Substituting SPME for noses in the detection and quantification of mothball vapors from textiles in the National Museum of the American Indian collection

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ABSTRACT

Naphthalene is a volatile solid that often has been used as a pesticide to treat artifacts, particularly ethnographic material, and natural history collections. Naphthalene residues pose an ongoing health risk in museums and to communities that receive contaminated artifacts through cultural repatriation. The Museum of the American Indian disposed of its supply of "moth flakes" in 1985, but noxious residues remain on collections. It has been demonstrated that naphthalene vapors become stronger in humid air, which increases the potential health risk. A method for quantifying naphthalene vapor concentrations using SPME-GC/FID is presented, and the relationship between naphthalene, water vapor, and textile fibers is explored as a step in the development of a treatment to remediate the pesticide residues.

INTRODUCTION

From the early 1900s into the 1980s the Museum of the American Indian Heye Foundation (MAI) in New York City sought to preserve its collection of Native American cultural material from insect infestation with a variety of pesticides. Museum records show that naphthalene moth flakes were the pesticide of choice, even though para-dichlorobenzene also was available at the time, and that the flakes were used heavily and routinely on the collection. Naphthalene was discontinued decades ago, but noxious residues remain on organic materials in the collection, which is now part of the Smithsonian Institution's National Museum of the American Indian (NMAI). Assessment of the potential health risk of naphthalene-contaminated collections to museum staff, visiting researchers, and Native American recipients of repatriated museum objects has required cross-disciplinary collaboration between conservators, Native American tribal representatives, materials scientists, chemists, medical toxicologists, risk assessors, and specialists in cultural repatriation. These efforts have been hampered by difficulties in quantifying the contamination. Of particular concern are the elevated concentrations of naphthalene vapor experienced during the humidification and wet-cleaning of the museum's textiles in preparation for exhibition (Heald 2005). Similarly, repatriated ceremonial regalia while worn may become warm and damp with perspiration and emit more naphthalene vapors. Earlier mothball contamination identification studies at NMAI used solid-phase microextraction (SPME) sampling with gas chromatography-mass spectrometry (GC/MS), but did not quantify the vapors present (Heald 2005, Ormsby 2006). Quantification is key to evaluating health risk and assessing the efficacy of mitigation techniques being developed by the authors, particularly rapid air exchange. The aims of the current research have been to develop quantitative methods to measure naphthalene vapors emitted by contaminated textile artifacts, and to investigate why naphthalene vapors are stronger in humid conditions. A series of experiments explores the relationships between naphthalene vapor, temperature, humidity, and different textile fibers.

BACKGROUND

Naphthalene

Naphthalene is a bicyclic aromatic hydrocarbon that was discovered in coal tar in 1820 (Kidd 1821). The white crystalline solid sublimes into a

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distinctive strong smelling gas and has pesticidal properties. It is insoluble in water in concentrations above approximately 30 mg/L but is soluble in many organic solvents including alcohols, benzene, and substituted benzenes.

Reported cases of naphthalene poisoning are uncommon, but its toxicity is well documented. For most of the population, acute exposure by inhalation, ingestion, or skin contact can cause headache, nausea, vomiting, diarrhea, malaise, confusion, anemia, jaundice, convulsions, and coma (USEPA 2000). Acute symptoms usually abate upon removal of the naphthalene, although hemolytic anemia, a serious blood complication that shreds red blood cells, can occur days after exposure. Newborns and people with glucose-6-phosphate dehydrogenase (G6PD) deficiency are much more susceptible to these conditions, which can result in death (Cock 1957, Beutler 1994, Bates 2002, Frank and Maj 2005, Bronstein et al. 2008). G6PD deficiency is a common hereditary disease that affected 6% of the world population (i.e., 1 out of 17 people) in 2005. Women who are pregnant or breast feeding are also at risk; a mother's anemia can affect the unborn child, and naphthalene can be passed through the blood or breast milk (ATSDR 2005). Chronic exposure has been linked to cataracts and "is reasonably anticipated to be a human carcinogen" based on studies in animals (ATSDR 2005, NTP 2011). Naphthalene is a regulated pesticide in the United States and subject to legal and recommended exposure limits by health agencies (Table 1). The United States is not alone in addressing the health risks of naphthalene. For instance, the European Union banned the sale of naphthalene products in 2008 (CEC 2008).

Table 1

Occupational Exposure Guidelines in the United States

Threshold Limit Value-Short Term Exposure Limit (TLV) (USEPA)	15 ppm
Permissible Exposure limit (PEL) (USEPA)	10 ppm
General Public Acceptable Limit (USEPA)	1 ppm
Odor Threshold (USEPA)	85 ppb
Occupational Exposure Level (OEL) (Makos 2001)	15 ppb

Naphthalene first was documented as a moth repellent in museums in 1887 (Odegaard and Sondongei 2005), with apparent widespread use soon after. Records show that MAI purchased naphthalene as early as 1918 and that 200 pound drums were purchased annually from 1945 to 1975. The museum disposed of all naphthalene as toxic waste in the mid-1980s under growing concerns about its toxicity (Pool 2001). The collection since has been moved from New York and re-housed in a specially built facility in Maryland, but mothball odors remain, especially on rolled textiles. This residual contamination is an ongoing concern for people who work closely with collections on a daily basis and for tribal communities receiving ceremonial objects through repatriation legislation.

A further challenge is the observed increase of naphthalene vapors in humid conditions. NMAI conservators have noted that naphthalene fumes become stronger during washing and humidification of certain textiles, under warm lights during photography, and in ceremonial use. This was proven analytically by Heald et al. (2005) and Ormsby et al. (2006). Heald et al. (2005) demonstrated that the increased naphthalene concentration is related to the type of textile fiber.

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Vapor detection

Previous research at NMAI used SPME-GC/MS to detect and identify naphthalene vapors, and a clear description is provided by Heald et al. (2005) and Ormsby et al. (2006). Current research builds upon that technique with two modifications. First, the GC is equipped with a flame ionization detector (FID) that has high sensitivity but only identifies compounds by comparison to known standards. In contrast, a mass spectrometer is less sensitive but identifies substances from characteristic fragmentation patterns in the chromatogram.

Second, the SPME fiber sampling method was changed from *exposed mode* to *time-weighted average (TWA) mode* (Lee et al. 2002, Chen and Pawliszyn 2003, Ouyang et al. 2005, Ormsby et al. 2006). In exposed mode, the SPME fiber is exposed completely out the end of its needle. The advantage is that more naphthalene can be collected in a shorter time frame, but the calibration process is more involved. Each fiber must be calibrated to a range of vapor concentrations using the same exposure time, and generating the known concentrations is challenging (Ouyang et al. 2005, Ormsby et al. 2006). In TWA mode, the SPME fiber is retracted to a specific distance inside its needle. The same amount of naphthalene can be collected, but over a longer duration. The calibration can be built from a single known vapor concentration (e.g., saturated atmosphere) because the geometry of the collection mode obeys Fick's law of diffusion (Chen and Pawliszyn 2003). Another advantage is flexibility to adjust sampling duration without having to recalibrate the SPME fiber. Thus, for highly concentrated atmospheres, the measurement can be made quickly (so the fiber does not saturate), and for very low concentrations, sampling time can be extended, which improves the lower detection limit.

EXPERIMENTAL

Solid naphthalene, naphthalene saturated textile swatches, and historic textiles in the NMAI collection were enclosed in glass jars or plastic bags, and the air within was sampled using principles and bagging methods described by Ormsby et al. (2006), but with the SPME TWA mode. A series of experiments was performed that varied the amount of naphthalene present in the containers, relative humidity (RH), and textile fiber.

GC/FID

Ormsby's (2006) method was used as a starting point for the GC/FID method. Naphthalene concentrations were measured with an Agilent 6890N GC with a split/splitless injection port and FID. The injection port used a Restek 1.0 mm Drilled Uniliner inlet liner and was operated at 310°C with helium flowing at 1 mL/min. Chromatographic separations used a Supelco SLB-5ms column, 30 m × 0.25 mm i.d., 0.25 µm film thickness, and an Alltech deactivated FSOT guard column, 10 m × 0.32 mm i.d. The oven was programmed: 40°C for 2 minutes, then ramped at 12°C/min to 140°C and held for 3 minutes, then ramped at 30°C/min to 250°C and held for 1 minute. Signals were collected, recorded, and integrated with Agilent Chemstation Rev. B.1.03. High-purity helium, hydrogen, and air were supplied by Airgas, Inc.

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The GC/FID was calibrated with commercially prepared liquid standards of naphthalene (analytical standard, Supelco 200 μg/mL ampule) dissolved in GC grade methanol to 5, 10, 50, 100, and 200 μg/mL concentrations and injected by autosampler in triplicate. A calibration curve was generated by plotting peak areas against mass of naphthalene injected and using classical least squares regression in Microsoft Excel.

SPME

Vapor samples were collected onto 85 μm carboxen/PDMS Stableflex Supelco SPME fibers that were held with a Supelco SPME fiber holder for use with manual sampling. The SPME fibers were checked by GC between analyses to ensure a clean fiber and to collect a blank chromatogram.

Each jar septum or plastic bag was pierced with a 20 gauge disposable needle to create an opening into which the SPME needle was inserted. At the end of the exposure, the SPME needle was removed and capped with a septum until analysis.

The TWA mode can be calibrated using one known concentration because the geometry of the fiber position in the needle obeys Fick's law of diffusion (Chen 2003). SPME fibers were calibrated at 0% and 100% RH to evaluate whether humidity influenced their absorption rate. For the dry condition, solid naphthalene (450 mg) and Drierite dessicant (anhydrous calcium sulfate doped with cobalt chloride indicator, W.A. Hammond Drierite Co., Ltd.) were sealed in a jar. For the 100% RH condition, another jar was set up with 450 mg of naphthalene crystals and 500 μL deionized water in a 5 mL beaker. Each jar was sampled for 5 minutes. The sampling rate (μL/min) was calculated using the equation:

$$SR = \frac{(StdPeakArea - BlankPeakArea) \bullet A + B}{((44.935 \bullet T) - 531.81) \bullet min}$$

where SR is the sampling rate, A and B are the slope and intercept from the GC calibration curve, T is temperature in Celsius, and min is minutes the SPME fiber was exposed in TWA mode. The results from at least three SPME fiber exposures were averaged to determine the SPME sampling rate for naphthalene.

Calculation of detected naphthalene vapor concentrations

Naphthalene vapor concentrations were calculated with the equation:

$$C(ppm) = \frac{(SamplePeakArea - BlankPeakArea) \bullet A + B}{SR \bullet min \bullet 5.24}$$

where 5.24 is the conversion from ng/ml (equivalent to mg/m³) to ppm by volume.

Unsaturated naphthalene vapor experiments

Unsaturated naphthalene atmospheres were created with swatches of woven cotton, linen, silk, and wool textiles (1.25" × 1.25") that had been exposed to saturated naphthalene vapor for nine years (Heald et al. 2005, Ormsby et al. 2006). Swatches were sealed in individual jars and allowed to equilibrate for at least 24 hours before sampling with the SPME needle

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Figure 1
Zuni sash, National Museum of the American Indian, Smithsonian Institution (01/2040). Photo: NMAI Photo Services

for 5 to 20 minutes and analyzing with GC/FID. Each swatch was measured at ambient conditions (22°C, 40% RH) and again after 1.5 hours at humid conditions (22°C, 100% RH).

Experiments with historic textiles

Five Navajo and Puebloan wool textiles in the NMAI collection were analyzed. Each was enclosed in a heat-sealed bag of polyvinyl fluoride plastic (Dupont Tedlar) and allowed to equilibrate at ambient conditions for 24 hours before air sampling. One textile was humidified for 1.5 hours to simulate a typical humidification treatment (Figure 1). For both ambient and humid conditions, the air was sampled for 1.5 hours. The punctured bags were sealed with pressure sensitive tape.

Relative humidity manipulation

Humidity in all enclosures was manipulated by drying with Drierite desiccant or humidifying with deionized water. For experiments with saturated naphthalene vapor atmospheres, water was held in a small beaker. For experiments with swatches or historic textiles, water-soaked blotter paper was isolated from the textile with Gore-Tex, a waterproof but vapor permeable membrane.

RESULTS AND DISCUSSION

Effect of temperature on saturated naphthalene concentration

Saturated naphthalene vapor concentrations ($C_{(ppm)}$) were calculated based on published vapor pressures (Dortmund 2013) using the following:

$$C_{(ppm)} = 8.57T - 101.49 \qquad T = 18.4\text{-}26.4°C\ (65.12\text{-}79.52°F)$$

where T is the temperature (°C). Based on this relationship where concentration increases with temperature, the saturated naphthalene vapor concentration at 22°C is 87 ppm. This concentration exceeds all exposure limits (Table 1) and should caution anyone working in the presence of naphthalene crystals.

Effect of humidity on SPME fiber absorption rate and saturated naphthalene concentration

For one fiber, the sampling rate for naphthalene was 7.3 ± 0.3 μL/min at 0% RH and 7.1 ± 0.1 μL/min at 100% RH. This verifies the SPME fiber sampling rate does not change with humidity and indicates that moisture has a negligible effect on saturated naphthalene vapor concentration. The observation is significant because the authors had considered the possibility that naphthalene might also exist as a *liquid* in a humid space, which could drastically increase the amount of inhalable naphthalene in the air (Smolìk and Schwarz 1997). The presented experiments did not support this hypothesis.

Effect of humidity on naphthalene vapor concentration in unsaturated naphthalene atmospheres

Textile swatches at ambient conditions emitted between 4 and 40 ppm naphthalene vapors (Figure 2, Table 2). All swatches emitted higher

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concentrations of naphthalene vapor than the general public limit, odor threshold, and occupational exposure limit (OEL). Boosting the humidity to 100% RH increased the naphthalene vapors by 2-fold for the cotton, 2.9-fold for the linen and silk, and 9.1-fold for the wool swatches, which approaches the 87 ppm calculated saturated concentration. The experiment did not control for weight, volume, or surface area of the textiles, so these values are not generalizable. However, the results suggest that the type of fiber in a textile (and artifact materials in general) affects the concentration of naphthalene vapor in a space.
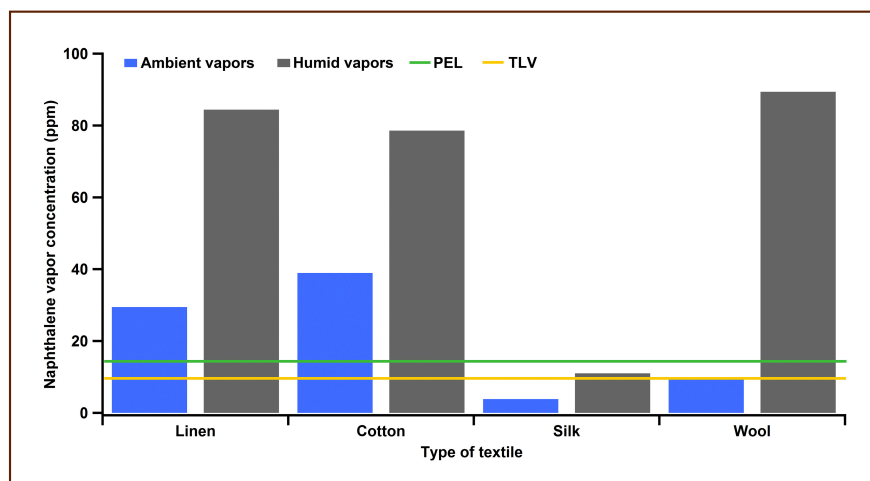

Figure 2
Naphthalene concentration measured at ambient and humid conditions

Table 2
Naphthalene concentrations for swatches and historic textile in ambient and high-humidity conditions

Textile	Ambient (ppm)	Humid (ppm)	Increase (%)
Linen	29	84	287%
Cotton	39	79	202%
Silk	3.8	11	287%
Wool	9.8	89	908%
NMAI Textile 01/2040	1.1	1.8	166%

Comparison of naphthalene vapors emitted from historic textiles at ambient and humid conditions

In ambient conditions, naphthalene vapors emitted by four of the historic textiles ranged from 500–1100 ppb and breeched the OEL (Figure 3). For the most contaminated textile (NMAI Object 01/2040), the measured concentration was just over the general public limit, and the vapors increased by 66% upon humidification to slightly less than 2 ppm (Figure 4, Table 2). It is possible that this last textile did not have sufficient time to equilibrate, so the equilibrium concentration may have been higher (Fusek 1985). The measurement of the fifth textile was below the detection limit of 400 ppb for a 1.5 hour exposure (Figure 3). Even though vapors emitted by the historic textiles were significantly lower than the doped textile swatches, the vapor concentrations were still too high to safely work with the textiles without protection or sufficient ventilation.

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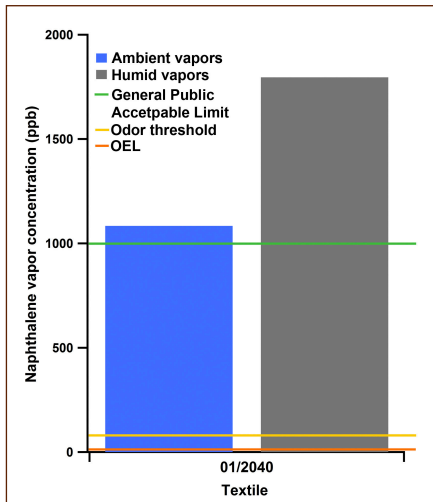OF THE AMERICAN INDIAN COLLECTION**

Figure 4

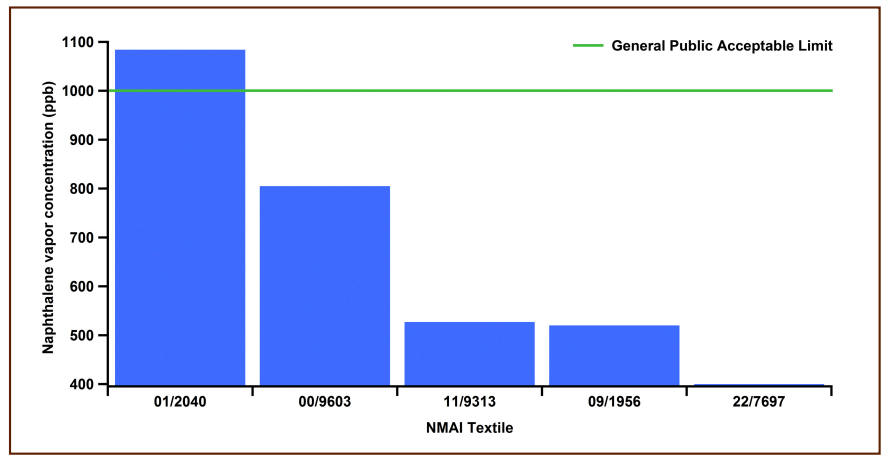Naphthalene concentrations measured from NMAI Object 01/2040 at ambient and humid conditions

Figure 3

Naphthalene concentrations measured over historic textiles at ambient humidity

CONCLUSION

A straightforward method to calibrate and measure naphthalene vapor concentrations was developed and implemented using SPME-GC/FID. This progress is part of a long-term project to measure and remove naphthalene from museum collections, to protect museum staff and researchers who work with collections and Native communities that receive repatriated objects from the collection. Unlike earlier work, a more sensitive FID was used instead of a mass spectrometer, and the SPME sampling was changed to TWA mode. The benefits of TWA sampling are a simpler calibration process and freedom to adjust the exposure duration, for example to enhance detection of high or low concentrations.

The relationship between the saturated vapor concentration of naphthalene and temperature is well established. The observation that naphthalene vapors emanating from textiles increases with humidity suggests that interactions between the textile, air, water, and/or naphthalene are taking place. The experiments of Heald et al. (2005) were replicated and the naphthalene vapor concentrations were determined. In an enclosed space with naphthalene crystals and no textile, air will saturate with naphthalene vapors at 87 ppm (at 22°C) regardless of the ambient humidity. This is an important finding as it suggests that the *maximum* concentration for naphthalene in air is determined by temperature. However, with experimentally prepared textile swatches, it was observed that the humidified naphthalene concentration was twice the ambient for cotton, nearly 2.9 times for linen and silk, and 9.1 times for wool. After a brief humidification, a naphthalene-contaminated, historic textile gave off 66% more naphthalene vapors than in ambient conditions. These values confirm the dependence of naphthalene vapors on humidity when the air is not saturated with the moth ball vapor and when linen, cotton, wool, or silk textiles are present. This suggests that the mechanism may involve absorption and desorption of the naphthalene from the fibers in the presence of water molecules.

This research is part of an ongoing investigation of rapid air exchange as a passive, chemical-free treatment to reduce naphthalene residues on artifacts. Next steps in this research include applying the measurement method described here to the experiments to remove naphthalene residues

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from textiles. This improved understanding of naphthalene's behavior in a humid environment will help optimize remediation of the pesticide and offers a new perspective on safe environmental conditions for interacting with contaminated objects.

GLOSSARY

Exposed sampling mode: SPME sampling mode that extends the SPME fiber outside its metal needle while collecting vapors, and is time dependent.

General public acceptable limit: 10% of PEL, used for general public exposure levels in a museum setting (Makos 2001).

Fick's law of diffusion: Law that describes diffusion and assumes a concentration gradient, used in TWA calculations.

Occupational exposure limit (OEL): Maximum concentration for museum staff working in a naphthalene-treated collection area for a 40-hour work week (Makos 2001).

Odor threshold: Lowest concentration a person can detect by smell.

Permissible exposure limit (PEL): Maximum concentration to which a worker in the United States can be legally exposed, expressed as a time-weighted average over an 8-hour work day (USEPA 2000).

Solid phase microextraction (SPME) fiber: Fused silica fiber with a polymeric coating that absorbs or adsorbs vapors, and desorbs them into a GC upon heating.

Threshold limit value short term limit (TLV): Recommended maximum concentration for 15-minute workplace exposure (USEPA 2000).

Time weighted average sampling mode (TWA): SPME sampling mode that keeps the SPME fiber inside its metal needle while collecting vapors, and is not time dependent.

ACKNOWLEDGMENTS

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