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# The Treatment of Archaeological Papers Affected by Iron Corrosion Using Calcium Phytate

**Amanda Gould**

Canadian Museum of History, 100 Laurier Street, Gatineau, Quebec K1A 0M8, Canada; amanda.gould@historymuseum.ca

*The calcium phytate/calcium bicarbonate treatment method developed for delaying iron gall ink corrosion on paper was applied to the paper components of two archaeological artifacts: a soup tin label that was unearthed from the site of a storehouse built on Dealy Island by British sailors searching for the lost Franklin Expedition, and a pocket watch that was retrieved from the waters of the St. Lawrence River after the wreck of the RMS* Empress of Ireland*. The soup tin label showed highly three-dimensional rust accretions and staining with both stained and unstained areas of the paper testing positive for free iron(II) and iron(III) ions. The pocket watch label showed significant yellow-orange coloured staining throughout the remains of the primary paper support and also tested positive for both iron(II) and iron(III) ions. Although neither artifact was inscribed with iron gall ink, the calcium phytate/calcium bicarbonate treatment was applied to each of them to combat the degradation of paper that results from iron(II)-catalysed oxidation and acid-catalysed hydrolysis of cellulose, while at the same time maintaining the visual cues to the archaeological context of the two objects. The treatment decision-making process and treatment steps are discussed.*

*Le traitement au phytate de calcium et bicarbonate de calcium développé pour retarder la corrosion des encres ferro-galliques a été appliqué aux composantes de papier de deux artefacts archéologiques: une étiquette de boite de conserve découverte sur le site d'un entrepôt construit sur l'île de Dealy par des marins britanniques à la recherche des navires perdus de l'expédition Franklin, ainsi qu'une montre de poche retrouvée dans les eaux du fleuve Saint-Laurent suite au naufrage du RMS* Empress of Ireland*. L'étiquette de la boite de conserve avait d'importantes taches et concrétions de rouille, et tant les zones tachées que non-tachées réagissaient positivement au test pour la présence d'ions de fer(II) et de fer(III). L'étiquette de la montre de poche avait de nombreuses taches jaune-orangées sur la quasi-totalité du support papier et réagissait également positivement à la présence d'ions de fer(II) et de fer(III). Bien qu'aucun des artefacts n'ait été libellé à l'encre ferro-gallique, le traitement au phytate de calcium et bicarbonate de calcium a été appliqué à chacun d'eux pour lutter contre la dégradation du papier qui résulte de l'oxydation et l'hydrolyse acide de la cellulose, catalysée par la présence d'ions de fer(II), tout en maintenant l'aspect visuel témoignant du contexte archéologique des deux objets. Le processus de prise de décision du traitement et les étapes de traitement sont discutés.*

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## Introduction

Two archaeological artifacts, a John Henry Gamble Preserved Provision Manufacturer label from a tin of Ox Cheek Soup and fragments of a label from the interior of an Ingersoll pocket watch were examined and treated in the context of the preparation for two Canadian Museum of History (CMH) exhibitions: *Northwest Passage and Sir John Franklin*, displayed at the Prime Minister's Office in 2013 and early 2014, and *Canada's Titanic – The Empress of Ireland*, an exhibition which opened at the CMH on May 29, 2014 to correspond with the 100th anniversary of the sinking of the steamship.[1] Both objects are made of paper that had been in contact with metal in a wet environment: mixed salt and fresh water in the river in the case of the watch label, and seasonal cycles of precipitation and thaw in the case of the provision label. Both objects thus exhibited staining, accretions and losses directly attributable to the metallic, specifically iron, components of the composite artifacts.

Both objects are present in the National Collection at the CMH because of an increase in accessibility to Arctic and underwater archaeological sites that occurred in the early 1960s.[2] Coincidentally, though the two objects would not be accessioned until the years 2000 and 2012 respectively, the Ox Cheek Soup label was found by a visitor to the Dealy Island site in the same year, 1964, that the wreck of the *Empress of Ireland* was rediscovered by sport divers in the St. Lawrence River. It was also only by coincidence that the two labels were to be treated in the CMH paper conservation lab at roughly the same time, and that very similar treatment protocols were established for the two paper objects.

## History of the Artifacts

### *Ox Cheek Soup Label (CMH Catalogue Number 2000.98.8)*

The John Henry Gamble[3] provision manufacturer label was collected from a location dubbed "Captain Kellett's Storehouse", a depot built on Dealy Island, southeast of Melville Island in the Canadian Arctic.[4] The storehouse was built in the spring of 1853 under order of Captain Henry Kellett by the crew of HMS *Resolute*, a British Navy ship there in search of the lost Franklin Expedition (1845–1848). A drystone building with a canvas roof, the cache "was originally stocked with provisions and supplies sufficient to sustain 66 men for 210 days on full royal Navy allowance."[5] About 15 metres east of the cache is a midden[6] containing refuse, including bottle glass, crockery and opened tin cans, deposited there by the crews of the two ships under Captain Kellett's command.[7]

It is not known from exactly what part of the small site the CMH label originated, as the artifact's donor, a Crown

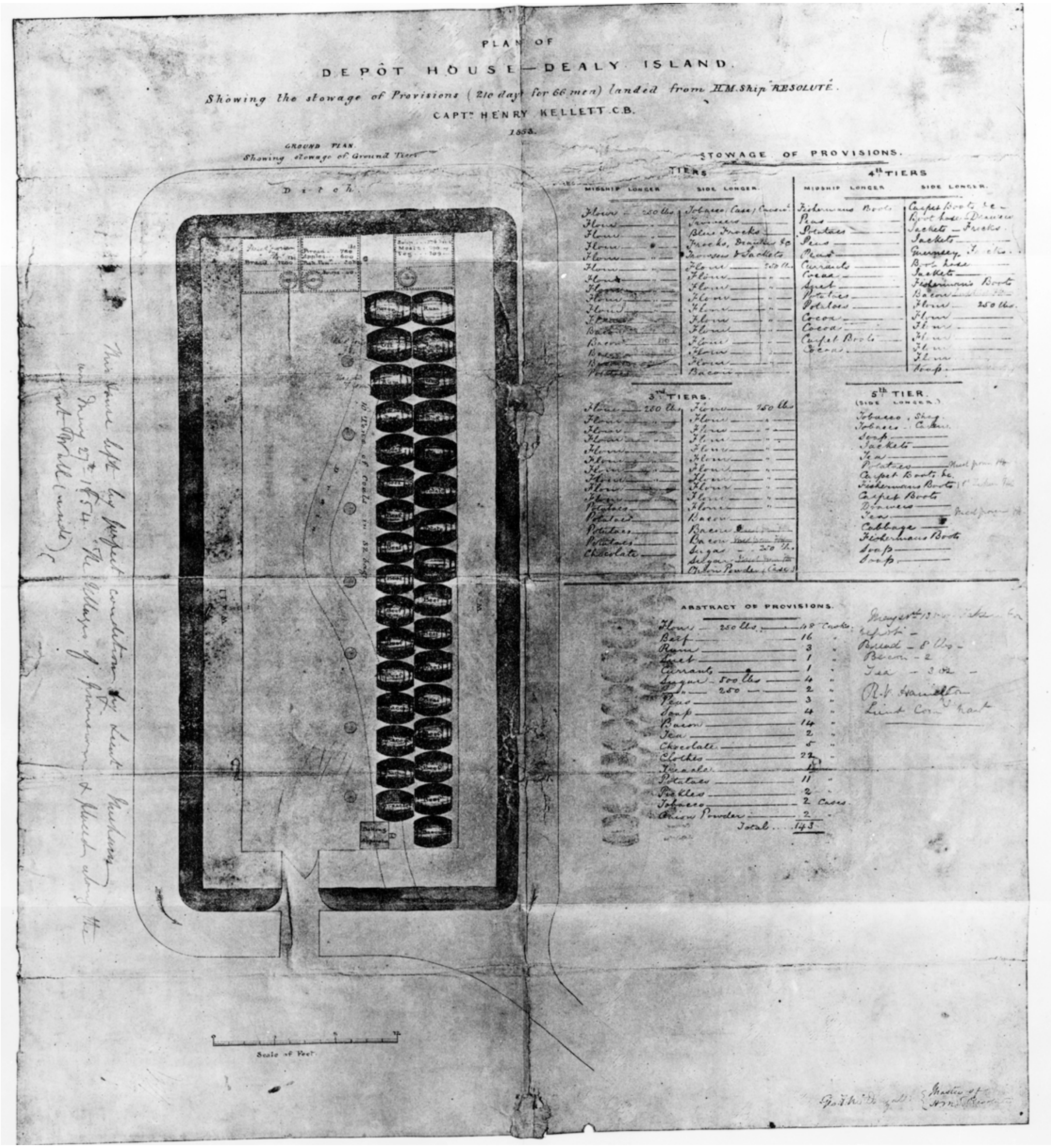


**Figure 1.** Plan of Depot House - Dealy Island showing the Stowage of Provisions (210 days for 66 men) landed from H.M. Ship "Resolute" Captn. Henry Kellett C.B. 1853. Note the reference to "Soup" in the 4th Tiers (bottom), 5th Tier (bottom) and Abstract of Provisions (middle) lists and in the box directly above the casks on the Ground Plan. [Facsmile of plan found by Capt. Bernier in 1908.] [Cartographic material.] Copyright expired. Microfiche NMC15283.

Geologist, told the museum that "he just happened upon [it] in the course of his travels" in 1964 while doing field work for the Geological Survey of Canada.[8] It could be that it was attached to a tin that was deposited with its contents intact in the cache for the use of future European explorers (**Figure 1**). It might also have been attached to an empty tin used to construct part of the north wall of Kellett's storehouse, or have originated from the large midden.

The discovery and removal of the label from the site would have occurred prior to the existence of Federal or Territorial regulations regarding the removal of archaeological materials. It was removed many years prior to the assessment, excavation and preservation of the site carried out by a team from the Prince of Wales Northern Heritage Centre (PWNHC) in Yellowknife led by Robert R. Janes, and which included Charles E.S. Hett of the Canadian Conservation Institute (CCI).[9] This was a concentrated effort in the field seasons of 1977 and 1978 that resulted in the majority of the objects found above the permafrost being sent to the CCI for treatment, and others sent to the PWNHC (see **Appendix I** for a list of tin labels in the collection of the PWNHC). The remaining artifacts were left in situ and covered by an insulated floor devised and installed in the storehouse by the team for the purpose of protecting the site, while accommodating future visits to it by either human or bear.[10]

*Pocket Watch Label (CMH Catalogue Number 2012.21.198)*

The watch was part of the 2012 accession by the CMH of approximately five hundred objects and archival documents related to the sinking of RMS *Empress of Ireland*, a luxury vessel launched by the Canadian Pacific in 1906 to handle the burgeoning traffic in transatlantic travel. At 1:55 in the early morning of May 29, 1914, when most of the ship's passengers had stowed their small personal items near their bunks and gone to sleep, the *Empress* was rammed amidships by the *Storstad*, a Norwegian collier en route to Montreal from Sydney, Nova Scotia. Navigating through a thick fog off the coast of Sainte-Luce-sur-Mer, the two ships had lost sight of one another and each was unable to keep the other vessel apprised of its movements.[11] The *Empress* then listed strongly to starboard, a gaping 4 metre wide by 14 metre high puncture in her hull. Within 14 minutes of the collision the ship had sunk, killing an estimated 1012 of the 1477 people aboard.[12]

Many of the artifacts from the *Empress* wreck were brought to the surface in the mid-1970s when the *Empress*' location on the bed of the St. Lawrence River, rediscovered in 1964, was a popular sport-diving destination. It was not until April 15, 1999 that the site became subject to the provisions in the Quebec Cultural Property Act that prohibit divers from removing material or otherwise damaging the wreck and its contents.[13]

In addition to artifacts salvaged from the ship, the CMH accession includes primary and secondary archival materials on the subject of the *Empress* and other vessels such as her sister ship, the *Empress of Britain*, as well as the similarly ill-fated *Titanic*. Materials on the subject of the Canadian Pacific Railway (CPR) Steamship Company and the passengers of the *Empress of Ireland*'s last voyage are among the many and varied items amassed over several decades and then deposited with the CMH by the collector who, as a sport diver, descended to the wreck many times after its rediscovery.

According to the markings on the inside verso of the case, the pocket watch was manufactured in the U.S. by "ROBT. H. INGERSOLL & Bro." The brothers, Robert and Charles, were two farm boys from Michigan who moved to New York in the early 1880s to make their fortunes. They specialized in the manufacture of dollar items, such as toy typewriters, retailed through a mail-order business. They first advertised a pocket watch in their catalogue in 1892.[14] A year later the Ingersolls were selling watches at the World's Columbian Exposition in Chicago.

In 1896, Ingersoll introduced a dependable watch, the Ingersoll Yankee, which no matter the retail location was sold for the price of one dollar.[15] By 1914, the "Watch Makers to the American People" had sold 35 million of their "dollar" watches using the slogan "The Watch That Made the Dollar Famous." A dollar being roughly equivalent to a working man's daily wage in this period in the U.S., the CMH's *Empress* watch could conceivably have belonged to any of the *Empress*' passengers, though the pocket watch was primarily a man's accessory at that time. "Dollar" watches may have been a common possession even among crew members or those with third class passage, as "the dollar price put the possession of a real timepiece within the reach of multitudes who were engaged in forms of activity wherein a delicate timepiece would be apt to get out of order."[16] Recognized as having had a revolutionary impact in widening the market in personal time pieces, an Ingersoll Yankee "dollar" watch, ironically, is not considered by collectors today to be worth the price of repair.

## Condition Prior to Treatment

*Ox Cheek Soup Label (CMH Catalogue Number 2000.98.8)*

Measuring 8.4 cm high by 12.4 cm wide, the label (**Figure 2**) is printed in black ink on Western machine made wove paper with a matte surface. The colour of the paper approximates PCAPSB BEIGE (I) locally discoloured, the thickness approximates PCAPSB MEDIUM (2), and the surface texture approximates PCAPSB SLIGHTLY TEXTURED (I).[17] The label is inscribed as follows:

> 137, Leadenhall-Street, London / John Henry Gamble / Patent Preserved Provision Manufacturer. / 137 Leadenhall-Street, / Opposite the East India House, LONDON / OX CHEEK SOUP. / Directions – Cut round on the top close to the edge with a chisel and hammer, having previously scraped off the paint to prevent it mixing with the contents. / Empty the Soup into a saucepan and stir it gently to prevent it getting burned while warming. Some prefer heating it in the Canister and opening afterwards. This dish is admirably adapted for making Pies; or if there be more gravy than required, it can be used as Soup. Perhaps a little more seasoning may be added. / Manufactory – 4, 5, & 6, Morrison's Quay, Cork.

The label as collected was encapsulated in polyester film. The enclosure was removed in order to complete a condition report, which was done using normal and transmitted light, as well as 10x magnification. Examination revealed overall loose surface soiling accompanied

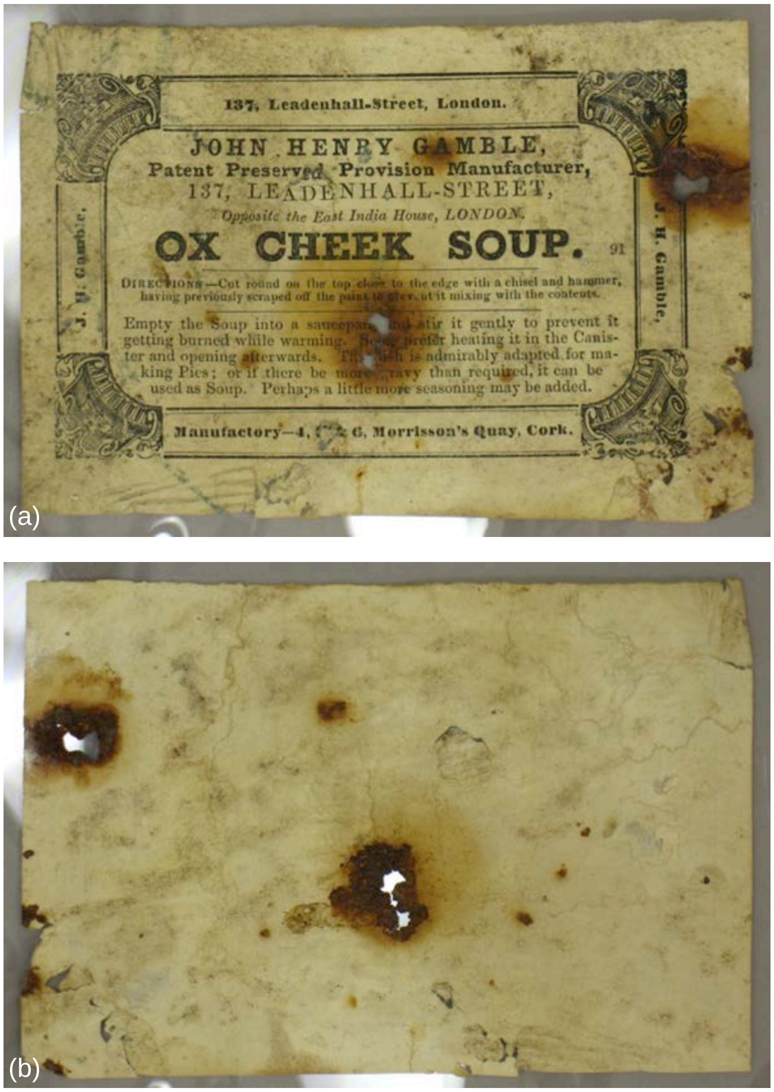


**Figure 2.** Ox Cheek Soup label before treatment, recto (a) and verso (b). Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

by local areas of ingrained dirt. Colour change in the form of overall yellowing was considered probable. Local colour change included numerous stains of differing colours and sources, most notably areas of orange coloured staining associated with rust accretions. Accretions included highly three-dimensional rust attached to the verso of the label in addition to green and red coloured media (**Figure 3**). Some of the accretions on the label seem to indicate that the soup tin was painted green (**Figure 4**). Evidence of previous active iron corrosion was visible under magnification on the accretions in the form of tell-tale hollow shells of dark red corrosion bubbles.[18] The tin itself would have been composed of wrought iron[19] sheets forming a core that was plated with tin and joined with a lead-tin solder.[20] Planar deformations included numerous small creases and associated draws.[21] Holes, losses, cuts and tears were apparent, as was surface damage that included areas of abrasion on both the recto (**Figure 2a**) and verso (**Figure 2b**) of the label.

The verso showed skinning possibly associated with the removal of the label from its tin can. Adhesive residue was not observed, though it is assumed that the label was at one time adhered to the exterior of a tin or its paper wrapper. It is not known at what point the label was separated from its tin of soup.

Standard photo-documentation, including overall recto and verso images as well as photomicrographs of various accretions, was completed prior to treatment. Surface pH, bathophenanthroline colour indicator and solubility tests were all recorded on laboratory testing summary forms. The average pH of the paper, tested[22] on both the stained and unstained areas, was 4.0. Tests for free iron(II) ions

**Figure 3.** Accretions on soup label. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

**Figure 4.** Accretions on the label indicate that the soup tin was painted green. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

using bathophenanthroline colour indicating test papers were positive for both the stained and the unstained areas of the paper, showing an average of 25 units[23] of free iron(II) ions according to the CCI colour comparison chart.[24] Tests of the used test strips with ascorbic acid showed a maximum of 25 units of free iron(III) ions. Solubility tests showed that the black printing ink and other stray media on the label were *not* soluble in water, ethanol, or the chelation and deacidification solutions proposed for use during treatment.

*Pocket Watch Label (CMH Catalogue Number 2012.21.198)*

Prior to treatment, the pocket watch label consisted of one larger, kidney-bean shaped fragment, found loose within the cover of the watch (**Figure 5**), and numerous smaller fragments, including one reading "THIS" that remained adhered to the interior of the cover. Assuming that the original size corresponded to the interior dimensions of the cover of the pocket watch, approximately half of the label was missing. The thickness of the larger fragment is between PCAPSB MEDIUM (2) and MODERATELY THICK. Most of the smaller fragments were found inside the mechanism cavity of the watch. The paper manufacture of the label is likely wove; however, colour and texture remain difficult to determine as better than ninety percent of the paper surface is orange in colour as a result of liquid staining associated with the corrosion product formed by the watch components. The pocket watch, though not made primarily of iron, contains iron[25] screws, rivets and spring. The largest fragment had many small tears and folds. The paper surface was abraded in some areas such that it appeared as a mat of fibres.

The text on the largest label fragment read as follows:

GUAR_/ TO KEEP GOO_/ YEAR AND IF_/ ___TO DO_S/ BY US FREE_/ OPTION FOR_/ WRAP CAREFU_/ RETURN POST_/ YOUR NAME_/ LID ONLY/ ______SIGNED BY/ DEALER WHEN SOLD/ DEALER SIGN NAME HERE [________]/ ROBTH_/ 315_AVE., NE/ AFTER FIRST YE_/ IF WATCH NEEDS REPAIR_/ IT TO US AND WE WILL I_/ YOU WHAT REPA_/ WILL COST/ WAT______AND DATE IS/

Prior to the treatment of the label, the watch itself was examined and treated by conservator Kristen Stockstill.[26] Inscriptions on the verso of the watch, transcribed by Stockstill after performing light cleaning, read:

JAN 29 01 APR 11 05 JUNE 4 07
JUNE 20 09 MAY 24 10 NOV 12 12
MADE IN USA/ ROBTH INGERSOLL & BRO NEW YORK
32121779

The first two lines of characters, machine-etched into the metal, are likely patent dates. The last seems to indicate that the most recent patent renewal before the manufacture of the watch occurred on or was valid until November 12, 1912. However the serial number, "32121779," etched below the patent dates suggests, according to one source,[27] a date of manufacture in 1911. The *Empress* watch was thus likely only about two years old when it boarded the ship on the person of a passenger or crew member.

The surface pH, bathophenanthroline colour indicator and solubility tests for the watch label were all recorded on laboratory testing summary forms. The average pH for the three locations tested was 4.0. The largest fragment was tested for the presence of iron ions and the bathophenanthroline iron(II) colour indicating test papers gave positive results for free iron(II) and iron(III) ions in the paper (**Figure 6**). Solubility tests showed that the medium of the printed text was *not* soluble in the solutions proposed for use during treatment.

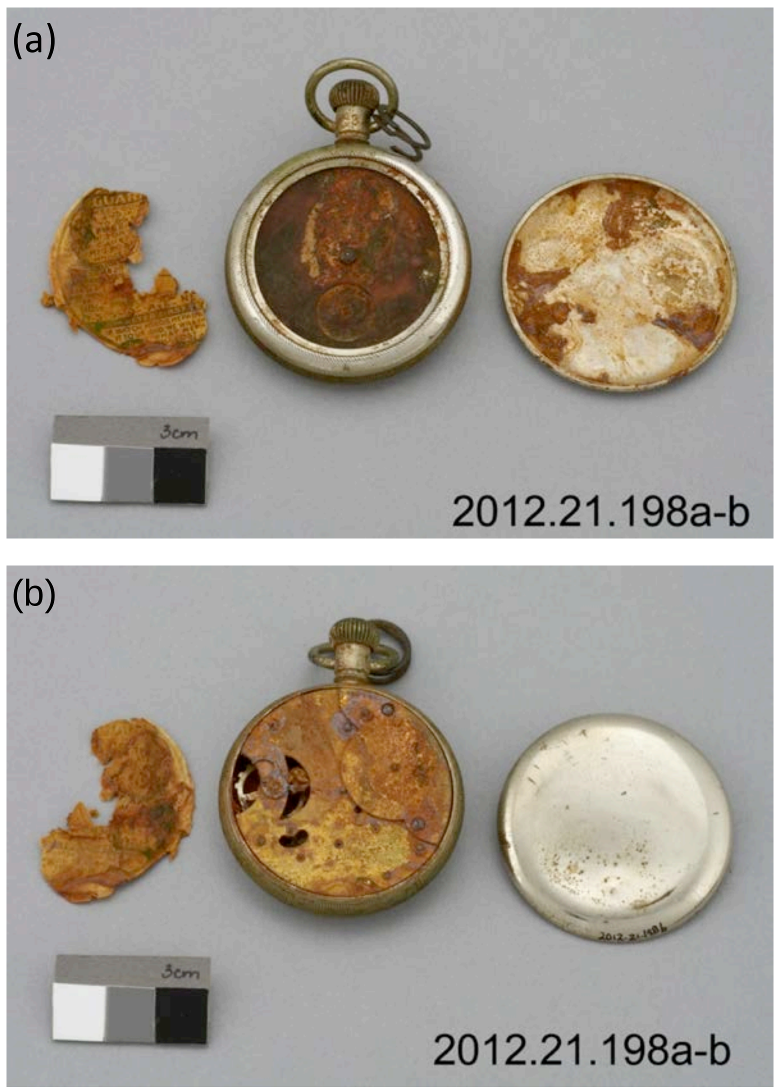


**Figure 5.** Watch and label fragment as acquired, recto (a) and verso (b). Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

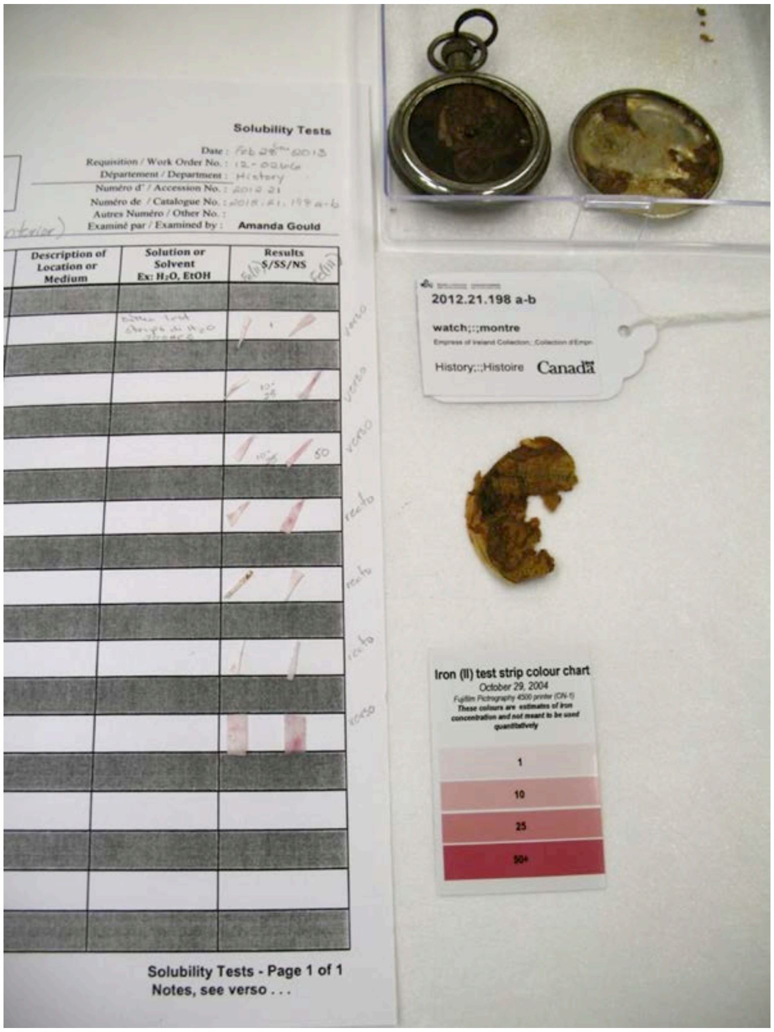


**Figure 6.** Results of bathophenanthroline colour indicating paper tests for free iron ions on the pocket watch label. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

## Treatment Decision Making

A conventional paper conservation treatment goal for objects similar to the two labels discussed in this article is to reduce staining to the paper substrate, especially staining that obscures an image or printed text. Past experimental conservation treatments, performed circa 1980 for Arctic materials removed from Dealy Island and other Franklin sites, involved reduction of the iron staining through bleaching with sodium hydrosulphite (also called sodium dithionite) or, in older cases, using oxalic acid.[28] In projects managed by the Antarctic Heritage Trust (AHT) preserving Antarctic exploration expedition bases, food tin labels, if they could be safely removed from their tins, have been treated according to a conventional paper conservation protocol: washing, stain reduction (using EDTA or oxalic acid, depending on ink solubility),[29] deacidification, tear repair and lining, and resizing.[30] The current practice for Antarctic materials treated by paper conservators employed by the AHT also involves reducing iron staining on paper labels from food tins through bleaching with sodium hydrosulphite. In the case of the AHT tinned provision labels, this makes sense "because the intent of [their] collection within the expedition bases is to show the items as they were when the last users walked away from the building[;] evidence of deterioration occurring since then is not deliberately kept."[31] These Antarctic objects are, however, numerous. Only some of the labels are considered rare because they occur with lesser frequency within the larger collection of tinned food cans.[32]

By comparison, the two CMH labels are both rare. It was unusual that tinned provisions provided to 19th century Arctic expeditions searching for the Northwest Passage were labelled using paper labels. It was far more common that the contents of tins were indicated by a metal label or lettering that had been applied with stencil and paint.[33] The Ox Cheek Soup label is the only example in the CMH collection of a relatively whole paper provision tin label from the Henry Gamble Preserved Provision Manufacturer.[34] There are only two examples in Canadian museum collections of Gamble Ox Cheek Soup labels associated with Arctic exploration.[35] Other possible examples remain in situ, such as the tins that remain entombed in ice in Kellett's Storehouse.[36] Though there are many examples of Ingersoll "dollar" watch labels extant, owing to the one-time success of the company and its product, only one in the CMH's collection is associated with the wreck of the *Empress of Ireland*.

The immediate treatment goal for these two artifacts was stabilisation for exhibition. Although housed in a controlled museum storage environment since their acquisition, the two artifacts were to be included in exhibits that would travel outside the CMH, introducing the possibility of exposure to environmental changes during transport or while on exhibit at other venues. Simple housing options were considered but deemed insufficient, and so a conservation treatment intervention was pursued. After considering the unique nature of the CMH labels and after a discussion of the possible treatment approaches for them, both the responsible archaeologist and curator preferred to let the labels retain the visible markers of their archaeological context, i.e. the rust staining and accretions. The challenge was then to devise treatment protocols aimed at physically and chemically stabilising the two paper labels while retaining these markers.

In devising treatment protocols for the labels, aqueous interventions appeared to offer the most satisfactory outcome. In the case of the pocket watch label, which consisted of many small, highly distorted fragments, the introduction of moisture for the purpose of relaxing and flattening the fragments of the label was a desired treatment step. In the case of the soup tin label, the holes in the support, surrounded as they are by fragile, accretion-bound areas of paper, as well as the overall "cracked" appearance that is most apparent on the verso, made local tear repair seem an insufficient and potentially problematic method of physical stabilisation. In both cases the author felt that overall lining was called for, despite the challenges posed by the metal accretions on the verso of the soup tin label. If the accretions were to be maintained, then they should remain visible. Avoiding the application of moisture through the use of, for example, a solvent reactivated lining tissue might not have allowed for the sought-after visibility of the accretions and surrounding paper of the verso of the label.[37] In addition, as reported above, pH testing indicated that both labels were acidic and called for deacidification.[38] Though non-aqueous deacidification is possible, aqueous methods are in general considered preferable due to the perception that better penetration of the alkaline solution can be achieved. The use of moisture was therefore considered preferable for several reasons and also posed little risk to the media: there was no evidence of the printed lettering on the labels having bled during previous exposures to water, and solubility testing confirmed that the media remained insoluble.

Although aqueous treatment steps were considered preferable, there existed one more significant challenge to including them in the treatment protocols. Due to the presence of the iron stains and accretions, both labels had been tested and, as indicated above, yielded a positive indication of the presence of free iron(II) and iron(III) ions (**Figure 6**). It is understood that the introduction of moisture allows iron(II) ions to migrate to previously unaffected areas of a paper substrate and to catalyze detrimental reactions in the cellulose chains.[39] Conventional stain removal would have dealt with these free ions through washing, reduction bleaching and chelation. Since stain removal was not a treatment goal, a step was needed that would provide another form of chelation that would not result in a significant loss of the visible markers of iron ion presence, while at the same time permit stabilisation using aqueous methods.

The treatments of the two labels thus had the same goals as those in the treatment of iron gall ink inscribed documents.[40] According to Selwyn, the presence of iron(II) ions is an important factor in the degradation of papers inscribed with iron gall ink.[41] Presumably the presence of such free ions in the labels would be a factor in the degradation of the two paper labels affected by iron corrosion. To combat the forms of degradation to cellulose caused by the free iron ions in so many iron gall ink formulations, the Instituut Collectie Nederland (ICN)[42] calcium phytate protocol was developed[43] over the course of several years and is now in fairly common use amongst conservators. Applying the calcium phytate protocol on the labels might therefore provide the same protection afforded documents inscribed with iron gall ink, and allow for the desired aqueous treatment steps.

The idea of using the ICN calcium phytate protocol led to the use of the “Treatment Options for Iron Gall Ink on Paper” flow chart published in volume 37 of the *Journal of the Canadian Association for Conservation*.[44] In treating the label like an iron gall ink inscribed document for the purposes of treatment planning, a comparison with the ICN Condition Rating System[45] was made. A Condition Rating of 4, which indicates physical damage and loss of substrate,[45] was selected for both labels due to the existing loss of paper support in the case of the watch label and the two areas affected by accretions on the soup tin label, and the risk of further losses of the paper, the accretions, or both. Using the treatment options flow chart helped to solidify the potential process in the mind of the author. Though the risks posed by mechanical manipulation of artifacts of this rating need to be considered before undertaking a wet treatment, the small size of the labels allowed for mitigation of the risks through careful handling. The author thus felt comfortable proceeding with the treatments despite a condition rating that might deter a conservator from treating other artifacts in this manner. As the printing inks on the papers proved not to be soluble in water or a combination of water and ethanol, the ICN calcium phytate protocol would allow the author to achieve the treatment goals. It was hoped and intended that the combination of chelation and deacidification delivered through the application of the ICN protocol would address issues posed by the free iron ions and low pH while allowing for the preferred methods of relaxation, realignment and physical stabilisation (overall lining) of the papers. All this was to be achieved without the undesirable result of loss of the accretions or a significant reduction of the associated staining.

**Treatment Procedure**

*Ox Cheek Soup Label (CMH Catalogue Number 2000.98.8)*

The treatment began with surface cleaning, which was carried out only to the extent that very loose debris was removed. A temporary light-weight (4 g/m$^2$) Japanese tissue facing was then applied with gelatin to portions of the recto in order to support the very fragile areas surrounding the two interior losses (**Figure 7**). The label was then allowed to dry overnight in a weighted blotter stack. Gelatin was chosen as the adhesive at this point in order to minimize the lateral migration of soluble iron ions.[46] It was speculated that when the object was wetted out more than 24 hours later in order to perform the phytate application, the gelatin would be partially solubilized and any binding of the metal ions that was achieved by the gelatin would be of minimal impact to the chelation by phytate portion of the treatment as a whole. Note that the final treatment step was to be sizing of the recto with gelatin.

When it came time to perform the calcium phytate treatment, the label was placed recto side down between pieces of nonwoven polyester (Reemay) on the suction table (**Figure 8**). All necessary solutions had been prepared ahead of time according to the ICN protocol. The label was then wetted out with a spray application of 50:50 water and ethanol. The treatment then proceeded with the use of a modified version of the ICN protocol: rather than the object being immersed in a bath, the calcium phytate solution was applied by brush to the object while it was held in place by the draw of a suction table. Though temporarily faced with tissue, the label was still fragile and the manipulation necessary to place the label in a bath deemed unnecessarily risky when a suction table was available.

**Figure 7.** Temporary facing paper on soup label. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

**Figure 8.** Soup label during treatment. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

The goal of a modified calcium phytate treatment is to approximate the amount of phytate that would have been deposited in an immersion treatment. Tests performed at the Canadian Conservation Institute (CCI) have shown that multiple applications of an *ethanol modified* calcium phytate solution deposit more phytate than a single application.[47] This logic was followed in deciding to do multiple brush applications of an unmodified phytate solution when using the suction table. The planned treatment protocol was to continue this pattern until no positive results registered with bathophenanthroline indicator paper. In devising the protocol,[48] it was decided that if the indicator papers still tested positive after ten rounds, then the application would be stopped.

A soft, two inch wide goat hair brush (*hake*) was used to apply a total of approximately 300 mL of calcium phytate solution to the surface of the verso of the paper. This was done in three applications: a solution sodden brush was used to apply approximately 100 mL of solution in three passes of the brush. The brush was reloaded with solution after each pass, and each pass required careful manipulation of the brush so as not to dislodge the accretions. About one minute after the third pass of the brush, a test with bathophenanthroline indicator paper was made in three locations on the paper, both the stained and unstained areas. When an obvious pink colour was seen on the indicator paper – comparable to a 25 unit (positive) or 1–10 unit (mildly positive) rating on the CCI chart – the application continued with another round. After three rounds of three passes of a freshly loaded brush (nine passes), the test paper did not turn pink, so the calcium phytate application was halted. As per the ICN protocol, calcium bicarbonate was then applied by brush to the verso of the label for the purpose of

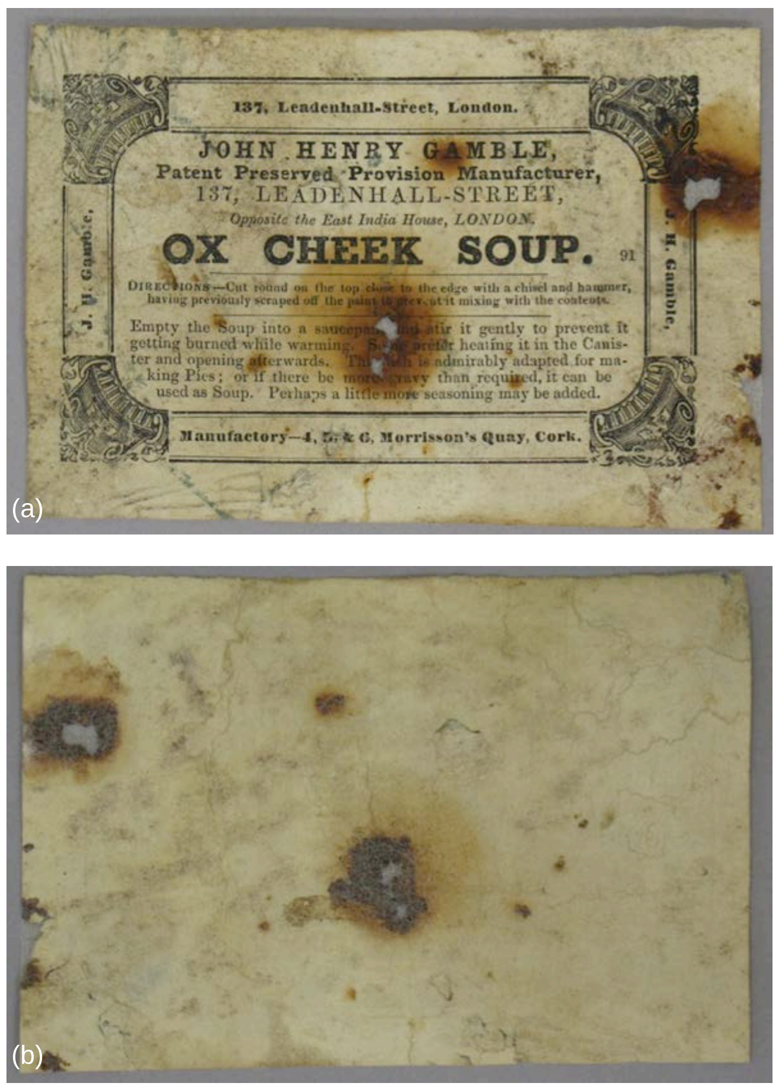


**Figure 9.** Ox Cheek Soup label after treatment, recto (a) and verso (b). Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

deacidification. Though other methods of deacidification could have been considered owing to the absence of iron gall ink and other pH sensitive media, the author did not deviate from the ICN protocol in this regard.

The planar deformations were realigned while still damp, and after partial drying of the paper on the suction table, small tears were repaired with kozo fibre Japanese paper (4 $g/m^2$) and 50:50 wheat starch paste and methyl cellulose. This adhesive combination was chosen for its workability or "slip" as compared to paste alone. The verso of the label was then lined overall with a lighter weight, toned kozo fibre Japanese paper (2 $g/m^2$). The facing papers were removed and proper alignment of the text on the recto was ensured. The label was then allowed to dry in a weighted blotter stack. After drying, the front of the label was sized with gelatin (**Figure 9a**). The overall lining of the verso supports the paper adjoining the two holes and edge losses without obscuring the fact that there are accretions on the verso of the label that likely indicate the colour of the paint that coated the tin to which the label was once attached (**Figure 9b**).

*Pocket Watch Label (CMH Catalogue Number 2012.21.198)*

Prior to the treatment of the label, the watch itself was examined and treated by Kristen Stockstill. The heavier iron corrosion on the gear side of the watch was reduced using cotton swabs dampened with ethanol in order to reveal the surface detail, including the information transcribed above.

All fragments of the label were wetted out on the suction table. Tears and folds on the major fragment were then realigned. Washing and realignment of the smaller fragments through multiple applications of small quantities of distilled water resulted in the transfer of orange-brown discolouration products[49] from the fragments to the blotter below. Prior to treatment, many of the smaller fragments were a darker orange colour as compared to the major fragment of the label (**Figure 10**). The smaller paper fragments had been found in the form of small, matted balls inside the mechanism cavity of the watch and had to be wetted out for the purpose of relaxation and subsequent realignment (**Figure 11**). Some of these fragments had corroded metal parts of the spring embedded in them and this metal was removed as best as possible.

After wetting out and alignment, the calcium phytate solution was applied by brush on the suction table in the same manner as described for the Ox Cheek Soup label. The solution was applied three times followed by testing with indicator strips, and then applied three more times followed again by testing with strips. Only two rounds of three passes with the brush were required before the test papers no longer showed any indication of pink colour. This may make sense intuitively: though both objects exhibited similar bathophenanthroline iron(II) colour indicating paper test results, the pocket watch label with fragments has less than half the surface area of the soup tin label. Calcium bicarbonate was then applied by brush, again in the same manner as to the other label.


**Figure 10.** Watch label fragments during treatment. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

A piece of kozo fibre Japanese paper (approximately 20 $g/m^2$) was wetted out and a 50:50 mixture of wheat starch paste and methyl cellulose was used to paste out one side. The Japanese paper was then placed paste-side up on the suction table. The largest fragment of the watch label was adhered to the lining paper first, followed by smaller fragments, aligning the fragments as work progressed (**Figure 12**).[50] In this case, due to significant losses and the absence of significant features on the verso of the label, a paper with a weight that is closer to the weight of the artifact was selected for the lining. The recto of the object, which had a somewhat "fuzzy"[51] appearance prior to treatment, was then sized overall with gelatin which imparted an improved surface character. It was then placed in a weighted blotter stack


**Figure 11.** Small label fragments found within the mechanism cavity were wetted out and realigned. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

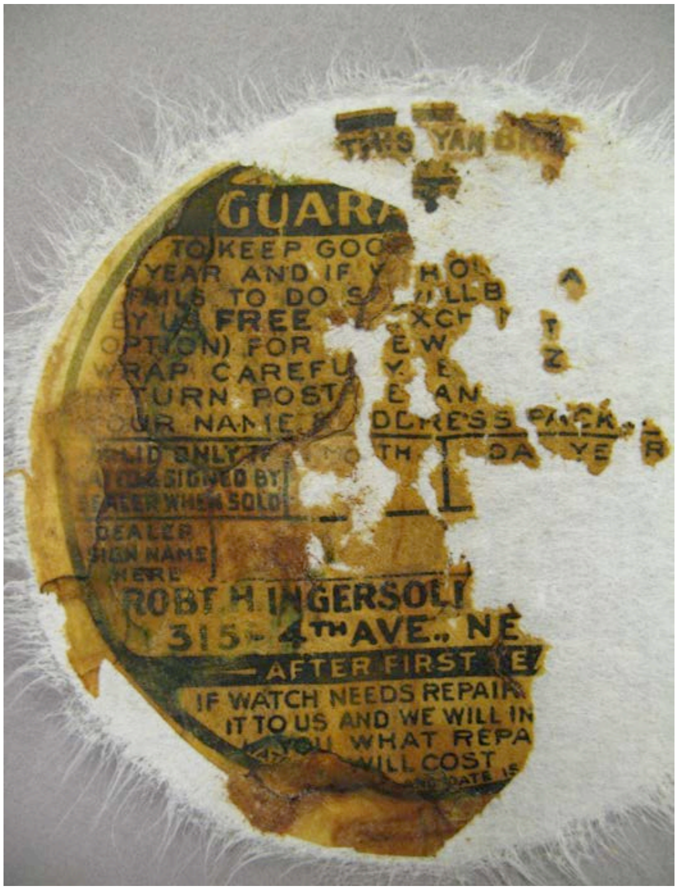


**Figure 12.** Label fragments lined with Japanese paper. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

between nonwoven polyester to dry and flatten. After drying, the lined label was trimmed and the visible areas of lining paper toned with coloured watercolour pencil to blend with the fragments (**Figure 13**). For exhibition, the label was placed back into the lid of the watch. A piece of polyester film cut to the shape of the label acts as a barrier between the paper and the interior of the lid.

**Figure 13.** Pocket watch and label after treatment. Photograph courtesy of the Canadian Museum of History, Gatineau, Quebec.

## Conclusion

The calcium phytate/calcium bicarbonate treatment method was applied to the paper components of two artifacts. These paper artifacts were not inscribed with iron gall ink but affected by iron corrosion. The purpose of the treatments was in both cases to, among other goals, combat future oxidative iron(II)-catalysed degradation of cellulose, while altering the visual cues to the artifacts' history and archaeological context as little as possible. This meant maintaining the orange coloured staining and corrosion accretions affecting the papers. The treatment was successful in that both artifacts were exhibited amongst related objects in CMH exhibitions. Both objects were retested with bathophenanthroline colour indicating test papers one year after their treatment, yielding no colour indication of free iron ions. This demonstrates that the application of the calcium phytate/calcium bicarbonate treatment method to paper not bearing iron gall ink, but likewise negatively affected by the presence of free iron ions, is a viable treatment alternative when stain reduction is not a desired outcome.

## Acknowledgements

The author wishes to thank the following colleagues for their contributions: Dr. Karen Ryan and Dr. John Willis of the CMH; Sherry Guild, Season Tse and Tara Grant of the CCI; all Conservation and Collections Management Section staff at the CMH, but especially, with regards to this project, Emily Lin, Rebecca Latourell and Caroline Marchand; Jan Hawley, Lizzie Meek, Colleen Healey, Sylvie Leblanc and Charles Hett for replying to personal communications; Kate Westbury for recent and future collaboration; and to the several reviewers, family and friends, known and anonymous, who helped to hone this paper.

## Materials

*Ammonium hydroxide (9721-02, 28-30%, 500 mL, certified ACS)*: J.T. Baker, Mallinckrodt Baker Inc., distributed by Alphachem Ltd., Milltower Court, Mississauga, Ontario L5N 5Z6, Canada; Tel.: 905-821-2995; <http://www.alphachem.ca/>

*Bathophenanthroline indicator paper for iron ions*: Preservation Equipment Ltd., Vinces Road, Diss, Norfolk, IP22 4HQ, UK; Tel.: 44 1379 647400; <www.preservationequipment.com> or University Products of Canada (Catalogue No. 539-3000), BFB Sales, 2957 Inlake Court, Mississauga, Ontario L5N 2A4, Canada; Tel. 905-858-7888; <http://www.archivalproducts.ca/>

*Calcium carbonate powder (1288-01, 500 g, certified ACS)*: J.T. Baker, Mallinckrodt Baker Inc.

*Calcium hydroxide powder (1372-01, 500 g, certified ACS)*: J.T. Baker, Mallinckrodt Baker Inc.

*Colour chart for bathophenanthroline indicator paper*: Canadian Conservation Institute, 1030 Innes Road, Ottawa, Ontario K1B 4S7, Canada; contact: season.tse@pch.gc.ca

*Ethanol (Anhydrous ethyl alcohol, 500 mL)*: Commercial Alcohols Inc., 2 Chelsea Lane, Brampton, Ontario L6T 3Y4, Canada; Tel.: 905-790-7500; <http://www.comalc.com/>

*Japanese paper*: The Japanese Paper Place, 77 Brock Avenue, Toronto, Ontario M6K 2L3, Canada: Tel: 416-538-9669; <http://www.japanesepaperplace.com/>

*Methyl cellulose powder* (*Dow Methocel A4C Cellulose Ether, 1 lb*): Talas, 330 Morgan Avenue, Brooklyn, New York 11211, USA; Tel.: 212-219-0770; <http://talasonline.com/>

*Phytic acid (28966-3, 40%, 1 L)*: Sigma-Aldrich Canada Ltd., 2149 Winston Park Drive, Oakville, Ontario L6H 6J8, Canada; Tel.: 800-565-1400; <www.sigmaaldrich.com>

*Reemay non-woven polyester fabric (0.005" thickness)*: Talas

*"Zen Shofu" wheat starch (1 lb)*: Talas

**Notes and References**


1. *Northwest Passage and Sir John Franklin*, curated by Dr. Karen Ryan and displayed at the Prime Minister's Office in 2013 and early 2014; *Canada's Titanic – The Empress of Ireland*, curated by Dr. John Willis and on exhibit at the Canadian Museum of History from 29 May 2014 to 6 April 2015.

2. Hett, Charles E.S., "Conservation Considerations on Archaeological Sites of the Franklin Era," in: *The Franklin Era in Canadian Arctic History 1845–1859*, National Museum of Man Mercury Series, Archaeological Survey of Canada Paper no. 31, edited by Patricia D. Sutherland (Ottawa: National Museums of Canada,1985), p. 186.

3. Of note is that in addition to owning the English patent on canning food, which he was awarded with partners Bryan Donkin and John Hall in about 1810, John Henry Gamble was also responsible for the first production of machine-made paper in England, having brought the drawings for Louis-Nicolas Robert's endless wire papermaking machine there from France and then partnering with the Foudrinier brothers in 1801–1802 to build a machine. See "John Gamble," in: *Grace's Guide* website, <www.gracesguide.co.uk/John_Gamble>. Accessed December 2014. The author wonders whether the labels and/or wrappers used on the Gamble provision tins were printed on or made of papers manufactured in Britain on a Foudrinier papermaking machine (English patent 2487 issued 20 April 1801). Commercial production of the paper began in earnest in January 1812 according to Dard Hunter in *Papermaking, The History and Technique of an Ancient Craft* (New York: Dover, 1978), p. 532.

4. Borden Number QfNo-1; 74 57 04 N latitude and 108 41 00 W longitude.

5. Hett, Charles and Martin E. Weaver, "The conservation of Captain Kellett's storehouse, Dealy Island, N.W.T.," in: *Conservation within Historic Buildings*, Preprints of the Contributions to the Vienna Congress, 7–13 September 1980, edited by N.S. Brommelle, Garry Thomson and Perry Smith (London: International Institute for Conservation of Historic and Artistic Works, 1980), p. 73.

6. The word midden is commonly used by archaeologists to describe a feature containing waste products related to day-to-day human domestic activity. See "Midden" in: *Wikipedia* website, <http://en.wikipedia.org/wiki/Midden>. Accessed December 2014.

7. Janes, Robert R., "The Preservation and Ethnohistory of a Frozen Historic Site in the Canadian Arctic," *Arctic*, vol. 35, no. 3, 1982, p. 367.

8. From the Canadian Museum of Civilization (History) Catalogue Card.

9. Janes, Robert R., Brian Walker-Yorga, Charles E.S Hett, Martin E. Weaver and Robert G. Lemon, "The Archaeology and Conservation of a Frozen Historic Site in the Canadian Arctic," paper presented at the 44th annual meeting of the Society for American Archaeology, Vancouver, BC, Canada, 23–25 April 1979.

10. Hett and Weaver, "The Conservation of Captain Kellett's Storehouse," p. 76.

11. Willis, John, *Canada's Titanic – The Empress of Ireland* (Gatineau, QC: Canadian Museum of History, 2014), pp. 73–82.

12. Willis, *Canada's Titanic – The Empress of Ireland*, p. 94.

13. Récifs Artificiels de L'Estuaire du Québec, "Wreck of Empress of Ireland – Protected Cultural Resource – Diving Guidelines," <http://www.mcc.gouv.qc.ca/fileadmin/documents/publications/regions/guideplongee.pdf>. Accessed February 2015.

14. Brearley, Harry, *Time Telling Through the Ages* (New York: Doubleday, Page & Co. for Robert H. Ingersoll & Bros., 1919), pp. 196–213.

15. McDermott, Kathleen, *Timex: A Company and its Community 1854–1998* (Middlebury, CT: Timex Corporation, 1998), p. 54.

16. Brearley, *Time Telling Through the Ages*, p. 203.

17. *The Print Council of America Paper Sample Book* (PCAPSB) by Elizabeth Lunning and Roy Perkinson (Boston: Print Council of America, 1996) is a publication that offers an annotated selection of antique and modern

papers with the goal of providing a standard for their description. The papers included in the sample book were chosen to represent the range of colour, thickness and texture of the papers one is likely to encounter among the prints and drawings of the past 600 years.

18. Selwyn, Lyndsie, *Metals and Corrosion: A Handbook for the Conservation Professional* (Ottawa: Canadian Conservation Institute, 2004), pp. 107–108.

19. Wrought iron is an almost pure iron that contains less than 0.035 wt% dissolved carbon and up to 2.5 wt% slag inclusions and ". . . was a readily available commercial product until the end of World War II." Selwyn, *Metals and Corrosion*, pp. 91, 96.

20. Hawley, J.K., E.A. Kawai and C. Sergeant, "The removal of rust stains from arctic tin can labels using sodium hydrosulphite," *Journal of the International Institute for Conservation – Canadian Group*, vol. 6, no. 1–2, 1981 p. 17.

21. The noun "draw" is used to describe the deformation in the primary support that results from the action "to draw" meaning to wrinkle or change shape by pulling.

22. The CMH Paper Lab is currently equipped with an Orion 8135BNUWP ROSS Ultra Flat Surface pH electrode and Orion model SA 720 meter.

23. "The colour chart range is from 1–10 (mildly positive), 25 (positive) and 50+ (very positive)." Guild, Sherry, Season Tse and Maria Bedynski, "Technical Note on Treatment Options for Iron Gall Ink on Paper with a Focus on Calcium Phytate," *Journal of the Canadian Association for Conservation*, vol. 37, 2012, p. 20.

24. For a discussion of the application and interpretation of the test see Neevel, Johan G., "Application Issues of the Bathophenanthroline Test for Iron(II) Ions," *Papier Restaurierung*, vol. 30, no. 1–2, 2009, pp. 3–15.

25. Type of iron (wt% of carbon) unknown.

26. Extract from the condition report by Kristen Stockstill, Conservator, 28 March 2013: "Watch face is dented and has a thin layer of iron corrosion on top. Hands and numbers are missing. Silver (?) rim is in good condition however there is some tarnishing and [some] dark red accretions present. Verso – dark red, shiny accretions – possible original adhesive residue that has been re-deposited during underwater burial. Is very flaky and seems easily removable. Evidence of previous active iron corrosion – hollow shells of dark red corrosion bubbles around gears. . . . Cap – Outer surface: very good condition. Inside surface: three large areas of bright orange and dark red corrosion (flash rusting) / adhesive residue."

27. See the table of Ingersoll Watch Company serial numbers and dates in "Brief History: Ingersoll Watch Company," in: PocketWatchRepair.com website, <www.pocketwatchrepair.com/histories/ingersoll.html>. Accessed December 2014.

28. Hawley, et al., "The removal of rust stains from arctic tin can labels using sodium hydrosulphite," pp. 17–24.

29. Antarctic Heritage Trust, *The Antarctic Heritage Trust Artifact Conservation Programme Manual of Methodologies*, vol. 2 (Christchurch, New Zealand: Antarctic Heritage Trust, January 2009), p. 15. Unpublished document. The manual states that "corrosion staining is a significant factor for many can labels – often simple washing will remove some staining . . . chelation/ corrosion treatments on tin labels have shown that EDTA is slow, but can be used with coloured inks whereas oxalic [acid] works much more effectively at removing staining but should only be used with black inks. Both should only be used if multiple rinses can be undertaken to remove residual corrosion remover."

30. Antarctic Heritage Trust, *The Antarctic Heritage Trust Artifact Conservation Programme Manual of Methodologies*, pp. 33–35.

31. Meek, Lizzie (Conservator, Antarctic Heritage Trust), personal communication, 16 February 2014.

32. Meek, personal communication, 16 February 2014.

33. See image of various labels including paper Ox Cheek Soup label on provision containers removed from Dealy Island in 1954 on p. 81 in Harington, C.R., "The Cache on Dealy Island," *Canadian Geographical Journal*, vol. 3, March 1964, pp. 75–83, <www.canadiangeographic.ca/franklin-expedition/docs/1964_March_Canadian_Geographic.pdf>. Accessed June 2014.

34. Perhaps, as a manufacturer of machine-made paper, Gamble found it convenient to use paper labels on the packaging of his tinned provisions. This would, one assumes, allow for more advertising on the container than a stencilled label, and was perhaps more economical than a metal one.

35. The other resides in the Glenbow Museum: "M-3302 McClintock Expedition. – 1852. – 1 item. – Consists of label from can of ox-cheek soup, from a Dealy Island cache of Sir Francis mcClintock [sic], who headed the 1852–1853 expedition to Melville Island to search for the missing Arctic explorer, Sir John Franklin. Collected by Hugh Dempsey, a member of the Glenbow salvage team (1968). NA-1813 is a photograph of the label." From "Franklin Expedition and Arctic Exploration collection" archives finding aid in: Glenbow Museum website, <www.glenbow.org/collections/search/findingaids/archhtm/franklin.cfm>. Accessed June 2014.

36. The author hopes that the insulated plywood floor constructed in July 1978 to protect and keep frozen the remaining contents of Kellett's Storehouse is still intact and performing this task. The site was last officially visited in 1981 (Sylvie Leblanc (Territorial Archaeologist, Nunavut), personal communication, 5 May 2014). At that time the floor had been performing well (Charles Hett (Conservator (retired), Canadian Conservation Institute), personal communication, 20 May 2014), but there is no official confirmation of the current state of preservation of the site.

37. Solvent reactivated mending tissues are conventionally prepared in the lab by coating one side of a tissue with a selected adhesive or combination of adhesives and then drying it between pieces of silicone coated polyester film, under weights, to prevent curling. Though very thin, light-weight tissues can be used for this purpose to aid in visibility of the primary support through the repair tissue, the thinner the tissue, the more one sees the adhesive layer itself. Prepared and dried against the polyester film, the adhesive forms a very smooth layer that can appear shiny, or even in some cases sparkly when viewed at certain angles of light. Successful use of a thin solvent-reactivated tissue would in this instance have required experimentation to select the combination of tissue, adhesive, application and drying technique that resulted in the least visible layer of adhesive between the finished tissue, the accretions and the primary support. Traditional lining with aqueous adhesives was deemed more reliable.

38. The term deacidification is used here to include the concepts of both neutralization and alkalization. Alkalization, or the deposition of an alkaline reserve in the paper, being the preferred outcome in the majority of paper treatments.

39. Jacobi, Eliza, Birgit Reissland, Claire Phan Tan Luu, Bas van Velzen and Frank Ligterink, "Rendering the Invisible Visible," *Journal of Paper Conservation*, vol. 12, no. 2, 2011, pp. 25–34.

40. The author consulted with CCI Senior Conservator Sherry Guild and Senior Conservation Scientist Season Tse to confirm this rationale. Season Tse and Sherry Guild, series of personal communications, ending February 2014.

41. Selwyn, *Metals and Corrosion*, p. 102. See section describing the Fenton reaction.

42. Note that in 2011, the Netherlands Institute for Cultural Heritage (ICN) was incorporated into the Netherlands Cultural Heritage Agency, Rijksdienst voor het Cultureel Erfgoed (RCE). The author has chosen to continue to use the shorthand of "ICN Protocol" as she believes that it continues to be in common usage amongst paper conservators in North America.

43. Reissland, Birgit and Frank Ligterink, *The Iron Gall Ink Website*, <http://www.ink-corrosion.org/>. Last updated 13 February 2011. Accessed February 2015.

44. Guild, Sherry, Season Tse and Maria Bedynski, "Technical Note on Treatment Options for Iron Gall Ink on Paper with a Focus on Calcium Phytate," *Journal of the Canadian Association for Conservation*, vol. 37, 2012, pp. 17–21. Note that the author had access to early drafts of the flow chart prior to its publication.

45. Reissland, Birgit and Judith Hofenk de Graaff, *Condition rating for paper objects containing iron gall ink*, ICN-Information, no. 1 (Amsterdam: Instituut Collectie Nederland, 2001).

46. "Research has shown that gelatin size may offer some protection against iron gall ink corrosion by binding transition metal ions and inhibiting their migration when they are exposed to high humidity conditions." Guild et al., "Technical Note on Treatment Options for Iron Gall Ink," p. 19.

47. Tse, Season, Sherry Guild and Amanda Gould, "A Comparison of Aqueous Versus Ethanol Modified Calcium Phytate Solutions for the Treatment of Iron Gall Ink Inscribed Paper," *Journal of the Canadian Association for Conservation*, vol. 37, 2012, pp. 3–16.

48. Thanks to CCI Senior Paper Conservator Sherry Guild and CCI Senior Scientist Season Tse.

49. Presumably the orange coloured products that were removed from the fragments were the result of various mechanisms of deterioration, including the rust stains, though exactly what was being removed from the label fragments by the washing procedure was not investigated. This level of removal was considered acceptable in order to achieve the relaxation and realignment of the fragments.

50. At the time of the treatment the author had not seen an example of an intact Ingersoll pocket watch label. It was after the treatment was completed that the author realized that the placement of three of the small fragments is incorrect relative to the other fragments. This issue was relayed to the curator, but neither the author nor the curator felt that re-doing the lining to fix the placement justified the risk of damaging the fragments. This information is documented in the database record for the object as an addendum to the electronic treatment record.

51. Fuzzy, the technical term to describe the piece of paper that has gone through the wash in the pocket of one's pants.

**Appendix I: Tinned Provision Labels from QfNo-1 "Kellett's Cache," Government of Nunavut Museum Collections, Prince of Wales Northern Heritage Centre.**
**(**Compiled by Joanne Bird, Curator of Collections, Prince of Wales Northern Heritage Centre, April 24, 2014.)

| Accession No. | Object | Paper label | Painted (stencilled) label |
|---|---|---|---|
| 978.036.013 | can | "Tripe and Onions" | |
| 978.036.015 | can; label | "CARROTS" | |
| 978.036.017 | can; label | illegible | |
| 978.036.028 | can | illegible | |
| 978.036.029 | can | illegible | |
| 978.036.030d | label | legible | |
| 978.036.065b | can | illegible | |
| 978.036.070a | can | partial; "JO" and "...ROTS" visible | |
| 978.036.074d | can | partial; "...EEN PEAS" visible | |
| 978.036.076 | can | | partial; "...ROTS" |
| 978.036.078c | can | | partial; "MULLIG...SO" |
| 978.036.078b | can | | partial; "TA…P" |
| 978.036.092b | can | remnant; illegible | |
| 978.036.101b | can | | "..MILK.." |
| 978.036.106 | can | | "MILK" |
| 978.036.107 | can | remnant; some letters visible; "SHR EFER ADMIR THA" | |
| 978.036.148b | can fragment | remnant; some letters visible; "CKSON .. HOG... ... RDEEB" | |
| 978.036.166 | can fragment | remnant; illegible | |
| 978.036.266 | label | rust stained; faint | |
| 978.036.631 | label fragment | | |
| 977.080.011 | can | | "ROAST MUTTON" |
| 982.114.098 | can | | "PARSNIPS PLAIN" |