like all proteins, gelatin possesses mild complexing properties, thus being able to bind a certain amount of soluble iron (II) ions. It is also a good consolidant.[37] Small tears in the paper support were repaired from the verso with an ultralight *kozo* tissue (RK-0); wheat starch paste was used as an adhesive. Losses in the paper support were filled with a paper pulp on a suction table. Most pages in the volume had breaks or were separated along the ink line border close to the gutter. To repair them locally would create too much swell in the rebound book, so it was decided to line all the pages with the same *kozo* tissue. Methylcellulose was used as an adhesive.

Following the paper treatment and during the rebinding of the pages, the focus was on creating flexibility in the spine. The text block was resewn on linen tapes and the book was rebacked with a hollow spine. This would result in a greater ease of opening, as well as pages that would turn without any stress to the paper along the gutters. The worn boards were consolidated but not modified, as it was deemed preferable to show the wear and tear the book had received due to heavy use.

Conclusion

The Mackay sketchbook, which was suffering from iron gall ink corrosion, was treated using the simmering water treatment to prevent this unique book from perishing and to allow it to be further studied. It is hard to imagine any other treatment that could have fulfilled the conservation requirements demanded of this severely damaged artifact. We chose the simmering treatment over the calcium phytate method because it has a longer history of use on original historical documents, it does not add a new compound to the document, and because no surface deposits requiring mechanical removal would be left on the paper. One of the main drawbacks of the simmering treatment as compared to the phytate treatment is that it does not stabilize the iron gall ink against future corrosion. The potential risks associated with the simmering water treatment were, admittedly, serious. Any aqueous treatment for artifacts with degraded inks has disadvantages, as discussed earlier. The use of very hot water makes the process even more unpredictable. Bleeding and physical losses of ink flakes from the movement of air bubbles were additional potential risks. We were aware of these risks and carried out as many analyses, tests and trials as we could. A search of available literature, personal communications with other conservators and conservation scientists, and information gathered while attending the Iron Gall

(a)



(b)

(c)

Figure 9. The simmering treatment: **a)** after simmering, pages transferred onto suction table and removed from silk envelope, **b)** initial drying on suction table while misting with ethanol, **c)** pages placed between blotters for complete drying and flattening.

Ink Meeting in Newcastle in September 2000 all increased our confidence and courage during the decision making process.

A component of the phytate method was incorporated into our treatment: in order to prevent an unacceptable rise of pH levels associated with solutions containing magnesium, we chose calcium bicarbonate solution as a deacidification agent. Although this solution did not leave an alkaline reserve in the paper, we thought the paper support was sufficiently stabilized. The sketchbooks will be kept in the climate controlled storage at the Gatineau Preservation Centre (constant conditions of 50% RH and 18°C, free of air pollutants, visible light and UV radiation). Since the simmering water treatment washes out iron (II) ions but offers no future protection against corrosion, the lower storage temperature, 18°C, will also help to slow down oxidative degradation.

The research and effort that went into the determination of a suitable treatment for this sketchbook, taking into account levels of acceptable risk, were well worth the results. The paper support is much brighter and very flexible, even in areas that were previously extremely brittle and impossible to handle without the risk of causing more damage. The dimensions of the pages have not been changed as a result of the treatment. The inks' colour has not changed, and inks did not bleed nor penetrate to the opposite side of the paper support. The tests for iron (II) content indicate that large quantities of harmful iron (II) ions were removed from the inks into the wash water solution. This is supported by the results of wash water analysis. The simmering treatment did result in removing large quantities of the calcium sulphate filler as well, but this filler has no buffering capacity and does not contribute to the stability of the paper. The treatment caused the surface pH of the paper to increase, which is a good indicator of increased stability. The results of a follow-up experiment investigating effects of simmering on the chemical and mechanical properties of paper are also reassuring. The greatest proof, however, is the book itself. Today, the James G. Mackay sketchbook can be handled and copied safely for the first time since it was acquired by the National Archives (**Figure 10**).

Acknowledgements

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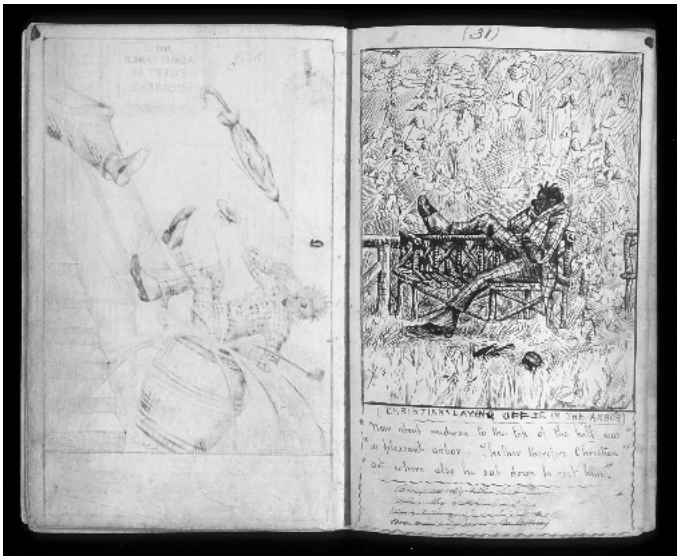

Figure 10. James G. Mackay, *The Pilgrim's Progress Colored*. Restored book open on page 31. Library and Archives Canada, Accession No. 1988-033.

photodocumenting the simmering treatment of the Mackay sketchbook; Agata Bedynski for her comments and proof-reading the first draft of this manuscript; Betty Jaquish, Paper Conservator of Library and Archives Canada for comments and editorial advice and for preparing digital images.

Materials

Bathophenanthroline iron II test papers: University Products of Canada, 2957 Inlake Court, Mississauga, Ontario, L5N 2A4, Tel:1-800-667-2632 or 905-858-7888.

Calcium carbonate: Fisher Scientific Limited, 112 Colonnade Road, Nepean, Ontario, K2E 7L6, Tel: 613-228-6387 or 1-800-267-1000, http://www.fishersci.ca/.

Calcium hydroxide: Fisher Scientific Limited.

Ethanol: Fisher Scientific Limited.

Gelatin: Talas, 569 Broadway, New York, New York 10012, USA, Tel: 212-219-0770, info@talasonline.com.

Hollytex (non-woven, spun bonded polyester fabric): Carr McLean, 461 Horner Avenue, Toronto, Ontario, M8W 4X2, Tel:1-800-268-2123, cmclean@carmclean.ca.

Methylcellulose METHOCEL A4M: Dow Chemical Company, Midland, Michigan 48674, U.S.A.

Silk Crepeline: Talas.

Ultralight *Kozo* Tissue (RK-O): Paper Nao, 4-37-28 Hakusan Bunkyo-Ku, Tokyo 112-0001, Japan, Tel: 03-3944-4470.

Wheat starch paste: University Products of Canada.

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