

Occurrence of Parabens and Paraben Transformation Products in Sediment and Water Systems of the Brazos River Above and Below a Low Water Dam

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Research Article

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Abstract

Parabens are hydroxybenzoic acid esters, used as preservatives in personal care products, pharmaceuticals, and food. Parabens have been classified as endocrine-disrupting compounds due to their estrogenic activity. Parabens enter aquatic environments primarily through wastewater effluent and runoff, and continuous inputs allow their pseudo-persistence despite relatively rapid degradation. While the environmental occurrence and toxicity of parent parabens are well characterized, little is known about their transformation products. This study quantified parabens and transformation products in water (BDL-10.6 ng/L) and sediment (BDL-4.5 ng/g) upstream and downstream of a low water dam on the Brazos River, Texas, USA, and characterized their partitioning coefficients (K_d , K_{oc}). PHBA and DHBA were the most abundant analytes in both matrices. Measured partitioning coefficients differed from literature and modeled predictions, likely reflecting nonequilibrium conditions and interactions between polar functional groups and sediment organic matter, highlighting limitations of models predicting sorption behavior.

Introduction

Since the 1950s, parabens have been used as preservatives in personal care products, food, and pharmaceuticals (Chatterjee et al. 2024). Parabens are chemically defined as hydroxybenzoic acids with C-4 esterification (Neri et al. 2022). They exhibit broad spectrum antimicrobial activity, high chemical stability (Wei et al. 2021), and are odorless, colorless, and tasteless (Chatterjee et al. 2024). However, parabens have been identified as emerging chemicals of concern and classified as endocrine-disrupting compounds (EDCs), due to their similar structure to estrogen (Chatterjee et al. 2024) and potential to interfere with both endocrine and intracrine targets (Hager et al. 2022). Parabens have been documented in human urine, serum, placenta, breast tumor tissue, and adipose tissue (Liao et al. 2019). Although the United States considers parabens safe, strict regulation has emerged elsewhere. In 2014, the European Union limited paraben concentrations in various products and banned them entirely from products for children aged 3 and under (Maia et al. 2023). Measured amounts of parabens are increasing globally, with surface water concentrations reaching 171 $\mu\text{g/L}$ (Vale et al. 2022). Methyl paraben (MeP) and propyl paraben (PrP) are present in the highest concentrations in aquatic systems (Haman et al. 2015). These chemicals are introduced to the environment via disposal of pharmaceuticals, cosmetics, foods, and agricultural materials into aquatic environments (Pereira et al. 2023). When parabens transit wastewater treatment plants, paraben transformation occurs and the resulting transformation products are released into the environment along with diminished concentrations of parent parabens (Figure S1). Parabens have a high affinity for free chlorine in water and commonly produce halogenated (chlorinated) products through phenolic hydroxyl groups reacting with free chlorine involved in wastewater treatment (Haman et al. 2015) (Figure S1). While the health effects of parabens are relatively well studied, health effects of these transformation products are lesser known (Penrose and Cobb 2022). Parabens are not entirely removed via wastewater treatment, with discharge rates of 23% for parent compounds and 49% for transformation products, including halogenated parabens (Karthikraj et al. 2017), released through effluent streams and sludge (Maia et al. 2023). The ubiquitous presence of parabens and their

transformation products in aquatic environments is concerning, as half-lives for these compounds can approach 36,000 hours for chlorinated transformation products (Cl-MetPB, Cl₂-MetPB, Br₂-MetPB) in certain marine environments (Vale et al. 2022). Parabens with lower half-lives are pseudo persistent as they are constantly released into the environment, especially near wastewater treatment plants (Haman et al. 2015). Although paraben concentrations in drinking water are typically below 6 µg/L, the presence and persistence of parabens in aquatic systems may pose a risk for both human and environmental health (Pereira et al. 2023).

Previous studies found paraben and paraben transformation products in river water and biota downstream of wastewater treatment plants in the Brazos River, Waco, Texas, United States (Penrose 2023). However, the effects of paraben transport and transformation on partitioning to sediments have not been evaluated near these plants, where waters pass through features such as low water dams and shallow riffle areas. This study characterizes partitioning of parabens and their transformation products between sediment and water (K_d and K_{oc}) at several sites (Figure 1) upstream and downstream of a low water dam, located 8 river miles southeast of Lake Waco (USGS 2024) and is used to regulate water volume in Lake Brazos and flow into downstream reaches of the Brazos River.

Methods and Materials

Sample Site Determination and Collection: Two sampling sites were chosen based on accessibility and available transportation, one located above and one located below a low water dam on the Brazos River (Figure 1). These sites were chosen to augment previously sampled sites (Penrose and Cobb 2023b) surrounding a municipal wastewater treatment plant. Water samples above the dam were collected from kayaks, and all other samples were collected on foot. Sediment and water samples were collected on three separate days during the summer of 2023, with three samples taken per site on each sampling day. Samples were collected from distinct spots within an estimated 25-30 meter radius at each site to better represent the broader location and avoid stirred up sediment influenced by prior sample collection. Sediment samples were collected from the top 2-3 inches within 3 feet of the riverbank in 50 mL polypropylene centrifuge tubes. Water samples were collected in 500 mL amber glass bottles as grab samples. In preparation, bottles were washed with Citranox soap and water and then rinsed three times with GenPure ultrapure water and three times with methanol. Bottles were allowed to dry completely before use. In the field, bottles were rinsed with river water three times before collecting the sample.

Standards and Supplies: Analytical standards for methyl paraben (MeP, 97.5%) and propyl paraben (PrP, 99%) were obtained from AccuStandard. Analytical standards for ethyl 3-chloro-4-hydroxybenzoic acid (ClEtP), 3,5 dichloro-4-hydroxybenzoic acid (Cl₂PHBA), para-hydroxybenzoic acid (PHBA), 3,4-dihydroxybenzoic acid (DHBA), methyl 3,4 dihydroxybenzoic acid (MePOH), methyl 3,5-dichloro-4-hydroxybenzoic acid (Cl₂MeP), and ethyl 3,5-dichloro-4-hydroxybenzoic acid (Cl₂EtP) were acquired in 97% purity from Sigma Aldrich. All analytical standards were obtained as neat compounds and made into 1000 ppm solutions with methanol. Internal standard solutions for MeP-d₄, EtP-d₄, PrP-¹³C, BuP-d₉,

and PHBA-d₄ were obtained from Cambridge Isotopes, and prepared in methanol before storing in amber glass vials at -20°C.

Solid Phase Extraction Analytes were isolated from water using filtration and solid phase extraction as previously described (Penrose and Cobb 2023a). In summary, samples were filtered to remove solids, run through OASIS HLB cartridges, and parabens were eluted with methanol. Extracts were concentrated to a smaller volume for analysis. For sediment extraction, 1 g was weighed into a 50 mL polypropylene centrifuge tube. A 4.5 mL aliquot of water was added to each sample before acidifying to a pH of 3 with nitric acid. Each sample, blank, and spiked blank received 20 µL of the internal standard (5 ppm). The blank spike was received 100 µL of a solution containing each analyte of interest (100 ppb). Extraction mixtures were allowed to equilibrate for 30 min before adding 2 mL of acetone and vortexing with a Fisher Scientific Vortex Mixer. A 4 mL aliquot of 50:50 methanol water was added before shaking on an orbital shaker (100 rpm for 30 m). Samples were centrifuged (Beckman Coulter Avanti JXN-26) at 3000 rpm for 5 m. The supernatant was decanted into a new centrifuge tube, the pellet discarded, and 0.5 mL of 50:50 methanol water and 2 mL of ultrapure water was added to each extraction mixture. OASIS HLB Extraction cartridges were prepped with 5 mL of 50:50 methanol water and gravity filtered, using the vacuum to initiate flow. Extraction mixtures were eluted through cartridges under vacuum, and the rinse was discarded. Cartridges were dried for 5 min under vacuum, before eluting with 5 mL of methanol into 15 mL centrifuge tubes. Nitrogen evaporation (Thermo Scientific Reacti-Vap III #TS-18826) was used to concentrate extracts to 1 mL before diluting to 2 mL with methanol.

Instrumentation: Extracts from each sample were analyzed using an Acquity UPLC (Waters Corp., Milford, MA, USA) coupled to a Xevo TQS mass spectrometer as described earlier (Penrose and Cobb 2023b). That publication includes all relevant operating parameters. Briefly, the technique utilized an Acquity UPLC BEH C18 column with a gradient of 0.01% formic acid and acetonitrile as mobile phase and a flow rate of 300 µL/min. Limits of quantification (LOQs) ranged from 0.10-0.843 ng/L for water (Penrose and Cobb 2023b) and 1.0 to 2.0 ng/g dry weight for sediment (Penrose 2023).

Organic Matter and Wet Weight Determination: To determine the fraction of organic matter, 34 sediment samples were oven-dried for 48 h at 60°C. They were homogenized using a mortar and pestle. Rocks and other large organic matter were removed before weighing. Two 1 g aliquots were weighed for each sample before firing in aluminum foil weigh pans at 550°C for 4 h. Samples were weighed again to determine the ash mass. The difference between the soil mass and the ash mass was found as the ash free dry mass (AFDM). This protocol was derived from an existing standard method and modified slightly (Ameel et al. 1998). Wet weight was measured by drying preweighed sediments (1g) to constant weight in an oven at 90°C for at least 16 hours. Sediments were dried until weights on consecutive days did not change (± 0.0001).

Quality Control: A method blank and blank spike were analyzed alongside each set of samples for both water and sediment. Each sampling day's set consisted of all six samples collected that day (three upstream and three downstream), run together with one method blank and one blank spike per method.

The method blank was included, and analyte concentrations from the method blank were subtracted from all other samples in the set. The blank spike was used to assess recovery of each analyte. A blank and a standard were run every 10 samples for instrumental quality control. Isotopically labeled internal standards were added to every sample prior to extraction to determine recovery. Some analytes did not have internal standards, so the recovery from the most structurally similar parent compound was used. Calibration curves were constructed with 8 calibration standards from 0.5 ng/mL to 100 ng/mL for all analytes except PHBA and DHBA, which had concentrations ranging from 5 ng/mL to 1000 ng/mL.

Data Analysis: Upstream and downstream concentrations of each paraben and transformation product were compared to determine if there were differences in water and sediment above and below the dam. Concentrations were reported in ng/L for the water samples and ng/g for the sediment samples with confidence intervals to indicate variance. Shapiro–Wilk's tests and Bartlett's tests were used to determine if the values met the ANOVA assumptions of normal distribution and homogeneity of variances, respectively. Untransformed and log-transformed data remained non-normal, so non-parametric tests were used. Statistical comparisons were performed using Kruskal–Wallis and Dunn's tests. Average maximum likelihood estimations (MLEs) of concentrations were used in place of non-detects during statistical analysis. Analyses were performed using Microsoft Excel (2016) and R (Version 3.4.1). Maps were constructed in ARCGIS 10.8.1 and QGIS 3.14.16.

To calculate K_d , the concentration of each analyte in the sediment sample was divided by its corresponding concentration in the water sample. This was done for both upstream and downstream samples separately. The K_d value for each analyte was averaged and reported in L/g. K_d was determined using dry weight. K_d was only calculated for compounds that had a quantifiable concentration in water or sediment. EtP was excluded as it had non-detects for both water and sediment. To calculate K_{oc} , the K_d value for each analyte in every sample was divided by the fraction of organic carbon (f_{oc}). The f_{oc} values were calculated by dividing the AFDM value for each sample by the total soil mass before firing. K_{oc} values were log transformed and averaged for each analyte both upstream and downstream, before being reverse transformed.

Results and Discussion

Alkyl paraben concentrations in sediment ranged from below detection limits (BDL) to 3.7 ng/g upstream and from BDL to 4.5 ng/g downstream (Fig. 2), with no significant difference between sites for any paraben and transformation product studied. PHBA and DHBA had the highest concentrations of measured species, exceeding 1 ng/g at both sites. PHBA was the most abundant analyte overall, with average concentrations of 1.56 ng/g and 1.80 ng/g in downstream and upstream samples, respectively. Though this pattern was not mirrored by DHBA. MePOH had the lowest average concentrations at both sites.

Few studies have investigated paraben occurrence in sediments (Haman et al. 2015). The highest concentrations reported globally were 476 ng/g for MeP and 60 ng/g for EtP in the Turia River Basin,

Spain (Carmona et al. 2014), and 64.5 ng/g for PrP in Korea (Liao et al. 2013), but concentrations across various U.S. aquatic sediments were much lower with MeP, EtP, and PrP ranging from 0.312–45.5 ng/g, ND-0.459 ng/g, and ND-3.52 ng/g, respectively (Liao et al. 2013). Concentrations in this study aligned more closely with the U.S. data.

Paraben concentrations in water ranged from BDL to 10.6 ng/L upstream and from BDL to 4.7 ng/L downstream (Fig. 2). PrP was the only compound with a statistically significant difference ($p < 0.05$) between sites. Oxygenated and hydrolytic transformation products were detected above the limit of detection more frequently than parent parabens, reaching concentrations up to 19 ng/L upstream and 10.5 ng/L downstream. Dichloroparaben species were detected more frequently than monochloroparaben species, with ClMeP remaining below the detection limit in all samples, likely due to ring deactivation from the attachment of two chlorines to the phenyl group, rendering dichloro transformation products much less reactive.

Maximum MeP and PrP concentrations in this study's surface water were orders of magnitude smaller than values reported in China (154 ng/L), Argentina (1105 ng/L), and Poland (1600 ng/L). Our results more closely matched riverine water in Pittsburgh: MeP (2.2–17.3 ng/L) and PrP (ND-12 ng/L) (Renz et al 2013). MeP and PrP followed the same pattern of limited retention in sediment relative to surface water observed elsewhere (Ramos et al. 2026). PHBA and DHBA had the highest concentrations of all the measured analytes across both matrices, consistent with previous Brazos River findings and likely reflecting their role as natural degradation products of all parent parabens (Penrose and Cobb 2023b). Because relatively few studies report environmental concentrations of halogenated paraben transformation products, with the literature focusing predominantly on parent parabens, this study helps address an important gap in understanding their environmental occurrence.

Paraben partitioning between sediment and water

K_d values were smaller upstream than downstream for all species except EtP, indicating that higher sediment sorption downstream of the low water dam. K_{oc} values followed a similar pattern, with all but two parabens showing increased values downstream. MeP showed the largest upstream-downstream variation.

As these compounds are released into surface water and continuously contact suspended solids and sediment which typically contain a significant organic fraction, paraben deposition occurs. Paraben adsorption onto suspended solids or sediment is strongly dependent on the organic content of the solid matter and the hydrophobicity of the sorbing species (Haman et al. 2015). Estimated log K_{ocs} suggested MeP (1.96) and EtP (2.47) should not be expected to adsorb to suspended solids and sediment, unlike PrP (3.04) (Andersen and Larsen 2013). However, *in situ* concentrations of parabens and their chlorinated derivatives were found to be several times higher in the suspended solid phase than the dissolved phase (Terasaki et al. 2012), consistent with our K_d and K_{oc} values not following patterns predicted by controlled laboratory or *in silico* techniques (Table S2).

We attribute the differences between our data and predicted literature values to three factors. First, analyte concentrations in bed sediments and river water are unlikely to be at equilibrium. The waterway above the dam is approximately 130 m wide and 4 m deep, and this distance between surface water and sediment sampling locations likely prevented equilibrium between the two compartments. Flow dynamics are also a key factor controlling this. Above the dam, water flows in a controlled fashion and moves across the bed sediments with limited turbulence. Second, suspended solids likely act as the key intermediate influencing analyte partitioning between bed sediments and water. Immediately downstream of the dam, turbulent flow increases aeration and enhances paraben partitioning to both suspended solids and bed sediments, while also amplifying biotic and abiotic transformation rates of all analytes, further disrupting partitioning equilibria. Even though mean partitioning values were not statistically different, MeP, MePOH, Cl₂MeP, and Cl₂EtP all showed numerically higher absorptivity in downstream sediment, with MePOH showing the largest difference from upstream. Third, sorption may also be influenced by interactions between the polar functional groups of the acids and polyhydroxylated parabens and the non-organic moieties of the sediment or the polar functions of organic matter, which could explain why environmental K_d and K_{oc} values do not always follow patterns predicted by controlled laboratory or *in silico* techniques which often use the n-octanol water partitioning coefficient, K_{ow}, a measure of the compound's hydrophobicity. Together, these results suggest that *in situ* distribution of parabens and their transformation products between sediment and water is influenced by dynamic environmental processes not captured by modeled estimates, highlighting the importance of considering site-specific conditions when evaluating chemical transport, transformation, and uptake, as such assessments increasingly rely on modeled data to describe contaminant fate.

Declarations

No large language models were used in the creation of this manuscript, outside of LLMs embedded in search engines used during literature review.

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Competing Interests: The authors have no conflicts of interest to disclose.

Author Contributions: George Cobb and Michael Penrose contributed to study conception and design. Michael Penrose and Amanda Ellis developed the protocol, with input from George Cobb. Sample collection and processing, data analysis, and construction of figures and tables were done by Michael Penrose, Amanda Ellis, and Caroline Graham, with input and advice from George Cobb. Amanda Ellis and Caroline Graham wrote the manuscript with input from George Cobb. All authors read and approved the final manuscript.

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Figures

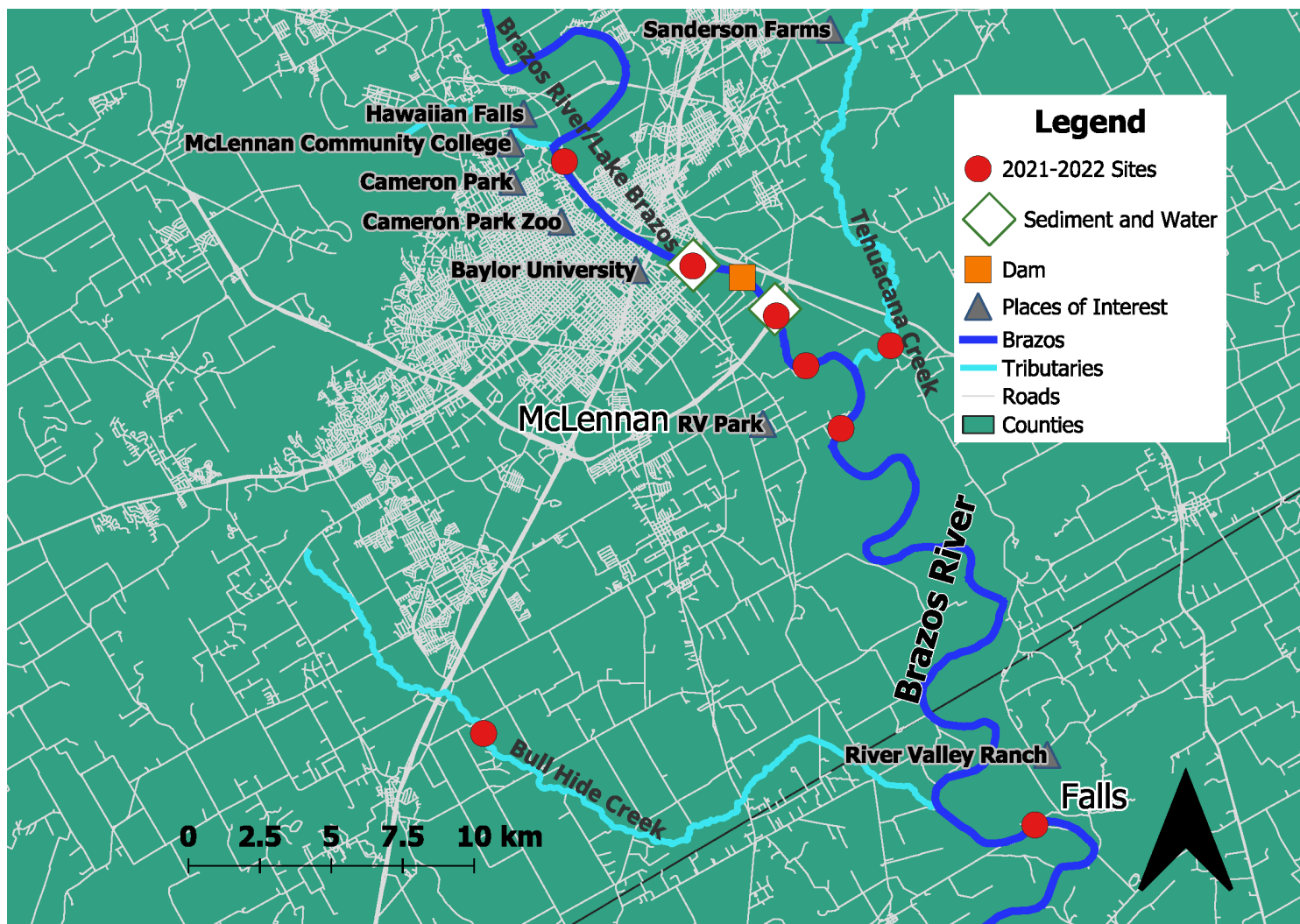


Figure 1

Water and sediment sampling sites along the Brazos River. White diamonds indicate the two sites sampled in this study (above and below the low water dam); additional sampling sites were from Penrose and Cobb 2023b.

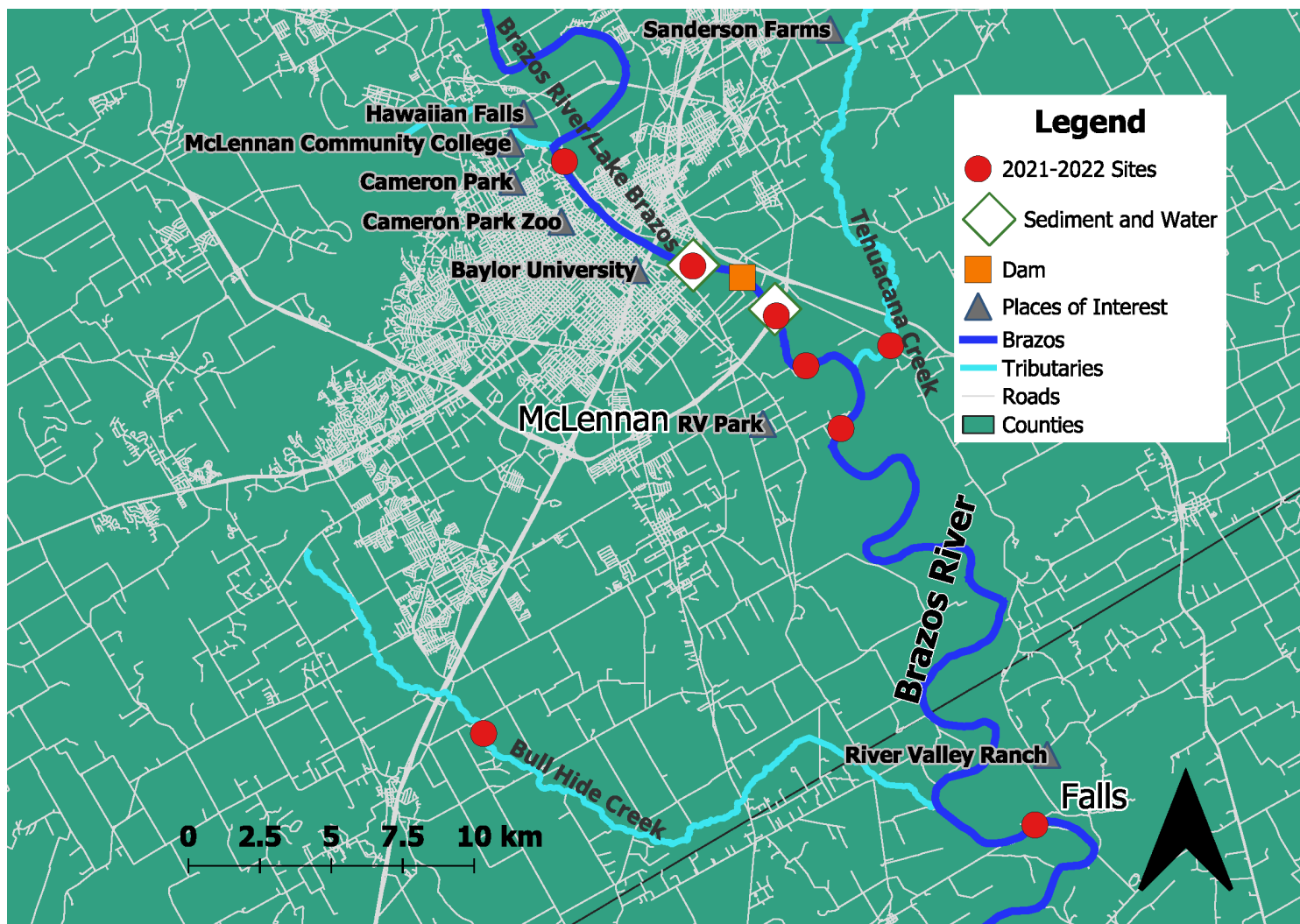


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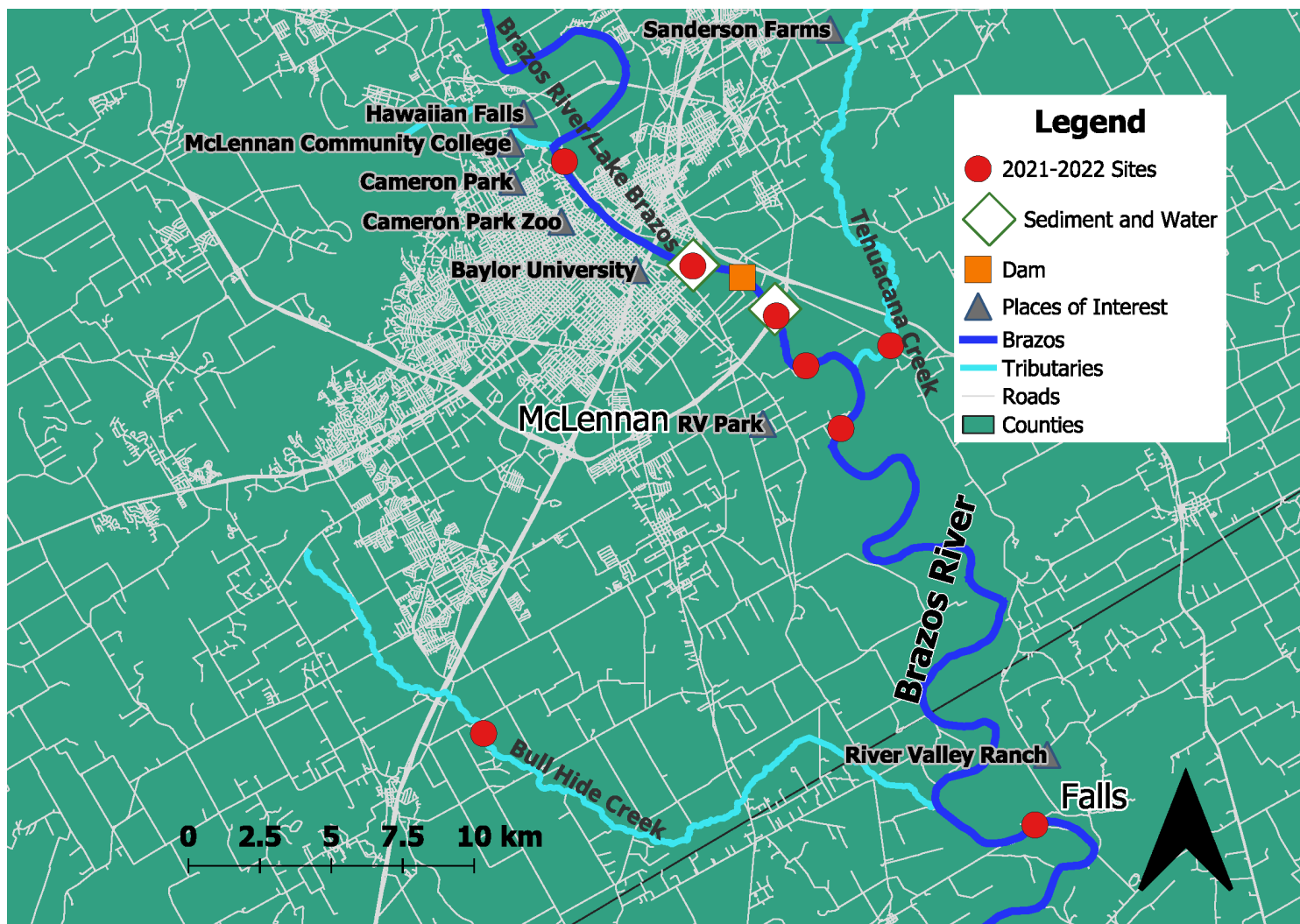


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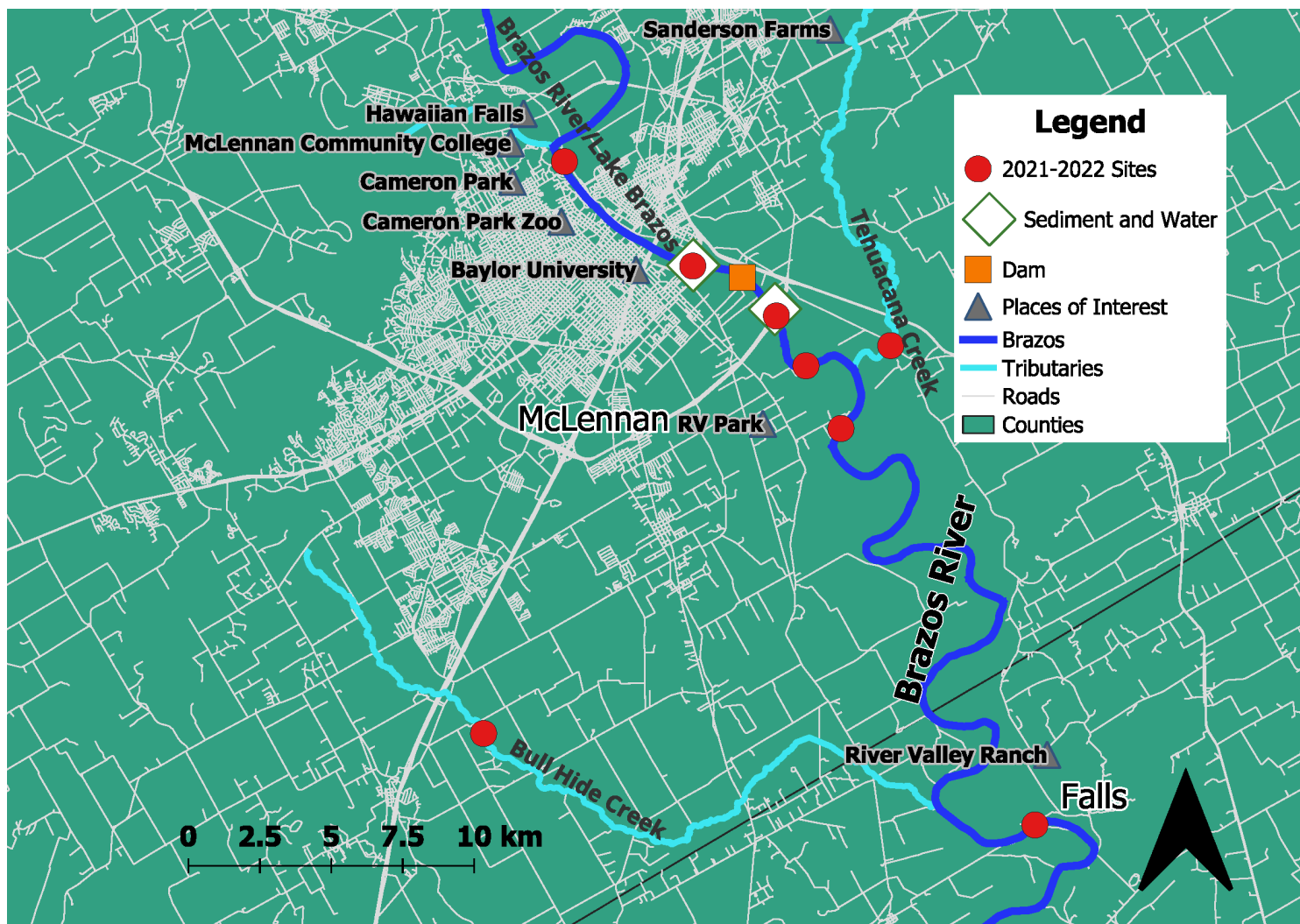


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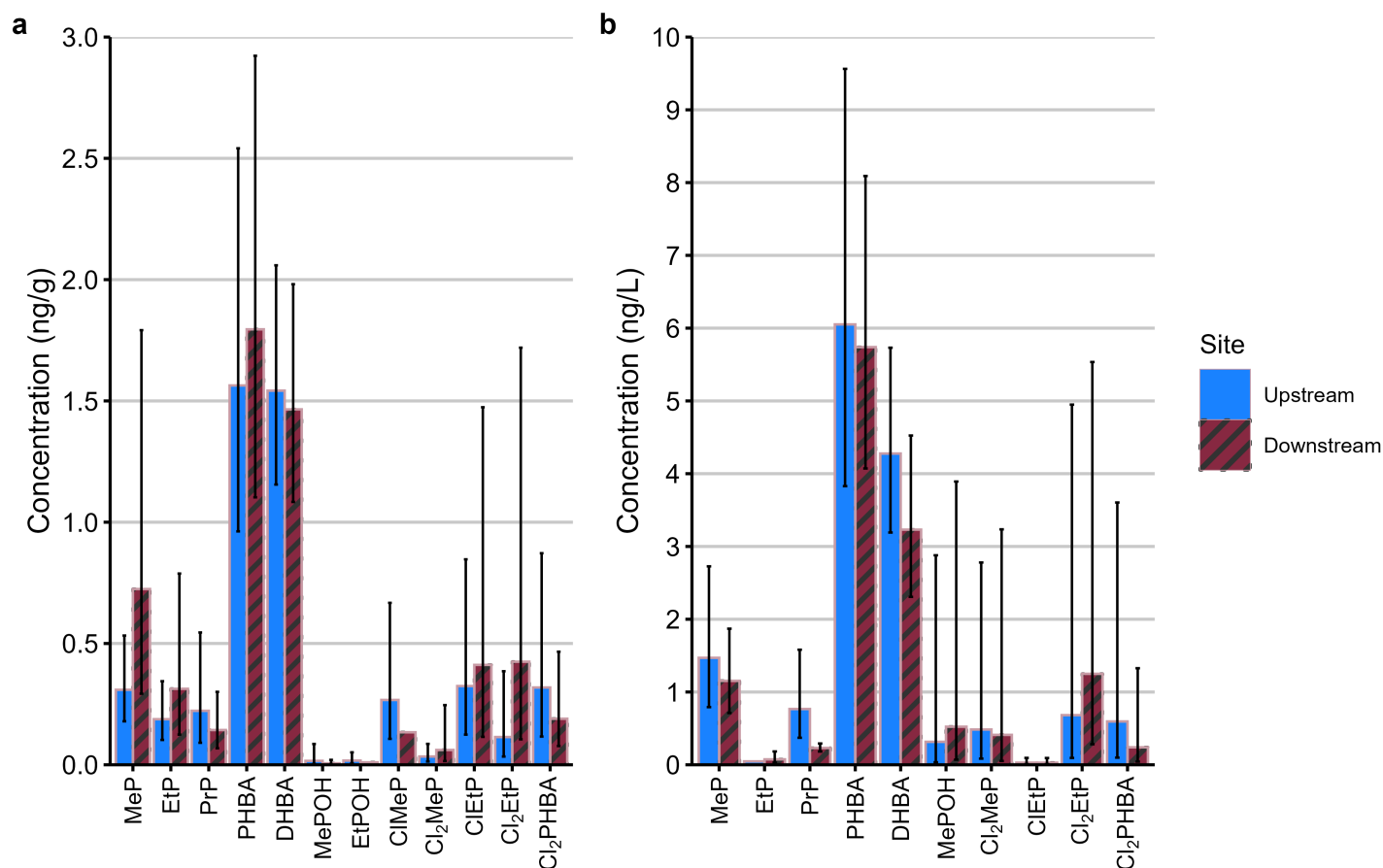


Figure 2

a Paraben and paraben transformation product concentrations in sediment upstream and downstream of a low water dam. A missing error bar indicates all values for that compound were below the detection limit. **b** Paraben and paraben transformation product concentrations in water upstream and downstream of a low water dam. A missing error bar indicates all values for that compound were below the detection limit; EtPOH and ClMeP were excluded from the water panel because all measured values were below the detection limit. Asterisks denote compounds with statistically different concentrations between upstream and downstream sites.

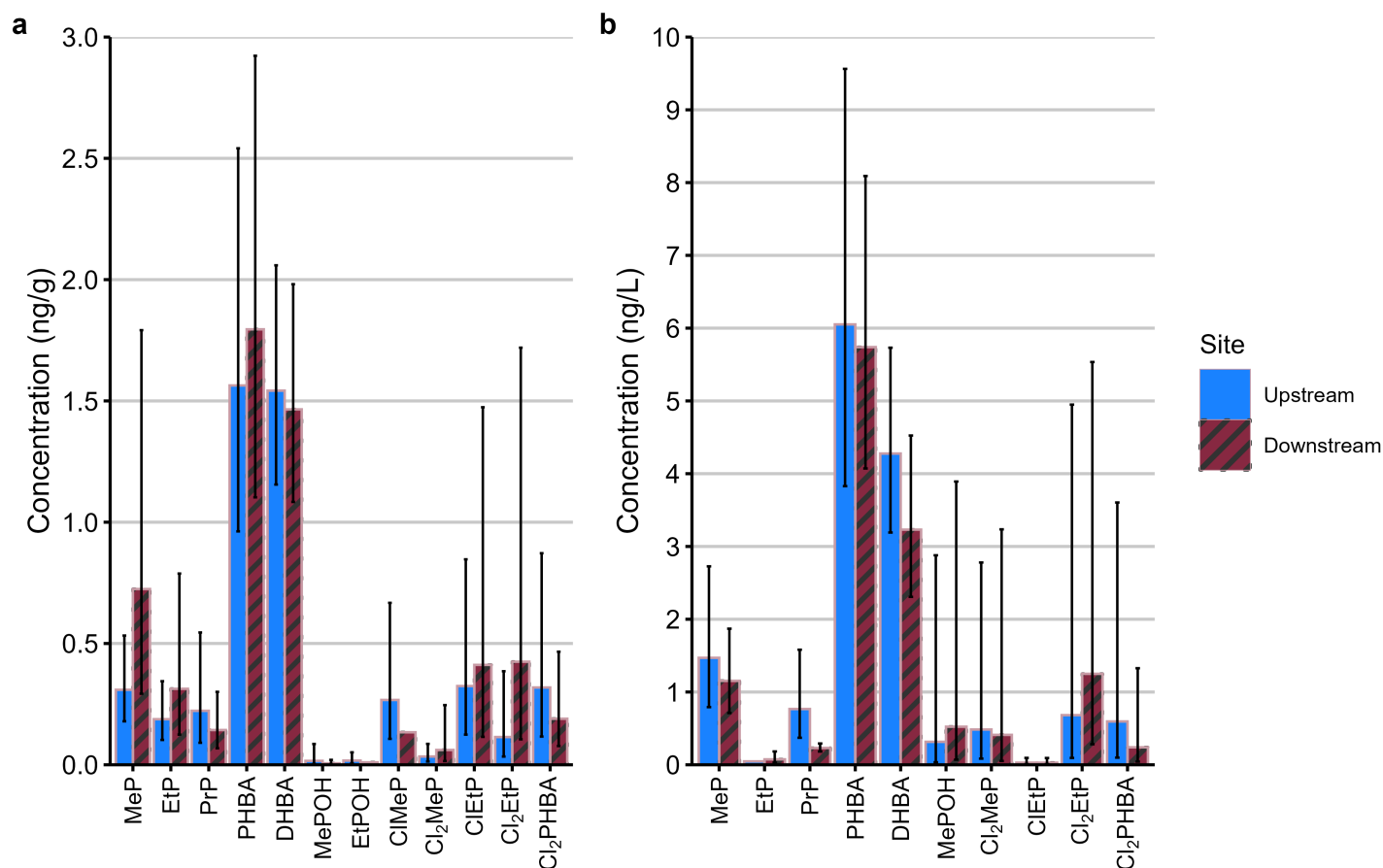


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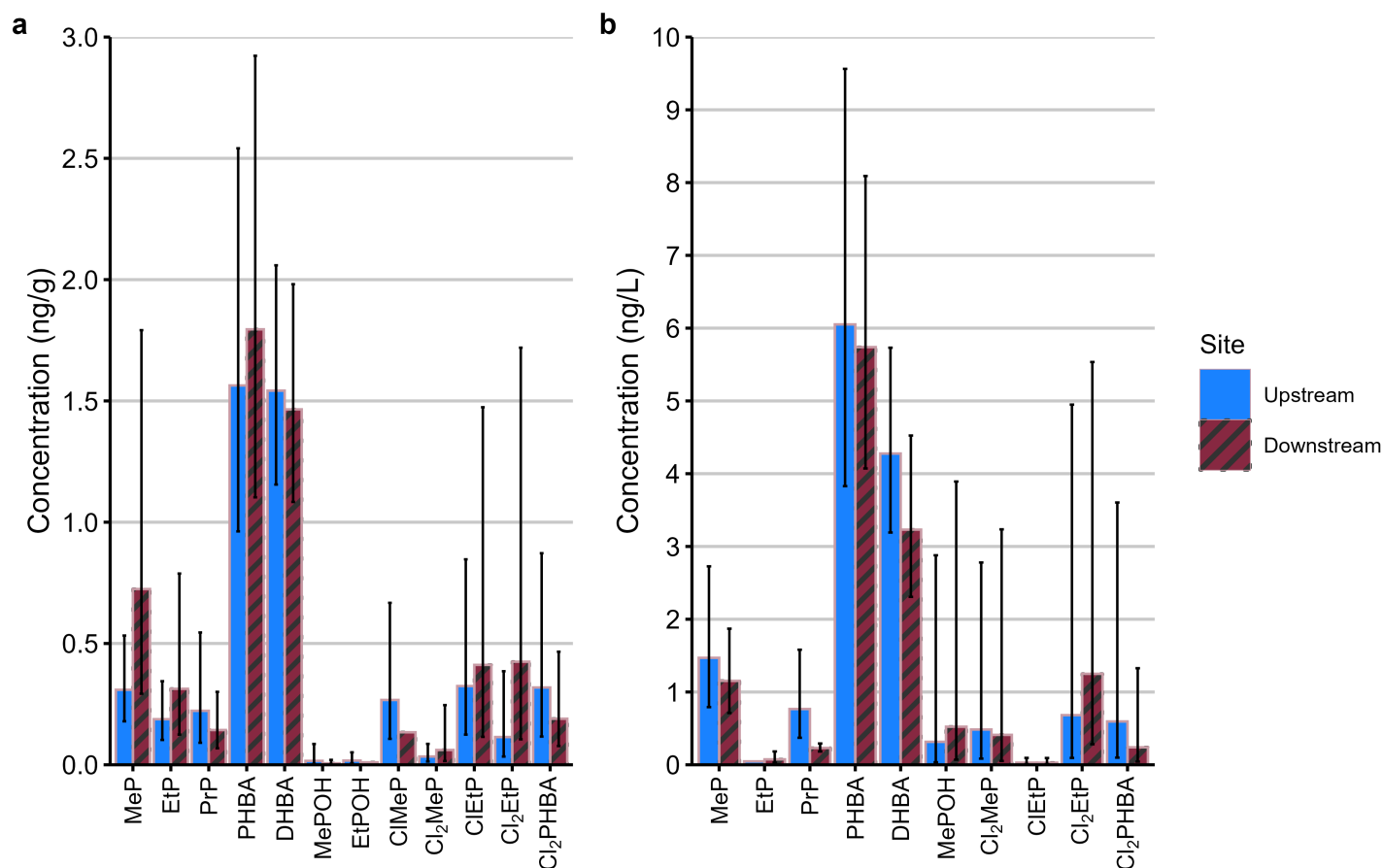


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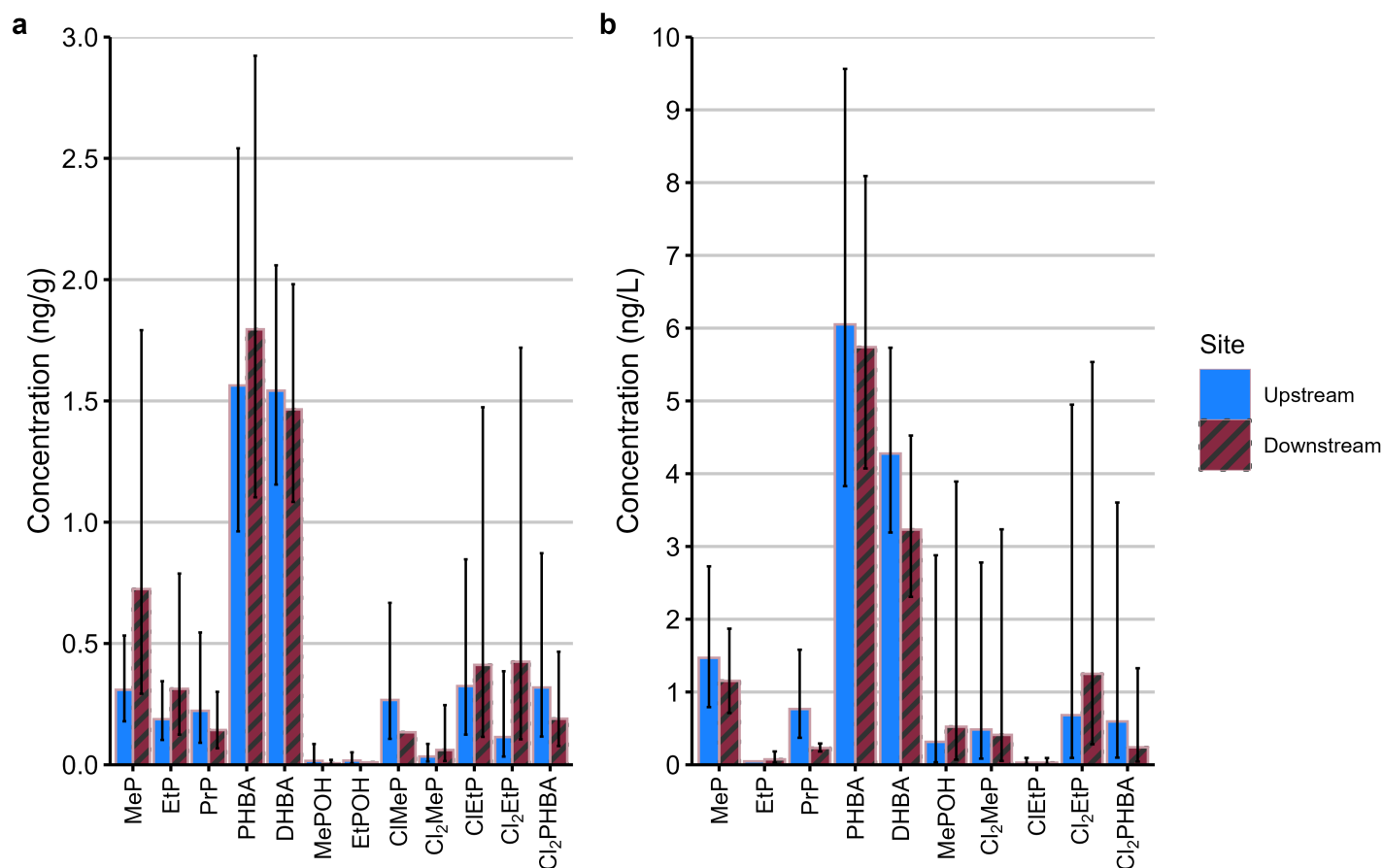


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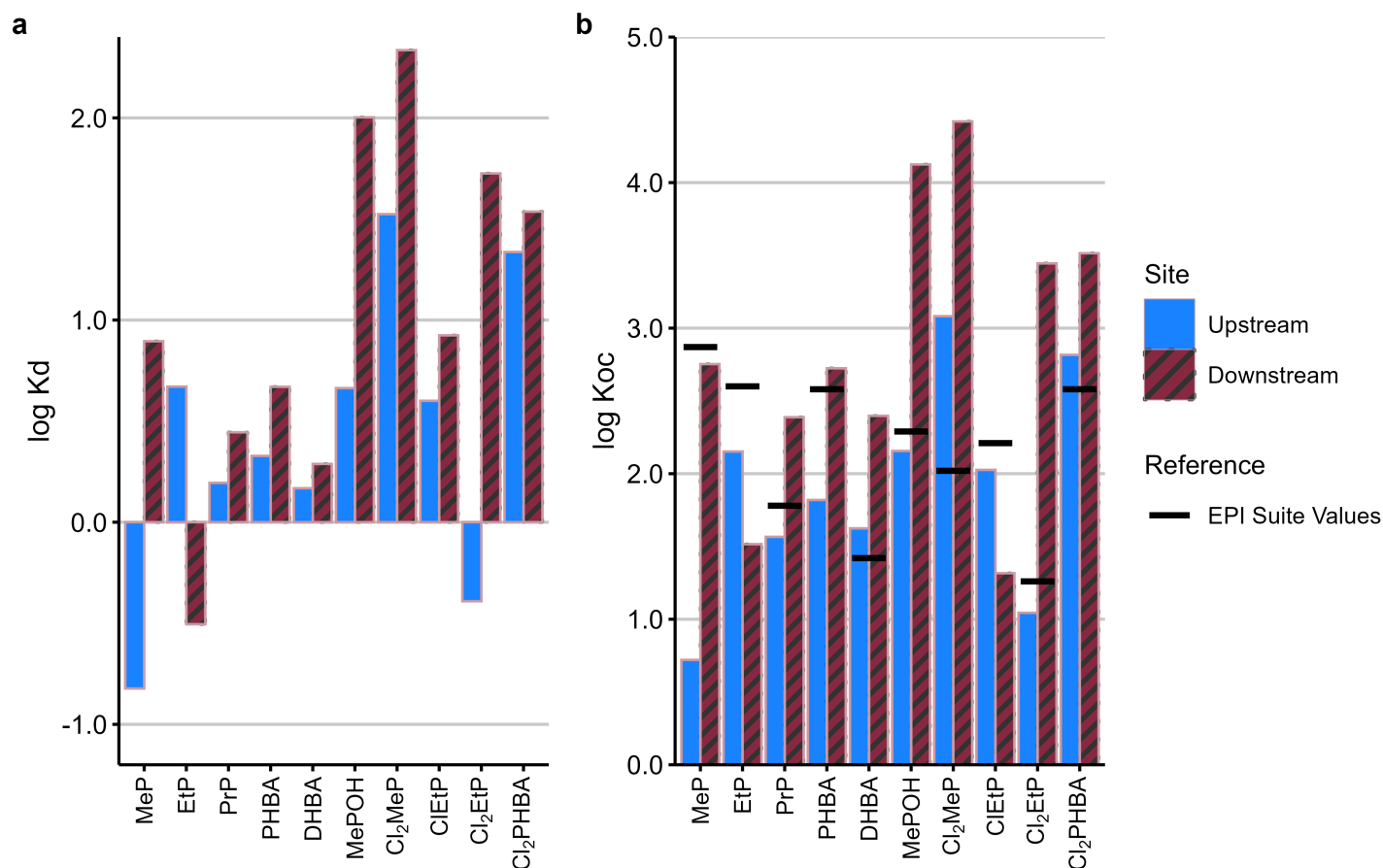


Figure 3

a Log Kd values for parabens and transformation products upstream and downstream of a low water dam. Blue bars represent upstream measurements, while striped red bars represent downstream measurements. Average log Kd values are provided in Table S1. **b** Log Koc values for parabens and transformation products upstream and downstream of a low water dam. Black segments indicate the estimated average log Koc values for each compound from EPI Suite. Average log Koc values from the present study and EPI Suite are provided in Table S2.

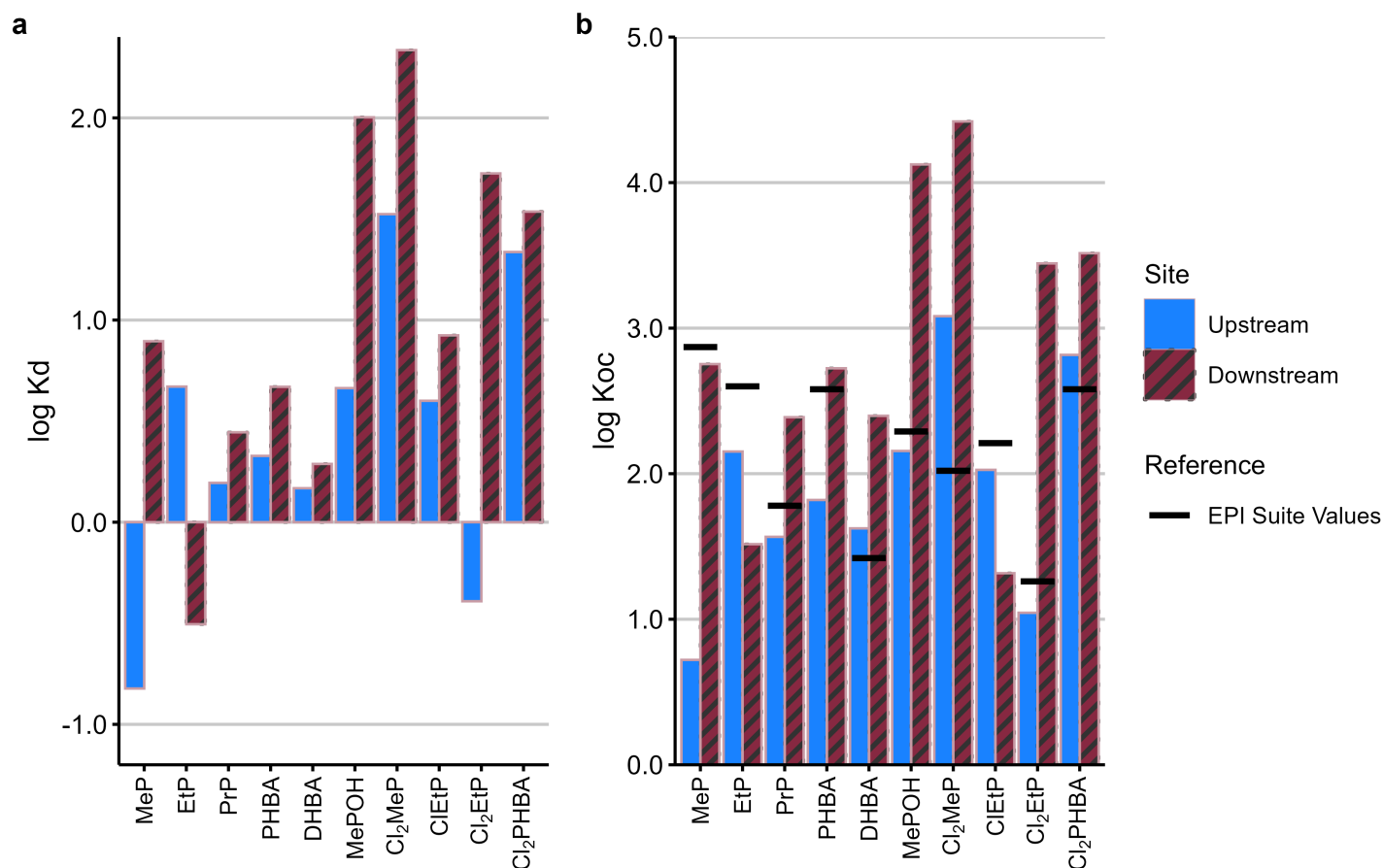


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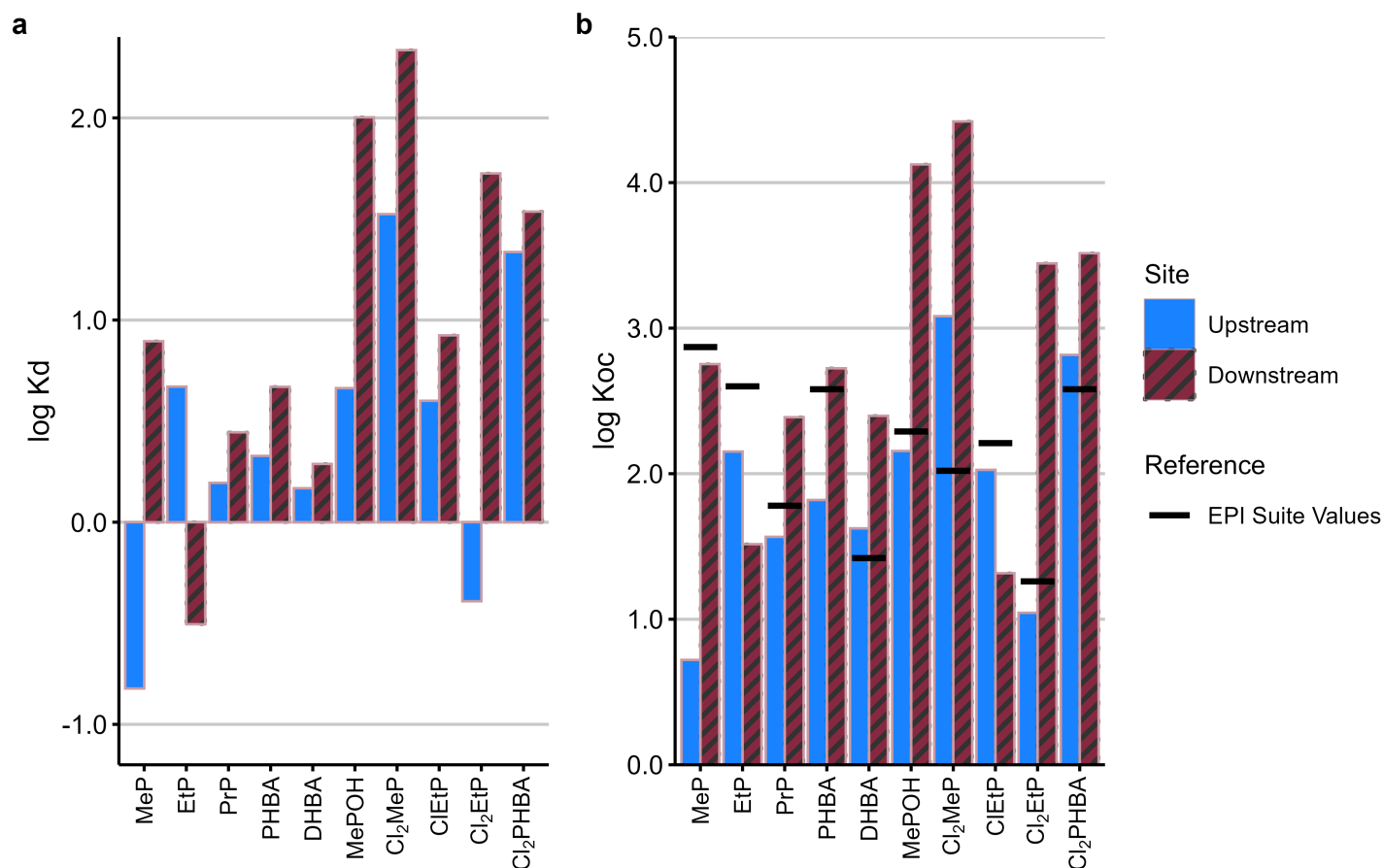


Figure 3

a Log K_d values for parabens and transformation products upstream and downstream of a low water dam. Blue bars represent upstream measurements, while striped red bars represent downstream measurements. Average log K_d values are provided in Table S1. **b** Log K_{oc} values for parabens and transformation products upstream and downstream of a low water dam. Black segments indicate the estimated average log K_{oc} values for each compound from EPI Suite. Average log K_{oc} values from the present study and EPI Suite are provided in Table S2.

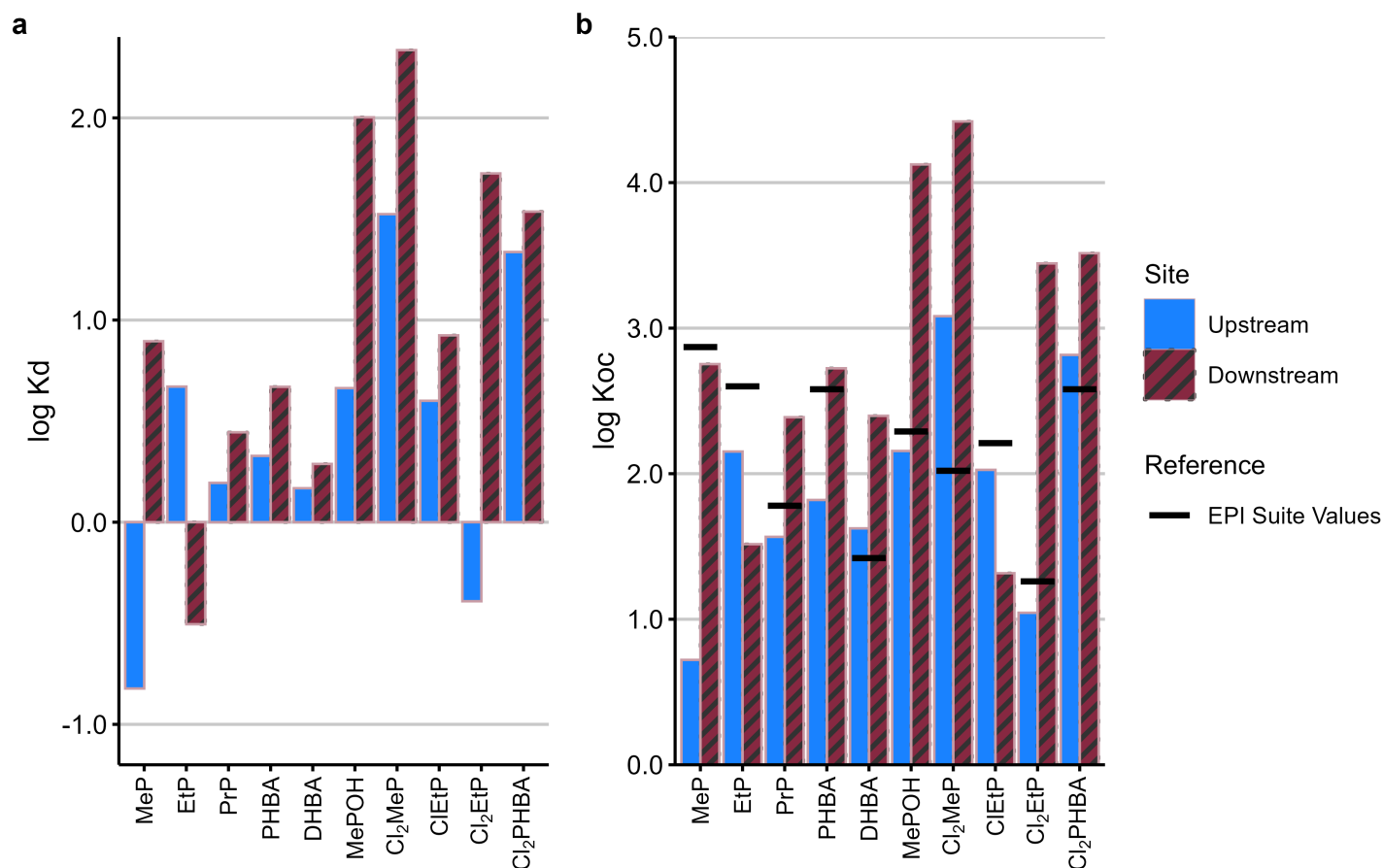


Figure 3

a Log Kd values for parabens and transformation products upstream and downstream of a low water dam. Blue bars represent upstream measurements, while striped red bars represent downstream measurements. Average log Kd values are provided in Table S1. **b** Log Koc values for parabens and transformation products upstream and downstream of a low water dam. Black segments indicate the estimated average log Koc values for each compound from EPI Suite. Average log Koc values from the present study and EPI Suite are provided in Table S2.

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