How should we characterize emissions, transport, and the resulting exposure to SVOCs in the indoor environment?

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ABSTRACT

A systematic and efficient strategy is needed to assess and manage the potential risks to human health that arise from the manufacture and use of thousands of chemicals. For both volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs), exposure is strongly influenced by the types of materials and products in which the VOC or SVOC occur, the concentration of the VOC or SVOC in the material or product, the way in which the material or product is used or applied indoors, and the ventilation rate within the room or building. For VOCs, the rate of emissions from various materials and products are generally characterized in small chambers. The results are scaled up to represent what happens in a larger room, and then used to estimate the expected gas-phase concentration and associated health effects. For SVOCs, however, the characteristics of the room play a much larger role in controlling the process of emissions, transport, and exposure. For example, partitioning of SVOCs to interior surfaces including walls, windows, furniture, clothing, dust and airborne particles, all substantially affect the emissions and transport processes, as well as the resulting exposure. Given the inherent complexity of the situation, what is needed is a screening-level approach which is based on rapid estimates of exposure to a specific SVOC that is present within a specific material or product. These rapid estimates of exposure are then combined with estimates of toxicity to create a screening-level estimate of the risk associated with the specific SVOC/product combination. The SVOC/product combinations with relatively high risks can then be evaluated, focusing on more accurate measurements of the key parameters that govern emissions, transport, and the resulting exposure to SVOCs in the indoor environment. Among available tools for evaluating these key parameters, there are significant gaps associated with the SVOCs. To close these gaps, we have developed simple methods to measure some of the key parameters. These parameters include the gas-phase concentration in equilibrium with the product surface, the partition coefficient between air and indoor surfaces, the vapor pressure of liquid SVOCs, and the partition coefficient between air and airborne particles. Phthalates in polyvinyl chloride flooring were selected to test these measurement methods, with results that agree well with those measured in previous, more sophisticated tests. As increasing numbers of these measurements are completed, the overall approach should substantially improve our ability to estimate the potential exposure to SVOCs in indoor environments and can help with the risk-based prioritization of a wide range of chemicals and products.

KEYWORDS

Semi-volatile organic compounds (SVOCs), Screening-level estimates, Exposure, Risk

1 INTRODUCTION

Modern indoor environments contain a vast array of products, many of which emit semi-volatile organic compounds (SVOCs) such as plasticizers, flame retardants, and biocides (Weschler and Nazaroff, 2008). As a result, SVOCs are ubiquitous indoors, redistributing from their original sources to indoor air and all indoor surfaces including skin, airborne particles, and dust. Many SVOCs are associated with adverse health effects, and several are considered potential endocrine disrupters (Rudel and Perovich, 2009). Products such as building materials, furnishings, household goods and those used for personal-care contain a vast array of SVOCs. In some cases, SVOCs are sprayed as a liquid or applied as a powder. Such applications are the common means by which pesticides, termiticides, herbicides, and sealants are introduced to indoor environments. In other cases, SVOCs are present as additives or solvents in materials used indoors. These include plasticizers, flame retardants, anti-oxidants, preservatives, and coalescing agents. In addition to these two primary classes, SVOCs can be generated by incomplete combustion, by high-temperature heating processes and by chemical processes.

To prioritize testing and to manage risks of industrial chemicals including SVOCs, methods are being developed and applied to rapidly evaluate compounds for their potential hazard. The ToxCast^{TM} program applies recent advances in high-throughput screening, computational chemistry, and toxicogenomic techniques to profile bioactivity and prioritize toxicity testing for hundreds of chemicals (Judson et al., 2010). When combined with high-throughput estimates of toxicity, the development of efficient exposure tools should enable a more complete risk-based prioritization of chemicals in commerce. Of the roughly 100,000 chemicals catalogued in the Aggregated Computational Toxicology Resource (ACToR), for which there is at least limited toxicity information, some form of exposure information is available for less than 20%. Readily accessible data on concentrations in exposure-related media (air, dust, food) are available for a much smaller proportion (Egeghy et al., 2012). Consequently, exposure-based prioritization currently requires the use of simple models to estimate exposure potential.

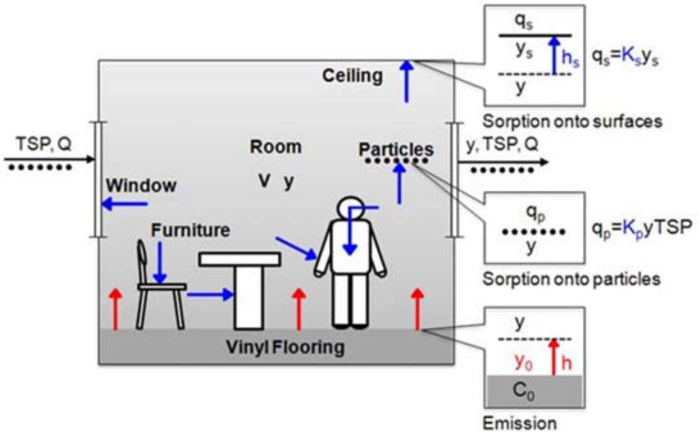

Figure 1: Schematic showing mechanisms governing emissions of SVOCs present in products as additives.

With this in mind, Little et al. (2012) proposed a method to obtain screening-level estimates of potential indoor exposure to SVOCs from a single source through a range of potential exposure pathways (including inhalation, ingestion and dermal sorption) based on a mechanistic understanding of the emissions, transport, and human uptake processes. SVOCs that are either present as additives in products used indoors or as ingredients in products directly applied to interior surfaces were considered in this method because these two classes are the major contributors to the broad spectrum of manufactured SVOCs found indoors (Little et al., 2012). Figure 1 shows the mechanisms governing emissions of SVOCs from a solid material in a room. The mechanisms affecting transport of SVOCs include emissions of SVOCs from the surface of the source to the air in immediate contact with the source, mass-transfer through the boundary layer into the bulk room air, and sorption to airborne particles and interior surfaces. The room air is assumed to be well-mixed. In Figure 1, Q is the ventilation rate, V is the room volume, TSP is the total airborne particle concentration, C_0 is the concentration of an SVOC in the source, y_0 is the equilibrium concentration in the air immediately adjacent to the source, y is the gas-phase concentration in the room air, q_s is the sorbed surface concentration on the interior surface, y_s is the gas-phase concentration in equilibrium with q_s, q_p is the sorbed particle-phase concentration, K_s is the surface/air partition coefficient ($K_s = q_s/y_s$), K_p the particle/air partition coefficient ($K_p = q_p/(y \cdot TSP)$), and h and h_s are gas-phase mass transfer coefficients. The system is further simplified by assuming that C_0 is effectively constant (so much SVOC is present in the source that the concentration does not deplete significantly) (Xu and Little, 2006), and by assuming that gas-phase SVOCs can instantaneously reach equilibrium partitioning with airborne particles and interior surfaces.

With reference to Figure 1, the accumulation of SVOC in the air obeys a mass balance, or:

$$\frac{dy(t)}{dt} \cdot V + \frac{dq_p}{dt} \cdot V = h \cdot (y_0 - y) \cdot A - h_s \cdot (y_0 - y_s) \cdot A_s - Q \cdot y - Q \cdot q_p \qquad (1)$$

where A is the source surface area, and A_s is the exposed interior surface area of the room.

A similar model can be applied for SVOCs that are sprayed or applied (Little et al., 2012). The models can be used to predict y, the gas-phase concentrations of SVOCs, as well as their concentrations in airborne particles and house dust, and to then estimate SVOC exposures through inhalation, ingestion and dermal sorption. Equation (1) indicates that y is largely determined by y_0, K_s, K_p, and other parameters that are relatively easy to quantify (A, A_s, h, h_s, Q, and TSP). However, because the values of several parameters (y_0, K_s, and K_p) is in general unknown, simple and accurate methods for measuring these parameters are needed.

2 NOVEL METHODS TO MEASURE KEY PARAMETERS

2.1 Diffusion sampler method to measure y_0

For consumer products containing a high SVOC weight percentage, y_0 may in some cases be approximated by the vapor pressure of the pure SVOC (Clausen et al., 2012). Unfortunately, y_0 cannot be approximated by vapor pressure in all cases (Liang and Xu, 2014). Experimental methods are therefore necessary to determine y_0. The methods developed by Liang and Xu (2014) and Cao et al. (2017) are currently the best time-saving methods for y_0 measurements via small-scale environmental chambers. For emission of di-(2-ethylhexyl) phthalate (DEHP) from polyvinyl chloride (PVC) flooring, the experimental time can be as short as 1 – 5 days. However, the complexity of the chamber tests limits the practicality of these two approaches. The material has to be flat and relatively soft in order to be properly installed in the chamber. Therefore, a simple method for directly measuring y_0 without a chamber may be used (Wu et al., 2016b). In this method, a standard stainless steel thermal desorption tube is vertically placed on the surface of an SVOC source material, SVOCs will emit from the source into the tube air, transfer through the air in the tube, be adsorbed on the tube interior surface and finally be adsorbed on the sorbent at the far end of the tube, as shown in Figure 2.

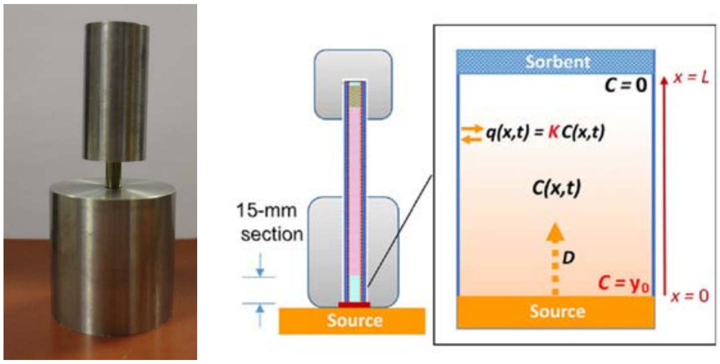

Figure 2: Photo and schematic representation of the model of the experimental system for measuring y_0.

Based on these mechanisms, we developed a model to predict the uptake rate of SVOCs emitted from the product surface. The model accounts for gas-phase diffusion of SVOC through the air gap in the tube, as well as adsorption of SVOC onto the interior surface and the sorbent of the tube. Values of y_0 and an additional unknown parameter, K, the tube surface/air partition coefficient, are obtained by fitting a diffusion model to the sampling data. As increasing numbers of such experiments are completed, the ability to reliably predict K for specific SVOCs according to their vapor pressure should improve substantially, leaving y_0 as the only unknown. Phthalates in two types of PVC flooring were selected to test the method. There is very good agreement between the experimental results and fitted curves based on the model for all measurements (Figure 3). In addition, the values of y_0 and K agree well with those measured in independent chamber tests, and the experimental time is comparable to existing methods (several days, as shown in Figure 3). This method is shown to be applicable to chemicals with a wide range of vapor pressures and the shape of the source material is not limited by this method, both facilitating the measurement of y_0 for as many SVOC/product combinations as possible.

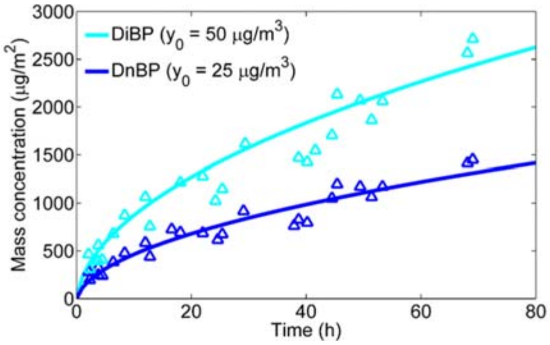

Figure 3: Measured DiBP and DnBP in the tube as a function of time and best-fit model curves.

2.2 Simple method to measure K_s on impervious surface

To date, few methods are available to measure the partition coefficient between air and indoor surfaces, K_s. The most common approach is to pass an air stream with a constant concentration of target compounds through a ventilated chamber initially containing clean test materials. The values of K_s are determined after adsorption equilibrium is established between the material and air. For example, Morrison et al. (2015) measured the K_s values for diethyl phthalate (DEP) and di-n-butyl phthalate (DnBP) on cotton fabrics in small chamber experiments. Since equilibrium partitioning is a prerequisite for these measurements, a noticeable constraint is the long experimental duration, which is usually more than 10 days. Furthermore, the time required to reach equilibrium could be impractically long (>85 days) for SVOC/surface pairs with large K_s (Cao et al., 2016). To alleviate this problem, Cao et al. (2016) recently developed a sealed-chamber approach without the requirement of reaching equilibrium to measure K_s values of DEHP, di-iso-butyl phthalate (DiBP), and DnBP partitioning onto cotton clothing. However, the sink of SVOCs on the chamber wall is neglected, which could compromise its accuracy. In addition, the required time to make reliable measurements can still be long for compounds with large K_s values such as DEHP (more than 72 days at 25 °C).

To solve this problem, we recently developed a simple method based on a passive sampling technique to measure the K_s values for SVOCs (Wu et al., 2017). The cylindrical diffusion chamber consists of an aluminum shim, an SVOC source material, and a test material disk, as shown in (Figure 4). The circular shim is placed between the SVOC source and the test material disk. In this way, one side of the test material is exposed to the SVOCs emitted from the source material. The thickness of the shim defines the diffusion distance between the emission source and the surface of the test material. A weight is placed on top of the material disk to hold the shim and the disk firmly against the flat source material surface (Figure 4). The driving force for mass flux across the air gap is the concentration gradient between the emission source and the air adjacent to the test material surface.

Figure 4: Elements of the diffusion chamber sampler for measuring K_s.

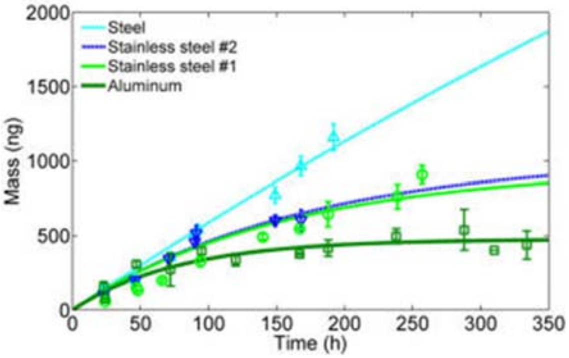

Figure 5: Measured DEHP mass accumulated on the impervious surface as a function of time and best-fit model curves for steel, stainless steel #1, stainless steel #2, and aluminum.

A mass transfer model, which is very similar to that for the measurement of y_0 (Wu et al., 2016b), was developed to predict the sorption amount of SVOC on the test material surface as a function of time. The model implies that the experimental time will increase as the shim thickness increases. Therefore, the shim thickness was selected as 2 mm in order to shorten the experimental time as much as possible. K_s can be obtained by fitting the model to the SVOC mass collected on the test material surface over a series of sampling times. The Levenberg − Marquardt algorithm was used to find the best-fit K_s through an iterative fitting procedure. Sorption of DEHP on typical impervious surfaces including aluminum, steel, glass, and acrylic was selected to test the method. The relative standard deviation (RSD) of most replicates ranged from 1% to 26%. The coefficients of determination R^2, which provides a measure of how well the sampling data are fitted by the model, were all larger than 0.84, indicating good agreement between the experimental results and fitted curves (Figure 5). The requisite time to make reliable measurements for DEHP was about 15 days, which is shorter than existing methods (e.g., 30 days (Cao et al., 2016)).

2.3 Tube chamber method to measure K_p

In existing studies, K_p is usually predicted by empirical equations that include the saturation vapor pressure of pure sub-cooled SVOC liquid or the octanol-air partition coefficient of SVOCs (K_{oa}). However, the accuracy of the predicted K_p is doubtful (mainly due to the uncertainties in saturation vapor pressure of pure sub-cooled SVOC liquid or K_{oa} and the various assumptions made for the equations (Wei et al., 2016). Therefore, accurate measurement of K_p is vital for successfully characterizing the interaction between gas-phase SVOCs and particles.

Currently, few studies focusing on measuring K_p are available for typical indoor SVOCs. The study of Benning et al. (2013) was the first to quantify K_p of SVOCs under well-controlled chamber conditions, but its accuracy was in part reduced by the complexity of the air velocity field within the chamber, especially when equilibrium between SVOCs and particles cannot be attained at the chamber outlet. Therefore, we proposed a novel method for measuring K_p using a tube chamber (inner diameter: 1.72 cm, length: 3.60 m) coated with a thin layer of pure SVOC liquid on the inner surface (Wu et al., 2018). Figure 6 shows the schematic of the experimental system. Particle-laden air at a controlled flow rate, which ensures the air flow in the chamber to be laminar, was introduced into the chamber. Two sorbent tubes were connected in parallel to the chamber outlet, one with a filter measuring the effluent gas-phase SVOC concentration (C_g), the other without a filter measuring the sum of gas- and particle-phase SVOC concentration (C_{tot}), and the effluent particle-phase SVOC concentration (C_{sp}) can be obtained by ($C_{tot} - C_g$). As fresh air (free of SVOCs) is introduced into the chamber, the air flow will carry the SVOCs toward the chamber outlet and dilute the SVOCs. Due to the dilution, more SVOCs will emit from the liquid surface coating and subsequently diffuse toward the centerline of the tube. When particles are present in the air, uptake of SVOCs by particles will occur as they move toward the chamber outlet and diffuse toward the tube centerline. Given that the tube is sufficiently long, equilibrium between gas-phase SVOC and particles was attained at the chamber outlet, and K_p was determined by $C_{sp}/(C_g \cdot TSP)$.

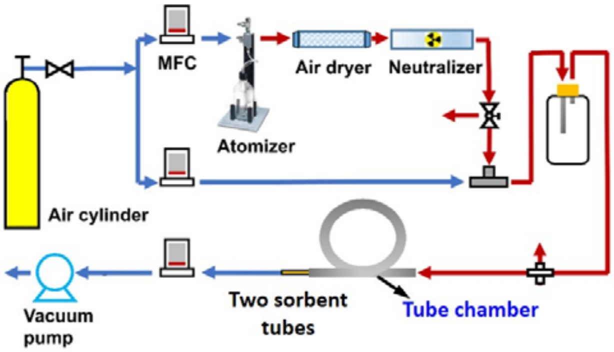

Figure 6: Schematic of the experiment setup for measuring K_p.

Uptake of DEHP by one inorganic (ammonium sulfate) and two organic (oleic acid and squalane) particles was selected to test the method. The values of K_p for the ammonium sulfate, oleic acid, and squalane particles were determined to be 0.011 ± 0.004 $m^3/\mu g$, 0.2321 ± 0.13 $m^3/\mu g$, and 0.11 ± 0.10 $m^3/\mu g$, respectively (Wu et al., 2018). The precision of this method is acceptable considering that the accurate determination of SVOC concentrations and the separation of gas-phase and particle-phase SVOCs are experimentally very challenging.

2.4 Measurement of vapor pressure of liquid SVOCs

Even though we have developed a simple method to measure y_0, it is still impossible to obtain for every SVOC/product combination (e.g., for materials with irregular shape including toys). For such cases, an estimate of y_0 based on existing product characteristics, typically the material-phase concentration C_0 of the SVOC in the product, would be very useful. Recently, Eichler et al. (2018) investigated the relationship between y_0 and C_0 for different SVOC/product combinations through theoretical analysis and experimental measurements. The results showed that the material/gas equilibrium relationship follows Henry's Law. In addition, the Henry's Law Constant for all target SVOCs consisted of the respective pure liquid vapor pressure (V_p) and an activity coefficient, which accounts for the non-ideal nature of the solution. Thus, if insufficient y_0 data is available for a specific SVOC, other similar SVOCs with available data could be used to establish the relationship between y_0, C_0, and V_p (i.e., obtain the corresponding activity coefficient), and then y_0 can be estimated based on the resulting activity coefficient and V_p for the specific compound. However, even though some V_p data for certain well-researched SVOCs are available in the literature, there are significant differences in V_p for SVOCs (especially for SVOCs with very low volatility) between different measurements and estimation results (Liang and Xu, 2014). More reliable vapor pressure measurements are clearly needed.

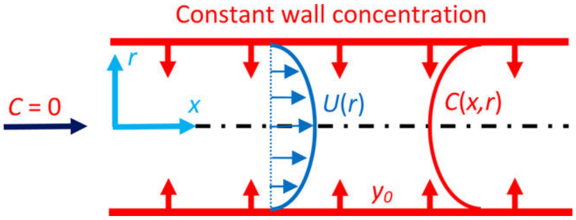

Figure 7: Laminar forced convection mass transfer of SVOC in the tube for measuring V_p.

We developed a new approach to determine V_p of SVOCs by using a tube chamber coated with a thin layer of pure SVOC liquid on the inner surface (similar to the apparatus used for measuring K_p, as introduced above) (Wu et al., 2016a). After emission from the surface of the cylindrical tube wall, the forced convection mass transfer of SVOCs within the tube flow can be modeled using a laminar flow mass transfer model (Figure 7). V_p of the analytes can be calculated by fitting the model to the SVOC concentration measured at the tube outlet. Using n-hexadecane as reference compound, the measured V_p at temperature of 298 K is determined as 0.16 Pa, which is in good agreement with the literature value (0.19 Pa), giving confidence in the accuracy of our method. We have also measured V_p values for ten different phthalates and alternate plasticizers. Duplicate measurements of V_p were made for each compound, and the relative standard deviation (RSD) of the replicate samples ranged from 0.1% to 20%, implying that the method is stable.

3 ESTIMATING EXPOSURE TO SVOCS PRESENT IN A SINGLE PRODUCT

For prioritization purposes, a general estimate of total exposure potential is sufficient. However, our approach provides further insight into significant exposure pathways based on product characteristics (Little et al., 2012). In addition to inhaling SVOCs in indoor air, exposure via inhalation of airborne particles, ingestion of dust, and dermal absorption may play significant roles, contributing to total human intake rates. For SVOCs present as additives, the following equation (2) can be used to estimate the gas-phase concentration at steady state (derived from equation (1)), with emissions considered to persist for the entire time during which the source is present in the indoor environment.

$$y = \frac{h \times y_0 \times A}{h \times A + Q^*}, \text{ with } Q^* = \left(1 + K_p \times TSP\right) \times Q \tag{2}$$

For SVOCs that are sprayed or applied, equation (3) can be used to estimate y at steady state, with concentrations that persist for the duration given by equation (4).

$$y = \frac{h \times y_0 \times A}{h \times A + h_s \times A_s + Q^*} \tag{3}$$

$$\tau \sim \frac{M_0}{y \times Q^*} = \frac{\left(h \times A + h_s \times A_s + Q^*\right) \times M_0}{h \times y_0 \times A \times Q^*} \tag{4}$$

where M_0 is the initial SVOC mass applied, and τ is the duration.

We next illustrate the approach for exposure analysis using three “additives” (DEHP, DnBP, and BDE-47) and one “sprayed or applied” SVOC (chlorpyrifos) as examples. A recent study suggests that direct transport of SVOCs from bulk air through skin to dermal capillaries can sometimes be important and, if so, can be modeled considering three mass-transport resistances in series: the resistance from bulk air to the skin-surface lipids, the resistance through the stratum corneum, and the resistance through the viable epidermis. Therefore, dermal exposure from the gas phase is estimated using an “overall permeability”, k_{p_g}, based on permeabilities through the boundary layer adjacent to skin and through the stratum corneum/viable epidermis composite to dermal capillaries (Weschler and Nazaroff, 2012). Exposure via inhalation, oral ingestion of dust, and dermal absorption from the gas phase for a 3-year old child are calculated. Table 1 summarizes the calculated results, with detailed parameters used for calculation and their determination methods provided in Little et al. (2012).

Table 3: Estimated exposure ((µg/kg)/d) to DEHP, DnBP, BDE-47, and chlorpyrifos via indoor air and intake of particles/dust for a 3-year old child.

Exposure pathway	DEHP	DnBP	BDE-47	Chlorpyrifos	Equation
Inhalation (air)	0.013	0.34	1.4×10^{-4}	0.32	$y \times InhR \times ED$
Inhalation (particles)	0.82	0.02	3.5×10^{-5}	0.01	$y \times K_p \times TSP \times InhR \times ED$
Ingestion (dust)	75	1.6	3.2×10^{-3}	0.59	$y \times K_{dust} \times IngR$
Dermal absorption (from air)	0.12	2.7	1.0×10^{-5}	0.23	$(y \times k_{p\_g} \times SA \times f_{SA} \times ED)/BW$
Total	76	4.6	3.5×10^{-3}	1.15	

The primary difference between exposure to DEHP and exposure to DnBP (76 and 4.6 (µg/kg)/d, respectively; Table 1) is the volatility of the compounds, with DnBP having a vapor pressure that is two orders of magnitude higher than DEHP. The result is that for DEHP, 99% of the exposure occurs through ingestion of dust (due to its low volatility DEHP partitions strongly onto dust and airborne particles), while for DnBP, 55% of the exposure occurs via dermal absorption with 35% via ingestion of dust. In the case of BDE-47, the surface area of the source is somewhat lower (15 m^2, compared to 20 m^2 of DEHP and DnBP), but y_0 (0.0005 $µg/m^3$, compared to 1 $µg/m^3$ of DEHP and DnBP) is considerably lower than for either DEHP or DnBP. The lower y_0 explains the substantially lower exposure (0.0035 (µg/kg)/d), but the fact that the volatility of BDE-47 is relatively low (falling between DEHP and DnBP) explains why 90% of the exposure also occurs through ingestion of dust. For chlorpyrifos, the high y_0 (350 $µg/m^3$) means it is a much stronger source than the others, but the source area is far smaller (0.75 m^2). The two effects tend to compensate, with an overall exposure of 1.15 (µg/kg)/d, which is distributed almost equally among inhalation (23%), ingestion of dust (44%), and dermal absorption (31%).

4 CONCLUSIONS

There is an urgent need to estimate exposure to SVOCs, but the vast number of SVOC/product combinations poses a serious challenge. To solve this problem, we propose a simple and rapid method to estimate exposure, in which we characterize a specific indoor product and then estimate exposure, which can be used to rapidly characterize specific SVOC/product combinations, and estimate their contributions to exposure. We have also identified several parameters that strongly affect the exposure estimates, including the gas-phase concentration in equilibrium with the product surface (y_0), the partition coefficient between air and indoor surfaces (K_s), the partition coefficient between air and airborne particles (K_p), and the vapor pressure of liquid SVOCs (V_p) that strongly influences y_0 values. However, to date, reliable values of these key parameters are scarce and existing methods for measuring them are time-consuming and inaccurate. To close this gap, we have developed several simple and accurate methods to measure them, which have been tested by our laboratory measurements (mainly using phthalates as the example). These methods should be helpful for accurately assessing potential exposure to indoor SVOCs, as well as for exposure-based prioritization of indoor SVOCs. However, more measurements for more SVOC/product combinations are required in the future. When combined with the rapid estimates of toxicity being developed in ToxCast™, our rapid estimates of exposure will allow risk-based prioritization of a wide range of SVOCs. This information can then be used to either remove specific products from the market, or make substitutions, introducing alternate low toxicity SVOCs that impart similarly desirable product performance at equivalent cost.

5 ACKNOWLEDGEMENTS

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