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Electronic Supporting Information

Pyrolysis removes common microconstituents triclocarban, triclosan, and nonylphenol from biosolids

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Section ES1. Selection of Pptimized Solvent for Accelerated Solvent Extractions

Five different solvent combinations were tested to determine the optimal solvent and cycle numbers for analyte recovery. Methanol and dichloromethane were tested separately and in combination. Ultimately two heating cycles with methanol was chosen for the extraction method.



Figure ES1-1. Extraction efficiency using dichloromethane and one extraction cycle. Vertical bars represent average recovery and the error bars represent the standard deviation between duplicate runs.



Figure ES1-2. Extraction efficiency using dichloromethane and two extraction cycles. Vertical bars represent average recovery and the error bars represent the standard deviation between duplicate runs.



Figure ES1-3. Extraction efficiency using 1:1 methanol:dichloromethane at two cycles. Vertical bars represent average recovery and the error bars represent the standard deviation between duplicate runs.



Figure ES1-4. Extraction efficiency using methanol at one cycle. Vertical bars represent average recovery and the error bars represent the standard deviation between duplicate runs.



Figure ES1-5. Extraction efficiency using methanol at two cycles. Vertical bars represent average recovery and the error bars represent the standard deviation between duplicate runs.

Section ES2. LC-MS-MS Operation and Spike-Recovery Results

Microconstituent concentrations in extracts for the temperature experiments were determined by injecting 20 μL into a Shimadzu Prominence HPLC coupled to an AB Sciex tandem mass spectrometer (LC-MS/MS). Chromatography was performed with a Phenomenex Luna C18 column (3 μm particle size, 150 x 3 mm). The flow rate was 300 μL/min using mobile phase A of 100% HPLC grade water and mobile phase B of 100% methanol. The method began with 70% B from zero to three minutes and then linearly increased to 100% B from three minutes to 20 minutes, and held at 100% B until 22 minutes. The gradient dropped down to 70% B at 22.01 minutes and was maintained at 70% until 25 minutes.

The precursor and product mass-to-charge (m/z) targets for TCS, TCC, and NP were 289 and 35, 313 and 160, and 219 and 133, respectively. A methanol blank and a standard were injected every 12 samples to check for column contamination and consistency in MS response. Seven point standard curves were used for analyte quantification. Analytes were only quantified if they yielded responses greater than the lowest point in the standard curve and had a signal-to-noise ratio greater than 10. The quantification limits (based on a signal-to-noise ratio of 10:1) was 10 μ g/L for all compounds.

Spike-and-recovery tests were performed to verify that the analytes were recovered in the biosolids and biochar matrices using the ASE extraction method followed by the LC-MS-MS method. Analytes were recovered in both matrices, indicating that the absence of a compound in an experimental sample could be contributed to removal by pyrolysis.

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Matrix	TCS	тсс	NP
Biosolids	49 ± 19	50 ± 24	105 ± 16
Biochar	44 ± 1	31 ± 6	84 ± 6

Table ES2-1: Recovery of Analytes from Spike and Recovery Tests for LC-MS/MS(n=3; average % recovery ± standard deviation).

Section ES3. LC-MS Operation and Spike-Recovery Results

Microconstituent concentrations in extracts from the time experiments and sand experiments were determined by injecting 20 μ L into a Shimadzu LCMS-2020. Chromatography was performed with a Phenomenex Luna C18 column (3 μ m particle size, 150 x 3 mm). The flow rate was 400 μ L/min using mobile phase A of 100% HPLC grade water and mobile phase B of 100% methanol. The method began at 80% methanol and increased linearly over 13 minutes to 100% methanol. The mass to charge (m/z) ratios used for detection of TCS, TCC, and NP were 288, 312, and 219, respectively. The same QA/QC steps described in S1 were followed, and the quantification limits for TCS and TCC were 10 μ g/L and 25 μ g/L for NP.

Spike-and-recovery tests were performed to verify that the analytes were recovered in the biosolids and biochar matrices using the ASE extraction method followed by the LC-MS method. Analytes were recovered in both matrices, indicating that the absence of a compound in an experimental sample could be contributed to removal by pyrolysis.

Matrix	TCS	тсс	NP
Biosolids	86 ± 12	73 ± 13	88 ± 27
Biochar	61 ± 5	34 ± 12	65 ± 6

Table ES3-1: Recovery of Analytes from Spike and Recovery Tests for LC-MS(n=3; average % recovery ± standard deviation).



Section ES4. Room-temperature Control for Spiked Sand Fate Experiments

Figure ES4-1. Microconstituent recovery in room-temperature control for spiked-sand pyrolysis fate experiments. Microconstituents were spiked into sand and held in the system at room temperature. The microconstituents were only recovered in the sand; none of the microconstituents were found in the methanol collection system.



Section ES5. Chromatograms for Triclocarban and Triclocarban-Dechlorinated Products





Figure ES5-2. Triclocarban chromatogram from impinger sample.



Figure ES5-3. 1-(3,4-Dichlorophenyl)-3-phenylurea chromatogram from standard.



Figure ES5-4. 1-(3,4-Dichlorophenyl)-3-phenylurea chromatogram from impinger sample. Triclocarban was dechlorinated during pyrolysis. The shift to earlier retention times for these products is indicative of the decrease in hydrophobicity that occurs when a chlorine atom is replaced with a hydrogen atom.



Figure ES5-5. 1-(4-Chlorophenyl)-3-phenylurea chromatogram from standard.



Figure ES5-6. 1-(4-Chlorophenyl)-3-phenylurea chromatogram from pyrolysis system tubing rinse.



Section ES6. Chromatograms for Triclosan and Suspected Triclosan-Dechlorinated Products

Figure ES6-1. Triclosan chromatogram from standard.



Figure ES6-2. Triclosan chromatogram from impinger sample.



Figure ES6-3. Likely 5-Chloro-2-(3-chlorophenoxy)phenol chromatogram from impinger sample.



Figure ES6-4. Likely 5-Chloro-2-phenoxyphenol chromatogram from impinger. The second peak is likely another triclosan dechlorinated product that has a different chlorine atom remaining. The large peak at 6 minutes belonged to nonylphenol and was removed from this image for clarity.

Section ES7. Vapor Pressures of Microconstituents and Legacy Pollutants

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Microconstituent	Vapor Pressure (mm Hg)
Nonylphenol	6.86E-04
Triclosan	4.65E-06
Triclocarban	3.61E-09
Legacy Pollutant	
2,3,7,8-2,3,7,8-tetrachlorodibenzo-p-dioxin	1.50 E-09
Decachlorobiphenyl	1.06E-07
2,2',3,3',4,4'-Hexachlorobiphenyl	2.56E-06
2,3,3'-Trichlorobiphenyl	4.00E-05

Table ES7-1: Vapor Pressures of Common Microconstituents and Legacy Pollutants. Values are taken from Estimation Programs Interface Suite, US EPA 2012.