

Passive/Diffusive Samplers for Pesticides in Residential Indoor Air

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Pesticides applied indoors vaporize from treated surfaces (e.g., carpets and baseboards) resulting in elevated air concentrations that may persist for long periods after applications. Estimating long-term respiratory exposures to pesticide vapors in residential indoor environments using active (pump-based) sampling systems has been costly and burdensome on occupants. Diffusion-controlled passive sampling devices (PSDs), which do not require a noisy pump and can be situated unobtrusively within the home, would offer distinct advantages. The U.S. EPA is investigating the applicability of diffusive PSDs, semipermeable membrane devices (SPMDs), solid-phase microextraction (SPME), and other gas-sorbent partitioning samplers for estimating semivolatile organic compound (SVOC) pesticides in homes. This paper presents the results of the initial evaluations of two diffusion-controlled PSDs for determination of three pesticides in room air.

Keywords: pesticides, semivolatile, residential indoor, diffusive, gas-sorbent partitioning

1. Introduction

The United States Environmental Protection Agency (EPA) has for two decades been interested in potential applications of passive sampling devices (PSDs) for estimating the concentrations of various pollutants in air. During the early 1980s, several commercial PSDs designed for workplace use were evaluated for determination of volatile organic compounds (VOCs) in ambient air but were found to be limited by excessive background contamination and inadequate sensitivity [1]. This led to the development of a stainless steel diffusion-controlled PSD capable of high sensitivity for VOCs through thermal desorption [2]. While this PSD was manufactured during the 1980s and 1990s by Scientific Instrumentation Specialists, Inc., Moscow, Idaho, it saw little use [3, 4]. A version of the EPA PSD modified to collect nitrogen oxides also had limited use for indoor and outdoor air monitoring [5]. By the early 1990s, EPA had adopted a simple diffusive sampler for inorganic gases manufactured in Japan and marketed by Ogawa & Company USA, Pompano Beach, Florida, and Rupprecht & Patashnick Co., Inc., Albany, New York [6]. The Ogawa 3300 passive sampler for ozone, developed jointly by EPA and Harvard University, has enjoyed widespread use for ambient air monitoring in the U.S. [7, 8, 9]. Until very recently, however, there has been little interest in passive sampling for organic compounds.

Exposure to pesticides in indoor air has been a concern for many years, especially after a large study conducted by EPA in the 1980s showed that 85% of the total daily adult exposure to airborne pesticides was from breathing air inside the home [10]. Indoor residential sampling can be restricted because of the lack of available space for sampler placement or by homeowner objections, particularly regarding equipment noise. Therefore, PSDs, which do not depend on an air pump, would be attractive for estimating respiratory exposures to pesticides

inside occupied homes. Furthermore, unlike active samplers, PSDs may be deployed for days, weeks, or months to obtain integrated exposure estimates.

Although the current trend in the U.S. is toward the use of nonvolatile or low-volatility insecticides for residential pest control, most pesticides fall into the semivolatile category (vapor pressure range 10^{-2} – 10^{-8} kPa at 25 °C). This is especially true for disinfectants and room deodorizers. Semivolatile pesticides will be present in air primarily as vapors, which may be collected by diffusion onto a sorbent or by gas-sorbent partitioning. PSDs based on these sampling principles may be employed for estimation of respiratory exposures to pesticides within the home.

2. Materials and Methods

Two diffusive samplers with which the EPA has extensive experience were chosen for initial evaluation: the thermally desorbable PSD developed by Lewis et al. [2] and the Ogawa 3300 sampler (see Fig. 1). Both PSDs are cylindrical, dual-faced samplers that employ a series of diffusion plates and screens to control the sampling rate. The EPA PSD is constructed primarily of stainless steel and measures 3.8 cm o.d. by 1.2 cm (sorbent bed size 3.0 cm by 0.2 cm). The sorbent selected for this work was Amberlite™ XAD-2 resin (Supelpak-2™, Supelco, Belafonte, Pennsylvania); 0.73 g was used. The 2-cm o.d by 3-cm Ogawa PSD, which is designed to collect inorganic gases on treated filters by reactive sampling, was modified by hollowing out the solid body and filling the 1.5-cm by 1.5-cm void with 1.21 g of XAD-2. One of the two Ogawa stainless steel diffusion screens was placed on either end of the sorbent bed, and the snap-in diffusion caps were replaced to contain the sorbent.

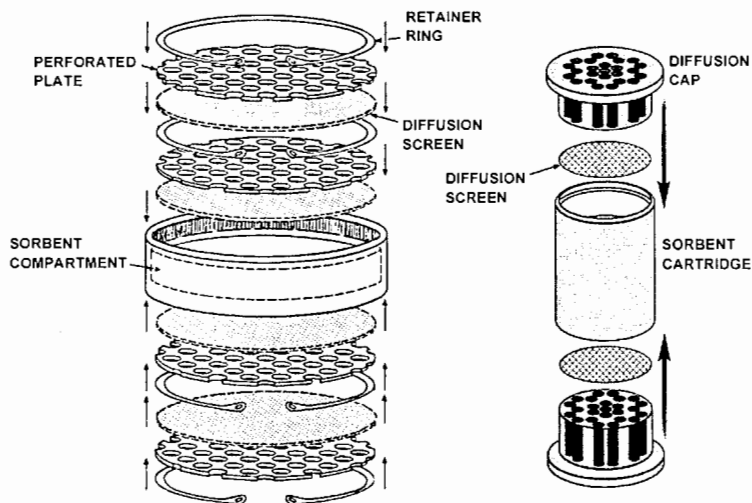


Figure 1. EPA (left) and modified Ogawa passive samplers.

Three insecticides were chosen for evaluation of the PSDs: diazinon (O,O-diethyl O-[6-methyl-2-(1-methylethyl)-4-pyrimidinyl]phosphorothioate, CAS No. 333-41-5; vapor pressure = 1.1×10^{-5} kPa at 25 °C), chlorpyrifos (O,O-diethyl-O-(2-isopropyl-6-methyl-4-pyrimidinyl) phosphorothioate, CAS No. 2921-88-2; vapor pressure = 2.6×10^{-6} kPa), and permethrin (O,O-diethyl-O-[6-methyl-2-(1-methylethyl)-4-pyrimidinyl]phosphorothioate, CAS

No. 333-41-5; vapor pressure = 4.5×10^{-8} kPa) [11]. The diffusion coefficients at 25 °C for these compounds were estimated by the method of Fuller et al. [12] to be 0.045, 0.043, and 0.040 cm²/s, respectively. (Note: The Fuller method was modified to utilize the molecular volume increments of Bondi [13]; in cases where volume increments were not available for specific molecular groups, they were estimated by comparison with values for other similar groups.) The sampling rate for the EPA PSD, R_{EPA} , was estimated by multiplying the ratio of the diffusion coefficient of test pesticide to that of chlorobenzene ($D_{CB} = 0.075$ cm²/s) by the empirically-determined sampling rate for chlorobenzene ($R_{CB} = 66.8$ cm³/min [2]). The calculated sampling rates for diazinon, chlorpyrifos, and permethrin were 39.6, 37.8, and 35.2 cm³/min, respectively. The sampling rate for the Ogawa PSD was calculated from Fick's first law of diffusion ($D_{Cp} \times A/L$) to be 6.2, 5.9, and 5.2 cm³/min for diazinon, chlorpyrifos, and permethrin, respectively. These values are in approximate agreement with the values derived by multiplying the ratio of the known sampling rate of the Ogawa PSD for NO₂ (21 cm³/min) to that of the EPA PSD (154 cm³/min) by R_{EPA} .

The performance of the PSDs was evaluated in a 30-m³ (2.2-m W by 5.9-m L by 2.4-m H) room (mobile office) with smooth composite wall paneling, vinyl flooring, and painted gypsum board ceiling. Heating and air conditioning were provided by a 12,100 kJ heat pump operating on 100% recycled air. All cracks and crevices except the door and the windows were sealed. Exposures were performed in the dark (except when retrieving samples). Continuous temperature, humidity, and air velocity were monitored with a Davis Weather Monitor II (Davis Instruments Corp., Hayward, California). The PSDs were deployed at a height of 1.8 m above the floor by suspending them from wires, each PSD separated by at least 20 cm. The EPA PSDs were held in a metal protective cage with openings covered with perforated plates containing 3-mm openings (no significant effect on sampling rate) and O-ring-sealed metal caps for closure before and after sampling [5]. The Ogawa PSD was held in a plastic mounting bracket. Three active samplers, with inlets 1.8-m above the floor and situated 117 cm apart, were evenly placed between the PSDs to determine reference air concentrations of the study pesticides. The active samplers sampled air at a rate of 1.2 L/min through a 2.5-cm quartz-fiber filter followed by a 2.2-cm by 7.6-cm polyurethane (PUF) vapor trap [14] and were operated continuously for 3-d intervals. The emitter source was a cut-pile nylon carpet, 1.8 m by 4.2 m, placed on the floor and uniformly broadcast-sprayed with aqueous emulsifiable concentrate pesticide formulations made up from Dursban™ L.O. (Dow Elanco, Indianapolis, Indiana), 41.5% (w/w) chlorpyrifos; Diazinon 4E (Bonide Products, Inc., Yorkville, New York), 47.5% (w/w) diazinon; and Spectracide Pro™ (Spectrum Group Division of United Industries Corp., St. Louis, Missouri), 10.0% (w/w) permethrin.

All samplers were prepared in a clean room free of pesticide contamination. Prior to exposure, the assembled samplers were enclosed in glass jars with PTFE-lined lids sealed with PTFE tape. After exposure, the PSDs were returned to their respective containers and kept at -80°C until extracted. The jars were allowed to equilibrate to room temperature for 2 h before opening. Once opened, PSDs were handled with tweezers or nitrile-gloved hands and received only brief exposures to laboratory air. The EPA PSD was removed from its protective cage, one sampling face was spiked with 400 µL of a 2.0-ng/µL standard of *p*-terphenyl-d₁₄ in *n*-hexane (surrogate recovery standard), and the intact device was quickly placed in a 500-mL Soxhlet extractor and immediately covered with the extraction solvent. The Ogawa PSD was removed from its mounting bracket, wiped with hexane (since its outer surfaces were exposed), spiked with the recovery standard, placed in a 250-mL extractor, and covered with solvent. The filter and PUF trap were removed from the active sampler cartridge and likewise placed in a 250-mL extractor. The Ogawa PSDs and active samples were extracted with 125 mL of 6% ethyl ether/94% *n*-hexane (pesticide quality) for 16 h at 7–12 cycles/h. The EPA PSDs were extracted with the same solvent in the same manner, except that 250 mL of solvent was

required (4–6 cycles/h). All extracts were concentrated to approximately 10 mL by rotary evaporation and reduced to a final volume of 2 mL at room temperature under a stream of high-purity nitrogen. Immediately prior to analysis, the final extract was spiked with an internal standard consisting of either a mixture of d_{14} -labeled polycyclic aromatic hydrocarbons for analysis by gas chromatography/mass spectrometry (GC/MS) or 2,4,5-tribromobiphenyl for gas chromatography/electron capture detection (GC/ECD). Recoveries of the test pesticides spiked onto XAD-2 were quantitative. Laboratory and field blanks of both PSDs and the active sampling cartridge were nearly always below levels of detection.

3. Results and Discussion

The EPA and modified Ogawa PSDs were exposed in triplicate for 3, 6, 12, and 21 days in the test room to vapors of the study pesticides emitted following broadcast spray application of formulation containing 1% (w/w) each of chlorpyrifos, diazinon, and *cis, trans*-permethrin (application rate 1.1 g/m²). In addition, one set of three of each PSD was exposed from day 3 to day 21 postapplication. Sampling commenced 2 h after application, when the carpet was dry to the touch. Active samples were also taken in triplicate for sequential 3-d periods starting on day 1 after application and continuing through day 21. Included also were three field blanks of each sampler type, which were taken from the preparation area to the exposure room and returned without opening their containers. The temperature in the exposure room ranged from 17 to 31 °C (mean 21 °C) and the relative humidity varied from 44% to 55% (mean 50%).

Results from the room test presented in Table 1 show that air concentrations were relatively constant over the 21-d test period, indicating that the room was well sealed. While the air levels of chlorpyrifos and diazinon, averaging 8.3 and 22.2 µg/m³, respectively, were higher than those typically encountered in residential indoor air, they were comparable to those found in residences within the first several days of an indoor application [15]. Diazinon, which is approximately four times more volatile than chlorpyrifos, was generally found at levels 3–4 times higher than those of chlorpyrifos. Permethrin, which is a nonvolatile compound, was not detected in any of the passive samples, but was detected in all active samples at 2–5 ng/m³. Mean air concentrations calculated from the vapors collected by the EPA PSD agreed very well with the active sampler data for chlorpyrifos, ranging from 77.5% to 98.8% of the active measurement (overall mean 85.9%, $\sigma = 7.2$), although precision between PSDs was poor (21–38% relative standard deviation [RSD] vs. 4–11% RSD for the active samplers). However, the results were less favorable for diazinon (range 49.1–70.4%, overall mean 61.9%, $\sigma = 7.7$; precision between PSDs 8–31% RSD vs. active 1.5–9% RSD). The low bias for the EPA PSD may be attributable to boundary layer resistance during quiescent periods in the test room, where air velocities near the PSDs ranged from 0.2 to 4.2 cm/s (mean 1.2 cm/s) when the heat pump was off to 6.1 to 116 cm/s (mean 20.8 cm/s) when it was on [2]. Air concentrations calculated for the Ogawa PSD were 5–12 times greater than the active sampler measurements for chlorpyrifos and 4–5 times those for diazinon, making it apparent that the device was not sampling according to Fick's first law of diffusion. Rather than sampling at the predicted 5.9–6.2 cm³/min (approximately 15% that of the EPA PSD), the Ogawa PSD collected 1–2 times as much pesticide as the EPA PSD. These data are presented graphically in Figure 2.

The large amount of oversampling by the Ogawa PSDs implies sorption of pesticide vapors by the materials from which the devices were constructed. This possibility was investigated by suspending three of the empty PSDs inside sealed 3.8-L metal cans (paint cans) over approximately 5 mL of the aqueous pesticide formulation contained in open petri dishes. After three days in the can, the empty Ogawa PSDs had collected 7.5–7.8 µg (mean 7.6 µg) of chlorpyrifos and 27–29 µg (mean 28 µg) of diazinon, some of which may have been from condensation under the highly humid conditions inside the cans (condensed moisture was

visible on the interior walls of the cans). Simultaneous exposures of empty EPA PSDs also resulted in collection of lesser quantities of the pesticides, presumably by condensation or surface adsorption; i.e., 0.8–2.7 μg (mean 2.0 μg) of chlorpyrifos and 4.0–11.0 μg (mean 8.1 μg) of diazinon. Examination of the reconstructed mass chromatograms from the test room samples revealed a large amount of contamination in the extracts from the Ogawa PSDs, suggesting an origin in the construction materials. Extracts of EPA PSDs were largely free of extraneous contamination. It was also observed that the diffusion plates of the Ogawa PSD did not fit tightly after extraction, suggesting that exposure to the solvent had adversely affected the plastic. The manufacturer's literature and distributors' Internet sites stated that the Ogawa

Table 1. Comparison of EPA and Ogawa PSDs with active sampler, n = 3.

| Active Sampler | | | Passive Samplers | | | Estimated* Air Conc., $\mu\text{g}/\text{m}^3$ | | |
|-----------------------|--|--|-----------------------|---|---------------|--|-------|--------|
| Sampling Period, Days | Mean Mass Collected μg (%RSD) | Mean Air Conc., $\mu\text{g}/\text{m}^3$, 3 d | Exposure Period, days | Mean Mass Collected, μg (%RSD) | | EPA | Ogawa | Active |
| | | | | EPA | Ogawa | | | |
| <i>Chlorpyrifos</i> | | | | | | | | |
| 0–3 | 44.5 (4.1) | 8.2 | 0–3 | 1.3 (34.3) | 1.0 (65) | 8.1 | 40.0 | 8.2 |
| 3–6 | 32.7 (5.1) | 6.0 | 0–6 | 1.8 (34.9) | 4.2 (6.2) | 5.5 | 83.1 | 7.1 |
| 6–9 | 47.1 (0.8) | 8.5 | 0–12 | 4.4 (33.5) | 7.9 (14.5) | 6.6 | 77.4 | 7.6 |
| 9–12 | 57.1 (4.4) | 10.6 | 0–21 | 8.2 (20.9) | 12.8 (1.1) | 7.1 | 71.9 | 8.3 |
| 12–15 | 38.2 (5.4) | 6.9 | 3–21 | 5.7 (38.5) | 11.4 (8.9) | 6.8 | 74.5 | 8.4 |
| 15–18 | 69.8 (10.6) | 12.8 | | | | | | |
| 18–21 | 30.6 (6.1) | 5.6 | | | | | | |
| <i>Diazinon</i> | | | | | | | | |
| 0–3 | 166.8 (5.0) | 30.7 | 0–3 | 3.7 (30.6) | 1.9 (33.9) | 21.6 | 69.6 | 30.7 |
| 3–6 | 117.2 (5.6) | 21.5 | 0–6 | 6.0 (23.8) | 4.6 (6.0) | 17.5 | 85.7 | 26.1 |
| 6–9 | 147.1 (1.5) | 26.5 | 0–12 | 11.6 (15.4) | 7.9 (20.3) | 16.9 | 74.3 | 26.8 |
| 9–12 | 155.6 (5.5) | 16.9 | 0–21 | 17.1 (8.3) | 9.8 (1.2) | 14.2 | 52.2 | 23.6 |
| 12–15 | 93.3 (8.2) | 26.2 | 3–21 | 11.4 (23.7) | 8.0 (10.8) | 11.0 | 49.7 | 22.4 |
| 15–18 | 143.2 (9.2) | 10.4 | | | | | | |
| 18–21 | 57.0 (4.0) | 23.6 | | | | | | |

*Calculated based on 5.9 and 37.8 cm^3/min sampling rates for chlorpyrifos for the Ogawa and EPA PSDs, respectively; 6.2 and 39.6 cm^3/min for diazinon. Air concentrations determined with the active sampler are mean values calculated from 3-d samples.

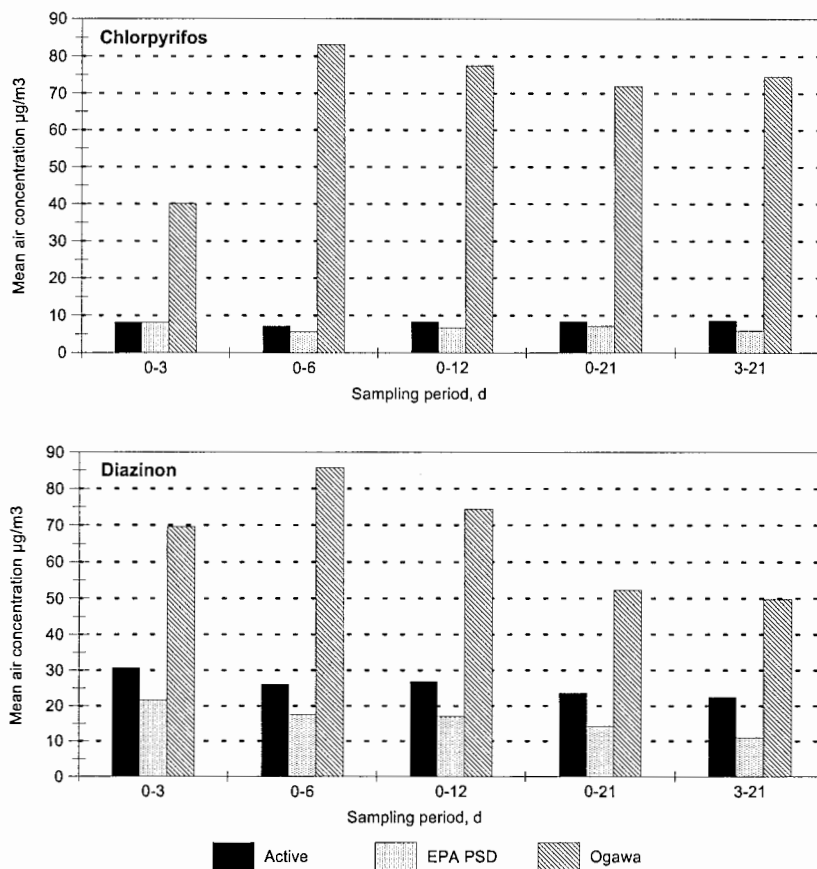


Figure 2. Air concentrations of chlorpyrifos (top) and diazinon in test room as measured by active and passive samplers.

bodies were made of Teflon™ or polytetrafluoroethylene (PTFE), but the composition of the diffusion plates was unspecified. However, discussions with the machinist who drilled out the Ogawa bodies cast doubt that the bodies were made of PTFE. Density measurements confirmed this doubt, revealing that the bodies had a density of $1.41 \pm 0.03 \text{ g/cm}^3$, comparable to the published [16] value of 1.43 g/cm^3 for Delrin⁷, an acetal plastic. The diffusion plate had a density of $0.8 \pm 0.07 \text{ g/cm}^3$, suggesting that they were made from low-density polyethylene (LDPE). Both of these plastics, but especially LDPE, are sorptive [17] and susceptible to damage by organic solvents.

4. Conclusions

The results of this initial test eliminate consideration of the Ogawa 3300 as a potential PSD for semivolatile pesticides, although the PSD might perform well if constructed of PTFE or high-quality stainless steel. The EPA PSD must be further evaluated before it can be accepted as constructed or modified to improve its performance. Dry deposition of semivolatile compounds onto the surfaces of PSDs, whatever their composition, is a potential problem not likely to be encountered with VOCs. Surface adsorption of vapors by construction materials, including

stainless steel (especially high-carbon types) may also compete with diffusional sampling processes.

Studies are currently under way to investigate the potential of the EPA PSD construction materials to collect pesticide vapors from room air. Additional room tests are being conducted to evaluate the performance of the EPA PSD at lower air concentrations. A 122-cm ceiling fan has been installed to assure that air velocities near the PSDs are always above 10 cm/s. In addition, two other devices are undergoing tests: the semipermeable membrane device (SPMD) developed by the U.S. Geological Survey for water monitoring [18] and the MPS badge, a gas-sorbent partitioning sampler developed by Midwest Research Institute for organophosphate vapors. The SPMD, which collects vapors passing through LDPE tubing into triolein by the processes of permeation and air-solvent partitioning, has been found to be highly efficient at recovering semivolatiles compounds, including pesticides, from ambient air [19]. The SPMDs currently under study contain 0.25 mL of triolein encased in flat tubes of LDPE measuring 2.5 cm by 15 cm, and have estimated sampling rates for diazinon and chlorpyrifos of 1100 and 660 cm³/min, respectively. Pesticides collected by the SPMD are recovered by dialysis in *n*-hexane. Because of its very high sampling rate, boundary layer resistance may have a profound effect on the SPMD. The MPS device is a simple 1.6-cm by 3-cm by 1-mm-thick piece of material made from a proprietary carbon-based mixture cross-linked to a high-temperature polymeric material that is directly exposed to air. Its estimated sampling rate for the study pesticides is on the order of 100 cm³/min. Analytes are recovered by extraction with a polar organic solvent. Also planned are investigation of semipermeable membrane extractors (SPMEs), which can be used to sample in either the gas-sorbent partitioning or diffusive modes [20, 21]. PSDs with satisfactory test room performance will be further tested in the EPA IAQ Test House, a single-story 121 m² home, and subsequently subjected to field evaluations in occupied homes in which pesticides are routinely used.

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