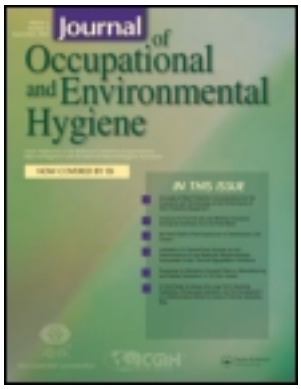

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B. S. Marpoe [a], W. A. Groves [a], E. G. Lee [b], J. E. Slaven [c] & M. Harper [b]

[a] The John and Willie Leone Family Department of Energy and Mineral Engineering, Penn State University, University Park, Pennsylvania

[b] Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Morgantown, West Virginia

[c] Department of Biostatistics, Indiana University School of Medicine, Indianapolis, Indiana

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Effects of Covered Solid Sorbent Tube Sample Holders on Organic Vapor Measurements

B.S. Marpoe,[1] W.A. Groves,[1] E.G. Lee,[2] J.E. Slaven,[3] and M. Harper[2]

[1]The John and Willie Leone Family Department of Energy and Mineral Engineering, Penn State University, University Park, Pennsylvania
[2]Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Morgantown, West Virginia
[3]Department of Biostatistics, Indiana University School of Medicine, Indianapolis, Indiana

A study was conducted to examine whether there are significant differences between organic vapor concentrations measured using charcoal tubes with three different configurations: uncovered sample holder (open tube), SKC, and Buck brand covered sample holders. A fractional factorial experimental design was used with the following factors and levels: vapor (n-hexane vs. m-xylene), pump type (pulsating vs. continuous), exposure profile (variable vs. constant), flow rate (30 mL/min vs. 200 mL/min), duration (30 min vs. 80 min), and sample placement (mannequin vs. free hanging). Two of each sampler configuration (six total) were placed in an exposure chamber, and a dynamic test-atmosphere generation system was used to prepare atmospheres containing approximately 12–15 ppm n-hexane or m-xylene with exposure profiles and sampling conducted according to a run sheet generated for the experimental design. A total of 24 runs were completed with six samplers per run, yielding 144 samples that were analyzed by gas chromatography/flame ionization detector. Concentration results for each pair of SKC and Buck covered sample holders were averaged and normalized by dividing by the average result for the open tube sampler from the same run to eliminate the effect of daily variation in chamber concentrations. The resulting ratio of covered sample tube holder and open tube concentrations was used as the response variable. Results of analysis of variance using the general linear model (MINITAB 16) identified statistically significant main effects and/or interactions for pump type, exposure profile, flow rate, and sample holder. However, the magnitude of the effects was generally less than 10%, and overall mean concentration ratios were 0.989 and 1.02 for the Buck and SKC sample holders, respectively. These results show good agreement between covered sample holder results and open tube measurements and demonstrate that exposure assessment errors resulting from the use of covered sorbent tube sample holders for organic vapor monitoring are relatively small (<10%) and not likely to be of practical importance.

Keywords air sampling, sorbent tube sample holder, sorbent tube sampling, vapor sampling

Correspondence to: W.A. Groves, 223 Hosler Building, Penn State University, University Park, PA 16802-5000; e-mail: wag10@psu.edu.

INTRODUCTION

Industrial hygienists have relied extensively on the use of solid sorbent tubes for monitoring organic vapors since the initial application of gas chromatography to airborne contaminant monitoring[(1)] and subsequent development of analytical methods based on charcoal tubes with carbon disulfide desorption.[(2–5)] It has been noted that solid sorbent-based collection methods have been the most common approach to volatile organic monitoring since the mid-1970s[(6)] with potential application to hundreds of analytes. Collection typically employs a sampling train consisting of a personal sampling pump connected by Tygon® tubing to the solid sorbent tube, which may or may not be enclosed in some type of tube holder assembly. Tube holders differ in design and function. Some contain a needle valve at the base that adjusts flow rate when used with a sampling pump in constant pressure mode, while other holders simply provide a fitting and tube holder sleeve to facilitate connection of tubing to the sorbent tube (Figure 1a). Holders also typically include some type of protective cover that encloses the potentially sharp open inlet of the glass sorbent tube, and a clip for attaching the sampler assembly to the worker.

While good practice generally dictates minimizing contact between sampled air and any materials/surfaces upstream from the collection medium, the use of a sample holder with protective cover would violate this premise as the contaminant must first pass through an opening in the cover before entering the sorbent tube. Presumably, any resulting bias is expected to be negligible as there does not appear to have been a description or study of this topic in the literature, manufacturers' product information, or published analytical methods. Present interest grew out of a separate project that focused on the development of a physiological sampling pump (PSP) and the associated customized sorbent tube holder.[(7–9)] Preliminary laboratory testing of this pump suggested the possibility of sample holder effects when measured concentrations were compared with

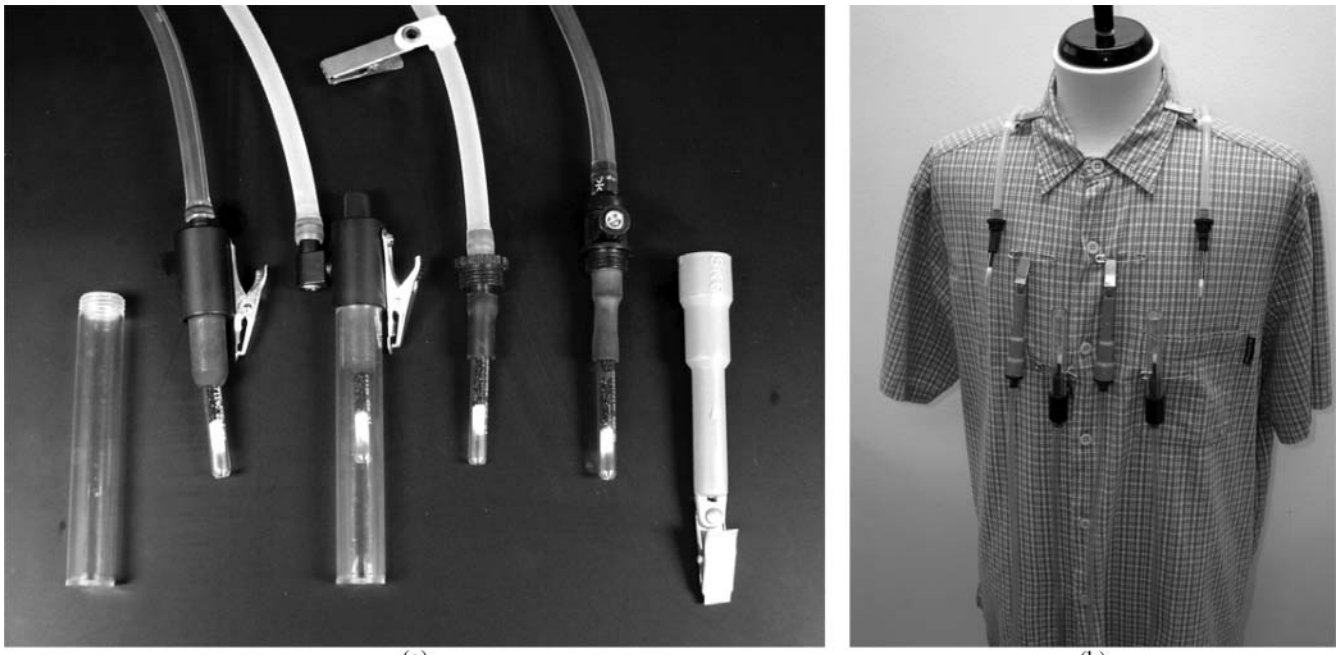


FIGURE 1. Photos showing (a) various sorbent tube configurations from left to right: non-adjustable Buck holder with cover removed, adjustable covered Buck holder, open tube (uncovered) with non-adjustable SKC holder, and adjustable SKC holder with cover removed; (b) two each of covered Buck and SKC holders, and open tube (uncovered) sample holders on mannequin.

those from an open sorbent tube (uncovered holder assembly). Therefore, further exploration of the possible effects of sample holders was undertaken.

The specific aims of this study were to examine whether there are substantial differences (>10%) between concentrations measured using activated carbon sorbent tubes with three different sample holder configurations: (1) uncovered sample holder, (2) covered SKC brand sample holder, and (3) covered Buck brand sample holders, and to characterize the nature of any significant effects by examining different vapors (n-hexane vs. m-xylene), types of sampling pumps (pulsating stroke-counter type vs. "continuous" type pumps), exposure profiles (variable vs. constant), sampling flow rates (200 mL/min vs. 30 mL/min), sample placement (mannequin vs. hanging freely), and sample durations (80 min vs. 30 min). Differences greater than 10% were considered substantial based on the generally accepted levels of the total coefficient of variability (CV_T) for sampling and analytical methods, which is on the order of 10% for most organic vapors.[10] The selection of SKC and Buck sample holders for inclusion in the study was arbitrary. The SKC holder had been used in the previous project and so it was included, while the Buck sample holder was identified and selected after a search of vendors for readily available sorbent tube holders.

METHODS

A fractional factorial experimental design was used in this study.[11] This allowed for testing between several factors while keeping the number of runs minimized and while maintaining the appropriate power level to detect significant differences. The seven factors examined and the respective

TABLE I. Factors and Levels Examined in Study

Factor	Levels	
Vapor	Low bp (n-hexane)	High bp (m-xylene)
Pump type	Pulsating	Continuous
Exposure profile	Variable	Constant
Sampling flow rate	Low (30 mL/min)	High (200 mL/min)
Sample placement	Mannequin	Free hanging
Duration	Short (30 min)	Long (80 min)
Sample holder	Buck	SKC Open tube (uncovered)

TABLE II. Study Design Matrix

Run	Vapor	Pump Type	Exposure	Flow Rate	Mannequin	Duration
1	n-Hexane	Pulsating	Variable	30 mL/min	Yes	80 min
2	m-Xylene	Pulsating	Variable	30 mL/min	No	80 min
3	n-Hexane	Continuous	Variable	30 mL/min	No	80 min
4	m-Xylene	Continuous	Variable	30 mL/min	Yes	80 min
5	n-Hexane	Pulsating	Constant	30 mL/min	No	30 min
6	n-Hexane	Pulsating	Constant	30 mL/min	No	80 min
7	m-Xylene	Pulsating	Constant	30 mL/min	Yes	30 min
8	m-Xylene	Pulsating	Constant	30 mL/min	Yes	80 min
9	n-Hexane	Continuous	Constant	30 mL/min	Yes	30 min
10	n-Hexane	Continuous	Constant	30 mL/min	Yes	80 min
11	m-Xylene	Continuous	Constant	30 mL/min	No	30 min
12	m-Xylene	Continuous	Constant	30 mL/min	No	80 min
13	n-Hexane	Pulsating	Variable	200 mL/min	No	80 min
14	m-Xylene	Pulsating	Variable	200 mL/min	Yes	80 min
15	n-Hexane	Continuous	Variable	200 mL/min	Yes	80 min
16	m-Xylene	Continuous	Variable	200 mL/min	No	80 min
17	n-Hexane	Pulsating	Constant	200 mL/min	Yes	30 min
18	n-Hexane	Pulsating	Constant	200 mL/min	Yes	80 min
19	m-Xylene	Pulsating	Constant	200 mL/min	No	30 min
20	m-Xylene	Pulsating	Constant	200 mL/min	No	80 min
21	n-Hexane	Continuous	Constant	200 mL/min	No	30 min
22	n-Hexane	Continuous	Constant	200 mL/min	No	80 min
23	m-Xylene	Continuous	Constant	200 mL/min	Yes	30 min
24	m-Xylene	Continuous	Constant	200 mL/min	Yes	80 min

levels are summarized in Table I. Factors and levels were selected as those most likely to be relevant to both routine volatile organic monitoring and potential mechanisms for sample holder effects on measured concentrations. The resulting study design matrix required 24 experimental runs (Table II). The effect of the type of sample holder was evaluated by including two duplicate sampling units for each type of sample holder for every experimental run (Figure 1). This required six sampling trains, two each of the three sample holder arrangements: (1) open (uncovered) sorbent tube, (2) covered SKC sample holder (SKC Inc., Eighty Four, Pa.), and (3) covered Buck sample holder (A.P. Buck Inc., Orlando, Fla.). The six samplers were suspended with the inlets at the same level, spaced evenly across a span of 23 cm at a height of approximately 122 cm, with the same spacing used for both the free hanging and mannequin placements (Figure 1b). The free hanging sample assembly consisted of a T-shaped section of fabricated tubing designed to fit into the base of the mannequin stand after removal of the torso. The design of the assembly provided a horizontal section of tubing with the same height and spacing as the mannequin for the six samplers that were then suspended and allowed to hang freely. Sample holders were randomly assigned to six possible positions for each experimental run.

A test-atmosphere generation system and exposure chamber similar to that used for the evaluation of the PSP[9] was used for these experiments (Figure 2). Vapor was generated using a gas washing bottle containing the solvent of interest (either n-hexane or m-xylene) in a water bath maintained at constant temperature. A metered stream of air was passed through the bubbler and introduced into a dilution airstream to achieve the desired concentration in the exposure chamber. For runs that specified a variable exposure profile, vapor generation was turned on and off at 10-min intervals to produce a concentration profile varying from approximately 0–30 ppm as shown in Figure 3 (top). The dynamic test-atmosphere generation system parameters were adjusted so that average concentrations for both vapors and each exposure profile were similar at approximately 15 ppm. This concentration is somewhat arbitrary and was selected to be similar to the concentrations generated in the previous study in which the possible sample holder effects were first observed. These concentrations correspond to approximately 0.15× and 0.30× the threshold limit values (TLVs®) of m-xylene and n-hexane, respectively.

Selected chemical and physical properties for the two vapors examined are presented in Table III. Inclusion of m-xylene was based on the previous PSP study, and n-hexane was selected as an additional vapor having different chemical and physical properties. Table III shows the more volatile nature of n-hexane as demonstrated by the lower boiling point (bp) and higher vapor pressure (vp) compared with m-xylene. It was expected that if sample holders did significantly affect

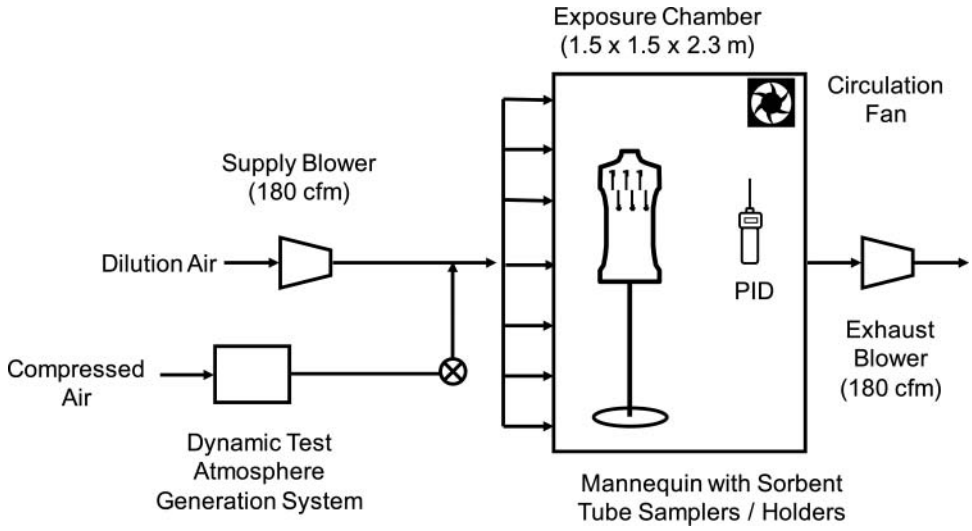


FIGURE 2. Schematic of test atmosphere generation system and exposure chamber

measured concentrations, vapor uptake and release through some sorption mechanism would likely play a role, and therefore, bp and vp would be relevant parameters.

All samples were collected on a standard (6 × 70 mm, 100/50 mg) coconut charcoal tube (Cat. No. 226-01, SKC). The pulsating pumps used were the SKC 222 Series (Cat. No. 222-3), while the continuous flow pumps were the SKC Airlite model (Cat. No. 110-100) equipped with the low flow adapter kit (Cat. No. 110–500). The low flow mode of the Airlite pump requires the use of an adjustable sample holder equipped with a needle valve for setting the flow rate. Therefore, the adjustable Buck holder (Cat. No. APB-109030) and the SKC

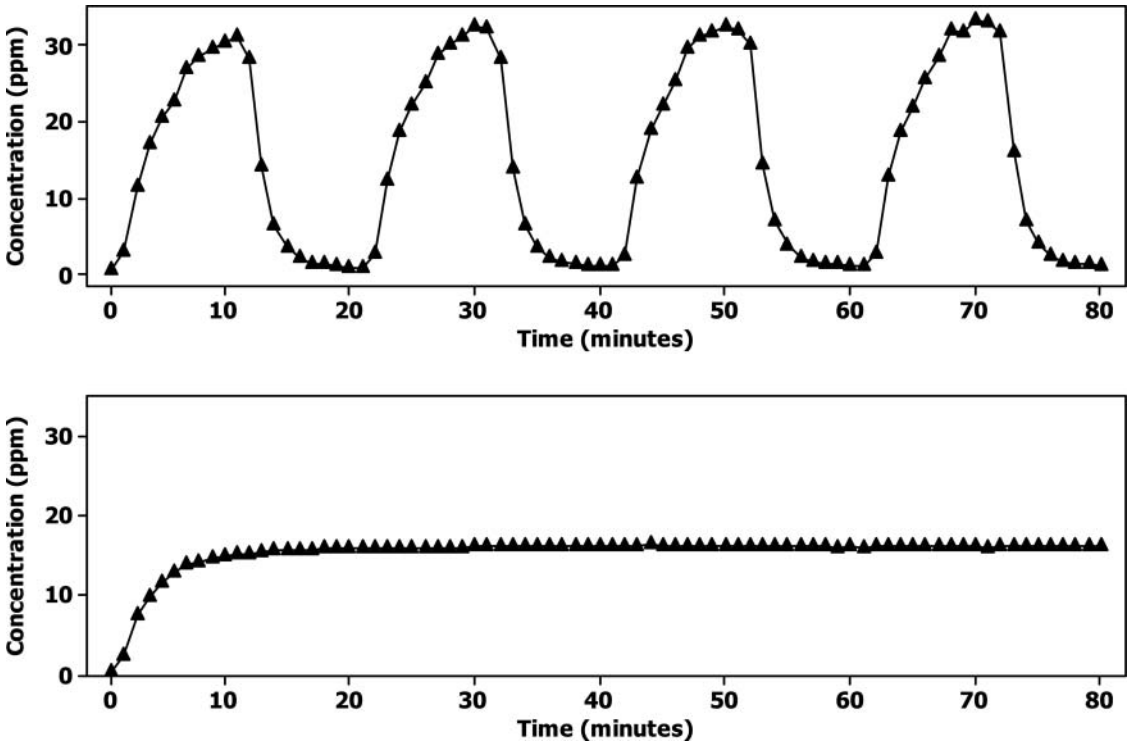
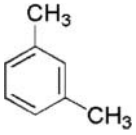
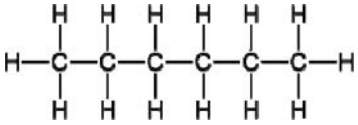


FIGURE 3. Time series plots showing typical variable (top) and constant (bottom) concentration exposure profiles as measured by calibrated PID for m-xylene

TABLE III. Chemical and Physical Properties of Vapors Examined in Study

Property	m-Xylene	n-Hexane
CAS#	108-38-3	110-54-3
ACGIH TLV	100 ppm	50 ppm
MW	106.2	86.2
BP, °C	139	69
VP @ 20°C	6.0 mm Hg	128 mm Hg
Chemical Class	methylated aromatic	straight chain alkane
Structure		
PID Correction Factor 10.6 eV	0.43	4.3

(Cat. No. 224-26-01) holder were used with the Airlite pump for continuous flow runs, while the non-adjustable Buck (Cat. No. APB-109032) and SKC (Cat. No. 222-3-1) holders were used with the SKC 222 Series pump for the pulsating flow runs. SKC holders were used for all open tube (uncovered) samples for reasons elaborated in the following section.

It was originally planned that the open tube (uncovered) sample would be collected by simply inserting the sorbent tube directly into tubing connected to the pump. However, since the type of continuous flow pump employed required the use of a needle valve to adjust flow, the open tube sample had to include this downstream element in the sampling train as well. Therefore, the decision was made to use the SKC holders with the covers removed for all open tube samples. Selection of the SKC holders was based solely on the availability of an adequate number of holders for both covered SKC and open tube samples at the beginning of the study. A consequence of this approach is that the experimental comparisons being made across the samplers is focused specifically on the portion of the sample train that is upstream from the sorbent tube, i.e., the sample holder cover assembly and inlet. Given that downstream factors potentially affecting sampler performance would be limited to leaks and/or related pump calibration errors, and since as previously stated the most likely source of differences between covered and uncovered sample results would be related to an upstream sorptive mechanism, this approach was not expected to hinder the goals of the experiment.

Pumps and sampling trains were pre- and post-calibrated using primary standards, and samples were labeled, capped, and stored in a freezer until analyzed on site by GC/FID according to NIOSH Methods 1500 (Hydrocarbons, BP 36°-216°C) and 1501 (Hydrocarbons, Aromatic). The 24 experimental runs with six samplers per run yielded a total of 144 sorbent tube samples for analysis. Calibration standards and spiked QC samples were analyzed with each set of experimental samples. A calibrated PID (MiniRAE 2000; RAE Systems, San Jose Calif.) was placed in the exposure chamber for each run, and concentrations were logged and subsequently downloaded and stored so that the time concentration profile (Figure 3) could be examined and compared across runs. Exposure chamber temperature and humidity were recorded (but not controlled) for all runs using a digital hygrometer (Cat. No. 35519-047; VWR International, LLC, Radnor, Pa.).

No human subjects were involved in the experiments. All samples were placed on a stand or mannequin within the chamber for the duration of the experimental runs, and at the completion of runs, flow through the vapor generation system was turned off and the chamber was flushed with clean air prior to re-entry.

RESULTS AND DISCUSSION

Results for measured air concentrations categorized by sample holder and vapor are summarized in Table IV. Concentrations ranged from approximately 9–17 ppm with coefficients of variability (CV) of approximately 12% overall. The within-run variability across the six samplers ranged from 0.74–10%, with an average of 3.6%. The relative magnitude of the concentrations measured by the different samplers was consistent across vapors and for the overall results as seen

TABLE IV. Concentrations (ppm) by Sampler and Vapor

Variable	N	Mean	Std. Dev.	CV%	Min	Max
Sampler						
Buck	48	13.8	1.7	12.2	8.95	17.0
Open tube	48	13.4	1.6	11.9	9.43	16.6
SKC	48	13.3	1.5	11.5	9.24	16.3
Vapor						
n-hexane	72	12.9	1.8	14.1	8.95	16.8
m-xylene	72	14.1	1.1	7.5	12.4	17.0

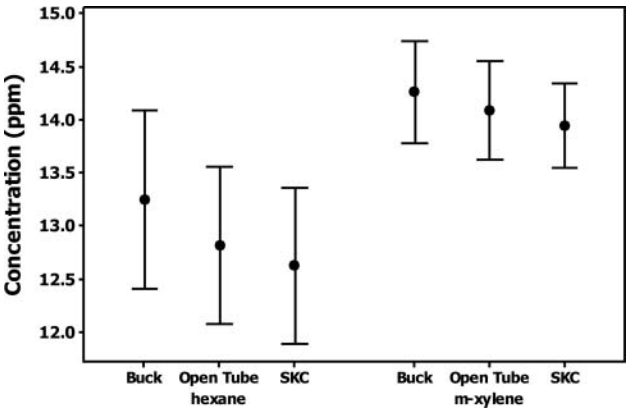


FIGURE 4. Interval plot of average measured concentration with 95% CI by sample holder and vapor

in Table IV and Figure 4. The Buck sample holder had the highest results, the SKC sampler the lowest, with the open tube results in between. Although it was intended that the average chamber concentrations would be relatively constant from day to day and similar for the two vapors, summary statistics and the interval plots indicate lower and more variable concentrations for n-hexane compared with m-xylene. These results reflect differences in the characteristics of the test atmosphere generation system for the two vapors rather than sample holder effects on measured concentrations. To control for this variability, the concentrations were normalized by dividing the average result for each of the covered sample holders by the average result for the open tube samples for each experimental run. The resulting normalized concentration ratios (e.g., SKC result/open tube result) were then used as the response variable for additional statistical analyses.

Summary statistics for the normalized concentrations are presented in Table V, and the results show a significant reduction in the variability of the response. CVs ranged from approximately 3–7%, which represents a reduction of approximately one-half compared with the variability of the original concentrations. Normalized concentrations ranged from approximately 0.94–1.2 with mean values very close to a value of one, which would represent perfect agreement with the reference open tube sample result. The relative magnitude of the sample holders was consistent with the previous results for concentrations: the Buck sample holder yielded the highest average results (1.02) and the SKC holder had the lowest (0.99).

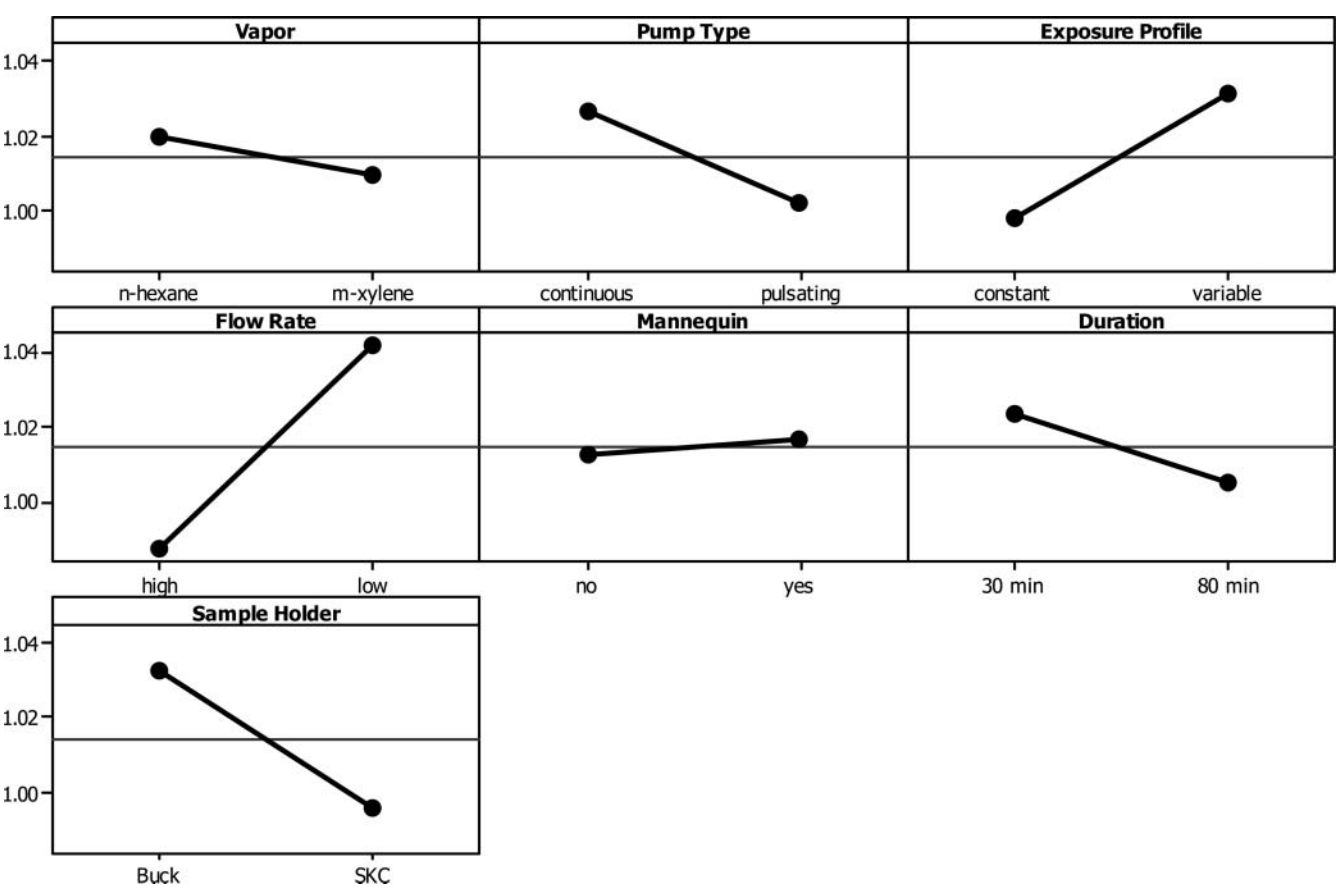


FIGURE 5. Main effects plot for normalized concentration. Exposure Profile ($p = 0.014$), Flow Rate ($p < 0.001$), and Sample Holder ($p = 0.009$) were identified as significant.

TABLE V. Normalized Concentrations by Sampler and Vapor

Variable	N	Mean	Std. Dev.	CV%	Min	Max
Sampler						
Buck	24	1.02	0.07	7.1	0.944	1.20
SKC	24	0.988	0.031	3.1	0.939	1.08
Vapor						
n-hexane	24	1.01	0.07	6.6	0.944	1.20
m-xylene	24	1.00	0.05	4.8	0.939	1.16

Note: Average covered sample holder concentration divided by average open-tube (uncovered) result for each run.

Analysis of variance methods were applied to the normalized concentration response variable with the seven factors and levels: Vapor (n-hexane vs. m-xylene), Pump (continuous vs. pulsating), Exposure Profile (constant vs. variable), Flow Rate (200 mL/min vs. 30 mL/min), Sample Placement (mannequin vs. free hanging), Duration (80 min vs. 30 min), and Sample Holder (Buck vs. SKC), using the General Linear Model (Minitab Release 16; Minitab, Inc., State College, Pa.). The specified model included main effects and two-factor interaction terms. Results indicated three significant main effects: Exposure Profile ($p = 0.014$), Flow Rate ($p < 0.001$), and Sample Holder ($p = 0.009$), and three significant interaction terms, Pump Type*Flow Rate ($p = 0.004$), Pump Type*Sample Holder ($p < 0.001$), and Exposure Profile*Flow Rate ($p = 0.004$). The associated main effects and interaction plots are presented in Figures 5 and 6. The main effects plots generally show fitted means ranging from approximately 0.99–1.04 for the normalized concentration response variable. This means that on average the concentrations measured using the covered sample holders did not differ from the open tube sample results by more than 5%. The plots also show that average differences between covered sample holder results and the reference open tube samples were greatest for the variable exposure profile, low sample flow rate, and the Buck sample holder (Figure 5).

Referring to the interaction plots in Figure 6, the largest effects are seen when a low sampling flow rate was combined with the continuous flow pump or the variable exposure profile, and when the Buck sample holder was used with the continuous flow pump. For each of these three significant interaction terms, the normalized concentrations were within the approximate range of 0.99–1.08, meaning that average covered sample holder-measured concentrations generally did not differ from

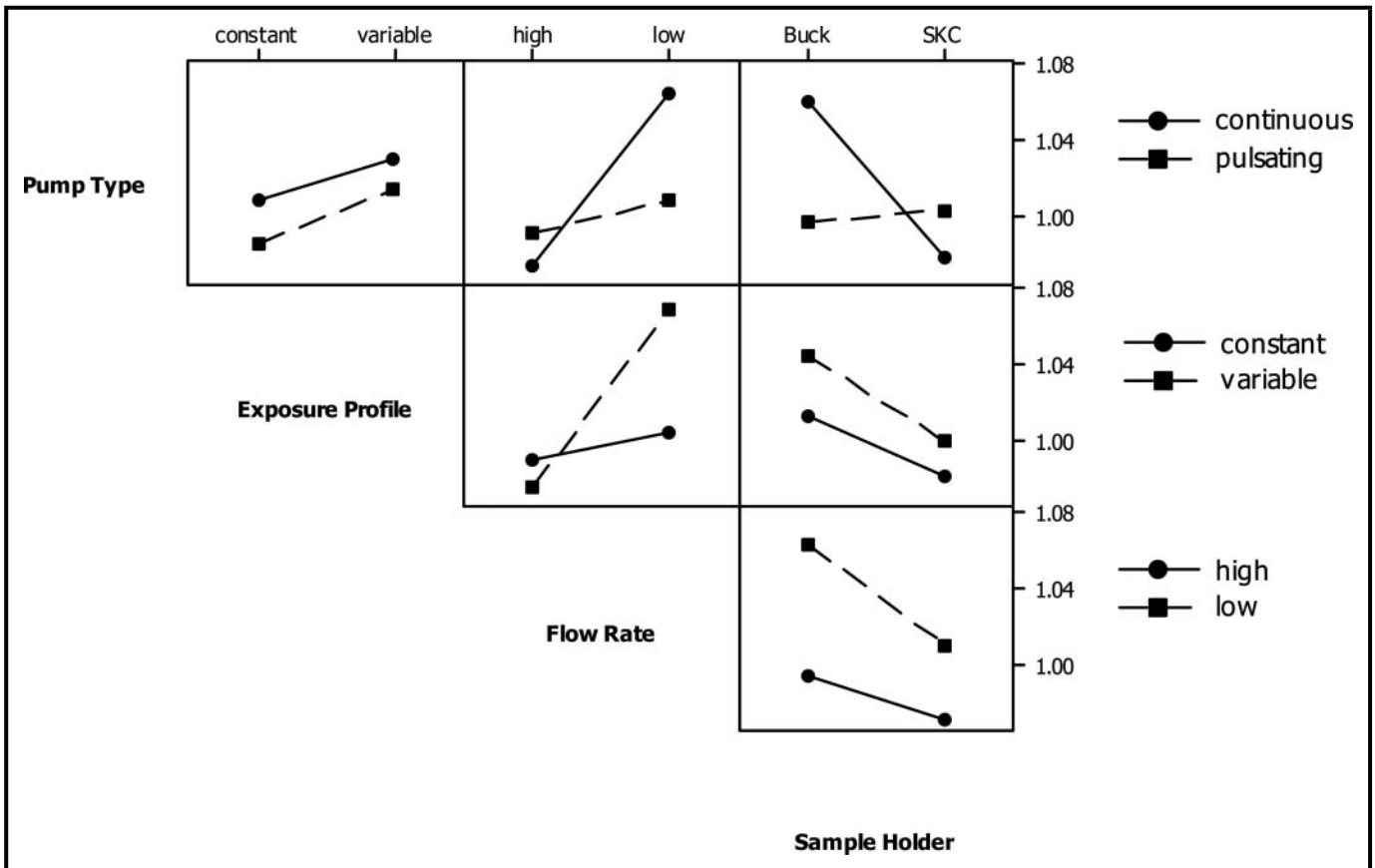


FIGURE 6. Interaction plot for normalized concentration. Statistically significant interactions identified for Pump Type*Flow Rate ($p = 0.004$), Pump Type*Sample Holder ($p < 0.001$), and Exposure Profile*Flow Rate ($p = 0.004$)

the reference open tube results by more than 10%. Two of the significant interactions do not include factors related to the atmosphere being sampled, i.e., Pump Type*Flow Rate and Pump Type*Sample Holder interactions are independent of the vapor or exposure profile characteristics, duration, and sample placement. This suggests the possibility of some type of systematic difference in calibration of the samplers with the greatest discrepancies occurring for a continuous pump, low flow rate, and the Buck holder. It is possible that this combination of factors merely highlights the conditions under which calibration differences between samplers are greatest. The open tube and SKC samples employ the same base to hold the tube and adjust flow and may, as a result, be more likely to produce equivalent results than the Buck sample holder.

Alternatively, the interaction between Flow Rate and Exposure Profile could reflect characteristics of the environment being sampled with the largest apparent effect seen for low flow rate and variable exposure profile. It seems possible that the dead volume created by the sample holder protective covers could contribute to this effect. Any resulting lag in the rise and fall of concentrations within the covered sample holder would likely be greatest for the lowest flow rate.

Despite the lack of a determination of definitive mechanisms for the significant interactions identified, the magnitude of the potential effects was relatively small. While statistically significant, the difference between normalized concentrations for covered SKC and Buck holders was less than 5% (95% confidence interval [CI] = 0.2–6.8%), and the overall average normalized concentration was not significantly different ($p = 0.499$) from a value of one (95% CI = 0.99–1.02). These differences are well within expected levels of variation for charcoal tube sampling and analysis ($CV_T = 0.10$)[10] and would not be expected to adversely affect the overall accuracy of these methods.

CONCLUSIONS

Although limited in scope, this study examined the possible effects of covered sorbent tube sample holders on measured organic vapor concentrations. Results indicate that while factors such as the nature of the exposure/concentration profile, sampling flow rate, and the type of sample holder used can influence agreement with open tube-measured concentrations, the magnitude of these effects are relatively small. It is not expected that the use of covered sample tube holders would contribute significantly to accepted levels of sampling and analytical variability for sorbent tube-based methods.

DISCLAIMER

The findings and conclusions in this report are those of the authors and do not necessarily represent the views of the National Institute for Occupational Safety and Health. Mention of sampling devices does not constitute an endorsement and does not imply that other devices are not fit for the same purpose.

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REFERENCES

1. **Rushing, D.E.:** Gas chromatography in industrial hygiene and air pollution problems. *Am. Ind. Hyg. Assoc. J. 19*:238–245 (1958).
2. **Otterson, E.J., and C.U. Guy:** "A Method of Atmospheric Solvent Vapor Sampling on Activated Charcoal in Connection with Gas Chromatography." *Transactions of the Twenty-Sixth Annual Meeting of the American Conference of Governmental Industrial Hygienists, Philadelphia, PA*. Cincinnati, Ohio: American Conference of Governmental Industrial Hygienists, 1964. p. 37.
3. **Fraust, C.L., and E.R. Hermann:** Charcoal sampling tubes for organic vapor analysis by gas chromatography. *Am. Ind. Hyg. Assoc. J. 27*:68–74 (1966).
4. **Van Houten, R., and L. George:** A method for the collection of air samples for analysis by gas chromatography. *Am. Ind. Hyg. Assoc. J. 30*:465–469 (1969).
5. **White, L.D., D.G. Taylor, P.A. Mauer, and R.E. Kupel:** A convenient optimized method for the analysis of selected solvent vapors in the industrial atmosphere. *Am. Ind. Hyg. Assoc. J. 31*:225–232 (1970).
6. **Perkins, J.L.:** *Modern Industrial Hygiene—Recognition and Evaluation of Chemical Agents*, Vol. 1. New York: Van Nostrand Reinhold, 1997. pp. 426–428.
7. **Lin, M.I., W.A. Groves, A. Freivalds, et al.:** Laboratory evaluation of a physiologic sampling pump (PSP). *J. Environ. Monit. 12*(*7*):1415–1421 (2010).
8. **Lee, L., M. Flemmer, E.G. Lee, et al.:** A novel physiologic sampling pump capable of rapid response to breathing. *J. Environ. Monit. 11*(*5*):1020–1027 (2009).
9. **Lin, M.I., W.A. Groves, A. Freivalds, et al.:** Exposure assessment by physiologic sampling pump—prediction of minute ventilation using a portable respiratory inductive plethysmograph system. *J. Environ. Monit. 10*(*10*):1179–1186 (2008).
10. **U.S. Department of Health and Human Services (DHHS), National Institute for Occupational Health and Safety (NIOSH):** *Occupational Exposure Sampling Strategy Manual*. DHHS (NIOSH) Pub. No. 77-173, Technical Appendix D, pp. 78–81, by N.A. Leidel, K.A. Busch, and J.R. Lynch. Cincinnati, Ohio: DHHS, NIOSH, 1977.
11. **Montgomery, D.C.:** Two-level fractional factorial designs. In *Design and Analysis of Experiments*. Hoboken, N.J.: John Wiley & Sons, Inc., 2005, pp. 282–335.