Supporting Information

Beyond Phthalate Di-esters: Existence of Phthalate Mono-esters in South China House Dust and Implications for Human Exposure

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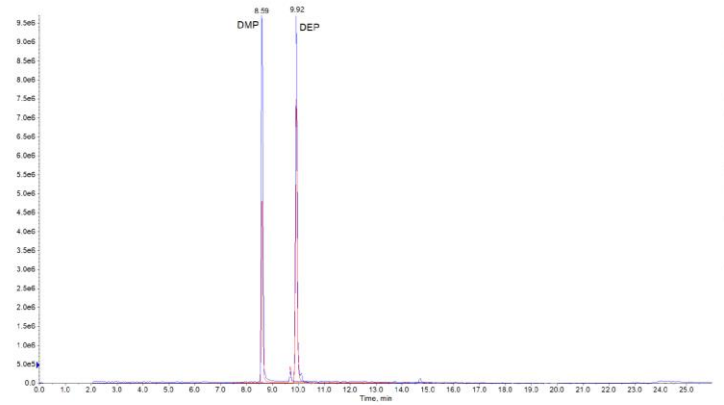
Details of instrument analysis

Determination of phthalate diesters (di-PAEs, except for DEHP) and phthalate monoesters (mono-PAEs) in dust was conducted on an Agilent HPLC coupled to an AB Sciex 3200 triple quadrupole mass spectrometry (MS/MS, Toronto, Canada). Determination of mono-PAEs in urine was conducted on a Shimadzu UPLC coupled to an AB Sciex 5500 Q Trap MS/MS (Toronto, Canada).

Chromatographic separation was achieved by using a Luna® 2.5 μm C18(2) 100 Å column (100 mm × 2.1 mm, 2.5 μm particle size; Phenomenex, Torrance, CA, U.S.). The mobile phase consisted of water (A) and methanol (B), both spiked with 0.02 mM ammonium acetate for mono-PAEs or 0.1% formic acid for di-PAEs. A flow rate of 200 μL/min was used. The gradient for di-PAEs was programmed as: 0-2 min, 5% B; 2-3 min, 5% ramped to 60% B (linear); followed by a linear increase to 100% B in 20 min (held for 5 min) and then a change to 5% B in 1 min (held for 5 min). The gradient for mono-PAEs was programmed as: 0-3 min, 10% B; 3-4 min, 10% ramped to 60% B (linear); followed by a linear increase to 100% B in 11 min (held for 5 min) and then a change to 5% B in 1 min (held for 4 min). The MS was equipped with a TurboIonSpray® electrospray ionization (ESI) probe operated in the multiple reaction monitoring (MRM) mode (Table S2).

Determination of DEHP was conducted on a 7890B gas chromatograph interfaced with a 5977A mass spectrometer in electron impact (EI) mode (GC-MS, Agilent, USA). The GC was equipped with a 30-m HP-5MS column (0.25 mm i.d., 0.25 μm, J&W Scientific, Agilent, USA). The GC injector was operated in spitless mode and held at 260 °C. Initial oven temperature was held at 80 °C for 3 min, ramped to 150 °C at 10 °C/min, and finally increased to 300 °C at 5 °C/min (held for 10 min). The heated transfer line temperature and quadrupole temperature were set as 280 °C and 150 °C, respectively. The monitored m/z ions for DEHP and deuterated DEHP-d4 were 149/167 and 153/171, respectively.

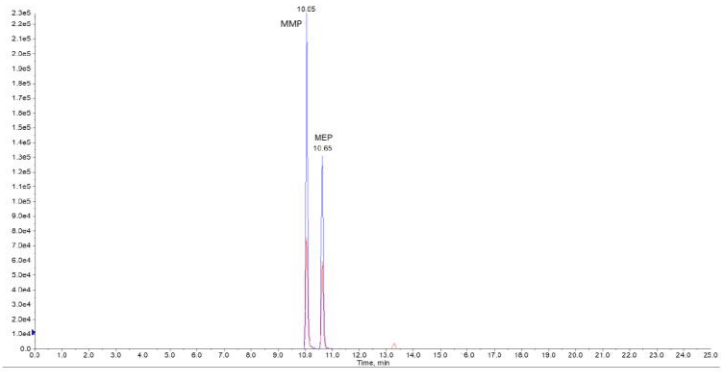


Instrument: AB Sciex 5500 Q Trap MS/MS

Column: Luna® 2.5 µm C18(2) 100 Å column (100 mm × 2.1 mm, 2.5 µm particle size; Phenomenex, Torrance, CA, U.S.)

Mobile Phase: A: 0.2 mM ammonium formate (aq); B: 0.2 mM ammonium formate (methanol)

Gradient Details: 0-3 min, 10% B; 3-4 min, 10% ramped to 60% B (linear); followed by a linear increase to 100% B in 11 min (held for 5 min) and then a change to 10% B in 1 min (held for 4 min).



Diester	RT (min)	Monoester	RT (min)
DMP	8.59	MMP	10.05
DEP	9.92	MEP	10.63
DiPrP	11.63	MiPrP	11.24
DiBP	13.72	MiBP	12.44
DBP	13.92	MBP	12.60
BBzP	13.78	MBzP	12.69
DHxP	16.71	MHxP	14.85
DiHeP	17.44	MiHeP	15.45
DiNoP	18.85	MiNP	16.87
DPeP	15.51	MPeP	13.71
DCHP	15.67	MCHP	13.41
DEHP	18.23	MEHP	16.42
DEHP	18.23	MECPP	12.38
DEHP	18.23	MEOHP	12.11
DEHP	18.23	MEHHP	12.59

Figure S1 Extract ion chromatography of DMP, DEP, MMP and MEP in the multiple reaction monitoring mode; retention time (RT) information of all target phthalate diester and monoester pairs under the same chromatographic conditions

Table S1. Physicochemical properties of phthalate diesters and monoesters.

Abbreviation	Full name	CAS	Molecular weight	Biological half-lives (h)[a]	*Log* K_{oa}[b]	*Log* K_{ow}[c]
Phthalate diesters						
DMP	Dimethyl phthalate	131-11-3	194	0.3	6.7	1.7
DEP	Diethyl phthalate	84-66-2	222	0.5	7.0	2.7
DiPrP	Diisopropyl phthalate	605-45-8	250	0.6	7.4	3.5
DPrP	Dinpropyl phthalate	131-16-8	250	0.8	8.1	3.6
DAlP	Diallyl phthalate	131-17-9	246	0.9	8.0	3.4
DiBP	Diisobutyl phthalate	84-69-5	278	1.4	8.4	4.5
DBP	Dibutyl phthalate	84-74-2	278	2.2	8.6	4.6
DPeP	Dipentyl phthalate	131-18-0	306	4.5	9.7	5.6
DiPeP	Diisopentyl phthalate	605-50-5	306	3.5	9.5	5.5
iBCHP	isobutylcyclohexyl phthalate	5334-09-8	304	4.3	9.7	5.3
DiHxP	Diisohexyl phthalate	68515-50-4	334	9.4	10.3	6.5
DHxP	Dihexyl phthalate	84-75-3	334	10.0	9.8	6.6
DCHP	Dicyclohexyl Phthalate	84-61-7	330	10.2	11.6	6.2
DPP	Diphenyl phthalate	84-62-8	318	1.0	10.0	4.1
DBzP	Dibenzyl phthalate	523-31-9	346	0.4	12.3	5.1
DiHeP	Diisoheptyl phthalate	71888-89-6	363	12.6	11.0	7.4
DHeP	Diheptyl phthalate	3648-21-3	363	15.9	11.1	7.6
DEHP	Di-2-ethylhexyl phthalate	117-81-7	391	15.9	12.6	8.4
DiNP	Diisononyl phthalate	68515-48-0	419	45.4	13.6	9.4
DNP	Dinonyl phthalate	84-76-4	419	57.4	12.6	9.5
DiDP	Diisodecyl phthalate	26761-40-0	447	85.9	14.7	10.4
DUP	Diundecyl phthalate	3648-20-2	475	206.4	14.1	11.5

BBzP	Butyl benzyl phthalate	85-68-7	312	0.8	9.0	4.8
Phthalate monoesters						
MMP	Monomethyl phthalate	4376-18-5	180	0.7	8.7	1.4
MEP	Monoethyl phthalate	2306-33-4	194	1.2	9.3	1.9
MiPrP	Monoisopropyl phthalate	35118-50-4	208	1.5	9.6	2.3
MiBP	Monoisobutyl phthalate	30833-53-5	222	2.1	9.9	2.8
MBP	Monobutyl phthalate	131-70-4	222	2.4	10.0	2.8
MPeP	Monopentyl phthalate	24539-56-8	236	3.3	10.4	3.3
MHxP	Monohexyl phthalate	24539-57-9	249	4.5	10.7	3.8
MCHP	Monocyclohexyl phthalate	7517-36-4	248	7.2	11.3	3.7
MiHeP	Mono-2-heptyl phthalate	129171-03-5	264	6.0	11.0	4.2
MEHP	Monoethylhexyl Phthalate	4376-20-9	278	8.2	11.4	4.7
MECPP	Mono-2-ethyl-5-carboxypentyl phthalate	40809-41-4	308	7.1	16.2	4.0
MEOHP	Mono-2-ethyl-5-oxohexyl phthalate	40321-98-0	292	1.0	12.8	2.7
MEHHP	Mono-2-ethyl-5-hydroxyhexyl phthalate	40321-99-1	294	1.2	14.3	3.2
MiNP	Monoisononyl phthalate	N/A	292	10.5	11.8	5.2
MBzP	Monobenzyl phthalate	2528-16-7	256	0.9	11.7	3.1

The properties are estimated based on the U.S. Environmental Protection Agency Estimation Program Interface (EPI) Suite Version 4.11;

[a] normalized to 10 gram fish;

[b] octanol-air partition coefficient;

[c] octanol-water partition coefficient;

Table S2. The multiple reaction monitoring (MRM) ions or selected ion monitoring and limit of quantification (LOQ) of phthalate di- and monoesters.

Abbreviation	Collision energy	Declustering potential	MRM/SIM	Surrogate standards	LOQ	
					dust (ng/g)[a]	urine (ng/mL)[b]
Diesters						
BBzP	40	20	313.1/91.1	DBzP -d4	55	-
	30	20	313.1/149.2			
DAlP	20	40	247.1/189.1	DEP-d4	80	-
	35	20	247.1/149			
DiPeP	30	30	307.2/71.1	DPeP -d4	4.1	-
	40	30	307.2/149.2			
DPeP	30	25	307.2/149.1	DPeP -d4	51	-
	60	25	307.2/65.1			
DBzP	30	20	347.1/91.1	DBzP -d4	4.0	-
	20	20	347.1/181.1			
DiBP	30	25	279.2/149.1	DiBP-d4	24	-
	60	25	279.2/65.2			
DBP	20	10	279.1/149.2	DBP-d4	63	-
	20	60	279.1/205.1			
DCHP	20	30	331.2/149.2	DCHP-d4	1.7	-
	20	30	331.2/167			
DEP	25	25	223.2/149.1	DEP-d4	15	-
	20	20	223.2/177.1			
DiHxP	30	20	335.2/149.2	DHxP-d4	4.4	-
	20	20	335.2/85.2			
DHxP	30	25	335.2/149.1	DHxP-d4	3.9	-
	20	20	335.2/85.1			
DiHeP	30	25	363.2/149.2	DHeP -d4	56	-
	20	20	363.2/99.2			-
DHeP	30	20	363.2/149.1	DHeP -d4	4	
	80	25	363.2/65.2			-
DiNP	30	20	419.3/85.1	DEHP-d4	14	
	30	20	419.3/149.1			-
DNP	30	20	419.3/149.1	DEHP-d4	50	
	30	20	419.3/71.1			-
DiPrP	30	25	251.2/149.1	DPrP-d4	3.1	
	45	25	251.2/65.1			-
DPrP	40	20	251.2/149.1	DPrP-d4	24	-
	20	25	251.2/191.1			
DMP	20	20	195.1/163.2	DMP-d4	50	
	40	20	196.1/77.1			-
DPP	20	20	319.1/225.2	DPP-d4	0.6	

	45	20	319.1/77.1			
DiDP	30	20	447.3/85.2	DEHP-d4	66	-
	30	20	447.3/149.2			
DUP	30	25	475.4/149	DEHP-d4	6	-
	35	20	475.4/121.1			
iBCHP	30	20	305.2/149.2	DBP-d4	26	-
	20	25	305.2/223.2			
DEHP	-	-	149/167	DEHP-d4	35	-
Monoesters						
MMP	-10	-100	178.9/107	MBP-d4	6.9	0.2
	-20	-20	178.9/77.1			
MHeP	-20	-30	263/113.1	MBP-d4	42	3.4
	-30	-30	263/77			
MBzP	-30	-30	254.9/77	MBzP-d4	9.0	2.6
	-25	-25	254.9/107			
MBP	-25	-25	221/71	MBP-d4	30	1.7
	-25	-25	221/77			
MCHP	-25	-25	247/97	MBP-d4	12	4.4
	-30	-25	247/77			
MEP	-30	-30	192.9/77	MBP-d4	4.0	3.4
	-20	-25	192.9/121			
MEHP	-30	-40	277/77	MBP-d4	15	7.2
	-30	-40	277/134.1			
MHxP	-30	-30	249/77	MBP-d4	44	1.0
	-25	-30	249/99.1			
MiBP	-25	-25	221/77	MBP-d4	22	0.9
	-25	-25	221/134			
MiNP	-50	-45	291.1/141.1	MBP-d4	59	5.1
	-50	-45	291.1/77			
MiPrP	-25	-25	207/77	MBP-d4	5.5	0.9
	-25	-25	207/120.9			
MPeP	-30	-25	235/77	MBP-d4	98	1.4
	-25	-30	235/85			
MECPP	-20	-30	307.1/159.1	MBP-d4	33	0.3
	-40	-30	307.1/113.1			
MEHHP	-20	-30	293.1/121	MBP-d4	6.4	0.2
	-10	-25	293.1/145.1			
MEOHP	-30	-45	291.1/143.1	MBP-d4	18	1.0
	-40	-45	291.1/121			

[a] based on 50 mg dust

[b] based on 1 mL urine

Table S3. Characteristics of study populations and dwelling environment.

Children	n (%)	Adults	n (%)
Sex		*Sex*	
Female	35 (73)	Female	21 (51)
male	13 (27)	male	20 (49)
Age (year)		*Age (year)*	
0.5-3	24 (50)	20-35	20 (49)
3-6	24 (50)	35-70	21 (51)
Weight (kg)		*Weight (kg)*	
8.2-15.0	22 (46)	47-68	23 (56)
15.1-32	26 (54)	70-80	18 (44)
Height (m)		*Height (m)*	
0.50-0.99	27 (56)	1.50-1.69	27 (65)
1.00-1.33	21 (44)	1.70-1.88	14 (35)
Hours per day spent in home (h)		*Hours per day spent in home (h)*	
8-16	24 (50)	8-16	31 (76)
17-24	24 (50)	17-24	10 (24)

Table S4. Concentrations of phthalate monoesters (μg/g) in five replicate samples of the NIST Standard Reference Material 2585 and their relative standard deviation (RSD)

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Average	RSD (%)
MMP	4.2	3.6	3.8	4.2	3.9	3.9	6
MEP	4.4	5.7	5.0	5.3	4.7	5.0	9
MiPrP	0.22	0.26	0.24	0.24	0.23	0.24	6
MiBP	6.0	6.4	6.2	6.6	6.7	6.4	4
MBP	18	19	19	20	20	19	4
MBzP	1.2	1.2	1.3	1.3	1.6	1.3	10
MHxP	1.3	1.0	1.2	1.1	1.3	1.2	10
MHeP	1.8	1.5	1.5	1.6	1.9	1.7	11
MiNoP	2.5	2.2	2.9	2.4	2.8	2.5	10
MPeP	0.14	0.15	0.17	0.18	0.18	0.17	11
MCHP	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	-
MEHP	10	10	10	10	13	11	9
MECPP	0.74	0.80	0.61	0.74	0.69	0.7	9
MEOHP	0.35	0.36	0.47	0.48	0.48	0.43	14
MEHHP	3.5	3.5	3.4	3.9	3.8	3.6	6

Table S5. The rate of degradation (%) from phthalate diesters to respective monoesters during sample treatments.[a]

Mono- and di-PAE pairs	S1	S2	S3	S4	S5
MMP-DMP	0.4	0 [b]	0	0	1.5
MEP-DEP	0	0	0	0	0
MiPrP-DiPrP	0	0	0	0	0
MiBP-DiBP	2.0	1.3	3.8	2.0	2.7
MBP-DBP	0.5	0.1	1.7	0.5	0.9
MPeP-DPeP	0.2	0	0.2	0.1	0.3
MHxP-DHxP	0.1	0	0.1	0	0.1
MCHP-DCHP	3.2	2.6	3.0	3.3	3.6
MiHep-DiHeP	0	0	0.1	0.1	0.1
MBzP-[BBzP+DBzP]	0	0	0.2	0.1	0
MEHP-DEHP	0	0	0.1	0	0.1
MECPP-DEHP	0	0	0	0	0
MEOHP-DEHP	0	0	0	0	0
MEHHP-DEHP	0	0	0	0	0
MiNP-DiNP	0	0	0	0	0

[a] 500 ng each of the target phthalate diesters spiked into 0.1g sodium sulfate (n = 5; S1 – S5);

[b] mono-PAE was not detected.

Table S6. Concentrations and detection frequencies of phthalate diesters without respective monoesters screened in house dust (μg/g).

	% detected	median	5th percentile	95th percentile
DiAlP	0	<LOQ	<LOQ	<LOQ
DPrP	96	0.45	0.03	2.16
DPP	0	<LOQ	<LOQ	<LOQ
iBCHP	0	<LOQ	<LOQ	<LOQ
DiPeP	65	0.03	<LOQ	0.15
DiHxP	0	<LOQ	<LOQ	<LOQ
DHeP	98	0.09	0.03	0.59
DiDP	100	7.68	2.54	22.22
DUP	95	0.48	0.05	1.63
DNP	100	1.10	0.51	2.94

Table S7 Coefficient of variation (CV, %) of the molar concentration ratios of a mono-PAE to its respective di-PAE

	average	standard deviation	CV (%)
MMP-DMP	5.4	5.9	109
MEP-DEP	0.4	0.5	135
MiPrP-DiPrP	3.6	6.2	170
MiBP-DiBP	0.4	0.4	105
MBP-DBP	0.5	0.4	82
MEHP-DEHP	0.02	0.01	59
MECPP-DEHP	0.001	0.002	135
MEOHP-DEHP	0.001	0.001	148
MEHHP-DEHP	0.004	0.01	149

Table S8. Spearman correlation coefficients for phthalate mono- and diesters in house dust and mono-esters in urine for children and adults

urine-dust	children	adults	urine-dust	children	adults	urine-dust	children	adults
MMP-MMP	-0.03	-0.11	MMP-DMP	0.10	0.03	MMP-[DMP+MMP]	-0.06	0.06
MEP-MEP	-0.20	-0.02	MEP-DEP	-0.11	-0.14	MEP-[DEP+MEP]	-0.12	0.04
MiBP-MiBP	**0.29***	0.29	MiBP-DiBP	0.12	0.16	MiBP-[DiBP+MiBP]	0.22	0.20
MBP-MBP	0.23	0.21	MBP-DBP	0.10	0.08	MBP-[DBP+MBP]	0.13	0.10
MEHP-MEHP	0.21	-0.25	MEHP-DEHP	0.28	0.21	MEHP-[DEHP+MEHP]	0.28	0.03
MEOHP-MEOHP	0.11	-0.28	MEOHP-DEHP	0.12	0.20	MEOHP-MEHP	0.04	-0.26
MEHHP-MEHHP	0.13	-0.14	MEHHP-DEHP	0.16	0.18	MEHHP-MEHP	0.03	-0.25
MECPP-MECPP	0.03	-0.21	MECPP-DEHP	0.12	0.12	MECPP-MEHP	-0.01	-0.33

* $p < 0.05$

Table S9. Regression analyses of predictors of child urinary phthalate monoesters (ng/mL)

Predictors	MMP		MEP		MiBP		MBP		MEHP		MEOHP		MEHHP		MECPP	
	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*
Gender																
male	reference		reference		reference		reference		reference		reference		reference		reference	
female	0.73 (0.24, 2.2)[a]	0.56	0.69 (0.19, 2.5)	0.57	0.44 (0.18, 1.1)	0.08	0.60 (0.2, 1.5)	0.21	0.74 (0.16, 2.4)	0.48	0.78 (0.33, 1.9)	0.57	0.66 (0.27, 1.6)	0.35	0.77 (0.32, 1.9)	0.55
Age	0.90 (0.76, 1.1)	0.25	0.92 (0.73, 1.1)	0.42	0.89 (0.76, 1.1)	0.17	0.96 (0.84, 1.2)	0.91	1.1 (0.88, 1.4)	0.35	1.0 (0.87, 1.2)	0.92	0.97 (0.82, 1.1)	0.66	0.97 (0.83, 1.1)	0.67
BMI																
≤16.6	reference		reference		reference		reference		reference		reference		reference		reference	
>16.6	0.71 (0.33, 1.6)	0.39	0.84 (0.31, 2.3)	0.73	0.67 (0.32, 1.4)	0.27	0.67 (0.32, 1.4)	0.28	0.88 (0.32, 2.2)	0.73	1.1 (0.59, 2.1)	0.72	1.1 (0.54, 2.2)	0.78	1.2 (0.61, 2.3)	0.61
Time spending in home per day (h)																
≤16	reference		reference		reference		reference		reference		reference		reference		reference	
>16	1.0 (0.43, 2.4)	0.98	0.71 (0.26, 1.9)	0.48	0.97 (0.48, 2.0)	0.94	0.96 (0.45, 2)	0.91	0.81 (0.28, 2.1)	0.59	0.89 (0.47, 1.8)	0.77	0.90 (0.46, 1.8)	0.76	0.83 (0.42, 1.6)	0.57
Mono-PAEs in dust																
low	reference		reference		reference		reference		reference		reference		reference		reference	
high	0.98 (0.4, 2.4)	0.97	0.93 (0.34, 2.6)	0.88	1.7 (0.79, 3.6)	0.17	1.9 (0.85, 4.5)	0.11	0.80 (0.25, 2.2)	0.59	1.0 (0.53, 1.9)	0.96	1.2 (0.57, 2.5)	0.61	1.1 (0.53, 2.1)	0.87
Di-PAEs in dust																
low	reference		reference		reference		reference		reference		reference		reference		reference	
high	1.3 (0.51, 3.2)	0.60	0.70 (0.24, 2)	0.50	1.0 (0.51, 2.1)	0.76	1.1 (0.48, 2.4)	0.84	1.0 (0.31, 2.8)	0.90	0.84 (0.45, 1.7)	0.67	0.95 (0.46, 2)	0.90	0.91 (0.45, 1.8)	0.79

[a] 95% confidence interval

Table S10. Regression analyses of predictors of adult urinary phthalate monoesters (ng/mL)

Predictors	MMP		MEP		MiBP		MBP		MEHP		MEOHP		MEHHP		MECPP	
	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*	β coefficient	*p*
Gender																
male	reference		reference		reference		reference		reference		reference		reference		reference	
female	0.58 (0.28, 1.3)[a]	0.16	1.1 (0.49, 2.7)	0.75	1.4 (0.75, 2.5)	0.30	1.3 (0.6, 2.7)	0.52	1.1 (0.48, 2.5)	0.84	1 (0.53, 1.9)	0.99	1.1 (0.55, 2)	0.87	0.88 (0.49, 1.7)	0.74
Age	1.0 (0.99, 1.1)	0.20	1.0 (0.99, 1.1)	0.18	1.0 (0.97, 1.0)	0.79	1.0 (0.96, 1.0)	0.81	1.0 (0.95, 1.0)	0.44	1.0 (0.96, 1.0)	0.54	1.0 (0.96, 1.0)	0.47	1.0 (0.96, 1.0)	0.59
BMI																
≤25	reference		reference		reference		reference		reference		reference		reference		reference	
>25	0.68 (0.26, 1.7)	0.40	0.72 (0.25, 2.1)	0.54	2.0 (0.91, 4.2)	0.08	1.6 (0.63, 4.3)	0.30	0.87 (0.29, 2.4)	0.72	0.85 (0.38, 1.9)	0.67	0.86 (0.36, 2)	0.71	0.84 (0.38, 1.9)	0.67
Time spending in home per day (h)																
≤12	reference		reference		reference		reference		reference		reference		reference		reference	
>12	0.67 (0.3, 1.5)	0.33	1.3 (0.50, 3.1)	0.62	0.80 (0.41, 1.5)	0.49	1.2 (0.53, 2.6)	0.69	0.59 (0.23, 1.3)	0.18	0.5 (0.27, 1.1)	0.07	0.51 (0.26, 1.1)	0.09	0.56 (0.31, 1.2)	0.15
Mono-PAEs in dust																
low	reference		reference		reference		reference		reference		reference		reference		reference	
high	0.96 (0.42, 2.2)	0.91	1.2 (0.48, 3.2)	0.66	1.4 (0.75, 2.6)	0.27	1.8 (0.84, 3.8)	0.13	1 (0.42, 2.4)	0.99	0.63 (0.36, 1.3)	0.22	0.81 (0.41, 1.7)	0.62	0.81 (0.46, 1.6)	0.60
Di-PAEs in dust																
low	reference		reference		reference		reference		reference		reference		reference		reference	
high	1.7 (0.64, 3.4)	0.35	1.1 (0.41, 2.7)	0.90	1.1 (0.60, 2.1)	0.69	0.87 (0.41, 1.9)	0.73	1.5 (0.64, 3.8)	0.32	1.5 (0.75, 2.7)	0.27	1.5 (0.68, 2.8)	0.36	1.3 (0.66, 2.3)	0.50

[a] 95% confidence interval