



Be alert for vapor intrusion of 1,4-dioxane from contaminated groundwater

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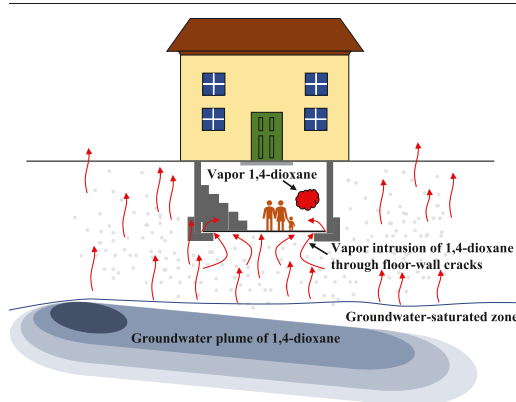
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HIGHLIGHTS

- Vapor intrusion of 1,4-dioxane from contaminated groundwater is a concern.
- A sensitive and rapid analytical method for airborne 1,4-dioxane is developed.
- The method is suitable for identifying 1,4-dioxane in high humidity environments.
- High levels of 1,4-dioxane vapor could invade basements in case of shallow plumes.
- 1,4-Dioxane vapor should be monitored in the coming flood season.

GRAPHICAL ABSTRACT



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ABSTRACT

Vapor intrusion (VI) poses significant environmental problems that can degrade indoor air and pose human health risks. This study focuses on 1,4-dioxane, a widely-used volatile organic compound (VOC) that is found in groundwater, however, this compound has not received much attention in indoor air and measurement methods are not well developed. 1,4-dioxane is sufficiently volatile and highly mobile in groundwater, and thus can present a VI risk. In this study, we develop a sensitive analytical method for quantifying airborne 1,4-dioxane, provide a performance evaluation of the method, and initiate preliminary field measurements above a 1,4-dioxane groundwater plume. The method uses passive sampling, automated thermal desorption, and gas chromatography/mass spectroscopy. Numerous other VOCs can be simultaneously measured. A low detection limit ($0.067 \mu\text{g}/\text{m}^3$) is attained, which allows quantification at concentrations below health-based guidelines. The performance evaluation suggests limits to sampling times in high humidity environments and other means to ensure good performance. The scenario analyses demonstrate potential impacts from shallow plumes, especially in flooded basements, and thus monitoring of 1,4-dioxane vapor intrusion in the flood season is an urgent need.

1. Introduction

The solvent and stabilizer 1,4-dioxane has been used widely, is water soluble and highly mobile in the environment, resistant to degradation,

relatively volatile, and toxic (Simonich et al., 2013; Sun et al., 2016; US EPA, 2006). Low level human exposure to 1,4-dioxane is widespread, mainly arising from the use of consumer products, such as cleaning agents and personal care products (Lin et al., 2020; Lin et al., 2021). Environmental exposures also occur, largely through surface and ground water that has been contaminated by industrial uses of 1,4-dioxane. The largest known area of contamination is a portion of the Huron watershed in Washtenaw County, Michigan, a result of industrial disposal practices from 1966 to

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1986 where waste solvent was dumped into unlined lagoons, which subsequently leaked into the groundwater aquifer that flows into the Huron River in Ann Arbor, Michigan. Both groundwater and surface water (from the Huron River) are used for drinking water for several hundred thousand people. Recent monitoring of 1,4-dioxane in groundwater in Scio Township and western Ann Arbor show concentrations up to 800 $\mu\text{g/L}$; higher levels had been seen earlier (EGLE, 2021a). Due to the large extent of the contamination, the area is being considered for Superfund listing. The U.S. Environmental Protection Agency (EPA) and several states (including Michigan) have established drinking water and groundwater guidelines for 1,4-dioxane that range between 0.25 and 77 $\mu\text{g/L}$; the large range is a result of differences in the risk assumptions used to formulate the guideline (Sun et al., 2016; US EPA, 2017). Notably, these levels consider only ingestion exposure, and they do not consider the potential from inhalation exposure, which can result from volatilization from consumer products, soil vapor intrusion (VI) into buildings from contaminated groundwater, and volatilization associated with domestic water use such as showering.

1,4-dioxane's toxicity includes adverse effects on the liver and kidney. For inhalational exposure, occupational studies, case reports and animal experiments have reported liver pathology and kidney lesions (Kasai et al., 2009; Kasai et al., 2008; Torkelson et al., 1974). The International Agency for Research on Cancer (IARC) has classified 1,4-dioxane as a possible carcinogen, and the United States Environmental Protection Agency (EPA) considers it "likely to be carcinogenic to humans" (IARC, 1999; US EPA, 2020) and lists a regional screening level (IRSL) of 0.56 $\mu\text{g}/\text{m}^3$ for the residential air screening level (although a 0.2 $\mu\text{g}/\text{m}^3$ level is implied for a 1×10^{-6} lifetime excess cancer risk from chronic inhalation exposure using the current unit risk estimate), and a drinking water limit of 0.46 $\mu\text{g/L}$ (US EPA, 2020). The State of Michigan follows the US EPA and lists an initial risk screening level (IRSL) of 0.2 $\mu\text{g}/\text{m}^3$ for 1×10^{-6} lifetime excess cancer risk (EGLE, 2021c). At equilibrium at 25 °C, 1 $\mu\text{g/L}$ in water corresponds to 0.196 $\mu\text{g}/\text{m}^3$ in air (using Henry's Law with constant = 4.80×10^{-6} atm- m^3/mol). Given levels found in groundwater, and especially considering houses with flooded basements, inhalation exposure to 1,4-dioxane from VI may represent a significant risk.

As an environmental pollutant, 1,4-dioxane has been most frequently measured in aqueous environments. Measurements in air are uncommon, although three methods have been published (Francis et al., 2009; Harkov et al., 1983; NIOSH, 1994). For occupational applications, the National Institute for Occupational Safety and Health (NIOSH) has developed a method using active sampling into a glass tube containing charcoal with subsequent analysis using gas chromatograph and flame ionization detection (GC-FID) (NIOSH, 1994). This method's detection limit, ~0.01 mg per sample, is too high for most environmental applications. The method presented by Harkov et al. (1983) which uses active sampling into a stainless steel tube containing Tenax-GC and Spherocharb and analysis by GC-FID, can be as low as 0.01 ppb (0.036 $\mu\text{g}/\text{m}^3$), however, the evaluation of this method is incomplete, e.g., recovery was not reported. Further, this and the NIOSH methods use active sampling (i.e., with sampling pumps), which are less convenient than passive sampling methods. Francis et al. (2009) used selected ion flow tube mass spectrometry, which allows real-time measurements with a reported detection limit of 530 ppt (1.91 $\mu\text{g}/\text{m}^3$). While real-time measurements can be very useful, the method's detection limit is too high for many environmental applications, and the technology requirements may preclude wide use. A simple, rapid, sensitive, and validated method for trace level airborne measurements of 1,4-dioxane is required.

In order to achieve the identification of vapor 1,4-dioxane, the goals of this study are to first develop a sensitive method of sampling and quantifying vapor-phase 1,4-dioxane over a wide concentration range, and then to evaluate its performance with a focus in high humidity environments that are common in basements and other environments relevant to VI applications, and finally to conduct a preliminary field measurement and evaluate application scenarios.

2. Materials and methods

2.1. Chemicals

1,4-Dioxane (99%, extra dry) was purchased from Acros Organics of Thermo Fisher Scientific (New Jersey, USA). Benzene, tetrachloroethene, fluorobenzene, and pentane (HPLC grade) were purchased from MilliporeSigma (Burlington, Massachusetts, USA). Methanol (HPLC grade) were purchased from Fisher Chemical (Pittsburgh, PA, USA).

2.2. Sampling

Samples were collected using passive sampling directly into one end of a thermal desorption tube which was subsequently used in analysis. The 10 cm long (0.4 cm of inner diameter) stainless sorbent tubes were packed with 160 mg 60/80 mesh Tenax-GR with a 1.0 cm or 0.5 cm diffusion gap. A glass wool plug retained the sorbent at both ends (Fig. S1). Before sampling, tubes were conditioned at 325 °C for 6 h with a 30 mL/min flow of high purity N_2 , sealed with a stainless-steel cap, wrapped in baked aluminum foil, placed in a clean glass jar with an activated carbon pack, and refrigerated. During sampling, the cap was removed and the tube was placed in a stand allowing free air circulation. The sampling uptake rate was calculated using a diffusion model that accounted for the tube configuration, temperature, and the diffusion coefficient. Analyte masses (ng) were converted to concentrations ($\mu\text{g}/\text{m}^3$) by dividing by the calculated sampling volume (m^3 , determined as the diffusion coefficient of the chemical in air \times porosity of diffusion medium (air or glass wool) \times tortuosity of diffusion medium (air or glass wool) \times diffusion area \times sampling time \div diffusion distance, Table S1) (Batterman et al., 2012; Jia et al., 2012). After sampling, the cap was put back on, and the tube stored as described earlier.

2.3. Analysis

In the laboratory, 1,4-dioxane was analyzed using an automated thermal desorption (ATD) system (Model 2000, Scientific Instrument Services, Ringoes, NJ, USA), and a gas chromatograph/mass spectrometer (GC/MS, Model 6890/5973, Agilent Technologies, Santa Clara, CA, USA). We incorporated the measurement of 1,4-dioxane into an existing method for 100 VOCs, which is detailed elsewhere (Zhong et al., 2019). The GC used a capillary column (DB-VRX, 60 m \times 0.25 mm, 1.40 μm film thickness, Agilent Technologies, Santa Clara, CA, USA) and ChemStation software (G1701BA, Agilent Technologies). The cryotrap cooling, heating and desorption temperatures were -140, 250 and 200 °C, respectively, and the injector, detector, MS quadrupole, and MS source temperatures were 250, 250, 150, and 230 °C, respectively. The GC oven temperature was initially 45 °C for 10 min, increased at 8 °C/min to 140 °C, held for 10 min, then increased at 30 °C/min to 225 °C, and held for 13 min. The MS was operated in scan mode from 29 to 270 AMU (atomic mass unit). Fluorobenzene was used as internal standard (IS) and 2 ng of IS was injected into each tube before thermal desorption.

2.4. Calibration standards and quality control samples

A stock standard solution (SSS) (2 mg/mL) was prepared by dissolving neat 1,4-dioxane in methanol and storing at 4 °C. A primary dilution standard was prepared at 200 $\mu\text{g}/\text{mL}$ by diluting SSS in methanol, from which duplicate calibration standards (0.5–50 $\mu\text{g}/\text{mL}$) in pentane were prepared to build 5-point calibration curves. To confirm calibrations and method stability, daily quality control (QC) checks were performed by injecting 2 μL of a 5 $\mu\text{g}/\text{mL}$ standard (in pentane) prior to the analysis of samples and blanks, using an acceptance criterion of 30%.

2.5. Performance tests

Test atmospheres of 1,4-dioxane at the desired humidity and room temperature used a dynamic dilution setup with an exposure chamber

(0.31 L glass chamber with Teflon caps, concentration of 10 ng/L, and flow rate of 1 L/min, 10 ng/L·min) (Fig. 1). In a sequence of experiments, the relative humidity (RH) was set to 25, 50 and 75%, as confirmed by measurements in the chamber (using a HOBO temperature relative humidity data logger, Onset Computer Corp, USA). Before inserting VOC tubes for sampling, the system was stabilized by running for 1–2 h. Sampling tubes with 0.5 and 1.0 cm diffusion gaps were then deployed in the chamber for periods of 12, 24, and 36 h. Each sampling period and gap length used duplicate tubes. Recoveries were estimated as the ratio of the measured concentration to 10 ng/L (the theoretical concentration in the chamber). Thus, these recoveries include possible losses in the exposure system, sample collection, sample desorption, and instrument analysis.

The method detection limit (MDL) was determined as the standard deviation of 7 replicate low concentration injections (0.4 ng) into the sampling tube, multiplied by 3.14 (Student's t-value at the 99% confidence level). Additional tests were conducted for method comparison and assessment. Several of these used injections of benzene and tetrachloroethene (TCE) into the chamber at a concentration of 10 ng/L (along with 1,4-dioxane), which were collected and analyzed using the same sampling tubes. The sampling and analysis method have been well validated for benzene and TCE. Recoveries using active and passive sampling were compared using tests at 50% RH, a flow rate of 50 mL/min (for active sampling), and sampling period of 20 min for active sampling (12, 24 and 36 h set for passive sampling) (Fig. 1). Active sampling also was used to investigate potential losses in the chamber by comparing up- and downstream measurements.

2.6. Scenario analysis

The houses targeted for sampling were in the impacted area, and included residential sites south of M-14, north of West Liberty Street, east of South Zeeb Road, and west of the Huron River, all which refer to the 1,4-dioxane plume map in Washtenaw County, Michigan (EGLE, 2021b). Using the local conditions, we estimated concentrations in basements for two scenarios: VI of 1,4-dioxane from an aquifer 5 m below ground surface (bgs), and for a flooded basement. In both scenarios, sampling sites were in basements, at least 0.5 m from floor, ceiling, walls, furniture, and other surfaces, and in the opposite corner from water or space heaters. These locations were expected to provide representative levels, given that typical VI entry routes for houses in the study region include cracks and openings in the basement floor (especially around the foundation joint along the building perimeter), floor drains, and utility service entries (e.g., for gas, water, waste).

1,4-Dioxane levels were estimated using attenuation coefficients, Henry's law, and the ideal gas law as below (Yao et al., 2013). We assumed that vapor from groundwater followed Henry's law and the ideal gas Law,

and passed through the soil to surface house with attenuation. The vapor concentration C_v (g/m^3) is calculated using Henry's law:

$$C_v = \frac{C_w \times M \times H}{R \times T} \quad (1)$$

where C_w is the concentration in groundwater (mol/m^3), M is 1,4-dioxane's molecular weight (88.11 g/mol), R is the ideal gas constant ($0.0000821 \text{ atm}\cdot\text{m}^3/\text{mol}\cdot\text{K}$), T is the temperature (298.15 K), and H is the Henry's constant of 1,4-dioxane ($4.80 \times 10^{-6} \text{ atm}\cdot\text{m}^3/\text{mol}$).

The air concentration in the house, C_H , due to vapor intrusion, was calculated using an attenuation coefficient μ (dimensionless) that accounts for diffusion through the soil, sorption and other processes in the soil, barriers that limit entry of soil gas into the house, advection, and air change rates in the house that dilute the incoming soil gas:

$$C_H = C_v \times \mu \quad (2)$$

3. Results and discussion

3.1. Chromatogram and mass spectrum

The ion chromatogram of 1,4-dioxane (Fig. S2) had a retention time of 18.54 min. From the mass spectrum, we selected ions with m/z of 88 for the quantitation (Q-ion) and m/z of 28, 58 and 43 as qualifier ions, which had nominal ratios to the Q-ion of 1.00, 0.55 and 0.18, respectively. These four ion peaks were symmetrical, separated from background noise, and distant from other compounds found in the samples. Fluorobenzene, used as the IS, had a similar retention time (17.31 min; Fig. S2), and we used m/z of 96 as the Q-ion and m/z of 50 and 70 as qualifier ions (ratios to the Q-ion of 0.07 and 0.17, respectively). The program run time was ~60 min, which included ~7.00 min for thermal desorption and 47.7 min for the GC/MS program (which incorporated analyses of 100 VOCs in addition to 1,4-dioxane).

3.2. Calibration and detection limit

The 1,4-dioxane calibration curve (Fig. S3) was linear over three orders of magnitude ($R^2 = 0.998$). The MDL was 0.10 ng, which corresponds to a 1,4-dioxane concentration of $0.067 \mu\text{g}/\text{m}^3$ for a sampling volume of 1.5 L of air (which can be obtained by either active sampling or passive sampling using a diffusion gap of 1.0 cm and sampling time of 54 h at 25 °C). This value is well below the 0.20–0.56 $\mu\text{g}/\text{m}^3$ range for current cancer screening levels for chronic inhalation exposure (EGLE, 2021c; US EPA, 2020).

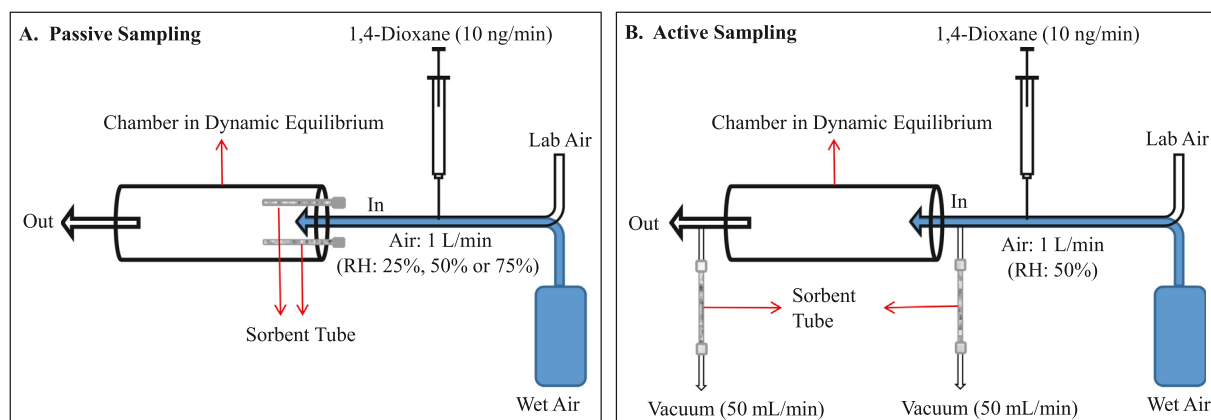


Fig. 1. Passive (left) and active (right) recovery test system for 1,4-dioxane at different RHs.

3.3. Effect of humidity

The recovery of 1,4-dioxane across the tested experimental conditions (various diffusion gaps, sampling duration, and relative humidity) averaged $51.6 \pm 10.6\%$ (\pm standard deviation). We used water vapor's diffusion coefficient ($0.256 \text{ cm}^2/\text{s}$ at 25°C) to calculate the mass of water vapor absorbed by the adsorbent for each condition, assuming complete uptake of water vapor (the worst case), disregarding the relatively hydrophobic properties of the Tenax adsorbent. The calculated adsorbed water mass and measured 1,4-dioxane recoveries showed a weak negative association (Fig. 2). Excessive water uptake can interfere with system performance. Each 10 mg of water (worst case estimate representing 6.2% of the adsorbent weight) collected by the adsorbent yielded a 5.8% ($p = 0.071$, 95% CI: $-12.3\% \sim 0.6\%$) decrease of 1,4-dioxane recovery. For example, considering a 9.8-hour exposure at 75% RH (1.0 cm diffusion gap), the adsorbent would collect 10 mg of water, and induce a 5.8% decrease in recovery.

Effects of water vapor on the recovery of benzene were negligible ($p = 0.87$; Fig. 2). TCE results were similar to those of 1,4-dioxane, e.g., recovery was reduced by 8.8% with 10 mg of water vapor absorbed ($p = 0.12$). Water absorbed on the adsorbent can affect sampling, e.g., inhibit uptake due to competition between adsorbates and the presence of a water film, as well as degrade the analysis, e.g., skew peak shapes and detection. The three compounds tested varied considerably in hydrophilicity, e.g., $\log K_{ow}$ values for 1,4-dioxane, benzene and TCE are -0.27 , 2.13 and 3.40 , respectively (US EPA, 2017). While the recovery of more hydrophilic compounds like 1,4-dioxane was expected to be more sensitive to the water loading, we saw mostly minor effects and small differences between the three compounds for the experimental conditions. While humidity had small effects for the tested conditions, we still suggest limiting the sample volume and water uptake in high humidity environments by either shortening the sampling time or using an appropriately sized air gaps while accounting for the detection limits of 1,4-dioxane. Field measurements might be confirmed using distributed volume sampling, e.g., sampling using two passive tubes that have different air gaps and thus different sampling rates.

In active sampling, recoveries averaged 75.8, 90.9, and 90.0% for 1,4-dioxane, benzene, and TCE, respectively (Fig. S4), showing higher losses of 1,4-dioxane than the two other VOCs. For the NIOSH method using

active sampling, a bias of 9% and accuracy of ± 18.7 was noted; recovery data were not presented (NIOSH, 1994). The other two methods mentioned previously did not evaluate method recovery (Francis et al., 2009; Harkov et al., 1983). Active sampling was conducted at 50% RH with 1 L volume sampling. Assuming all water vapor in the sample was absorbed, the adsorbent would have picked up 8.6 mg water, which is similar to the passive sampling condition using the 12 h period, 50% RH and a 1.0 cm diffusion gap (8.2 mg water absorbed) where recoveries were 62, 63 and 96% for 1,4-dioxane, benzene and TCE, respectively. Thus, active sampling improved recoveries of 1,4-dioxane and benzene, but TCE performance was essentially unchanged. Active sampling also verified that VOC levels entering and exiting the sampling chamber were statistically unchanged (Fig. S4), thus demonstrating neglectable losses in the chamber.

3.4. Test scenarios

The former industrial disposal site in Ann Arbor, Michigan is now surrounded by forests and parking lots. We conducted field measurements in several houses approximately 1.5 to 3 km distant during non-flooding season. We did not detect 1,4-dioxane in basement air. The nearest groundwater monitoring well showed that 1,4-dioxane concentration in groundwater near these houses was around 200 ppb. As noted earlier, recent measurements of 1,4-dioxane levels in residential wells Michigan ranged to 800 ppb (EGLE, 2021a). Assuming a concentration of 800 ppb at the capillary fringe (top of the surface water table), then the equilibrium soil gas concentration would be $157 \mu\text{g}/\text{m}^3$. The U.S. Geological Survey maps show that the affected area has a shallow water table, typically about 5 m bgs; basement flooding also has been reported. Groundwater temperatures average 13.3°C in summer (July and August). Basements are common in the area, and reach about 2 m bgs, thus, soil gas has to travel 3 m from the vapor source to enter a residence.

Indoor levels and the potential for human exposure from VI contaminants depend on many factors, including the level and distribution of the contaminant in groundwater (e.g., concentration), subsurface characteristics that affect diffusion, advection, sorption and degradation (e.g., water content, porosity, temperature, barometric pressure), building factors that affect soil gas entry and distribution (e.g., footprint, floor penetrations, volume, air change rates, internal air flows, pressurization), and human

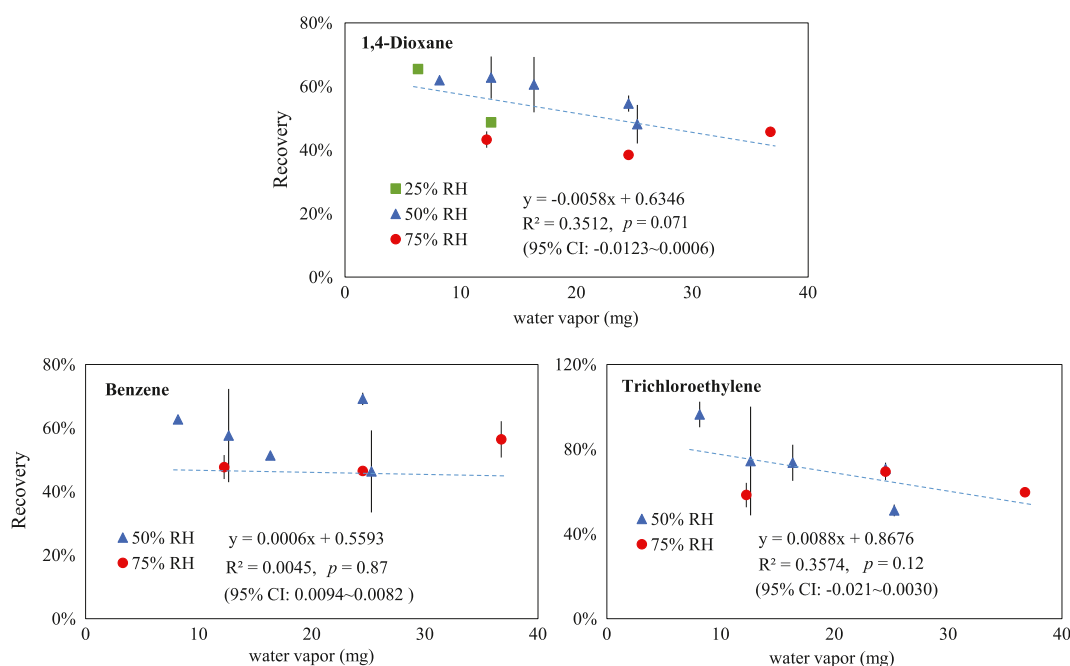


Fig. 2. Scatter plots showing recovery of 1,4-dioxane, benzene and tetrachloroethene (TCE) versus calculated mass of water vapor absorbed by the adsorbent in the sampling tube.

factors that determine exposure (e.g., breathing rates, time spent in places). The subsurface and building factors affecting VI are often summarized using attenuation coefficients that relate soil gas levels (at the water table) to indoor air levels; this coefficient is at least 0.001 for 3 m deep source (Abreu and Johnson, 2005). With this value, the estimated 1,4-dioxane concentration in the basement is $0.16 \mu\text{g}/\text{m}^3$. Actual concentrations will depend on site-specific conditions, e.g., lower temperatures, greater depth to groundwater, and lower groundwater concentrations would lower indoor concentrations, but spatial and temporal heterogeneity might increase levels at some houses. While this VI scenario approaches the Michigan IRSL of $0.2 \mu\text{g}/\text{m}^3$, which represents a 10^{-6} lifetime cancer risk due to inhalation exposure of 1,4-dioxane from VI, the results do not suggest a severe threat.

The scenario for a flooded basement gives very different results. During the flood season (typically May through August), basements in low-lying areas may flood, allowing 1,4-dioxane to volatilize directly into the house. Assuming a groundwater concentration of $222 \mu\text{g}/\text{L}$, the mean level reported in this residential area for March 2021 (EGLE, 2021a), basement flooding would lead to $44 \mu\text{g}/\text{m}^3$ of 1,4-dioxane in basement air. This considerably exceeds the Michigan IRSL ($0.2 \mu\text{g}/\text{m}^3$) (EGLE, 2021c), the U.S. EPA screening level for chronic inhalation exposure ($0.56 \mu\text{g}/\text{m}^3$) (US EPA, 2020), and the EPA screening levels for non-cancer effects ($30 \mu\text{g}/\text{m}^3$) (US EPA, 2020), but the much higher ATSDR minimum risk levels for no adverse effects for 1 to 14-day (acute) exposures ($7200 \mu\text{g}/\text{m}^3$) (ATSDR, 2012) is not exceeded. (This groundwater concentration considerably exceeds the current U.S. EPA screening level for domestic water $0.46 \mu\text{g}/\text{L}$ (US EPA, 2020)). These results are based on equilibrium partitioning and other simplifying assumptions, but given the low air change rates in basements (Du et al., 2015) and duration of flooding (often multiple days), monitoring of indoor air for 1,4-dioxane during flooding events in this area is strongly recommended.

4. Conclusions

The sensitive and rapid analytical method for quantifying airborne 1,4-dioxane developed in this study is applicable to general environmental applications, and the performance evaluation indicates the method's suitability for high humidity environments where this solvent is likely present. As noted earlier, contamination of groundwater by 1,4-dioxane is a widespread, significant and long-lasting environmental problem in southeast Michigan, and possibly in other areas where this solvent was inappropriately discharged. For deeper subsurface plumes, indoor concentrations of 1,4-dioxane due to VI are likely to be below chronic and acute health-based guidelines. However, in cases of shallow plumes where the soil attenuation factor is large, basement flooding, and potentially off-gassing from the use of contaminated water, the potential to produce high levels of 1,4-dioxane vapor exists, and monitoring is needed or desirable to quantify levels, exposures and health risks. We also noted 1,4-dioxane's common use in personal care products, which may increase the potential for inhalation exposure.

CRediT authorship contribution statement

Nan Lin: Conceptualization, Methodology, Software, Validation, Formal Analysis, Investigation, Data Curation, Writing - Original Draft, Writing - Review & Editing, Visualization.

Lexuan Zhong: Methodology, Investigation, Data Curation.

Christopher Godwin: Methodology, Investigation.

Stuart Batterman: Conceptualization, Methodology, Validation, Formal Analysis, Resources, Writing - Original Draft, Writing - Review & Editing, Supervision, Project Administration, Funding Acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2022.153713>.

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