| Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q |
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| Matrix | Detection Limit | Container & Storage | Internal or Surrogate Standards | Sample/Pretreatment, Extraction, and Cleanup | Instrumental Analysis | LC or GC | MS | Quantitation Ion | Confirmation Ion | Reference |
| Storm and Surface Waters | ML: 2 ng/L | Aqueous: full 250 mL amber glass bottle, samples held up to 14 days in the fridge | EIS: 6PPD‑q-13C6Non-extracted internal standard: D5-6PPD-q | SPE with ACN elution | LC-MS/MS | C18 column using a 0.2% formic acid in water and ACN mobile phase  | ESI+ (MRM) | 299.2/215.1 | 299.2/241.1 | (USEPA 2023) |
| Storm, Surface Water, and Solids | MRL:Aqueous: 2 ng/LSolids: 0.25 ng/g  | Aqueous: full 250 mL amber glass bottle, samples held up to 14 days in the fridge Solids: Full 120 mL amber glass jar | EIS: 6PPD‑q-13C6Non-extracted internal standard: D5-6PPD-q | Serial sonication with ACN and SPE with ACN elution | LC-MS/MS | C18 column using a 0.2% formic acid in water and ACN mobile phase  | ESI+ (MRM) | 299.2/215.1 | 299.2/241.1 | Eurofins |
| Water, Fish Bile, and Plasma,  | MDL:Plasma: 0.0075–0.025 ng/mLBile: 0.015–0.05 ng/mLFish: 0.07 ng/g | Glass vials or bottles | Surrogate: D5-6PPD-qInternal: Progesterone-d9 | Water: Liquid/liquid extraction: concentration/solvent exchangeBile and Plasma: Water dilution, SPE, analysis | LC-MS/MS | Phenomenex Kinetex C18 EVO (100×2.1mm, 1.7 µm particle size) | ESI+ (MRM) | 299/241 | 299/215 | Bile/plasma: (da Silva et al. In preparation) |
| Storm, Surface Waters, Effluent, Soil and Biosolid | MRL: 0.1 ng/L and 0.05 ng/g | Amber glass bottles (HDPE not recommended), store aqueous sample at 4°C and solids at -20°C. Aqueous sample stable for at least 35 days. Solids and biosolids are stable for at least 40 days.No significant recovery difference in river water stored at -20°C and 4°C for 35 days | D5-6PPD-q | Water Extraction: Liquid-liquid extraction. with DCMSoil and biosolid: Ultrasonic extraction. with MeCNCleanup: 2 g, 6 mL Silica SPE | 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 0.1% formic acid in ACN mobile phase  | Positive mode ionization | 299.4/241.0 | 299.4/215.0 | SGS AXYS Analytical Ltd |
| Water | MRL: 1 ng/LInstrument LOQ: 0.025 ng/mL | 250 mL bottles, no preservative, stored above freezing to 6°C for 28 days | D5-6PPD-q | SPE: Waters Oasis HLB 6 cc (200 mg) SPE cartridge (WAT 106202) or Bakerbond Speedisk H2O-Philic DVB (8072-07) cartridges, eluted with ACN  | LC-MS/MS | Phenomenex 00D-4622-AN (100 mm×2.1 mm, 2.6 μm biphenyl 100 Å) with 0.1% formic acid/water and 0.1% formic acid/ACN mobile phase | ESI+Dynamic MRM | 299.1/215.1 | 299.1/215.1 | Standard Operating Procedure MEL730136, Version 1.2 (Washington State Department of Ecology 2023) |
| 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+ | 299.1/241.2 | 299.1/215.1 | (Lane et al. 2024) |
| Influent and Effluent of WWTP | MDL: 4 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 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= 299.1754 | Information not available | (Maurer et al. 2023)  |
| Snow from Urban Street | LOQ: 25 ng/L | 250 mL glass bottles; kept frozen until further treatment and analyzed on the same day | Not available as of the publication date | Snow: thawed, centrifuge, supernatant direct injection, remaining snow particles ultrasonic bath with methanol, diluted 1:1 with water | UPLC-TOF-MS | HSS T3 column; 100×2.1 mm, 1.7 μm and Atlantis T3 (3 µm, 2.1mm x 100mm); (A) water with 0.1% formic acid (v/v) and (B) methanol with 0.1% formic acid (v/v) mobile phase | ESI(+) modeMRM | 299.0/215.1 | 299.0/241.1 | (Seiwert et al. 2022) |
| Influent and Effluent WWTP during Snow Melt, Rain, And Dry conditions | LOQ: 25 ng/L | 250 mL glass bottles | Not available as of the publication date | 2 mL of wastewater filtered using syringe filters (0.45 µm, RC membrane, Minisart RC4, Sartorius) | UPLC-TOF-MS | HSS T3 column; 100×2.1 mm, 1.7 μm and Atlantis T3 (3 µm, 2.1mm × 100 mm); (A) water with 0.1% formic acid (v/v) and (B) methanol with 0.1% formic acid (v/v) mobile phase | ESI(+) modeMRM | 299.0/215.1 | 299.0/241.1 | (Seiwert et al. 2022) |
| Air from 18 Major Cities That Comprise the GAPS Network | Instrument LOQ: 0.122 ng/mLMethod LOQ: 0.169 pg/m3 | PUF disk samplers collect 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) Water and 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 | 299.1754/ 187.0866 | Information not available | Johannessen, Saini, et al. 2022) |
| Fine Particulate Matter (PM2.5) | MQL: 0.08 pg/m3MDL: 0.02 pg/m3 | Quartz fiber filter | Surrogate standard diphenylamine-d10 and internal standard D5-6PPD-q | Serial ultrasonication with dichloromethane and ACN, concentrated with nitrogen in ACN and PTFE filtered | UHPLC-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 modeMRM | 299.2/241.1 | 299.2/215.1299.2/187.1 | (Wang et al. 2022) |
| Fine Particulate Matter (PM2.5) from Megacity | LOD: 5 pg/mL | Whatman medium-volume quartz fiber filters | Pyrene-d10 and benzophenone-d10 | 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 CH3COONH4 (A)/ MeOH (B) mobile phase | ESI(+) modeMRM | 299/187 | 299/ 215 | (Y. Zhang et al. 2022) |
| Size-Fractioned Atmospheric Particles and Dust of Different Indoor Environments | Ambient Particles LOD: 0.03 pg/m3Dust LOD: 0.03 ng/g | Eight-stage nonviable Anderson cascade impactor (TISCH-Model TE-20-800, USA) with a glass substrate membrane for ambient particle collection | Internal standard: 6PPD-q-13C6 | Ultrasonic extraction with n-hexane and acetone, centrifuged, and concentrated to near dryness, redissolved in methanol, and filtered 0.22 μm poly(ether sulfone) membrane | UPLC-MS/MS | Betasil C18 column (100×2.1 mm, particle size 3 μm, Thermo Scientific) with water (A) and ACN (B) mobile phase | ESI(+) modeMRM | 299.2/241.1 | 299.2/187.1 | (Y.-J. Zhang et al. 2022) |
| Airborne Particulate Matter Along a Highway in Mississippi, USA | LOD: 2.90 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 (5×2.1 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  | Not available as of the publication date | Precleaned nylon bag (pore size of 25 μm) | ISTD: coumaphos-d10SSTD: benzophenone-d10 | 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(+) modeMRM | 299.18/ 215.08 | 299.18/241.09299.18/256.12299.18/187.09 | (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 | 6PPD‑q-13C6 | 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(+) modeMRM | 299.2/241.1 | 299.2/215.1 | (Zeng et al. 2023) |
| Fish  | LOD: 0.0003 mg/kgLOQ: 0.001 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/80:10  | ESI(+) modeMRM | 299.2/215.2 | 299.2/187.2299.2/241.2 | (Ji, Li, et al. 2022)  |
| 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 μmMobile phase: 5 mmol/L ammonium formate solution (A), methanol (B) | ESI(+) modeMRM | 299.25/ 215.25 | 299.25/187.15 | (Fang et al. 2023)  |
| Embryonic Zebrafish | LOD: 0.089 ng/mLLOQ: 0.439 ng/mL | Glass trays | Not available as of the publication date | FastPrep homogenizer, sonication, and centrifuging | HPLC/MS-MS | Atlantis T3 C18-phase column (2.1mm × 50mm, 3μm; Waters) with an Atlantis T3 Security Guard column (2.1×10mm, Waters) | ESI(+) modeMRM | 298.979/ 241.10 | 298.979/ 215.10 | (Grasse et al. 2023) |
| Embryonic Zebrafish | LOD: 5 pg/mL | Well plate exposures | 6PPD-q-13C6 | 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) mobile phase | ESI(+) modeMRM | 299/187 | 299/ 215 | (S.-Y. Zhang, Gan, Shen, Jiang, et al. 2023)  |
| 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 um). 0.1% formic acid in ultrapure water (A) and 0.1% formic acid in methanol mobile phase | ESI(+/-)full-scan mode | Not available as of the publication date | Not available as of the publication date | (Nair et al. 2023) |
| Fish Tissue (*S. l. pluvius*, *S. curilus*, and *O. m. masou*) | Not available as of the publication date | Not available as of the publication date | 6PPD‑q-13C6 | Wet tissue into polypropylene tube, homogenized with glass beads, ACN centrifugation | LC-MS/MS | Shim-pack VP ODS column (150 mm×2.0 mm, silica-based C18 stationary phase); the mobile phase was 0.1% ammonium acetate (pH: about 5) and methanol (1:8 v/v)  | Full-scan mode | Transitions from m/z 299 to 241, m/z 305 to 247, and m/z 315 to 231 were used for the quantification of 6PPD‑q | Not available as of the publication date | (Hiki and Yamamoto 2022) |
| Lumpfish Blood | LOD: 0.1 pg | Not available as of the publication date | Internal: D5-6PPD-qRecovery: 6PPD‑q‑13C6 | Vortex-sonication and centrifuged | HRGC/HRMS | TG-5SILMS column (30 m, 0.25 mm ID, film thickness—0.25 μm) | Non-targeted screeningFull-scan mode | Not available as of the publication date | Not available as of the publication date | (Hägg et al. 2023) |
| Exposure Concentrations in Toxicity Studies | LOD: 0.05 ug/L | Aerated 45 L rectangular glass tanks, 150 L inert glass-fiber Krescel tanks, 700 L glass-fiber Min-o-Cool tanks | D5-6PPD-q | Direct injection of exposure concentrations | UPLC-HRMS | Phenomenex Kinetex 1.7 µm XB-C18 column and SecurityGuard C18 guard column) with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase | Positive HESI mode | 299.1754/ 215.0819 | 299.1754/ 187.0869299.1754/ 243.1132 | (Brinkmann et al. 2022) |
| Human Urine from General Adults, Children, and Pregnant Women | MDL: 0.021 ng/mL | Urine immediately transferred to the laboratory, stored at −40°C until analysis; glass used during laboratory extractions | 6PPD‑q-13C6 | 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(+) modeMRM | 299.2/241.1 | 299.2/215.1 | (Du et al. 2022) |
| Honey | LOD: 0.0003 mg/kgLOQ: 0.001 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/80:10  | ESI(+) modeMRM | 299.2/215.2 | 299.2/187.2299.2/241.2 | (Ji, Li, et al. 2022) |
| Lettuce (*Valerianella locusta*) plant and roots & 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 filterRoots: Freeze-dried roots, suspended in nutrient solution, reciprocal shaker, centrifuge, nylon syringe filterNutrient Solution: serial liquid−liquid extraction, nylon syringe filter | UPLC-MS/MSUPLC-HRMS | C18 column (ACQUITY HSS T3, 1.8 µm, Waters), ultrapure water (phase A) and ACN (phase B), both containing 0.1% formic acid mobile phase | ESI(+) modeMRM | 299/256.1 | 299/241299/215299/187 | (Castan et al. 2023) |
| Soil, Water, Atmospheric Particles; Urban Runoff Water Samples Were Collected in a Dense Traffic Urban Area | IQL: 0.023 ng/mL | Soil: stainless-steel shovel, transported to lab within 2 hours, freeze-dried, homogenized, sieved through a 60 meshAtmospheric particle: collected 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-d10Surrogate: D5-6PPD-q | Soil: serial ultrasonication with ACN, concentrated to dryness with nitrogen, redissolved in methanol and 0.45 μm nylon filteredAtmospheric particles: serial ultrasonication with dichloromethane and ACN, concentrated to near dryness with nitrogen, redissolved in ACN and filteredWater: 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(+) modeFull-scan and data-dependent acquisition mode | 299.2/241.1 | 299.2/215.1 | (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 SPE or the bioaccessible fraction: 50 mg of Oasis HLB eluted with ethyl acetateUltrasound-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 m×0.25 mm×0.25 μm film)  | SRM | Suspect screening | Not available as of the publication date | (Armada et al. 2023) |
| Solubilization of Organic Compounds from Tire Particles Using FishIn VitroDigestive Model | Digestate LOD: 0.1 μg/LLOQ: 0.3 μg/LCryogenically milled tire treadLOD: 0.2 μg/LLOQ: 0.5 μg/L | Amber glass vessels | D5-6PPD-q, benzothiazole-d4, aniline-d5, and diphenylurea-d10, | FishIn Vitro Digestive Model and coingestion experiments, then serial liquid/liquid extraction with dichloromethane 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(+) modeMRM | 299.00/ 187.00 | 299.00/241.00 | (Masset et al. 2022) |
| Road Runoff | 98% Confidence MDL: 13.98 ng/L | 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(+) modeMRM | 299.0/215.1 | 299.0/187.0 | (Rodgers et al. 2023) |
| Urban River with Stormwater-influenced Flows; Upstream, Downstream, and near WWTP Discharge | LOQ: 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, and Solvent B, methanol with 0.1% of formic acid mobile phase | Orbitrap HRMS, positive ionization mode with a HESI source (HESI-II probe). PRM for data acquisition | Targeted select ion monitoring: 299.17540 | Not available as of the publication date | (Johannessen et al. 2022) |
| Urban Runoff from Cold Climate: Stormwater, Snowmelt, River Water | LOD: 1.2 ng/mLLOQ: 3.3 ng/mL | 4 L and 1 L Nalgene bottles | D5-6PPD-q | Whatman Grade GF/F glass microfiber filters (0.7 μm) SPE with Waters Oasis HLB (500 mg, 6 cc), eluted with methanol and DCM, concentrated to dryness with nitrogen, redissolved in 1:1: methanol:water  | UHPLC-HRMS | UHPLC: Phenomenex Kinetex 1.7 µm XB-C18-LC (100×2.1 mm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase | Positive mode HESI PRMSuspect screening: full MS/ddMS2 | 299.1754/ 215.0819 | Ions monitored during the suspect screening299.1754/ 187.0869299.1754 /241.0974299.1754/ 256.1210299.1754/ 200.0071299.1754/ 243.1132299.1754/ 100.1122 | (Challis et al. 2021) |
| Surface Water at Five Urban Centers in Queensland, Australia; Surface Waters and Stormwater Australian Urban Tributary | MDL: 0.05 ng/L | 600 mL polypropylene jars, frozen (−20°C) until analysis | Internals: d6-5-methylbenzotriazole and d5-atrazineInject Internal: Caffeine-13C3 | Water: filtered through Whatman 47 mm, 1 μm, GFF/B, SPE with Waters Oasis 6 cm3 HLB cartridges, eluted with methanol concentrated with nitrogenParticles: 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/MSParticles: Pyro-GC/MS (not analyzed for 6PPD‑q) | LC: Phenomenex Kinetex biphenyl 100 Å analytical column (2.6 μm, 50 mm×2.1 mm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phasePryo-GC/MS: Particulates captured on the 1 μm filter analyzed for TRWPs and polymers with pryo-GC/MS | ESI(+) modeMRM | LC-MS/MS: 299/241PRYO-GC/MS: Full-scan mode over a mass range of 40 to 600 m/z | 299/215299/187 | (Rauert, Vardy, et al. 2022; Rauert, Charlton, et al. 2022)  |
| Fish Media during Acute Toxicity Studies with Atlantic Salmon (*Salmo salar*) and Brown Trout (*Salmo trutta*) alevins | LOD: 0.006 μg/LLOQ: 0.020 μg/L | Water samples were collected in preprepared probes 1.5‐mL polypropylene tube with a 2×2 cm piece of dust‐free paper wipe; 1‐mL aliquots of fish media were collected and transferred to individual probes. Probes were kept at 5°C and were analyzed 5 days after the exposure trial | 6PPD‑q-13C6 | Direct injection and analysis of probes | LC-MS/MS | Agilent Eclipse Plus C18 RRHD ultra(U)HPLC column (3×50‐mm, 1.8‑µm) column with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase | Jet Stream electrospray interface operated in positive-ion mode | 299.1/187.3 | 299.1/77.3 | (Foldvik et al. 2022) |
| Exposure Concentrations during Acute Toxicity Studies of Freshwater Fish and Crustacean Species (*Danio rerio, Oryzias latipes, Daphnia magna*, and *Hyalella azteca*) | LOD: 0.05 μg/LLOQ: 0.17 μg/L | Glass tanks, beakers, and bottles | Not available as of the publication date | Direct-inject and direct-inject with dilutions  | 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(+) modeMRM | 299/241 | 299/187299/215 | (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(+) modeMRM | 299.2/215.2 | 299.2/187.2299.2/241.2 | (Ji, Huang, et al. 2022)  |
| Surface Water from Two Urbanized Watersheds  | LOQ: 0.0065 μg/L | PE bottles, held for 72 hours refrigerated, and then frozen  | Atrazine-d5 and melamine--13C3 | 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 | 299.1754 |  | (Johannessen, Helm, and Metcalfe 2021) |
| Surface Water, Groundwater, and Stormwater, and Suspended Material  | MDL: 0.029 ng/LMQL: 0.098 ng/L | Stainless-steel bucket, 0.7 μm GFFs to collect suspended particles, water samples in HDPE bottles NaN3 (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) Mobile phase: water (0.1%formic acid) and methanol (0.1% formic acid)  | ESI(+) modeMRM | 299.15/ 241.10 | 299.15/187.10 | (R. Zhang, Zhao, Liu, Tian, et al. 2023) |
| Water Samples from Coexposures of Coho and Chum with Nominal 320 mg/L TWP | Not available as of the publication date | 4-L precleaned amber glass bottles | Not available as of the publication date | SPE (3 mL, 100 mg Infinity Osorb), eluted with methanol, and concentrated to 1 mL with nitrogen | LC-QTOF-HRMS | Agilent ZORBAX Eclipse Plus 2.1×100 mm (analytical), 2.1×5 mm (guard), 1.8-µm particle size gradient 5 mM ammonium acetate plus 0.1% acetic acid in each of deionized water and methanol mobile phase | Two methods were applied to estimate the ‘equivalent concentration’ of TWPs in exposure water: nontarget HRMS features and measured concentrations tire-derived chemicals | Not available as of the publication date | Not available as of the publication date | (McIntyre et al. 2021) |
| Mammalian Cells | Not available as of the publication date | Not available as of the publication date | Internal: coumaphos-d10 (ISTD) Surrogate: benzophenone-d10 | 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 modePRM mode | 299.18/ 215.08 | 299.18/241.09299.18/256.12299.18/187.09 | (Wu et al. 2023) |
| Influent, Effluent, and Biosolids in Four WWTPs in Hong Kong | InfluentLOQ: 0.02 ng/LLOD: 0.005 ng/LOtherLOQ: 0.01 ng/LLOD: 0.002 ng/LBiosolidsLOQ: 0.04 ng/gLOD: 0.012 ng/g | Glass bottles, held on ice and transferred to lab within 2 hoursWastewater: glass microfiber filtered (1.2 μm, Whatman, Hillsboro, USA) to remove suspended particulate matter, added 5% (v/v) methanol to inhibiting microbial growth, stored in the dark at 4°C until extractionBiosolids and filtered suspended particulate matter: freeze-dried, homogenized, 60-mesh sieve, stored at−20°C until extraction | Surrogate: diphenylamine-d10Internal: D5-6PPD-q | Glass bottles, held on ice and transferred to lab within 2 hoursWastewater: 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 filteredBiosolids 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 (1.8 μm, 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(+) modeMRM | 299.2/241.1 | 299.2/215.1299.2/187.1 | (Cao et al. 2023) |
| Runoff Samples from Tunnel Washing, Runoff Treatment Plant, and Downstream of the Plant Drain; Two Water Samples from Puddles Were Included: One Was Runoff from an Artificial Soccer Turf Field and One from a Puddle on a Country Road | LOQ: 5 ng/L | Water samples were kept at 5°C prior to sample preparation and were analyzed within 2 weeks after collection | 6PPD‑q-13C6 | Clean Samples: transfer to polypropylene tube, centrifuge, SPE with Oasis HLB 30 mg/1 mL, eluted with methanol, evaporated to dryness with nitrogen, redissolved in ACNDirty Water Samples: transfer to polypropylene tube, centrifuge, lipophilic constituents extracted with dichloromethane and orbital shaker, centrifuged. Organic phase collected and diluted with hexane and SPE Phenomenex Strata Si-1 silica (55 μm, 70 Å, 100 mg/1 mL), eluted with dichloromethane, evaporated to dryness with nitrogen, redissolved in ACN | LC-MS/MS | Eclipse Plus C18 RRHD LC column (1.8 µm, 3.0×50 mm, Agilent Technologies). Mobile phase solvent A was 0.1% formic acid in water, and mobile phase B was 0.1% formic acid in ACN | ESI(+) modeMRM | 299.1/187.3 | 299.1/77.3 | (Kryuchkov et al. 2023) |
| Surface Water and Stormwater Samples and Tire/Artificial Turf Particle Suspensions | LOQ: 8 ng/L | Amber glass bottles; raw samples stored at 4°C, analyzed within 1 week of sampling | Not available as of the publication date | A CP-MIMS immersion probe was constructed from a 7.6 cm length of dense PDMS hollow fiber membrane (inside diameter of 190 μm, outside diameter of 300 μm, Permselect, Medarray Inc., Ann Arbor, MI) | HRMS | CP-MIMS membrane permeate was collected off-line and directly infused | ESI(+) modeHSAID source | 299/215 | 299/243299/256299/100 | (Monaghan et al. 2021) |
| Influent and Effluent from Municipal, Hospital, and Industrial WWTPs  | LOD: 0.098 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 | 299.15/ 241.10 | 299.15/187.10 | (R. Zhang, Zhao, Liu, Thomes, et al. 2023, 410) |
| 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: 0.05 ng/LLOQ: 0.17 ng/L | Glass amber bottles, immediately adjusted to pH 3.0 with 4 M H2SO4, 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(+) modeMRM | 299.28/ 241.10 | 299.28/214.98 | (H.-Y. Zhang, Huang, Liu, Hu, et al. 2023) |
| TWP Solvent Extracts, TWP Aqueous Leachate, Roadway Runoff, Roadway-Impacted Creek Samples | CreekLOD: 1.2 ng/LLOQ: 3.1 ng/LRoadway RunoffLOD: 2.1 ng/LLOQ: 5.7 ng/LTWP LeachateLOD: 2.4 ng/LLOQ: 6.8 ng/LTWP Methanolic ExtractsLOD: 0.12 μg/gLOQ: 0.4 μg/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, were used as mobile phases | ESI+ modedMRM | 299.2/215.1 | 299.2/187.1 | (Zhao et al. 2023)  |
| Stormwater Runoff | LOQ Stormwater: 5.1 ng/L | For exposures, glass aquaria | D5-6PPD-q | SPE: Waters Oasis HLB (200 mg, 6 mL) cartridges, with 0.5 g pre-cleaned micro glass beads (Filter Aid 400, 3 M, MN) to prevent clogging, eluted with methanol, concentrated with nitrogen | UHPLC-MS/MS | Agilent Poroshell HPH-C18 (2.1×100 mm, 2.7 μm) with C18 guard column and gradient with 0.1% formic acid in water and 0.1% formic acid in methanol mobile phase | ESI+MRM | 299.2/215.1 | 299.2/287.1 | (Tian et al. 2022) |
| Stormwater Runoff |  |  |  | Sand filtration, ion exchange, XAD-2 fractionation, silica gel fractionation, parallel HPLC fractionation, sequential HPLC fractionation | UPLC-QTOF-MS/MSReversed-phase C18 analytical column (Agilent ZORBAX Eclipse Plus 2.1×100 mm, 1.8 µm) with a C18 guard column (2.1×5 mm, 1.8 µm). For ESI+: 0.1% formic acid in each of water (A) and methanol (B). for ESI-: 1 mM ammonium fluoride in water (A) and methanol (B)UPLC-Orbitrap-MSn Agilent InfinityLab Poroshell 120 EC-C18 column (2.1×100 mm, 1.9 μm) with 0.1% formic acid in each of DI water (A) and MeOH (B)GC-QTOF-HRMS Agilent HP-5MS UI column (30 m×0.25 mm×0.25 μm film thickness) | UPLC-QTOF-MS/MSESI+/-Full-scan HRMS data acquired at the range of 100–1,700 m/z in 2 GHz Extended Dynamic Range mode. For structure elucidation, MS/MS data were acquired by data-dependent acquisition (m/z 50–1700, collision-induced dissociation at 10, 20, and 40 eV) using lists of preferred precursors prioritized during initial MS-only screeningUPLC-Orbitrap-MSnESI+ with targeted MS2 and MS3 acquisitionGC-QTOF-HRMSThe acquisition rate was 200 spectra/s over the range of m/z 45–550. USEPA Method 8270 internal standards were added to monitor the analytical performance |  |  |  | (Tian et al. 2021) |

Notes: μg=, microgram, µg/L=micrograms/liter, μL=microliter, μm=micrometer, ACN=acetonitrile, ASE=accelerated solvent extractor, BEH=bridged ethyl-siloxane/silica hybrid, cc=cubic centimeter, CP-MIMS=condensed phase membrane introduction mass spectrometry, DCM=dichloromethane, dMRM=dynamic multiple reaction monitoring mode, EIS=extracted internal standard, ESI=electrospray ionization, g=gram, GAPS=Global Atmospheric Passive Sampling, GC=gas chromatography, GC/MS=gas chromatography / mass spectrometry, GC-QTOF-HRMS=gas chromatography–quadrupole time-of-flight–high-resolution 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-MS/MS=high-performance liquid chromatography– tandem mass spectrometry, HRGC/HRMS= high-resolution gas chromatography / high-resolution mass spectrometry, HRMS=high-resolution mass spectrometry, HSAID=hot-surface induced desolvation, HSS T3= high-strength silica, trifunctionally bonded, ID=inner diameter, ISTD=internal standards, IQL=instrument quantification limit, L=liter, LC=liquid chromatography, LC-HRMS=liquid chromatography–high-resolution mass spectrometry, LC-MS=liquid chromatography–mass spectrometry, LC-MS/MS=liquid chromatography / tandem mass spectrometry, LC-QTOF-HRMS=liquid chromatography / high-resolution mass spectrometry, LOD=limit of detection, LOQ=limit of quantitation, MeOH=methanol, mg=milligram, mg=milligram, mg/kg=milligram/kilogram, mL=milliliter, ML=minimum level of quantification, mm=millimeter, mM=millimolar, mmol=millimol, mmol/L=millimole per liter; mol/L=mol per liter, MDL=method detection limit, MQL=method quantification limit, MRL=method reporting limit, MRM=multiple reaction monitoring mode, MS2=tandem mass spectrometry, MS/ddMS2=mass spectrometry / data dependent tandem mass spectrometry, ng/g=nanograms/gram ng/L=nanograms per liter, ng/mL=nanograms per milliliter, PAH=polynuclear aromatic hydrocarbon, PDMS=polydimethylsiloxane, PET=polyethylene terephthalate, pg=picogram, pg/m3=picogram per cubic meter, PRM=parallel reaction monitoring, PTFE=polytetrafluoroethylene, PUF=polyurethane foam, Pryo-GC/MS=pyrolysis gas chromatography / mass spectrometry, QuEChERs=A solid-phase extraction method: Quick, Easy, Cheap, Effective, Rugged, and Safe, SPE=solid-phase extraction, SRM=selected reaction monitoring, SSTD=surrogate standard, TRWP=tire- and road-wear particles, TWP=tire-wear particles, UHPLC-HRMS=ultra-high–performance liquid chromatography– high-resolution mass spectrometry, UHPLC-MS/MS=ultra-high–performance liquid chromatography– tandem mass spectrometry, UPLC=ultra-performance liquid chromatography, UPLC-HRMS=ultra-performance liquid chromatography, UPLC-MS/MS=ultra-performance liquid chromatography–tandem mass spectrometry, UPLC-QTOF-MSMS=ultra-performance liquid chromatography–quadrupole time-of-flight–tandem mass spectrometry, UPLC-TOF-MS=ultra-performance liquid chromatography–time-of-flight–mass spectrometry, v/v=volume per volume, WWTP=wastewater treatment plant.

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