Supporting Information

# Biomagnification of Higher Brominated PBDE Congeners in an Urban Terrestrial Food Web in North China Based on Field Observation of Prey Deliveries

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**Chemicals.** Individual non-labeled reference standards for PBDEs (BDE-28, -47, -77, -99, -100, -153, -154, -118, -128, -183, -196, -197, -202, -203, -206, -207, -208, -209) were purchased from AccuStandards, Inc. (New Haven, US). $^{13}$C-BDE-209 was obtained from Cambridge Isotope Laboratories (Andover, US). Dichloromethane (CNW technologies GmbH, Germany) and hexane (Honeywell, manufactured in Korea), were HPLC grade. Acetone was of analytical reagent and was re-distilled using glass system. Concentrated sulfuric acid was of guaranteed reagent.

**Cleanup for abiotic samples.** Extraction and clean-up procedures for abiota samples, i.e. grass and soil, were detailed in our previous studies (*1, 2*) with minor modification. For all abiotic samples, BDE-77, BDE-181 and $^{13}$C-BDE-209 as surrogate standards were spiked prior to Soxhlet extraction with 50% acetone in hexane (v/v) for 48 h. For grass individual, about 5 g of sample were natural air dried before extraction. The extracts were rotary-evaporated to 30–40 mL and then treated with concentrated sulfuric acid (60 mL) for 2 h. Target compounds were liquid-liquid extracted (LLE) 3 times (40 mL each) by hexane before overnight standing. Afterwards another twice of LLE were performed for insurance of fully extraction. All hexane fractions were gathered and then concentrated to 1–2mL before further cleanup on a multilayer alumina/silica gel column (*1*). The column was eluted with 80 mL of hexane/dichloromethane (1:1, v/v), and the effluent was finally evaporated to 200 μL in iso-octane. For soil, approximately 30 g of lyophilized samples were sieved and stored at -20 °C before Soxhlet extraction. Activated copper granules were added for removal of elemental sulfur. Concentrated extracts were further purified by using a

multilayer silica column (i.d. 1 cm) packed with neutral silica (8 cm) and 40% silica sulfuric acid (8 cm). The target fraction was obtained by elution with 30 mL hexane / dichloromethane (1:1, v/v) and redissolved to 200 μL in iso-octane after nitrogen dryness. A known amount of internal standards (BDE-118 and BDE-128) were injected to the final extracts of all abotic samples for response calibration before instrumental analysis.

**Instrumental analysis.** PBDEs were analyzed by gas chromatography-electron capture negative ionization mass spectrometry (GC/ECNI-MS) in selected ion monitoring (SIM) mode. For tri- to hepta-BDE congeners, a 30 m × 0.25 mm i.d. × 0.25 μm DB-XLB capillary column (J&W Scientific, CA) on an Agilent 6890 GC-5975 MS system was used and ions m/z = 79 and 81 were monitored. Initial column temperature was held at 110 °C for 1 min, and then increase to 180 °C at 8 °C/min (held for 1 min), to 240 °C at 2 °C/min (held for 5 min), to 280 °C at 2 °C/min (held for 15 min), and to 310 °C at 10 °C/min (held for 5 min). For the determination of octa- to deca-BDEs, a 15 m × 0.25 mm i.d. × 0.10 μm DB-5HT capillary column (J&W Scientific, CA) on a Shimadzu QP2010 series GC-MS system was used and ions m/z = 79 and 81 were monitored for octa- and nona-BDEs while m/z = 484.7/486.7 and 494.7/496.7 were monitored for BDE-209 and $^{13}$C-BDE-209, respectively. GC temperature program was performed as follows: The temperature was initiated at 110 °C (held for 5 min), and increased to 200 °C at 20 °C/min (held for 4.5 min), finally reached to 310 °C at 10 °C/min (held for 15 min).

Table S1. Method detection limit (MDL) in the present study [a]

| Congener | MDL( ng/g lw) | | | MDL(pg/g dw) | |
|---|---|---|---|---|---|
| | CK, ETS | BR | insect | grass | soil |
| BDE-28 | 0.17 | 0.15 | 0.095 | 6.4 | 1.1 |
| BDE-47 | 0.23 | 0.20 | 0.12 | 8.3 | 1.4 |
| BDE-99 | 0.30 | 0.26 | 0.16 | 11 | 1.8 |
| BDE-100 | 0.16 | 0.14 | 0.086 | 5.8 | 1.0 |
| BDE-153 | 0.21 | 0.18 | 0.11 | 7.6 | 1.3 |
| BDE-154 | 0.27 | 0.24 | 0.15 | 9.8 | 1.6 |
| BDE-183 [b] | 0.57 | 0.46 | 0.28 | 19 | 3.2 |
| BDE-196 | 2.0 | 1.8 | 1.11 | 75 | 12 |
| BDE-197 | 1.2 | 1.1 | 0.68 | 46 | 7.6 |
| BDE-201 | 1.2 | 1.1 | 0.66 | 44 | 7.4 |
| BDE-202 | 0.56 | 0.50 | 0.31 | 21 | 3.4 |
| BDE-203 | 2.4 | 2.2 | 1.3 | 89 | 15 |
| BDE-206 | 9.8 | 8.7 | 5.3 | 360 | 60 |
| BDE-207 | 12 | 10 | 6.4 | 430 | 71 |
| BDE-208 | 6.2 | 5.5 | 3.4 | 230 | 38 |
| BDE-209 | 29 | 26 | 16 | 1100 | 180 |

[a] CK: common kestrel; ETS: Eurasian tree sparrow; BR: brown rat; insect: including grasshoppers and dragonflies. [b] a signal-to-noise ratio of ten was used because it was not detectable in procedural blanks.

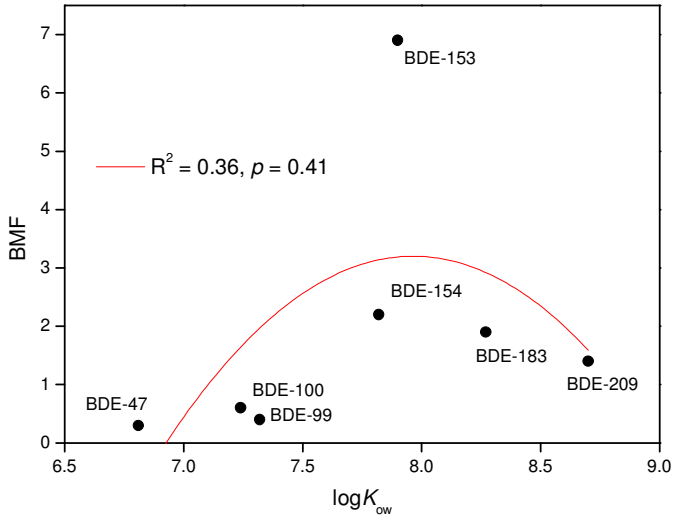


Figure S1. Relationship between BMFs and log $K_{ow}$ of PBDEs. Log $K_{ow}$ values are taken from Braekevelt et al. (*3*) and Wania et al. (*4*)

**Literature Cited**

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