Alkylphenols Persistently Remained at High concentrations in Landfilled Waste

Supplementary Material

Abbreviation table

Name	Abbreviation
Acetic acid--2,4-di-tert-butylphenol	Hac-2,4-DTBP
Bisphenol A	BPA
2,4-Bis(1-phenylethyl)phenol	2,4-BPTBP
2,6-Dimethoxyphenol	2,6-DTP
2,4-Di-tert-butylphenol	2,4-DTBP
3,5-Di-tert-butylphenol	3,5-DTBP
2-Ethoxy-4-methylphenol	2-ETMP
3-Ethyl-5-methylphenol	3-ETMP
5-Isopropyl-2-methylphenol	C_3H_4-2-MP
m-Cresol	CH_3-P
p-Cyclohexylphenol	p-CTBP
2-(1-Phenylethyl)phenol	2-1-PTBP
p-Isopropenylphenol	p-ITBP
4-tert-Butyl-2-methylphenol	4-tert-BTMP
3-tert-butylphenol	3-tert-BTP
2,4,6-Tri-tert-butylphenol	2,4,6-TtBP

Section S1 Background knowledge

In order to figure out the occurrence characteristics of alkylphenols (including concentration, medium, area, etc), this study searched literatures and listed the information in **Table S1-1.** Bisphenol A (BPA) is a relatively common and typical endocrine disruptor, so the concentration of BPA is also included in **Table S1-1** as a reference value.

Table S1-1 Occurrence characteristics of Alkylphenols (APs) and Bisphenol A (BPA) in literatures

Pollutants	Medium	Area	Concentration	Unit	Type	References
4-Tert-octylphenol	Indoor dust	Turkiye	35	ng/g	Median	1
4-N-nonylphenol			520			
Nonylphenol diethoxylate			1910			
Nonylphenols	Serum (pregnant women)	China	131.2	ng/mL	Median	2
4-N-octylphenol	Atmospheric fine particulates	China	0.66	pg/m^3	Median	3
2-Phenylphenol			3.9			
2-6-Di-tert-butyl-4-(hydroxymethyl)phenol			19.19			
3-Tert-butyl-4-hydroxyanisole			0.47			
2-Tert-butylbenzene-1,4-diol			0.12			
4-Nonylphenols	Lake sediments	USA	12.3	ng/g dry wt	Mean	4
4-Nonylphenol Monoethoxylates			11.5			
2,6-Di-tert-butyl-p-cresol	Wastewater	Canada	0.373	μg/L	-	5
4-Nonylphenolmonoethoxycarboxylic acid	Wastewater	Ohio, USA	8.5	μg/L	Mean	6
4-Nonylphenoldiethoxycarboxylic acid			73			
4-Nonylphenoltriethoxycarboxylic acid			2.2			
2,6-Di-tert-butyl-4-methylphenol	Sludge	China	4.14	μg/g	Mean	7
4-Tert-octylphenol			374	μg/g		
2,4,6-Tri-tert-bultylphenol			98.1	μg/g		
Nonylphenol	Freshwater		138.32	ng/L	Mean	8

Pollutants	Medium	Area	Concentration	Unit	Type	References
Nonylphenolmonocarboxylate		Northern Antarctic Peninsula region	2.89			
Nonylphenoldiethoxylate			6.86			
4-Tert-octylphenol	Multicereals breakfast	/	20 ± 2	ng/g	Mean	9
4-Hexylphenol			21 ± 2			
4-N-octylphenol			25 ± 1			
Phenol	Leachate from four landfill	Denmark	ND-2.2	µg/L	Range	10
CH3-P			ND-17			
o/p-Cresol			0.15-29			
2/3-Chlorophenol			ND-1.6			
4-Chlorophenol			ND-1.3			
3,5-Dimethylphenol			ND-27			
2,5-Dimethylphenol			ND-4.5			
2,4-Dimethylphenol			ND-13			
3,4-Dimethylphenol			ND-10			
2,6/2,3-Dimethylphenol			ND-1.9			
4-Chloro-*o*/*CH3-P*			0.26-5.3			
3,5-Dichlorophenol			ND-0.37			
Nonylphenol	Leachate	Beijing, China	11.2-14.2	ng/L	Range	11–13
Octylphenol		Beijing, China	40.5-54.3			
2-Phenylphenol		Guangdong, China	6.81-17.21			11–13
4-Nonylphenol	Leachate from four landfill	Sweden	<0.1-7.3	µg/L	Range	14
Nonylphenol monoethoxylate			<0.1-3.6			
4-tert-Octylphenol			<0.01-1.3			
Octylphenol monoethoxylate			<0.01-5.33			
4-tert-Butylphenol			0.027-8.1			

Pollutants	Medium	Area	Concentration	Unit	Type	References
4-tert-Pentylphenol			<0.1-0.66			
BPA	Leachate from four landfill	-	<0.01-107	-	-	14
	Bottled water	/	0.1	μg/L	Mean	15
	Tap water	/	0.06			
	Commercial milk products	Spain	8.3	pg/g	Median	16

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Table S1-2 shows the emerging pollutants detected in leachate and refuse reported in literature.

Table S1-2 Emerging pollutants reported in leachate and refuse

Pollutants		Medium	Concentration	Area	References
Microplastics	Total	Leachate	0.42–24.58 items/L	China	17
	Total	Refuse	62 (±23) items/g	China	18
	Polyethylene terephthalate		398 μg/g		19
	Polycarbonate		1.08 μg/g		
Antibiotics	Fluoroquinolone	Leachate	48326.67 ng/L	Shanghai, China	20
	Danofloxacin	Leachate	908 ng/L (average)	China	21
	Diclofenac		14300 ng/L		
	Sparfloxacin		1450 ng/L		
	Sulfadiazine		5680 ng/L		
	Sulfamethazine		1880 ng/L		
	Lincomycin		8160 ng/L		
	Norfloxacin	Landfilled refuse	258 μg/kg	China	22
	Enrofloxacin		390 μg/kg		
	Ciprofloxacin		61.2 μg/kg		
	Azithromycin		41.4 μg/kg		
	Sulfadiazine	Leachate	2056-22102 ng/L	Northern Italy	23

Pollutants		Medium	Concentration	Area	References
	Sulfamethoxazole		7978-95816 ng/L		
	Sulfadimidine		3898-8450 ng/L		
	Ciprofloxacin		0-434740 ng/L		
	Enrofloxacin		0-9074 ng/L		
	Erythromycin		8510-252824 ng/L		
	Sulfamethoxazole	Leachate	674–962 ng/L	Shanghai, China	24
	Sulfamerazine		1126–1192 ng/L		
	Sulfamethazine		60–1099 ng/L		
Phthalic acid ester (PAE)	Di(2-ethylhexyl) phthalate	Leachate	285 µg/kg	Anasco, Puerto Rico, America	25
	Di(2-Ethylhexyl) phthalate	Leachate	7.171 µg/L	Istanbul city, Turkey	25,26
	Benzyl butyl phthalate		0.24 µg/L		
	Di-n-octylphthalate		0.21 µg/L		
	Dimethyl phthalate (DMP)	Leachate	1.22-1.64 mg/L	China	27,28
	Di-ethyl phthalate (DEP)		1.40-1.73 mg/L		
	Diisobutyl phthalate (DIBP)		0.024-1.60 mg/L		
	Di-n-butyl phthalate (DnBP)		0.011- 1.28 mg/L		
	Di-(2-ethylhexyl) phthalate (DEHP)		0.016-1.03 mg/L		

Pollutants		Medium	Concentration	Area	References
Bisphenol A (BPA)		Leachate from four landfill	<0.01-107	Sweden	13
		Leachate	0.003-0.26 mg/L	China	27,28
		Leachate	238,188 ng/L (median)	Singapore	29
PPCP/EDCs	Acetaminophen	Leachate	2204 ng/L (median)	Singapore	29
	N, N-diethyl-m-toluamide		1496 ng/L (median)		
	Caffeine		419 ng/L		
	Clofibric acid		227 ng/L		
	Crotamiton		7.6 ng/L		
Per- and polyfluoroalkyl substances (PFAS)	Mass	Leachate	563-638 kg/yr	USA	30
	Per- and polyfluoroalkyl substances	Leachate	9700 ng/L	North central Florida, USA	31
	Perfluorobutyric acid (PFBA)		200 ng/L (median)		
	Perfluoropentanoic acid (PFPeA)		1200 ng/L (median)		
	Perfluorohexanoic Acid (PFHxA)		2400 ng/L (median)		
	Perfluorooctanoic acid (PFOA)		1100 ng/L (median)		
	Perfluorodecanoic acid (PFDA)		78 ng/L (median)		

Pollutants		Medium	Concentration	Area	References
	Perfluorononanoic acid (PFNA)		47 ng/L (median)		
Flame retardants	Hexabromocyclododecane	Leachate	5906 μg/kg dry weight	Netherlands	32
	Tetrabromobisphenol A		54 μg/kg dry weight		
	Bis(2-ethylhexyl) tetrabromophtalate	Sediment	11 ng/g dw	South Africa	33
	Mass of organophosphate flame retardants	Leachate	29.0-437 μg/L	China	34
	Triethyl phosphate		9.37 μg/L (median)		
	Tris(2methylpropyl) phosphate		0.766 μg/L (median)		
	Tributyl phosphate		1.70 μg/L (median)		
	Tris(2-butoxyethyl) phosphate		0.245 μg/L (median)		
	Tris(2-chloroethyl) phosphate		33.5 μg/L (median)		
	Tris(1-chloropropan-2-yl) phosphate		6.41 μg/L (median)		
APs	Nonylphenol	Leachate	11.2-14.2 ng/L	Beijing, China	11,12
	Octylphenol		40.5-54.3 ng/L	Beijing, China	
	2-Phenylphenol		6.81-17.21 ng/L	Guangdong, China	13
	Phenol		ND-2.2 ng/L	Denmark	12

Pollutants		Medium	Concentration	Area	References
	CH3-P	Leachate from four landfills	ND-17 ng/L		
	o/p-Cresol		0.15-29 ng/L		
	2/3-Chlorophenol		ND-1.6 ng/L		
	4-Chlorophenol		ND-1.3 ng/L		
	3,5-Dimethylphenol		ND-27 ng/L		
	2,5-Dimethylphenol		ND-4.5 ng/L		
	2,4-Dimethylphenol		ND-13 ng/L		
	3,4-Dimethylphenol		ND-10 ng/L		
	2,6/2,3-Dimethylphenol		ND-1.9 ng/L		
	4-Chloro-*o*/*CH3-P*		0.26-5.3 ng/L		
	3,5-Dichlorophenol		ND-0.37 ng/L		
	4-Nonylphenol	Leachate from four landfill	<0.1-7.3 ng/L	Sweden	13
	Nonylphenol monoethoxylate		<0.1-3.6 ng/L		
	4-tert-Octylphenol		<0.01-1.3 ng/L		
	Octylphenol monoethoxylate		<0.01-5.33 ng/L		
	4-tert-Butylphenol		0.027-8.1 ng/L		
	4-tert-Pentylphenol		<0.1-0.66 ng/L		

Figure S1-1 to S1-3 shows the proportion of landfilling of MSW.

Figure S1-1 Proportion of MSW landfilling in China cities.

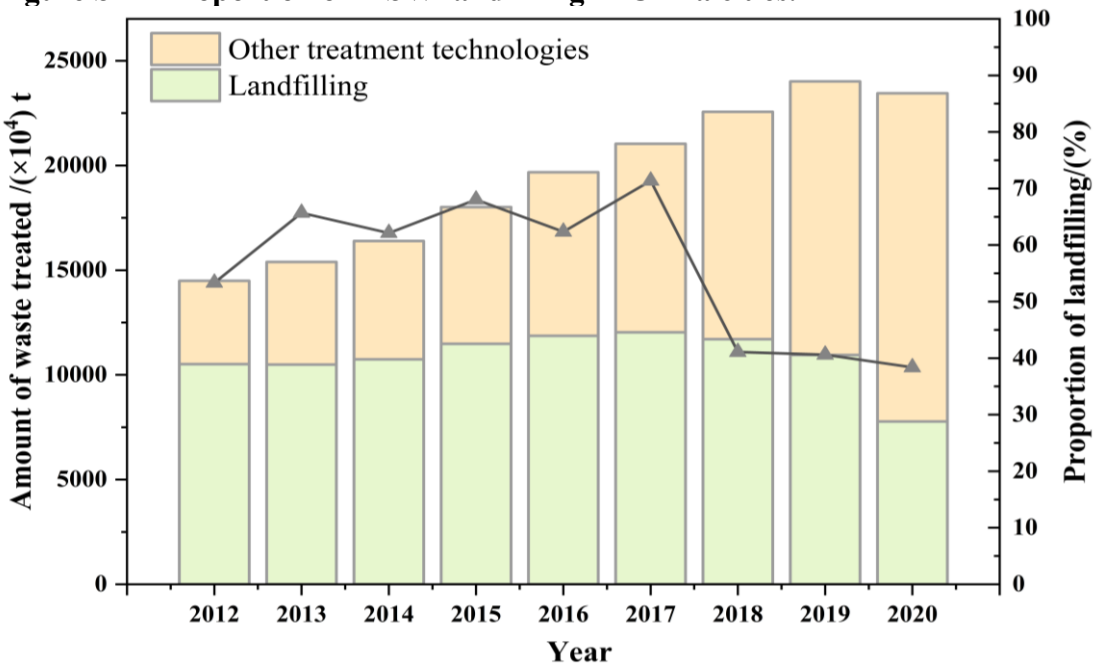


Figure S1-2 Proportion of MSW landfilling in Zhejiang Province.

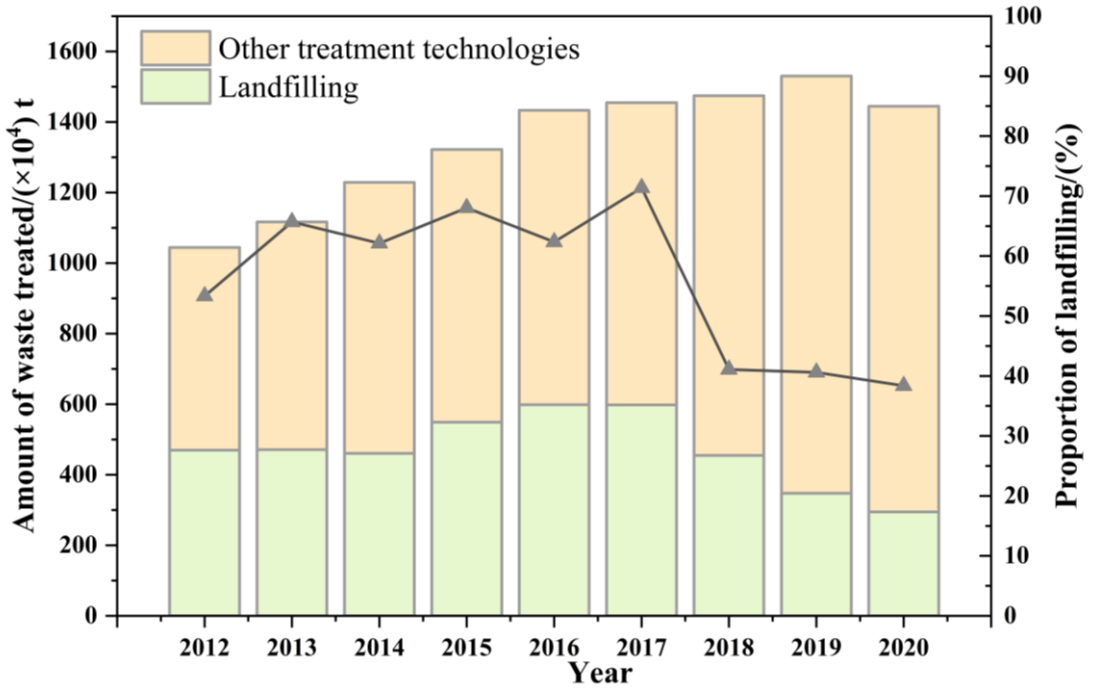
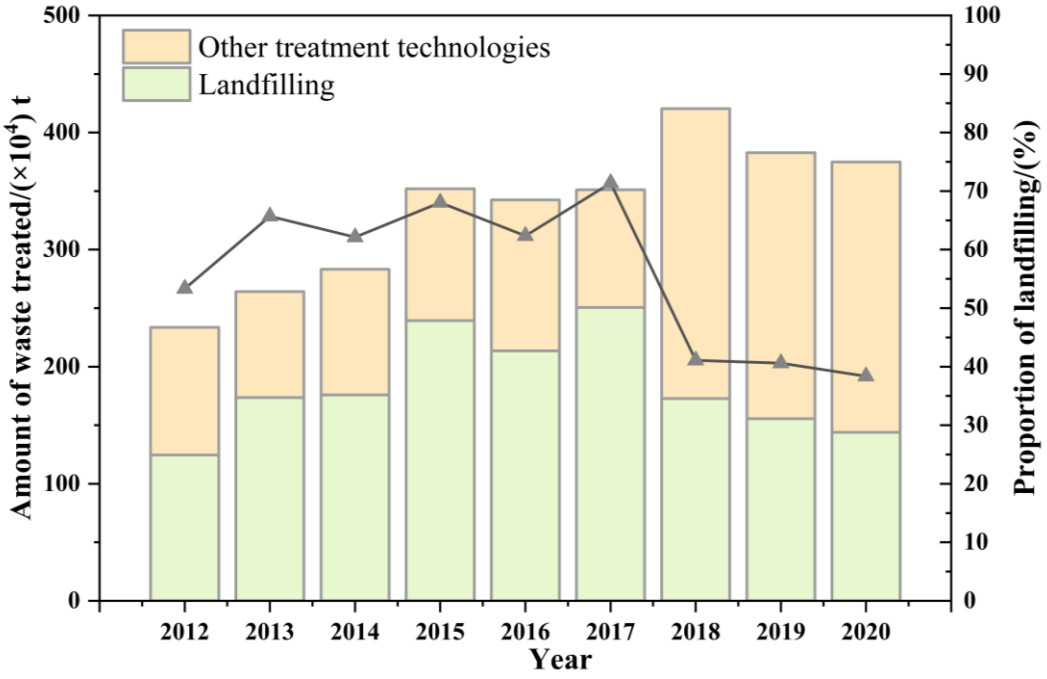


Figure S1-3 Proportion of MSW landfilling in Hangzhou City.



Section S2 Chemicals and reagents

Text S2-1 Details of the reagent

In this study, dichloromethane and n-hexane (HPLC grade) were used as the extract, and the Milli-Q pure water was prepared by an ultra-pure water purification system (Millipore, Billerica, MA, USA). Internal standards used for quantification was anthracene-d10 with the total concentration of 20 μg/mL. Analytical standards contained nonylphenol, 2,4-DTBP, 2,6-DTP, 2,4,6-tri-tert-butylphenol, 3-tert-butylphenol, etc. Details of the chemicals and reagents are shown in **Table S2-1**.

31 **Table S2-1 Details of chemicals and reagents**

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Name	Chemical formula	CAS	Supplier	Usage
2,4-DTBP	$C_{14}H_{22}O$	96-76-4	Sigma-Aldrich	Analytical standards
2,6-DTP	$C_8H_{10}O_3$	91-10-1		Analytical standards
2,4,6-TrBP	$C_{18}H_{30}O$	732-26-3		Analytical standards
3-tert-BTP	$C_{10}H_{14}O$	585-34-2		Analytical standards
Bisphenol A	$C_{15}H_{16}O_2$	80-05-7		Analytical standards
Nonylphenol	$C_{15}H_{24}O$	104-40-5		Analytical standards
Anthracene-d10	$C_{14}H_{10}$	1719-06-8		Internal standards
Dichloromethane	CH_2Cl_2	75-09-2		Extraction
n-Hexane	C_6H_{14}	110-54-3		Extraction
Sodium sulfate	Na_2SO_4	7757-82-6	Sinopharm Chemical Reagent Co., Ltd	Dehydrating agent
Milli-Q water	-	-	Millipore	-

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Section S3 Instrumental analysis

Quantitative analysis was performed with high-resolution gas chromatography and Orbitrap mass spectrometry (GC Orbitrap MS) with the TriPlus RSH automatic sampler (Thermo Fisher Scientific, Bremen, Germany). The substances were separated using a TG-5SILMS column (30 m length×0.25 mm id×0.25 μm film thickness, Thermo Fisher Scientific, Bremen, Germany). The column temperature was maintained at 60℃ for 2.00 min and then increased to 220℃ at 10℃/min for 3 min. Then it rose to 325℃ at 15℃/min and held for 5.00 min. The inlet temperature was 250℃, the carrier gas was helium, and the flow rate was 0.2 mL/min. The flow rate was 10 mL/min and the shunt time was 3 min. The ionization mode of electron bombardment source (EI, 70eV) was adopted, the ion source temperature was 280℃, and the transmission line temperature was 250℃. In the range of 33-350 m/z, the signal was acquired in full-scan mode with a mass spectral resolution of 60,000. The resulting raw data was collected by X calibur (Thermo Fisher Scientific, Bremen, Germany) and processed by Trace Finder 5.0 software (Thermo Fisher Scientific, Bremen, Germany).

Section S4 Substance identification

Text S4-1 Qualitative identification of substances by non-targeted analysis method

Substances from non-targeted analysis are screened in the following procedure:

(1) High resolution filtering (HRF), search index score (SI) and the deviation of retention index (ΔRI) based on the high-resolution mass spectrum by Trace Finder 5.0 software were used to screen the target substance.

HRF is the ratio of the exact mass of the possible substance to the exact mass of the corresponding substance in the spectrum library, the higher the HRF value indicates the greater the possibility of the compound.

SI is the index of positive match between the measured spectrum and the standard spectrum.

ΔRI is the deviation of the retention index calculated after comparing with the compound in the library, and a smaller ΔRI indicates a more accurate determination result.

(2) Through the obtained data, the molecular ion peak ([M+H]+), molecular formula and other information of the substance were determined to eliminate the interference of similar substances.

(3) Synthesize the contents of (1) and (2), the most likely substances were determined, and the semi qualitative results were obtained after screening.

(4) Further confirmation of suspected substances were made by comparing them with known standards.

Text S4-2 Quantitative information of substance

Combining with the substance test results, **Table S4-1** shows the CAS, retention time, quantitative ion, internal standard, calibration curves, R^2, method quantification limits (MQL) for quantitative substances. **Table S4-2** shows the retention time, quantitative ion, internal standard for semi quantitative substances. MQL was defined as 10 times the standard deviation of the procedural blank values. Substances that can be quantified by standards were accurately quantified, and other substances were semi-quantified according to similar structure criteria.

Table S4-1 Information sheet for quantitative substances

Chemicals	CAS	Retention time (min)	Quantitative ion (m/z)	Internal standard	Calibration curves	R^2	Method quantification limits (ng/g)
2,4-DTBP	96-76-4	15.989	191.14	Anthracene-d10	$y = 0.876\,x + 0.639$	0.9970	0.1-250
2,6-DTP	91-10-1	13.944	154.06		$y = (2E+08)\,x - (7E+07)$	0.9970	0.1-250
2,4,6-TtBP	732-26-3	16.998	247.21		$y = 0.0001\,x + (1E-04)$	0.9995	0.1-250
3-tert-BTP	585-34-2	13.149	135.08		$y = 0.4215\,x + 2.1564$	0.9927	0.1-250
Nonylphenol	104-40-5	16.845	107.05		$y = 0.0004\,x + 0.0002$	0.9972	0.1-250
Bisphenol A	80-05-7	23.536	213.09		$y = 0.0023\,x - 0.0999$	0.9906	40-250

Table S4-2 Information sheet for semi quantitative substances

Chemicals	CAS	Retention time (min)	Quantitative ion (m/z)	Recovery rate	Internal standard
Hac-2,4-DTBP	104316-22-5	15.965	191.143	101.2%	Anthracene-d10
2,4-BPTBP	2769-94-0	28.418	287.143	82.86%	
2,4-DTBP	96-76-4	15.989	191.143	77.38%	
3,5-DTBP	1138-52-9	15.103	163.111	106.4%	
2-ETMP	2563-07-7	12.525	175.148	77.38%	
3-ETMP	698-71-5	12.83	121.065	107.9%	
C_3H_4-2-MP	499-75-2	13.216	135.081	102.3%	
CH_3-P	108-39-4	9.728	107.049	54.7%	
p-CTBP	1131-60-8	15.068	120.093	87.43%	
2-1-PTBP	4237-44-9	19.456	212.156	72.62%	
p-ITBP	4286-23-1	21.436	119.049	92.07%	
4-tert-BTMP	98-27-1	14.035	149.096	55.9%	
2,4,6-TtBP	736-26-3	16.993	247.206	91.1%	

Section S5 Ecological risk assessment

Risk quotient (RQ) was used to calculated ecological risk assessment. It was first calculated as the quotient of measured concentration and predicted no-effect concentration (PNEC-soil) [35,36,37]. PNEC-water is the predicted no-effect concentration values in water.

In this study, PNEC-soil was calculated based on PNEC-water and shown in formula (**S5-1**). PNEC-water was calculated based on the acute toxicity of the minimum value among LC50-fish, LC50-daphnid, EC50-green algae (mg/L), and assessment factor (AF). Acute toxicity was obtained from Ecological Structure - Activity Relationships Program (ECOSAR, https://www.epa.gov/tsca-screening-tools/ecological-structure-activity-relationships-program-ecosar-operation-manual). Acute toxicity of 2,4,6-TtBP, Hac-2,4-DTBP and 3,5-DTBP cannot be found in ECOSAR, thus the study didn't consider their risk. The parameters are shown in **Table S5-1**.

$$PNEC - soil = \frac{PNEC-water \times Koc}{85} \quad \text{(S5-1)}$$

$$PNEC - water = \frac{\min\left(LC50\ fish, LC50 daphnid, EC\ green\ algae\right)}{AF} \quad \text{(S5-2)}$$

$$logKoc = 0.81 \times \log Kow + 0.1 \quad \text{(S7-3)}$$

Table S5-1 Parameters of ecological risk assessment

APs	LC50 (fish) /(mg/L)	LC50 (daphnid) /(mg/L)	EC50 (green algae) /(mg/L)	log Kow	log Koc	PNEC (water) /(mg/L)	PNEC (soil) /(mg/g)
2,4-BPTBP	9.10E-02	7.30E-02	2.37E-01	5.83E+00	4.82E+00	7.30E-05	5.67E-02
2,4-DTBP	1.73E-01	1.33E-01	3.54E-01	5.33E+00	4.42E+00	1.33E-04	4.10E-02
2-ETMP	5.74E+01	3.38E+01	2.90E+01	2.38E+00	2.02E+00	2.90E-02	3.61E-02
3-ETMP	1.15E+01	7.25E+00	8.22E+00	3.10E+00	2.61E+00	7.25E-03	3.47E-02
C_3H_4-2-MP	5.37E+00	3.51E+00	4.66E+00	3.52E+00	2.95E+00	3.51E-03	3.66E-02
CH_3-P	7.85E+01	4.48E+01	3.41E+01	2.06E+00	1.77E+00	3.41E-02	2.36E-02
p-CTBP	1.17E+00	8.24E-01	1.49E+00	4.33E+00	3.61E+00	8.24E-04	3.92E-02
2-1-PTBP	5.16E+00	3.42E+00	4.81E+00	3.67E+00	3.07E+00	3.42E-03	4.75E-02
p-ITBP	1.51E+01	9.35E+00	1.01E+01	2.96E+00	2.50E+00	9.35E-03	3.47E-02
4-tert-BTMP	2.30E+00	1.56E+00	2.47E+00	3.97E+00	3.32E+00	1.56E-03	3.79E-02
Assessment factor (AF): 1000							

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