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## Artificial turf fields act as recurring point sources of metals and emerging tire-derived contaminants in stormwater

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Artificial turf fields are widely used globally. Each field contains an estimated 125 tonnes of infill material, which is often composed of ground post-consumer vehicle tires. Tires contain chemicals that are deleterious to human health and aquatic ecosystems, which may be mobilized from artificial turf fields. We conducted laboratory leaching experiments and analyzed stormwater from multiple artificial turf fields to quantify contaminant release. We found that crumb rubber infill released several toxicants including *N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine-quinone (6PPD-Q), zinc, and copper, at higher levels than alternative infill materials. 6PPD-Q and 6PPD concentrations in infill leachates did not decrease with infill age. Non-targeted analysis identified fourteen compounds associated with crumb rubber infill material, including the putative identification of the bioaccumulative toxicant leucomalachite green. The maximum 6PPD-Q concentration in stormwater draining from an artificial turf field ( $130.2 \text{ ng L}^{-1}$ ) was over three times higher than the  $\text{LC}_{50}$  for juvenile coho salmon (*Oncorhynchus kisutch*), which could increase concentrations to concerning levels in receiving water bodies. Zinc and copper concentrations peaked at  $30.3$  and  $726.4 \text{ } \mu\text{g L}^{-1}$ , respectively. These concentrations are toxicologically relevant but lower than those typically observed in urban road runoff. Our results indicate that a typical crumb rubber-infilled field likely releases <1% of the total 6PPD in the infill material to stormwater each year, indicating persistent release beyond the field lifespan. Thus, artificial turf fields can act as recurring point sources of contaminants at ecologically significant levels.

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### Environmental significance

Artificial turf fields commonly use crumb rubber infill prepared from used vehicle tires, which can potentially release tire-derived contaminants into stormwater draining artificial turf fields. The acute ecotoxicity of tire-derived compounds such as 6PPD-quinone indicates a need to assess the impacts of crumb rubber infill on stormwater contaminant concentrations. We found that 6PPD-quinone and other contaminants can leach from crumb rubber infill materials even when materials were sampled from fields near their typical end-of-life, indicating the potential for ongoing contaminant release. We also measured contaminant concentrations in stormwater draining artificial turf fields and detected concentrations of 6PPD-quinone that exceeded the  $\text{LC}_{50}$  value for coho salmon, indicating the potential for adverse impacts of turf fields on aquatic ecosystems.

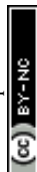
## 1 Introduction

Artificial turf fields were introduced in the 1960s as resilient and low-maintenance sports surfaces.<sup>1</sup> Today, there are more than 19 000 artificial turf fields in the United States and 600 to 750 new installations annually.<sup>2</sup> Most artificial turf fields utilize crumb rubber ground from post-consumer tires as infill material, and an average turf field contains 125 tonnes of crumb rubber, equivalent to approximately 20 000 passenger vehicle tires.<sup>3</sup> Crumb rubber or similar materials are commonly used for their elastomeric properties which provide a safer playfield surface and require less maintenance than grass. These fields

are commonly located in urbanized or semi-urbanized areas and connected to urban drainage systems that discharge to surface waters, creating potential pathways for contaminant transport to aquatic environments.

There are a range of toxicants associated with tire crumb rubber.<sup>4–13</sup> Ingredients with demonstrated toxicity include polycyclic aromatic hydrocarbons (PAHs),<sup>14–16</sup> carbon black (~28% by weight of tire mass),<sup>17</sup> and vulcanization accelerators (~4% by weight) including zinc oxide, *N,N'*-diphenylguanidine, and the probable human carcinogen 2-mercaptobenzothiazole (MBT).<sup>18</sup> Antiozonants (~1% by weight) including 6PPD (*N*-(1,3-dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine) are designed to migrate to the tire surface to react with ground-level ozone and prevent tire rubber from cracking. Additional additives (~1% by weight) can include compounds such as the corrosion inhibitor

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and UV-stabilizer 1,3-benzotriazole, a compound with moderate toxicity to aquatic organisms,<sup>19</sup> and the cross-linking agent hexamethoxymethylmelamine (HMMM). Chemical formulations for tire manufacturing are proprietary; the United States Environmental Protection Agency recently cataloged nearly 300 tire ingredients, but many compounds and their transformation products remain poorly characterized and poorly understood.<sup>20–22</sup>

Numerous studies have demonstrated that exposure to tire wear particles (TWPs) and their leachates can harm aquatic organisms.<sup>4,23–27</sup> Recent research has identified that the reaction between 6PPD and ambient ground-level ozone produces a quinone byproduct, 6PPD-Q,<sup>4</sup> which is acutely toxic to juvenile coho salmon (*Oncorhynchus kisutch*) and coastal cutthroat trout (*O. clarkii*) at levels as low as  $LC_{50} = 41 \text{ ng L}^{-1}$  and  $39.6 \text{ ng L}^{-1}$ , respectively.<sup>24,28</sup> Urban road runoff exposure can also be lethal to steelhead (*O. mykiss*) and chinook (*O. tshawytscha*) salmonids.<sup>27</sup> Embryonic zebrafish (*Danio rerio*) and water fleas (*Daphnia magna*) exhibited sublethal developmental abnormalities and mortality following exposure to tire leachate, with more severe effects associated with leachate from smaller particles.<sup>29</sup> Further, exposure to TWP has been shown to affect oxidative stress and energy metabolism in some crustaceans (*Eriocheir sinensis*)<sup>30</sup> and in blue mussels (*Mytilus edulis*).<sup>25</sup>

While recent research has examined TWPs and associated chemical release from roadways,<sup>6,9,11</sup> comparatively little is known about the chemical emissions from artificial turf fields.<sup>31</sup> Analyses of crumb rubber infill leachates have indicated elevated zinc and PAH concentrations,<sup>16</sup> and demonstrated the release of tire rubber additives such as 6PPD, 1,3-diphenylguanidine, MBT, and benzothiazole from crumb rubber material.<sup>32,33</sup> An analysis using non-targeted liquid chromatography and tandem mass spectrometry (LC-MS/MS) observed higher abundances of total rubber-associated compounds in leachates from crumb rubber infills less than two years of age.<sup>32</sup> Beyond laboratory experiments, Zhang *et al.* demonstrated the release of PAHs and microplastics from an artificial turf field during multiple rain events.<sup>15,34</sup> They measured event mean concentrations of PAHs approximately twelve times higher than values reported in urban roadway runoff.<sup>15</sup> A report released from the Connecticut Department of Environmental Protection<sup>35</sup> assessed the presence of polychlorinated biphenyls, semi-volatile and volatile organic compounds, and metals in storm-water runoff from artificial turf fields. Their research found that zinc levels emitted from the field exceeded acute aquatic toxicity criteria for surface waters.<sup>35</sup> Bryshun *et al.*<sup>36</sup> performed a pilot study which exposed crumb rubber infill material to real rainfall using a rooftop flow-through system. They report a median 6PPD-Q concentration of  $10.6 \mu\text{g L}^{-1}$  in rainwater following flow through crumb rubber material. Finally, Zhao *et al.*<sup>37</sup> assessed contaminant release from a variety of rubber-based consumer goods including crumb rubber and assessed PPD-related transformation products relative to total PPD concentration.

Alternative turf infill materials are commercially available but less common than crumb rubber due to the relatively low cost of crumb rubber material.<sup>3</sup> There is little information about these materials and their potential to induce aquatic toxicity.

Ethylene propylene diene terpolymer (EPDM) is a form of vulcanized rubber that is used as an alternative infill and may contain ingredients typically associated with tire crumb rubber.<sup>38</sup> Thermoplastic elastomer (TPE), another popular alternative infill, is not a vulcanized rubber but rather a copolymer composed of styrene, polyethylene, paraffin oil, chalk, carbon black, and other additives.<sup>38</sup>

Prior studies on turf infill materials indicate the potential for substantial contaminant leaching from artificial turf fields, but few data exist for emerging compounds like 6PPD-Q. Additional research is needed to assess the chemical profile of alternative turf materials in comparison to crumb rubber infill, and to validate trends between infill material age and chemical leaching. To the best of our knowledge, no studies have characterized the release of 6PPD or 6PPD-Q from artificial turf fields under field conditions during rainfall. Thus, this study aims to (1) assess the release of metals and emerging organic contaminants from crumb rubber and alternative artificial turf infill material *via* leaching experiments, (2) identify potential unknown contaminants *via* non-targeted analysis of infill leachates, and (3) quantify the temporal release of contaminants from artificial turf fields during rainfall events.

## 2 Methods

### 2.1 Infill material collection

We collected artificial turf field infill samples from twelve fields across the Vancouver Metropolitan Area in British Columbia, Canada. We selected sites representative of a range of ages (1–14 years post-installation) and infill materials. Field ages and corresponding cumulative precipitation data are provided in Table S1 and Fig. S1. We collected approximately 5–50 g of post-consumer ground tire rubber ( $n = 9$ ), EPDM ( $n = 1$ ), and TPE ( $n = 2$ ) infill materials by hand from the four corners of each field and transferred the material into amber glass jars pre-cleaned *via* triple rinsing and baking at  $400 \text{ }^\circ\text{C}$  for  $>4$  hours. We stored infill at  $-17 \text{ }^\circ\text{C}$  until analysis and equilibrated samples to room temperature before use. We also obtained a tire tread standard, produced *via* cryogenic milling of treads from a road-representative mix of new vehicle tires, from the U.S. Tire Manufacturers' Association<sup>40</sup> for use as a reference material.

### 2.2 Bench-scale leaching and extraction of infill material

To evaluate the potential for contaminant release from infill materials, we conducted bench-scale leaching and extraction procedures to characterize both inorganic and organic constituents.

To assess metals released from infill material, we deposited approximately 50 mg of infill material into a polypropylene tube with ultrapure water ( $>18 \text{ M}\Omega \text{ cm}$ ) at a 10 mg infill  $\text{mL}^{-1}$  water ratio. We inverted tubes and vortexed for 30 seconds, then placed them upright on an orbital shaker at 75 rpm for 24 hours at room temperature. Next, we syringe-filtered the supernatant through a  $0.45 \mu\text{m}$  PES filter to remove debris, acidified to 2% (v/v) with trace metals grade nitric acid ( $\text{HNO}_3$ ), and stored at room temperature for at least 24 hours until analysis by



inductively coupled plasma mass spectrometry (ICP-MS). We performed leaching in duplicate on individual field samples.

To assess organic chemicals present in the infill material, we deposited approximately 50 mg infill material into a pre-cleaned 20 mL glass vial at a ratio of 10 mg infill mL<sup>-1</sup> LC-MS grade methanol. We sonicated samples for 60 minutes, let them rest for 45 minutes, and collected and syringe-filtered supernatant using a 0.22 µm glass fiber syringe filter to remove debris. We added internal standards of 6PPD-Q (6PPD-Q-*d*<sub>5</sub>) and 1*H*-benzotriazole (BTZ-*d*<sub>4</sub>) at a final concentration of 1.2 µg L<sup>-1</sup>, and stored extracts at -17 °C until LC-MS/MS analysis. This method was developed with a goal of assessing near-surface organic concentrations, adapted from previous methods.<sup>37</sup> We performed extractions in triplicate on individual field samples. Method blanks were prepared following an identical procedure using no infill material.

### 2.3 Stormwater sample collection and processing

We collected stormwater from the subsurface drainage systems of three artificial turf fields (CR-H, CR-B, CR-D) across three storm events. At each field, we collected samples from a manhole isolated from other road runoff or wastewater inputs, which we confirmed *via* construction documents, site inspection, and consultation with field operators. See the SI for further information on the storm events sampled, including simplified construction documents. Notably, samples collected from fields CR-B and CR-D were influenced by stormwater draining from surrounding grass fields. We do not anticipate that this introduced metals or tire-derived contaminants, but it likely resulted in greater dilution and thus lower contaminant concentrations in our samples.

Storm Event 1 followed a 19 days antecedent dry period; we collected samples for metals analysis from field CR-H which contained 13.5 year-old infill at time of sampling. Storm Event 2 followed a 2.5 days antecedent dry period; we collected samples for metals analysis from field CR-B which contained 3.5 year-old infill at time of sampling. Storm Event 3 followed a 3 days antecedent dry period; we collected samples for metals and organics analysis from field CR-D which contained 6.5 year-old infill at time of sampling.

We used an autosampler (ISCO Model 6712) to collect stormwater samples into pre-cleaned bottles at time intervals ranging from 30 minutes to multiple hours, with the shortest intervals at the start of the precipitation events, intended to capture a high-resolution view of contaminant export following precipitation onset. The autosampler was triggered on a timer at the start of sampling. Before each sample was collected, the instrument flushed the sample line with stormwater, then the autosampler collected approximately 325 mL of stormwater. We retrieved samples within 36 hours following collection and aliquoted to remove large debris such as plant material or organisms when present. Samples were not filtered before analysis to avoid potential analyte loss, consistent with previously published methods for stormwater analysis.<sup>41–43</sup>

We transferred a subsample of 10 mL into a polypropylene tube, acidified to 2% (v/v) with trace metals-grade nitric acid

(HNO<sub>3</sub>), allowed the sample to rest for 24 hours, and stored vials at room-temperature before analysis of total metals concentration by inductively coupled plasma mass spectrometry (ICP-MS).

For Storm Event 3, we spiked the remaining stormwater sample (approximately 300 mL) to a final concentration of 7.2 µg L<sup>-1</sup> in SPE extract internal standard (6PPD-Q-*d*<sub>5</sub> and BTZ-*d*<sub>4</sub>) immediately following retrieval and stored samples at 4 °C before extraction within 7 days. Previous work has shown that portable autosamplers can sample organic contaminants like 6PPD-Q without substantial artifacts from sorption or contaminant transformation in storage.<sup>44</sup> We concentrated samples *via* manual solid-phase extraction using Oasis HLB cartridges (Waters). We conditioned cartridges sequentially with 6 mL LCMS-grade methanol and 6 mL ultrapure water, loaded samples at room temperature at approximately 1 mL min<sup>-1</sup>, washed cartridges with 6 mL DI water, dried cartridges, and subsequently eluted with 12 mL LCMS-grade methanol. We then aliquoted extracts into 2 mL glass vials and stored at -17 °C until LC-MS/MS analysis within 4 days.

### 2.4 Quantification of inorganic contaminants

We used an Agilent 7850 Inductively Coupled Plasma-Mass Spectrometer to analyze trace metals concentrations in stormwater and leachate samples. We verified instrument performance during batch tuning, and metals were quantified in helium collision mode. We prepared a seven-point external calibration curve (0–500 µg L<sup>-1</sup>) using an Environmental Calibration Standard (Agilent 5183-4688). We applied internal standardization using a multi-element internal standard mix (Agilent 5183-4681). For quality assurance and quality control, ultrapure water blanks or field blanks and known concentration spikes were analyzed at minimum every ten samples to assess background contamination, carryover, or instrument drift. Further information can be found in SI Section 1.3.

### 2.5 Quantification of organic contaminants

We obtained analytical standards for four organic compounds associated with tire rubber or urban runoff: 6PPD, 6PPD-Q, 1*H*-benzotriazole (BTZ), and HMMM. See the SI for details on chemical standard stocks and their associated deuterated internal standards. We purchased the analytical standards in solid form, dissolved them into LCMS-grade methanol at 1 mg mL<sup>-1</sup>, vortexed for 30 seconds, and stored samples in amber glass vials at -17 °C until use. We used an ultra performance liquid chromatograph (UPLC, Thermo Scientific Vanquish) and triple-quadrupole MS (Thermo Scientific Altis QqQ) to quantify organic contaminants. We performed separation on a Phenomenex LC Column (150 × 3 mm, Synergi 4 µm Hydro-RP A). Samples were quantified using an 8-point calibration curve across 0.01–10 µg L<sup>-1</sup> for stormwater samples and a 10-point calibration curve across 0.01–10 µg L<sup>-1</sup> for leachate, with a fit of *R*<sup>2</sup> > 0.99. We calculated the method detection limit (MDL) as the average of method blanks plus 3 times the standard deviation of method blanks. Surrogate recoveries ranged from 50–150% and samples with surrogate concentrations beyond this



range were rejected before data analysis. Full LC-MS/MS operating details and data processing details can be found in SI Section 1.4.

## 2.6 Non-target LC-MS/MS analysis of infill leachate

We used a UPLC (Thermo Scientific Vanquish) paired with an Orbitrap mass spectrometer (Thermo Fisher Orbitrap Exploris 240) to perform a non-targeted analysis of organic contaminants in infill leachate. Chromatographic separation was performed on a Phenomenex LC Column (150 × 3 mm, Synergi 4 μm Hydro-RP A). We operated the instrument in positive electrospray ionization (ESI) mode, with a full scan of 60 K resolution and subsequent data-dependent tandem mass spectra acquisition for up to 10 scans per cycle at 15 K resolution. Full LC-MS/MS operating details can be found in SI Section 1.5.

## 2.7 Data analysis

We conducted data analysis and visualization in R (v4.4.1) using the “tidyverse”,<sup>45</sup> “data.table”,<sup>46</sup> “gggh4x”,<sup>47</sup> and “pheatmap”<sup>48</sup> packages. In results, we report non-detect data as half of the MDL for statistical purposes but show non-detect data as zero in visualizations unless otherwise noted. Results are presented as median ± standard deviation (SD) of the replicates unless otherwise noted.

We retrieved precipitation data from an Environment Canada weather station<sup>49</sup> and from a university-operated weather station,<sup>50</sup> reported at hourly resolution and 15 minutes resolution, respectively. For comparison to contaminant concentration data, we interpolated precipitation time-point data linearly at 1 minute resolution using base R when necessary.

We performed a statistical assessment of quantitative LC-MS/MS data *via* a Wilcoxon rank-sum test to compare contaminant concentrations between crumb rubber and alternative infill materials, two-sample *t*-tests to assess differences between materials categorized as young (≤3 years) and old (>3 years), and linear regression analysis to evaluate the relationship between infill age and the relative abundance of 6PPD-Q to 6PPD. We define statistical significance as  $p < 0.05$ .

We utilized Compound Discoverer software (Thermo Fisher Scientific v3.3.3.200) to process non-target data. Spectra were filtered to a minimum precursor mass of 100 Da, a minimum peak intensity of  $1 \times 10^4$ , at least three scans per peak, and a minimum peak rating of 5/10 in at least two samples. Background features were identified by selectively removing spectra with a relative sample: background abundance less than 5 : 1 across sample groups within Compound Discoverer. We putatively identified chemical compositions based on accurate mass matching and MS/MS spectral similarity, and we classify features using a modified version of the Schymanski scale classification system.<sup>51</sup> For accurate mass matching, we queried ChemSpider and the EPA Tire Crumb Rubber list based on full scan *m/z*. When tandem mass spectra data were available, we assigned annotations by matching mzCloud and mzVault, where GNPS,<sup>52</sup> Mass Bank of North America,<sup>53</sup> and MassBank EU<sup>54</sup> spectra were included. Further details are available in Fig. S2 and Table S11.

# 3 Results

## 3.1 Trace contaminants in turf infill material leachates

Leaching trends from infill materials demonstrated substantial variability across compounds and infill age (Fig. 1). However, leachate from alternative infills generally exhibited lower concentrations of the quantified contaminants than crumb rubber leachates. Concentrations of 6PPD-Q (Wilcoxon rank-sum test,  $p = 0.016$ ), 6PPD ( $p = 0.016$ ), and zinc ( $p = 0.016$ ) were significantly higher in crumb rubber leachates than in alternative infill materials, consistent with previous studies demonstrating the presence of these compounds in tire-derived materials.<sup>14,16,32</sup> Concentrations of copper ( $p = 0.712$ ), HMMM ( $p = 0.09$ ), and BTZ ( $p = 0.701$ ) were not significantly different between crumb rubber and alternative materials. Copper is a common co-pollutant in crumb rubber typically released from brake pads but not directly used in tire rubber.<sup>55</sup> Detection of HMMM in our samples is consistent with findings from McMinn *et al.*<sup>32</sup> who found that high initial concentrations of HMMM in new crumb rubber rapidly decreased as the material aged, suggesting that HMMM may degrade relatively quickly under environmental conditions. BTZ was detected in few leachates, resulting in low statistical power to distinguish concentrations across material types. Further, BTZ is not commonly used in tire rubber, not listed on the EPA Tire Crumb Rubber List, and could be considered a co-contaminant for its more frequent use as a corrosion inhibitor, including in vehicle brakes.<sup>48</sup> We did not detect lead in any infill material leachates. Lead was historically used for tire vulcanization but has been largely replaced by zinc oxide,<sup>56</sup> although it is still listed on the EPA Tire Crumb Rubber List.

Contaminant concentrations observed in our leaching experiments were generally lower than previously reported concentrations. McMinn *et al.*<sup>32</sup> reported leachate concentrations of  $570 \pm 140 \text{ mg kg}^{-1}$  and  $27 \pm 6.5 \text{ mg kg}^{-1}$  of 6PPD and 6PPD-Q, respectively, from new crumb rubber infill retrieved prior to installation. The lower concentrations observed in our study may have been due in part to our use of less intensive leaching methods, which included a 105 minutes solvent extraction with sonication, intended to represent near-surface organic concentrations. Notably, the extraction protocol used by McMinn *et al.* employed a 24 hours leaching period into 1 : 1 acetone : methanol, substantially longer than our 105 minutes methanol leaching procedure; thus, their results may more accurately represent total organic concentrations rather than near-surface. In the context of aqueous leaching from fields, it is likely that both methods overrepresent the concentration of organics which may leach into stormwater. Similarly, the metals concentrations observed in our experiments were lower than those reported in a review of contaminant release from crumb rubber infill across various recent studies.<sup>14</sup> Reported zinc concentrations ranged from approximately 1000 to 10 000  $\text{mg kg}^{-1}$ . While these values are higher than levels observed in our sampling, most extractions were performed with the addition of nitric acid, which likely enhanced the leaching efficiency of metals from the rubber matrix.



## Leached compounds from alternative and crumb rubber

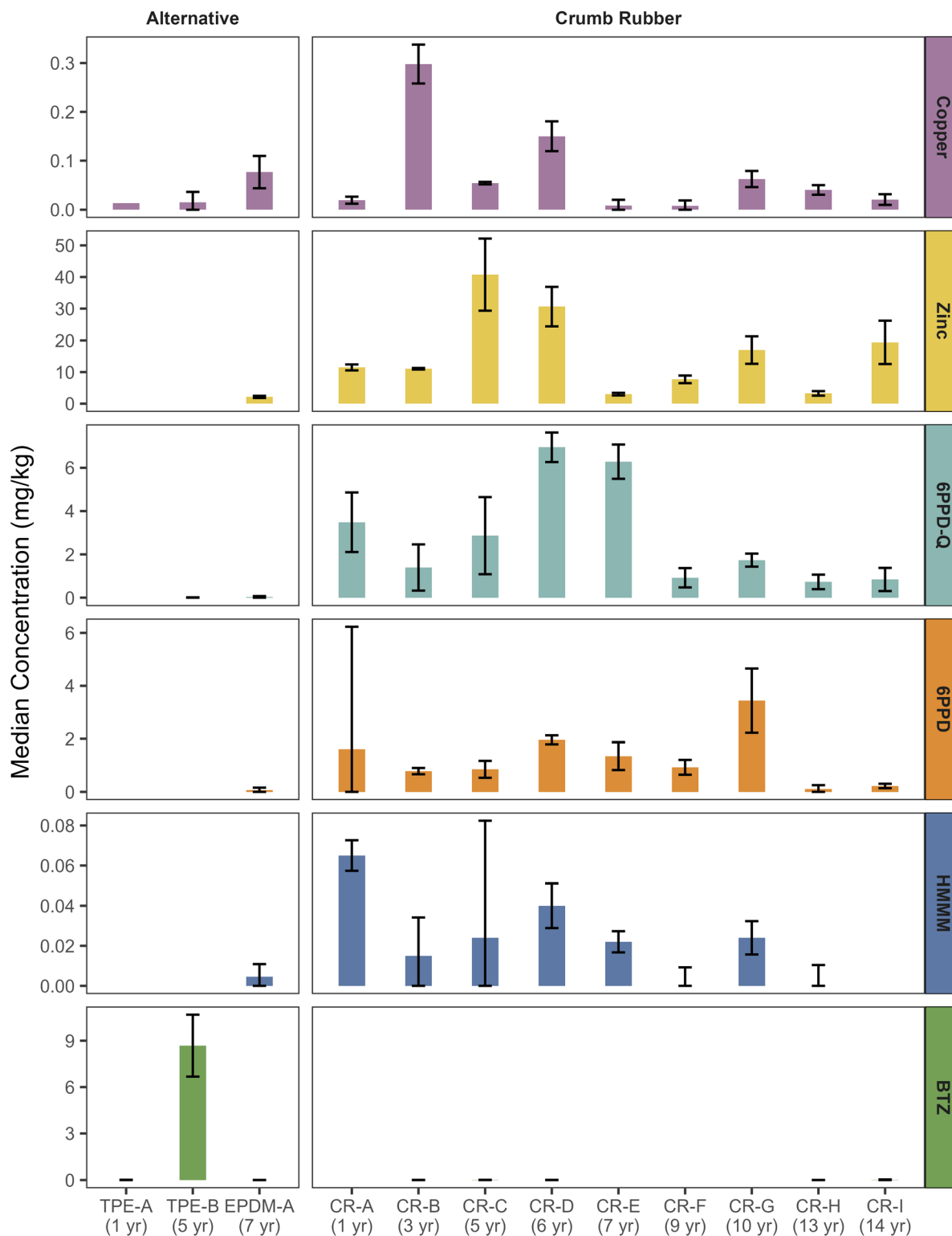


Fig. 1 Concentrations of trace contaminants in leachate from alternative and crumb rubber infill materials. Bars represent median concentration in  $\text{mg kg}^{-1}$  and error bars represent the standard deviation of the replicates.

We did not observe a significant relationship between leachate concentrations and crumb rubber infill material age. To assess correlation between infill age and chemical leaching

potential, we grouped crumb rubber infill data with a high number of detections into two categories: old ( $>3$  years,  $n = 7$ ) and young ( $\leq 3$  years,  $n = 2$ ). A comparison of the two groups



indicated that copper ( $t$ -test,  $p = 0.302$ ), 6PPD-Q ( $p = 0.760$ ), 6PPD ( $p = 0.502$ ), zinc ( $p = 0.136$ ), and HMMM ( $p = 0.461$ ) did not exhibit significant relationships with age. These results contrast with the McMinn *et al.* finding of an inverse relationship between age and 6PPD leaching potential, particularly in 0–2 years old turf infill.<sup>32</sup> We also indirectly evaluated exhaustion of 6PPD in infill materials over time by assessing whether there was a linear correlation between age and relative 6PPD-Q:6PPD abundance (Fig. S3) in our aged samples. We found no significant correlation (linear regression,  $R^2 = 0.003$ ,  $p = 0.890$ ). McMinn *et al.*<sup>32</sup> and Bryshun *et al.*<sup>36</sup> both assessed release from newer infill material and observed that concentrations of 6PPD-Q in extracts decreased over time, particularly for material less under 2 years in age. Our extraction method was most similar to that performed by Zhao *et al.*<sup>37</sup> who also performed a short extraction including sonication into methanol. They identified a weak correlation with respect to age for 6PPD-Q:6PPD and total concentration of PPD-related compounds; however, their results appeared modest, partially due to uncertainty with infill ages.

The lack of an observed relationship between age and leachate concentrations herein could be attributable to several factors. It is possible that age-dependent leaching trends may only be relevant for very young infill material, which was underrepresented in our dataset. Alternatively, total leachable concentrations (measured *via* the more intensive extraction conditions of McMinn *et al.*) may decline more quickly with age than near-surface 6PPD and 6PPD-Q, resulting in weak correlations between age and the leaching concentrations observed under the gentler extraction conditions we used. Total chemical content may change over the course of infill use, particularly considering environmental weathering processes that occur on artificial turf fields. For instance, a surface temperature of 93 °C was recorded on one artificial turf field during ambient temperatures of just 37 °C, indicating that high temperatures could contribute to physical-chemical degradation.<sup>57</sup> In another study, substantial physical degradation of crumb rubber was observed from aged (>10 years) infill<sup>58</sup> which implies the potential for chemical transformation or the release of nano-sized particles into stormwater.

Further, variability in annual precipitation may influence aging; we assessed if there was a trend between total cumulative precipitation and mean contaminant release (Table S12) and found no significant trend for 6PPD, 6PPD-Q, or BTZ, but a significant correlation ( $p = 0.01$ ) for HMMM, possibly indicating that historical precipitation may influence contaminant release, particularly for hydrophilic compounds. Finally, the chemical leaching potential of turf fields could also be partially replenished when field managers periodically add small quantities of new infill to counteract the loss of material that is inadvertently removed from fields *via* shoes or other incidental transportation. At our sites, infill replenishment likely minorly increased the available 6PPD mass reservoir. Detailed records for our sites were not kept, but typical practice would replace approximately 3–5% of the total field mass annually.<sup>39</sup>

Our results suggest that 6PPD exhaustion over time may not be the dominant mechanism determining 6PPD-Q and 6PPD

release for older infill material beyond one year of age. Instead, our results demonstrate that turf fields continue to release 6PPD and 6PPD-Q throughout their lifetime, potentially influenced by material surface area, infill material replenishment, and environmental variables including wet-dry cycling, cumulative precipitation, and UV or thermal exposure. In our experiments, we detected 6PPD and 6PPD-Q even in leachates from the oldest field in our study, indicating potential for release throughout the approximately 8 to 15 year lifetime of an artificial turf field.<sup>59,60</sup>

### 3.2 Tire-related compounds identified in leachate *via* non-targeted analysis

Non-targeted analysis of leachates from the 12 field infill materials and the cryomilled tire tread (CMTT) standard cumulatively yielded 110 833 features. After filtering to remove background noise *via* presence in blanks, 16 275 features remained. Using a modified version of the Schymanski classification system<sup>51</sup> for mass spectra, we assigned Level 1 confidence to compounds confirmed *via* comparison with analytical standards, specifically the four previously quantified organic analytes: 6PPD, 6PPD-Q, BTZ, and HMMM. We assigned Level 2 confidence to 95 features that exhibited full spectral matches with reference tandem mass spectra in mzCloud or mzVault databases. We assigned Level 3 confidence to 3410 features that had a single predicted molecular formula and at least one exact mass match within ChemSpider. We assigned Level 4 confidence to 574 features with a unique predicted molecular formula but no corresponding database match. Features classified as Level 5 lacked sufficient spectral or singular compositional predictions and were excluded to maintain brevity.

Of the 95 Level 2 features, we manually identified 39 unique compounds based on their spectral similarity and unique retention times. Prior to reporting, we removed features corresponding to substances unlikely to originate from infill materials, including naturally occurring compounds ( $n = 5$ ), pharmaceuticals ( $n = 12$ ), personal care products ( $n = 3$ ), pesticides ( $n = 2$ ), and laboratory reagents ( $n = 2$ ); see Table S13 for further information. We anticipate that these chemical contaminants were introduced to the turf field materials *via* environmental deposition or other inadvertent transportation. The remaining 15 compounds, considered relevant to infill materials, are summarized in Table 1. We classified compounds according to their predominant use: rubber manufacturing ( $n = 5$ ), plasticizers ( $n = 5$ ), flame retardants ( $n = 3$ ), UV-stabilizers ( $n = 2$ ), dyes ( $n = 1$ ), and various/unknown usage ( $n = 2$ ).

Nontarget analysis results demonstrate that within each infill type, the most abundant compounds are largely unique (Fig. 2A and B); generally rubber processing agents dominated the crumb rubber and CMTT leachates while UV stabilizers and plasticizers were more prevalent in alternative infill materials. Notably, octrizole, dibutyl phthalate, and 4-hydroxy-1-(2-hydroxyethyl)-2,2,6,6-tetramethylpiperidine were largely associated with TPE infill, whereas leucomalachite green and tris(2-ethylhexyl)phosphate were primarily concentrated in CMTT



**Table 1** Infill-relevant compounds putatively identified with Level 2 confidence, respective CAS numbers, predominant use, and whether the compound appears on the U.S. EPA Tire Crumb Rubber List (EPACR)

Predicted name	CAS	Use	Notes	EPACR
<i>N</i> -Cyclohexyl-2-benzothiazol-amine (NCBA)	28291-75-0	Rubber	Impurity of <i>N</i> -cyclohexyl-2-benzothiazole sulfenamide (CBS) vulcanization agent	No
Cyclohexylamine	101-83-7	Rubber	Vulcanization accelerator	Yes
<i>N,N'</i> -Diphenylguanidine	20277-92-3	Rubber	Vulcanization accelerator	Yes
Acridine-9(10 <i>H</i> )-thione	6540-78-9	Rubber	Suspected byproduct of vulcanization process	Yes
Triphenylphosphine oxide	791-28-6	Rubber, flame retardant	Catalyst in manufacturing	No
Di(2-ethylhexyl)phthalate (DEHP)	117-81-7	Plasticizer	Endocrine disrupting compound	Yes
Dibutyl phthalate	84-74-2	Plasticizer	Endocrine disrupting compound	Yes
Diisobutyl phthalate	84-69-5	Plasticizer	Endocrine disrupting compound	Yes
Tris(2-ethylhexyl)phosphate	78-42-2	Plasticizer, flame retardant	Aquatic toxicant	No
Diphenylphosphate	838-85-7	Plasticizer, flame retardant	Potential endocrine disrupting compound	No
4-Hydroxy-1-(2-hydroxyethyl)-2,2,6,6-tetramethylpiperidine	52722-86-8	UV-stabilizer	Used in paints	No
Octrizole	3147-75-9	UV-stabilizer	Used in paints and personal care products	No
Leucomalachite green	129-73-7	Dye/antifungal agent	Known to bioaccumulate in fish <sup>62</sup>	No
Dipropylene glycol dimethyl ether	34590-94-8	Variou	Used in paints, various other applications	Yes
3-Phenyl-2,3-dihydro-1,3-benzothiazol-2-imine	46493-66-7	Variou	Minimal information	No

samples. Di(2-ethylhexyl)phthalate and diethylamine exhibited the greatest mean abundance in EPDM infill.

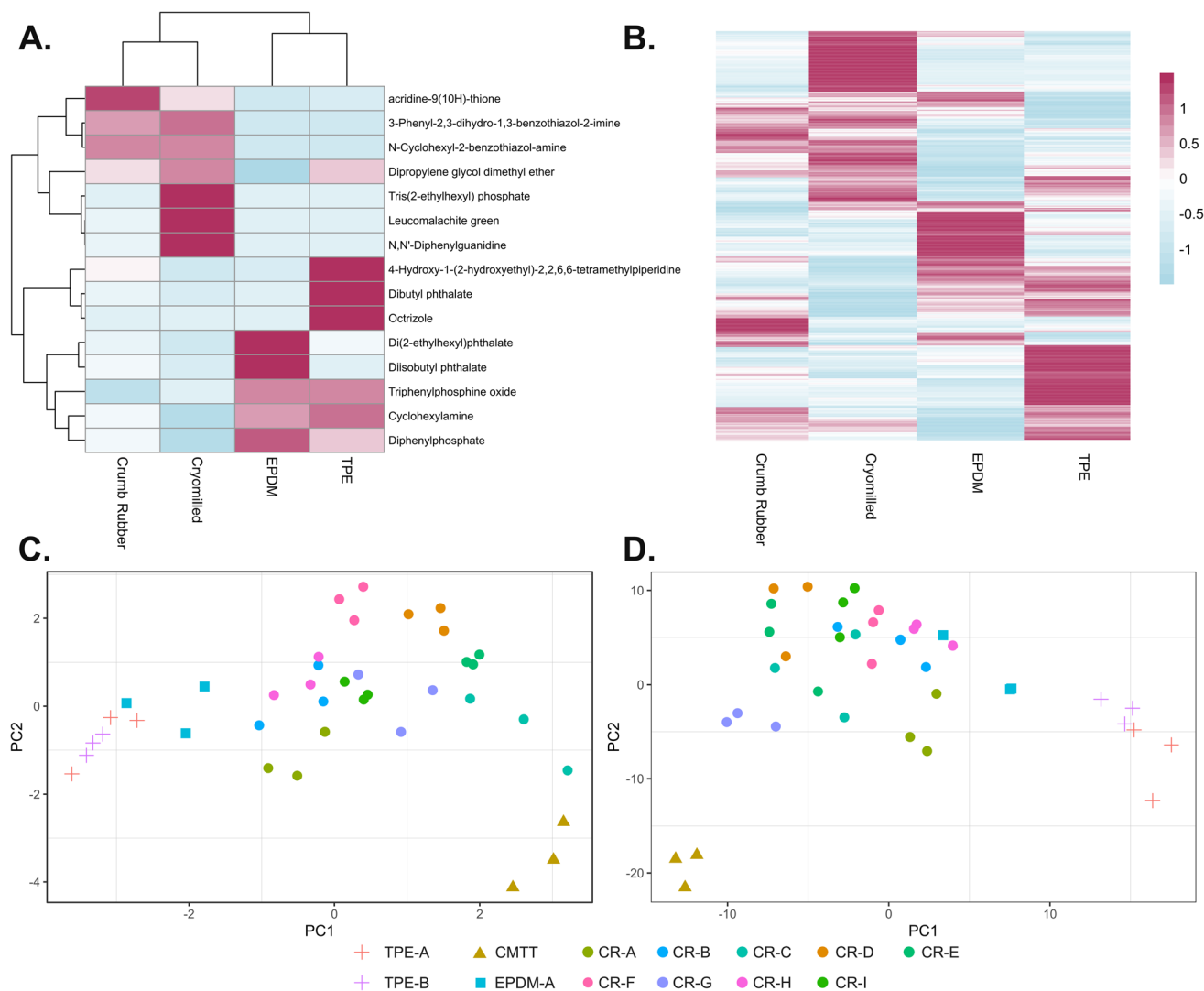
While the most abundant compounds varied by infill material type, we detected some compounds which were common to the leachates from several different materials (Fig. 2B). Several compounds typically associated with rubber vulcanization were present in leachates from multiple infill types. *N,N'*-diphenylguanidine was ubiquitous (CR = 27/27; CMTT = 3/3; TPE = 6/6; EPDM = 3/3). *N*-cyclohexyl-2-benzothiazol-amine (NCBA) (CR = 25/27; CMTT = 3/3) and acridine-9(10*H*)-thione (CR = 23/27; CMTT = 2/3) were frequently found in both tire rubber-derived materials. Cyclohexylamine was detected less frequently but found in crumb rubber and an alternative infill material (CR = 7/27; TPE = 1/6). It is possible that cyclohexylamine is associated with structural interior components or walls of tires rather than tread due to its presence in crumb rubber samples but not in CMTT.

Compounds used as plasticizers and UV stabilizers were also prevalent and common across material types, including several phthalates with known endocrine-disrupting behavior. Di(2-ethylhexyl) phthalate (DEHP) was detected in all samples (CR = 27/27; CMTT = 3/3; TPE = 6/6; EPDM = 3/3). Dibutyl phthalate (CR = 6/27; TPE = 3/6) and diisobutyl phthalate (CR = 3/27) were detected less frequently and may be associated

with interior tire components rather than tread due to their absence in CMTT. Tris(2-ethylhexyl) phosphate (CR = 7/27; CMTT = 3/3) and diphenyl phosphate (CR = 9/27; CMTT = 1/3; EPDM = 1/3), which are used as both plasticizers and flame retardants, were observed in leachates of multiple material types. Several UV stabilizers were identified, suggesting the incorporation of light-protective additives in both crumb rubber and thermoplastic elastomer infill. 4-Hydroxy-1-(2-hydroxyethyl)-2,2,6,6-tetramethylpiperidine was common across all infill types (CR = 22/27; CMTT = 2/3; TPE = 5/6; EPDM = 3/3). Octrizole was detected only in TPE and EPDM (TPE = 3/6; EPDM = 2/3) samples. The higher relative frequency of these compounds in alternative infill material may indicate their use in color-dyed polymers. Additional compounds include 3-phenyl-2,3-dihydro-1,3-benzothiazol-2-imine (CR = 23/27; CMTT = 3/3), a compound listed on the EPA CompTox HBM4EU database as a chemical of emerging concern,<sup>61</sup> and dipropylene glycol dimethyl ether which occurred sporadically (CR = 3/27).

Differences in chemical abundance between CMTT and crumb rubber indicate that CMTT leachate is not fully representative of runoff from artificial turf fields. CMTT and crumb rubber display some similarity in heatmaps (Fig. 2C and D). Principal component analyses (PCA) of Level 2 identified





**Fig. 2** (A) Heatmap and hierarchical clustering of median log-transformed feature intensities for the 15 infill-associated Schymanski confidence Level 2 features. Intensities are z-score-scaled by compound to highlight relative differences in abundance. Clustering is performed using Euclidean distance and complete linkage to group compounds and infills with similar normalized intensity profiles. The color scale spans from  $-1$  (blue; lower relative abundance) to  $+1$  (red; higher relative abundance), indicating deviations from each compound's mean signal across infills. Leaching was performed in triplicate and data are plotted for each replicate. (B) Heatmap of all detected features, using same color scale as in plot a. (C) Scores plot of principal component analysis of the 15 infill-associated Schymanski confidence Level 2 features. (D) Scores plot of principal component analysis of all detected features.

compounds (Fig. 2C) and of all detected features (Fig. 2D) indicate distinct clusters for CMTT that are substantially separated from the field-derived infill materials. While CMTT consists exclusively of the tire tread fraction, crumb rubber is ground from the full tire matrix including sidewall and inner materials which may contain a broader range of additives and fillers. As such, crumb rubber may more closely represent the chemical profile released from turf fields, while CMTT may more closely represent on-road emissions from tire wear while driving. However, this difference may be exacerbated by particle surface area and the degree of aging. While crumb rubber was made from post-consumer tires and collected from fields where it had been placed for years, the CMTT particles we used during leaching were substantially smaller, produced from new tire

tread, and stored in dark airtight conditions. Additionally, crumb rubber infill material collected may have accumulated chemicals from runoff, atmospheric deposition, or other environmental transportation pathways which contribute to the difference between CMTT and crumb rubber, relevant for the plot of all detected features (Fig. 2D).

When considering compounds identified at Level 3 confidence (Fig. S17), isovaleraldehyde, a rubber vulcanization agent, displayed the highest mean abundance in CMTT but was present across all infill types, suggesting widespread use during production and potential persistence under environmental conditions. Urea and 1*H*-1,2,4-triazole-3-sulfonamide showed relatively uniform mean peak areas across all four infill



materials, indicating broader use or their presence as background contaminants.

Fourteen of the fifteen Level 2 compounds were detected at least once in tire-derived material, of which only 50% (7/14) matched an entry in the US EPA Tire Crumb Rubber list (Table 1). Some compounds found at relatively high abundance in crumb rubber and CMTT (*e.g.*, NCBA) were not on the EPA list. This finding indicates that the present EPA crumb rubber list is not exhaustive and highlights the significant unknowns in tire chemical composition. More transparency in reporting tire manufacturing chemicals, and further studies to identify impurities and transformation products, would improve assessments of crumb rubber chemical occurrence and impacts in the environment.<sup>63</sup>

Leucomalachite green (LMG), an IARC Group 2B possible carcinogen,<sup>64</sup> was putatively identified with high frequency in tire rubber-derived material leachates (CR = 16/27; CMTT = 3/3). Leucomalachite green is a transformation product formed *via* the reduction of malachite green, a chemical which has historically been used in aquaculture as an antifungal and antibiotic agent, as well as a green dye. Leucomalachite green's detection in both field samples and CMTT suggests the potential use of malachite green as a dye in rubber manufacturing. Malachite green is known to harm aquatic species, with a 96 hours LC<sub>50</sub> of 99.8 µg L<sup>-1</sup> for rainbow trout (*Oncorhynchus mykiss*), and is persistent in fish flesh.<sup>65</sup> Malachite green was banned for aquaculture use in Canada in 1992;<sup>66</sup> despite bans, it is often found to be used illegally due to its efficacy in aquaculture.<sup>62</sup> LMG is not listed in the US EPA Tire Crumb Rubber database. To our knowledge, there has been one prior report of LMG in tire crumb rubber based on exact mass matching with predicted composition data.<sup>67</sup> Our MS/MS spectra with spectra matching to a reference library for LMG detection are available in Fig. S11.

### 3.3 Trace contaminant concentrations in stormwater samples from artificial turf field drainage systems

Our observed concentrations indicate substantial loadings of metals from turf field drainage systems. Copper, zinc, and lead were detected at high concentrations in turf field drainage systems across multiple locations and storm events (Fig. 3). Across the three events, maximum concentrations were 0.966 µg L<sup>-1</sup>, 30.3 µg L<sup>-1</sup> and 726 µg L<sup>-1</sup> for lead, copper, and zinc, respectively. These results are similar to values reported in previous work; a study in Connecticut<sup>35</sup> reported a maximum of 260 µg L<sup>-1</sup> and a mean of 84 µg L<sup>-1</sup> for zinc, and a maximum of 5 µg L<sup>-1</sup> and mean of 3.2 µg L<sup>-1</sup> for copper in stormwater collected from a turf field within 30 minutes of precipitation onset. We estimate a maximum of 19.64 g of zinc and 2.77 g of copper discharged into stormwater across a single storm events (Table S14). Considering that lead was not detected in our infill leaching experiments, we anticipate that it may have been released from subsurface infrastructure including metal fittings within the sampled pipe drainage systems.

During Storm Event 3, 6PPD-Q concentrations exceeded the reported LC<sub>50</sub> for juvenile coho salmon (41 ng L<sup>-1</sup>)<sup>24</sup> in 14/14 stormwater samples with concentrations above detection

limits, reaching a maximum of 130.23 ng L<sup>-1</sup> during a period of heavy rainfall. The local British Columbia Water Quality Guideline<sup>68</sup> for short-term acute 6PPD-Q exposure of 10 ng L<sup>-1</sup> was exceeded repeatedly throughout the rain event. Reported concentrations reflect conditions within the stormwater conveyance system; actual concentrations in salmon-bearing surface waters would be substantially lower due to dilution. Concentrations of 6PPD followed a similar temporal pattern to 6PPD-Q, peaking at 175.64 ng L<sup>-1</sup> with the second onset of rain. HMMM reached a maximum of 883.4 ng L<sup>-1</sup> rapidly following the onset of rain, then stabilized between 100 to 500 ng L<sup>-1</sup> during the remainder of the event. BTZ concentrations remained lower than the other trace organic contaminants (<80 ng L<sup>-1</sup>), except for an outlier of 418 ng L<sup>-1</sup>, possibly attributable to a grass blade piece within the collected stormwater; this data point was rejected from the dataset before analysis or visualization.

When comparing these results with our extract concentrations, there is a notable discrepancy with benzotriazole concentrations. We did not detect BTZ in extracts for field CR-E and anticipate that benzotriazole in stormwater was primarily released from UV-stabilizers used in the synthetic grass blades,<sup>69</sup> with co-contamination from brake pad residues on crumb rubber representing a smaller secondary source. In contrast, 6PPD and 6PPD-Q occurred at comparable levels in stormwater samples, consistent with our extraction experiments that showed similar concentrations of both of these compounds. It is possible that 6PPD was additionally released from other sources such as transfer from users' shoes, but we expect these contributions to be minor relative to direct release from infill. Overall, while the infill extractions characterize release from specific infill materials, stormwater sampling provides an understanding of contaminant mobilization from the entire turf field system.

When comparing these results with previous studies, our observed TrOC concentrations were close to observations in urban surface waters, but lower than concentrations observed in direct roadway runoff. A nationwide study from the U.S. Geological Survey reported 6PPD-Q concentrations between 2–290 ng L<sup>-1</sup> in urban surface waters,<sup>70</sup> while a Canadian study focusing on cold climate regions found a mean of 600 ng L<sup>-1</sup> of 6PPD-Q in urban runoff,<sup>7</sup> higher than the levels observed in our samples. For HMMM, one study reported that over 1000 ng L<sup>-1</sup> of was detected in urban surface water during cold-weather months<sup>71</sup> and a German study found up to 880 ng L<sup>-1</sup> in urban rivers.<sup>72</sup> BTZ has been detected in urban streams at 224–453 ng L<sup>-1</sup>,<sup>73</sup> although its ubiquitous use makes source attribution in urban environments difficult, particularly considering the widespread use of BTZ as a corrosion inhibitor in brakes.<sup>74</sup>

Overall, concentrations of contaminants in stormwater varied dynamically in response to precipitation. Copper concentrations peaked in all events shortly after the onset of precipitation. Zinc concentrations peaked shortly after copper and tended to remain elevated for longer, particularly during periods of heavy precipitation. Lead concentration profiles were less consistent, with the highest concentration observed soon after precipitation onset for Storm Events 1 and 3, but observed



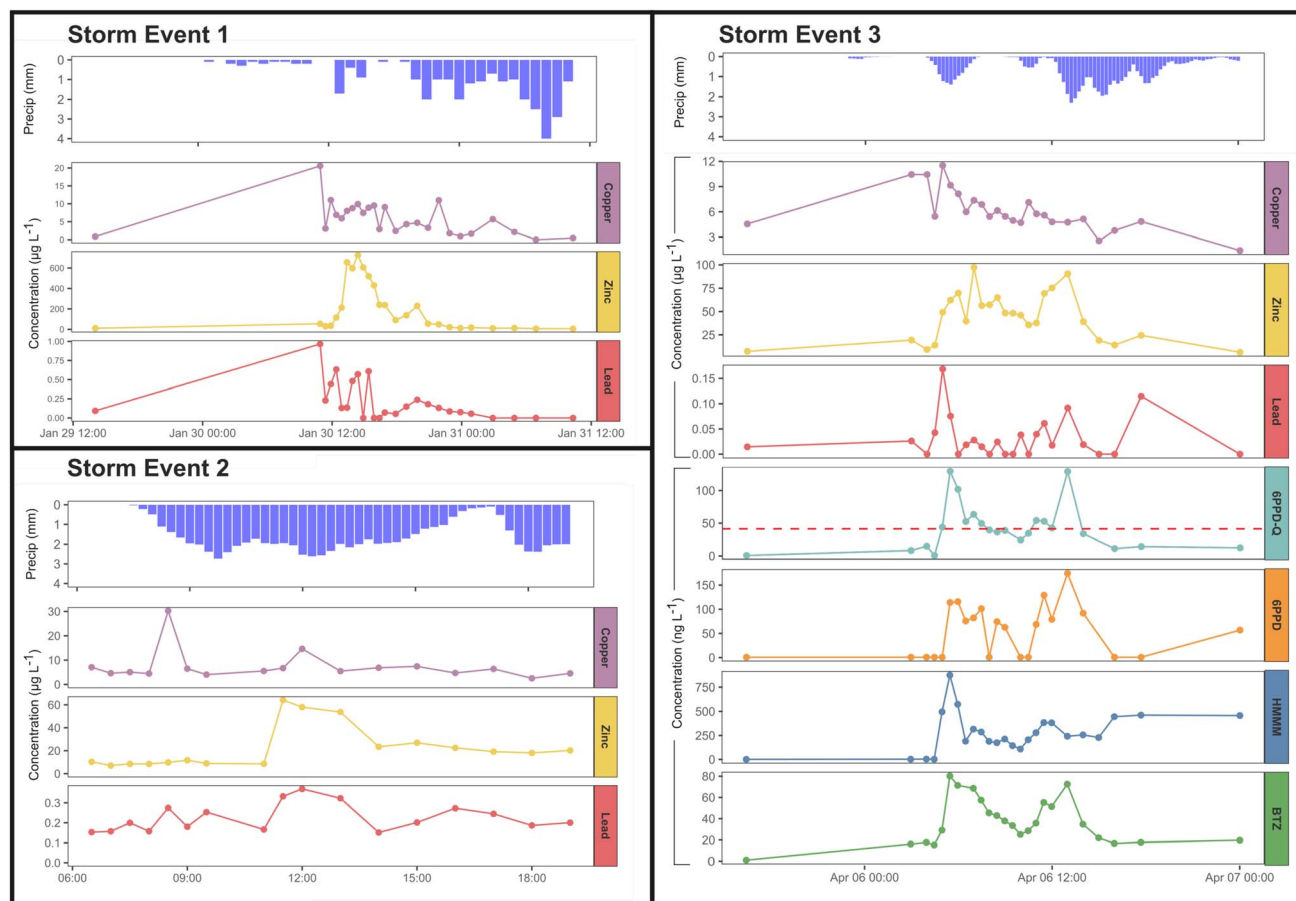


Fig. 3 Time-profile release of trace organic and metal contaminants across three Storm Events (notes: y-axis scales differ for each contaminant and event; non-detects are plotted as zero but assessed as half of the MDL for statistical reporting; for Storm Event 3 the 6PPD-Q  $LC_{50} = 41 \text{ ng L}^{-1}$  from Lo *et al.* is plotted as a red dashed line).

before onset in Storm Event 2, which could indicate dissolution from subsurface infrastructure or remobilization from accumulated sediment into stagnant water following the 2.5 days antecedent dry period.

To better understand how contaminant concentrations responded to precipitation intensity throughout rain events, we calculated cumulative release profiles for Storm Event 3, which had multiple peaks in precipitation that corresponded with peaks in contaminant concentrations (Fig. 4). Cumulative release profiles for metals in Storm Events 1 and 2 can be found in the SI (Fig. S18). Immediately following the initial precipitation onset during Storm Event 3, concentrations of all contaminants sharply increased (Fig. 4). During this initial precipitation period, 22–39% of total contaminant mass mobilized during the event was released, varying by contaminant. Subsequently, concentrations of most contaminants decreased until rainfall intensity peaked again  $\sim 7$  hours after the initial onset of rainfall. All of the target contaminants continued to be released throughout the event, with concentrations of some compounds tracking more closely with precipitation intensity. 6PPD-Q had the highest fraction released early in the event and it was mobilized quickly during subsequent rainfall. In contrast, a relatively small fraction of 6PPD was released during the first rainfall phase, and its release lagged most

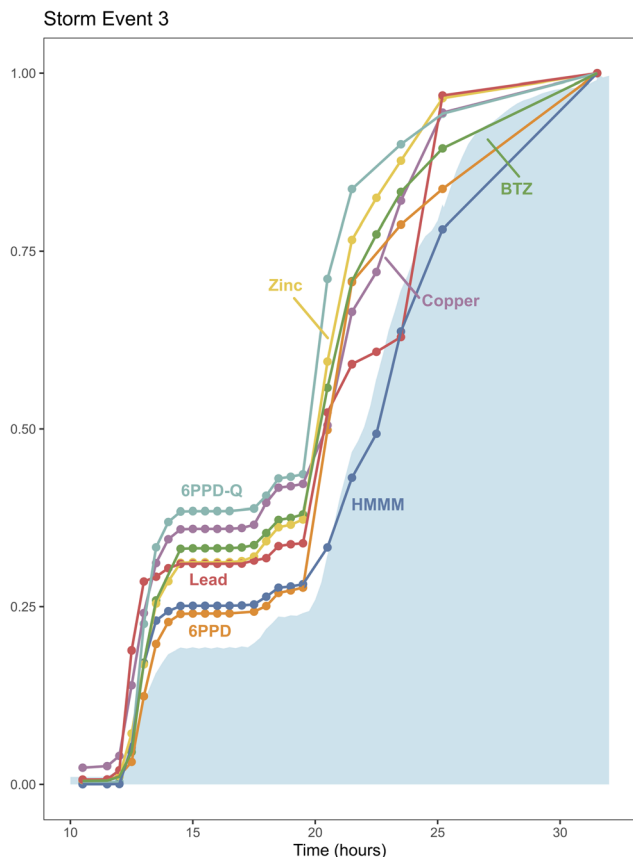
other contaminants after the second major onset of precipitation. BTZ, copper, and zinc exhibited intermediate behavior, with release occurring less rapidly in response to precipitation than 6PPD-Q but faster than 6PPD.

Physicochemical properties including  $\log K_{ow}$  may partially explain differences in mobilization dynamics for different contaminants. BTZ, a hydrophilic compound with a  $\log K_{ow}$  of 1.44, was released relatively quickly in response to rainfall and had a similar cumulative release profile to copper and zinc. In contrast, 6PPD ( $\log K_{ow} = 4.86$ ) and HMMA ( $\log K_{ow} = 1.61$ ) show comparatively lower release fractions in response to initial precipitation, perhaps due in part to their higher hydrophobicity relative to BTZ. However, 6PPD-Q ( $\log K_{ow} = 4.12$ )<sup>75</sup> was mobilized more quickly than BTZ or HMMA despite being more hydrophobic, perhaps because it is primarily present on the surfaces of infill material rather than being embedded within the infill polymer matrix.

## 4 Implications

This study demonstrates that artificial turf fields can act as long-term point sources of contaminants to stormwater and presents the first documented mobilization of 6PPD-Q from these





**Fig. 4** Cumulative release profiles for each contaminant across Storm Event Three (notes: x-axis begins at ten hours to improve plot visibility, contaminants are plotted as fraction of total contaminant release and rainfall is shown as fraction of total precipitation in light blue. Calculations and plotting were performed using half of MDL for compounds not detected above detection limit).

systems. 6PPD-Q concentrations in stormwater samples exceeded the  $LC_{50}$  for coho salmon by more than three times, which could result in toxicologically relevant concentrations in receiving waters, from a field with infill that had already been in place for 7 years. To understand whether 6PPD would be depleted in infill during the material's ~15 year lifetime, we estimated total emissions of 6PPD based on our stormwater sampling results. While exact makeup can vary, tires contain approximately 1% 6PPD by weight.<sup>76</sup> For a standard sports field with about 125 tonnes of crumb rubber infill, this equates to approximately 1250 kilograms of 6PPD. Assuming an average sized soccer pitch (7140 m<sup>2</sup>) and Vancouver's average annual precipitation (146 cm),<sup>49</sup> the estimated annual runoff volume is approximately 10.42 million liters. Based on the maximum 6PPD concentration we observed in stormwater (175.6 ng L<sup>-1</sup>), an upper limit of 1.83 g of 6PPD could be released from a field annually. Additional losses occur as 6PPD migrates to the surface and transforms into 6PPD-Q; assuming complete conversion of surface-bound 6PPD, this process could result in export of an additional 1.36 g annually. These values are likely overestimated as they utilize maximum instantaneous concentrations, but still indicate that leaching is a minor removal pathway and that 6PPD is unlikely to be exhausted in turf field

infill materials during their useful lifetimes (Table S14). This finding is substantiated by our leachate experiments, which indicated a lack of correlation between 6PPD and 6PPD-Q leachate concentrations with infill material age. Still, it is possible that leaching is not linear over time, and higher concentrations may be released shortly after installation.

Our non-targeted analysis revealed that infill materials contain a diverse suite of compounds, many of which do not exist in the USEPA Tire Crumb Rubber List database. Identified compounds included plasticizers, vulcanization accelerators, flame retardants, and UV stabilizers, and several of the detected chemicals are associated with aquatic or human toxicity. The putative identification of leucomalachite green is concerning given its persistence in fish tissue, and its presence in both crumb rubber and the cryomilled tire standard suggests its use in tire manufacturing. Alternative materials were observed to contain lower abundance of 6PPD-Q and metals and may reduce salmon exposure to 6PPD-Q; however, more research is needed to understand the impact of these materials in aquatic environments.

Artificial turf fields are contained sites with intentionally designed drainage systems that are often municipally owned, making them potential locations for targeted stormwater treatment. Recent work has shown that bioretention systems can reduce 6PPD-Q concentrations in stormwater by an order of magnitude *via* sorption to soil, substantially reducing lethality for vulnerable species.<sup>75,77</sup> Similar treatment approaches, based on sorption to organic surfaces, should be investigated as mitigation strategies for stormwater contaminants from turf fields. Alternative approaches to address contaminants from turf fields include limiting the use of crumb rubber as infill material or encouraging the use of safer chemicals in tires. In 2023, the EU banned intentionally added microplastics, mandating crumb rubber phase-out within eight years.<sup>78</sup> To support the development and implementation of effective legislation, further research is needed to better understand the impacts of artificial turf fields including contaminant leaching dynamics, the effects of environmental factors including weathering, and the environmental impacts of alternative materials that may replace crumb rubber infill.

## Author contributions

Katie J. Moloney: conceptualization, methodology, investigation, data curation, visualization, writing – original draft; Timothy F. M. Rodgers: conceptualization, supervision, writing – review & editing; Rachel C. Scholes: conceptualization, supervision, funding acquisition, writing – review & editing.

## Conflicts of interest

The authors declare that they have no competing financial interests.

## Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary



information: all contaminant concentrations, QA/QC data, and results of nontarget analysis are included in the uploaded SI PDF and Excel files. The exact locations of the sampled turf fields are withheld due to confidentiality concerns from partner municipalities, but the municipalities within which each field was located are disclosed in an SI table under the “location” heading. See DOI: <https://doi.org/10.1039/d5em01016k>.

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