Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Water, Fish Bile, and Plasma	MDL: Plasma: 0.0075–0.025 ng/mL Bile: 0.015–0.05 ng/ml	Glass vials and bottles	Surrogate: DPPD-d_{14} Internal: progesterone-d_9	Water: SPE or dilution in MeOH, analysis Bile and plasma: Water dilution, SPE, analysis	Water, bile, plasma: LC-MS/MS	Phenomenex Kinetex C18 EVO (100×2.1 mm, 1.7 µm particle size)	ESI+ (MRM)	268>211	268 >167	Bile/plasma: (da Silva et al. In preparation)
Groundwater, Stormwater, and Surface Water	MRL: 2 ng/L	Collection: amber glass bottles, no headspace. Storage: Analyzed within 72 hours of collection or frozen until analysis	Surrogate internal standard: D5-6PPD-q	0.7-micron GFF	UPLC-MS/MS	Waters ACQUITY UPLC BEH C18 (1.7 µm, 2.1×50 mm) plus Vanguard pre-column (1.7 µm, 2.1×5 mm) column with 0.1% formic acid in water and ACN mobile phase	ESI+	269.1/184.1	269.1/93.0	(Lane et al. 2024)
Influent and Effluent of WWTP	MDL: 60 ng/L	Large volume SPE	Not available as of the publication date	Filtered using a Sartopure GF + Midicap, 0.65 µm deep filter and extracted using cartridges filled with 10 g of Chromabond HR-X, eluted with 5 mL of ethyl acetate, 5 mL of methanol, 5 mL of methanol containing 1% of formic acid, 5 mL of methanol containing 2% of 7 N ammonia in methanol, evaporated under a nitrogen to near dryness, brought to 1 mL with methanol	LC-HRMS	Kinetex C18 EVO column (50×2.1 mm, 2.6 µm particle size, 83 Phenomenex, pre-column 4×2.1 mm, and in-line filter 0.2 µm) and a gradient elution with 0.1% of formic acid and methanol containing 0.1% of formic acid mobile phase	Ion mode ESI+	M+H= 269.2012		(Maurer et al. 2023)
Snow from Urban Street	LOQ: 100 ng/L	250 mL glass bottles	Not available as of the publication date		UPLC-TOF-MS	HSS T3 column; 100×2.1 mm, 1.7 µm, and Atlantis T3 (3 µm, 2.1×100 mm); (A) water with 0.1% formic acid (v/v) and (B) methanol with 0.1% formic acid (v/v) mobile phase	ESI(+) mode MRM	269.1/184.0	269.1/106.9	(Seiwert et al. 2022)
Influent and Effluent WWTP during Snow Melt, Rain, And dry Conditions	LOQ: 100 ng/L	250 mL glass bottles	Not available as of the publication date		UPLC-TOF-MS	HSS T3 column; 100×2.1 mm, 1.7 µm, and Atlantis T3 (3 µm, 2.1×100 mm); (A) water with 0.1% formic acid (v/v) and (B) methanol with 0.1% formic acid (v/v) mobile phase	ESI(+) mode MRM	269.1/184.0	269.1/106.9	(Seiwert et al. 2022)
Air from 18 Major Cities That Comprise the GAPS Network	Instrument LOQ: 1.95 ng/mL Method LOQ: 2.71 pg/m^3	PUF disk samplers collecting both gas- and particle-phase chemicals	Not available as of the publication date	ASE extraction with petroleum ether and acetone (83/17, v/v), rotary evaporation, reconstituted with iso-octane, silica column cleanup	UPLC-HRMS	Phenomenex (Torrance, CA, USA) Kinetex C18 column (2.6 µm in particle size, 50×4.6 mm in length and inner diameter); HPLC-grade methanol, with 0.1% of formic acid in both, were used as the mobile phase	Positive ionization mode with a HESI source (HESI-II probe) PRM	269.2012/ 185.1068	Not available as of the publication date	(Johannessen, Saini, et al. 2022)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Fine Particulate Matter ($PM_{2.5}$)	MQL: 0.25 pg/m^3 MDL: 0.07 pg/m^3	Quartz fiber filter	Surrogate standard: diphenylamine-d_{10} Internal standard: D5-6PPD-q	Serial ultrasonication with dichloromethane and ACN, concentrated with nitrogen in ACN and PTFE filtered	HPLC-HRMS	Waters acquity HSS T3 column (1.8 µm, 2.1×100 mm) with 0.1% formic acid in water and 0.1% formic acid in ACN mobile phase	Data-dependent MS2 mode MRM	269.2/93.1	269.2/184.1 269.2/212.1	(Wang et al. 2022)
Fine Particulate Matter ($PM_{2.5}$) from Megacity	LOD: 1 pg/mL	Whatman medium-volume quartz fiber filters	pyrene-d_{10} and benzophenone-d_{10}	Ultrasonication ACN and dichloromethane/hexane, taken to near dryness with nitrogen, redissolved in methanol and filtered with PTFE membrane	UHPLC-MS/MS	A Waters ACQUITY UPLC C18 column (1.7µm, 2.1 mm×100 mm) with 0.4 mM ammonium acetate (A)/ MeOH (B) mobile phase	ESI(+) mode MRM	269/184	269/107	(Y. Zhang et al. 2022)
Airborne Particulate Matter along a Highway in Mississippi, USA	LOD: 2.13 ng/L	Airborne particulate matter was collected using Sigma-2 passive samplers	Not available as of the publication date	Methanol and hexane extraction shaker table, filtered with polycarbonate gold-coated filters, rotary evaporation to 3 mL, nitrogen evaporation to near dryness and redissolved in 66% methanol	UHPLC-HRMS	Online filter cartridge with a 2.1 mm ID×0.2 µm porosity stainless-steel filter, an Eclipse Plus C18 RRHD (5 mm×2.1 mm ID; 1.8 µm) guard column followed by the analytical column with the same stationary; 1 mM ammonium formate and 0.1% formic acid (A) and methanol 0.1% formic acid (B) mobile phase	HESI mode; data-dependent product scan	Not available as of the publication date	Not available as of the publication date	(Olubusoye et al. 2023)
Dust: Road Dust, Interior Car Dust , Parking Lot Dust , Indoor Dust from Homes Near E-waste Dismantling Area	LOQ: 0.11 ng/g	Precleaned nylon bag (pore size of 25 µm)	ISTD: Coumaphos-d_{10} SSTD: Benzophenone-d_{10}	Serial sonication with ACN and 1:1 dichloromethane:hexane; concentrated by nitrogen into methanol and filtered	HPLC-MS/MS	HPLC: C18 column (100×2 mm, Luna 3 µm, Phenomenex) with 0.3 g/L ammonium acetate (A) and methanol (B) mobile phase	ESI(+) mode MRM	269.0/184.1	269.0/212.4 269.0/92.8	(Huang et al. 2021)
Sediments across Urban Rivers, Estuaries, Coasts, and Deep-Sea Regions	MDL: 0.043 ng/g	Sediment packed in aluminum foil and stored in polypropylene tubes; freeze-dried and 1.0 mm mesh screened	D5-6PPD	Transferred to glass tube, ultrasonicated with ACN, concentrated and filtered with PTFE membrane	LC–MS/MS	C8 column (Waters Xbridge BEH, 2.5µm,2.1 mm×100 mm) 0.1%formic acid in water and (B) methanol mobile phase at a flowrate of 0.3 mL/min	ESI(+) mode MRM	269.2/184.1	269.2/212.1	(Zeng et al. 2023)
Fish	LOD: 0.00025 mg/kg LOQ: 0.00043 mg/kg	Homogenized by the electric blender, frozen until extraction in centrifuge tube	Not available as of the publication date	Modified QuEChERS	HPLC-MS/MS	Athena C18-WP chromatographic column (2.1 mm×50 mm, 3.0 µm) Mobile Phase: MeOH: Deionized water/90:10	ESI(+) mode MRM	269.3/184.1	269.3/211.0	(Ji, Li, et al. 2022)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Larval Zebrafish and Water	LOQ: 0.1 ng/mL	Glass beakers	Not available as of the publication date	QuEChERS	HPLC/MS-MS	Luna Omega C18, 100×2.1 mm, 1.7 µm Mobile phase: 5 mmol/L ammonium formate solution (A), methanol (B)	ESI(+) mode MRM	269.40 > 184.25	269.40 > 107.15	(Fang et al. 2023)
Zebrafish Embryo	LOD: 0.130 ng/mL LOQ: 0.638 ng/mL	Glass trays	Not available as of the publication date	FastPrep homogenizer, sonication, and centrifuging	HPLC/MS-MS	Atlantis T3 C18-phase column (2.1 mm×50 mm, 3µm; Waters) with an Atlantis T3 Security Guard column (2.1×10 mm, Waters). Mobile phase of 0.1% formic acid in MilliQ water (A) and 0.1% formic acid in methanol (B)	ESI(+) mode MRM	269.079/ 106.90	269.079/184.0	(Grasse et al. 2023)
Embryonic Zebrafish	LOD: 1 pg/mL	Well plate exposures	6PPD-q-$^{13}C_6$	Homogenization, polyfiltration, sonication, concentrated to near dryness, and redissolved in methanol and filtrated through a 0.22 µm poly (ether sulfone) membrane	UPLC/MS-MS	A Waters ACQUITY UPLC C18 column (1.7µm, 2.1 mm×100 mm) with 0.4 mM CH3COONH4 (A)/ MeOH (B)	ESI(+) mode MRM	269/184	269/107	(S.-Y. Zhang, Gan, Shen, Jiang, et al. 2023) (Y. Zhang et al. 2022)
Rainbow Trout Tissue and Exposure Water Samples	MDLs: 0.1–0.6 ng/g in tissue	Plastic and glass	D5-6PPD-q	Whole fish body was homogenized into Eppendorf tube, serial sonication with ACN and centrifuged	UHPLC-HRMS	Hypersil GOLD C18 column (50×2.1 mm, 1.5 µm). Mobile phase of 0.1% formic acid in ultrapure water (A) and 0.1% formic acid in methanol (B)	ESI(+/-) Full-scan mode	Not available as of the publication date	Not available as of the publication date	(Nair et al. 2023)
Lumpfish blood	LOD: 0.5 pg	Not available as of the publication date	Internal: D5-6PPD-q Recovery: 6PPD-q-$^{13}C_6$	Vortex-sonication and centrifuged	HRGC/HRMS	TG-5SILMS column (30 m, 0.25 mm ID, film thickness—0.25 µm)	Nontargeted screening Full-scan mode	Not available as of the publication date	Not available as of the publication date	(Hägg et al. 2023)
Human Urine from General Adults, Children, and Pregnant Women	MDL: 0.012 ng/mL	Urine immediately transferred to the laboratory, stored at −40°C until analysis; glass used during laboratory extractions	6PPD-q-$^{13}C_6$	Salting-out assisted liquid−liquid extraction, concentrated with nitrogen and 0.22 µm filtered	LC-MS/MS	Ultra-Fast LC: Waters XBridge C8 column (2.1 mm×100 mm, 2.5µm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase	ESI(+) mode MRM	269.2/184.1	268.2/107.1	(Du et al. 2022)
Honey	LOD: 0.0003 mg/kg LOQ: 0.0001 mg/kg	Stored at room temp until extraction in centrifuge tube	Not available as of the publication date	Modified QuEChERS	HPLC-MS/MS	Athena C18-WP chromatographic column (2.1 mm×50 mm, 3.0 µm) Mobile Phase: MeOH: Deionized water/90:10	ESI(+) mode MRM	269.3/184.1	269.3/211.0	(Ji, Li, et al. 2022)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Lettuce (*Valerianella locusta*) Plant and Roots and TWPs in Nutrient Solution	Not available as of the publication date	Glass vials	Not available as of the publication date	Plant: serial bead beater with stainless-steel beads and ACN extraction, centrifuged, filtered with nylon filter Roots: Freeze-dried roots, suspended in nutrient solution, reciprocal shaker, centrifuge, nylon syringe filter Nutrient Solution: serial liquid−liquid extraction, nylon syringe filter	UPLC-MS/MS	C18 column (Waters ACQUITY HSS T3, 1.8 µm, Waters), ultrapure water (phase A) and ACN (phase B), both containing 0.1% formic acid mobile phase	ESI(+) mode MRM	269/184	269/107 269/93	(Castan et al. 2023)
Soil, Water, Atmospheric Particles; Urban Runoff Water Samples Were Collected in a Dense Traffic Urban Area	IQL: 0.035 ng/mL	Soil: stainless-steel shovel, transported to lab within 2 hours, freeze-dried, homogenized, sieved through a 60 mesh Atmospheric particle: collect on quartz fiber filters and stored at -20C Water: 200 mL collected in Teflon tubes, glass microfiber filter, acidified with 2% formic acid	Internal: diphenylamine-d_{10} Surrogate: D5-6PPD-q	Soil: serial ultrasonication with ACN, concentrated to dryness with nitrogen, redissolved in methanol and 0.45 µm nylon filtered Atmospheric particles: serial ultrasonication with dichloromethane and ACN, concentrated to near dryness with nitrogen, redissolved in ACN and filtered Water: HLB SPE Cartridge (60 mg, 3 mL), eluted with methanol−dichloromethane (1:9, v/v), concentrated to dryness with nitrogen, redissolved in ACN and 0.45 µm nylon filtered	UPLC-HRMS	Waters ACQUITY HSS T3 (1.8 µm, 2.1×100 mm) column with 0.1% formic acid in water and 0.1% formic acid in ACN mobile phase	ESI(+) mode full-scan and data-dependent acquisition mode	269.2/212.1	269.2/93.1	(Cao et al. 2022)
Recycled Tire Rubber Employed in Synthetic Football Fields	Suspect screening	Glass vial aluminum cap, stored in the dark at room temperature	Not available as of the publication date	In vitro simulation of digestion extraction, then solid-phase extraction or the bioaccessible fraction: 50 mg of Oasis HLB eluted with ethyl acetate. Ultrasound-assisted extraction for PAHs: crumb rubber in ethyl acetate, ultrasonic bath at 50 kHz for 20 min, PTFE filtered	GC/MS	Phenomenex Zebron ZB-Semivolatiles capillary column (30 mm×0.25 mm×0.25 µm film)	SRM	Suspect screening	Not available as of the publication date	(Armada et al. 2023)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Solubilization of Organic Compounds from Tire Particles Using Fish In Vitro Digestive Model	Digestate: LOD: 0.2 µg/L LOQ: 0.5 µg/L Cryogenically milled tire tread: LOD: 0.3 µg/L LOQ: 0.9 µg/L	Amber glass vessels	D5-6PPD-q, benzothiazole-d_4, aniline-d_5, and diphenylurea-d_{10},	Fish In Vitro Digestive Model, then liquid/liquid extracted twice DCM, then concentrated for analysis	UHPLC-HRMS	Waters ACQUITY UPLC HSS T3 (100×2.1 mm, 1.8 µm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase	ESI(+) mode MRM	269.05/ 106.99	269.05/184.09	(Masset et al. 2022)
Road Runoff	Not available as of the publication date	Water extracted within 48 hours of collection; glass bottles used for spikes	D5-6PPD-q	Filtered with 0.7-mm glass microfiber filters (Grade GF/F, cytiva), then SPE with Oasis 6 cc, 500 mg HLB cartridges, eluted with methanol	HPLC-MS/MS	Agilent InfinityLab Poroshell 120 EC-C8 LC (30 mm, 2.1 mm, 2.7 µm) column with 1 mM ammonium formate in water and methanol mobile phase	ESI(+) mode MRM	269.3/184.2	Not available as of the publication date	(Rodgers et al. 2023)
Urban River with Stormwater-Influenced Flows; Upstream, Downstream, and near WWTP Discharge	0.0098 µg/L	PET bottle, stored frozen (−18°C) *protocol established prior to discovery of 6PPD-q*	Not available as of the publication date	SPE with Waters HLB cartridges, 6 cc, 500 mg, eluted with methanol and concentrated	UPLC-HRMS	Kinetex 2.6 µm C18 column (50×4.6 mm). Solvent A, Milli-Q water (pH=7) with 0.1% of formic acid (A), and methanol with 0.1% of formic acid (B) mobile phase	Orbitrap HRMS, positive ionization mode with a HESI source (HESI-II probe). PRM for data acquisition	Targeted select ion monitoring: 269.20123	Not available as of the publication date	(Johannessen, Helm, et al. 2022)
Surface Water at Five Urban Centers in Queensland, Australia; Surface Waters and Stormwater in Australian Urban Tributary	Not available as of the publication date	600 mL polypropylene jars, frozen (−20°C) until analysis	Internal: d_6-5-methylbenzotriazole and d_5-atrazine Inject Internal: Caffeine-$^{13}C_3$	Water: filtered through Whatman 47 mm, 1 µm, GFF/B, SPE with Waters Oasis 6 cm^3 HLB cartridges, eluted with methanol concentrated with nitrogen Particles: Filter papers with particles dried in an incubator at 60°C for 3 hours and stored at 4°C for analysis; filters were cut into eight equal segments, and one segment was loaded into an 80 µL pyrolysis cup	Water: LC-MS/MS Particles: Pryo-GC/MS (not analyzed for 6PPD-q)	LC: Phenomenex Kinetex biphenyl 100 Å analytical column (2.6 µm, 50×2.1 mm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase pryo-GCMS: Particulates captured on the 1 µm filter analyzed for TRWPs and polymers by pryo-GC/MS	ESI(+) mode MRM	LC-MS/MS: 269/184 Pyro-GC/MS: Full-scan mode over a mass range of 40 to 600 m/z	269/107269/93	(Rauert et al. 2022)
Exposure Concentrations during Acute Toxicity Studies	LOD: 0.5 µg /L LOQ: 1.8 µg /L	Glass tanks, beakers, and bottles	Not available as of the publication date	Direct-inject	LC-MS/MS	Shim-pack VP ODS C18 (150×2.0 mm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase	ESI(+) mode MRM	269/185	Not available as of the publication date	(Hiki et al. 2021)
Exposure Concentrations during Zebrafish Behavior and Neurotransmitter Studies	Not available as of the publication date	Not available as of the publication date	Not available as of the publication date	Extracted with ACN	HPLC-MS/MS	Athena C18-WP (2.1×50 mm, 3.0 µm) column with water and methanol mobile phase	ESI(+) mode MRM	269.3/184.1	269.3/211.0	(Ji, Huang, et al. 2022)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Surface Water from Two Urbanized Watersheds	LOQ: 0.0065 µg/L	PE bottles, held for 72 hours refrigerated, and then frozen	Atrazine-d_5 and melamine-$^{13}C_3$	To ensure efficient extraction of transformation products with unknown chemical structures, three different SPE methods were employed	UPLC-MS/MS	Kinetex 2.6 µm C18 column (50×4.6 mm), mobile phase A consisting of Milli-Q water (pH=7), and mobile phase B consisted of methanol	HESI source (HESI-II probe) operated in positive ionization mode. Data acquisition was achieved using PRM	269.20123	Not available as of the publication date	(Johannessen, Helm, and Metcalfe 2021)
Surface Water, Groundwater, Stormwater, and Suspended Particles from Stormwater	MDL: 0.048 ng/L MQL: 0.160 ng/L	Stainless-steel bucket, 0.7 µm GFFs to collect suspended particles, water samples in HDPE bottles NaN_3 (0.05%) to inhibit microbial activity, stored at 4°C	D5-6PPD-q	Filtered through 0.7 µm GFFs (Grade GF/F; Whatman, UK), NaN_3 (0.05%) was added into water samples to inhibit microbial activity. The water samples and suspended particles were stored at 4°C until they were extracted. SPE Water samples: adjusted to pH 2 and EDTA added, then Waters Oasis HLB cartridges (6 mL, 200 mg), eluted with methanol, taken to dryness with nitrogen and redissolved in 10% methanol and filtered with 0.22 µm nylon microfiltration membrane Suspended Particles: freeze-dried and sequential ultrasonicated with methanol, dryness with nitrogen and redissolved in 10% methanol and filtered with 0.22 µm nylon microfiltration membrane	UPLC-MS/MS	Waters XBridge BEH C18 column (2.1×100 mm, 2.5 µm) column with 0.05% formic acid in water and 0.05% formic acid in ACN mobile phase	ESI(+) mode MRM	269.20/ 184.10	269.20/185.15	(R. Zhang, Zhao, Liu, Tian, et al. 2023)
Surface Water, Groundwater, and Stormwater, and Suspended Material	MDL: 0.048 ng/L MQL: 0.160 ng/L	Stainless-steel bucket, 0.7 µm GFFs to collect suspended particles, water samples in HDPE bottles NaN_3 (0.05%) to inhibit microbial activity, stored at 4°C	D5-6PPD-q	Water samples were adjusted to pH=2 using 3 mol/L HCl, SPE Oasis HLB cartridges, eluted with methanol, evaporated to almost dryness, redissolved in 10% methanol and nylon filtered	LC-MS/MS	Column: Poroshell HPH-C18 column (2.1×100 mm, 2.7 µm) with a C18 guard column (2.0 × 4 mm). Mobile Phase: water (0.1%formic acid) and methanol (0.1% formic acid)	ESI(+) mode MRM	269.20/ 184.10	269.20/185.15	(R. Zhang, Zhao, Liu, Tian, et al. 2023)
Mammalian Cells	LOQ: 0.11 ng/g	Not available as of the publication date	Internal: Coumaphos-d_{10} Surrogate: Benzophenone-d_{10}	Digestion mixtures were extracted by SPE with Waters Oasis HLB 1 cc 30 mg cartridges, eluted with 8:2 methanol: ACN, and concentrated by vacuum concentrator	UPLC-HRMS	Waters ACQUITY BEH C18 UPLC column (2.1×100 mm, 1.7 µm in particle size and 130 Å in pore size) with 0.1% formic acid in water and 0.1% formic acid in ACN mobile phase	Positive-ion mode PRM mode	269.0/184.1	269.0/212.4 269.0/92.8	(Wu et al. 2023)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
Influent, Effluent, and Biosolids in Four WWTPs in Hong Kong	Influent: LOQ: 0.12 ng/L LOD: 0.037 ng/L Other: LOQ: 0.06 ng/L LOD: 0.018 ng/L Biosolids: LOQ: 0.31 ng/g LOD: 0.092 ng/g	Glass bottles, held on ice and transferred to lab within 2 hours Wastewater: glass microfiber filtered (1.2 µm, Whatman, Hillsboro, USA) to remove suspended particulate matter, added 5% (v/v) methanol to inhibit microbial growth, stored in the dark at 4°C until extraction. Biosolids and filtered suspended particulate matter: freeze-dried, homogenized, 60-mesh sieve, stored at−20°C until extraction	Surrogate: diphenylamine-d_{10}, Internal: D5-6PPD-q	Glass bottles, held on ice and transferred to lab within 2 hours Wastewater: serial liquid/liquid dichloromethane extraction, purification with Envi-carbSPE cartridge and eluted with ethanol/dichloromethane(2:8, v/v), taken to near dryness with nitrogen, redissolved with ACN and nylon filtered Biosolids and filtered suspended particulate matter: serial ultrasonication with dichloromethane and ACN, purification with Envi-carbSPE cartridge and eluted with ethanol/dichloromethane(2:8, v/v), taken to near dryness with nitrogen, redissolved with ACN and nylon filtered	LC-MS/MS	Waters ACQUITY HSS T3 column (1.8µm, 2.1×100 mm), where the mobile phase consisted of 0.1% formic acid in deionized water (A) and 0.1% formic acid in ACN (B)	ESI(+) mode MRM	269.2/93.1	269.2/ 184.1269.2/ 212.1	(Cao et al. 2023)
Influent and Effluent from Municipal, Hospital, and Industrial WWTPs	LOD: 0.120 ng/L	Upon arrival at lab, hydrochloric acid added to a pH 2, stored at −20°C	D5-6PPD-q	0.7 µm GFF then SPE Oasis HLB (6 mL, 200 mg) eluted with methanol, evaporated to almost dryness with nitrogen, redissolved with 10% methanol	LC-MS/'MS	Waters Xbridge BEH C18 column (2.1 mm ID, 100 mm, 2.5 µm), 0.05% formic acid in Milli-Q water (mobile phase A) and ACN (mobile phase B)	MRM	269.2/184.10	269.2/185.15	(R. Zhang, Zhao, Liu, Thomes, et al. 2023)
Urban Water System: Surface Water, Surface Rainfall Runoff (Hardened Pavement, Road, Farmland), Influents and Effluents WWTP, and Six Points Along Drinking Water Treatment Sections	LOD: 0.04 ng/L LOQ: 0.12 ng/L	Glass amber bottles, immediately adjusted to pH 3.0 with 4 M H_2SO_4, added 5% methanol (v/v) to inhibit microbial growth, transported in cold ice boxes, stored at (4°C) before processing and extracted within 48 hours	Not available as of the publication date	Filtered through 0.7 µm GFF membranes, filter membrane serial sonication extraction with methanol and 0.1% formic acid, added to filtered water. SPE Oasis HLB cartridges (500 mg, 6 mL), eluted methanol ethyl acetate, and dichloromethane. Taken to dryness with nitrogen and redissolved with methanol and PTFE filtered	UPLC-MS/MS	Column not listed; 0.1% formic acid; (A) and methanol (B) mobile phase	ESI(+) mode MRM	269.05/92.7	269.05/ 185269.05/ 183.8	(H.-Y. Zhang, Huang, Liu, Hu, et al. 2023)

Table 5-2. Summary of sample collection and analytical method information for studies of 6PPD

Matrix	Detection Limit	Container and Storage	Internal or Surrogate Standards	Sample/Pretreatment, Extraction, and Cleanup	Instrumental Analysis	LC or GC	MS	Quantitation Ion	Confirmation Ion	Reference
TWP Solvent Extracts, TWP Aqueous Leachate, Roadway Runoff, Roadway-Impacted Creek Samples	Creek LOD: 3.3 ng/L LOQ: 4.9 ng/L Roadway Runoff LOD: 2.4 ng/L LOQ: 3.9 ng/L TWP Leachate LOD: 2.8 ng/L LOQ: 4 ng/L TWP Methanolic Extracts LOD: 3.4 ug/g LOQ: 11 ug/g	Roadway Runoff: grab and ISCO sampler, stored at 4°C and extracted within 24 hours of sample collection	D5-6PPD-q	TWP samples methanol-extracted; TWP leachate, roadway runoff, and creek water samples were SPE extracted (Oasis HLB cartridges, eluted with methanol and concentrated to 1 mL)	LC-MS/MS	Agilent Poroshell HPH-C18 column (2.1×100 mm, 2.7 µm) preceded with a C18 guard column(2.0×4 mm). LC-MS grade water (A) and methanol (B), both with 0.1% formic acid as the mobile phase	ESI+ mode dMRM	269.2/184.0	269.2/107.1	(Zhao et al. 2023)

Notes: µL=microliter, µm=micrometer, ACN=acetonitrile, ASE=accelerated solvent extractor, BEH=bridged ethyl-siloxane/silica hybrid, cc=cubic centimeter, DCM= dichloromethane, dMRM=dynamic multiple reaction monitoring mode, EDTA= ethylenediaminetetraacetic acid, ESI=electrospray ionization, g=gram, GAPS=Global Atmospheric Passive Sampling, GC=gas chromatography, GC/MS=gas chromatography / mass spectrometry, GFF=glass fiber filter, g/L=grams per liter, HDPE=high-density polyethylene, HESI=heated electrospray ionization, HLB=hydrophilic-lipophilic-balanced, HPLC=high-performance liquid chromatography, HPLC-HRMS=high-performance liquid chromatography–high-resolution mass spectrometry, HPLC-MS/MS=high-performance liquid chromatography– tandem mass spectrometry, HRGC/HRMS= high-resolution gas chromatography / high-resolution mass spectrometry , HRMS=high-resolution mass spectrometry, HSS T3= high-strength silica, trifunctionally bonded, ID=inner diameter, ISTD=internal standards, LC=liquid chromatography, LC-HRMS=liquid chromatography–high-resolution mass spectrometry, LC-MS/MS=liquid chromatography / tandem mass spectrometry, LOQ=limit of quantitation, MeOH=methanol, MDL=method detection limit mg=milligram, mg/kg=milligrams/kilogram, mL=milligrams per liter, mm=millimeter, mM=millimolar, mmol=millimol, mol/L=mol per liter, MQL=method quantification limit, MRL=method reporting limit, MRM=multiple reaction monitoring mode, MS2=tandem mass spectrometry, ng/L=nanograms per liter, PAH=polynuclear aromatic hydrocarbon, PE=polyethylene, pg/m^3=picograms per cubic meter, PRM=Parallel reaction monitoring, PTFE=polytetrafluoroethylene, PUF=polyurethane foam, Pyro-GC/MS=pyrolysis gas chromatography / mass spectrometry, SPE=solid-phase extraction, SRM=selected reaction monitoring, SSTD=surrogate standard, TRC= Toronto Research Chemical, TRWP=tire- and road-wear particles, TWP=tire-wear particles, UPLC=ultra-performance liquid chromatography, UPLC-HRMS=ultra-performance liquid chromatography–high-resolution mass spectrometry, UPLC-MS/MS=ultra-performance liquid chromatography– tandem mass spectrometry, UPLC-TOF-MS=ultra-performance liquid chromatography–quadrupole time-of-flight–tandem mass spectrometry, v/v=volume per volume, WWTP=wastewater treatment plant.

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