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Article

Transfer of Chlorinated Organophosphate Esters from Furniture Fabric to Indoor Dust via Direct Contact

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Abstract: Chlorinated organophosphate esters (Cl-OPEs)—specifically tris(2-chloroethyl) phosphate (TCEP), tris(2-chloroisopropyl) phosphate (TCIPP), and tris(1,3-dichloroisopropyl) phosphate (TDCIPP—are added to the foam fillings of furniture in some countries to meet fire safety regulations. Recent reports of the capacity of Cl-OPEs to elicit adverse effects on the health of humans and wildlife have focused attention on their potential emission from furniture and consequent human exposure. Given studies demonstrating substantial human exposure via the ingestion of house dust, and that direct source: dust contact is a highly effective mechanism via which brominated flame retardants are emitted from source materials into dust; this study examines the transfer of TCEP, TCIPP, and TDCIPP from furniture fabric into dust in direct contact with the fabric. To do so, we exposed indoor dust to a furniture fabric covering in a sealed stainless steel emission chamber held at room temperature and recorded Cl-OPE concentrations in both dust and fabric at 0, 1, 2, 4, 7, and 10 days. Consistent with the intentional application of TCIPP to the furniture foam that our test fabric was used to cover, the concentration of TCIPP in the fabric exceeded that of TCEP and TDCIPP by ~2 orders of magnitude. Results show a significant decline ($p < 0.05$) in concentrations of TCIPP in the fabric from 44.6 ± 2.84 mg/g at day 1 to 22.7 ± 6.0 mg/g at day 10. This contrasted to a significant ($p < 0.05$) increase in concentrations of TCIPP in dust from 86.4 ± 23.2 µg/g at day 1 to 195 ± 10.8 µg/g at day 10. Combined, these data strongly suggest that direct dust:fabric contact is an important pathway via which Cl-OPEs contaminate indoor dust.

Keywords: indoor dust; flame retardants; TCIPP; furniture fabric; source-to-dust transfer

1. Introduction

In the United Kingdom, domestic and office furniture is required to comply with the Furniture and Furnishings (Fire Safety) Regulations 1988 (FFRs). While not essential to meet the requirements of the FFRs, evidence both from Ireland (where furniture fire safety is governed by similar regulations to the UK FFRs) and the UK, demonstrates that chlorinated organophosphate esters (Cl-OPEs) have been widely applied to furniture to achieve regulatory compliance [1,2]. Although Cl-OPEs are added to furniture foam fillings, likely because of chemical migration to fabric covers that are in direct contact with treated foam fillings, substantial concentrations of Cl-OPEs have also been detected in furniture fabric coverings [1]. This is of concern, given evidence that Cl-OPEs in furniture fabric can transfer across the human skin barrier following dermal contact [3], and that Cl-OPEs like tris(2-chloroethyl) phosphate (TCEP), tris(2-chloroisopropyl) phosphate (TCIPP), and tris(1,3-dichloroisopropyl) phosphate (TDCIPP)

can elicit human health impacts including carcinogenicity, as well as developmental, immune, neurological, and reproductive toxicity [4–8].

In addition to direct human exposure via skin contact with furniture fabrics containing Cl-OPEs, research also reports that exposure to these chemicals also occurs via inhalation of indoor air [9], as well as ingestion of indoor dust [10] and food [11]. Moreover, this demonstrable presence of Cl-OPEs in indoor air and dust, is clear evidence of their facile emission from indoor sources. Research into the mechanisms via which related chemicals, such as phthalate diester plasticisers and brominated flame retardants (BFRs) transfer from products within which they are incorporated into indoor dust suggests three principal processes are involved. In summary, these comprise: (a) volatilisation followed by sorption to dust; (b) abrasion of product fibres/particles that are deposited to dust; and (c) transfer via direct product-dust contact [12,13]. While similar evidence does not, to our knowledge, exist for Cl-OPEs, we hypothesise that similar mechanisms govern their transfer from furniture fabrics into indoor dust. Moreover, given a previous report of substantial transfer of HBCDD from a furniture fabric to indoor dust via direct fabric:dust contact, we further hypothesise that over time, similar transfer will result in reduced Cl-OPE concentrations in furniture fabric. While Cl-OPEs are added intentionally to furniture foam rather than fabric coverings, this is important as substantial transfer from fabric coverings may result in older furniture items failing to meet fire safety regulations.

Against this backdrop, this study examines the transfer of Cl-OPEs from furniture fabric into indoor dust in direct contact with the fabric, quantifying its impact on concentrations of Cl-OPEs in both the fabric and indoor dust.

2. Experimental

2.1. Measurement of Cl-OPEs in Dust and Fabric

The native compounds (TCEP, TDCIPP, and TCIPP), $TnBPd_{27}$ and $TPhP_{d15}$ used as internal standards and D_{12}-benz[*a*]anthracene employed as recovery determination (syringe) standards were purchased from Wellington Laboratories (Guelph, ON, Canada) as stock solutions in toluene at 1 mg/mL. HPLC grade acetone and hexane were supplied by Fisher Scientific UK Ltd. (Leicestershire, UK), while ethyl acetate, iso-octane, florisil, and glass wool were supplied by Sigma Aldrich (St. Louis, MI, USA). Nitrogen used for solvent evaporation was oxygen free and supplied by BOC Gases (London, UK).

2.2. Dust Sample Preparation, Extraction and Clean Up

The dust samples were homogenised by sieving through 500 µm mesh aluminium sieve and removing undesirable fibres using acetone rinsed tweezers. After sieving, the dust was stored in glass jars with aluminium foil lined lids and stored at 4 °C until extraction. The extraction method used followed that developed in our laboratory [10]. An aliquot of dust (50 mg) was spiked with 150 µL internal (or surrogate) standard solution (containing 100 ng each of $TnBP_{d27}$ and $TPHP_{d15}$). Samples were extracted with 2 mL hexane:acetone (3:1 *v*/*v*) via vortexing for 1 min, followed by ultrasonication for 5 min (2 cycles). Between cycles, the dust samples were centrifuged at 2000 rpm for 2 min and the supernatants collected in a clean glass tube. The combined extracts were carefully concentrated under a gentle N_2 stream to incipient dryness and reconstituted in 1 mL hexane. This concentrated extract was then added (with two × 1 mL hexane rinses) to a pasteur pipette containing 1 g Florisil that, before use, was prewashed using 8 mL of methanol followed by 4 mL of hexane. Following addition of the sample extract, the column was eluted with 8 mL of hexane (discarded) to remove PBDEs. OPEs were then eluted with 10 mL ethyl acetate and the eluate evaporated to incipient dryness before resolubilisation with 100 µL of iso-octane containing 100 ng of D_{12}-benz[*a*]anthracene as recovery determination standard, ready for injection into the GC-MS.

2.3. Fabric Extraction and Clean Up

Fabric samples were extracted in accordance with a previously reported soaking extraction procedure [14]. Approximately 0.2 g of fabric was placed in 20 mL of toluene in a glass bottle with a lid then vortexed for 2 min, followed by storage in the dark at room temperature for 2 days. After 2 days, an aliquot (10 µL) was removed, spiked with 1 µg internal (surrogate) standards and diluted to 10 mL with toluene, before a 2 µL aliquot of the diluted extract was injected onto the GC-MS and analysed using the procedures outlined below. Textile samples were analysed in triplicate.

2.4. GC/MS Analysis

Analysis was conducted on an Agilent 5975 GC/MS fitted with a 30 m DB-5 MS column (0.25 mm id, 0.25 μm film thickness). The carrier gas was helium with a constant flow rate of 1.0 mL/min. Mass spectrometer temperatures used were: injector 290 °C under splitless conditions and a solvent delay of 3.8 min. The ion source, quadrupole and interface temperatures were 230 °C, 150 °C and 300 °C respectively. The GC temperature programme was: 100 °C, held for 1.25 min, ramp 10 °C/min to 240 °C, ramp 20 °C/min to 310 °C, and held for 5 min, equating to a total run time of 23.75 min. The MS was operated in electron ionisation (EI) selected ion monitoring (SIM) mode. The ions monitored for quantification (Q) and identification (I) purposes were: 249 (Q) and 251 (I); 277 (Q) and 279 (I); 381 (Q) and 379 (I); 103 (Q) and 167 (I); and 341 (Q) and 349 (I) for TCEP, TCIPP, TDCIPP, d_{27}-TNBP, and d_{15}-TPHP respectively. Ion 240 was used for measurement of the recovery determination standard D_{12}-benz[*a*]anthracene. D_{27}-TNBP was used to quantify TCIPP and TCEP while TDCIPP was quantified using D_{15}-TPhP.

2.5. QA/QC

Recoveries of the internal (surrogate) standards were: (average = 91%; range = 42–135%) for D_{27}-TNBP and (average = 92%; range = 44–137%) for D_{15}-TPHP. During method validation, 5 replicate aliquots of NIST SRM2585 (organics in indoor dust) were analysed, with 1 further aliquot analysed every 20 samples. In the absence of certified or indicative concentrations for our target OPEs, we compared our data with previous reports for this SRM as shown in Table 1. This comparison reveals our method to be precise (relative standard deviations between 3 and 15% for individual OPEs) and to agree well with previous studies [15–17].

Table 1. Cl-OPE concentrations (μg/g) in dust SRM2585 compared with literature data.

	TCEP	TCIPP	TDCIPP	Reference
Mean	1.07	1.06	2.02	This study
STDEV [a]	0.12	0.11	0.15	
Mean	0.70	0.82	2.00	Van den Eede et al., 2011 [15]
STDEV	0.17	0.10	0.26	
Mean	0.84	0.88	2.30	Bergh et al., 2012 [16]
STDEV	0.06	0.14	0.28	
Mean	0.81	0.75	2.50	Brandsma et al., 2014 [17]
STDEV	0.04	0.02	0.01	

[a] standard deviation.

2.6. Analysis of Blanks, LOD and LOQ

To further assess the quality of the method, every 6th sample was a reagent blank, which consisted of 50 mg pre-baked Na_2SO_4 extracted and cleaned as a sample. For dust, field blanks were also analysed, consisting of pre-baked Na_2SO_4 vacuumed from the surface of aluminium foil into a sampling sock and thereafter treated as a dust sample. In all field and reagent blanks, analyte concentrations were <5% of those present in samples and thus no blank correction was necessary. Limits of quantification for TCEP were 0.06 and 1.5 μg/g in dust and fabrics respectively, with values for TCIPP 0.044 and 1.1 μg/g, and for TDCIPP 0.03 and 0.75 μg/g respectively.

2.7 Chamber Experiments

Our experiments were conducted using the same stainless steel bespoke emission chambers as used to examine transfer of HBCDDs from a curtain fabric into indoor dust via direct fabric: dust contact [18]. Figure 1 depicts the experimental configuration employed. The fabric used in this study was a wool covering taken from our research group sample archive where it had been stored wrapped tightly in aluminium foil in a cold store (4 °C) since collection in 2011 as part of a study measuring chemical flame retardants (including TCEP, TCIPP, and TDCIPP) in discarded UK soft furnishings [2]. The indoor dust sample was a mixture of five living room dust samples collected by one of the authors from Ciudad Victoria, Mexico, between December 2013 and January 2014. We used this composite dust sample as our in-house analyses revealed it to contain concentrations of TCEP, TCIPP, and TDCIPP much lower than those reported in the UK [10]. Concentrations of Cl-OPEs were measured in triplicate in the fabric and in quintuplicate in the dust sample before use in our experiments and are reported in Table 2. Concentrations of TCIPP in the fabric exceed by 2 orders of magnitude those of TCEP and TDCIPP, strongly implying that TCIPP was intentionally added to the foam filling of the chair from which the fabric was taken.

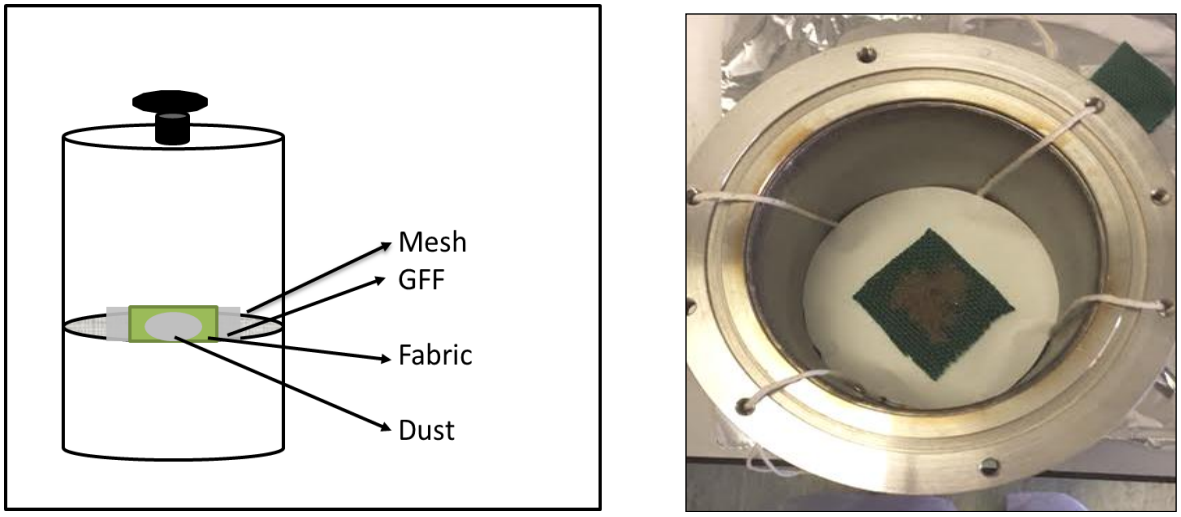

Figure 1. Configuration (**L**) and photo (**R**) of the chamber experiments studying source-to-dust transfer of Cl-OPEs.

Table 2. Initial concentrations of target Cl-OPEs in fabric and dust used in this study.

Analysis #	TCEP	TCIPP	TDCIPP
Dust (µg/g) (n = 5)			
1	1.7	7.6	5.3
2	1.9	8.0	5.3
3	2.0	6.1	4.7
4	1.9	6.2	4.8
5	2.1	6.4	5.2
Average	1.9	6.9	5.1
SD	0.15	0.88	0.29
Fabric (mg/g) (n = 3)			
1	0.12	52	0.41
2	0.19	49	0.31
3	0.19	52	0.32
Average	0.17	51	0.35
SD	0.04	1.8	0.02

Transfer of Cl-OPEs from the fabric into the overlying dust was examined in triplicate by using three separate identically dimensioned test chambers. In each chamber, a known amount of dust containing known concentrations of target Cl-OPEs was placed on the surface of the fabric. Aliquots of the dust were removed at various time points and analysed for their Cl-OPE content. To avoid volatilisation and atmospheric inputs during the experiment, the chambers were sealed to the air. Chamber temperature was maintained at 22 ± 1 °C throughout.

In each test chamber, 5 × 5 cm square of the test wool fabric was weighed accurately and placed on a clean filter paper (Whatman, UK) into the chamber with a known mass of dust (~0.3 g, accurately weighed) spread as gently and evenly as possible over the fabric surface, before the chamber was sealed [18]. Five different contact times were studied, 1, 2, 4, 7 and 10 days. Each experimental duration was studied in triplicate (i.e., in 3 separate test chambers), with fresh aliquots of fabric and dust used in each chamber and for each experimental duration examined. At the end of each experiment, the dust was collected very carefully with a soft brush avoiding the removal of fabric fibres with the dust. After removal of our dust sample and homogenisation, two subsamples were weighed accurately, extracted and analysed for Cl-OPE content. In addition, duplicate sub-samples of the residual fabric from each test chamber were analysed.

3. Results and Discussion

3.1. Influence of Fabric:Dust Contact Time on Concentrations of Cl-OPEs in Fabric

Table 3 reports the concentrations of our target Cl-OPEs detected in the duplicate sub-samples of fabric taken from each of the three test chamber experiments for each fabric:dust contact time examined. A summary of these concentrations is depicted in Figure 2. There is an evident steady reduction in concentrations of all three target Cl-OPEs as fabric:dust contact time increases. Statistical analysis via ANOVA with a post-hoc Tukey test revealed

this reduction to be statistically significant ($p < 0.05$). Of particular note, after 10 days of fabric:dust contact, the concentration of TCIPP has fallen to 22.7 ± 6.0 mg/g from the day 1 concentration of 44.6 ± 2.84 mg/g.

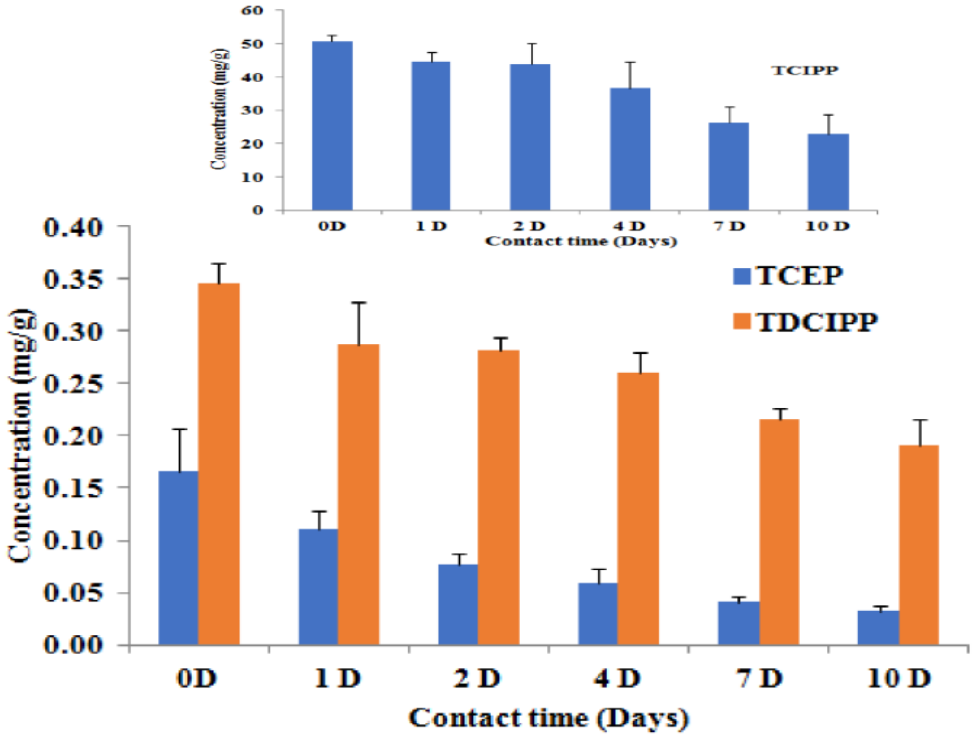

Figure 2. Average concentrations (mg/g) of Cl-OPEs in fabric over contact time (y-error bars denotes 1 standard deviation).

Table 3. Concentrations (mg/g) of target Cl-OPEs in fabric after different fabric:dust contact times.

Fabric Contact Time	TCEP	TCIPP	TDCIPP
1 day contact			
1A [a]	0.11	47.2	0.29
1B	0.09	48.3	0.29
2A	0.09	45.1	0.29
2B	0.12	44.0	0.29
3A	0.111	41.3	0.29
3B	0.14	41.7	0.28
Average	0.11	44.6	0.29
SD	0.02	2.84	0.004
2 days contact			
1A	0.070	47.7	0.28
1B	0.067	47.2	0.26
2A	0.076	43.6	0.28
2B	0.070	51.3	0.29
3B	0.080	33.9	0.30
3C	0.098	39.5	0.28
Average	0.08	43.9	0.28
SD	0.01	6.3	0.01
4 days contact			
1A	0.045	41.7	0.27
1B	0.051	28.6	0.29
2A	0.042	29.0	0.23
2B	0.063	45.0	0.25
3A	0.073	45.0	0.26
3B	0.075	29.9	0.27
Average	0.06	36.7	0.26
SD	0.01	8.00	0.02

Table 3. *Cont.*

Fabric Contact Time	TCEP	TCIPP	TDCIPP
7 days contact			
1A	0.038	23.2	0.21
1B	0.036	30.0	0.22
2A	0.039	29.3	0.22
2B	0.046	17.7	0.22
3A	0.041	27.6	0.22
3B	0.049	29.8	0.22
Average	0.04	26.3	0.22
SD	0.01	4.92	0.01
10 days contact			
1A	0.024	15.0	0.18
1B	0.027	18.3	0.18
2A	0.031	19.1	0.18
2B	0.035	27.4	0.21
3A	0.039	27.3	0.20
3B	0.035	29.3	0.20
Average	0.03	22.7	0.19
SD	0.01	5.99	0.01

[a] A and B denote duplicate analyses of the fabric sample taken from a given test chamber at the same contact time.

3.2. Influence of Fabric:Dust Contact Time on Cl-OPE Concentrations in Dust

Table 4 and Figure 3report concentrations of Cl-OPEs detected in dust samples collected from each test chamber at each fabric:dust contact time. There is a clear increase in Cl-OPE concentrations with increasing contact time, that ANOVA with a Tukey post-hoc test showed to be statistically significant ($p < 0.05$). Coupled with the corresponding decrease in Cl-OPE concentrations in the fabric sample, this suggests strongly that Cl-OPEs undergo substantial fabric:dust transfer during the course of our chamber experiments. Consistent with the Cl-OPE pattern in the fabric, concentrations in all dust samples collected from our chamber experiments were in the order: TCIPP > TDCIPP > TCEP. However, the TCIPP:TDCIPP:TCEP ratio in the dust samples (47.6:2.9:1 after 10 days contact) differed markedly from that observed in the fabric (757:6.3:1 after 10 days contact). This not only provides strong evidence that the elevated concentrations in dust in these experiments have not arisen via transfer to dust of abraded fabric fibres but clearly suggests that the efficiency of fabric-to-dust transfer follows the order: TCEP > TDCIPP > TCIPP. As the order of vapour pressures of these Cl-OPEs is: TCEP ≥ TCIPP >> TDCIPP [19], this does not suggest that uptake of Cl-OPEs by dust occurs solely via contact with the boundary layer of the source:air interface [20]. Likewise, our data is inconsistent with Cl-OPE transfer occurring solely in accordance with the hypothesis that a dust layer disrupts the boundary layer surrounding the source (fabric), allowing direct uptake to dust particles of a chemical from the source surface [12].

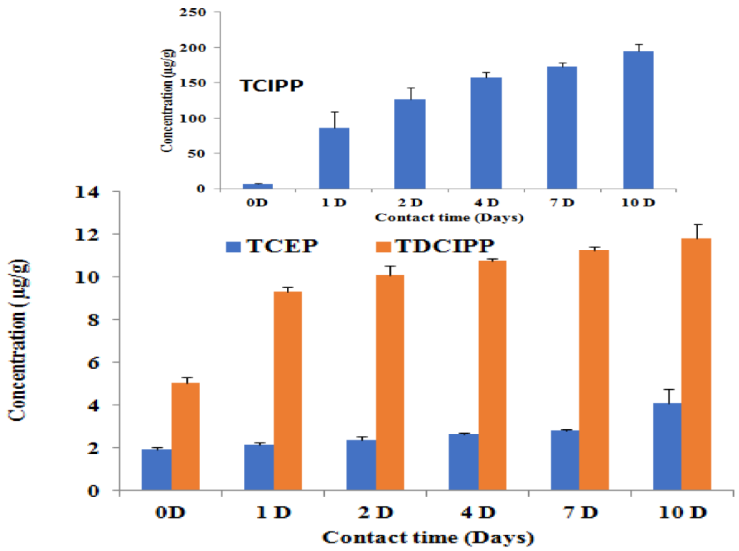

Figure 3. Average concentrations (µg/g) of CL-OPEs in dust over contact time.

Table 4. Variation of concentrations (µg/g) of Cl-OPEs in dust with fabric:dust contact time.

Fabric Contact Time	TCEP	TCIPP	TDCIPP
1 day contact			
1A	2.02	57.5	9.02
1B	2.06	65.6	9.19
2A	2.21	74.2	9.28
2B	2.21	105	9.41
3A	2.24	108	9.30
3B	2.24	108	9.74
Average	2.16	86.4	9.32
SD	0.10	23.2	0.24
2 days contact			
1A	2.24	111	9.59
1B	2.20	112	9.97
2A	2.30	114	9.98
2B	2.48	129	10.01
3B	2.59	144	10.54
3C	2.59	149	10.66
Average	2.40	127	10.12
SD	0.17	17.1	0.40
4 days contact			
1A	2.59	146	10.60
1B	2.69	154	10.88
2A	2.72	153	10.87
2B	2.70	161	10.87
3A	2.70	162	10.58
3B	2.73	168	10.78
Average	2.69	157	10.76
SD	0.05	7.66	0.14
7 days contact			
1A	2.72	174	11.08
1B	2.75	171	11.18
2A	2.76	180	11.20
2B	2.88	177	11.42
3A	2.85	172	11.50
3B	2.87	167	11.07
Average	2.80	173	11.24
SD	0.07	4.49	0.18
10 days contact			
1A	3.59	207	11.49
1B	3.86	209	12.54
2A	3.39	191	12.72
2B	3.98	188	11.04
3A	4.83	184	11.76
3B	4.96	188	11.33
Average	4.10	195	11.81
SD	0.65	10.79	0.68

The average concentration of TCIPP in dust increases from 86.4 µg/g on day 1 up to 195 µg/g after 10 days of contact. Similar observations were made for TCEP and TDCIPP, where after 1 day contact with fabric, the average concentrations in dust were 2.16 µg/g and 9.32 µg/g respectively, and after 10 days contact were 4.10 µg/g and 11.81 µg/g respectively. While transfer to dust occurs in the early stages of contact, it continues to increase (most markedly for TCIPP due to its far greater starting concentration in the fabric) throughout the experiments, suggesting that fabric:dust equilibrium takes longer than 10 days to be attained.

These observed increases in Cl-OPE concentrations in dust have concomitant implications for human exposure via dust ingestion. Based on our experimental data for example, and assuming that 25% of the typical mass of dust ingested by an adult (20 mg/day) was contaminated with TCIPP at the concentrations reached after 10 days fabric:dust contact; exposure via ingestion of dust present on furniture fabrics alone would be 975 ng/day. This figure exceeds the high-end exposure scenario dose for UK adults of 910 ng/day [10].

Statistical analysis via ANOVA with a post-hoc Tukey test reveals concentrations of Cl-OPEs of interest in the analysed dust all significantly ($p < 0.05$) increase with increasing contact time. The transfer seen in our experiments is likely due to the Cl-OPE concentration gradients between the fabric and the dust, which may be expressed in terms of the fugacity of the compounds [18]. The fugacity potential of a chemical can be defined as the escape potential of that chemical in a given phase (air, water, solid, dust, fabric). When the fugacities of the chemical in two phases are equal the chemical is in equilibrium between the two phases and no net chemical transfer between the phases (e.g., fabric and dust) occurs; however, when chemical fugacities in the phases are different there is net transfer of the chemical from the phase in which its fugacity (concentration) is higher into the other phase. This net transfer will continue until equilibrium is reached—i.e., fugacities of the chemical in the two phases are equal [18]. The exact mechanisms governing the migration of SVOCs from source to dust via direct contact are not completely understood; however, it has been suggested that transfer occurs as a result of contact between dust and gas phase FRs present in the boundary layer directly above the source (e.g., fabric) [20]. According to this theory, compounds with low vapour pressures will be less abundant in this layer and thus are less efficiently transferred. Alternatively, it has been suggested that source-dust transfer may occur as a consequence of direct contact between the source and dust particles that replace the boundary layer [12]. In such a scenario, the influence of vapour pressure on the efficiency of transfer is negligible. Unfortunately, our experiments could not provide insights into which of these mechanisms govern the Cl-OPE transfer from the fabric to dust, owing to the very different concentrations of the 3 target Cl-OPEs in the fabric. Experiments to generate evidence to better understand the mechanisms of source-dust transfer of Cl-OPEs and related compounds are thus a research priority. Such experiments could include use of fabrics spiked with similarly elevated concentrations of several halogenated flame retardants covering a wide range of vapour pressures. Such experimental approaches should be augmented with mathematical modelling.

4. Dedication

This work is dedicated to the memory of Dr Yessica Ortiz. The work reported here is taken from the PhD thesis of Yessica Carrizales Ortiz. Ortiz, Yessica "Factors influencing human exposure assessment of organophosphorus flame retardants (OPFRs) via indoor dust ingestion" PhD thesis, University of Birmingham, 2020. https://etheses.bham.ac.uk//id/eprint/8071/ (accessed on 22 May 2025).

Author Contributions

Y.O.: Writing—original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. S.H.**:** Writing—review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Data Availability Statement

Data are available on request.

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Conflicts of Interest

The authors declare no conflict of interest.

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