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Multi-Analytical Investigation of Arsenical Transfer and Remediation on Nineteenth-Century Green Books

Susan Russick ¹, Lea Faoro ¹, Maria Kokkori ², Samuel M. Webb ³, Hortense De La Codre ², Madeline Meier ² and Marc Vermeulen ⁴

¹Northwestern University Libraries, Northwestern University, Evanston, IL, USA; ²NU-ACCESS – Center for Scientific Studies in the Arts, Northwestern University, Evanston, IL, USA; ³SLAC National Accelerator Laboratory, Stanford University, Menlo Park, CA, USA; ⁴Collection Care Department, The National Archives, Richmond, UK

ABSTRACT

Books containing heavy metals, specifically nineteenth-century green arsenical books, have been identified at Northwestern University Libraries, raising health and safety concerns related to handling. Copper acetoarsenite pigments, such as emerald green, were detected on book covers, decorative page edges, labels, and other components using noninvasive analytical techniques including X-ray fluorescence (XRF), Raman spectroscopy, and Fourier-transform infrared (FTIR) spectroscopy. Further examination of selected volumes using synchrotron radiation (SR) techniques revealed pigment migration, degradation, and arsenic transfer to adjacent books. This paper expands on initial findings through two related experiments. The first explored the transfer of arsenic using mechanical friction; Staedtler Mars® white vinyl erasers rubbed on arsenical books generated crumbs which were analyzed via scanning electron microscopy–energy-dispersive X-ray spectroscopy (SEM-EDX). Results confirmed the transfer of arsenic, copper, and lead, with decorative page edges being particularly prone to shedding arsenic onto other materials. The second experiment tested remediation methods on a book contaminated by prolonged exposure to an arsenical neighbor. Surface cleaning using erasers and a vacuum removed flecks of pigment but did not eliminate non-chromophoric arsenic as confirmed by SR analyses, which highlights its presence either as a degradation product embedded within the paper or present in the paper as part of its production process. Findings demonstrate the acute toxicity risk posed by arsenical books and support the need for safe handling protocols. However, materials with only trace levels of arsenic embedded during production may pose a lower risk of transfer. Cross contamination beyond prolonged direct contact appears limited. These results highlight critical considerations for library preservation practices and future research on arsenic in historical materials.

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Introduction

Books and manuscripts have been produced using heavy metals such as arsenic, chromium, lead, and mercury for millennia, posing legacy safety concerns in libraries today (Delbey et al. 2019; Tedone and Grayburn 2020). While these elements may exist as hidden residues of the material's production process or even be applied as a pesticide after production (Angelova et al. 2023), they are mostly recognized as vividly colored pigments.

Due to arsenic's acute toxicity (ATSDR 2014), the identification of arsenical materials in the collection has been the first step to providing a safe environment for both library staff and patrons. The yellow arsenic sulfide pigment, orpiment, has been found on book covers at other institutions (Delbey et al. 2019), but among Northwestern University manuscripts, has only been identified

in small quantities and in areas of the object unlikely to be touched during handling. However, copper acetoarsenite pigments, such as emerald green and Scheele's green, are more prevalent in Northwestern's collection (Vermeulen et al. 2023; Russick et al. 2025) and are the focus of this study. They were used on frequently touched areas such as book covers, page edges, and labels. These pigments may be tentatively identified due to their distinctive green hue and the limited time-frame of use during the nineteenth century (Douma 2008; Emerald green 2022), however, color alone is inadequate for positive identification.

X-ray fluorescence (XRF) was used to positively identify the presence of arsenic. XRF was selected as the main testing method since it can identify all the heavy metals of interest, does not require sampling of the object, and does not produce hazardous waste. Additional analytical

CONTACT Susan Russick  susan-russick@northwestern.edu  Northwestern University Libraries, 1970 Campus Drive, Evanston, IL 60208

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techniques are needed to positively identify that the pigment is a copper acetoarsenite, however; within Northwestern's collection, whenever additional testing such as Raman spectroscopy has been done on nineteenth-century books containing both arsenic and copper, the pigment has always been identified as emerald green (Vermeulen et al. 2023).

According to Northwestern's Office of Environmental Health and Safety, there is no safe level of exposure to heavy metals. The greatest risk arises from ingestion after transfer by touch, which includes the potential transfer to hands and adjacently shelved books. Surface and air testing at Northwestern University Libraries has demonstrated that arsenic, chromium, and lead are transferred to gloves during handling of large quantities of books (Carnow and Assoc., Ltd 2021). However, it is still unclear how likely and to what extent arsenic can be transferred to hands from a single book or from books that have been secondarily contaminated.

To estimate arsenic transfer, books previously analyzed using XRF were selected for further investigation of their surfaces. Books were selected based on XRF analyses of different book parts (page edges, cover) and materials (paper, cloth, leather). It is important to note that results from XRF analysis are impacted not only by materials present on the book's surface, but may also include elements in any support materials, sizings, or inks present. To investigate the transfer of elements only from the book's surface, an eraser technique was used as a proxy for user handling and abrasion related to sliding adjacently shelved books against each other.

The eraser technique is a minimally invasive, using electrostatic molecular extraction from the surface of the book onto a PVC polymer eraser, and has been used for zooarchaeology by mass spectrometry peptide mass fingerprinting by Fiddymment et al. (2015). The presence of the transferred material on the eraser crumbs is then assessed using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), to characterize elements present within the transferred samples. From a scientific perspective, the eraser technique is optimal because it is minimally invasive, produces a miniscule amount of hazardous waste, and allows us to further understand what elements are present on the surface of a material that will also transfer via touch. From the conservator's point of view, the eraser technique is straightforward and accessible and simply involves gently surface cleaning the book. Surface cleaning is a common conservation treatment and white vinyl or PVC polymer erasers, a vetted product designed to capture friable particles, are well known in the conservation community (Northeast Document Conservation Center (NEDCC) 2007; American Institute for Conservation (AIC) 2024).

Secondary exposure to arsenic through touching contaminated surfaces is another significant concern. Previous studies have shown arsenic transfer from

one book to an adjacently shelved book (Vermeulen et al. 2023), but it remained unknown whether this transferred arsenic could be effectively remediated to allow safe access to contaminated books. Here, the effectiveness of two widely available remediation methods, namely the eraser technique described above and a vacuum method (NEDCC 2007; AIC 2024), were assessed using a multi-analytical approach using SEM-EDX, synchrotron radiation XRF (SR-XRF), and X-ray absorption near edge structure (XANES).

Eraser test: assessing transfer of arsenic during handling

To understand the extent of arsenic transfer that occurs during the handling of arsenic-containing books, erasers were used to mimic the contact and friction that occurs during handling on selected books. Areas of interest of the books were surface cleaned using white vinyl erasers, producing eraser crumbs. The eraser crumbs, which collect loose particles from the books' surfaces, were then analyzed using analytical techniques, specifically SEM-EDX, to assess the transfer of heavy metals.

Selection of books

Books were selected for sampling based on elemental components identified using XRF, the type of material support for the green pigment, and years of publication (Appendix 1). Nearly 50% of the arsenical books identified at Northwestern have paper covers, and nearly 40% have green decorative page edges. The books selected in this study reflect the need for a deeper understanding of these supports. The samples included a control of the eraser only; nine paper covered books with varying levels of arsenic and lead detected; three arsenical page edges (one with lead), one leather spine; and one textile cover.

The XRF data was collected from the survey completed in 2021 (Vermeulen et al. 2023). The energy emitted from the XRF instrument does not exclusively stop at the surface of the substance being tested and can detect substances up to about 3.5 mm beneath the surface (Bloch 2015). The mass attenuation coefficient, or depth to which the XRF includes data, varies with the density, elements present, instrument settings, and other variables (Bloch 2015). A zero-background plate may be used to help isolate the target; however, in the case of the typical book cover, the pigment coating, paper, adhesives, and book board would all be incorporated into a shared spectrum. No quantitative or semi-quantitative analyses were performed as it fell outside the project's scope.

Sample collection

Sampling was performed using materials and techniques typical for surface cleaning of rare collection

materials (AIC 2024; NEDCC 2007). To prepare samples, new Staedtler Mars® white vinyl erasers were cut into 15 small wedges (~2 cm L x 0.5 cm W x 0.5 cm D) and individually placed in labeled Eppendorf™ Safe-Lock Microtubes. Each book was placed on a new, unused sheet of paper with a smooth surface, intended to capture eraser crumbs during cleaning. One pre-cut wedge of white vinyl eraser was removed from its microtube and rubbed on the surface of a book for 45 s, mimicking the motion and pressure conservators used for surface cleaning. Depending on the location of the green pigment on the book, different techniques were used to clean a similar surface area and attempt to produce similar amounts of eraser crumbs.

- Book boards – Paper window mats (window area 6 × 6 cm) were used to define the surface area of the back book covers cleaned. The least degraded area of the back cover was chosen (Figure 1).
- Book spine – The spine covering material was cleaned where it extended onto the boards for a flatter surface.
- Page edges – The entire tail of the text block was cleaned.

After surface cleaning the area, the eraser crumbs were gently brushed off the surface of the book onto the smooth paper below and funneled into the microtubes.

Irregularities were noticed during the eraser crumb collection. Books with smooth surfaces did not produce as many crumbs as more textured surfaces. Books that were more worn or dirty produced crumbs that appeared gray in color, and some books seemed to shed more pigment and the eraser crumbs appeared green.

Eraser crumb sample analysis: SEM-EDX

SEM-EDX indicated that arsenic and lead had transferred onto eraser crumbs for some samples. When arsenic was detected, there was also the presence of copper (Figure 2). This is hypothesized to occur because of the use of a copper acetoarsenite pigment, such as emerald green.

The results of the EDX analysis are summarized in Table 1. The presence of calcium (Ca), magnesium (Mg), silicon (Si), and titanium (Ti) are likely associated with the erasers and are therefore italicized to differentiate them from other elements detected. Factors including the quantity of each element present in the book; the media binder used; the book's surface texture and condition; and the sampling technique may all influence the transfer of arsenic. Arsenic was not always positively identified on eraser crumbs used on arsenical books.

Using XRF, nine book covers or page edges had clear peaks for arsenic and copper, a combination that is found in copper acetoarsenite pigments. SEM-EDX identified arsenic and copper simultaneously on six eraser samples taken from those areas. Copper was found on an additional three eraser samples, without the presence of arsenic, suggesting its presence is not related to a copper acetoarsenite pigment. The copper may instead be related to another pigment or to the paper-making process.

XRF detected both arsenic and lead in eight books (Figure 3). Using SEM-EDX, lead and arsenic were not simultaneously found on any eraser crumbs, which is likely attributed to the overlap of La1 lead and Ka1 arsenic peaks with EDX. Lead was found on two eraser crumb samples. Arsenic was found on six eraser samples.

All three decorative page edge samples, samples 4, 5, and 6, resulted in the transfer of arsenic to eraser crumbs. XRF indicated the presence of trace lead on one of these books, sample 4, however no lead was detected on the eraser crumbs.

More paper covers than any other support material were tested. Of the nine paper covers tested, SEM-EDX identified arsenic on eraser crumbs from the six items for which XRF showed clear peaks of arsenic unless obvious peaks of lead were also present. Lead alone was positively identified on eraser crumbs from two of the three samples where both lead and arsenic were evident using XRF, where the La1 lead peak could possibly be obfuscating the Ka1 arsenic peak. SEM-EDX detected neither arsenic nor lead on eraser crumbs generated from items with XRF trace levels of either substance.

The leather spine, sample 3, had a low signal for both arsenic and lead using XRF, but neither was evident on the eraser crumbs.

The textile cover, sample 16, had evident arsenic and trace lead detected with XRF, but only arsenic was detected on the eraser crumbs.

Eraser tests – remarks

While the sample size is too small to make strong generalizations, transfer of arsenic and lead to eraser crumbs occurred, and it is therefore likely that arsenic and lead can transfer to hands or surrounding surfaces. The transfer of elements is likely impacted by the support material.

The likelihood of arsenical transfer from a book through primary exposure depends on various factors, including the location of the pigment on the book (covers, page edges, labels, etc.), the materials used during book production (paper, textile, leather), the condition of the book, and the abrasiveness of the handling process. While the erasers approximate the transfer of arsenic by touch, it is unknown



Figure 1. Eraser crumb sample produced by rubbing gently on textured paper covered book within the window mat. Courtesy of Northwestern University Libraries.

whether moisture from ungloved hands could influence the transfer or absorption of oxidized substances. The duration of exposure and the purpose of handling could also impact transfer. For example, the act of quickly reshelving an item results in much shorter exposure when compared to a reader holding a book for several hours.

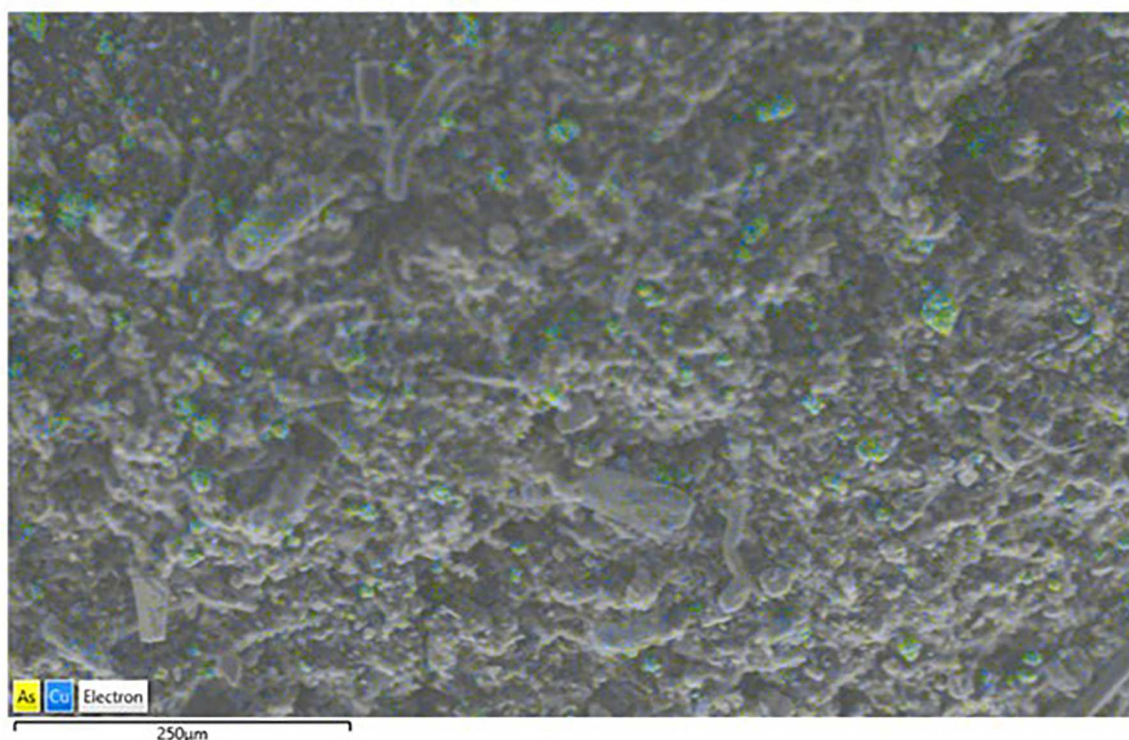
Arsenic consistently transferred from decorative arsenical page edges to erasers, suggesting that colored page edges present a probable risk. Transfer from paper covers only occurred with a high arsenic signal, although there were inconsistencies. The one leather and one textile books that were tested are too small a sample size to generalize.

While the sample size is small, no books produced with trace levels of arsenic or lead (as identified using XRF) had SEM-EDX detectable levels of those substances. This finding suggests that there may be a positive relationship between low XRF peaks heights for the overall book and lack of arsenic or lead transfer as seen in the eraser test. Although further study is needed, it could suggest that trace heavy metals as identified using XRF on books may not pose a significant risk for transfer. This negative finding is also reassuring, given the myriad of opportunities for cross contamination

(Biblioteksverband and Bestandserhaltung 2023) and the likelihood that even books without arsenical permanent neighbors may have been in contact with arsenical books while on a book cart or during use.

It seems possible to use swab tests to successfully detect the transfer of arsenic alone (Wetten et al. 2024), however tracking multiple metals at once remains elusive. It is unclear why arsenic, copper, and lead were not always transferred to erasers at detectable levels by SEM-EDX. There was variability in eraser crumb production related to the book's surface texture and condition, which could have impacted results. The detection of heavy metals on the books using XRF may have been due to an artifact of the leather, cloth, or paper production process rather than a pigment applied to the surface, and therefore not easily transferrable. The transfer of only paired arsenic and copper or only lead could be related to the binder used, the use of lead as an undercoating, an interaction between arsenic and lead, or because of the overlap of L α 1 lead and K α 1 arsenic peaks in SEM-EDX, but it is beyond the scope of this project. Further study is needed to determine the relationship between the book and the transfer of metals to eraser crumbs.

a) EDS Map Electron Micrograph Overlay



b) EDS Elemental Maps

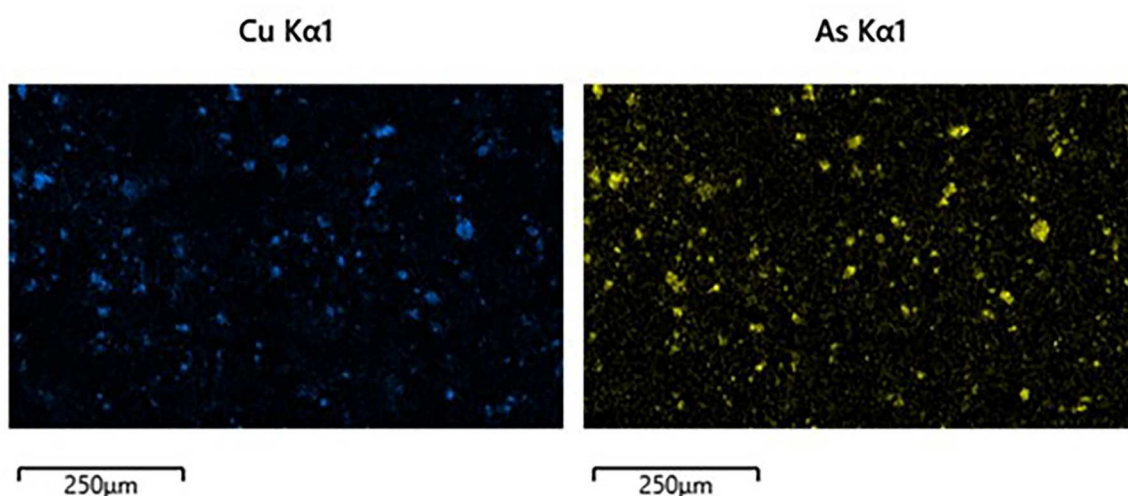


Figure 2. (a) Representative SEM-EDX elemental map overlays of arsenic and copper on an eraser crumb from book number 5 sampled via the eraser method; (b) Copper and arsenic elemental maps from EDX analysis.

Whatever the reason, this eraser test with SEM-EDX analysis did not reliably detect all heavy metals present and cannot be considered an adequate method of determining the presence of heavy metals on books. Using this eraser test as a means of identifying heavy metals in books is not viable; however, it may still be useful in determining whether a specific book transfers a heavy metal via touch and therefore requires a special protocol when handling.

Assessing the remediation of arsenic transfer from adjacently shelved books

Selection of book

A book, *Aristée, ou De la divinité*, Hemsterhuis, François, Paris, 1779, known to have been shelved adjacent to an arsenical book for decades, was scanned on front and back covers using the Elio XRF. The book is covered in a cream-colored paper with a dark blue surface coating. Trace arsenic and lead were detected on the covering paper. This book was previously

Transfer Test: Elements Identified on Eraser Crumbs using SEM-EDS

[illegible]

Arsenic and Lead detected using XRF and SEM/EDS



Figure 3. Samples (numbered) are roughly distributed based on the XRF signal strength of arsenic and lead as detected on books. The eraser crumbs test is represented in two ways: the border color indicates the part of the book or support material sampled, and the fill color indicates deposits identified using SEM-EDX.



Figure 4. *Aristée, ou De la divinite*, a non-arsenical book that was contaminated with arsenic from adjacent shelving. Remediation was attempted in the upper third of the book using a vacuum and in the lower third of the book using erasers. Other areas were protected using plastic strips.

analyzed at the Stanford Synchrotron Radiation Light-source (SSRL) which revealed the transfer of arsenic (Vermeulen et al. 2023). The study showed arsenic is likely present as a pigment and in a colorless degraded form. After the first SSRL scan, the object was placed in a plastic zip bag and was not removed from the bag until remediation.

Remediation processes

Conservators have an arsenal of remediation techniques; however, dry cleaning is preferred in most circumstances due to the risk of further damage with wet and chemical means. The most common techniques involve brushing, use of erasers or sponges, and vacuuming (NEDCC 2007; AIC 2024). Given the desire to capture removed arsenic particles, surface cleaning using erasers and vacuuming were selected as the two potential remediation techniques requiring further testing. White vinyl PVC erasers were used since they have been vetted by conservators, do not contain the pigment elements of interest, and have been used in other scientific inquiries (Fiddymment et al. 2015).

Aristée, ou De la divinite, the selected book, was removed from the plastic zip bag in which it was stored and wrapped horizontally with three strips of polyethylene (Figure 4). Remediation of the top and bottom areas was conducted using different methods, respectively vacuum and eraser, while the

center strip was left protected as a control, where no remediation was attempted. Gloves were changed between each step.

Head – The top protective plastic area was removed, and the upper front cover of the object was vacuumed using a HEPA vacuum cleaner and a new vacuum cleaner brush. A fresh protective plastic strip was added after cleaning.

Center – No remediation attempt.

Tail – The bottom protective plastic area was removed, and the lower front cover of the object was surface cleaned using Staedtler Mars® white vinyl erasers in two phases:

- 6 × 6 cm area cleaned following the protocol described in the transfer of arsenic eraser test, with eraser crumbs retained and examined using SEM-EDX.
- The remaining portion of the tail section of the cover was cleaned using the same method, but without retaining the crumbs. A fresh protective plastic strip was added after cleaning.

Following remediation cleaning, the book was placed in a fresh plastic zip bag until the analytical assessment was performed.

Assessment of eraser as remediation technique: SEM-EDX analysis

The instruments and techniques used were the same as those described in the above section on *Eraser crumb sample testing: SEM-EDX*.

Arsenic and copper were detected on the eraser crumbs, but lead was not (Table 2). The arsenic and copper combination supports the hypothesis of the transfer of the copper acetoarsenite pigment. As lead was not detected in the adjacently shelved book, the

Table 2. Energy-dispersive X-ray spectroscopy (EDX) results for *Aristée, ou De la divinite*, Sample 2, the remediated book. Each sample was analyzed twice to account for heterogeneity across the sample. Elements likely to be from the eraser material based on presence in the control (sample 1) are italicized. Arsenic, copper, and lead are highlighted showing detection using XRF and/or SR-XRF on book covers.

Remediation Test: Elements Identified on Eraser Crumbs using EDX		
Element	Sample 1 Eraser Control	Sample 2 Remediated Book
Aluminum (Al)		X
Arsenic (As)		X
Calcium (Ca)	X	X
Copper (Cu)		X
Iron (Fe)		X
Magnesium (Mg)	X	X
Lead (Pb)		
Silicon (Si)	X	X
Titanium (Ti)	X	X

lead detected on both the front and back covers via XRF of *Aristée, ou De la divinite* can be presumed to be from the book itself, rather than transfer from the neighboring arsenical volume. Arsenic on the eraser crumbs suggests that erasers may be an effective remediation method for secondary arsenic contamination but would need to be further explored.

Assessment of remediation techniques using synchrotron radiation XRF and XANES

SR-XRF and XANES were employed to compare the effects of remediation using a similar approach published by Vermeulen et al. in 2023. SR-XRF imaging was chosen over conventional scanning macro XRF (MA-XRF) due to the higher beam intensity, spatial resolution, and sensitivity offered by the former allowing precise localization of a small amount of arsenic-based material. Furthermore, SR-XRF allows for the speciation of the arsenic, distinguishing the chemical forms, a capability not achievable with other methods available for this study. The same book scanned in the original study (Figure 5a, b) was re-evaluated following the application of the previously described remediation techniques.

MA-XRF mapping (Figure 5) revealed that arsenic distribution did not exhibit significant post-remediation changes using either the vacuum or eraser methods. Although arsenic and copper were detected on the eraser crumbs, indicating some remediation of the copper acetoarsenite pigment, the overall levels of arsenic on the document remained largely unchanged, suggesting that these methods were not sufficiently effective.

Random XANES analyses at the As-K edge across the front cover showed three distinct peaks with energy at maximum intensity ($E_{\max}$) at 11,868.2, 11,872.6, and 11,876.4 eV (Figure 5c). The peak position is sensitive to the spectral oxidation state of As, and the $E_{\max}$ or edge peak (white line) photon energy position increases with an increase in oxidation state. The white line's position is also influenced by the cation to which the arsenic species will be associated. Consequently, these three peaks are likely to correspond to three arsenic species of various natures (both in oxidation state and composition). The peak at 11,876.4 eV is likely associated with an arsenate species (As^{V}), which can be a product of the degradation of the original green arsenic-containing pigment. The peaks at 11,868.2 and 11,872.6 eV are more difficult to assign but are likely associated with two different reduced species (As^{III}). The peak at 11,868.2 eV could be associated with non-degraded copper acetoarsenite whereas the peak at 11,872.6 eV may be associated with arsenic oxide as it is always shifted toward higher energies compared to other arsenite species (Diacomanolis et al. 2016; Vermeulen et al., 2016; Keune 2020).

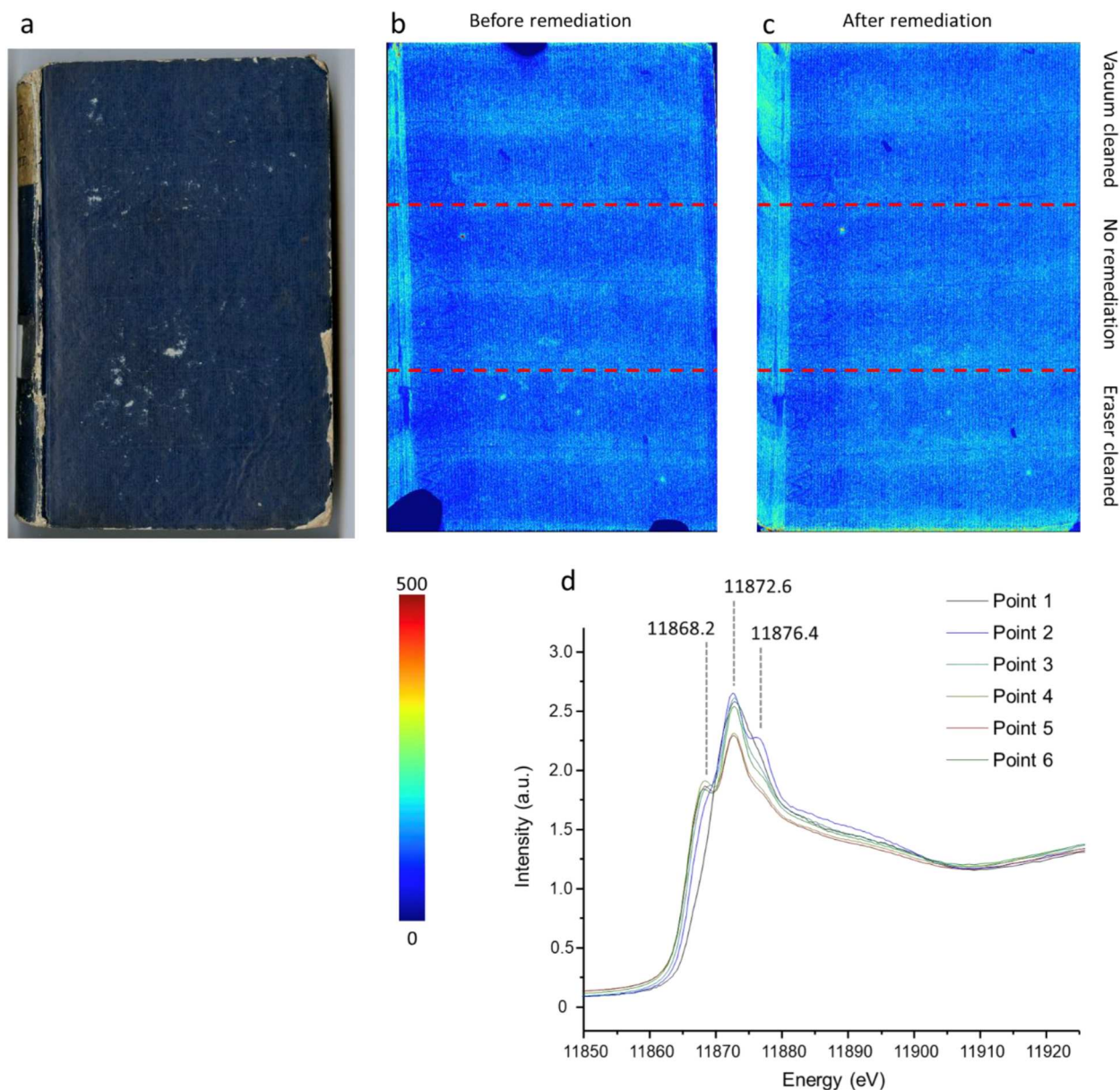


Figure 5. (a) Visible light image of the studied book and SR-XRF distribution maps of arsenic; (b) before remediation; and (c) after remediation, showing in red dotted lines the three areas of interest: top: vacuum cleaned; middle: no remediation; and bottom: eraser cleaned; (d) presents a random selection of XANES measurements realized after remediation, showing three white line energies at 11,868.2, 11,872.6, and 11,876.4 eV.

The presence of all three arsenic species identified in XANES is consistent with the transfer from the adjacent book, containing either non-degraded arsenic pigment or its degradation products, as demonstrated by Vermeulen et al. in 2023 and further demonstrates the overall lack of effectiveness of the remediation techniques. While confirming that arsenic was transferred, it does not preclude that arsenic from the papermaking process may also be a contributor.

Arsenic associated with the papermaking process could be present in the cover paper independently of the green pigment transferred from the adjacent cover and complicating the interpretation of SR-XRF results, as shown in Figure 6. Arsenic occurs naturally in virtually all soil and groundwater and is 'ubiquitous in the plant kingdom' (National Research Council (US

Committee on Medical and Biological Effects of Environmental Pollutants 1977; Hughes et al. 2011), making the raw materials for paper likely to include trace arsenic (Michon et al. 2011). Arsenic was inadvertently introduced to some papers in the pulping process and, during the nineteenth century, may have been purposely introduced as a fungicide. Arsenic could also have been introduced in the form of smalt, a 'blueing' agent. 'Blueing' was often used to counteract the yellowing of the paper substrate. In this process, blue pigments such as smalt or Prussian blue, or blue dyes (both natural and synthetic), were added to neutralize the yellowing effect (Leclerc and Fliedner 1992; Mustalish 2000). As a result, many papers contain trace levels of arsenic, evident when tested using sensitive instrumental analysis.

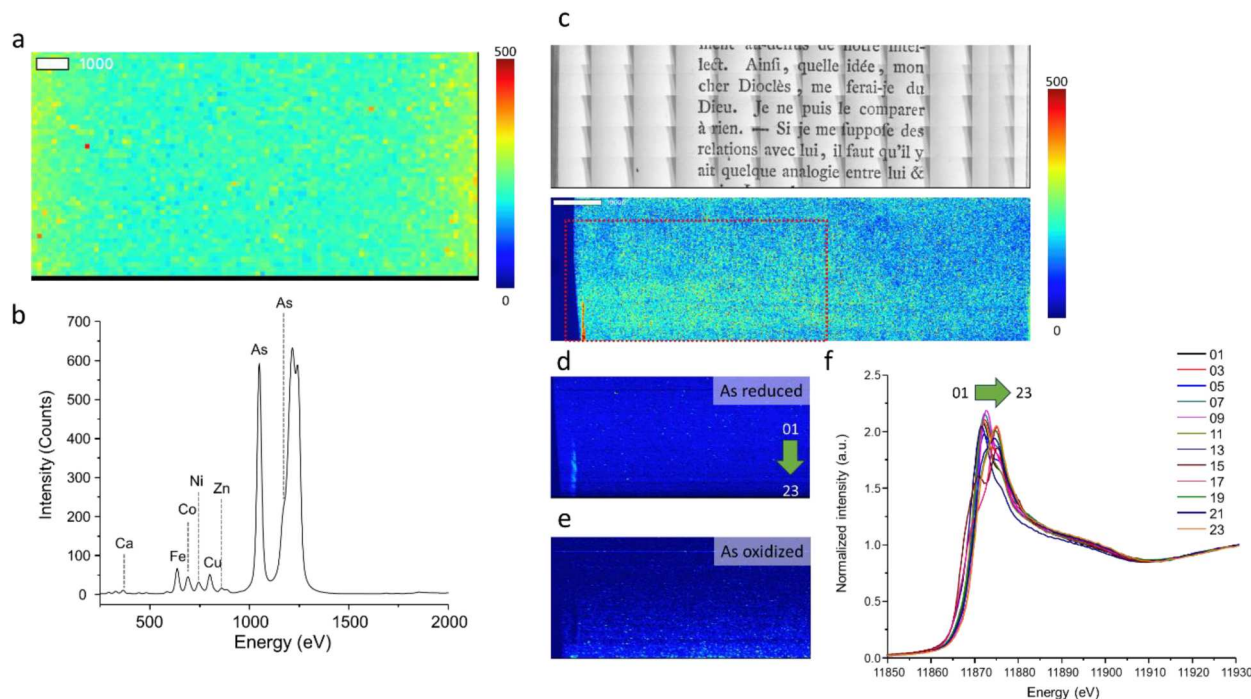


Figure 6. (a) SR-XRF distribution map of arsenic; (b) associated multi-channel analyzer (MCA) spectrum representative of the elements being present; (c) mosaic visible light image of page 128 and associated XRF distribution map of arsenic with, in red dotted line, the selected area for XANES analysis showing the presence of arsenic distributed across the pages independently of the text area; (d) As-XRF intensities high-resolution maps recorded at 11,872.6 eV, characteristic for reduced As; (e) As-XRF intensities high-resolution maps recorded at 11,876.4 eV, characteristic for oxidized As; and (f) extracted XANES spectra showing the increase in oxidized As when moving from areas inside the page (01) toward the edge of the page (23) as indicated by the green arrow.

The copper acetoarsenite pigment applied to the surface of the fully formed paper sheet adds a form of arsenic, bound to copper, which is distinct from integrated arsenic.

Some copper acetoarsenite pigment was remediated, as demonstrated using SEM-EDX, but XANES revealed that a significant amount of arsenic-based material remains in the book. While the possibility exists that the arsenical pigment transferred from the neighboring book was completely removed and the remaining arsenic originates from the papermaking process, further research is needed to confirm this hypothesis but falls outside of the scope of this article. Alternatively, some of the transferred arsenic pigment could have migrated deeper into the paper, some degrading, and is no longer possible to remediate. The co-presence of several oxidation states observed after remediation within a single measurement point in XANES (Figure 6d) suggests a complex mixture of materials. However, the peak at 11,868.2 eV, corresponding with a likely peak for a copper arsenite species, suggests that at least some emerald green pigment transferred and was not remediated.

Elevated concentrations of arsenic align with the laid lines of the paper cover. This pattern could reflect additional contact with the neighboring book at those high points, or it may result from arsenic-containing fibers settling into the laid mold during

papermaking. A ~1 cm vertical band of lower arsenic concentration paralleling the spine of the book supports the theory that rubbing against the adjacent book contributed to arsenic transfer. However, the overall uniformity of the laid pattern suggests that arsenic was also part of the papermaking process. Since the thicker areas of fiber deposit would form the same high points, distinguishing these effects using SR-XRF alone is challenging, and further analysis is required to draw definitive conclusions.

In worn areas of the book's covering paper, the arsenic signal is higher where the blue surface coating is disrupted, revealing the abraded cream-colored paper surface and the tan book board underneath. If this signal originates from transferred arsenic pigment, the rough paper fibers may have facilitated particle embedding compared to the smooth-finished blue surface. Conversely, if arsenic from the papermaking process is present, the abraded areas may reveal arsenic embedded in the paper fibers. Again, further experimental work is necessary to clarify these observations but falls beyond the original scope of this research project.

It is impossible to remediate arsenic that was part of the papermaking process. The existence of non-degraded arsenic pigment in the post-remediation scan confirms that remediation of transferred pigment was not completely effective. More

aggressive remediation attempts may be possible for sturdy volumes but could cause damage to more fragile materials. Evaluation of remediation success on individual volumes via SR-XRF would be unsustainable and difficult to determine through other means.

To explore the possibility that arsenic could also be associated with paper production rather than external contamination, the text block inside the book was scanned using XRF and XANES. It is important to note that the paper used for the text block is different from the one used in the cover but their elemental composition and trace elements may be similar. The general presence of arsenic in nineteenth-century paper production is a hypothesis that would require a larger survey of paper blocks from that time period, which was beyond this research's scope. This is, however, an assumption made based on repeated XRF analysis of paper material often showing small levels of arsenic. The first page was analyzed with the entire paper block behind it to enhance the detection of elements, revealing a non-negligible peak for arsenic, among other elements such as Ca, Fe, Co, Cu, and Zn, all with lesser intensities, based on the multi-channel analyzer (MCA) spectra (Figure 5a, b). A similar result was obtained when scanning page 60 along with the remaining paper block (data not shown), indicating that the presence of arsenic seems to be associated with the paper itself and is likely the same across the book. To better understand the distribution and nature of the arsenic within a single page, similar scans were carried out on page 128, placing a lead sheet behind it to isolate the page from the remainder of the paper block. Here again, arsenic was still detectable and appeared distributed throughout, despite some richer areas (Figure 6c). This likely indicates arsenic to be part of the paper production process, potentially from contaminated water (Michon et al. 2011), a bleaching process, or more likely to the use of a smalt, used as an optical brightener for the paper (Brückle 1993). This is consistent with the presence of cobalt (Co) and nickel (Ni) along with the arsenic in the MCA analysis (Figure 6b).

XANES spectra (Figure 6f) show that points selected further inside the page (away from the page edge, smaller numbers) show mainly peaks at energy 11,872.2 eV, characteristic for reduced arsenic. When moving toward the page edge (spectra with larger numbers), an increase in the peak at energy 11,875.8 eV, characteristic for oxidized arsenic, is observed. When As-XRF intensities high-resolution maps are recorded at both energies (Figure 6d and e), it becomes clear that arsenic near the page edge is more oxidized (Figure 6e), suggesting that the arsenic present in the pages is primarily arsenic (III), which oxidizes to arsenic (V) upon exposure to oxygen and humidity from the environment.

The presence of arsenic as smalt in the paper does not exclude its presence in another form, but this study does not ascertain this. The absence of Co and Ni in the XRF spectra of the cover does not seem to indicate the use of smalt. Thus, in the case of the cover, the presence of arsenic could be linked either to external contamination through contact to arsenic-containing books or to the contamination through the paper production process.

Remediation techniques – concluding remarks

Despite the clear indications that arsenic was picked up by the eraser, neither of the remediation techniques (eraser and vacuum methods) proved to be entirely efficient in removing arsenic from the book surfaces according to the synchrotron study. This suggests that while these methods can capture some arsenic particles, a significant amount remains embedded within or on the surface of the paper. Additionally, the arsenic from the neighboring book seems to have accumulated differently in damaged areas of the book's surface, where the raw paper fibers under the coating potentially picked up more arsenic than the smoother, coated areas. Additional remediation passes and cleaning methods were not attempted due to time constraints and because a single remediation campaign would be more realistic in a library setting.

Furthermore, our analyses indicate that some of the arsenic detected might be inherent to the paper itself rather than solely a result of transfer from the adjacent green book. This inherent arsenic, likely originating from the paper production process, presents a challenge for remediation efforts as it is deeply embedded within the paper fibers and impossible to remove. Polypropylene zip bags and plastic strips used throughout the remediation process could have also contributed to the unrecorded removal of arsenic due to their static charge. However, despite this potential factor, the overall arsenic levels in the book appeared unchanged when assessed at the SSRL, emphasizing the limited effectiveness of the applied remediation techniques.

Conclusions

Heavy metals are commonly found in pigments used to produce nineteenth-century books, creating a public safety issue in libraries. The transfer of arsenic from books made using copper acetoarsenite pigments, emerald green and Scheele's green, is of the highest concern due to the element's acute toxicity. Transfer of arsenic through primary and secondary exposure was demonstrated through the eraser test and analysis of remediation techniques

on a contaminated book, *Aristée, ou De la divinite* (sample 2).

In the eraser test, SEM-EDX confirmed the transfer of arsenic, copper, and lead to eraser crumbs, suggesting that the risk of transferring heavy metals is possible after touching a single arsenical volume. Of the locations and materials tested, arsenical green page edges consistently resulted in the transfer of arsenic to erasers and therefore seem most likely to transfer arsenic to other materials. Further research is needed to investigate the relationship between the substrate holding the green pigment, the pigment's binder, and the shedding of arsenic.

SEM-EDX did not detect heavy metals on erasers from books with trace levels of arsenic and lead, suggesting that they may not present as significant a risk of transfer. It is possible that the trace level of arsenic corresponds to paper production, rather than pigmentation, and is more firmly embedded in the paper, leather, or textile. While further testing is needed, materials with trace levels of arsenic and lead may be safe to handle with relaxed safety protocols.

SEM-EDX confirmed that arsenic was removed from the contaminated book, *Aristée, ou De la divinite* (sample 2) via erasers, although remediation was not evident when examining the book itself through synchrotron radiation analyses. Although the presence of some arsenic detected with SR-XRF and XANES requires further research to completely understand, the non-degraded arsenic remaining on the remediated book suggests that total removal of the transferred arsenic pigment from a neighboring book may not be possible using Staedtler Mars® erasers or a HEPA vacuum cleaner. *Aristée, ou De la divinite* still contains arsenic after remediation attempts. At least a portion of the remaining arsenic seems related to paper production, more embedded, and potentially a lower risk of transfer. Further research is needed in the study of arsenic in paper production but is beyond the scope of this research.

Aristée, ou De la divinite had the special shelving circumstance of having the same arsenical neighbor for decades of continued contact. The neighbors of most other books in this study were not recorded and shelf positions for circulating collections are always in flux. Aside from *Aristée, ou De la divinite*, no other cross contamination was detected. This finding is reassuring, given the opportunities for cross contamination with arsenical books during short exposure on a book cart or during use.

As investigations of heavy metals in library materials continue, the authors hope this research, which builds on evidence of arsenic and lead transfer and provides an initial glimpse at remediation efficacy, will help to inform safe handling practices, library policy, and provide a starting point for other research topics.

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No potential conflict of interest was reported by the author(s).

ORCID

Susan Russick  <http://orcid.org/0000-0002-2203-8830>

Lea Faoro  <http://orcid.org/0009-0007-2018-3763>

Maria Kokkori  <http://orcid.org/0009-0007-4832-5313>

Samuel M. Webb  <http://orcid.org/0000-0003-1188-0464>

Hortense De La Codre  <http://orcid.org/0000-0002-0038-1929>

Madeline Meier  <http://orcid.org/0000-0003-1608-0810>

Marc Vermeulen  <http://orcid.org/0000-0003-1000-3259>

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APPENDICES

Appendix 1. XRF analysis and sample selection

XRF analysis was done using XGLab Elio energy-dispersive X-ray fluorescence analyzer to acquire spectra for samples 2 and 4–16. The instrument is equipped with a transmission Rh anode X-ray tube. A collimator allowing a 1 mm diameter focused spot size at the object's surface was used. The instrument was operated at 40 kV and 100 μ A. Spectra were acquired for 120 s and further processed using the Elio software. Samples 2 and 4–16 were analyzed as part of the pilot project with the help of scientists at Northwestern University Center for Scientific Studies in the Arts (NU-ACCESS).

A Bruker Tracer 5 g X-ray fluorescence analyzer was used to acquire spectra for sample 3 (Appx. 1 Figure A1). The instrument is equipped with a transmission Rh anode X-ray tube. A collimator allowing an 8 mm diameter focused spot size at the object's surface was used. The instrument was operated at 45 kV, 30 μ A with a Ti/Al filter. The spectra were acquired for 30 s and further processed with Artax software. Sample 3 was tested with a different XRF spectrometer than the rest of the books because the NUL conservation lab acquired its own XRF analyzer for the remainder of the project.

Books were selected for XRF testing because they were published during the nineteenth century and contained one or more green components in the binding. They were selected for inclusion in further testing for reasons stated in Appendix 1, Table A1.

Table A1. Books selected for sampling, including identifying information, the green support material, and the reason for including in testing.

Sample	Identifying information	Material	Reason for selection
01.	Control – eraser crumbs only	Control	Control
02.	<i>Aristée, ou De la divinité.</i> Hemsterhuis, François. Paris. 1779.Schulze 104 H49	Paper cover	– Remediated contamination – Arsenic not detected using XRF, detected using SSRL – Shelved next to arsenical book sample 10
03.	<i>Archiv der Mathematik und Physik, mit besonderer Rücksicht auf die Bedürfnisse der Lehrer an höheren Unterrichtsanstalten.</i> Grunert, Johann August. Greifswald. 1857.L510.5 A67 Ser.1 v.28-30	Leather spine	Trace arsenic and trace lead
04.	<i>Almanach dramatischer Spiele zur geselligen Unterhaltung auf dem Lande.</i> Kotzebue, August von. Hamburg. 1834.832.08 A445 1834	Page edges	High arsenic, trace lead
05.	<i>Anna Grenwil, das tapfre Mädchen aus Schottland; oder, Die Enthüllung im schwarzen Gewölbe. Eine historisch-romantische Geschichte aus Cromwells Zeitalter.</i> Leipzig. 1802833.6 A613 v.1 1802	Page Edges	High arsenic, no lead
06.	<i>Archäologische beiträge, von Otto Jahn.</i> Jahn, Otto. Berlin. 1847.733 J25 1847	Page Edges	High arsenic, no lead
07.	<i>Apologetik der kirche, oder, Begründung der wahrheit</i>	Paper Cover	High arsenic, lead

(Continued)

Table A1. Continued.

Sample	Identifying information	Material	Reason for selection
	<i>und göttlichkeit des Christenthums in seiner fortpflanzung und entwicklung.</i> Berlage, Anton. Münster, Theissing. 1834.Schulze 239 B51		
08.	<i>Frühlingsalmanach.</i> Lenau, Nicolaus. Stuttgart. 1835.Taschenbuch 838 F94 1835	Paper Cover	High arsenic, higher lead
09.	<i>Immergrün.</i> Wien. 1842.Taschenbuch 838 I33 v.6	Paper Cover	High arsenic, higher lead
10.	<i>Aphorismen aus Theodor Parows nachlass; hrsg von Eduard Mätzner.</i> Parow, Theodor Gottlieb. Berlin. 1839.Schulze 104 P25	Paper Cover	– High arsenic, no lead – Adjacently shelved to sample 2
11.	<i>Berliner Kalendar auf das Jahr.</i> Berlin. 1843.Taschenbuch 838 B51 1843	Paper Cover	High arsenic, trace/no lead
12.	<i>Die idee der freiheit und der begriff des gedankens.</i> Bayer, Karl. Nürnberg. 1837.Schulze 110 B35	Paper cover	No arsenic, high lead
13.	<i>Skizzen Zur Kunstgeschichte der Modernen Medaillen-Arbeit (1929-1840).</i> Berlin. 1840.Schulze 737 B69s	Paper cover	No arsenic, high lead
14.	<i>Brittischer Damen-kalendar und Taschenbuch für das Jahr.</i> Frankfurt am Mayn. 1801.Taschenbuch 838 B862 1801	Paper Cover	Trace arsenic, no lead
15.	<i>Berliner Kalendar.</i> Berlin. 1835.Taschenbuch 838 B51 1829	Paper Cover	Trace arsenic, trace lead
16.	<i>Irish Melodies.</i> Moore, Thomas. London. 1866.Large 821.7 M82i 1866	Textile	High arsenic, trace lead

Appendix 2: SEM-EDX analysis

Scanning electron micrographs (SEMs) were obtained with a Hitachi S-3400N-II with a working distance of 10 mm. Samples were mounted on carbon tape on an aluminum pin stub mount. Micrographs were acquired using an accelerating voltage of 25.00 kV in the variable pressure mode. Energy-dispersive X-ray spectroscopy (EDX) was performed in the SEM using an accelerating voltage of 25.00 kV and a working distance of 10 mm. Spectra were collected and processed using the 'AZtec' software package (Oxford Instruments). Utilizing the mapping feature of the software, maps of samples were generated using a frame count of 20 for all samples to provide better consistency when comparing across sites and samples. The micrographs depicted in the SEM-EDX analysis visualized the surface morphology of the eraser samples. Those in Figure 2 show arsenic and copper distributed on the eraser crumb surface after remediation and those in Figure A2 show calcium, magnesium, silicon, and titanium on an eraser crumb surface from the control eraser with the underlying carbon type used for mounting the samples visible in the top left corner.

Where lead is detected via the La peak(s) in EDX, the presence of arsenic cannot be confidently determined without



Figure A1. The Bruker Tracer 5 g is mounted on a copy stand using a rail mount. A mirror allows users to see the flashing red light which indicates X-ray emission. Not shown in this image is a dosimeter mounted on the wall, which is changed on a quarterly basis. Courtesy of Northwestern University Libraries.

the $K\beta$ peak(s) because of the overlap of the $L\alpha_1$ lead and $K\alpha_1$ arsenic peaks. Arsenic is therefore not listed in the Elements column of Table 1 when lead is present and no

$K\beta$ arsenic peak was detected. In samples with prominent peaks of lead, the SEM-EDX test could not definitively confirm the presence of arsenic.

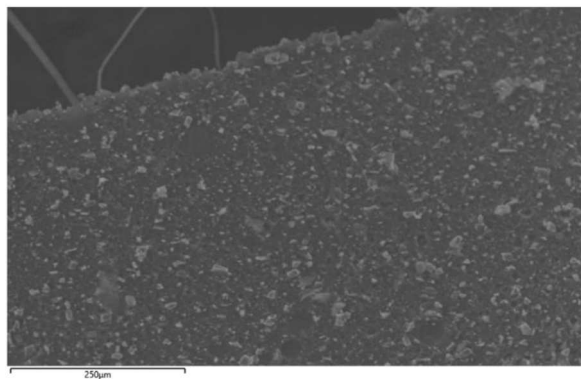
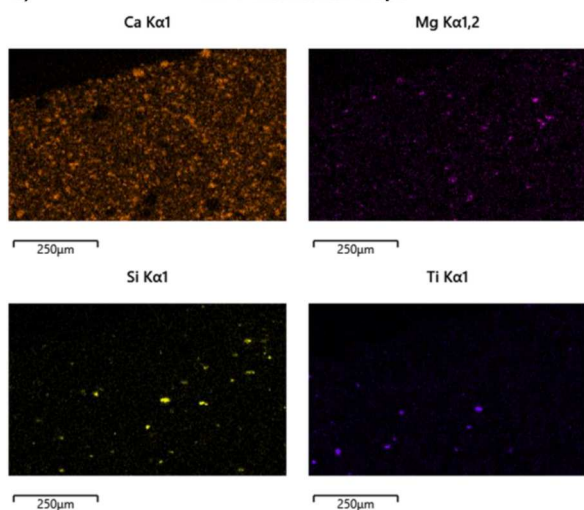
a) EDS Map Scanning Electron Micrograph**b) EDS Elemental Maps**

Figure A2. (a) Representative SEM of control eraser sample and (b) corresponding EDX maps. Individual EDX elemental maps for the control sample (b) showing calcium (Ca), magnesium (Mg), silicon (Si), and titanium (Ti).