Supporting Information for "Detection, Identification, and Quantification of Hydroxylated Bis(2-ethylhexyl)-Tetrabromophthalate Isomers in House Dust"

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This supporting information provides figures addressing (1) Chromatogram of extracted ions with m/z 640.9946 (10 ppm window) in negative ion using pure methanol as mobile phase; (2) Chromatogram of extracted ions with m/z 640.9946 (10 ppm window) for highly purified standard (AccuStandard, New Haven, CT, USA); (3) Chromatograms and mass spectra for OH-TBPH in positive ion mode; (4) NMR results for purified OH-TBPH; (5) TBPH and OH-TBPH in different fractions from florisil cartridges; (6) Comparison of SIM of full scan mode for detection of OH-TBPH and TBPH.

Purification of OH-TBPH by HPLC fractionation. HPLC fractionation was used to isolate OH-TBPH from technical product BZ-54 which contained only TBB and TBPH compared to FM-550. Fractions were collected at 2-min interval from 0 min to 120 min, and then OH-TBPH2 in each fraction was quantified by use of UHPLC-Q Exactive after 10,000-fold dilution with a mixture of methanol and acetone (v/v, 1:1). Fractions which contained OH-TBPH2 were collected and combined, and then evaporated. Fractionation was conducted by use of a Betasil C18 column (5 μm; 22.1 mm × 150 mm; Thermo Fisher Scientific) which was maintained at 30 °C. The flow rate and the injection volume were 6 mL/min and 100 μL, respectively. Mixture of methanol and ultrapure water (v/v, 8:2) containing 0.1% NH4OH (v/v) was used as mobile phase. After purification, the OH-TBPH2 was characterized by ^{1}H NMR spectra (Figure S4). The purified OH-TBPH2 (0.1 mg/L) was also characterized using UHPLC-Q Exactive with full scan range from m/z 200-2000. The intensity of OH-TBPH2 was 100-folds higher than TBPH, indicated the relatively high purity of the OH-TBPH2 standard (Figure S4).

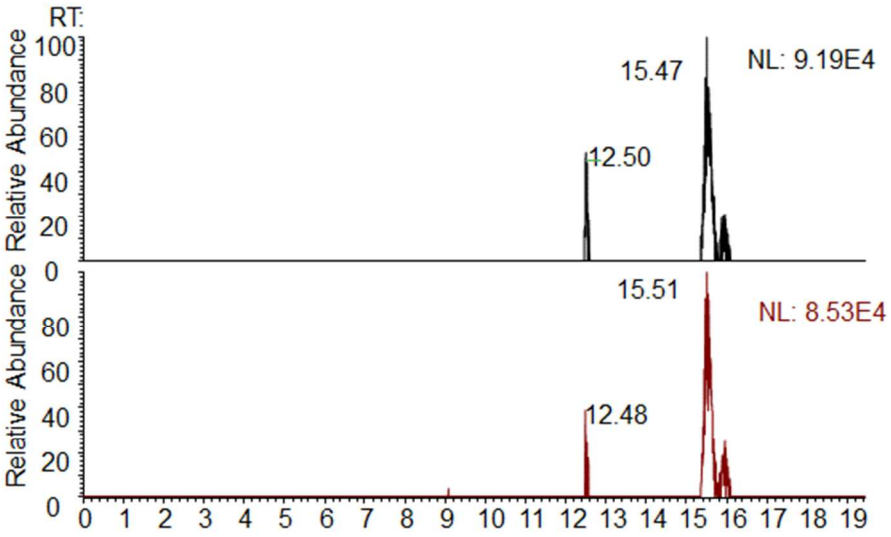

Figure S1. Chromatogram of extracted ions with m/z 640.9946 (10 ppm window) in negative ion mode for commercial standard using pure methanol as mobile phase.

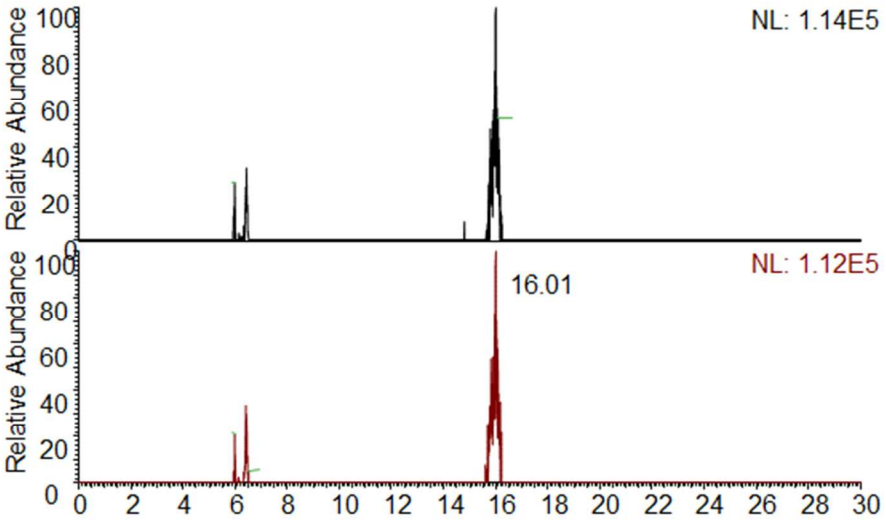

Figure S2. Chromatogram of extracted ions with m/z 640.9946 (10 ppm window) in negative ion mode for highly purified standard (AccuStandard, New Haven, CT, USA).

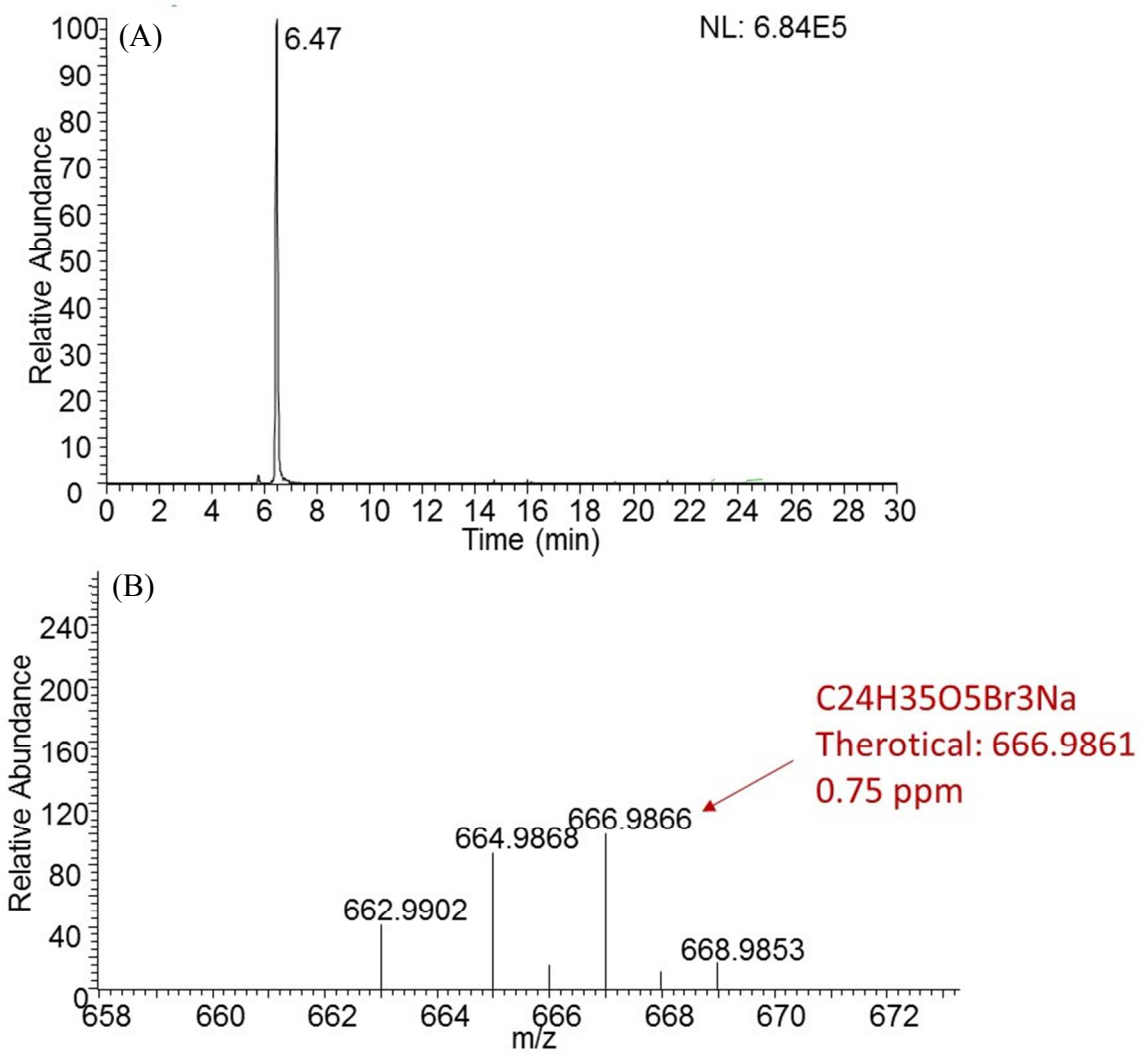

Figure S3. (A) Chromatogram of extracted ions with m/z 666.9861 (10 ppm window) in positive ion mode for BZ-54 standard. (B) Mass spectra of OH-TBPH in positive ion mode with mass error of 0.75 ppm to sodium adduct.

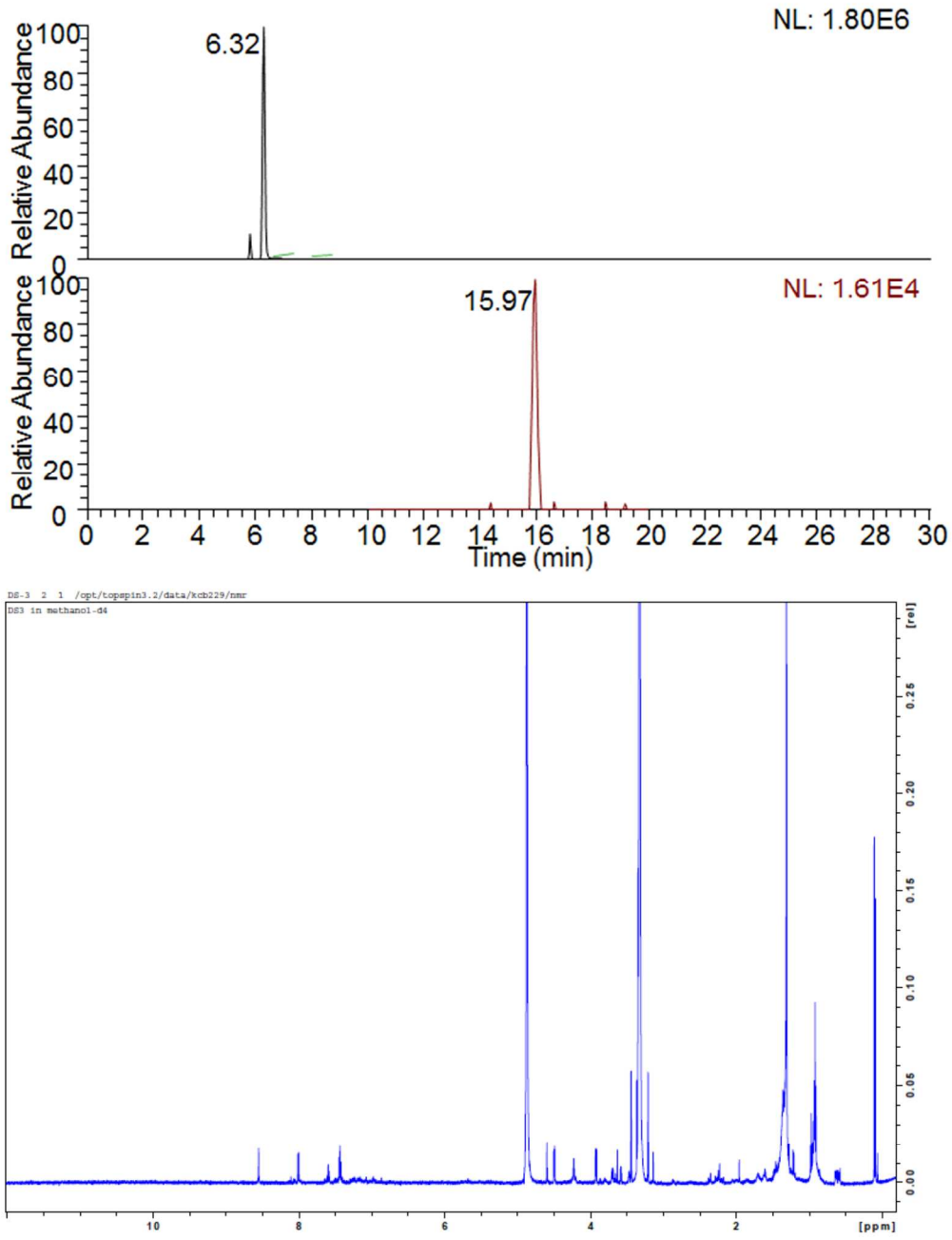

Figure S4. Ultra-High Resolution LC/mass spectrometry (above) and ^{1}H NMR (bottom) analysis of purified OH-TBPH standards. The impurity of TBPH was 100 folds lower than OH-TBPH2 in purified standard.

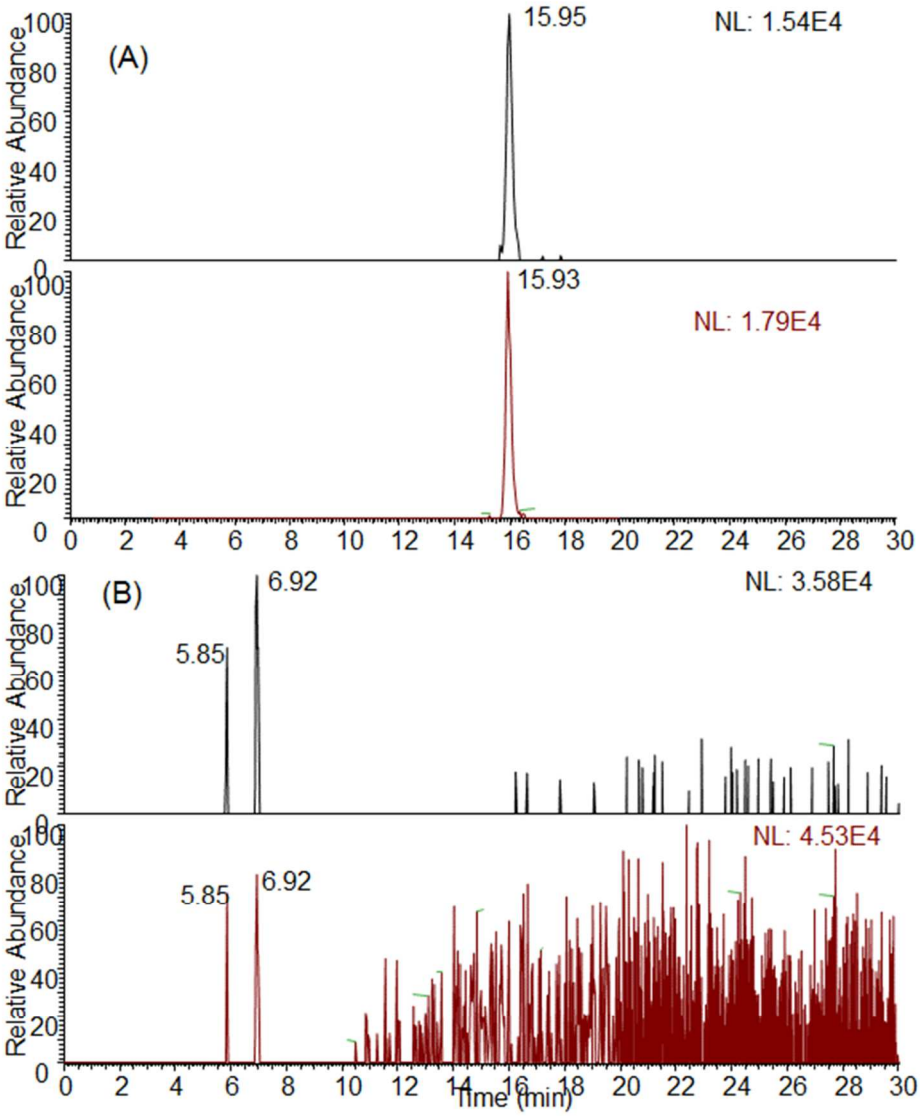

Figure S5. (A) TBPH was eluted in the first fraction from Florisil cartridges using DCM; (B) OH-TBPH isomers were eluted in the third fraction from Florisil cartridges using a mixture of methanol:DCM (v/v, 1:1).

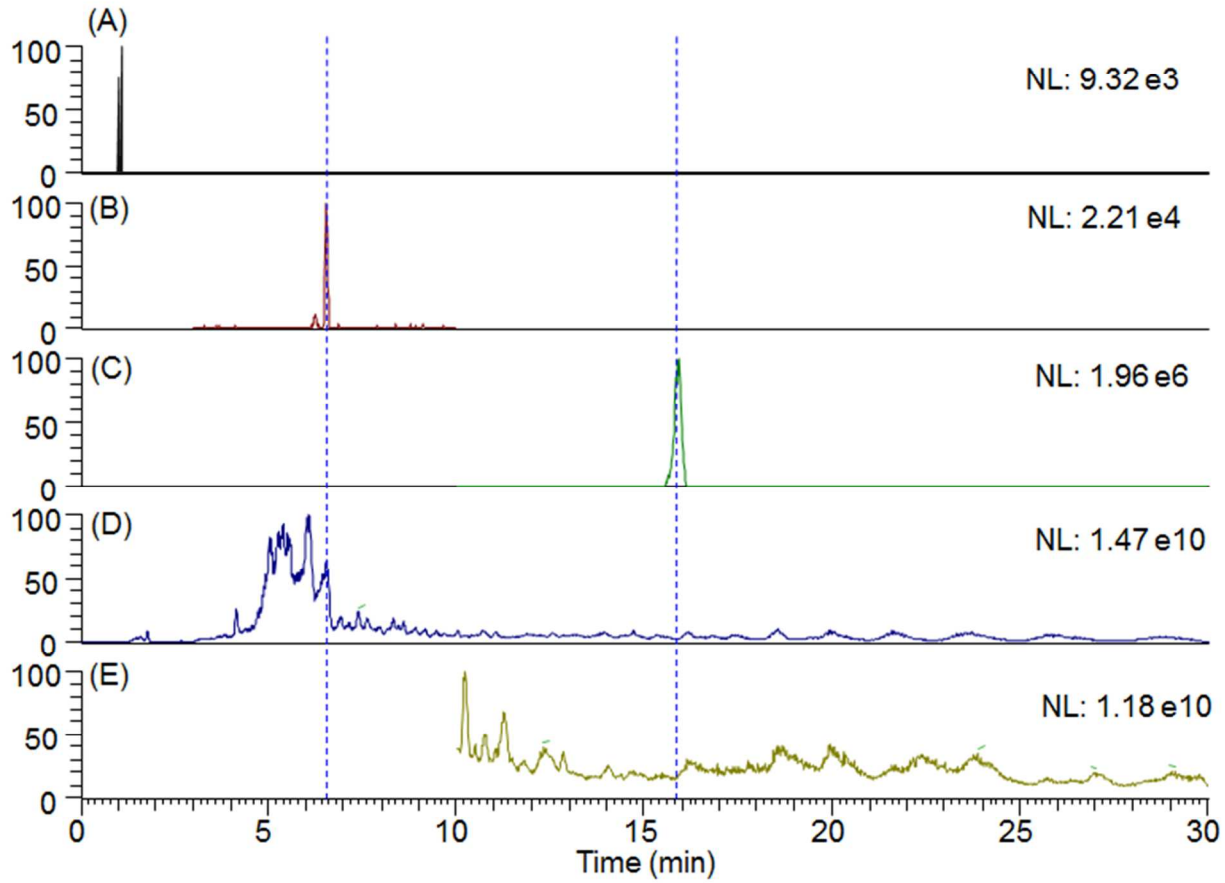

Figure S6. Comparison of the SIM mode and full scan mode for OH-TBPH analysis in dust samples. (A) OH-TBPH isomers could not be detected under full scan mode when extracted the ions at 10 ppm window. (B) Two OH-TBPH isomers were successfully detected using SIM mode when extracted the ions at 10 ppm window. (C) TBPH was observed in full scan mode. (D) The total ion intensity in negative ion mode was much greater than those of OH-TBPH at the similar elution time. (E) Total ion intensity in positive ion mode and comparison to TBPH intensity.