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A Portable Vapor/Particle Sampler

The airborne particle and vapor phases of a volatile organic chemical (VOC) often coexist in the real workplace environment. Assessment of worker exposure to a VOC requires measuring not only the total airborne concentration but also the phase distribution because the deposition efficiency of the material in the respiratory tract will depend on the form in which it is inhaled. A prototype portable vapor/particle sampler (PVPS) has been designed for sampling and quantifying the phase distribution of volatile components in micrometer-sized airborne particles and coexisting gaseous phase based on differential inertia. The sampler was laboratory tested and validated. Tests included sampler performance assessment and comparison with current sampling methods for particles and organic vapors, i.e., glass fiber filter, charcoal sorbent tube, and diffusion monitors. The PVPS is a low-cost and lightweight device that can be driven by a single standard personal sampling pump. The mass quantities of materials collected by the sampler can be determined by standard analytical procedures. Combined with an appropriate size-selective inlet, the PVPS may be used as a personal inhalable or respirable volatile aerosol sampler for occupational VOC exposure assessment, especially in industrial, or household, spray work environments where the particle sizes are frequently large.

Keywords: aerosol phase distribution, exposure assessment, personal sampler, volatile aerosol, volatile organic chemicals

Volatile organic chemicals (VOCs) are used in many industrial spraying processes. The particle phase and vapor phase of a VOC often coexist in the ambient workplace environment. Malek et al.⁽¹⁾ found that the styrene in aerosol droplets could contribute up to 30% of the total styrene concentration in the air during a resin spraying process in the reinforced plastic industry. Cohen et al. further demonstrated that xylene contained in paint droplets during automobile paint spray operations could represent up to 50% of the total airborne xylene concentration.⁽²⁾ Therefore, the actual worker exposure to a VOC is to both vapor phase and particle phase inhalation. Assessment of worker exposure to a VOC may require measuring not only the total airborne concentration but also the phase distribution in certain workplace conditions.⁽³⁾ The deposition efficiency of a material in the respiratory tract depends on the form in which it is inhaled. The lung dose resulting from equal inhaled concentrations can differ substantially. The American Conference of Governmental Industrial Hygienists Chemical Substances Threshold Limit Value (TLV) Committee has recognized this issue and suggested establishing dual TLV[®]s for the two phases of a

volatile substance.^(4,5) Proper personal and portable field sampling devices are needed for determination of the phase distribution of a VOC component in an industrial spray environment. However, because a volatile substance can be absorbed by, and evaporate from, particle surfaces during the sampling process, air samplers presently in use are not adequate for quantitative phase separation of a VOC component.⁽³⁾

Charcoal sorbent tubes (CSTs) are recommended by the National Institute for Occupational Safety and Health (NIOSH) for sampling airborne vapors of volatile hydrocarbons. However, Cohen et al.⁽²⁾ found that the air concentrations of xylene sampled by CSTs were higher than those determined by 3M diffusion monitors (DMs) at paint spray operations. This is attributed to the capture of not only vapor molecules but also particles by CSTs.^(6,7) Experiments for examining the particle filtration characteristics of CSTs were made previously with Arizona road dust particles at a flow rate of 400 mL/min. The results show overall CST filtration efficiencies in the range of 12 to 33%. The CST filtration efficiency increases with particle size, ranging from 10% for 0.5 μm particles to about 100% for particles greater than 2 μm in diameter.⁽⁸⁾ Measurement data of

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particle size distribution in automobile paint spray applications by Brosseau et al. showed that the paint droplets were large and polydisperse with geometric means from 20–40 μm and geometric standard deviations of 2–3.⁽⁸⁾ Similar results were found by Chan et al.⁽⁹⁾ It is suspected that the majority of paint droplets could be filtered by either the glass wool tip or the charcoal granules of CSTs. The concentration of an organic solvent sampled by a CST represents not only its airborne concentration in the gaseous phase but also an indeterminate portion of the particulate phase. Conventional sampling methods use filters for collecting particles and prefiltered charcoal sorbent tubes (F-CST) for collecting both vapor and particles when they coexist in the environment. In view of the collection characteristics of CSTs for particles, F-CSTs together with CSTs cannot provide efficient separation of the two phases of a VOC.

Gunderson and Anderson⁽¹⁰⁾ developed a two-stage vapor/particle sampler for separating airborne vapor molecules and particles based on their differential diffusion rates. The sampler consists of a sorbent-coated tube preceding a filter. When sample air flows through the tube, vapor molecules diffuse faster than particles to the tube wall to be collected by the sorbent, and particles pass through the tube to be collected by the filter. Vapor collection efficiency by a sorbent-coated tube mainly depends on the ratio of the residence time in the tube to that required for vapor molecules to diffuse to the tube wall. A pressure gradient of the vapor is established due to removal of vapor molecules by the sorbent-coated wall and causes further evaporation of the volatile components from the particles. Especially in a spray workplace or other indoor misty environments, where the VOC particle phase concentration is likely to be higher, the particle evaporation is not negligible.⁽³⁾

A prototype portable vapor/particle sampler (PVPS) was developed for sampling and quantifying the phase distribution of VOC components in micrometer-sized airborne particles and coexisting gaseous phase based on differential inertia. The design phase included the experimental determination of the optimum geometric parameters and operating conditions for the sampler, sampler calibration, sampler performance assessment, and inter-sampler comparison with current existing sampling methods for particles and organic vapors, i.e., glass fiber filter, CST, and DM.

DESIGN

A schematic diagram of the prototype PVPS is shown in Figure 1. The sampler consists of a sample flow inlet nozzle (1.0 mm in diameter), an anisokinetic tube, a 25-mm glass fiber filter, and two CSTs. The flow inlet nozzle is aligned at the centerline of an anisokinetic tube and at the center of the filter disk. At the throat of the inlet nozzle, sample inlet flow, Q_0 , splits into two flows, Q_p and Q_v , which instantly go in opposite directions. The Q_p flow travels in the same direction as the inlet flow while the Q_v flow turns 180° abruptly. Particles that possess larger inertia deviate from the Q_v streamline and follow the Q_p flow to be collected on the filter. The vapor in the air is divided into two flow portions, which are collected by two in-line CSTs, CST1 and CST2. CST1 also serves as a filter backup and collects vapor that evaporates from the particles collected on the filter during the sampling process. The constant flow rates are controlled by needle valves and calibrated using a Gilibrator Primary Flow Calibrator (Gilian Instrument Co., Wayne, N.J.). The VOCs collected by the glass fiber filter and the two CSTs can be separately desorbed with carbon disulfide (CS_2) and analyzed by gas chromatography.

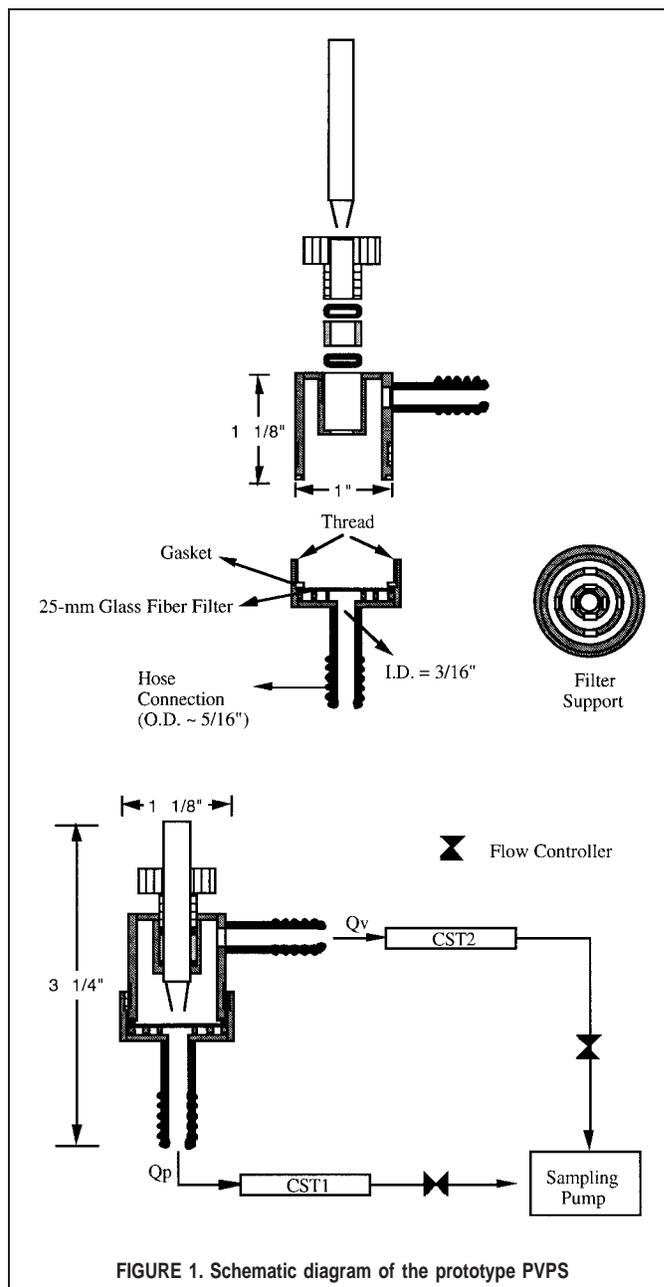


FIGURE 1. Schematic diagram of the prototype PVPS

Determination of the Airborne Concentrations of a VOC in Particulate Phase (C_p) and in Gaseous Phase (C_v)

The air concentrations of a VOC in the two phases and the total particle mass sampled (M_p) can be determined based on measured values of the masses collected on the filter (M_f) and the two CSTs (M_{CST1} and M_{CST2}), sampling time (t), the sample inlet flow rate (Q_0), and the dichotomous flow rates (Q_p and Q_v), as:

$$C_v = \frac{M_{\text{SCT2}}}{Q_v t} \quad (1)$$

$$M_p = (M_f + M_{\text{CST1}}) - \left[\frac{Q_p}{Q_v} \right] M_{\text{CST2}} \quad (2)$$

$$C_p = \frac{M_p}{Q_0 t} \quad (3)$$

$$Q_0 = Q_p + Q_v \quad (4)$$

Sample Inlet Flowrate (Q_0)

The sampler is designed to operate at a sample inlet flow rate of 1.8 L/min, which can be conveniently and reliably provided by lightweight, commercial personal sampling pumps.

Particle Cut-Off Size

The particle cut-off size, D_{50} , is mainly determined by the sampling flow rate and the inlet nozzle size. It can be roughly estimated as shown below for select flow rates and nozzle diameters:⁽¹¹⁾

$$\sqrt{c}D_{50} = \sqrt{\frac{St_{50}9\mu W}{\rho_p V_0}} \quad (5)$$

$$c = 1 + 1.257\frac{2\lambda}{D_{50}} + 0.40\frac{2\lambda}{D_{50}}\exp\left(-1.10\frac{D_{50}}{2\lambda}\right) \quad (6)$$

where c = Cunningham slip correction coefficient

St_{50} = Stokes number at 50% collection efficiency

μ = fluid viscosity

W = nozzle diameter

ρ_p = particle density

V_0 = average air velocity of sample flow in the nozzle throat

λ = mean free path of the air molecules.

For the dichotomous flow sampler, the cut-size is also determined by the dichotomous flow ratio ($\alpha = Q_p/Q_v$). The actual cut-size is very close to the theoretical calculation value. For $\alpha = 1/8$, theoretical and actual values are 0.93 and 0.90, respectively.

EXPERIMENTAL

Calibration

The PVPS was tested and calibrated using laboratory-generated particles and vapors. A vibrating orifice aerosol generator (VOAG-1 3050, TSI, St. Paul, Minn.) was employed to produce monodisperse fluorescein-tagged oleic acid particles in the particle size range greater than 1 μm from oleic-alcohol solutions containing less than 2% fluorescein in weight. Monodisperse particles were dried, diluted, and charge-neutralized before entering the sampler. An extra filter was placed between the side arm of the sampler and CST2 to collect the particles passing through the sampler. The size of the particles generated (D_p) can be calculated based on the vibration frequency (f), the volumetric rate of liquid feeding (Q), and the concentration of the solution (C) from the formula:⁽¹²⁾

$$D_p = \left(\frac{6QC}{\pi f}\right)^{1/3} \quad (7)$$

The quality and size of the monodisperse particles were also monitored on-line by a particle size analyzer (CI-208, Climat Instruments, Redlands, Calif.) equipped with a multichannel analyzer and an interfacing computer. The mass of fluorescein-tagged oleic acid particles collected on the two filters were quantified and analyzed by fluorometry with a fluorescence spectrophotometer (LS-5, Perkin-Elmer, Oak Brook, Ill.). The particle collection efficiencies can be determined and expressed as a function of particle aerodynamic diameter.

Monodisperse submicrometer fluorescein particles were generated from a system that included a Collison nebulizer and a differential mobility analyzer (DMA 3071, TSI) and measured by a condensation particle counter (CPC 3022, TSI). The particle

TABLE I. Particle Collection Efficiency of the PVPS with 1.0-mm Diameter Nozzle at a Total Sampling Flow Rate of 1.8 L/min

Aerodynamic Particle Diameter (μm)	Particle Collection Efficiency (%)	
	$\alpha = 1:8$	$\alpha = 1:1$
0.1		54
0.2		60
0.3		67
0.4	10	72
0.5	18	77
0.6	25	81
0.7	33	84
0.8	41	86
0.9	50	88
1.0	59	89
≥ 2.0	≥ 93	≥ 95

collection efficiencies were obtained by comparing the particle concentration passing through the sampler with that in an equivalent path without the sampler.

Vapor collection efficiency was measured with gaseous phase toluene, xylene, and styrene in a chamber equipped with a vapor generating device.

Performance Evaluation

To verify sampler performance, measurements of the PVPS were compared with other well-established reference samplers side by side. The intersampler comparison tests were carried out by comparing (a) the PVPS measurements of airborne vapor concentration from CSTs and 3M DMs and (b) the PVPS measurements of nonvolatile monodisperse test particle concentrations from glass fiber filters.

Chemical Analytical Method for Organic Solvents

The organic solvent collected by both the front and back sections of the CSTs (as separate portions), glass fiber filters, or the DMs were desorbed with CS_2 and analyzed by gas chromatography according to the NIOSH *Manual of Analytical Methods*.⁽¹³⁾ Desorption efficiencies, which vary with the types of collectors and chemicals, were determined by applying a known amount of the chemical to the collectors.

A gas chromatograph (Varian Aerograph model 2700, Walnut Creek, Calif.) was used in this study. The instrument is equipped with a flame ionization detector and an interfaced integrator (Reporting Integrator 3390A, Hewlett Packard, Avondale, Pa.). Optimum GC operating conditions, such as column types; carrier gas flow rates; injection, column, and detector temperatures, etc. were chosen according to the types of chemicals to be analyzed. Calibration curves were determined for all chemicals of interest. A 6 ft \times 1/8 inch o.d. stainless steel column containing 10% UC-98 on 80/100 mesh Chromosorb W AW DMCS was used in this work for analysis of toluene, xylene, and styrene samples. The temperature settings were 170°C for injection, 100°C for column, and 200°C for detector.

RESULTS AND DISCUSSION

Particle Collection Efficiency

The particle collection efficiencies (η) of the PVPS were measured for particle aerodynamic diameters of 0.03–10 μm at a total sampling flow rate of 1.8 L/min and two dichotomous flow ratios,

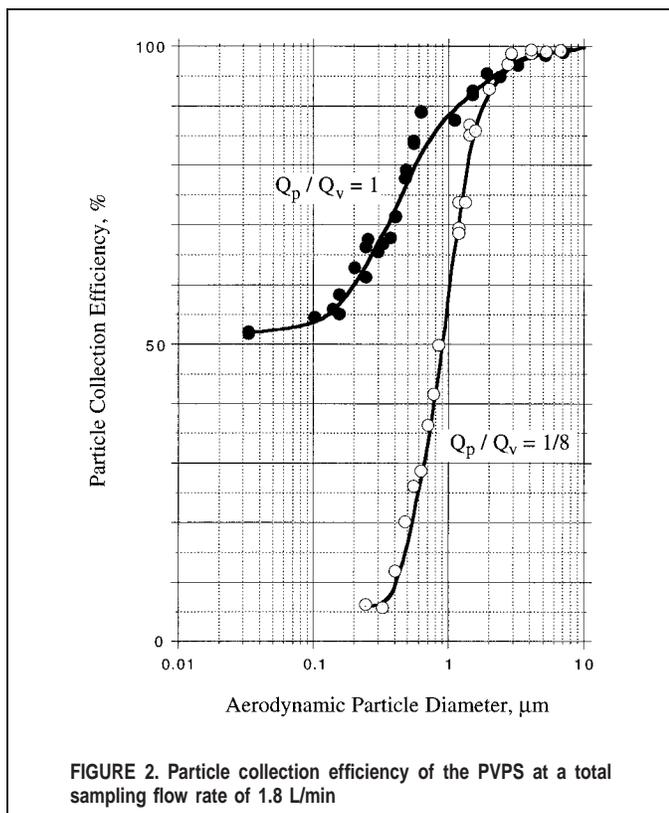


FIGURE 2. Particle collection efficiency of the PVPS at a total sampling flow rate of 1.8 L/min

$\alpha = 1/8$ and 1, by using fluorescein-tagged oleic acid particles (Table I). Here, η is defined as the total particle mass collected on the filter (M_f) against the total particle mass sampled (M_t):

$$\eta = \frac{M_f}{M_t} \quad (8)$$

The calibration curves are shown in Figure 2. The sampler has a relatively sharp particle size cut when it operates at $\alpha = 1/8$ with a cut size (D_{50}) of 0.9 μm . The particle collection efficiencies of

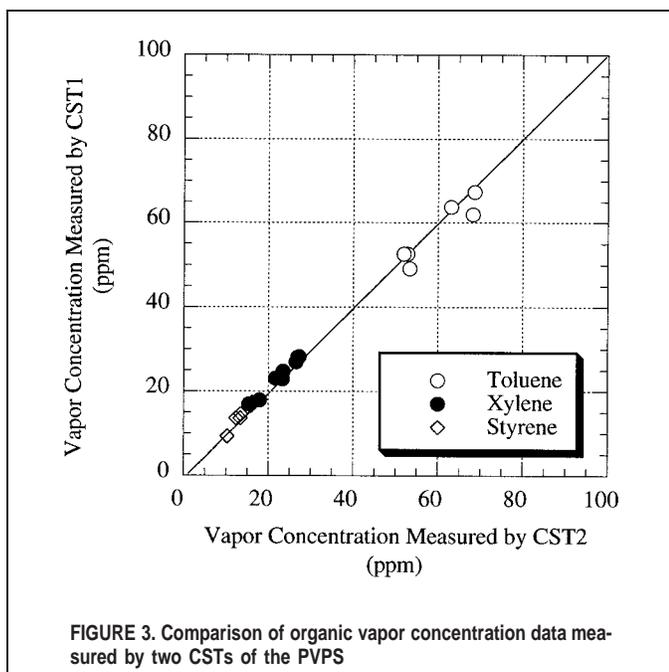


FIGURE 3. Comparison of organic vapor concentration data measured by two CSTs of the PVPS

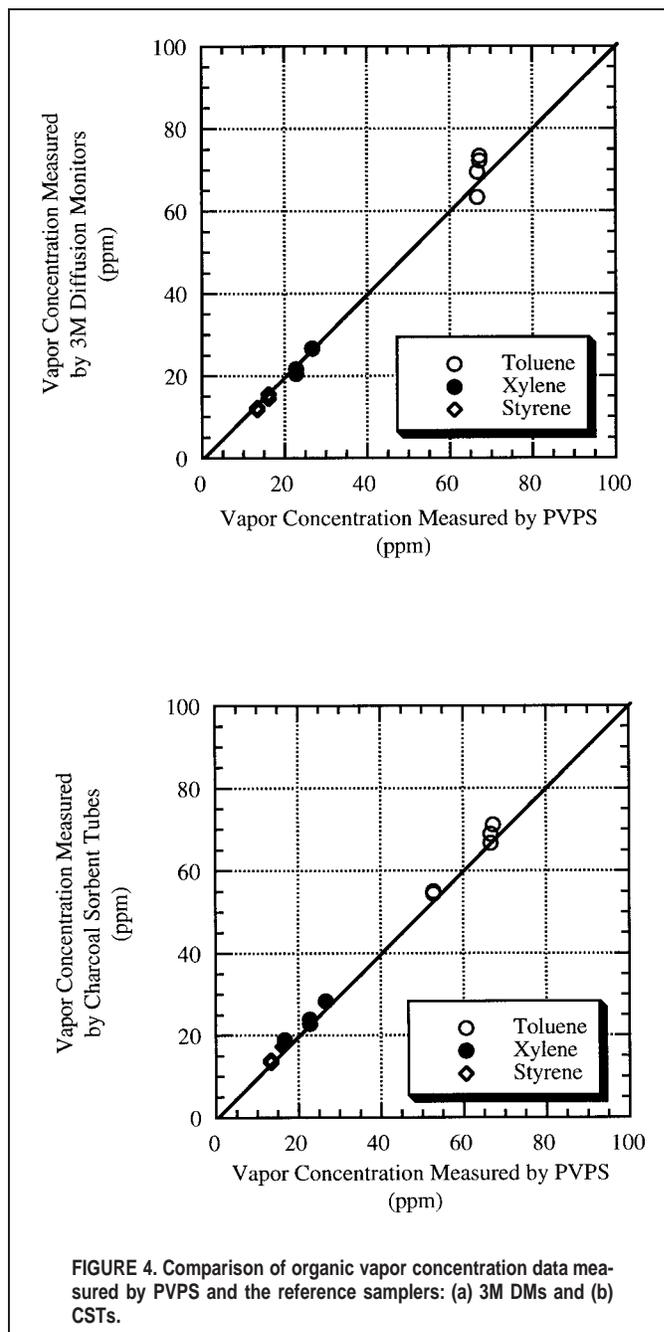


FIGURE 4. Comparison of organic vapor concentration data measured by PVPS and the reference samplers: (a) 3M DMs and (b) CSTs.

the filter with $\alpha = 1$ are greater than 50% for all particle sizes, and greater than 89% and 95% for particle size larger than 1 μm and 2 μm , respectively.

Particles generated in industrial spray processes are frequently large. The particles smaller than 2 μm hold less than 2% of the total mass concentration according to the data measured in automobile paint spray process by Brosseau et al.⁽⁸⁾ Therefore, the PVPS will separate particle and gaseous phases properly in these spray work environments.

Vapor Collection Efficiency

Testing of the vapor collection efficiency of the PVPS was focused on the ratio of the vapor fractions collected on CST1 and CST2, which should be unity after being normalized by the dichotomous flow rates. The 3M DM and CST were also used in the tests as

TABLE II. Comparison of the Organic Vapor Concentration Data Measured by PVPS and the Reference Samplers, 3M Diffusion Monitors and Charcoal Sorbent Tubes

Sampler A	Sampler B	C_A/C_B (mean \pm SD)	Average
PVPS-CST1	PVPS-CST2	1.02 \pm 0.02	1.02 \pm 0.02
PVPS CST1	CST	0.96 \pm 0.01	0.95 \pm 0.01
PVPS CST2		0.93 \pm 0.02	
PVPS CST1	DM	1.08 \pm 0.03	1.06 \pm 0.02
PVPS CST2		1.03 \pm 0.02	

reference samplers. The results are shown in Figures 3 and 4. Agreement between these measurements was within 6% (Table II).

Comparison with Reference Glass Fiber Filters

Intersampler comparison results showed a good agreement between the mass concentration data of monodisperse fluorescein particles measured by the PVPS and the reference filters, 37-mm glass fiber (Whatman GF/A), with a correlation of

$$\frac{C_{PVPS}}{C_{Reference}} = 0.96 \pm 0.06$$

(95% confidence interval, $n = 13$).

Wall Loss

The mean wall loss of the sampler is (2.2 \pm 0.9)% with a maximum value of 5.2% for a test set of 13 samples (95% confidence interval).

Loss of VOC Components from Particulate Portion Collected on Filter

Due to the rapid equilibrium between gaseous phase and particle phase of a VOC, there is no conventional method for separating the particle-bound VOC and its coexisting vapor without disturbing the equilibrium between the two phases. The innovative concepts of the PVPS are (1) to separate particles from the airstream in a relatively short time (milliseconds time scale) to minimize disturbing the phase equilibrium and (2) to evaporate most particle-bound VOC components from the filter into a backup CST, CST1, with an airflow passing the filter (Q_p). However, there may be some transfer of vapor from the particles collected on the filter to the side-stream air. Under normal sampling conditions, the concentrations of the pollutants in the environment are relatively stable. The particle-bound VOC will not be scrubbed into the side airstream (Q_v) due to the existing dynamic equilibrium. However, in a situation where ambient VOC concentrations change greatly during the sampling process, for example, from highly contaminated air to relatively clean air, particle-bound VOCs may release not only into the airstream passing the filter but also the side airstream.

The quantity of the particle-bound VOC loss is difficult to determine since there are no standard volatile aerosol sources. These laboratory tests were done by dosing a small quantity (10 μ l) of liquid styrene onto the glass fiber filter of the PVPS, immediately closing the sampler, then sampling. Some VOCs will evaporate from the filter and escape to the ambient air during the sampler assembling process. This fraction will then be sampled by the CST tubes. The quantities of the VOC vapor loss and collection are unknown. An estimate was made by assuming that the

TABLE III. Loss of Styrene from the Glass Fiber Filter

Q_p (L/min)	$\left(\frac{M_{CST1}}{M_0}\right)_{exp}$ (%)	$\left(\frac{M_{CST1}}{M_0}\right)_{est}$ (%)	$\left(\frac{M_{CST1}}{M_0}\right)_{exp} / \left(\frac{M_{CST1}}{M_0}\right)_{est}$ (mean \pm SD)
	82.8		
0.9	86.0	83.6	1.03 \pm 0.05
0.9	92.3		
	83.5		
0.85	81.5	84.2	0.96 \pm 0.01
0.95	80.0		
0.80	80.5	85.1	0.94 \pm 0.02
1.00	78.5		
0.2	28.3	56.2	0.45 \pm 0.05
1.6	21.9		
	25.3		

Note: M_{CST1} and M_{CST2} are the masses collected on the two CSTs, respectively. M_0 is the total styrene mass sampled ($M_0 = M_{CST1} + M_{CST2}$). The subscripts "exp" and "est" represent the experimental value and the estimated value, respectively.

value of the total vapor mass (M_v) to be collected is within a 1-inch radius spherical volume (comparable to the dimension of the PVPS), then:

$$M_v = \frac{PV}{RT} M_w \quad (9)$$

where P = partial pressure of the VOC
 V = volume
 R = gas constant
 T = temperature
 M_w = molecular weight.

For styrene, P = 6.10 mmHg at 25°C, $V = 4/3\pi r^3 = 69 \text{ cm}^3$, $M_w = 104.16$, so the total vapor mass is about 2.35 mg.

As shown in Table III, the quantity of VOC loss to the side airstream depends on the dichotomous flow rate ratios, α . Therefore, the problem can be overcome by increasing α . However, corresponding to this increase, the measurement error will be increased. Although the above experiment is only an estimation and represents an extreme variation condition of the ambient environment, these results are used as the sample design criterion since there is no other standard. For the selection of the dichotomous flow ratio, α , a value of 1/8 will be recommended if the ambient environment is relatively stable during the sampling process, since the lower α values are associated with lower sampling errors, and an optimum value of 1 is recommended for operating the PVPS under all other circumstances to prevent the loss of the particle-bound VOC to the side-stream air.

ESTIMATION OF THE SAMPLING ERRORS

The expected sampling error when mixed atmospheres are tested has been estimated by propagation of errors in terms of (a) the concentration ratio of the particle portion to the vapor portion of a VOC in air, C_p/C_v , and (b) the ratio of the dichotomous flow rates of the two fractions. The variances of means of the vapor and particulate phase concentrations of a VOC, $S_r(C_v)$, and $S_r(C_p)$

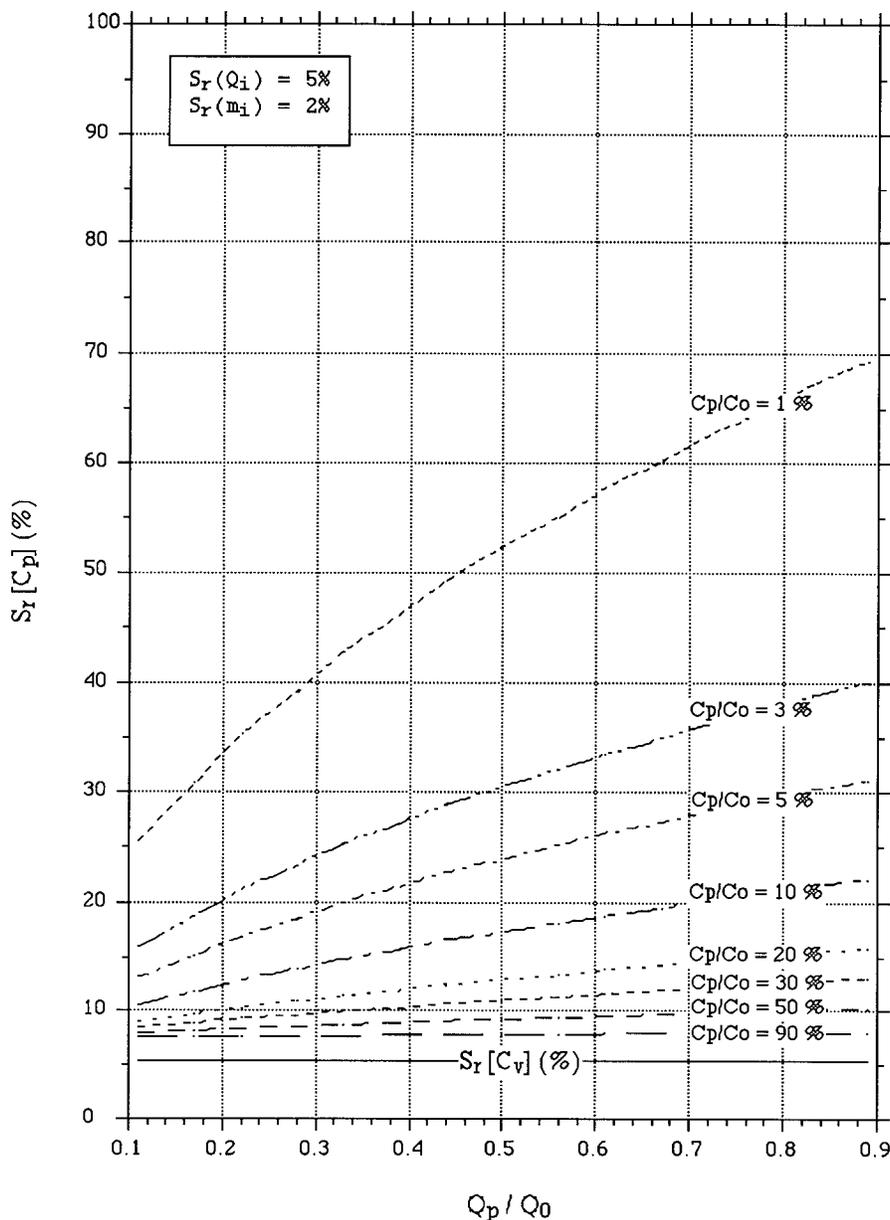


FIGURE 5. Estimated sampling error as a function of the flow rate ratio, Q_p/Q_0

can be derived from Equations 1–4 by using the propagation of error formulas:⁽¹⁴⁾

$$[S_r(C_v)]^2 = \left(\frac{S(C_v)}{C_v}\right)^2 = \left(\frac{S(M_{CST2})}{M_{CST2}}\right)^2 + \left(\frac{S(Q_v)}{Q_v}\right)^2 \quad (10)$$

$$\begin{aligned}
 [S_r(C_p)]^2 &= \left(\frac{S(C_p)}{C_p}\right)^2 \\
 &= \frac{[S(M_f)]^2 + [S(M_{CST1})]^2}{(M_p)^2} + \left(\frac{S(Q_0)}{Q_0}\right)^2 \\
 &\quad + \left(\frac{Q_p}{Q_0}\right)^2 \left(\frac{C_v}{C_p}\right)^2 \left(\left(\frac{S(M_{CST2})}{M_{CST2}}\right)^2 + \left(\frac{S(Q_p)}{Q_p}\right)^2 + \left(\frac{S(Q_v)}{Q_v}\right)^2 \right)
 \end{aligned} \quad (11)$$

where M_p , M_{CST1} , and M_{CST2} are the measurement values of VOC collected on the filter and two CSTs and M_p is the particle-bound VOC mass. SM_{CST1}/M_p , SM_{CST2}/M_{CST2} , and SM_f/M_p are the mass measurement errors with a typical value of 2% for VOCs.⁽¹³⁾ The variation of sampling flow rates, $S(Q_0)/Q_0$, $S(Q_p)/Q_p$, and $S(Q_v)/Q_v$ are generally within 5%.

As shown in Figures 5 and 6, the sampling errors are increased as Q_p/Q_0 increases and C_p/C_v decreases. However, even when the dichotomous flow ratio is increased to 1:1, i.e., $Q_p = Q_v = 0.9$ L/min, for a total sampling flow rate of 1.8 L/min, the estimated sampling errors are (1) under 10% when the particulate fraction of the total airborne concentration is higher than 40%, (2) 15% when the particulate fraction is in the range of 15–40%, and (3) 20% when the particulate fraction range is 8–15%, and are still in an acceptable range based on the theoretical analyses.

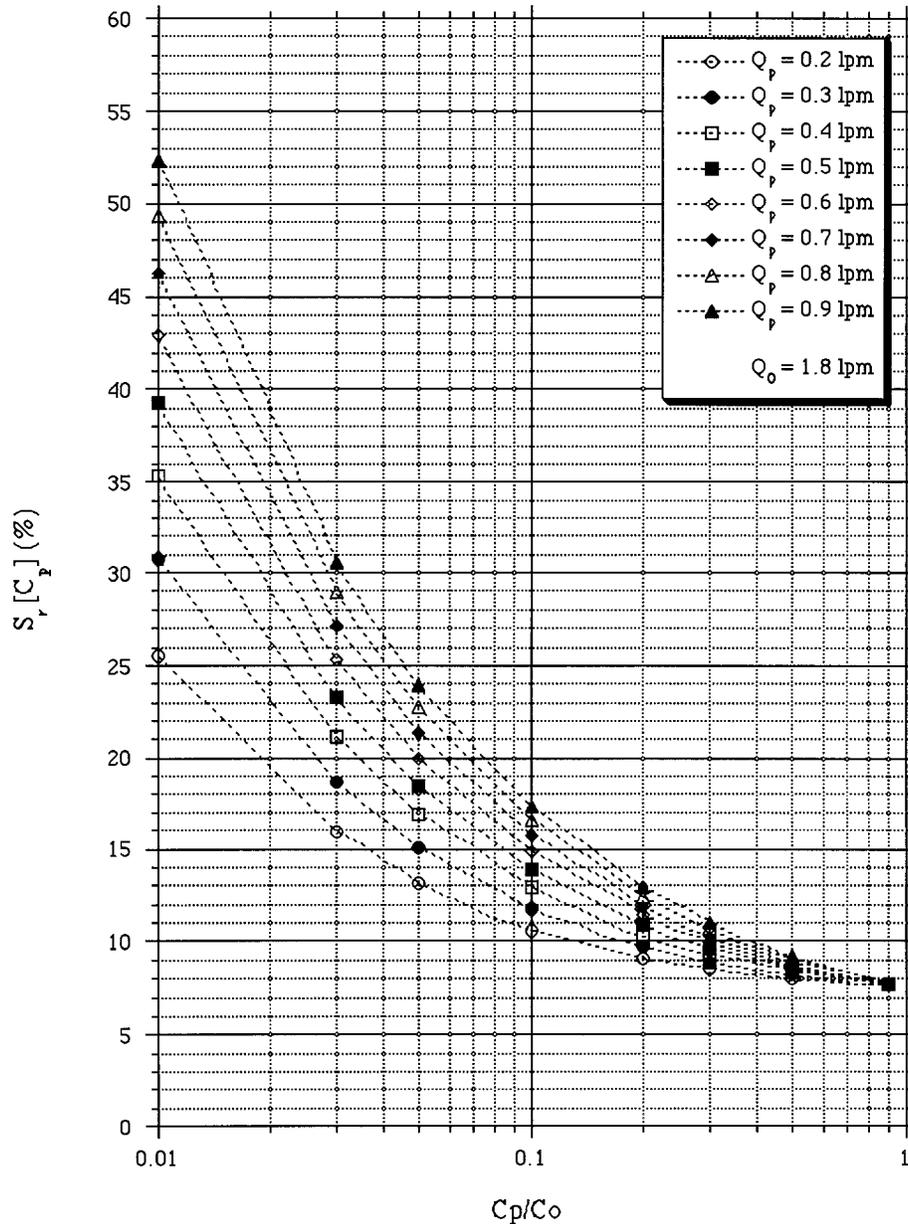


FIGURE 6. Estimated sampling error as a function of the concentration ratio, C_p/C_0 .

CONCLUSION

A prototype PVPS has been developed and characterized for sampling and quantifying the phase distribution of volatile components in micrometer-sized particles and coexisting vapors. Such knowledge is essential for proper evaluation of inhalation exposure for materials in both gaseous and particle phases. The PVPS is a relatively simple and inexpensive personal and field sampling device. The sampler is quite small, about 1 inch in diameter and 3 inches long, and operates with a standard personal sampling pump. The quantities of materials collected by the sampler can be determined by standard chemical analytical procedures.

The particle cut-size is designed to be $0.9 \mu\text{m}$ at a sampling

flow rate of 1.8 L/min and a dichotomous flow ratio of 1:8. Combined with an appropriate size-selective inlet, the PVPS may be used as a personal inhalable or respirable volatile aerosol sampler.

The application of the PVPS may include (a) industrial spray operations, such as the reinforced plastic industry and paint spray booths; (b) indoor sprays, such as household chemicals (air fresheners, cleaning solvents, insecticides) and products used in beauty salons (hairsprays, nail dryers); and (c) organic pesticide sprays on farmland or in greenhouses. The same principle may be applied for sampling the particles that contain volatile or semivolatile components that are traditionally sampled with other sorbent collectors (silica gel, porous polymer, etc.).

When there are large variations in ambient VOC concentration

or the phase distribution during the PVPS sampling, particle-bound VOC components collected on the filter may escape to the side-stream air. Laboratory test results showed that the problem could be overcome by increasing the dichotomous flow ratio to 1:1. The test results also showed that some design modifications would improve the performance of the prototype PVPS; additional laboratory and field studies are needed to validate and modify the sampler. Since there are no volatile aerosol standards, validation of the sampler may be performed by comparing the phase partition data of volatiles or semivolatiles as measured by the PVPS and by other sampling methods.

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