



Legacy and alternative flame retardants in Norwegian and UK indoor environment: implications of human exposure via dust ingestion

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Accepted Version

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Kademoglou, K., Xu, F., Padilla-Sanchez, J. A., Haug, L. S., Covaci, A. and Collins, C. D. (2017) Legacy and alternative flame retardants in Norwegian and UK indoor environment: implications of human exposure via dust ingestion. *Environment International*, 102. pp. 48-56. ISSN 0160-4120 doi: <https://doi.org/10.1016/j.envint.2016.12.012> Available at <http://centaur.reading.ac.uk/69170/>

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To link to this article DOI: <http://dx.doi.org/10.1016/j.envint.2016.12.012>

Publisher: Elsevier

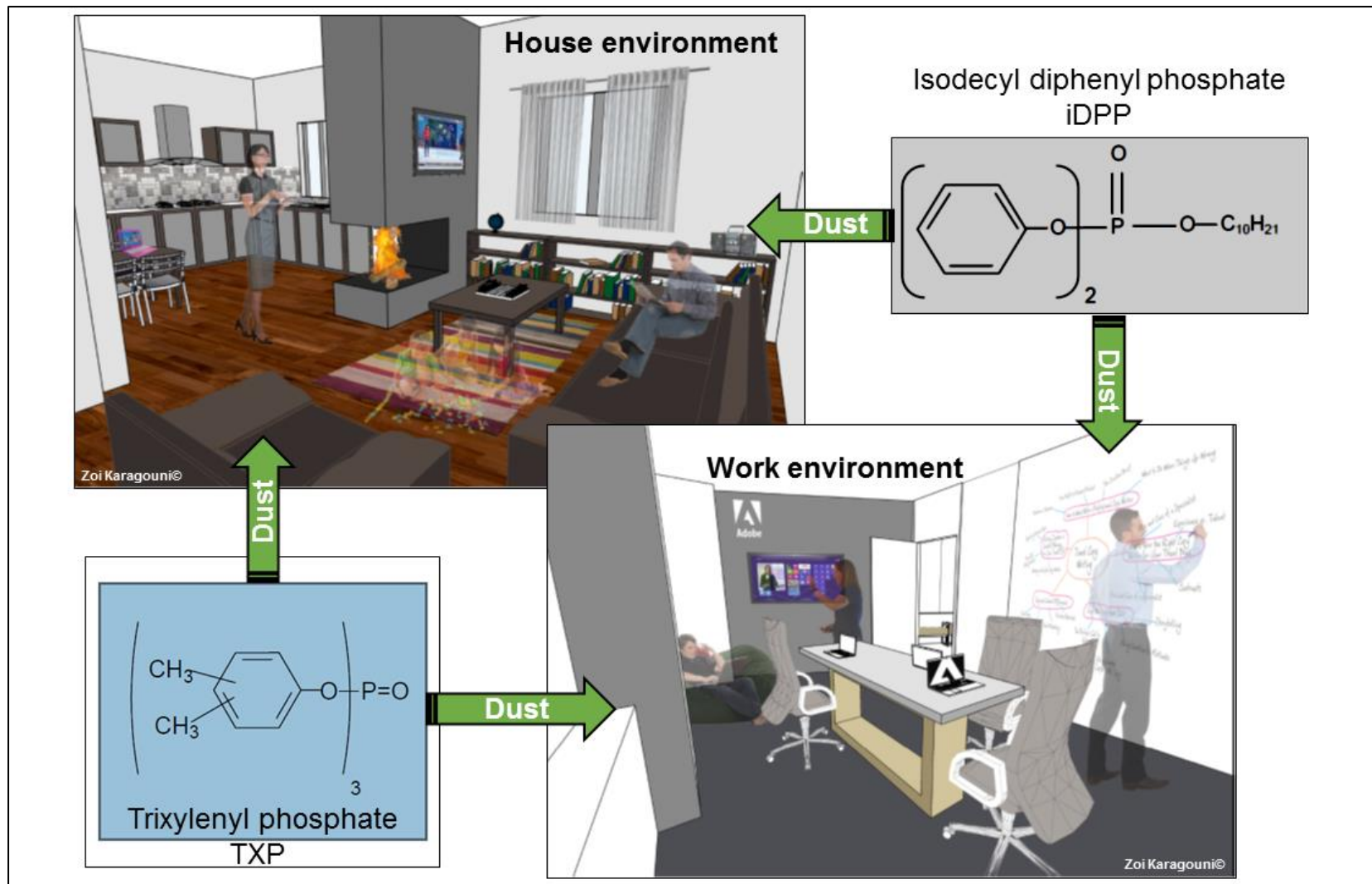
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1 Legacy and Alternative Flame Retardants in Norwegian and UK Indoor Environment: Implications of
2 Human Exposure via Dust Ingestion

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10 **Abstract**

11 Indoor dust has been acknowledged as a major source of flame retardants (FRs) and dust ingestion is
12 considered a major route of exposure for humans. In the present study, we investigated the presence of
13 PBDEs and alternative FRs such as emerging halogenated FRs (EHFRs) and organophosphate flame
14 retardants (PFRs) in indoor dust samples from British and Norwegian houses as well as British stores
15 and offices. BDE209 was the most abundant PBDE congener with median concentrations of 4,700 ng
16 g⁻¹ and 3,400 ng g⁻¹ in UK occupational and house dust, respectively, 30 and 20 fold higher than in
17 Norwegian house dust. Monomeric PFRs (m-PFRs), including triphenyl phosphate (TPHP),
18 tris(chloropropyl) phosphate (TCPP) and tris(2-chloroethyl) phosphate (TCEP) dominated all the
19 studied environments. To the best of our knowledge, this is the first report of isodecyldiphenyl
20 phosphate (iDPP) and trixylenyl phosphate (TXP) in indoor environments. iDPP was the most abundant
21 oligomeric PFR (o-PFR) in all dust samples, with median concentrations one order of magnitude higher
22 than TXP and bisphenol A bis(diphenyl phosphate (BDP). iDPP and TXP worst-case scenario
23 exposures for British workers during an 8h exposure in the occupational environment were equal to 34
24 and 1.4 ng kg bw⁻¹ day⁻¹, respectively. The worst-case scenario for BDE209 estimated exposure for
25 British toddlers (820 ng kg bw⁻¹ day⁻¹) did not exceed the proposed reference dose (RfD) (7,000 ng
26 kg bw⁻¹ day⁻¹), while exposures for sum of m-PFRs (Σ m-PFRs) in British toddlers and adults (17,900
27 and 785 ng kg bw⁻¹ day⁻¹ respectively) were an order of magnitude higher than for Norwegian toddlers
28 and adults (1,600 and 70 ng kg bw⁻¹ day⁻¹).

29 **Keywords:** PBDEs; alternative flame retardants; UK; Norway; indoor dust; human exposure

30 **Highlights**

- 31 • PBDEs, EHFRs and PFRs were analysed in Norwegian and UK house, store & office dust
- 32 • First report of iDPP and TXP in indoor dust with several o-PFRs also detected
- 33 • m-PFRs dominated all indoor environments, followed by EHFRs, PBDEs, and o-PFRs
- 34 • BDE209 levels were significantly higher in British than Norwegian house dust
- 35 •
- 36 • o-PFR estimated intakes for employees in Britain were higher than stay-home adults

37 **Abbreviations**

38 EHFRs: emerging halogenated flame retardants

39 m-PFRs: monomeric PFRs

40 o-PFRs: oligomeric PFRs

41 1. Introduction

42 Flame retardants (FRs) are widely used in everyday consumer products including carpets, electronic
43 appliances, clothing and textiles, thermal insulation and cable coatings. Since the 1970s, polybrominated
44 diphenyl ethers (PBDEs) have been widely used in consumer products as FRs (Alaee et al., 2003).
45 Various human health effects are associated with PBDEs exposure such as disruption of the endocrine
46 and thyroid homeostasis (Legler and Brouwer, 2003) and neurodevelopmental growth of children (Costa
47 and Giordano, 2007). The commercial mixtures Penta-BDE and Octa-BDE have been listed as persistent
48 organic pollutants (POPs) for elimination under the Stockholm Convention (Stockholm Convention,
49 2009a, 2009b), while the Deca-BDE mixture is currently under review. The use of Deca-BDE was
50 banned in Norway in 2008 (EBFRIP, 2008), while it was included by the EU in the amended Annex
51 XVII of REACH (EC No 1907/2006), banning its production, use and marketing in the EU (European
52 Commission, 2016). As a result of the REACH amendment, furniture and fire safety regulations in the
53 UK are currently under review by the national competent authorities (UK Department for Business,
54 Energy & Industrial Strategy, 2016). Due to legislative restrictions on their commercial use, PBDEs
55 have been replaced with alternatives, known as “emerging” halogenated flame retardants (EHFRs)
56 including 2-ethylhexyl-2,3,4,5-tetrabromobenzoate (EH-TBB; Penta-BDE replacement), bis(2-
57 ethylhexyl)-3,4,5,6-tetrabromophthalate (BEH-TEBP; Penta-BDE replacement), 1,2-bis(2,4,6-
58 tribromophenoxy) ethane (BTBPE; Octa-BDE replacement), decabromodiphenyl ethane (DBDPE;
59 Deca-BDE replacement) and Dechlorane Plus (DPs; Deca-BDE replacement) (Stapleton et al., 2008;
60 Wang et al., 2011) and organophosphate flame retardants (PFRs) such as tris(2-chloroethyl) phosphate
61 (TCEP) and tris(chloropropyl)phosphate (TCPP) (van der Veen and de Boer, 2012).

62 Several studies have indicated that also EHFRs and PFRs may pose potential risks to humans. EH-TBB
63 and BEH-TEBP, major components in the commercial product Firemaster 550[®], have been proven to
64 act as endocrine disruptors and obesogens when orally administered to rats (Patisaul et al., 2013) and
65 can bind and activate the transcription of peroxisome proliferator-activated receptor γ (PPAR γ) ligands,
66 while triphenyl phosphate (TPHP)-induced *in vitro* adipocyte differentiation and diverted osteogenic
67 differentiation towards lipid accumulation has been reported (Pillai et al., 2014). DPs, EH-TBB, BEH-
68 TEBP and PFRs, such as TCEP and tris(1,3-dichloro-2-propyl) phosphate (TDCIPP) have been detected
69 in human breast milk and blood in Asian populations (Ben et al., 2013; Kim et al., 2014), as well as in
70 blood, hair and nails in USA residents (Liu et al., 2016). TDCIPP has been linked with reduction in free
71 thyroxine and increase in prolactin secretion in US men, while TPHP was associated with weakening
72 sperm quality (Meeker and Stapleton, 2010). An *in vitro* estrogenic and anti-androgenic potency of
73 TDCIPP, tris(2-butoxyethyl) phosphate (TBOEP), and TPHP on human osteosarcoma (U2OS) cell line
74 exposed to indoor dust extracts has also been reported (Suzuki et al., 2013). In the EU, restrictions on

75 the use of chlorinated PFRs, such as TDCIPP and TCPP, have been issued based on toxicological
76 concerns related to their carcinogenic potency (ECHA, 2008a, 2008b).

77 Monomeric PFR (m-PFRs), including TDCIPP, TCPP and TCEP, are routinely used as FRs in flexible
78 polyurethane foams (PUFs) and textiles (Ali et al., 2012; Cao et al., 2014b). TPHP can be used as a
79 plasticiser and a FR in PVC, thermoplastics and synthetic polymers, while TBOEP is exclusively used
80 as a plasticiser in floor polish and rubber products (Marklund et al., 2003; Stapleton et al., 2009; van der
81 Veen and de Boer, 2012). The use of EHFRs and m-PFRs in consumer products has thus increased and
82 this is reflected by their high abundance in indoor dust in the UK (Brommer and Harrad, 2015), China
83 (Cao et al., 2014a), Japan (Tajima et al., 2014), Sweden (Newton et al., 2015) and Norway (Cequier et
84 al., 2014). PFRs such as TCPP, TCEP and TBOEP dominate house, office and hotel environments, with
85 levels in hotel dust six fold higher than office dust from China (Cao et al., 2014b). A few studies have
86 reported oligomeric PFRs (o-PFRs) in considerable amounts in dust, such as tetraakis(2-chlorethyl)-
87 dichloroisopentyl diphosphate (V6), an alternative of Penta-BDE, TCPP and TDCIPP (ECHA, 2008c),
88 along with resorcinol bis(diphenyl phosphate (RDP) and bisphenol A bis(diphenyl phosphate (BDP) as
89 Deca-BDE alternatives in electronic and plastic consumer products (Ballesteros-Gómez et al., 2014;
90 Brandsma et al., 2013; Matsukami et al., 2015). Since house dust acts as a repository sink for EHFRs
91 and PFRs, dust originating from indoor environments (e.g. houses, offices, stores) is considered as a
92 major source of human exposure to FRs (Alves et al., 2014; Jones-Otazo et al., 2005).

93 In April 2016, the Washington State House Bill 2545 (Toxic-free Kids and Families Act) was approved
94 to ban children's products and residential upholstered furniture from the market containing more than
95 0.1% of TCEP, TDCIPP, Deca-BDE, hexabromocyclododecane (HBCD) and tetrabromobisphenol A
96 (TBBPA) with an effective date set for June 2016. Additional six FRs, including TPHP, TCPP, V6, EH-
97 TBB, BEH-TEBP, and isopropylated triphenyl phosphate (IPTPHP) will be evaluated and
98 recommended to the Legislature for possible restriction in consumer products (State of Washington,
99 2016). The implementation of this bill may potentially trigger the phasing out PBDE alternatives, thus
100 initiate the development and use of newer FRs. Therefore, the continuous and rigorous assessment of

101 legacy and alternative FRs, especially oligomeric PFRs (o-PFRs), in the indoor environment is essential
102 due to their potential adverse effects on human health.

103 To bridge this knowledge gap, the main objectives of the present study are:

104 a) To assess the presence of legacy and alternative FRs in three different indoor environments from
105 two European countries (the UK and Norway)

106 b) To estimate and compare human intakes to a wide range of FRs via dust ingestion using the
107 same dust samples for non-working adults and toddlers in Norwegian and British houses, as
108 well as for working adults in British stores and offices.

109 **2. Materials and Methods**

110 **2.1 Sampling**

111 Ten indoor dust samples were collected from pre-existing vacuum cleaner bags (houses) in Norway
112 (Oslo) as a part of the A-TEAM cohort sampling during November 2013 – April 2014 (Papadopoulou
113 et al., 2016). Twenty-two indoor dust samples from pre-existing vacuum cleaner bags (10 houses, 6
114 stores and 6 offices; Table SI-1) were collected in Reading (UK) during August – December 2013. The
115 UK house dust samples were collected from the homes of University of Reading employees, while UK
116 office and store vacuum cleaner bags were collected in Reading with respect to the participant's approval
117 and willingness to cooperate in the present study. All dust samples were sieved to <250 µm using a
118 methanol-washed metallic sieve; this size fraction of dust is likely to be ingested according to (Yu et al.,
119 2012). Oven-baked Na₂SO₄ (granular) was also sieved as field blank. All dust samples were kept in
120 hexane-washed amber glass bottles and stored at 4°C till analysis.

121 **2.2 Extraction and clean-up**

122 The method was based on a previous study (Van den Eede et al., 2012a) with some modifications.
123 Briefly, 30 mg of dust was extracted with 2.5 mL hexane:acetone (3:1) using ultra-sonication extraction
124 for 10 min and vortexing for 1 min three times. The combined extract was concentrated on aminopropyl
125 (NH₂) silica cartridges (500 mg, 3 mL, Agilent, USA) and further fractionated with 10 mL hexane (F1)
126 and 12 mL of ethyl acetate (F2). F1 was further concentrated, following a clean-up on an acidified silica
127 cartridge (5%, 1 g, 6 mL) and elution with 12 mL dichloromethane. F2 was equally aliquoted into two
128 portions, F2a and F2b. Then, F1, F2a and F2b were evaporated, reconstituted with 100 µL of iso-octane
129 (F1 & F2a) and methanol (F2b), respectively, and then filtered. Finally, the extracts were transferred to
130 injection vials and analyzed on GC-ECNI-MS (F1, for PBDEs and EHFRs), GC-EI-MS (F2a, for m-

131 PFRs, except TXP) and LC-QqQ-MS (F2b, for o-PFRs and TXP). More details about sample
132 preparation and instrumental analysis are found in SI.

133 **2.3 QA/QC and Data Analysis**

134 Overall, 28 and 31 compounds (out of 33) were detected in house and occupational dust samples,
135 respectively (Tables SI-2, SI-3, SI-4, and SI-5). SRM 2585 (n=2, NIST, USA) was used for QC testing
136 and the results were in line with the literature (Table SI-6). Four Na₂SO₄ samples (30mg) were used as
137 field blanks for background checking and results were blank corrected for all analytes by subtraction of
138 the mean field blank values from the raw FR values (expressed in ng/g) according to (Abdallah and
139 Covaci, 2014). Method limits of detection (mLOD) were calculated as three times the standard deviation
140 of the field blanks. For non-detected analytes, mLOD was calculated based on signal-to-noise-ratio 3:1
141 (Table SI-7). GraphPad Prism[®] version 7.00 for Windows, (GraphPad Software, La Jolla CA, USA) was
142 used for statistical analysis. Compounds with detection frequencies (DF) lower than 40% were excluded
143 from statistical analysis. Where needed, non-detections were replaced by half of mLOD for statistical
144 analysis. All data were checked for normality using the D'agostino and Pearson tests, data that failed
145 the normality test were log-transformed and checked for normality again. Not all data were normally
146 distributed after log-transformation. Ordinary two-way ANOVA (Uncorrected Fisher's test, p<0.05)
147 was performed to assess statistically significant differences of FRs between UK house and occupational
148 concentrations and between UK and Norwegian houses. Due to some data failing to pass normality tests,
149 Spearman's correlation (p<0.05) was employed to assess statistical dependence and correlation between
150 FRs in the three different dust categories.

151 **3. Results and discussion**

152 This study reports concentrations of four groups of FRs in dust from UK stores and offices (n=6 offices
153 and n=6 stores), UK houses (n=10) and Norwegian houses (n=10). Studied chemicals included nine
154 PBDE congeners, eight EHFRs, ten monomeric PFRs, and six oligomeric PFRs (Tables S1-8, SI-9, SI-
155 10, SI-11, SI-12, and SI-13). Overall, the UK occupational dust samples had the highest FR
156 contamination, followed by UK and Norwegian house dust. In an attempt to define newly identified
157 PFRs, this group is divided in monomeric (m-PFRs), including TPHP, TnBP, TCPP, TDCIPP etc., and
158 oligomeric (o-PFRs), including V6, BDP and RDP, using the abbreviation nomenclature as suggested
159 by (Matsukami et al., 2015). In our study, monomeric PFRs presented the highest levels in total,
160 followed by EHFRs, PBDEs and oligomeric PFRs.

161 **3.1 PBDEs**

162 Most PBDEs were frequently detected in UK houses and occupational dust with DF>50%, unlike in the
163 Norwegian house dusts (Fig. 1A, B, C; Table SI-8, SI-10, and SI-12). BDE28 to BDE183 levels were

164 relatively lower compared to BDE209, probably because of the global phase out of Penta- and Octa-
165 BDE commercial mixtures (Dodson et al., 2012). Similar to indoor dust samples from Belgium, China
166 and Sweden (Ali et al., 2011; Newton et al., 2015; Zheng et al., 2011) median level of BDE47 was four-
167 fold higher in UK (12 ng g^{-1}) than in Norwegian house dust samples. Median concentrations of BDE47
168 (9.1 ng g^{-1}) and BDE183 (11 ng g^{-1}) in occupational dust were within the concentration range of studies
169 from Belgium and Germany (Ali et al., 2011; Brommer et al., 2012), but lower than the USA (Michigan)
170 and China (Batterman et al., 2010; Cao et al., 2014b). BDE209, the most abundant PBDE congener,
171 was detected in all samples, with median concentrations of $4,700 \text{ ng g}^{-1}$ and $3,400 \text{ ng g}^{-1}$ in UK
172 occupational and house dust, respectively, which is much higher than a recent study of Norwegian
173 classroom dust (507 ng g^{-1}) (Cequier et al., 2014) and also in the Norwegian house dust samples from
174 the present study (160 ng g^{-1}) (Fig 1A). A statistically significant difference of BDE209 concentrations
175 was observed between UK and Norwegian house dust ($p=0.014$). Since DBDPE acts a major
176 replacement of BDE209, the BDE209/DBDPE ratio is indicative of the progress of phasing out Deca-
177 BDE. The median BDE209 /DBDPE ratio was <1 for Norwegian house dust while it was >3 in UK
178 house dust. Such findings can be possibly attributed to low Deca-BDE usage in Norway and its unilateral
179 ban since 2008 (EBFRIP, 2008), contrary to the EU where Deca-BDE was added to the candidate list
180 of substances of very high concern for authorisation under the REACH regulation in 2012 with its use
181 in consumer products eventually banned within the REACH framework earlier in 2016 (ECHA, 2012;
182 European Commission, 2016). Unlike the Nordic indoor environment where hard-surfaced wooden
183 flooring is more frequently applied (Roos and Hugosson, 2008), an evident preference towards carpet
184 flooring in UK houses could potentially contribute to the higher BDE209 levels, hence the high
185 BDE209/DBDPE median ratio in UK house dust (Jonsson, 2005). However, the median BDE209
186 /DBDPE ratio in the UK occupational dust was <1 , probably due to the replacement of Deca-BDE in
187 newer products in stores and offices compared to house environment.

188 **3.2 Emerging halogenated FRs**

189 Nearly all EHFRs were frequently detected ($DF>50\%$) in all three types of dust (Fig. 1A, B, C, Table
190 SI-8, SI-10, SI-12). DBDPE and BEH-TEBP were the most abundant EHFRs ($DF>80\%$). In house dust,
191 DBDPE median concentration was two-fold higher in UK ($1,100 \text{ ng g}^{-1}$) than Norway (686 ng g^{-1}) (Fig
192 1A&B), which was in agreement with a previous Norwegian study (Cequier et al., 2014) and
193 considerably higher than DBDPE levels in dust from Belgium and Sweden (Ali et al., 2011; Newton et
194 al., 2015). The median concentration of BEH-TEBP in UK house dust (110 ng g^{-1}) was equivalent to
195 recent studies from USA and Sweden (Dodson et al., 2012; Newton et al., 2015). However, BEH-TEBP
196 median in our Norwegian dust samples was lower than a previous Norwegian study (Cequier et al.,
197 2014). The small sample size of the Norwegian dust collection analysed in the present study ($n=10$) may
198 act as a limiting factor. Our dust samples were collected from pre-existing vacuum cleaner bags, whereas

199 in (Cequier et al., 2014) dust samples (n=48) were collected using forensic filters. In UK occupational
200 dust, DBDPE had the highest median concentration (5,400 ng g⁻¹), followed by BEH-TEBP (250 ng g⁻¹),
201 both of which were higher than previous studies on Belgian and German office dust (Ali et al., 2011;
202 Brommer et al., 2012), but lower than a recent Chinese study (Cao et al., 2014b). EH-TBB was several
203 folds lower than BEH-TEBP in all three types of dust. Additional sources of BEH-TEBP in consumer
204 products other than Firemaster 550[®] (EH-TBB/BEH-TEBP ratio 4:1 in commercial mixture (Stapleton
205 et al., 2008) are suspected to be Great Lakes DP-45[™] and Firemaster[®] BZ-54 (Chemtura Inc., USA),
206 (Barr et al., 2012; Zheng et al., 2015). A statistically significant difference between UK house and
207 occupational dust concentrations was found for two Deca-BDE alternatives, DBDPE (p<0.05) and *anti*-
208 DP (p<0.05) (Stapleton et al., 2008; Zhu et al., 2007). *Anti*-DP (median: UK occupational 43.1 ng g⁻¹;
209 UK house 6.8 ng g⁻¹; Norwegian house 4.5 ng g⁻¹) was the predominant DP isomer compared to *syn*-DP
210 (median: UK occupational 15.2 ng g⁻¹; UK house 4.6 ng g⁻¹; Norway 2.6 ng g⁻¹), in agreement with other
211 studies (Cequier et al., 2014; Newton et al., 2015; Zheng et al., 2015). TBECH isomers were less
212 frequently detected (DF<60%), with concentrations of α -TBECH consistently higher than β -TBECH,
213 although the β -TBECH isomer was not detected in Norwegian house dust samples. This may be
214 attributed to β -TBECH being less volatile compared to α -TBECH, leading to lower β -TBECH levels in
215 indoor dust, unlike the 50:50 α/β TBECH isomer ratio in the commercial mixture. ([Tao et al., 2016](#);
216 [Wong et al., 2015](#)).

217 **3.3 Monomeric PFRs**

218 All m-PFRs were frequently detected (DF>50%) in all three types of samples, apart from TnPP which
219 was found <mLOD in all samples (Fig. 1 D, E, F; Table SI-9, SI-11 & SI-13). The median concentration
220 of sum of 10 m-PFRs (Σ_{10m} -PFRs) (88,000 ng g⁻¹) in UK occupational dust was similar to UK house
221 dust (79,000 ng g⁻¹), but four-fold higher than in Norwegian house dust (23,000 ng g⁻¹). Individual PFR
222 levels in our UK house dust samples were in agreement with a recent study of UK house dust (Brommer
223 and Harrad, 2015). TCPP and TBOEP presented the highest median concentrations in UK houses
224 (65,000 ng g⁻¹ and 8,100 ng g⁻¹, respectively) (Fig.1E), with TCPP median in UK houses two-fold higher
225 than house dust from Japan (30,900 ng g⁻¹) and considerably lower from another Japanese house study
226 (1,570,000 ng g⁻¹) (Kanazawa et al., 2010). In Norwegian houses, TBOEP ranked first (18,000 ng g⁻¹),
227 nearly two-fold higher than previously reported data from USA house dust (11,000 ng g⁻¹) (Dodson et
228 al., 2012) and in agreement with (Cequier et al., 2014). TBOEP (median 33,000 ng g⁻¹) and TCPP
229 (median 25,000 ng g⁻¹) were also two predominant m-PFRs in UK occupational dust. Used as a
230 plasticiser in flexible PVC, thermoplastics and food packaging, EHDPHP median concentration (20,000
231 ng g⁻¹) ranked as the third highest m-PFR in occupational dust, one to two orders of magnitude higher
232 than its median in UK house dust. This may suggest that EHDPHP usage in the UK occupational
233 environment and its application in new consumer products are steadily increasing. EHDPHP median
234 concentration in UK house dust (2,400 ng g⁻¹) was 12-fold higher than in the Norwegian house dust, yet

235 marginally lower than recently reported UK house dust concentrations (Brommer and Harrad, 2015).
236 No statistically significant difference was observed between UK and Norwegian house dust
237 concentrations ($p=0.07$) or between UK house and occupational dust samples ($p=0.055$) for EHDPHP.

238 TCPP median concentration in UK houses from our study was 30 times higher compared to Norwegian
239 house median concentration, while TCPP in Norwegian house dust was lower than levels from Belgium
240 and another Norwegian house dust study (Cequier et al., 2014; Van den Eede et al., 2011). A statistically
241 significant difference was found for TCPP ($p=0.016$) when comparing UK and Norwegian house dust
242 concentrations. This may be possibly attributed to higher TCPP usage in the UK where TCPP is a TCEP
243 replacement, while TCPP production and use in Norway have decreased during the past decade (ECHA,
244 2008a; van der Veen and de Boer, 2012). Median concentrations of TPHP and TDCIPP in UK house
245 dust were two-fold higher (1,500 and 750 ng g^{-1} , respectively) than in Norwegian houses (830 and 340
246 ng g^{-1} , respectively), but lower than TPHP and TDCIPP levels reported from the USA (Betts, 2013;
247 Stapleton et al., 2009). TMPP and TEHP were marginally different between house dusts in the two
248 countries, while the median concentration of TnBP was two-fold higher in Norwegian house dust
249 compared to UK house dust. Concentrations of m-PFRs have recently been reported in floor and surface
250 dust, sampled with dust collection filters, from the same Norwegian population group ($n=61$) (Xu et al.,
251 2016). The range of m-PFRs levels in floor and surface dust (collected from the living room) from Xu
252 et al (2016) is of the same order as the vacuum cleaner dust in the present study ($n=10$, Norwegian
253 house dust). TBOEP dominated the Norwegian house environment both in our study and in Xu et al
254 (2016). The TBOEP concentration range in the present study (1300-48,000 ng g^{-1}) was within the range
255 of floor (727-311,000 ng g^{-1}) and settled dust ($<\text{mLOD}$ -540,000 ng/g) from Xu et al (2016), yet with
256 higher median concentrations (our study: 18,000 ng g^{-1} ; Xu floor dust: 8,100 ng g^{-1} ; Xu settled dust:
257 6,800 ng g^{-1}). Such results may be attributed to: a) differences in sample size; b) vacuum cleaner dust
258 was sieved, but floor and settled were not; the large particles, like sand and hair, might dilute the
259 contamination in dust sample; c) vacuum cleaner dust is representative of the entire house, while Xu et
260 al (2016) only studied the living room; d) vacuum cleaner dust represents long term indoor
261 contamination, while floor and settled dust represent short term contamination. This suggests that the
262 sampling strategy factors such as collection season, area, tools and population selection, could
263 potentially influence the study outcome. Therefore, researchers are advised to choose a sampling
264 strategy firmly based on the aim and scope of their study.

265 **3.4 Oligomeric and monomeric PFRs**

266 Although TXP and TDBPP are considered as monomeric PFRs (Table SI-5), we will discuss them
267 together with oligomeric PFRs (o-PFRs) due to the novel character of their environmental emissions
268 and their usage in similar FR products (Matsukami et al., 2015). To the best of our knowledge, this is
269 the first report of iDPP and TXP in the indoor environment. Most o-PFRs were detected in all three

270 types of dust (DF>80%) (Fig.1 D, E, F; Table SI-9, SI-11, and SI-13), apart from RDP (no detection in
271 Norwegian house dust) and TDBPP (DF<50% in UK and Norwegian house dust samples). All o-PFRs
272 were frequently detected in occupational dust samples with substantially higher concentrations
273 compared to the house dust samples. iDPP was the most abundant o-PFR in our dust samples, ranging
274 from 600-145,000 ng g⁻¹, 110-1,700 ng g⁻¹ and 6-260 ng g⁻¹ in UK occupational dust, UK house dust
275 and Norwegian house dust, respectively. Also, median concentrations of BDP (UK occupational dust
276 480 ng g⁻¹; UK house dust 66 ng g⁻¹; Norwegian house 35 ng g⁻¹), TXP (UK occupational dust 240 ng
277 g⁻¹; UK house dust 26 ng g⁻¹; Norwegian house dust 9.1 ng g⁻¹) and V6 (UK occupational dust 40 ng g⁻¹
278 ⁻¹; UK house dust 17 ng g⁻¹; Norwegian house dust 4 ng g⁻¹) were relatively higher than RDP and TDBPP,
279 which were in general close to the mLOD. Maximum values for iDPP and BDP were close to 145,000
280 ng g⁻¹ and 6,000 ng g⁻¹, respectively, both found in dust from a toy store. In a personal computer (PC)
281 store, the maximum concentration of TXP was near 6,000 ng g⁻¹. iDPP concentrations of UK house and
282 occupational dusts were statistically significantly different (p=0.019). We can assume that this is a result
283 of the faster replacement rates of consumer products in the occupational environment compared to UK
284 houses. No significant difference was found for TXP and BDP (p=0.07), possibly as a result of the small
285 sample size analysed in the present study (10 UK houses and 12 stores).

286 A few studies have reported the presence of oligomeric PFRs in indoor dust and SRM 2585. RDP and
287 BDP have been identified in our dust samples, but not in SRM2585 (Table SI-6). (Brandsma et al., 2013)
288 reported higher concentrations of BDP and RDP in house dust when collected on/around electric items
289 than in distance. Although it has been reported in baby products and car dust since 2011, V6 may have
290 been used in consumer products since the early 1990s considering that SRM 2585 was prepared using a
291 pool of samples collected during mid to late 1990s (Fang et al., 2013; Stapleton et al., 2011). An average
292 concentration of 117 ± 6 ng g⁻¹ for V6 was reported by (Fang et al., 2013) in SRM2585 with LC-APCI-
293 MS/MS, two-fold higher than our result (47 ± 23 ng g⁻¹) where LC-ESI-MS/MS was employed for
294 instrumental analysis. Since TDBPP, iDPP, and TXP were also present in SRM 2585 with very low
295 levels (Table SI-6), we can assume their commercial use has been ongoing earlier than has been
296 generally perceived or that such compounds may be impurities of PFRs, such as TPHP, TMPP and
297 EHDPHP (Derouet et al., 1996; UK Environment Agency, 2009a).

298 **3.5 iDPP and TXP commercial mixtures**

299 According to the UK Environment Agency (2009), iDPP, an alkyl diaryl phosphate ester, is
300 manufactured in the UK and distributed by Ferro UK Ltd. and ICL-IP Europe B.V. in unknown amounts
301 so far and is used as a FR plasticiser in flexible PVC, synthetic rubber, textiles and pigment products.
302 The registered trademarks for iDPP available in Europe are Phosflex[®] 390 and Santicizer[®] 148 with the
303 commercial mixture composition set as 90% iDPP and <5% TPHP as a technical mixture impurity (UK
304 Environment Agency, 2009b). Newer PFRs such as iDPP have a general impurity due to their

305 manufacture process which potentially causes a diverse contamination profile indoors with similarly
306 structured PFRs, e.g. iDPP with EHDPHP. In the present study, iDPP highest concentrations were found
307 in British-based toys ($145,000 \text{ ng g}^{-1}$) and kitchenware stores ($15,000 \text{ ng g}^{-1}$). Extensive use of laminated
308 wooden flooring, plasticised vinyl polymer products and displays was observed in the two stores. As
309 legislation on the use of PBDEs and their alternatives in consumer products gets stricter, higher levels
310 of “newer” FR are likely to be observed in the indoor environment, including iDPP. TCEP and TDCIPP
311 will be partly restricted to 0.1% by weight in children’s products from 2017 by Washington State (USA)
312 (State of Washington, 2016), which might pave the way for replacement of earlier PBDE alternatives
313 with newer FRs in consumer products. We may also assume that low levels of iDPP in Norwegian
314 house dust could be due to limited commercial availability of iDPP in consumer products in the
315 Norwegian market by comparison with the UK.

316 Trixylenyl phosphate (TXP) is a triaryl phosphate ester currently manufactured by Chemtura Inc.
317 (formerly Great Lakes Chemical Corp., USA) under the registered trademark Kronitex[®] TXP (Chemtura
318 Corp, 2013) and by ICL-IP Ltd. (Israel) as Syn-O-Ad[®] 8475 (ICL IP Inc., 2008) with an estimated usage
319 in Europe between 100 – 1000 tonnes/year (ECHA, 2015). In 2013, the European Chemicals Agency
320 (ECHA) indicated the use of TXP as a tricresyl phosphate (TCP) substitute and formally listed it as a
321 “substance of very high concern” because of its potential reproductive toxicity (ECHA, 2013). Xylenols
322 such as TXP are naturally derived alcohols with recommended application in wire and cable insulation,
323 fire resistant lubricants and PVC applications where low volatility and high resistance products are
324 essential (Harper, 2003). In our study, TXP maximum concentration ($5,800 \text{ ng g}^{-1}$) was reported in a
325 dust sample collected from a computer store. The store’s interior design was covered with PVC and
326 carpet flooring, numerous computer displays and repair rooms where cables and wires are frequently
327 found.

328 **3.6 Correlation between FRs present in dust from different environments**

329 Spearman’s correlation revealed significant and positive correlations among low brominated PBDEs in
330 all environments (Fig.2) in agreement with (Cequier et al., 2014) as they formulate a group of
331 compounds with similar structural and physico-chemical characteristics and are present in the same
332 commercial mixtures. In the occupational environment, where oligomeric PFRs were more abundant
333 than PBDEs and EHFRs, iDPP, RDP and BDP were strongly correlated between each other, EHDPHP
334 and TPHP ($\rho > 0.9$; $p < 0.01$) and TnBP, TCEP and TBOEP ($0.6 < \rho < 0.8$; $p < 0.01$) also occurred together
335 probably due to their application as plasticisers and FRs in similar consumer products and RDP being
336 used as TCEP substitute due to its low release to the environment (van der Veen and de Boer, 2012).
337 Also, TPHP is present in the indoor environment either as an individual FR or as an impurity in the BDP
338 and RDP technical mixtures (Mihajlović, 2015; UK Environment Agency, 2009c; van der Veen and de
339 Boer, 2012). In the UK house environment, V6 was highly correlated with TCEP ($\rho > 0.7$; $p < 0.01$)

340 probably due to TCEP impurity in V6 formulation, while no significant correlation was achieved for
341 Norway, where TCEP use has significantly decreased since 2003 (van der Veen and de Boer, 2012). In
342 UK houses, oligomeric PFRs including RDP and BDP, were strongly correlated with each other ($\rho > 0.9$;
343 $p < 0.01$), while only BDP was correlated with BDE-209 ($\rho > 0.79$; $p < 0.01$), although they are both
344 proposed as Deca-BDE alternatives in electronics (Ballesteros-Gómez et al., 2014). In Norwegian
345 houses, TXP was moderately to highly correlated with Tetra-BDEs, Hepta-BDEs, and BDE-209
346 ($0.6 > \rho > 0.7$; $p < 0.01$), with *anti*-DP and TCEP ($\rho > 0.7$; $p < 0.01$) and with TDCIPP ($\rho > 0.7$; $p < 0.05$).
347 Alpha- and β -TBECH isomers were highly correlated with each other and BDE-28 ($\rho > 0.9$; $p < 0.01$) in
348 all environments and with BEH-TEBP in the occupational environment ($\rho > 0.7$; $p < 0.01$) may be caused
349 by the banned Tri-BDE formulations and parallel Firemaster 550[®] or Great Lakes DP-45TM and
350 Firemaster[®] BZ-54 applications in electronic products.

351 **3.7 Human exposure assessment**

352 Different scenarios of human exposure via dust ingestion have been estimated for oligomeric PFRs in
353 the present study (Table-1, Tables SI-15, SI-16, and SI-17). More information on equations and
354 parameters used for exposure estimates are presented at SI Table 14. To the best of our knowledge, this
355 is the first study providing multi-scenario exposure assessment for this wide variety of FRs based on the
356 same samples from two different countries. However, we recognise that the small number and
357 representativeness of samples analysed in the present study represents a major uncertainty in these intake
358 calculations.

359 In all scenarios (Tables SI-15, SI-16, and SI-17), much higher intakes from dust ingestion have been
360 calculated for m-PFRs than for PBDEs, EHFRs and o-PFRs. Toddlers were found to have much higher
361 estimated exposure to all FR than adults, due to higher dust ingestion rates (average exposure scenario
362 20 and 50 mg per 24 h, for adults and toddlers, respectively; high exposure scenario, 50 and 200 mg per
363 24 h for adults and toddlers, respectively) and lower body weight (12.3 kg for toddlers and 70 kg for
364 adults). Close-to-floor activity and more frequent hand-mouth-contact are rationales behind using higher
365 dust ingestion rates for toddlers. In the worst case scenario, estimated exposure of British toddlers from
366 dust ingestion were 890, 17,900, and 650 ng kg bw⁻¹ day⁻¹ for Σ PBDEs, Σ m-PFRs and Σ EHFRs,
367 respectively; while the estimated exposures for Norwegian toddlers were equal to 60, 1,600, and 40 ng
368 kg bw⁻¹ day⁻¹ for Σ PBDEs, Σ m-PFRs and Σ EHFRs, respectively. Contrary to the exposure of Norwegian
369 toddlers, the estimated exposure of BDE209 for British toddlers in the worst case scenario was equal to
370 820 ng kg bw⁻¹ day⁻¹, about 12% of the daily reference dose (RfD) (7,000 ng kg bw⁻¹ day⁻¹) (Table SI-
371 17). Based on our assessment, Norwegian stay-home adults and toddlers, have one order of magnitude
372 lower exposure of Σ PBDEs and Σ m-PFRs from average dust ingestion (50 mg) than British adults and
373 toddlers. For TBOEP exposure with average dust intake rate, Norwegian stay-home adults (median 5.3
374 ng kg bw⁻¹ day⁻¹) and toddlers (median 75 ng kg bw⁻¹ day⁻¹) were two-fold higher compared to British

375 counter parts (2.3 and 33 ng kg bw⁻¹ day⁻¹ respectively). However, Norwegian adults and toddlers were
376 found to have lower exposure for other m-PFRs and o-PFRs, such as TPHP and BDP, set below the
377 proposed RfD values (Table SI-16&17). (Ali et al., 2013) reported slightly higher exposure to ΣPBDEs
378 for both adult and toddler via house dust ingestion in Kuwait compared to Norway, but lower than our
379 British non-workers. For Σm-PFRs and ΣEHFRs, the calculated intake for adults and toddlers from
380 Kuwait and Pakistan were lower compared to our study for Norwegians and British non-workers. In
381 another study from Norway, slightly higher median exposure of ΣPBDEs (female 0.4 ng kg bw⁻¹ day⁻¹,
382 children 1 ng kg bw⁻¹ day⁻¹) and Σm-PFRs (female 16 ng kg bw⁻¹ day⁻¹, children 133 ng kg bw⁻¹ day⁻¹)
383 were reported compared to our assessment for Norwegians (Cequier et al., 2014), but lower than our
384 British stay-home adult.

385 With 8 h of exposure (50 mg day⁻¹ dust intake rate), the estimated exposures of British workers for
386 ΣPBDEs, Σm-PFRs and ΣEHFRs were higher than Norwegian non-workers (Table SI-15&16).
387 Estimated exposure for British workers to ΣEHFRs was nearly two-fold higher (median 0.55 ng kg bw⁻¹
388 day⁻¹) than British stay-home adults (median 0.36 ng kg bw⁻¹ day⁻¹), unlike ΣPBDEs and Σm-PFRs
389 exposures in these population groups. Given the small sample size in our study from UK stores and
390 offices, we present here FR estimated intakes for the two dust sample groups combined as a general
391 exposure scenario for British employees with 8h exposure duration. A more elaborate view on estimated
392 intakes for all FRs for individual offices and stores is available in SI. Briefly, in average and high dust
393 ingestion rate scenarios, exposure for ΣPBDEs, Σm-PFRs and ΣEHFRs between UK office employees
394 (SI Table 18) and UK stores employees (SI Table 19) were found to be within a comparable range. In
395 worst case scenario, estimated intakes for British-based toys store employees were 14-fold lower for
396 EHDPHP than TBOEP, reaching 30 and 434 ng kg bw⁻¹ day⁻¹, respectively. The second highest
397 estimated intake for TBOEP was found for employees in a British-based store selling office supplies,
398 electronics and furniture equal to 147 and 368 ng kg bw⁻¹ day⁻¹ in average and high dust ingestion rates,
399 respectively. The two cases of store employees (in toys store and office supplies store) did not exceed
400 the proposed RfD for TBOEP (1.5x10⁴ ng kg bw⁻¹ day⁻¹) in both dust ingestion rate scenarios and 8h of
401 exposure. Human exposure via dust ingestion has never been estimated for most o-PFRs, except BDP
402 and RDP (Brandsma et al., 2013). Among all o-PFRs, in most scenarios, the highest intakes via dust
403 ingestion were calculated for iDPP, followed by BDP or TXP (Table 1). Considering 8h of exposure
404 during a workday, British employees were found to have higher estimated exposure of individual o-
405 PFRs than British and Norwegian stay-home adults (24 h) (Table 1). The worst-case scenario for iDPP
406 was estimated for employees in a British-based toy store, where the estimated exposure was 34.6 ng kg
407 bw⁻¹ day⁻¹, nearly three-fold higher than the average dust intake scenario, set considerably below the
408 proposed LOAEL (Table 1). In the worst case scenario for toddlers, Norwegian toddlers may have an
409 exposure of equal to 11.3 ng kg bw⁻¹ day⁻¹ for BDP, while British toddlers have TXP exposure equal to
410 8.7 ng kg bw⁻¹ day⁻¹. In contrast, for Dutch and Greek toddlers (worst case scenario), higher BDP

411 exposures were reported equal to 1,100 ng kg bw⁻¹ day⁻¹ and 750 ng kg bw⁻¹ day⁻¹, respectively; while
412 their RDP exposure were also thousand-fold higher than our assessment (Brandsma et al., 2013). Based
413 on findings in this study, exposure to TDBPP does not seem to raise major toxicological concerns for
414 humans, as TDBPP was rarely detected in our dust samples or other environmental samples (Lopez et
415 al., 2011).

416 **4. Conclusions**

417 Our study reports levels of legacy and alternative FRs in house dust samples from Norway and the UK,
418 as well as from British stores and offices. The median levels of m-PFRs were found to be considerably
419 higher in all environments compared to EHFRs, PBDEs and o-PFRs. Due to higher FR concentrations
420 in British house dust samples, the estimated human intakes for FRs for toddlers in Britain were found
421 to be higher than toddlers in Norway. However, the small number and representativeness of samples
422 analysed in the present study should be carefully considered as it represents a major uncertainty in these
423 intake calculations. In the worst case scenario, BDE209 estimated intake for British toddlers did not
424 exceed the proposed RfD, yet it was considerably higher than for Norwegian toddlers, thus setting
425 British toddlers more prone to potentially adverse health effects related to BDE209 exposure compared
426 to Norwegian ones. This is the first study reporting human exposure via dust ingestion for most o-PFRs.
427 Toddler estimated intakes for o-PFRs were found to be higher than stay-home adults in both countries.
428 In the worst case scenario, iDPP estimated intake for employees in a British-based toy store was
429 considerably higher than for other o-PFRs, together with TDBPP and TXP. This is the first study
430 reporting considerable concentrations of iDPP and TXP in the indoor environment of Norway and the
431 UK. iDPP and TXP together with other halogen-free alternatives such as EHDPHP, are likely to be
432 considered in the future as substances of high toxicological interest for two reasons: a) their potential
433 for human exposure via dust ingestion is considerable and b) their toxicological potency to humans
434 remains unresolved. TXP reproductive toxicity to humans has been reported (ECHA, 2013; Latendresse
435 et al., 1994), while signs of teratogenic alterations have been observed when iDPP and EHDPHP were
436 orally administered in rodents (Robinson et al., 1986) . Also, inhalation has been proposed as a
437 significant route of exposure for several m-PFRs (Cequier et al., 2015; Schreder et al., 2016). Therefore,
438 future research should be considered on the possible adverse health effects of o-PFRs in humans and
439 potential alternative routes of exposure such as inhalation and dermal uptake, as well as measuring their
440 levels in the indoor environment.

441 **Acknowledgments**

442 The research leading to these results has received funding from the European Union Seventh Framework
443 Programme FP7/2007–2013 under grant agreement n° 316665 (A-TEAM project). The authors would

444 like to acknowledge Dr. Eleni Papadopoulou from NIPH (Norway) for her help during the ATEAM
445 sampling campaign. Walid Maho and Dr. Alin Ionas are acknowledged for the support on instrumental
446 analysis. Fuchao Xu acknowledges the financial support of FWO travel grant for study
447 abroad (V422716N) during the manuscript-writing period. Katerina Kademoglou would like to thank
448 prof. Adrian C. Williams for his useful comments on the manuscript and acknowledge the financial
449 support from the Mediterranean Scientific Association of Environmental Protection (MESAEP) during
450 her PhD studies by means of the Emmanuel Lahaniatis Award for Young Scientists.

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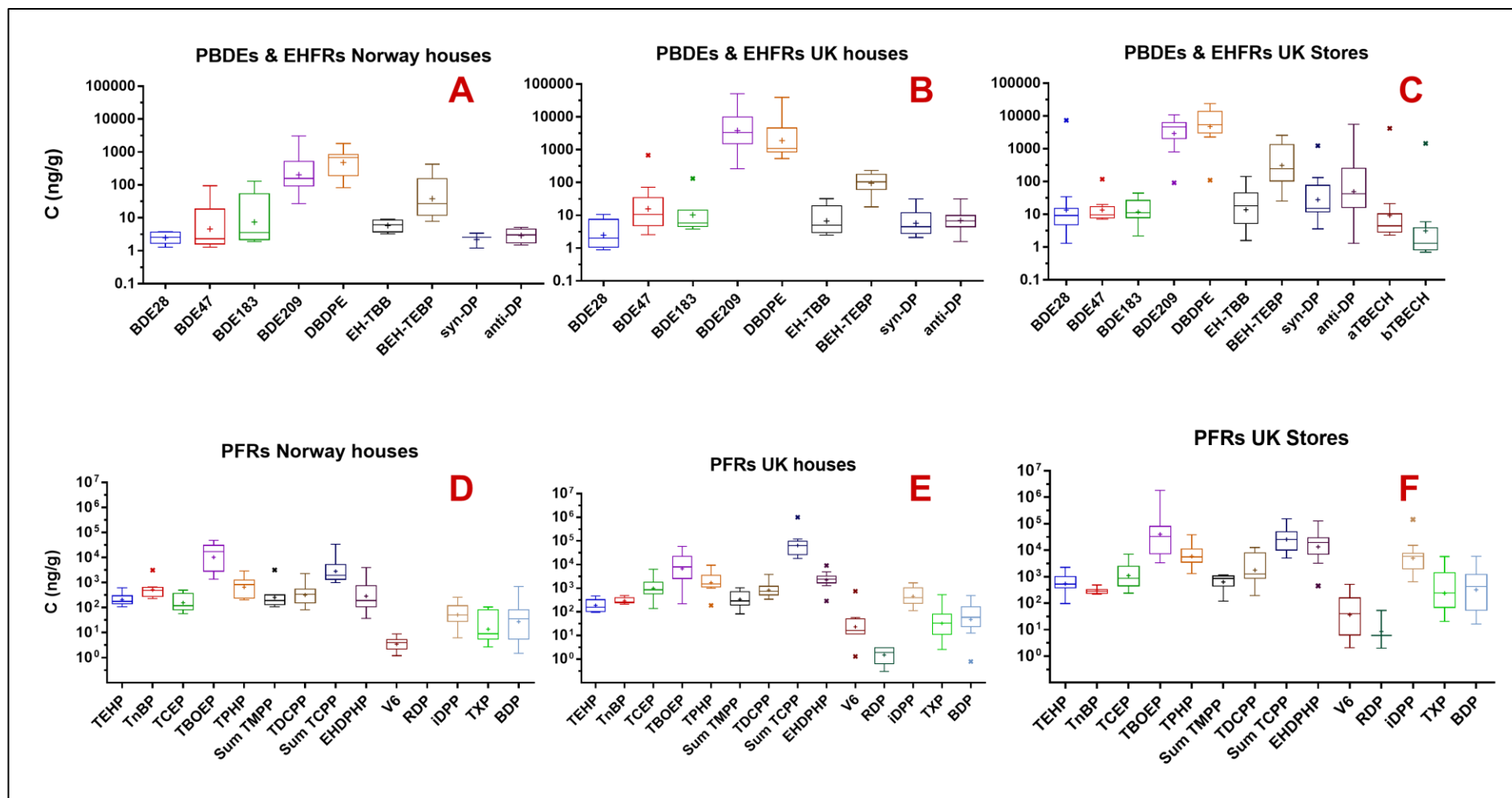
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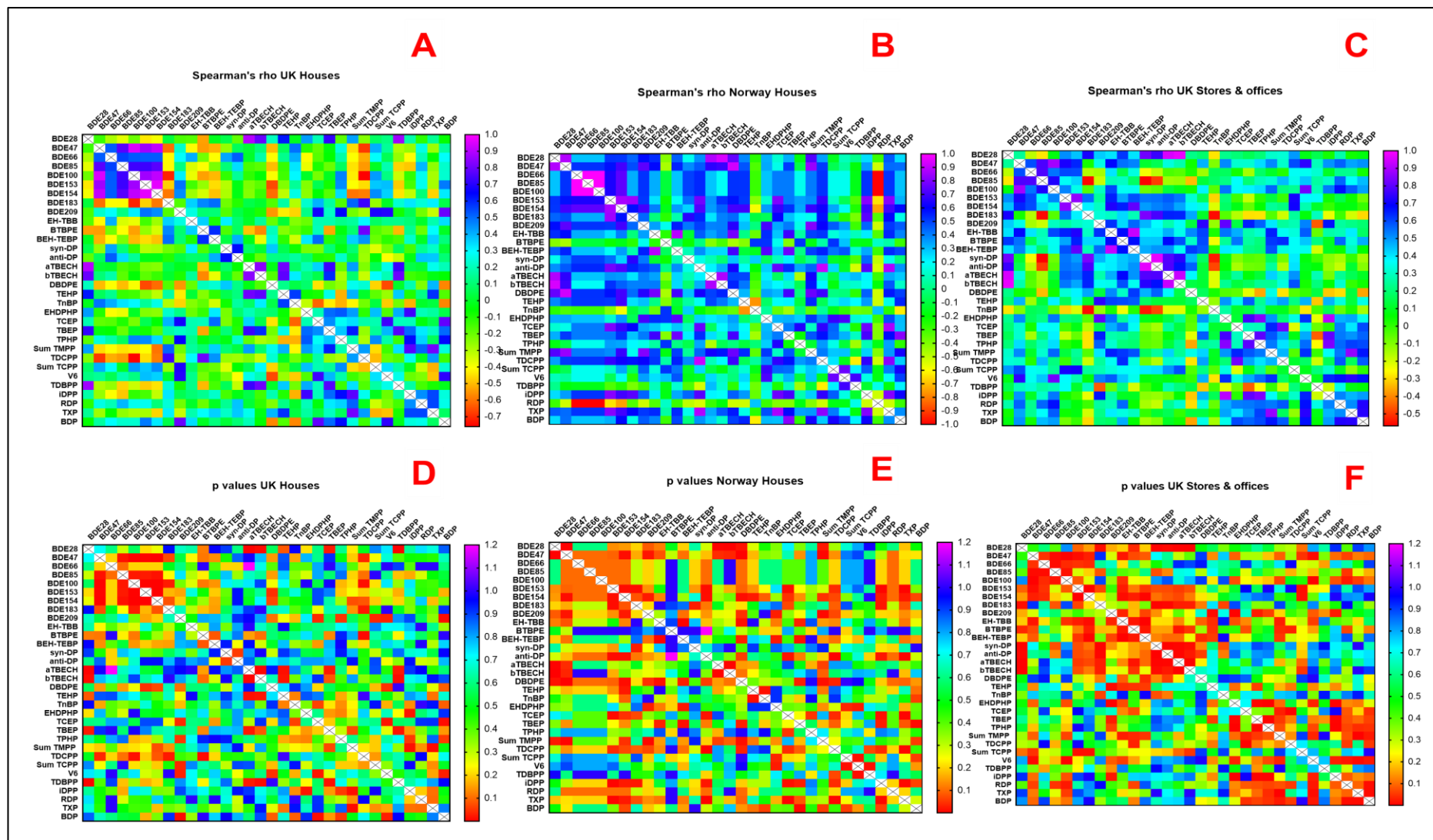
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720 Figure 1 - Boxplots of indoor dust concentrations for selected PBDEs, EHFRs and PFRs from Norwegian houses (A&D) (N=10), UK houses (B&E) (N=10) and
 721 UK stores and offices (C&F) (N=12). Shown in the whiskers are 25th and 75th percentiles, median (central line), mean (+ symbol) and outlier (x symbol) values.
 722 All data shown are log transformed. Please note the linear scale for concentration (ng g⁻¹) on y axis.



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Figure 2 - Heat maps presenting Spearman's rank correlation (ρ) and p-values for all FRs in UK houses (2A&D,) Norwegian houses (2B&E) and UK stores and offices (2C&F).

726 Table 1 - The estimated daily human intake (median and maximum) to selected PFRs in different scenario (ng kg bw⁻¹ day⁻¹).

Human exposure assessment for selected PFRs (ng kg bw⁻¹ day⁻¹)											
	Compound	Stay-home toddler (t=24h)				Stay-home adult (t=24h)				Adult at work (t=8h)	
		UK houses		Norway houses		UK houses		Norway houses		UK offices and Stores	
		Median	Maximum	Median	Maximum	Median	Maximum	Median	Maximum	Median	Maximum
Normal exposure scenario (dust intake 50 mg/d)	V6	0.067	3.073	0.016	0.036	0.005	0.216	0.001	0.003	0.004	0.049
	TDBPP	0.002	0.002	0.002	0.003	<0.001	<0.001	<0.001	<0.001	<0.001	0.001
	iDPP	1.63	6.858	0.209	1.065	0.115	0.482	0.015	0.075	0.562	13.853
	RDP	0.008	0.013	0.007	0.007	0.001	0.001	0.001	0.001	0.001	0.005
	TXP	0.108	2.183	0.037	0.427	0.008	0.153	0.003	0.03	0.023	0.554
	BDP	0.272	1.972	0.144	2.833	0.019	0.139	0.01	0.199	0.046	0.565
High exposure scenario (dust intake 200 mg/d)	V6	0.27	12.293	0.066	0.143	0.012	0.54	0.003	0.006	0.010	0.122
	TDBPP	0.008	0.008	0.01	0.011	<0.001	<0.001	<0.001	0.001	0.001	0.004
	iDPP	6.52	27.431	0.834	4.26	0.286	1.205	0.037	0.187	1.404	34.632
	RDP	0.031	0.05	0.029	0.029	0.001	0.002	0.001	0.001	0.001	0.013
	TXP	0.431	8.732	0.148	1.707	0.019	0.384	0.007	0.075	0.058	1.386
	BDP	1.086	7.886	0.576	11.333	0.048	0.346	0.025	0.498	0.115	1.412

727 * Normal scenario was considered for dust intake of 20 mg and 50 mg per 24 h, for adults and toddlers, respectively; while for high exposure scenario, 50 mg and 200 mg per 24
728 h, respectively, dust exposures were considered for stay-home adults and toddlers. Daily exposure for working adults in UK stores and offices has been estimated as well using
729 the same parameters (median, maximum, ingestion rate), but for 8h exposure duration. Body weights, 70 kg for adults and 12.3 kg for toddlers, were applied for the estimation