

Supplementary Information for:

Concentrations of Halogenated Flame Retardants and Polychlorinated Biphenyls in house dust from Lagos, Nigeria

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Table S1a: GC oven temperature program for Brominated Flame Retardants (BFRs)

| | Rate (°C / min) | Temperature (°C) | Hold time (min) | Run time (min) |
|----------------|------------------------|-------------------------|------------------------|-----------------------|
| Initial | | 80 | 2 | 2 |
| Ramp 1 | 20 | 170 | 5.5 | 10 |
| Ramp 2 | 25 | 320 | 10 | 16 |

Table S1b: GC oven temperature program for Polychlorinated Biphenyls (PCBs)

| | Rate (°C / min) | Temperature (°C) | Hold time (min) | Run time (min) |
|----------------|------------------------|-------------------------|------------------------|-----------------------|
| Initial | | 65 | 0.75 | 0.75 |
| Ramp 1 | 20 | 250 | 1 | 11 |
| Ramp 2 | 5 | 250 | 0 | 13 |
| Ramp 3 | 30 | 320 | 1 | 16 |

Table S2a: m/z ions monitored for target BFRs and PCBs

| BDEs | | | NBFRs | | | PCBs | | |
|----------------|-------|-------|----------------|-------|-------|----------------|-------|-------|
| BDE 28 | 405.8 | 407.8 | PBBz | 471.6 | 469.5 | PCB 11 | 222 | 224 |
| BDE 47 | 483.7 | 485.7 | PBT | 487.7 | 485.7 | PCB 28 | 255.9 | 257.9 |
| BDE 100 | 403.8 | 405.8 | PBEB | 499.7 | 501.7 | PCB 52 | 289.9 | 291.9 |
| BDE 99 | 403.8 | 405.8 | HBBz | 549.7 | 547.7 | PCB 101 | 325.9 | 327.9 |
| BDE 154 | 483.7 | 485.7 | Syn-DP | 271.8 | 269.8 | PCB 118 | 325.9 | 327.9 |
| BDE 153 | 483.7 | 485.7 | Anti-DP | 271.8 | 269.8 | PCB 153 | 359.9 | 361.9 |
| BDE 183 | 561.5 | 563.5 | DPDPE | 484.6 | 486.6 | PCB 138 | 359.9 | 361.9 |
| BDE 209 | 799.4 | 801.4 | | | | PCB 180 | 393.8 | 395.8 |

Table S2b: Precursor to product ion m/z transitions monitored for HBCDDs

| Diastereomer | Ion transition (m/z) |
|--|-----------------------------|
| α -, β -, γ -HBCDD | 640.6 \rightarrow 78.0 |
| ^{13}C α -, β -, γ -HBCDD | 652.4 \rightarrow 79.0 |
| d_{18} γ -HBCDD | 657.6 \rightarrow 78.9 |

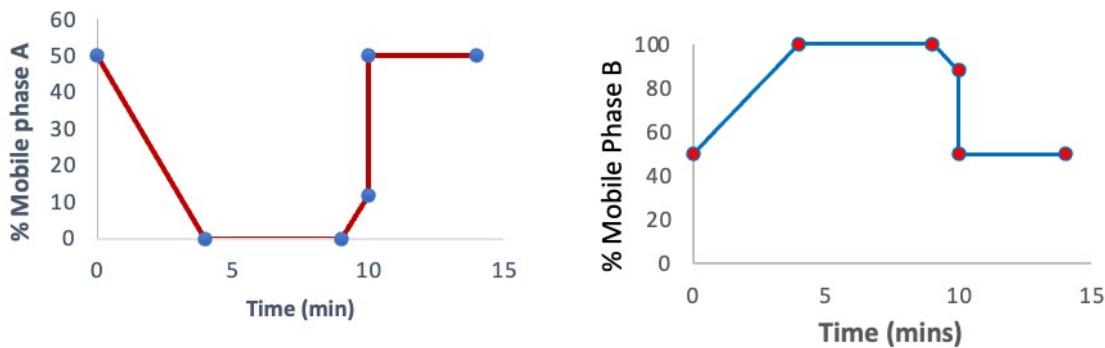


Figure S1: Mobile phase gradient for the analysis of HBCDDs

* Mobile phase A: methanol : water (1:1 v/v)
Mobile phase B: methanol

Table S3a: Concentration of PBDEs determined in SRM 2585 ($n=10$)

| | Mean±Std | Cert. value ^a |
|---------|------------|--------------------------|
| BDE 28 | 35.9±4.9 | 46.9±4.4 |
| BDE 47 | 508.0±30 | 498±46 |
| BDE 99 | 1004.6±97 | 892±53 |
| BDE 100 | 175.1±11 | 145±11 |
| BDE 153 | 153.9±13 | 119±1 |
| BDE 154 | 87.1±9.1 | 83.5±2.0 |
| BDE 183 | 39.9±2.6 | 43.5±3.5 |
| BDE 209 | 2228.8±310 | 2510±190 |

Table S3b: Concentration of PCBs determined in SRM 2585 ($n=10$)

| Congener | Mean±Std | Cert. value ^a |
|----------|----------|--------------------------|
| PCB 28 | 12.3±0.9 | 13.4 ± 0.5 |
| PCB 52 | 23.8±1.4 | 21.8 ± 1.9 |
| PCB 101 | 26.6±1.4 | 29.8 ± 2.3 |
| PCB 118 | 26.8±1.6 | 26.3 ± 1.7 |
| PCB 153 | 38.0±1.7 | 40.2 ± 1.8 |
| PCB 138 | 33.6±2.5 | 27.6 ± 2.1 |
| PCB 180 | 17.7±3.2 | 18.4 ± 3.2 |

^a Stapleton et al., 2006

Table S4a: Target BFRs and the corresponding Internal standards

| BFRs | Internal Standard (IS) (Concentrations – 500 ng/mL) | Recovery Determination Standard (RDS) (Concentrations – 500 ng/mL) |
|-----------------|--|---|
| BDE 28 | | |
| BDE 47 | | |
| BDE 99 | | |
| BDE 100 | | |
| PBBz | BDE 77 | |
| HBBz | | |
| PBT | | |
| PBEB | | ¹³ C – BDE - 100 |
| BDE 153 | | |
| BDE 154 | | |
| BDE 183 | BDE 128 | |
| <i>Anti</i> -DP | | |
| <i>Syn</i> -DP | | |
| BDE-209 | | |
| DPDPE | ¹³ C - BDE 209 | |
| α -HBCDD | ¹³ C – α - HBCDD | |
| β -HBCDD | ¹³ C – β - HBCDD | |
| γ -HBCDD | ¹³ C – γ - HBCDD | d ₁₈ - γ -HBCDD |

Table S4b: Target PCBs and the corresponding Internal standards

| PCBs | Internal Standards (Concentrations – 200 ng/mL) | Recovery Determination Standard (Concentrations – 200 ng/mL) |
|---------|--|--|
| PCB 11 | PCB 14 | |
| PCB 28 | PCB 34 | PCB 29 |
| PCB 52 | PCB 62 | |
| PCB 101 | PCB 119 | |
| PCB 118 | | |
| PCB 153 | PCB 147 | PCB 129 |
| PCB 138 | | |
| PCB 180 | PCB 173 | |

Table S4c: Recoveries of Internal Standards

| IS | Recoveries |
|---------------------------|------------|
| BDE 77 | 110±11 |
| BDE 128 | 71±21 |
| ¹³ C - BDE 209 | 72±23 |
| α -HBCDD | 76±16 |
| β -HBCDD | 68±12 |
| γ -HBCDD | 75±25 |
| PCB 14 | 56±14 |
| PCB 34 | 67±16 |
| PCB 62 | 70±11 |
| PCB 119 | 89±14 |
| PCB 147 | 82±12 |
| PCB 173 | 88±15 |

Table S5a: Limits of quantification for target PBDEs

| PBDE | LOQ (ng/g) | Detection frequency (%) |
|---------|------------|-------------------------|
| BDE 28 | 0.78 | 0 |
| BDE 47 | 0.74 | 93 |
| BDE 100 | 0.21 | 67 |
| BDE 99 | 0.19 | 87 |
| BDE 154 | 0.26 | 100 |
| BDE 153 | 0.45 | 100 |
| BDE 183 | 0.49 | 100 |
| BDE 209 | 16 | 93 |

Table S5b: Concentrations of target PBDEs

| Sample # | Concentrations (ng/g) | | | | | | | | Σ_7 BDEs |
|----------|-----------------------|-----------|-------------|------------|------------|---------|-----------|-------------|-----------------|
| | BDE 28 | BDE 47 | BDE 100 | BDE 99 | BDE 154 | BDE 153 | BDE 183 | BDE 209 | |
| 1 | < 0.78 | <1.5 | < 0.21 | 7.7 | 1.8 | 2.0 | 2.6 | 260 | 280 |
| 2 | < 0.78 | 2.3 | < 0.21 | <0.19 | 0.64 | 1.5 | 4.5 | 290 | 300 |
| 3 | < 0.78 | 4.8 | 1.5 | 7.7 | 1.8 | 2.5 | 3.4 | 300 | 320 |
| 4 | < 0.78 | 1.6 | 1.6 | 4.3 | 1.1 | 2.0 | 3.1 | 540 | 550 |
| 5 | < 0.78 | 6.4 | < 0.21 | <0.19 | 4.6 | 4.6 | 3.9 | 280 | 300 |
| 6 | < 0.78 | 6.2 | 2.9 | 13 | 3.4 | 4.5 | 2.2 | 62 | 94 |
| 7 | < 0.78 | 6.3 | 2.0 | 6.2 | 0.75 | 1 | 8.1 | 130 | 150 |
| 8 | < 0.78 | 4.6 | 1.8 | 7.5 | 1.2 | 6.2 | 19 | 500 | 540 |
| 9 | < 0.78 | 14 | < 0.21 | 11 | 0.97 | 1.3 | 0.71 | <16 | 43 |
| 10 | < 0.78 | 5.5 | 1.8 | 6.9 | 1.7 | 1.9 | 3.7 | 130 | 150 |
| 11 | < 0.78 | 3.8 | < 0.21 | 5.4 | 0.89 | 1.5 | 3 | 160 | 170 |
| 12 | < 0.78 | 2.7 | 1.4 | 7.0 | 1.4 | 2.1 | 4 | 310 | 330 |
| 13 | < 0.78 | 2.2 | 1.1 | 5.4 | 1.2 | 1.8 | 3.1 | 430 | 450 |
| 14 | < 0.78 | 5.6 | 1.5 | 5 | 0.89 | 1.3 | 4.5 | 170 | 190 |
| 15 | < 0.78 | 20 | 3.1 | 12 | 1.2 | 1.5 | 2.0 | 770 | 810 |
| Mean | < 0.78 | 5.8 | 1.3 | 6.6 | 1.6 | 2.4 | 4.5 | 290 | 310 |
| Median | < 0.78 | 4.8 | 1.5 | 6.9 | 1.2 | 1.9 | 3.4 | 280 | 300 |
| Range | < 0.78 | <1.5 - 20 | <0.21 - 3.1 | <0.19 - 13 | 0.64 - 4.6 | 1 - 6.2 | 0.71 - 19 | <0.16 - 770 | 43 - 810 |

Table S5c: % Composition of each target PBDE congener to Σ_7 BDEs

| | BDE 47 | BDE 100 | BDE 99 | BDE 154 | BDE 153 | BDE 183 | BDE 209 |
|--------|----------|----------|-----------|-----------|-----------|-----------|---------|
| Mean | 4.1 | 0.6 | 4.4 | 0.8 | 1.2 | 1.8 | 87 |
| Median | 2.2 | 0.3 | 2.4 | 0.5 | 0.74 | 1.5 | 93 |
| Range | 0.3 - 32 | 0.05 - 3 | 0.06 - 25 | 0.2 - 3.6 | 0.2 - 4.8 | 0.2 - 5.4 | 35 - 98 |

Table S6a: Limits of quantification for target HBCDDs

| Isomer | LOQ (ng/g) | Detection frequency (%) |
|-----------------|------------|-------------------------|
| α -HBCDD | 5.3 | 13 |
| β -HBCDD | 11 | 0 |
| γ -HBCDD | 2.4 | 27 |

Table S6b: Concentrations of target HBCDDs

| Sample # | Conc. (ng/g) | | |
|---------------|-------------------|----------------|-------------------|
| | α -HBCDD | β -HBCDD | γ -HBCDD |
| 1 | <5.3 | <11 | 7.7 |
| 2 | <5.3 | <11 | <2.4 |
| 3 | <5.3 | <11 | <2.4 |
| 4 | 37 | <11 | 17 |
| 5 | <5.3 | <11 | 19 |
| 6 | <5.3 | <11 | <2.4 |
| 7 | <5.3 | <11 | <2.4 |
| 8 | 40 | <11 | 26 |
| 9 | <5.3 | <11 | <2.4 |
| 10 | <5.3 | <11 | <2.4 |
| 11 | <5.3 | <11 | <2.4 |
| 12 | <5.3 | <11 | <2.4 |
| 13 | <5.3 | <11 | <2.4 |
| 14 | <5.3 | <11 | <2.4 |
| 15 | <5.3 | <11 | <2.4 |
| Mean | 5.7 | <11 | 5.1 |
| Median | <5.3 | <11 | <2.4 |
| Range | <5.3-40 | <11 | <2.4-19 |

Table S6c: $f_{\alpha\text{-HBCDD}}$ in sample #s 4 and 8

| Sample | $f_{\alpha\text{-HBCDD}}$ |
|--------|---------------------------|
| 4 | 0.69 |
| 8 | 0.61 |

Table S7a: Limits of quantitation for target alt-HFRs

| | LOQ (ng/g) | Detection frequency (%) |
|-----------------|-------------------|--------------------------------|
| PBBz | 0.70 | 6.7 |
| PBT | 0.59 | 33 |
| PBEB | 0.71 | 53 |
| HBBz | 0.76 | 47 |
| <i>Syn</i> -DP | 0.10 | 93 |
| <i>Anti</i> -DP | 2.4 | 87 |
| DBDPE | 54 | 53 |

Table S7b: Concentrations of target alt-HFRs

| Sample # | Concentrations (ng/g) | | | | | | | |
|--------------------|------------------------------|--------------------|--------------------|--------------------|----------------------|-----------------------|------------------|-----------------------|
| | PBBz | PBT | PBEB | HBBz | <i>Syn</i>-DP | <i>Anti</i>-DP | DBDPE | $\sum_7 \text{NBFRs}$ |
| 1 | <0.7 | < 0.59 | 0.9 | 0.76 | 6.2 | 25 | 1400 | 1400 |
| 2 | <0.7 | 0.7 | 94 | 1.05 | 9.5 | 53 | 2400 | 2600 |
| 3 | <0.7 | 2.2 | 97 | 1.4 | 5.9 | < 2.4 | < 54 | 140 |
| 4 | <0.7 | < 0.59 | < 0.71 | < 0.76 | 8.7 | 68 | < 54 | 110 |
| 5 | <0.7 | 2.5 | 0.18 | < 0.76 | 18 | 28 | 1000 | 1000 |
| 6 | <0.7 | < 0.59 | < 0.71 | < 0.76 | 26 | 88 | < 54 | 140 |
| 7 | <0.7 | < 0.59 | < 0.71 | < 0.76 | 0.83 | 3.2 | < 54 | 34 |
| 8 | <0.7 | 0.69 | 36 | 3.5 | 7.2 | 21 | 320 | 390 |
| 9 | <0.7 | < 0.59 | < 0.71 | < 0.76 | < 0.1 | < 2.4 | < 54 | 32 |
| 10 | <0.7 | < 0.59 | < 0.71 | < 0.76 | 6.9 | 35 | 280 | 320 |
| 11 | 0.89 | 6.3 | 170 | 2.2 | 9.6 | 99 | < 54 | 320 |
| 12 | <0.7 | < 0.59 | 1.4 | 1 | 8.7 | 22 | 610 | 640 |
| 13 | <0.7 | < 0.59 | 1.5 | 1.1 | 7 | 27 | 820 | 860 |
| 14 | <0.7 | < 0.59 | < 0.71 | < 0.76 | 11 | 42 | 180 | 230 |
| 15 | <0.7 | < 0.59 | < 0.71 | < 0.76 | 11 | 32 | < 54 | 73 |
| Mean | 0.10 | 0.96 | 27 | 0.93 | 9.1 | 36 | 480 | 560 |
| Median | <0.7 | < 0.59 | < 0.71 | < 0.76 | 8.7 | 28 | 180 | 320 |
| | <0.7 – | < 0.59 – | < 0.71 – | < 0.76 – | < 0.1 – | < 2.4 - | < 54 - | |
| Range | 0.89 | 6.3 | 170 | 3.5 | 26 | 99 | 2400 | 32 - 2600 |

Table S7c: % Composition of each target NBFRs congener to $\sum_7 \text{NBFRs}$

| | PBBz | PBT | PBEB | HBBz | <i>Syn</i>-DP | <i>Anti</i>-DP | DBDPE |
|--------|-------------|------------|-------------|-------------|----------------------|-----------------------|--------------|
| Mean | 0.06 | 0.4 | 9.4 | 0.44 | 4.3 | 18 | 68 |
| Median | 0.02 | 0.18 | 0.31 | 0.25 | 2.1 | 6.5 | 85 |
| | 0.01 – | | | 0.03 – | 0.3 – | | |
| Range | 0.002 – 0.3 | 2.0 | 0.03 – 70 | 1.1 | 18 | 1.5 – 64 | 9.1 - 98 |

Table S8a: Limits of quantification for target PCBs

| PCB # | LOQ (ng/g) | Detection frequency |
|--------------|-------------------|----------------------------|
| PCB-11 | 0.19 | 0 |
| PCB-28 | 0.31 | 0 |
| PCB-52 | 0.49 | 87 |
| PCB-101 | 0.32 | 53 |
| PCB-118 | 0.29 | 80 |
| PCB-153 | 0.46 | 93 |
| PCB-138 | 0.57 | 100 |
| PCB-180 | 0.27 | 100 |

Table S8b: Concentrations of target PCBs

| Sample # | Concentration of PCB # (ng/g) | | | | | | | | ΣPCBs |
|-----------------|--------------------------------------|-----------------|-----------------------|-----------------------|-------------------|----------------------|------------------|------------------|-----------------|
| | 11 | 28 | 52 | 101 | 118 | 153 | 138 | 180 | |
| 1 | <0.19 | <0.31 | 1.8 | <0.32 | 0.59 | 1.3 | 4.1 | 1.5 | 9.4 |
| 2 | <0.19 | <0.31 | 2.2 | 0.79 | 1.3 | 0.83 | 2.6 | 0.97 | 8.6 |
| 3 | <0.19 | <0.31 | 3.6 | 0.51 | 1.4 | 2.8 | 8.8 | 2.2 | 19 |
| 4 | <0.19 | <0.31 | 2.1 | <0.32 | <0.29 | 1.2 | 4.1 | 0.93 | 13 |
| 5 | <0.19 | <0.31 | 4.7 | 1.4 | 2.1 | 11 | 22 | 9.2 | 51 |
| 6 | <0.19 | <0.31 | 1.7 | <0.32 | <0.29 | 0.67 | 2.3 | 0.93 | 5.9 |
| 7 | <0.19 | <0.31 | 2.1 | <0.32 | 0.39 | 1.6 | 3.4 | 1.7 | 9.4 |
| 8 | <0.19 | <0.31 | 1.6 | 1.2 | 4.2 | 4.4 | 18 | 5.8 | 35 |
| 9 | <0.19 | <0.31 | 3.8 | <0.32 | <0.29 | 2.5 | 5.5 | 1.7 | 14 |
| 10 | <0.19 | <0.31 | 0.58 | 0.68 | 1.2 | < 0.46 | 0.82 | 0.27 | 4.0 |
| 11 | <0.19 | <0.31 | <0.49 | 1.9 | 1.3 | 6.5 | 13 | 4.2 | 28 |
| 12 | <0.19 | <0.31 | 4.8 | <0.32 | 0.62 | 5.5 | 15 | 6.2 | 32 |
| 13 | <0.19 | <0.31 | 3.9 | <0.32 | 0.55 | 2.9 | 7.5 | 3.2 | 18 |
| 14 | <0.19 | <0.31 | 5.7 | 0.66 | 1.0 | 9.9 | 19 | 24 | 61 |
| 15 | <0.19 | <0.31 | <0.49 | 0.63 | 0.47 | 4.3 | 7.7 | 4.8 | 18 |
| Mean | <0.19 | <0.31 | 2.8 | 0.55 | 1.0 | 3.7 | 9.0 | 4.5 | 22 |
| Median | <0.19 | <0.31 | 2.1 | 0.51 | 0.62 | 2.8 | 7.5 | 2.2 | 18 |
| Range | <0.19 | <0.31 | <0.49 – 7.6 | <0.32 – 1.9 | 0.59 – 4.2 | <0.46 - 11 | 0.82 - 22 | 0.27 - 24 | 3.8 – 61 |

Table S8c: % Composition of each target PCB congener to Σ PCBs

| PCB # | 52 | 101 | 118 | 153 | 138 | 180 |
|--------|--------|--------|--------|----------|---------|----------|
| Mean | 17 | 3.3 | 6.1 | 16 | 40 | 17 |
| Median | 19 | 2.1 | 4.1 | 16 | 42 | 17 |
| Range | 0 – 33 | 0 - 18 | 0 - 31 | 7.1 - 24 | 22 - 51 | 7.1 – 40 |

Reference

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