Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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-$^{13}C_6$ Non-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/L Solids: 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-$^{13}C_6$ Non-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/mL Bile: 0.015–0.05 ng/mL Fish: 0.07 ng/g	Glass vials or bottles	Surrogate: D5-6PPD-q Internal: Progesterone-d9	Water: Liquid/liquid extraction: concentration/solvent exchange Bile 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 DCM Soil and biosolid: Ultrasonic extraction. with MeCN Cleanup: 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/L Instrument 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)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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(+) mode MRM	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(+) mode MRM	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/mL Method LOQ: 0.169 pg/m^3	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)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
Fine Particulate Matter ($PM_{2.5}$)	MQL: 0.08 pg/m^3 MDL: 0.02 pg/m^3	Quartz fiber filter	Surrogate standard diphenylamine-d_{10} 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 mode MRM	299.2/241.1	299.2/215.1 299.2/187.1	(Wang et al. 2022)
Fine Particulate Matter ($PM_{2.5}$) from Megacity	LOD: 5 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 CH3COONH4 (A)/ MeOH (B) mobile phase	ESI(+) mode MRM	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/m^3 Dust 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-$^{13}C_6$	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(+) mode MRM	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-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	299.18/ 215.08	299.18/241.09 299.18/256.12 299.18/187.09	(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-$^{13}C_6$	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	299.2/241.1	299.2/215.1	(Zeng et al. 2023)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
Fish	LOD: 0.0003 mg/kg LOQ: 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(+) mode MRM	299.2/215.2	299.2/187.2 299.2/241.2	(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 μm Mobile phase: 5 mmol/L ammonium formate solution (A), methanol (B)	ESI(+) mode MRM	299.25/ 215.25	299.25/187.15	(Fang et al. 2023)
Embryonic Zebrafish	LOD: 0.089 ng/mL LOQ: 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(+) mode MRM	298.979/ 241.10	298.979/ 215.10	(Grasse et al. 2023)
Embryonic Zebrafish	LOD: 5 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) mobile phase	ESI(+) mode MRM	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-$^{13}C_6$	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-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)	Non-targeted screening Full-scan mode	Not available as of the publication date	Not available as of the publication date	(Hägg et al. 2023)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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/ 187.0869 299.1754/ 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-$^{13}C_6$	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	299.2/241.1	299.2/215.1	(Du et al. 2022)
Honey	LOD: 0.0003 mg/kg LOQ: 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(+) mode MRM	299.2/215.2	299.2/187.2 299.2/241.2	(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 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 UPLC-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(+) mode MRM	299/256.1	299/241 299/215 299/187	(Castan et al. 2023)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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 mesh Atmospheric 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-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	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 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 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 Fish In Vitro Digestive Model	Digestate LOD: 0.1 µg/L LOQ: 0.3 µg/L Cryogenically milled tire tread LOD: 0.2 µg/L LOQ: 0.5 µg/L	Amber glass vessels	D5-6PPD-q, benzothiazole-d_4, aniline-d_5, and diphenylurea-d_{10},	Fish In 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(+) mode MRM	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(+) mode MRM	299.0/215.1	299.0/187.0	(Rodgers et al. 2023)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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/mL LOQ: 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 PRM Suspect screening: full MS/ddMS2	299.1754/ 215.0819	Ions monitored during the suspect screening 299.1754/ 187.0869 299.1754 /241.0974 299.1754/ 256.1210 299.1754/ 200.0071 299.1754/ 243.1132 299.1754/ 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: 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: 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 phase Pryo-GC/MS: Particulates captured on the 1 µm filter analyzed for TRWPs and polymers with pryo-GC/MS	ESI(+) mode MRM	LC-MS/MS: 299/241 PRYO-GC/MS: Full-scan mode over a mass range of 40 to 600 m/z	299/215 299/187	(Rauert, Vardy, et al. 2022; Rauert, Charlton, et al. 2022)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
Fish Media during Acute Toxicity Studies with Atlantic Salmon (*Salmo salar*) and Brown Trout (*Salmo trutta*) alevins	LOD: 0.006 µg/L LOQ: 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-$^{13}C_6$	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/L LOQ: 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(+) mode MRM	299/241	299/187 299/215	(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(+) mode MRM	299.2/215.2	299.2/187.2 299.2/241.2	(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-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	299.1754		(Johannessen, Helm, and Metcalfe 2021)
Surface Water, Groundwater, and Stormwater, and Suspended Material	MDL: 0.029 ng/L MQL: 0.098 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) Mobile phase: water (0.1%formic acid) and methanol (0.1% formic acid)	ESI(+) mode MRM	299.15/ 241.10	299.15/187.10	(R. Zhang, Zhao, Liu, Tian, et al. 2023)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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-d_{10} (ISTD) 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	299.18/ 215.08	299.18/241.09 299.18/256.12 299.18/187.09	(Wu et al. 2023)
Influent, Effluent, and Biosolids in Four WWTPs in Hong Kong	<u>Influent</u> LOQ: 0.02 ng/L LOD: 0.005 ng/L <u>Other</u> LOQ: 0.01 ng/L LOD: 0.002 ng/L <u>Biosolids</u> LOQ: 0.04 ng/g LOD: 0.012 ng/g	Glass bottles, held on ice and transferred to lab within 2 hours <u>Wastewater:</u> 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 extraction <u>Biosolids and filtered suspended particulate matter:</u> 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 <u>Wastewater:</u> 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 <u>Biosolids and filtered suspended particulate matter:</u> 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(+) mode MRM	299.2/241.1	299.2/215.1 299.2/187.1	(Cao et al. 2023)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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-$^{13}C_6$	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 ACN Dirty 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(+) mode MRM	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(+) mode HSAID source	299/215	299/243 299/256 299/100	(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)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
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/L LOQ: 0.17 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	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	<u>Creek</u> LOD: 1.2 ng/L LOQ: 3.1 ng/L <u>Roadway Runoff</u> LOD: 2.1 ng/L LOQ: 5.7 ng/L <u>TWP Leachate</u> LOD: 2.4 ng/L LOQ: 6.8 ng/L <u>TWP Methanolic Extracts</u> LOD: 0.12 μg/g LOQ: 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+ mode dMRM	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)

Table 5-3. Summary of sample collection and analytical method information for studies of 6PPD-q

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
Stormwater Runoff				Sand filtration, ion exchange, XAD-2 fractionation, silica gel fractionation, parallel HPLC fractionation, sequential HPLC fractionation	UPLC-QTOF-MS/MS Reversed-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/MS ESI+/- 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 screening UPLC-Orbitrap-MSn ESI+ with targeted MS2 and MS3 acquisition GC-QTOF-HRMS The 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/m^3=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.

References

Armada, Daniel, Antia Martinez-Fernandez, Maria Celeiro, Thierry Dagnac, and Maria Llompart. 2023. “Assessment of the Bioaccessibility of PAHs and Other Hazardous Compounds Present in Recycled Tire Rubber Employed in Synthetic Football Fields.” *Science of the Total Environment* 857 (January):159485. https://doi.org/10.1016/j.scitotenv.2022.159485.

Brinkmann, Markus, David Montgomery, Summer Selinger, Justin G. P. Miller, Eric Stock, Alper James Alcaraz, Jonathan K. Challis, et al. 2022. “Acute Toxicity of the Tire Rubber–Derived Chemical 6PPD-Quinone to Four Fishes of Commercial, Cultural, and Ecological Importance.” *Environmental Science & Technology Letters*, March, acs.estlett.2c00050. https://doi.org/10.1021/acs.estlett.2c00050.

Cao, Guodong, Wei Wang, Jing Zhang, Pengfei Wu, Han Qiao, Huankai Li, Gefei Huang, Zhu Yang, and Zongwei Cai. 2023. “Occurrence and Fate of Substituted P-Phenylenediamine-Derived Quinones in Hong Kong Wastewater Treatment Plants.” *Environmental Science & Technology*, October. https://doi.org/10.1021/acs.est.3c03758.

Cao, Guodong, Wei Wang, Jing Zhang, Pengfei Wu, Xingchen Zhao, Zhu Yang, Di Hu, and Zongwei Cai. 2022. “New Evidence of Rubber-Derived Quinones in Water, Air, and Soil.” *Environmental Science & Technology* 56 (7): 4142–50. https://doi.org/10.1021/acs.est.1c07376.

Castan, Stephanie, Anya Sherman, Ruoting Peng, Michael T. Zumstein, Wolfgang Wanek, Thorsten Hüffer, and Thilo Hofmann. 2023. “Uptake, Metabolism, and Accumulation of Tire Wear Particle–Derived Compounds in Lettuce.” *Environmental Science & Technology* 57 (1): 168–78. https://doi.org/10.1021/acs.est.2c05660.

Challis, J. K., H. Popick, S. Prajapati, P. Harder, J. P. Giesy, K. McPhedran, and M. Brinkmann. 2021. “Occurrences of Tire Rubber–Derived Contaminants in Cold-Climate Urban Runoff.” *Environmental Science & Technology Letters* 8 (11): 961–67. https://doi.org/10.1021/acs.estlett.1c00682.

Du, Bibai, Bowen Liang, Yi Li, Mingjie Shen, Liang-Ying Liu, and Lixi Zeng. 2022. “First Report on the Occurrence of *N*-(1,3-Dimethylbutyl)-*N*′-Phenyl-*p*-Phenylenediamine (6PPD) and 6PPD-Quinone as Pervasive Pollutants in Human Urine from South China.” *Environmental Science & Technology Letters*, November. https://doi.org/10.1021/acs.estlett.2c00821.

Fang, Chanlin, Liya Fang, Shanshan Di, Yundong Yu, Xinquan Wang, Caihong Wang, and Yuanxiang Jin. 2023. “Characterization of N-(1,3-Dimethylbutyl)-*N*′-Phenyl-*p*-Phenylenediamine (6PPD)-Induced Cardiotoxicity in Larval Zebrafish (*Danio Rerio*).” *Science of the Total Environment* 882 (July):163595. https://doi.org/10.1016/j.scitotenv.2023.163595.

Foldvik, Anders, Fedor Kryuchkov, Roar Sandodden, and Silvio Uhlig. 2022. “Acute Toxicity Testing of the Tire Rubber–Derived Chemical 6PPD-Quinone on Atlantic Salmon (*Salmo Salar*) and Brown Trout (*Salmo Trutta*).” *Environmental Toxicology and Chemistry* 41 (12): 3041–45. https://doi.org/10.1002/etc.5487.

Grasse, Nico, Bettina Seiwert, Riccardo Massei, Stefan Scholz, Qiuguo Fu, and Thorsten Reemtsma. 2023. “Uptake and Biotransformation of the Tire Rubber–Derived Contaminants 6-PPD and 6-PPD Quinone in the Zebrafish Embryo (*Danio rerio*).” *Environmental Science & Technology* 57 (41): 15598–607. https://doi.org/10.1021/acs.est.3c02819.

Hägg, Fanny, Dorte Herzke, Vladimir A. Nikiforov, Andy M. Booth, Kristine Hopland Sperre, Lisbet Sørensen, Mari Egeness Creese, and Claudia Halsband. 2023. “Ingestion of Car Tire Crumb Rubber and Uptake of Associated Chemicals by Lumpfish (*Cyclopterus Lumpus*).” *Frontiers in Environmental Science* 11. https://doi.org/10.3389/fenvs.2023.1219248.

Hiki, Kyoshiro, Kenta Asahina, Kota Kato, Takahiro Yamagishi, Ryo Omagari, Yuichi Iwasaki, Haruna Watanabe, and Hiroshi Yamamoto. 2021. “Acute Toxicity of a Tire Rubber–Derived Chemical, 6PPD Quinone, to Freshwater Fish and Crustacean Species.” *Environmental Science & Technology Letters* 8 (9): 779–84. https://doi.org/10.1021/acs.estlett.1c00453.

Hiki, Kyoshiro, and Hiroshi Yamamoto. 2022. “The Tire-Derived Chemical 6PPD-Quinone Is Lethally Toxic to the White-Spotted Char *Salvelinus leucomaenis pluvius* but Not to Two Other Salmonid Species.” *Environmental Science & Technology Letters* 9 (12): 1050–55. https://doi.org/10.1021/acs.estlett.2c00683.

Huang, Wei, Yumeng Shi, Jialing Huang, Chengliang Deng, Shuqin Tang, Xiaotu Liu, and Da Chen. 2021. “Occurrence of Substituted *p*-Phenylenediamine Antioxidants in Dusts.” *Environmental Science & Technology Letters* 8 (5): 381–85. https://doi.org/10.1021/acs.estlett.1c00148.

Ji, Jiawen, Jinze Huang, Niannian Cao, Xianghong Hao, Yanhua Wu, Yongqiang Ma, Dong An, Sen Pang, and Xuefeng Li. 2022. “Multiview Behavior and Neurotransmitter Analysis of Zebrafish Dyskinesia Induced by 6PPD and Its Metabolites.” *Science of The Total Environment* 838 (September):156013. https://doi.org/10.1016/j.scitotenv.2022.156013.

Ji, Jiawen, Changsheng Li, Bingjie Zhang, Wenjuan Wu, Jianli Wang, Jianhui Zhu, Desheng Liu, et al. 2022. “Exploration of Emerging Environmental Pollutants 6PPD and 6PPDQ in Honey and Fish Samples.” *Food Chemistry* 396 (December):133640. https://doi.org/10.1016/j.foodchem.2022.133640.

Johannessen, Cassandra, Paul Helm, Brent Lashuk, Viviane Yargeau, and Chris D. Metcalfe. 2022. “The Tire Wear Compounds 6PPD-Quinone and 1,3-Diphenylguanidine in an Urban Watershed.” *Archives of Environmental Contamination and Toxicology* 82 (2): 171–79. https://doi.org/10.1007/s00244-021-00878-4.

Johannessen, Cassandra, Paul Helm, and Chris D. Metcalfe. 2021. “Detection of Selected Tire Wear Compounds in Urban Receiving Waters.” *Environmental Pollution* 287 (October):117659. https://doi.org/10.1016/j.envpol.2021.117659.

Kryuchkov, Fedor, Anders Foldvik, Roar Sandodden, and Silvio Uhlig. 2023. “Presence of 6PPD-Quinone in Runoff Water Samples from Norway Using a New LC–MS/MS Method.” *Frontiers in Environmental Chemistry* 4. https://www.frontiersin.org/articles/10.3389/fenvc.2023.1194664.

Lane, Rachael Frances, Kelly L. Smalling, Paul M. Bradley, Justin B. Greer, Stephanie E. Gordon, John D. Hansen, Andrew R. Spanjer, Dana W. Kolpin, and Jason R. Masoner. 2024. “Tire-Derived Contaminants 6ppd and 6ppd-Q: Analysis, Sample Handling, and Reconnaissance of United States Stream Exposures.” https://doi.org/10.2139/ssrn.4824411.

Masset, Thibault, Benoit J. D. Ferrari, William Dudefoi, Kristin Schirmer, Alan Bergmann, Etienne Vermeirssen, Dominique Grandjean, Luke Christopher Harris, and Florian Breider. 2022. “Bioaccessibility of Organic Compounds Associated with Tire Particles Using a Fish In Vitro Digestive Model: Solubilization Kinetics and Effects of Food Coingestion.” *Environmental Science & Technology* 56 (22): 15607–16. https://doi.org/10.1021/acs.est.2c04291.

Maurer, Loïc, Eric Carmona, Oliver Machate, Tobias Schulze, Martin Krauss, and Werner Brack. 2023. “Contamination Pattern and Risk Assessment of Polar Compounds in Snow Melt: An Integrative Proxy of Road Runoffs.” *Environmental Science & Technology* 57 (10): 4143–52. https://doi.org/10.1021/acs.est.2c05784.

McIntyre, Jenifer K., Jasmine Prat, James Cameron, Jillian Wetzel, Emma Mudrock, Katherine T. Peter, Zhenyu Tian, et al. 2021. “Treading Water: Tire Wear Particle Leachate Recreates an Urban Runoff Mortality Syndrome in Coho but Not Chum Salmon.” *Environmental Science & Technology* 55 (17): 11767–74. https://doi.org/10.1021/acs.est.1c03569.

Monaghan, Joseph, Angelina Jaeger, Alon R. Agua, Ryan S. Stanton, Michael Pirrung, Chris G. Gill, and Erik T. Krogh. 2021. “A Direct Mass Spectrometry Method for the Rapid Analysis of Ubiquitous Tire-Derived Toxin *N*-(1,3-Dimethylbutyl)-*N*′-Phenyl-*p*-Phenylenediamine Quinone (6-PPDQ).” *Environmental Science & Technology Letters* 8 (12): 1051–56. https://doi.org/10.1021/acs.estlett.1c00794.

Nair, Pranav, Jianxian Sun, Linna Xie, Lisa Kennedy, Derek Kozakiewicz, Sonya Kleywegt, Chunyan Hao, et al. 2023. “In Process: Synthesis and Toxicity Evaluation of Tire Rubber–Derived Quinones.” Preprint. Chemistry. https://doi.org/10.26434/chemrxiv-2023-pmxvc.

Olubusoye, Boluwatife S., James V. Cizdziel, Matthew Bee, Matthew T. Moore, Marco Pineda, Viviane Yargeau, and Erin R. Bennett. 2023. “Toxic Tire Wear Compounds (6PPD-Q and 4-ADPA) Detected in Airborne Particulate Matter Along a Highway in Mississippi, USA.” *Bulletin of Environmental Contamination and Toxicology* 111 (6): 68. https://doi.org/10.1007/s00128-023-03820-7.

Rauert, Cassandra, Nathan Charlton, Elvis D. Okoffo, Ryan S. Stanton, Alon R. Agua, Michael C. Pirrung, and Kevin V. Thomas. 2022. “Concentrations of Tire Additive Chemicals and Tire Road Wear Particles in an Australian Urban Tributary.” *Environmental Science & Technology*, January. https://doi.org/10.1021/acs.est.1c07451.

Rauert, Cassandra, Suzanne Vardy, Benjamin Daniell, Nathan Charlton, and Kevin V. Thomas. 2022. “Tyre Additive Chemicals, Tyre Road Wear Particles and High Production Polymers in Surface Water at 5 Urban Centres in Queensland, Australia.” *Science of The Total Environment* 852 (December):158468. https://doi.org/10.1016/j.scitotenv.2022.158468.

Rodgers, Timothy F. M., Yanru Wang, Cassandra Humes, Matthew Jeronimo, Cassandra Johannessen, Sylvie Spraakman, Amanda Giang, and Rachel C. Scholes. 2023. “Bioretention Cells Provide a 10-Fold Reduction in 6PPD-Quinone Mass Loadings to Receiving Waters: Evidence from a Field Experiment and Modeling.” *Environmental Science & Technology Letters*, June. https://doi.org/10.1021/acs.estlett.3c00203.

Seiwert, Bettina, Maolida Nihemaiti, Mareva Troussier, Steffen Weyrauch, and Thorsten Reemtsma. 2022. “Abiotic Oxidative Transformation of 6-PPD and 6-PPD Quinone from Tires and Occurrence of Their Products in Snow from Urban Roads and in Municipal Wastewater.” *Water Research* 212 (April):118122. https://doi.org/10.1016/j.watres.2022.118122.

Silva, D. da, J. Gates, L. Harding, S. O’Neil, and I. Schultz. In preparation. “Analysis of Multiple Tire Rubber-Derived Chemicals (TRCs) in Fish Bile and Plasma: Analytical Method Field Assessment in Puget Sound, WA.”

Tian, Zhenyu, Melissa Gonzalez, Craig A. Rideout, Haoqi Nina Zhao, Ximin Hu, Jill Wetzel, Emma Mudrock, C. Andrew James, Jenifer K. McIntyre, and Edward P. Kolodziej. 2022. “6PPD-Quinone: Revised Toxicity Assessment and Quantification with a Commercial Standard.” *Environmental Science & Technology Letters*, January, acs.estlett.1c00910. https://doi.org/10.1021/acs.estlett.1c00910.

Tian, Zhenyu, Haoqi Zhao, Katherine T. Peter, Melissa Gonzalez, Jill Wetzel, Christopher Wu, Ximin Hu, et al. 2021. “A Ubiquitous Tire Rubber–Derived Chemical Induces Acute Mortality in Coho Salmon.” *Science* 371 (6525): 185–89. https://doi.org/10.1126/science.abd6951.

USEPA. 2023. “Draft Method 1634: Determination of 6PPD-Quinone in Aqueous Matrices Using Liquid Chromatography with Tandem Mass Spectrometry (LC/MS/MS).” EPA 821-D-24-001. Office of Water (4303T), Office of Science and Technology. https://www.epa.gov/system/files/documents/2024-01/draft-method-1634-for-web-posting-1-23-24_508.pdf.

Wang, Wei, Guodong Cao, Jing Zhang, Pengfei Wu, Yanyan Chen, Zhifeng Chen, Zenghua Qi, Ruijin Li, Chuan Dong, and Zongwei Cai. 2022. “Beyond Substituted *p*-Phenylenediamine Antioxidants: Prevalence of Their Quinone Derivatives in $PM_{2.5}$.” *Environmental Science & Technology*, July, acs.est.2c02463. https://doi.org/10.1021/acs.est.2c02463.

Washington State Department of Ecology. 2023. “Standard Operating Procedure (SOP): Extraction and Analysis of 6PPD-Quinone (Mel730136, Version 1.2).”

Wu, Jiabin, Guodong Cao, Feng Zhang, and Zongwei Cai. 2023. “A New Toxicity Mechanism of *N*-(1,3-Dimethylbutyl)-*N*′-Phenyl-*p*-Phenylenediamine Quinone: Formation of DNA Adducts in Mammalian Cells and Aqueous Organisms.” *Science of the Total Environment* 866 (March):161373. https://doi.org/10.1016/j.scitotenv.2022.161373.

Zeng, Lixi, Yi Li, Yuxin Sun, Liang-Ying Liu, Mingjie Shen, and Bibai Du. 2023. “Widespread Occurrence and Transport of *p*-Phenylenediamines and Their Quinones in Sediments across Urban Rivers, Estuaries, Coasts, and Deep-Sea Regions.” *Environmental Science & Technology*, January, acs.est.2c07652. https://doi.org/10.1021/acs.est.2c07652.

Zhang, Hai-Yan, Zheng Huang, Yue-Hong Liu, Li-Xin Hu, Liang-Ying He, You-Sheng Liu, Jian-Liang Zhao, and Guang-Guo Ying. 2023. “Occurrence and Risks of 23 Tire Additives and Their Transformation Products in an Urban Water System.” *Environment International* 171 (January):107715. https://doi.org/10.1016/j.envint.2022.107715.

Zhang, Ruiling, Shizhen Zhao, Xin Liu, Margaret William Thomes, Chui Wei Bong, Dilanka N. D. Samaraweera, Tilak Priyadarshana, Guangcai Zhong, Jun Li, and Gan Zhang. 2023. “Fates of Benzotriazoles, Benzothiazoles, and *p*-Phenylenediamines in Wastewater Treatment Plants in Malaysia and Sri Lanka.” *ACS ES&T Water* 3 (6): 1630–40. https://doi.org/10.1021/acsestwater.2c00410.

Zhang, Ruiling, Shizhen Zhao, Xin Liu, Lele Tian, Yangzhi Mo, Xin Yi, Shiyang Liu, Jiaqi Liu, Jun Li, and Gan Zhang. 2023. “Aquatic Environmental Fates and Risks of Benzotriazoles, Benzothiazoles, and *p*-Phenylenediamines in a Catchment Providing Water to a Megacity of China.” *Environmental Research* 216 (January):114721. https://doi.org/10.1016/j.envres.2022.114721.

Zhang, Shu-Yun, Xiufeng Gan, Baoguo Shen, Jian Jiang, Huimin Shen, Yuhang Lei, Qiuju Liang, et al. 2023. “6PPD and Its Metabolite 6PPDQ Induce Different Developmental Toxicities and Phenotypes in Embryonic Zebrafish.” *Journal of Hazardous Materials* 455 (August):131601. https://doi.org/10.1016/j.jhazmat.2023.131601.

Zhang, Yanhao, Caihong Xu, Wenfen Zhang, Zenghua Qi, Yuanyuan Song, Lin Zhu, Chuan Dong, Jianmin Chen, and Zongwei Cai. 2022. “*P* -Phenylenediamine Antioxidants in $PM_{2.5}$: The Underestimated Urban Air Pollutants.” *Environmental Science & Technology* 56 (11): 6914–21. https://doi.org/10.1021/acs.est.1c04500.

Zhang, Ying-Jie, Ting-Ting Xu, Dong-Min Ye, Ze-Zhao Lin, Fei Wang, and Ying Guo. 2022. “Widespread *N*-(1,3-Dimethylbutyl)-*N*′-phenyl-*p*-phenylenediamine Quinone in Size-Fractioned Atmospheric Particles and Dust of Different Indoor Environments.” *Environmental Science & Technology Letters* 9 (5): 420–25. https://doi.org/10.1021/acs.estlett.2c00193.

Zhao, Haoqi Nina, Ximin Hu, Zhenyu Tian, Melissa Gonzalez, Craig A. Rideout, Katherine T. Peter, Michael C. Dodd, and Edward P. Kolodziej. 2023. “Transformation Products of Tire Rubber Antioxidant 6PPD in Heterogeneous Gas-Phase Ozonation: Identification and Environmental Occurrence.” *Environmental Science & Technology* 57 (14): 5621–32. https://doi.org/10.1021/acs.est.2c08690.