

CLEANING PROTOCOL FOR MERCURIC CHLORIDE-CONTAMINATED HERBARIUM CABINETS AT THE SMITHSONIAN MUSEUM SUPPORT CENTER

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Abstract.—Mercuric chloride has been used to control insect and fungal infestations in natural history collections for the past two centuries. Due to health concerns, its use was discontinued in the mid-1980s, but specimens treated with mercuric chloride are commonly found in modern collections and present a hazard to collection staff and researchers. Cabinets used to store mercuric chloride-treated specimens also become contaminated with the substance and represent a source of exposure even after specimens are removed. A team at the US National Herbarium, in coordination with the Smithsonian's Office of Safety, Health and Environmental Management, developed a protocol to clean herbarium cabinets that were contaminated with mercuric chloride. Cabinets were cleaned with 70% ethanol and laboratory wipes, and effectiveness was measured using a portable mercury vapor analyzer and surface wipe sampling. Cleaning with ethanol was found to be more effective than just removing treated specimens, but the differences in reduction of airborne and surface mercury concentrations were not statistically significant. This study provides important insight and guidance for museums seeking to eliminate legacy mercuric chloride contamination from their herbarium cabinets.

Key words.—decontamination, herbarium specimens, mercuric chloride, United States National Herbarium

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INTRODUCTION

Mercuric chloride is an inorganic form of mercury known as corrosive sublimate because of its ability to sublime at room temperature (Webber et al. 2011). It was commonly used as a pesticide to treat natural history collections from the late 1700s to the mid-1980s (Briggs et al. 1983, Hawks et al. 2004, Purewal et al. 2007). Natural history specimens treated with mercuric chloride have been found in countries throughout the world, including the United Kingdom, Spain, South Africa, Canada, and the United States. These specimens are part of botany, anthropology, mammology, and ornithology collections (Sirois 2001, Oyarzun et al. 2007, Kataeva et al. 2009). Specimens were treated with mercuric chloride in various ways depending on the type of specimen and practice of the region. Application usually required the combination of mercuric chloride with a solvent, often ethanol, methanol, or phenol (Fosberg and Sachet 1965, Purewal 2001). Specimens were then sprayed, painted, or fully submerged in the solution and allowed to dry (Briggs et al. 1983, Hawks et al. 2004). Over time, mercuric chloride decomposes and sublimes, so some institutions retreated their specimens to ensure they remained pest free (Purewal 2001).

Mercuric chloride and its decomposition products are highly toxic to humans. Mercuric chloride exposure results in cell toxicity through direct contact and can cause

neurological and kidney damage (WHO 2013, EPA 2014). Mercuric chloride reacts with biologic material in museum specimens to yield elemental mercury and methyl mercuric chloride (Purewal 2001, Hawks et al. 2004, Oyarzun et al. 2007). Elemental mercury readily vaporizes and can cause respiratory failure at high concentrations. Organic mercury compounds, such as methyl mercuric chloride, are readily absorbed through the gastrointestinal tract and can cause neurological disorders and kidney toxicity. Although mercuric chloride has not been used commonly in more than 30 yr, its persistence and historic prevalence make it an ongoing hazard for conservators who care for natural history collections. To protect workers from the toxic effects of mercury in the United States, the Occupational Safety and Health Administration (OSHA) set a permissible exposure limit (PEL) of 0.1 mg/m^3 as an 8-hr time-weighted average (TWA) for elemental and inorganic mercury compounds and 0.01 mg/m^3 8-hr TWA for organic mercury compounds (OSHA 2005, 2006). The American Conference of Governmental Industrial Hygienists (ACGIH) recommends the same limit for organic mercury compounds but suggests a more protective limit of 0.025 mg/m^3 as an 8-hr TWA for elemental and inorganic mercury species (ACGIH 2011). No occupational or environmental health limits have been set for surface mercury concentrations, but the ACGIH assigned a “Skin” notation to all mercury compounds listed in the ACGIH Threshold Limit Value and Biological Exposure Indices guide (ACGIH 2011). This designates that a “significant contribution to the overall exposure” could occur via the cutaneous route (ACGIH 2011). Due to these risks, specimen cabinets should be well ventilated prior to access, and the use of gloves and other protective equipment has been recommended to limit dermal exposure (Hawks et al. 2004).

Most natural history museums around the world are aware of the dangers of housing mercuric chloride-treated specimens, and many have taken efforts to quantify and mitigate the potential exposures. One study at the Universidad Complutense de Madrid herbarium in Madrid, Spain, utilized a portable mercury vapor analyzer to measure airborne concentrations of elemental mercury vapor in the herbarium and found mercury concentrations up to $7.8 \text{ } \mu\text{g/m}^3$ in the main room (Oyarzun et al. 2007). A similar study from two herbaria in South Africa also found elevated airborne elemental mercury concentrations of up to $1.1 \text{ } \mu\text{g/m}^3$ near cabinets containing mercuric chloride-treated specimens (Kataeva et al. 2009). In the United States, a study at two university herbaria found concentrations of total airborne mercury over $100 \text{ } \mu\text{g/m}^3$ when opening herbarium cabinets. The US university study also collected samples of surface mercury and found concentrations up to 1.5 ng/cm^2 inside cabinets and up to 16.2 ng/cm^2 in the general workspace (Webber et al. 2011).

The Smithsonian Institution in Washington, DC, has conducted multiple mercury studies in the US National Herbarium at the Smithsonian’s National Museum of Natural History (NMNH). A study from 2004 at the National Herbarium developed a novel technique to detect mercury vapor in herbarium cabinets using mercury indicator powder. Their study found concentrations of elemental mercury vapor above $350 \text{ } \mu\text{g/m}^3$ within the herbarium cabinets (Hawks et al. 2004). Ongoing efforts led to a proposal and project, starting in 2012, that involved replacing the interleaving of unmounted herbarium specimens. The goal was to remove as much mercury saturated material as possible to preserve specimens in better archival storage. The long-term effects of this project are still being studied, and results on the impact of changing the interleaving are unknown at this time. Few other studies have investigated how to safely remediate specimens that have been treated with mercuric chloride. Many techniques have been

recommended, from direct manual removal to chemical methods and microwave heating, but the efficacy of these techniques for mercuric chloride removal from herbarium specimens is either inadequate or completely unknown (Odegaard and Zimmt 2008). The high concentrations of mercury vapor in many collections warrant an abundance of caution, and further investigation is necessary to determine how to effectively remediate mercuric chloride-treated collections.

Our study took place in Pod 2 of the Smithsonian Institution Museum Support Center (MSC) in Suitland, Maryland. The Smithsonian Institution MSC is an offsite facility that is separate from the permanent collections stored at other Smithsonian facilities. It was built to provide an upgraded storage facility for Smithsonian collections. The portion of the collection studied contained 144 herbarium bundles with approximately 5,025 specimens. The specimens were collected from portions of the United States, Mexico, South America, and New Caledonia. Collection dates range from as early as 1912 to the year 2000. This study built on the previous efforts of the NMNH staff to learn more about proper remediation techniques and the potential exposures during remediation. Our study examined how to best decontaminate herbarium cabinets after they have held mercuric chloride-contaminated specimens. This action has not been addressed in the literature but is an important step to ensure specimens are not recontaminated once they return to their storage cabinets.

MATERIALS AND METHODS

Sixteen herbarium cabinets were chosen from the collection in Pod 2 of MSC, containing 144 specimen bundles. All cabinets were manufactured by the Interior Steel Equipment Co. (Cleveland, OH) with internal dimensions of 72 in. (183 cm) × 26 in. (66 cm) × 19 in. (48 cm) and 24 equally sized compartments (see Fig. 1). Past work with the Interior Steel cabinets in Pod 2 confirmed that they have one full air exchange every 2–4 days (C. Hawks, personal communication, August 2014). MSC Pod 2 is a climate-controlled space with installed general mechanical ventilation. During the research project, the space was kept between 22.2°C and 24.2°C and at approximately 45% relative humidity. Cabinets were chosen based on mercury measurements from previous studies and were excluded if they had previous airborne mercury concentrations above 900 µg/m³ or below 100 µg/m³. Exclusion criteria were established based on maximum and minimum instrument limits of detection to provide detectable readings following the remediation method. Fourteen of the 16 cabinets were emptied of specimens. Seven of the emptied cabinets were cleaned while seven were left uncleaned. The remaining two of the 16 cabinets were used as controls and were left uncleaned with specimens inside. Due to staffing and time limitations, the sampling and cleaning was broken into two rounds. The first round sampled seven remediation cabinets and one control, and the second round sampled seven remediation cabinets and one control for a total of 14 sampled remediation cabinets and two controls. Each round took 4 wk to complete, with 1 wk between each stage of the experiment and used the methods listed below. A flowchart of the experimental stages is shown in Figure 2.

Week 1

Initial assessment.—Airborne mercury concentrations were measured with a Jerome J405 Gold Film Mercury Vapor Analyzer (Arizona Instrument LLC, Chandler, AZ). The operating principle of the Jerome is that air is pulled through an acidic gas filter to remove acidic gases that interfere with the gold film sensor, and then the air is passed over a gold film sensor that adsorbs any elemental mercury vapor. The adsorbed mercury

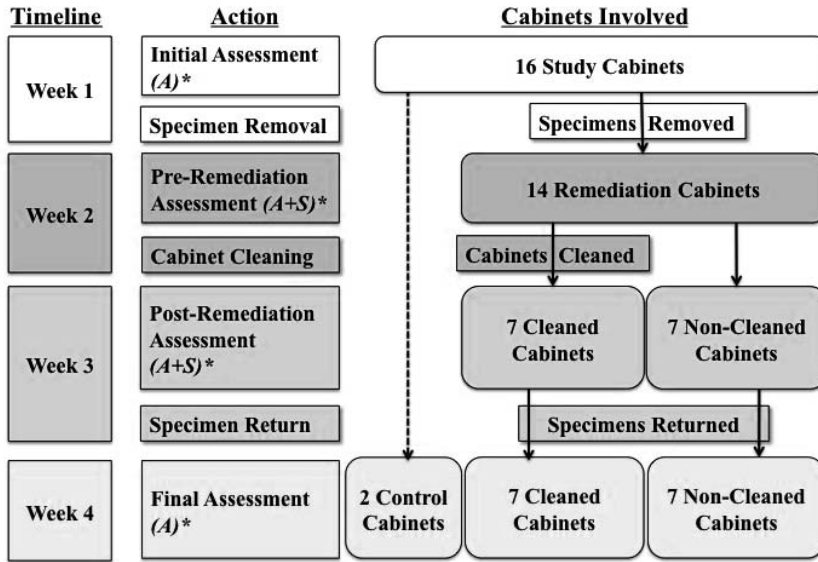


Figure 1. Herbarium cabinet showing specimen storage and shelf locations.

causes an increase in resistance on the gold film sensor that is proportional to the concentration of mercury vapor in the air. The gold film sensor eventually becomes saturated and must undergo a regeneration cycle, in which the sensor is heated to vaporize the adsorbed mercury. Each measurement takes 16 sec and can measure mercury vapor concentrations from $0.5 \mu\text{g}/\text{m}^3$ to $999 \mu\text{g}/\text{m}^3$ with an accuracy of $\pm 5\%$ at $25 \mu\text{g}/\text{m}^3$ (Arizona Instrument 2014).

The initial assessment required the measurement of airborne mercury concentrations to establish the initial airborne mercury concentration within each cabinet. Repeated measurements were not possible because the mercury vapor quickly dissipated once the cabinet doors were opened. To take each reading, the cabinet door was opened 1–2 in. to allow the Jerome probe to fully access the cabinet. The probe was moved approximately 2 ft above and below the midpoint of the cabinet during the 16-sec sample to achieve the most representative sample possible.

Specimen removal.—After the initial airborne mercury measurements were taken, all specimens were removed from their cabinets with the exception of the control cabinets. Prior to opening the cabinets for specimen removal, a Nilfisk SS Mercury Vacuum (Nilfisk Industrial Vacuums, Morgantown, PA) was used to remove mercury vapors from



**(A)* = Airborne Sampling, *(A+S)* = Airborne and Surface Sampling

Figure 2. Flowchart of experimental stages showing cabinets and sampling methods involved in each stage.

the cabinets and minimize exposure to study personnel. The mercury vacuum uses an activated-carbon filter and HEPA filter to remove mercury vapor and particulates and has a flow rate of 40.8 m³/hr (Nilfisk 2011). The vacuum hose was inserted into the bottom of each cabinet and allowed to run for approximately 5 min. The cabinets were then fully opened and specimens were removed and transported to chemical fume hoods for storage during the experiment. The location of each specimen was noted so they could be returned to their original location.

Week 2

Preremediation assessment.—After removal of the specimens, the cabinets were left undisturbed with their doors closed for 1 wk. Specimens were removed 1 wk prior to the preremediation assessment to minimize variability in subsequent measurements. Past experience with this collection showed that measurements taken immediately after the removal of specimens were often highly variable, and variability declined after cabinets were left undisturbed for several days. The preremediation stage involved sampling the cabinets for airborne and surface mercury concentrations prior to applying remediation methods. First, airborne mercury concentrations were measured using the same methods listed in the initial assessment section above. Then surface mercury wipe samples were collected from three locations within each cabinet. Surface mercury measurements were collected using SKC Smear Tabs in accordance with the wipe sample methods listed in OSHA Method ID-145 (OSHA 1989). Wipe samples were placed in digestion tubes provided by Analytics Laboratory (Ashland, VA) and shipped to Analytics Laboratory for analysis. Analysis was conducted using EPA Analytical Method SW846 7471A cold-vapor atomic absorption, which has a limit of detection of 0.075 µg total mercury



Figure 3. Wipe sampling using Smear Tab and disposable wipe template. Notice the black stains that suggest the presence of mercuric chloride.

(Analytics Laboratory 2011). Wipe samples were collected over an area of 100 cm^2 using disposable $10 \text{ cm} \times 10 \text{ cm}$ paper templates (Fig. 3). Wiping over this area gives a limit of detection of $0.00075 \text{ } \mu\text{g}/\text{cm}^2$ total mercury for this experiment. To ensure an even distribution of wipe samples, one wipe was taken from the top (shelf 1–3), middle (shelf 6–8), and bottom (shelf 10–12) of each cabinet on either the left or the right side of the central divider. To minimize bias, a random number generator was used to assign the left or right side and the exact shelf for sampling. Wipe sampling each cabinet required approximately 10–15 min.

Cabinet cleaning.—Seven of the 14 remediation cabinets were cleaned using 70% ethanol solution and lint-free laboratory wipes. Ethanol was chosen because it was commonly used as a solvent for mercuric chloride during specimen applications. Wipes were sprayed with ethanol prior to cleaning and were replaced often to minimize possible spreading of mercury from one shelf to another. Wipes were used to clean all exposed surfaces on the interior of each cabinet, starting from the top to the bottom and then the door.

A personal sampling pump was worn during cleanings to monitor the personal exposure of project personnel to mercury vapor and particulate. Personal monitoring was conducted using a Gilian Low Flow Sampler (Model LFS-1130) operated between 0.22 and 0.25 L/min. Samples were collected in accordance with NIOSH Method 6009 (for vapor) and OSHA Method ID-145 (for particulate) (NIOSH 1994, OSHA 1989) and analyzed by Analytics Laboratory. OSHA Method 145 and NIOSH Method 6009 both use cold-vapor atomic absorption for analysis and have limits of detection of $0.05 \text{ } \mu\text{g}$ and $0.025 \text{ } \mu\text{g}$ per sample, respectively (Analytics Laboratory 2011). During all cleaning and sampling, project personnel wore half-face air purifying respirators equipped with mercury vapor cartridges and nitrile gloves.

Week 3

Postremediation assessment.—Cabinets (both cleaned and noncleaned) were left undisturbed with the doors closed for 1 wk prior to the postremediation assessment. The

postremediation assessment of airborne and surface mercury concentrations employed the same methods used for the preremediation assessment. Postremediation surface wipes were taken from the same shelf that wipe samples were taken from in the preremediation assessment. Specimens were then returned to each cabinet in their original location and allowed to sit for 1 wk, with the doors closed, prior to the final assessment.

Week 4

Final assessment.—A final sample of airborne mercury was taken from all 16 cabinets using the method described in the initial assessment phase. This sample was collected to determine how the mercury concentration within each cabinet responded when the specimens were returned. Airborne mercury concentrations were measured in each control cabinet during the initial and final assessments.

DATA ANALYSIS

All statistical analysis was conducted using the statistical software package Stata/IC 13.1.

Mean Hypothesis Tests

Mean hypothesis tests (two-sample *t*-test and Wilcoxon rank-sum test) were used to compare the reduction in airborne and surface mercury between cleaned and noncleaned cabinets. Mercury reduction values were established using the data from the pre- and postremediation stages and calculated using the following equation:

$$\begin{aligned} &\% \text{Hg Reduction} \\ &= (1 - (\text{Postremediation Measurement} / \text{Preremediation Measurement})) \times 100. \end{aligned}$$

Values for the reduction in surface mercury were not calculated if the preremediation measurements were below the limit of detection ($0.00075 \mu\text{g}/\text{cm}^2$; 17/42 wipe locations [40%]). All of the locations with surface mercury concentrations below the limit of detection during the preremediation assessment also had surface mercury concentrations below the limit of detection during the postremediation assessment. Several techniques were used to compensate for surface mercury measurements that were below the limit of detection in the postremediation assessment ($0.00075 \mu\text{g}/\text{cm}^2$; 10/42 wipe samples [24%]). First, postremediation measurements that were below the limit of detection were set at the limit of detection, the most conservative approach. Second, postremediation measurements that were below the limit of detection were set to the limit of detection divided by the square root of 2. This method is a commonly used replacement method for censored data and is less error prone than substitution with the full limit of detection (Croghan and Egeghy 2003). Finally, full censoring of the postremediation data was conducted, and all measurements from the postremediation assessment were removed if they were below the limit of detection, resulting in a sample size of 15 wipe locations.

The calculated reductions in airborne mercury were normally distributed so a two-sample *t*-test was used to determine if a statistically significant difference in mean reduction existed between the cleaned and noncleaned cabinets. The calculated reductions in surface mercury were not normally distributed, so a Wilcoxon rank-sum test was used.

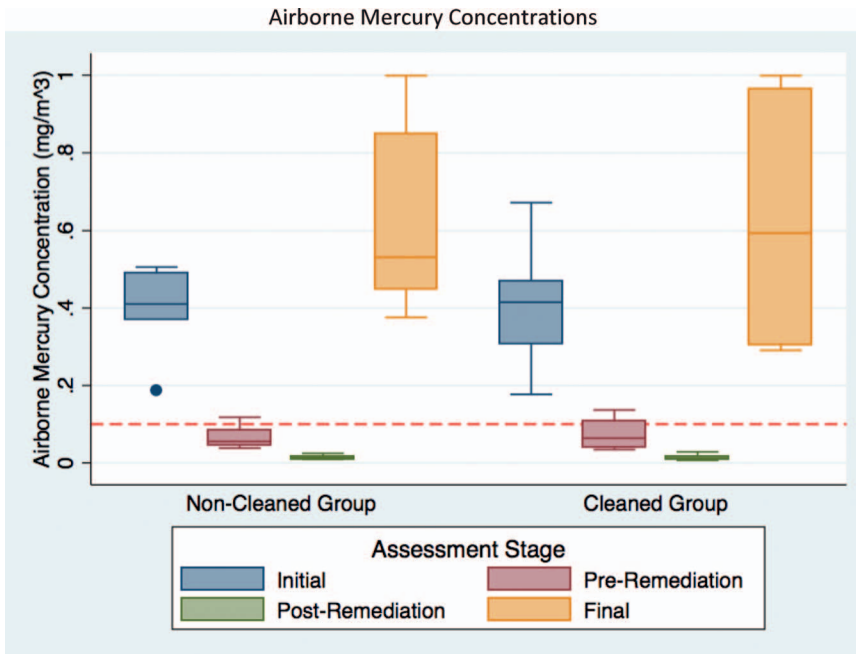


Figure 4. Box plots of the airborne mercury concentrations for the cleaned and noncleaned groups at each assessment stage. The dashed line designates the OSHA PEL 8-hr TWA for inorganic mercury vapor.

RESULTS

Box plots of the airborne mercury concentrations at each assessment stage are shown in Figure 4. Box plots of the surface mercury concentrations at each assessment stage are shown in Figure 5. The data in Figure 5 represent only the fully censored surface mercury concentrations that were included in the final hypothesis test.

Mean Hypothesis Tests

The data in Table 1 show the results from the hypothesis testing for reductions in airborne and surface mercury between the cleaned and noncleaned groups. The differences in mean reductions suggest better efficacy for ethanol cleaning, but these findings were not statistically significant. A spaghetti plot of the surface mercury concentrations from the pre- and postremediation assessments is shown in Figure 6. The data represented here represent uncensored surface mercury data and are linked by sample location. The cleaned cabinets tend to show greater reductions in surface mercury concentrations than the noncleaned cabinets, also suggesting that cleaning with ethanol is more effective at removing mercury contamination than removal of specimens alone.

The control cabinets did not show significant changes in airborne mercury concentrations during the experiment. One control increased from 0.369 to 0.500 mg/m³ while the other decreased from 0.554 to 0.469 mg/m³.

Personal Monitoring

Mercury vapor and particulates were below detectable limits for all breathing zone samples taken during case cleanings. Based on limits of detection, actual mercury

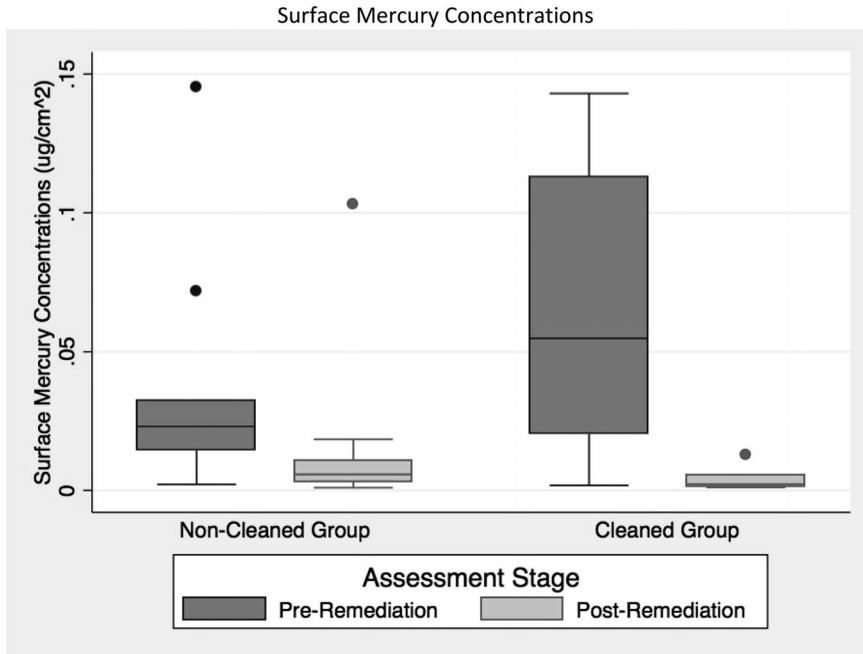


Figure 5. Box plots of the surface mercury concentrations for the cleaned and noncleaned groups at the pre- and postremediation assessments. Nondetectable measurements ($< 0.00075 \mu\text{g}/\text{cm}^2$) from the pre- and postremediation assessments were censored.

vapor exposure levels during case cleaning ranged between < 0.209 and $< 0.242 \mu\text{g}/\text{m}^3$ as an 8-hr TWA for total mercury. Actual mercury particulate exposure levels during case cleaning ranged between < 0.420 and $< 0.496 \mu\text{g}/\text{m}^3$ as an 8-hr TWA for total mercury.

DISCUSSION

The evidence gathered during this study presents interesting findings about the behavior of mercuric chloride-contaminated herbarium cabinets. The study aim was to

Table 1. Comparison of the reduction in airborne and surface mercury concentrations between cases that were or were not cleaned with ethanol after all herbaria specimens were removed.

Comparison	Average % reduction in cleaned (n)	Average % reduction in noncleaned (n)	Difference in mean (%) (95% CI)	t-test p value
Airborne Hg	82.4 (7)	79.6 (7)	2.8 (−0.7–6.4)	0.11
Surface Hg (nondetects = $0.00075 \mu\text{g}/\text{cm}^2$)	67.8 (14)	46.1 (11)	21.7 (−23.3–66.6)	0.44 ^a
Surface Hg (nondetects = $0.00075/\sqrt{2}$)	75.3 (14)	48.3 (11)	26.9 (−16.3–70.2)	0.19 ^a
Surface Hg (all nondetects censored)	84.8 (6)	43.4 (9)	41.4 (−33.3–116.0)	0.06 ^a

95% CI = 95% confidence interval.

^a p value from Wilcoxon rank-sum test.

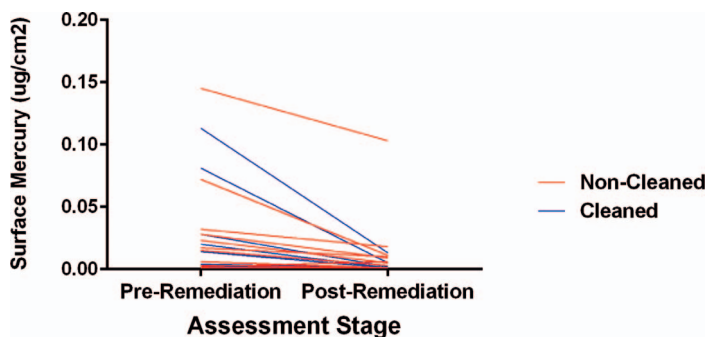


Figure 6. Spaghetti plot of the uncensored surface mercury concentrations from the pre- and postremediation assessments. Samples taken from the same location are connected with a line. Samples that were below the limit of detection were set at the limit of detection ($0.00075 \mu\text{g}/\text{cm}^2$) for this figure.

determine if cleaning with wipes and ethanol was more effective at removing mercury contamination than simply removing the specimens. The findings from the study suggest that cleaning with ethanol is more effective at removing both airborne and surface mercury contamination, although none of the differences in mercury reduction were found to be statistically significant ($p < 0.05$). It is possible that the airborne mercury was reduced considerably by opening and closing of the cabinets, and that cleaning with ethanol had little additional effect on reducing airborne mercury.

After the mercuric chloride-treated specimens were put back in their respective cabinets, airborne mercury concentrations quickly increased and surpassed the airborne mercury measurements from the initial assessment. During the experiment, the specimens were transported to and stored in chemical fume hoods. During removal, transport, and return of the specimens they were exposed to light. According to Oyarzun et al. (2007), light is an accelerating factor in the decomposition of mercuric chloride to elemental mercury. This may have contributed to the decomposition of mercuric chloride to elemental mercury, which then could have vaporized upon return to the cabinets. The sharp increase in airborne mercury concentrations following the return of specimens to their cabinets emphasizes the importance of specimen decontamination. If specimens are not properly decontaminated, then cabinet decontamination will have little lasting effect on the overall mercury contamination of mercuric chloride-treated collections.

Finding a statistically significant difference in surface mercury reduction was hampered by the limit of detection for the wipe analysis. Of the 42 paired wipe samples, 17 pairs (40%) were below the limit of detection during the prerediation assessment and were excluded. Ten additional pairs (24%) were below the limit of detection at the postremediation assessment, which limited our ability to determine the true reduction in surface mercury from prerediation to postremediation assessments. When these 10 pairs were also excluded, the sample size was reduced to only 15 paired wipe samples (36% of the original sample size) with an average of 84.8% reduction in the cleaned group and 43.4% reduction in the noncleaned group. Even though the sample size was greatly reduced, the statistical significance was greatly improved from 0.44 to 0.06 when we examined only the wipe sample pairs with two detectable concentrations. This suggests that cleaning with ethanol is an effective means of removing mercury contamination from within herbarium cabinets, providing encouraging evidence that mercuric chloride-contaminated herbarium cabinets can be efficiently remediated. Perhaps more

encouraging is that simply removing the contaminated specimens may be sufficient for the majority of decontamination needs. With the exception of sampling, the noncleaned cabinets were left undisturbed during the experiment and still achieved almost 80% reduction in airborne mercury contamination and greater than 40% reduction in surface mercury contamination. This may suggest that opening the cabinets for sampling provided sufficient ventilation to achieve significant reductions in mercury contamination. It may also be a function of the herbarium cabinet's natural air exchange rate and the mechanical ventilation within MSC Pod 2. Museums that have adequate ventilation may be able to achieve sufficient reductions in mercuric chloride contamination by removing the specimens from their cabinets and allowing several weeks for air exchange. Museums that use these techniques to decontaminate herbarium cabinets must exercise caution because differences in ventilation and mercuric chloride concentrations could lead to hazardous concentrations of mercury vapor in occupied spaces that are adjacent to collections.

CONCLUSIONS

Mercuric chloride continues to be a persistent contaminant in natural history collections that poses a health hazard to conservators and researchers. There has been little research on effective means of remediation for specimens and their storage containers. This study confirmed that it is possible to effectively decontaminate herbarium cabinets that are contaminated with mercuric chloride. Manual cleaning appeared to be more effective than removal of specimens alone, but both methods achieved significant reductions in mercury contamination. Although personal exposure to airborne mercury did not pose a serious risk during cleaning efforts, we caution that our findings may not translate directly to other museums due to differences in mechanical ventilation, cabinet material, and pesticide treatment history. More research is needed to determine effective remediation techniques that are safe for both employees and collections. Until effective methods have been determined and adopted, museum staff should wear gloves, lab coat, and respiratory protection whenever they access or handle specimens suspected of being treated with mercuric chloride.

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