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Screening Programme 2022

New Environmental Pollutants


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REPORT

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Summary

The 2022 Screening Programme followed up on some findings from the 2020 Screening Programme. This included further investigation of plastic-related substances, tyre-related substances, and disinfection agents. The 2022 Programme also included additional new PFAS, bisphenols, and new siloxanes.

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Screening Programme 2022
New Environmental Pollutants

Preface

On behalf of the Norwegian Environment Agency (NEA), the Norwegian Institute for Water Research (NIVA) and NILU have carried out the 2022 Screening Programme.

Coordinator at NEA was Bård Nordbø, the project manager at NIVA was Cathrine Brecke Gundersen with Pawel Rostkowski coordinating from NILU.

Sampling was carried out by Heidi Eikenes, Are Bäcklund, Sam-Erik Walker (NILU), Christian Vogelsang, Elisabeth Rødland, and Bjørnar Beylich (NIVA). Coordination of sampling equipment and chemical data was carried out by Kine Bæk (NIVA). Sondre Meland and Nina Engen contributed with scientific and administrative support, respectively. We would like to thank the Environmental Contaminants in an Urban Fjord monitoring programme and Eivind Stensrud at the University of Oslo for providing samples of marine biota, and Bjørn Aas at the Centre for sports facilities and technologies at the Norwegian University of Science and Technology (NTNU) for his contribution to site selection of artificial grass pitches.

Chemical analyses were performed by Thomas Rundberget and Laura Röhler (NIVA), Anders Røsrud Borgen, Vladimir Nikiforov, Alexander Håland, and Norbert Schmidbauer (NILU).
Data analyses and reporting were executed by Cathrine Brecke Gundersen, Malcolm Reid, Dorne Misaghian (NIVA), and Pawel Rostkowski (NILU). Quality assurance was performed by Merete Grung (NIVA). Reporting to Vannmiljø and the NORMAN Database was performed by Cleo Lisa Davie-Martin (NILU).

We thank everyone involved for a nice collaboration.

Oslo, 15.11.2023

Cathrine Brecke Gundersen

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Summary

The 2022 Screening Programme aimed at following up some of the significant findings from the 2020 Screening programme. This included further investigation into plastic-related substances, tyre-related substances, and disinfection agents. The project also included additional new PFAS, bisphenols and new siloxanes.

The objectives were to:

- investigate whether the above-mentioned substances were found at selected hot spot locations
- investigate whether the above-mentioned substances were introduced to and found in nature
- assess whether the levels found may pose a risk to the environment

Functional use of the substances and their physiochemical properties were critical factors guiding the design of the sampling campaign. Most of the substances are included as one of several components in various products such as car-tyre rubber, plastics, dyes, and textiles. Priority was therefore given to sampling locations associated with car tyre material such as artificial grass pitches and roads with heavy traffic. Sample types included stormwater, particles in air and water, the environment at tunnel treatment ponds, and biota (to investigate possible biological uptake). This resulted in 154 different environmental samples which were analysed for 59 different substances. Not all substances were analysed in all samples, but the total number of analytical results exceeded 1 300. Highlights of the results are herein presented, while the complete set of results is available in the Vannmiljø database (https://vannmiljo.miljodirektoratet.no/).

The substances were grouped depending on major area of use and/or physiochemical properties (bisphenols; disinfectants and solvents; long-chain chlorinated paraffins; pesticides and pharmaceuticals; rubber additives; siloxanes; surfactants including PFAS; and UV-additives). The presence of the substances in the various samples was evaluated by detection frequency, average concentration, and by comparing the measured concentration to Predicted No-Effect Concentrations (PNECs) when available. These are ecotoxicological threshold values derived by extrapolating from experimental data and, in some cases, from QSAR based prediction models.

The group of synthetic rubber-related substances included 25 compounds. These were analysed in all samples except marine biota. High concentrations, exceeding respective PNECs, were observed in all samples associated with vehicle tyres. This included all environmental compartments near busy roads, at a site for collection and recycling of used tyres, and artificial football fields (both indoor and outdoor). The highest concentrations were found for CAS 941-57-1 (Benzothiazole-2-sulfonic acid, BTSA), CAS 934-34-9 (2-hydroxybenzothiazole, OHBT), CAS 793-24-8 (6PPD) and its ozone bi-product 6PPD-Quinone, CAS 74-31-7 (DPPD), CAS 101-72-4 (IPPD), CAS 26780-96-1 (Antioxidant TMQ), CAS 3089-11-0 (Hexamethoxy methyl melamine, TMMAT) and CAS 102-06-7 (1,3-Diphenylguanidine, DPG). Most of these substances are added polymer/resin during manufacture of rubber tyre, at ratios of parts per hundred (%). The substances are integrated into the polymer particles so even very small amounts of solid, even as fine dust, can therefore show very high levels of these substances. Several of the relevant car tyre substances were found in the literature to be classified as leachables, and thus may also be present freely dissolved in the water phase.

None of the PFAS substances included were detected in this study. Only the surfactant CAS 2687-94-7 (Octyl-2-pyrrolidone, OP) was detected. It was found in rubber granule from football fields, and in

sludge from both tunnel environments and a tyre-recycling centre. Dust samples collected near busy roads were also found to contain OP. It is registered for use as an automotive degreaser which would corroborate the findings.

Two UV additives were included, but only CAS 1137-42-4 (4-Hydroxybenzophenone, 4-HBP) was detected frequently, but not at levels exceeding its PNEC values.

Of the five siloxanes measured (L6, L7, L8, L9, and L10), only the following three had available standard material for them to be quantified. L6, L7 and L8 were determined in samples of freshwater, sediment, and biota with concentration exceeding PNECs. L9 and L10 were detected using suspect screening, but not quantified. All the studied siloxanes were detected at high levels in air and dust samples. At the wastewater treatment plant, high degree purification was more efficient at removing siloxanes from the water phase than the low degree purification. Most of the siloxanes appeared to have ended up in the sludge.

Of the six substances included in the disinfectants and solvents group, only CAS 75-09-2 (Dichloromethane, DCM) and CAS 67-66-3 (Chloroform, TCM) were detected at significant levels. These substances were found in water samples from the urban river Alna catchment and in run-off from a vehicle tyre recycling centre. Compared to the findings from the 2020 Screening Programme, the levels in River Alna were much lower now.

The group of pesticides and pharmaceuticals included six substances. These were analysed in samples of treated wastewater and sludge from the wastewater treatment plant. Only amitriptyline (CAS 549-18-8) was detected in all wastewater effluent at concentrations exceeding those shown to have an impact on aquatic organisms (PNEC). Higher concentrations of this substance were observed in high treatment-grade effluent compared to those from the low treatment-grade. A possible explanation to this finding is that enzyme activity from microorganisms in the treatment deconjugate metabolites from human excretion of the pharmaceuticals back to the original pharmaceutical.

Long-Chain Chlorinated Paraffins (LCCP) were detected in water, sediment and biota at levels below the PNEC.

The bisphenol group included five substances. These were very rarely detected in any samples, and even then, at concentrations approaching detection limits.

Sammendrag

Tittel: Screening av nye miljøgifter
År: 2023
Forfatter(e): Cathrine Brecke Gundersen, Malcolm Reid, Pawel Rostkowski, Kine Bæk, Thomas Rundberget, Elisabeth Rødland, Bjørnar Beylich, Heidi Eikenes, Are Bäcklund, Sam-Erik Walker, Anders Røsrud Borgen, Vladimir Nikiforov, Alexander Håland og Norbert Schmidbauer.

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Miljødirektoratets screeningprogram 2022 hadde som mål å følge opp noen av hovedfunnene fra screeningprogrammet 2020. Dette inkluderte en nærmere undersøkelse av plastrelaterte stoffer, dekk-relaterte stoffer og desinfeksjonsmidler. I tillegg ble noen nye PFAS, bisfenoler og siloksaner inkludert.

Målsettingene var å:

- Undersøke om stoffene nevnt over ble gjenfunnet ved ulike «hot spot»-lokasjoner
- Undersøke om stoffene nevnt over introduseres til - og kan gjenfinnes - i naturen
- Vurdere om nivåene som observeres kan forårsake miljøskade

Prøvetakingsplanen ble designet basert på stoffenes kjente bruksområder samt fysiokjemiske egenskaper. De fleste av stoffene brukes som tilsetningsstoffer i ulike typer produkter som bildekk gummi, ulike typer plast, fargestoffer og tekstiler. Prøvetaking ble prioritert ved steder som assosieres med bildekk materiale, slik som kunstgressbaner og høytrafikkert vei. Ulike prøvetyper inkluderte overvann, partikler i luft og vann, miljø i rensebasseng og biota. Det resulterte i 154 ulike miljøprøver som ble analysert for 59 ulike stoffer. Alle stoffene ble ikke analysert i alle prøver, men det totale antall målte verdier utgjorde i overkant av 1300. Høydepunkter fra resultatene presenteres her, mens det komplette datasettet er tilgjengelig i Vannmiljø databasen (https://vannmiljo.miljodirektoratet.no/).

Stoffene ble gruppert avhengig av hovedbruksområde og/eller fysiokjemiske egenskaper (bisfenoler; desinfeksjonsmidler og løsemidler; lang-kjedede klorerte parafiner; plantevernmidler og legemidler; tilsetningsstoffer i gummi; siloksaner; tensider, inkludert PFAS; og UV-stoffer). Resultatene ble vurdert basert på deteksjonsfrekvens i de ulike prøvene, gjennomsnittlig konsentrasjon, og ved å sammenlikne målte konsentrasjoner med predikert konsentrasjon for ingen effekt i miljøet (PNEC), der det var tilgjengelig. Dette er økotoksikologiske terskelverdier som er utledet ved å ekstrapolere fra eksperimentelle data og, i noen tilfeller fra QSAR-baserte prediksjonsmodeller.

Stoffgruppen relatert til syntetisk gummi bestod av 25 ulike stoffer. Disse ble bestemt i alle prøvetyper bortsett fra marin biota. Høye konsentrasjoner, som oversteg tilhørende PNEC verdier, ble målt i alle prøver assosiert med dekk. Disse prøvene kom fra områder nær høytrafikkert vei, en lokalitet for innsamling og gjenbruk av brukte dekk og kunstgressbaner (både innendørs og utendørs). De høyeste nivåene ble målt for stoffene: CAS 941-57-1 (benzotiasol-2-sulfonsyre, BTSA), CAS 934-34-9 (2-hydroksybensotaisol, OHBT), CAS 793-24-8 (6PPD) og dets ozon bi-produkt 6PPD-Quinone, CAS 74-31-7 (DPPD), CAS 101-72-4 (IPPD), CAS 26780-96-1 (antioksidant TMQ), CAS 3089-11-0 (heksametoksy metyl melamin, TMMAT) og CAS 102-06-7 (1,3-difenylguanidin, DPG). De fleste av disse stoffene tilsettes til polymeren ved produksjon av gummi, og i mengder som utgjør hundredeler (%). Stoffene foreligger som integrerte bestanddeler av polymeren, og dersom kun en liten partikkel fra dekket er til stede i en prøve vil det føre til veldig høye målte konsentrasjoner. Flere av stoffene påvist, ble i litteraturen beskrevet til å kunne lekke ut fra partiklene og dermed gå over til vannfasen.

Ingen av de nye PFAS stoffene ble funnet i noen av prøvene. Derimot ble det ene tensidet CAS 2687-94-7 (oktyl-2-pyrrolidon, OP) observert i prøver av slam og gummigranulat fra kunstgressbaner, stedet for innsamling og gjenbruk av brukte dekk og fra tunnel. Støvprøver fra høytrafikkert vei inneholdt også dette stoffet. OP er registret for bruk i bilavfettingsmiddel, noe som kan forklare funnene.

Av de to UV-stoffene som var inkludert var det kun CAS 1137-42-4 (4-Hydroxybenzophenone, 4-HBP) som ble observert men ikke med nivåer som overskred tilhørende PNEC verdier.

Blant de fem siloksanene (L6, L7, L8, L9 og L10) inkludert ble kun de tre følgende bestemt kvantitativt: L6, L7 og L8 ble funnet i prøver av ferskvann, sediment og biota, og med nivåer som overskred respektive PNEC verdier. L9 og L10 ble målt ved såkalt "suspect screening”, men ikke kvantifisert. Høye nivåer av alle de fem siloksanene ble observert i prøver av luft og støv. Prøver fra det kommunale renseanlegget viste at høygrads rensing førte til mer effektiv fjerning av siloksanene sammenliknet med lav rensegrad. Mesteparten av siloksanene så ut til å havne i slammet.

Blant de seks stoffene i gruppen for desinfeksjonsmidler og løsemidler ble kun CAS 75-09-2 (Diklorometan, DCM) og CAS 67-66-3 (Kloroform, TCM) observert ved betydelige nivåer. Disse stoffene ble funnet i vann fra den urbane elven Alna, og i avrenning fra stedet for innsamling av brukte bildekk.

Gruppen med innsektsmidler og legemidler inneholdt seks ulike stoffer. Disse ble bestemt i prøver av vann og slam fra renseanlegg for avløpsvann. Stoffet amitriptylin (CAS 549-18-8) ble observert i vannet ved konsentrasjoner som overskrider nivåer som har vist å ha en påvirkning på akvatiske organismer (PNEC). Det ble funnet høyere konsentrasjoner i vannet fra høy rensegrad sammenliknet med lav rensegrad. Dette er et vanlig funn for noen legemidler, og skyldes at enzymer fra mikroorganismer brukt i prosessen kan tilbakeføre legemiddelmetabolitter fra mennesker til det originale legemiddelet.

Lang-kjedet klorerte parafiner (LCCP) ble målt i vann, sediment og biota men ikke med nivåer som overskred tilhørende PNEC verdier.

Gruppen med bisfenoler inkluderte fem stoffer. Disse ble kun observert i et fåtall av prøvene, og med lave nivåer som var nært analysemetodens deteksjonsgrense.

1 Introduction

1.1 Aim of the 2022 Programme

The 2022 Screening Programme of the Norwegian Environment Agency aims at following up some of the significant or worrying findings from the 2020 Screening Programme. This includes further investigation into plastic-related substances, tyre-related substances, and disinfection agents. The project also includes additional new PFAS, bisphenols, long-chain chlorinated paraffins, and siloxanes.

The objectives were to:
- investigate whether the substances were found at hot spot locations.
- investigate whether the substances were introduced to- and found in nature.
- assess whether the levels found may cause environmental damage.

The substances included in the 2022 Screening Programme were selected by the Norwegian Environment Agency's screening group based on external and internal input. The substances were prioritized based on previous investigations in the environment, physiochemical properties (including quantitative structure–activity relationship modelling) and the substances' use quantities.

See B. Appendix – List of substances, for a complete list of the substances.

1.2 Rationale for sampling locations and sample types

The main objective of the project was to close knowledge gaps about the discharge and distribution of the target substances, and to investigate the occurrence of these substances in biota. Functional use of the substances and their physiochemical properties were critical factors guiding the design of the sampling campaign. Knowledge from previous findings (including the 2020 Screening Programme) was also instrumental.

There was no information to indicate that any of the substances were produced in Norway, so hotspots resulting from factory emissions or releases during production were unlikely. Most of the substances are however included as one of several components in various products such as car-tyre rubber, plastics, dyes, and textiles. Priority was therefore given to sampling locations associated with car tyre material such as artificial grass pitches and heavily trafficked roads.

The sites covered, for abiotic samples:
- artificial grass pitch (inside and outside)
- tunnel environment
- small freshwater lake close to trafficked road
- highly trafficked road
- wastewater treatment plant with low and high degree of purification
- car tyre collection and recycling site
- urban river
- inside people's houses
- inside commercial buildings (furniture store, dentist's clinic, and facilities using 3D-printing)

For biota, the following locations were covered:

- a small freshwater pond close to trafficked road
- biota from the inner and outer Oslo fjord, representing a marine food web under pressure.

At each sampling site the selection of sample types was made mainly based on the physiochemical properties of the substances. For example, very hydrophilic substances can be expected to reside in the water phase while less hydrophilic substances may accumulate in sediments/sludges. Substances that are not very hydrophilic can reside in the water phase when in association with particles. Therefore, at some locations, the distinguishment has been made between a total water sample (water + particles) and a filtered water sample (water) and the particulate. Substances that are volatile can occur in air. Less volatile molecules can be distributed in air when bound to small particles and/or dust. Biotic samples are relevant for the less hydrophilic compounds that can occur in the biological tissue. The different types of samples covered water; filtered water; sediment; sludge; granule; air; air particles; dust; and biota.

2 Materials and Methods

2.1 Sampling strategy

For a rational sampling-design and selection of the most cost-effective and accurate analytical methodology, the set of compounds was divided into several groups. The grouping was based primarily on functional use of the substances, as well as their physiochemical properties. Functional use of the substances helps indicate the most appropriate sample-types to include in the study. For example, substances associated with rubber were analysed in most sample types, while the pharmaceuticals were only expected in samples from the municipal wastewater treatment plant. Also, physicochemical properties of the substances were relevant to decide the most appropriate sample-types. For example, LCCPs were expected to be found in dust rather than air, and in sediments and sludge rather than water. See Table 1 for an overview of the substance groups and from which sampling sites the samples were determined for the respective substance group. Note that some groups were based on classifications made by the Norwegian Environment Agency during prioritization. Note also that not all compounds in a group have been measured in all the same sample types. For a complete list of substances, see B. Appendix – List of substances. For a complete list of sampling sites, including the coordinates, see C. Appendix – List of sampling sites.

Here follows a description of the sampling locations and the types of samples collected. For more details on the methods used for sampling, see A. Appendix - Sampling. Note that due to instability of a few of the compounds, a special type of sample conservation was required (for details see Chapter 2.2).

Table 1: Overview over the sampling sites and sample types, and which samples that were analysed for the presence of the various substance groups (shaded blue). See B. Appendix for a complete list of the substances in each substance group.

		Substance group							
Sampling locations and sample types		**Bisphenols**	**Disinfectants and solvents**	**Long- chain chlorinated paraffins**	**Pesticides and pharmaceut**	**Rubber related**	**Siloxanes**	**Surfactants incl. PFAS**	**UV- additives**
Artificial grass pitch									
Outdoor	Water	🟦 ǂ		🟦	🟦 ϴ	🟦		🟦 ¤	
	Granule	🟦 ǂ		🟦	🟦 ϴ	🟦	🟦	🟦 ¤	
	Sludge	🟦 ǂ			🟦 ϴ	🟦	🟦	🟦 ¤	
Indoor	Air, particles	🟦 ǂ		🟦	🟦 ϴ	🟦 ǂ			
	Granule	🟦 ǂ		🟦	🟦 ϴ	🟦	🟦	🟦 ¤	
High trafficked road environments									
Tunnel wash	Water, filtered	🟦			🟦 ϴ	🟦			🟦
	Water, particles	🟦		🟦	🟦 ϴ	🟦			
	Sludge	🟦		🟦	🟦 ϴ	🟦	🟦	🟦 ¤	🟦
Recipient pond	Water, filtered	🟦			🟦 ϴ	🟦			
	Water, particles	🟦 ǂ		🟦		🟦			🟦
	Sediment	🟦		🟦	🟦 ϴ	🟦		🟦 ǂ	🟦
Road	Air, particles	🟦 ǂ		🟦		🟦 ǂ		🟦 ¤	

Wastewater treatment									
High quality	Water	🟦			🟦	🟦	🟦	🟦 ǂ	🟦
	Sludge	🟦		🟦	🟦	🟦	🟦	🟦	🟦
	Air	🟦 Ω	🟦		🟦 ϴ	🟦 ǂ	🟦		
Low quality	Water	🟦			🟦	🟦	🟦	🟦 ǂ	🟦
	Sludge	🟦		🟦	🟦	🟦	🟦	🟦 ǂ	🟦
Hotspots									
Car-tyre recycling site	Water, filtered	🟦			🟦 ϴ	🟦	🟦		🟦
	Water, particles	🟦 ǂ		🟦		🟦			
	Sludge	🟦		🟦	🟦 ϴ	🟦		🟦 ¤	🟦
	Dust	🟦		🟦		🟦		🟦 ¤	🟦
Urban river	Water	🟦 ǂ	🟦		🟦	🟦	🟦	🟦 ǂ	🟦
	Sediment	🟦 ǂ		🟦	🟦	🟦	🟦	🟦 ǂ	
Private homes	Air	🟦 ǂ			🟦 ϴ	🟦 ǂ	🟦		
	Dust	🟦		🟦	🟦	🟦		🟦	🟦
Dental clinic	Air	🟦 ǂ			🟦 ϴ	🟦 ǂ	🟦		
	Dust	🟦 ǂ		🟦		🟦	🟦	🟦 ¤	
3D-Printing facility	Air	🟦 ǂ			🟦 ϴ	🟦 ǂ	🟦		
	Dust	🟦		🟦		🟦	🟦	🟦 ¤	🟦
Furniture store	Air	🟦 ǂ			🟦 ϴ	🟦 ǂ	🟦		
	Dust	🟦		🟦		🟦	🟦	🟦 ¤	🟦
Biota (recipients)									
Marine	Seal blubber	🟦 ǂ		🟦	🟦 ϴ	🟦 ǂ	🟦		
	Seagull egg	🟦 ǂ		🟦	🟦 ϴ	🟦 ǂ	🟦		
	Cod liver	🟦 ǂ		🟦	🟦 ϴ	🟦 ǂ	🟦		
Freshwater	Perch liver	🟦 ǂ		🟦		🟦	🟦		🟦
	Duck mussel	🟦 ǂ		🟦		🟦	🟦		🟦
Reference samples									
Urban Park	Air	🟦		🟦	🟦 ϴ	🟦	🟦	🟦 ¤	🟦
	Air, particles	🟦		🟦	🟦 ϴ	🟦	🟦	🟦 ¤	🟦

\# Pesticides only
¤ Excluding PFAS
ǂ Not all substances
^ PFAS only
ϴ CAS 42074-68-0 (CTC/CTA) only
Ω CAS 80-07-9 (BCPS) only

2.1.1 Artificial grass pitch

Large amounts of rubber granules are used on artificial sports-fields in Norway, located both outdoor and indoor. The granules are typically made from used car tyres and are expected to contain several of the compounds of interest. These have been added to the tyre during production, to provide properties like enhanced UV-resistance or flexibility. Thus, artificial grass pitches are at risk of contaminating the nearby environment with these additives (in addition to the rubber itself).

Table 2 provides an overview of the samples collected from both outdoor (Apalløkka, Valle Hovin north, and Valle Hovin east) and indoor sites (OBOS and Lillestrøm Sportsklubb: LSK). Samples from the outdoor training facilities covered surface water runoff and sludge which were mainly collected from manholes. See Figures 1-4 for the locations and photos. The water samples were collected using a sampler installed inside the manhole which is designed to capture the first flush of a precipitation event (see Appendix A.1). One limitation to this sampling technique is a longer time from the sample reaches the sampler to arrival at the laboratory for necessary sample conservation. This may have influenced the measured level of the short-lived 6-PPD compounds (echa, 2023). At the Valle Hovin eastern site, the manhole was covered with a black filter cloth, apparently to prevent granules from entering the drainage system (Figure 4). This was not the case at Valle Hovin north, and thus, sampling covered two different types of manholes with regards to the distribution of granules. At the site Valle Hovin east, it was not possible to collect sludge from the manhole. Instead, one sample was collected from a stretch of scraped-off granules (Figure 3). Sampling at the indoor sports fields included granules and particle phase in air (Figure 5). At the Obos site, sampling was conducted two times, both during daytime and nighttime. Gas-phase air was not collected as the car tire additives are not expected to be found to any large extent in the gas phase given their limited volatility.

Table 2: Overview of the samples collected from artificial grass pitches.

Conditions	**Site name**	**Sample type**	**No.**
Outdoor	Apalløkka	Water	1
		Sludge	1
		Granule	1
	Valle Hovin north	Water	1
		Sludge	1
		Granule	1
	Valle Hovin east	Water	1
		Sludge	1
		Granule	1
Indoor	OBOS (daytime)	Air - particles	1
		Granule	1
	OBOS (nighttime)	Air - particles	1
		Granule	1
	LSK	Air - particles	1
		Granule	1
Total			**15**

Figure 1: Overview of the Apalløkka site. The manhole that was sampled is indicated by a red cross and the areas sampled for granules in blue. Source: GoogleEarth.

Figure 2: Photos of the manhole at Apalløkka from which water and sludge samples were collected. Photos: NIVA/Christian Vogelsang.

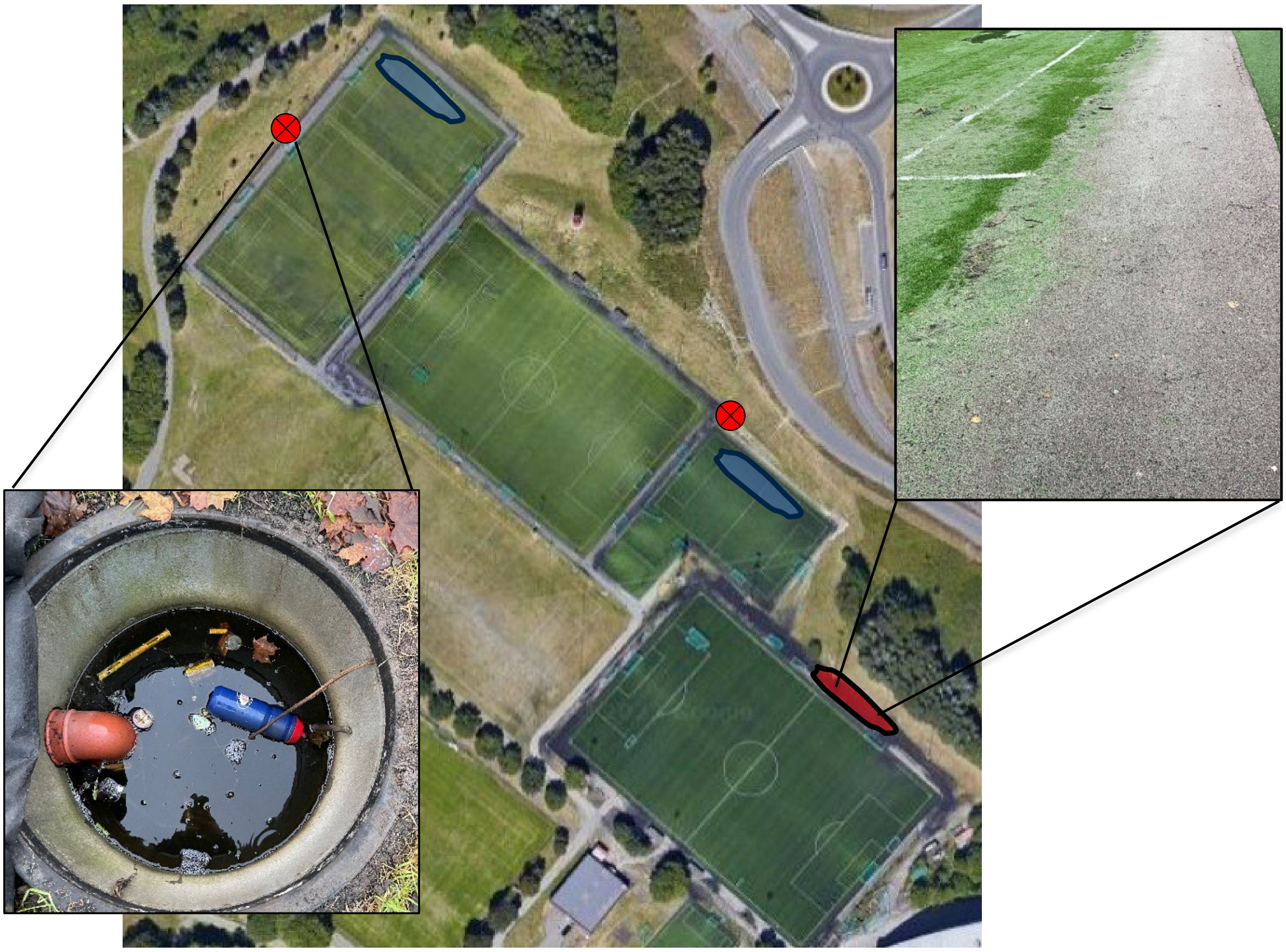


Figure 3: Overview of the Valle Hovin north and east sites illustrating locations for sampling of water and sludge (red filled and crossed circles); granules (grey areas); and one sludge (red area). Source: GoogleEarth. Photos: NIVA/Christian Vogelsang.

Figure 4: Photos of the sampled manhole at Valle Hovin east showing the black cloth used to prevent granules from entering the drainage system. Photos: NIVA/Christian Vogelsang.

Figure 5: Photos from the indoor sampling of particle phase in air (left) and granules (right). Photos: NILU.

2.1.2 Tunnel wash samples

Tunnels are hotspots for car tyre particles which are expected to contain several of the compounds of interest. The closed environment allows accumulation of the particles over time. During the event of washing, accumulated particles will be flushed out. Most of the > 1 200 road-tunnels in Norway do not have any form of treatment of the wash water. In its simplest form of treatment, gully pots are used to retain larger debris, rocks, and even some of the largest car tyre particles. For the tunnels that have some form of water treatment it is based on particle retention in the form of Sedimentation basins. The efficiency of the treatment is dependent on whether the contaminants are bound to particles or not, and on the size and morphology of the particles which governs their potential to settle within the designated time and conditions. Thus, small particles and pollutants not bound to particles may pass directly through the treatment.

Samples were collected from two sites in Oslo, the Smedstad and the Vålerenga tunnels, during the event of washing. See Table 3 for an overview of the samples. The Smedstad tunnel is 500 m long and with annual average traffic at 44,000 vehicles per day. Originally, all samples were planned to be collected from Smestad. However, due to multiple unforeseen events, including water restrictions during the 2022 summer drought, several of the samples had to be collected from the Vålerenga tunnel. The Vålerenga tunnel is 820 m long and with average traffic at 66,400 vehicles per day. The Smestad tunnel was also included in the 2020 Screening Programme (Schlabach et al., 2021).

A tunnel wash commonly proceeds by first sweeping the tunnel to remove loose material like dust and gravel, using a sweeping and vacuuming truck. Subsequently, the tunnel is washed, which may take two to three hours to complete depending on the length of the tunnel. The road surface, side walls, roof, and equipment of the tunnel is washed using soap and high-pressure water. The water from the cleaning will flow to the drainage system through gully pots and end up in a pump basin outside the tunnel. From there, the water is pumped into a sedimentation basin for treatment (~ 21 days) and subsequently released to the nearby environment or sewage system.

To represent conditions prior to the wash, samples of sludge were collected from the gully pot at the inlet (~ 100 m inside) and the outlet (~ 400 m inside) of the tunnel (Smestad, Figure 6). Sludge samples were also collected directly from the sweeping and vacuuming truck (Smestad). Water samples were collected from the pump basin at the beginning, during, and towards the end of the wash (Vålerenga), but before the water was transferred to the sedimentation basin (Figure 7). From the sedimentation basin, water samples were collected on day 14 during the sedimentation period (total duration of 21 days). On day 21 the treated water was released to the municipal storm water network which is connected to River Alna at Kværnerbyen where the river is piped. The treated water was collected from the outlet basin prior to release, at the beginning, during, and towards the end of the release. In addition, sludge samples from the sedimentation basin were collected.

Table 3: Overview of the samples collected from tunnels.

Site name	**Conditions**	**Sample type**	**No.**
Smestad	Inlet, sand trap, gully pot	Sludge	2
	Outlet, sand trap, gully pot	Sludge	2
	Sweeping and vacuuming truck	Sludge	2
Vålerenga	Pump basin, start of wash	Water filtered	1
		Water (total)	1
	Pump basin, during wash	Water filtered	2
		Water (total)	2
	Pump basin, end of wash	Water filtered	3
		Water (total)	3
	Sedimentation basin	Sludge	2
	Sedimentation basin, day 14 after cleaning	Water (total)	2
	Discharge treated water, start	Water filtered	2
		Water (total)	2
	Discharge treated water, during	Water filtered	2
		Water (total)	2
	Discharge treated water, end	Water filtered	2
		Water (total)	2
Total			**34**

Figure 6: Photo of sampling from the gully pot in the Smedstad tunnel (left) and sludge sampled from the gully pot (right). Photos: NIVA/Elisabeth Rødland.

Figure 7: Photo of water sampling using a telescope sampler from the pump basin (left) and from the Sedimentation basin (right), both from the Vålerenga tunnel. Photos: NIVA/Elisabeth Rødland.

2.1.3 Recipient pond: road and tunnel environment

A small pond located downstream of the Smestad tunnel presumably receives runoff from the nearby high-trafficked road as well as treated tunnel wash-water. The pond consists of two smaller basins, referred to as the upper and lower parts (Figure 8). Water and sediments were sampled from the two basins by the aid of a rubber boat (Table 4). Three sediment samples were mixed into one sample for each of the basins, before a subsample from each mixed sample was taken and placed into two separate sterile jars.

Table 4: Overview of the samples collected from the recipient pond.

Site name	Conditions	Sample type	No.
Smestaddammen	Upper part, closer to the road	Water filtered	1
		Water (total)	1
		Sediment	1
	Lower part of pond, further away from the road	Water filtered	1
		Water (total)	1
		Sediment	1
Total			**6**

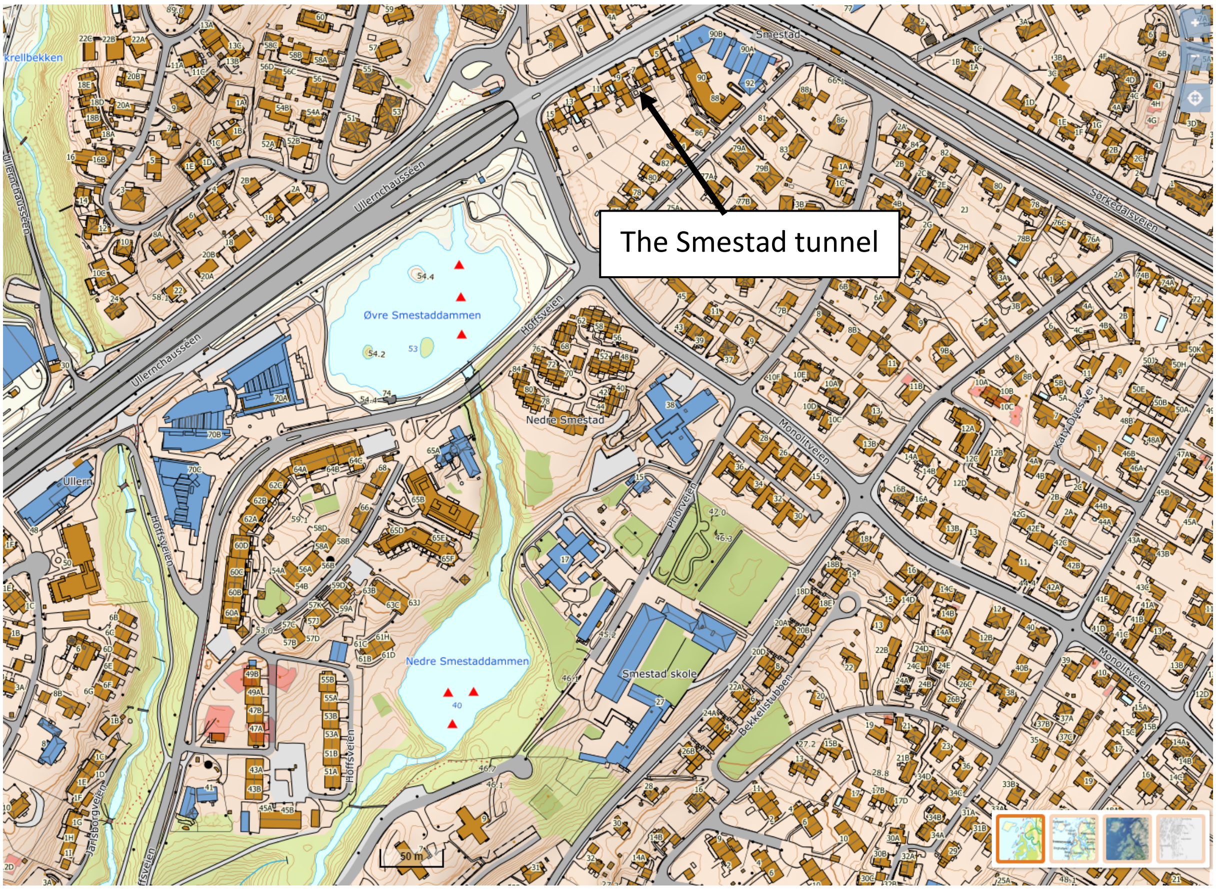


Figure 8: Map showing the location of the two basins of the Smestaddammen and the nearby Smestad tunnel. Sampling sites are indicated by red filled triangles. Source: norgeskart.no

2.1.1 High trafficked road and urban park

Airbourne car tyre-wear particles are likely to be present in air near high trafficked roads. Therefore, samples of particle phase in air were collected from three different locations of high trafficked roads in Oslo. The sites were Alnabru, Hjortneskaia, and Smestad, and with the less traffic affected urban location of the urban park, Sofienbergparken for reference. Samples were collected during summer and winter, and during weekday and weekend. This was done to capture the natural variation from seasons and from people's typical travelling habits. To get a broader picture of the presence of the compounds of interest in urban background air, gas phase samples were also collected at the urban park, Sofienbergparken. See Table 5.

The air particle samples were collected via low volume, single-filter samplers. The low volume samplers were placed at a height of two meters on top of existing monitoring stations (Figures 9 and 10). These filter samples were run for 72 hours each. To sample air gas phase at the urban park, ABN adsorbent samples were deployed and collected via a PTFE line connected to a pump with sampling for 72 hours.

Table 5: Overview of the samples collected from busy roads and an urban park.

Site name	Conditions	Sample type	No.
Alnabru	Summer - weekday	Air – particles	1
	Summer - weekend	Air – particles	1
	Winter – weekday	Air – particles	1
	Winter – weekend	Air – particles	1
Hjortneskaia	Summer - weekday	Air – particles	1
	Summer - weekend	Air – particles	1
	Winter – weekday	Air – particles	1
	Winter – weekend	Air – particles	1
Smestad	Summer - weekday	Air – particles	1
	Summer - weekend	Air – particles	1
	Winter – weekday	Air – particles	1
	Winter – weekend	Air – particles	1
Urban Park, Sofienbergparken (reference)	Summer - weekday	Air – particles	1
	Summer - weekend	Air – particles	1
	Winter – weekday	Air – particles	1
	Winter – weekend	Air – particles	1
	Summer - weekday	Air	1
	Summer - weekend	Air	1
	Winter – weekday	Air	1
	Winter – weekend	Air	1
Total			**20**

Figure 9: The air measurement station by high trafficked roads at Smestad. Photo: NILU.

Figure 10: The air measurement station by high trafficked road at Tittugrenda, Alnabru. Photo: NILU.

2.1.1 Wastewater treatment plant

Wastewater treatment plants are designed with treatment systems of different degrees of purification. The choice of treatment standard depends on factors such as capacity, the quality of the wastewater, and the conditions at the site for discharge. The wastewater treatment plant, Bekkelaget RA has sequential both simple (mechanical) and advanced (biological-chemical) treatment systems. Thus, the plant allows for sampling of wastewater treated with two different degrees of removal principles, and with the same incoming wastewater. Bekkelaget RA is the second largest wastewater treatment plant in Norway (ca. 300,000 person equivalents). The treated water is discharged into the Bekkelaget basin in the Oslo fjord (50 m depth). The site was also included in the 2020 Screening Programme (Schlabach et al., 2021).

The low degree purification consists of mechanical treatment by a coarse screen, a sand- and grease trap, and pre-sedimentation. Scrap material from the coarse screen and sand from the fat trap are landfilled, while grease from the fat trap and settled solids from the pre-sedimentation basin (primary sludge) are transferred to further sludge treatment (anaerobic sludge digestion for biogas production). To represent the simple treatment water samples were collected from the effluent from the pre-sedimentation basin and sludge samples from the primary sludge (from the transport belt). This corresponds to primary treatment.

High degree purification is achieved by subsequent biologic treatment in a co-precipitation step that also include nitrogen removal (anaerobic and aerobic treatment). The resulting effluent goes through a sand filter (polishing step) before the treated wastewater is discharged. Sludge from the biological treatment is co-digested with the primary sludge. To represent the advanced treatment, water samples were collected from the final effluent water and sludge samples from the final (digested and dewatered) sludge. Additionally, samples of air were collected but only from the high purification. Air samples were collected underneath the cap over the first part of the nitrifying basin (see Figure 13). This corresponds to tertiary treatment.

Sampling was carried out during summer and autumn, to capture seasonal variation. Sampling of water was done by time-proportional four-hour composite samples using automatic sample collectors (Figure 11, left). The sludge samples were daily grab samples and combined in equal amounts to one composite sample.

Table 6: Overview of samples collected from the wastewater treatment plant.

Site name	Conditions	Sample type	No.
Bekkelaget – high quality treatment	Summer	Water	4
		Sludge	1
		Air	2
	Autumn	Water	3
		Sludge	1
		Air	2
Bekkelaget - Low quality treatment	Summer	Water	2
		Sludge	1
	Autumn	Water	3
		Sludge	1
Total			**20**

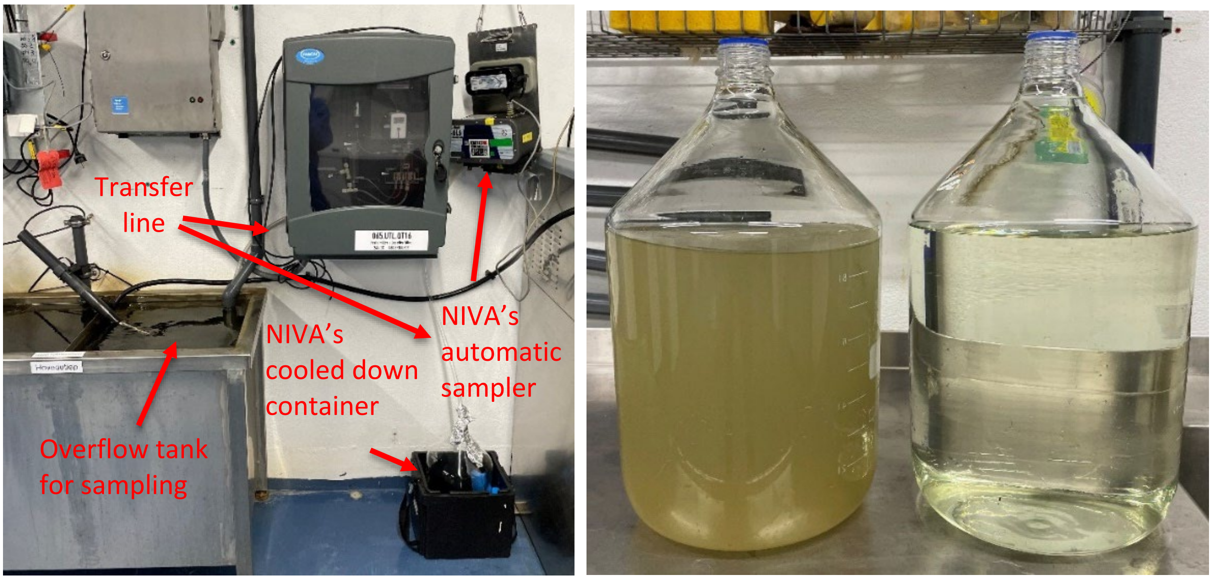


Figure 11: Photo of the automatic water sampler used at the wastewater treatment facility (left) and water samples collected (right) following low quality treatment (dark coloured) and high-quality treatment (light coloured). Photos: NIVA/Christian Vogelsang.

Figure 12: Photos of the sludge centrifuge (left) and with a close up of the entry point for sample collection (right). Photos: NIVA/Christian Vogelsang.

Figure 13: Photos of air sample collection using a low-volume active samples (left) equipped with ABN adsorbents (middle) into stainless stell canisters (right). Photos: NIVA/Christian Vogelsang.

2.1.2 Tyre collection site

Sites for collection and redistribution of used car tyres represent hotspots for release of particles and additives to the nearby environment. At Skjerkøy in Bamble municipality one such site is located, directly on the marine shoreline. Large amounts of whole tyres and shredded tyre material was stored at the outdoor location, and even on a barge outside the marina (Figures 14-16). The area was regularly flushed with large volumes of seawater to prevent tyre particles from spreading with the wind to nearby houses (which had previously been a problem). The water used for flushing was drained either to one single gully-pot located on site, or directly into the ocean.

Water samples were collected both from the gully pot and from the outlet pipe of the gully pot which was draining directly into the ocean. Samples of sludge were also collected from the gully pot. Samples of dust were collected from different types of surfaces at different locations within the area outdoors. See Table 7 for an overview of the samples.

Table 7: Overview of the samples collected from the car tyre recycling site.

Site name	Conditions	Sample type	No.
Skjerkøy	Runoff from the area	Water	1
		Water and particles	1
		Water	1
		Water and particles	1
		Water	1
		Water and particles	1
	Gully pot	Sludge	1
		Sludge	1
		Sludge	1
	House near shredder	Dust	1
	Outside workspace	Dust	1
	By the grab	Dust	1
Total			**12**

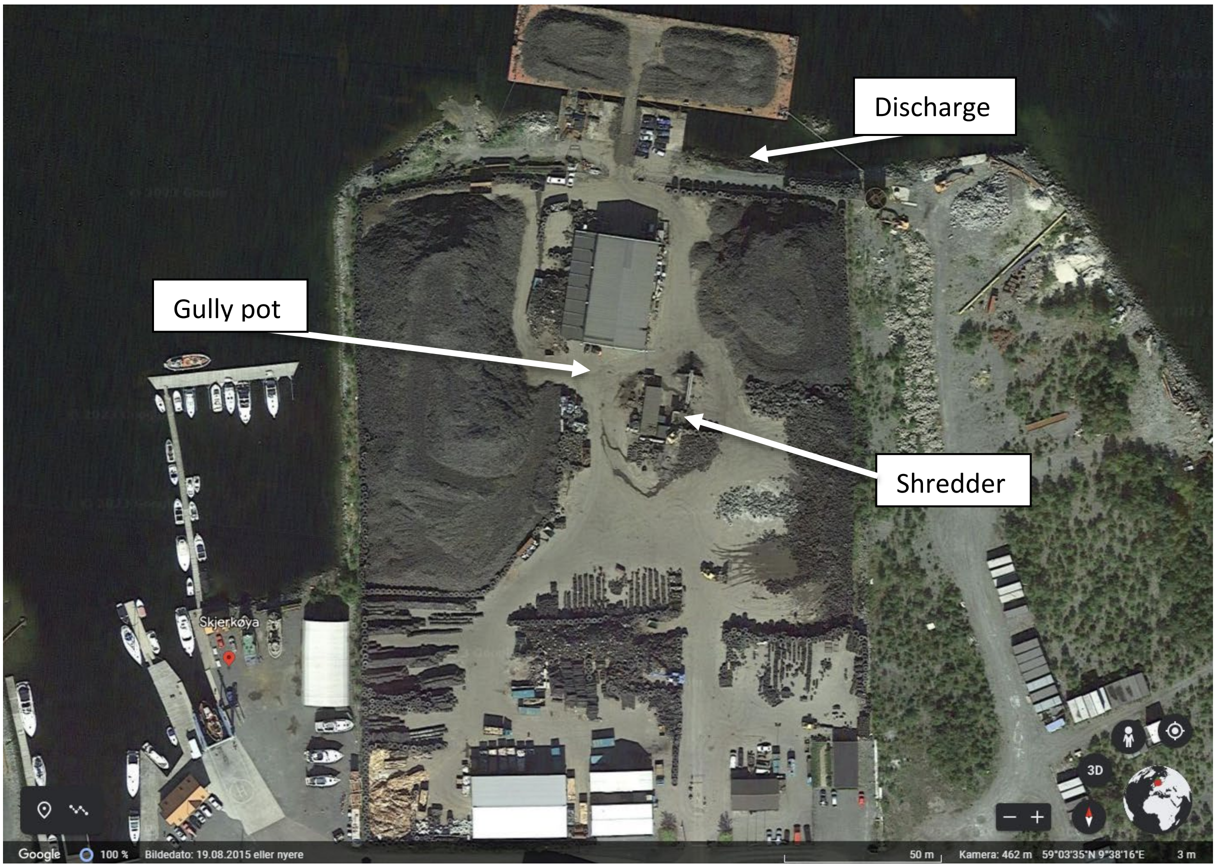


Figure 14: Satellite image of the site for collection and redistribution of used car tyres at Skjerkøya, Bamble. The large amounts of tyres can be seen in black colour. The approximate location of the gully pot in relation to the shredder as well as the discharge from the gully pot to the ocean is indicated. Source: Google Earth.

Figure 15: Sampling from the gully pot (left) and sample of sludge collected from the gully pot (right). Photos: NIVA/ Cathrine Brecke Gundersen.

Figure 16: Sampling of water discharging from the gully pot (left) and sampling of dust from a surface at the site (right). Photos: NIVA/ Cathrine Brecke Gundersen.

2.1.3 Urban River

River Alna is an urban river in Oslo, draining from the Alnasjøen and out into the Oslofjord. It receives runoff from high trafficked roads, different types of industrial activities, and generic urban activities. Four different sites were selected along the river for sampling of water and sediment (Table 8 and Figure 17). Furthest upstream is Kalbakken (nearby Apalløkka football field). Thereafter follows the stations Brubak, and Breivoll. The latter is located downstream the main industrial area at Alna. Furthest downstream is Kværner which represents the river before the river goes into a culvert that leads to the fjord (but does not include the main municipal overflow from the sewer network). The river was also included in the 2020 Screening Programme (Schlabach et al., 2021).

Sampling was conducted during both dry (low flow) and wet (high flow) weather conditions to capture some of the natural variation (Figure 18-20). The water samples were collected mainly as grab samples which deviated from the original plan of collecting temporal composite samples. One important reason for this was to adapt to the short lifetime of the car tyre related substance, 6PPD. Note that the sample at Brubak from dry conditions was collected as a four-hour composite sample (100 mL every 3 min) using automatic sampler (ISCO). See Figure 21. Composite sediment samples (5-10 subsamples from locations within 2-3 m^2) from the top 2 cm layer of wet sediments were collected at all four locations.

Table 8: Overview of the samples collected from the urban river.

Site name	**Conditions**	**Sample type**	**No.**
Kalbakken	Low flow	Water	1
	High flow	Water	1
		Sediment	2
Brubak	Low flow	Water	1
	High flow	Water	1
		Sediment	2
Breivoll	Low flow	Water	1
	High flow	Water	1
		Sediment	2
Kværner	Low flow	Water	1
	High flow	Water	1
		Sediment	2
Total			**16**

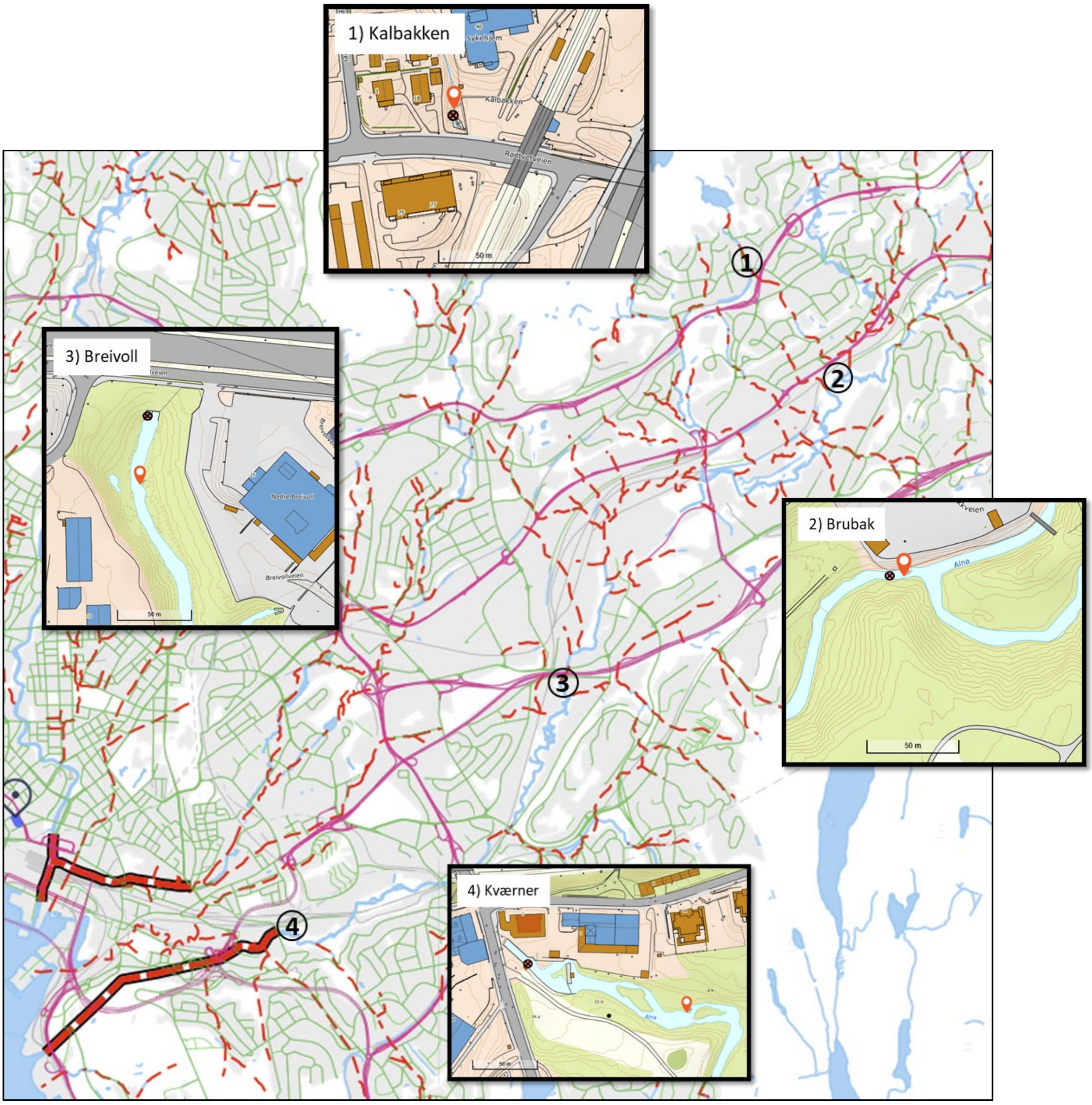


Figure 17. Locations of sampling points along Alna; 1) Kalbakken, 2) Brubak, 3) Breivoll and 4) Kværner. Source: "Plan innsyn", Oslo PBE and www.norgeskart.no (Kartverket)

Figure 18: Photos showing the conditions before (left) and after (right) a stormflow event at station Brubak. Photos: NIVA/Christian Vogelsang.

Figure 19: Photos showing the conditions before (left) and after (right) a stormflow event at station Breivoll. Photos: NIVA/Christian Vogelsang.

Figure 20: Photos showing the conditions before (left) and after (right) a stormflow event at station Kværner. Photos: NIVA/Christian Vogelsang.

Figure 21: Photos of the automatic sampling equipment (left) and the flow meter with the flow sensor mounted to a metal plate locked at the river floor (right), at station Brubak. Photos: NIVA/Christian Vogelsang.

2.1.4 Indoor (commercial and private)

A range of the substances of interest were expected additives in plastics as well as other types of consumer products. Therefore, the indoor environment at both commercial and private buildings were of interest, and subject to sampling of both dust and air (Table 9). The types of commercial buildings included 3-D printing facilities, dental clinics, and furniture warehouses. The identity of the sites has been anonymized. See Figure 22 for photos of the sampling.

For dust sampling, the participants were asked to not vacuum clean or wet clean the floors and the horizontal surfaces in the rooms during the last week before sampling.

Table 9: Overview of the samples collected from inside private and commercial buildings.

Site type	**Sample type**	**No.**
Private home 1	Dust	1
	Air	1
Private home 2	Dust	1
	Air	1
Private home 3	Dust	1
	Air	1
Private home 4	Dust	1
	Air	1
Private home 5	Dust	1
	Air	1
Private home 6	Dust	1
	Air	1
Dental clinic 1	Dust	1
	Air	1
Dental clinic 2	Dust	1
	Air	1
Dental clinic 3	Dust	1
	Air	1
3D-printing 1	Dust	1
	Air	1
3D-printing 2	Dust	1
	Air	1
3D-printing 3	Dust	1
	Air	1
Furniture store 1	Dust	1
	Air	1
Furniture store 2	Dust	1
	Air	1
Furniture store 3	Dust	1
	Air	1
Total		**30**

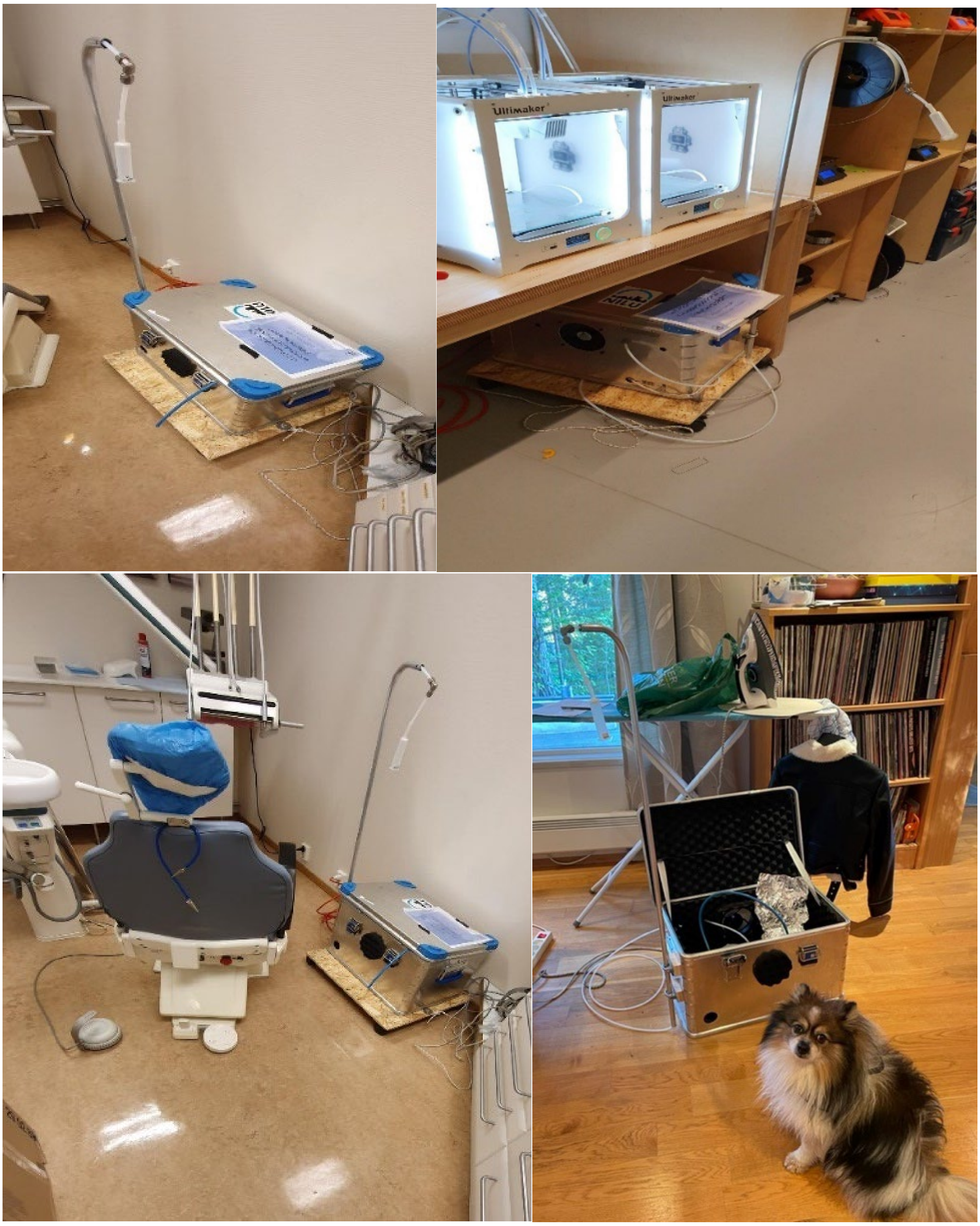


Figure 22: Photos from indoor air sampling with the sampling equipment (top left), sampling by a 3-D printer (top right); at a dental clinic (bottom left); and at a private home (bottom left). Photos: NILU.

2.1.5 Biota (marine and freshwater)

Recipient samples of biological material were collected from different freshwater and marine species (Table 10). The freshwater samples were collected from Lake Padderudvannet outside Oslo (Asker municipality) which is in proximity to one of the most trafficked roads in Norway (E18). The marine samples were from the Oslo fjord which receives runoff directly from the city of Oslo.

Freshwater samples of duck mussels (*Anodonta anatine)* and Perch (*Perca fluviatilis*) were sampled on the north side of lake Padderudvannet (Figure 23). The mussels were collected using an aqua-scope and a trash grabber, which resulted in a total of 15 mussels with a size between 15-57 cm (Figure 24, left). The soft tissue of the mussels was sampled using a stainless-steel scalpel and pooled into three. Perch (*Perca fluviatilis*) was sampled at three different locations using gillnets (Figure 25). Harvesting was conducted early morning the next day and resulted in a total of 15 fish with a length of 27 to 34 cm (Figure 24, right). Liver and filet from the fish was sampled outside (in the field), and three pooled samples from respective tissue were made.

The marine samples were obtained from the Oslofjord through the Urban Fjord monitoring programme. Eggs from Herring gull (*Larus argentatus*, Figure 26, left) were sampled at Søndre Skjælholmen, Raudskjæra and Husbergøya. A total of 15 specimens were pooled into three samples. Cod (*Gadus morhua*, Figure 26, right) was caught with trawl from RV Trygve Braarud at Midtmeie, southwest of Steilene. A total of 15 specimens were pooled into three samples. Blubber from Harbour seal (*Phoca vitulina*) was obtained from the University of Oslo through another research activity (Stensrud, 2022). Seals were collected by five different hunting groups, at Torbjørnskjær, Søndre Missingen, Singleøya and Garnholmen.

Table 10: Overview of the samples collected from freshwater and marine biota.

Conditions	**Specie**	**Sample type**	**No.**
Freshwater (Padderudvannet)	Mussels	Soft tissue	3
	Perch	Liver & Filet	3
Marine (Oslo fjord)	Cod	Liver	3
	Eggs from herring gull	Egg	3
	Harbour Seal	Blubber	3
Total			**15**

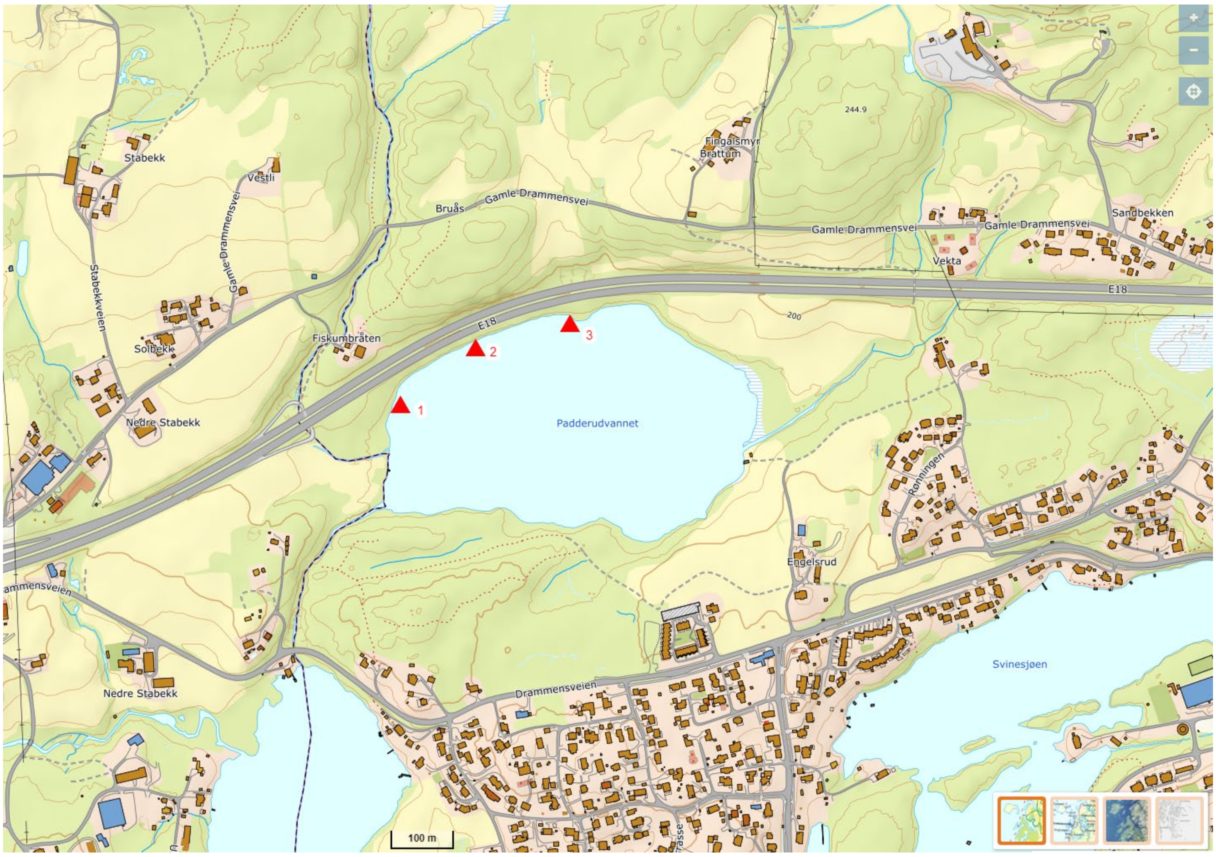


Figure 23: Map of Lake Padderudvannet showing the three stations where gillnets were placed. The duck mussels were collected at around the sampling site in the middle. Source: norgeskart.no

Figure 24: Photo of individuals of duck mussels (right) and Perch (left) and collected. Photos: NIVA/Johnny Håll.

Figure 25: Photo of gillnet harvesting. Photo: NIVA/Johnny Håll.

Figure 26: Photos of eggs from seagull (left) and sampled cod (right). Photos: NIVA/ Kine Bæk (left) and Sigurd Øxnevad (right).

2.2 Sample conservation and storage

Sample conservation is crucial to sustain the compound of interest at the environmental level from sampling and until analysis at the laboratory. Among the substances included, the 6PPD (CAS 793-24-8) gave some challenges due to its short lifetime under natural environmental conditions. Different options were evaluated and tested (acidification, freezing, etc.). The best option was for water samples to be conserved on solid phase extraction cartridges (Waters HLB) that had been activated by methanol (Figure 27). This was done in field or as soon as possible upon arrival at the laboratory. For water samples containing a high number of particles, such as those from tunnel wash, centrifugation or filtration using glass fibre filter (GF-A, 1.6 µm) was conducted prior to conservation using the solid phase extraction sorbent (Waters HLB). The different treatment methods were designated by “Water, total” = no filtration/centrifugation, “Water, filtered” = filtration or centrifugation was carried out, and “Water, particles” = the particulate fraction of the sample isolated by filtration. Note that the filter pore size applied is coarse meaning that small particles would pass through. The size of particles is operationally defined by filtration pore size 0.45 µm. All water and solid phase sample (sludge, sediment, granule, and biota), sample-containing HLB cartridges and sample-containing filters were stored frozen (-20^{o}C) prior to analysis.

Figure 27: Photo of samples with a high particle load being conserved using solid phase extraction cartridges (Waters HLB). Photo: NIVA/Kine Bæk.

2.3 Analytical methods

Analytical methods are summarised in Table 11. In general, solid samples including sediments, tyre granules, sludges, and biota were extracted with acetonitrile, hexane and methanol (alone or in combination) following the addition of internal standards. Liquid samples were extracted via solid phase extraction. Analysis for all substances was via either LCMS or GCMS. For further details see appendix A.3 Details of analytical methods.

Table 11: Overview of the techniques used for samples extraction and analysis for each substance and type of sample matrix.

Analytical method group	**Sample Matrix**						**Analysis Class**
	Air	**Air Dust / Particles**	**Water**	**SS/Sediment/ Sludge**	**Granule**	**Biota**	
Chloropariffines	-	ASE	-	ASE	ASE	ASE	LC-HRMS
SVOC	-	SE	SPE	SE	SE	SE	GC-HRMS
Rubber Additive	-	SE	SPE	SE	SE	SE	LC-MS
Bisphenol	-	SE	SPE	SE	-	-	LC-HRMS
VOC	TD	-	TD	-	-	-	TD-GC-ToF-MS
Siloxane	SE	SE	SPE	SE	-	SE	GC-HRMS
Pesticide/ Pharmaceutical	-	-	SPE	SE	-	-	LC-HRMS / GC-HRMS
Surfactant/PFAS	-	SE	SPE	SE	-	-	LC-HRMS

Glossary:

SS = Suspended Solids

ASE = Accelerated Solvent Extraction

SE = Solvent Extraction

SPE = Solid Phase Extraction
TD = Thermal Desorption
GC-(HR)MS = Gas Chromatography (High Resolution) Mass Spectrometry

LC-(HR)MS = Liquid Chromatography Mass Spectrometry
ToF = Time-of-Flight

2.4 Calculations and data presentation

All calculations were computed using the R programming language (R Core Team, 2023). The detection frequency was calculated based on different sample types and/or sampling sites. When the detection frequency was > 30%, any observations below the method limit of detection (LOD) was given the value of 0.5 * LOD, which was then included into the average value. For method concentration LODs, see Appendix A.3 Table A5. Note that we here use LOD rather than the limit of quantification (LOQ) due to the nature of the programme which is screening. Data is presented with one or two significant digits. Illustrations were made using the R packages ggplot2, plyr, dplyr, tidyr, egg, reshape2, and forcats.

2.5 Collection of PNECs

Toxicity data were obtained in the form of PNECs (predicted-no-effect concentrations). The PNECs are the concentrations of a chemical which marks the limit at which below no adverse effects of exposure in an ecosystem are measured. A PNEC is obtained through the application of an assessment factor to ecotoxicological endpoints (EC50 or no observed effect concentration, NOECs) using organisms from at least three trophic levels (usually algae, daphnids and fish). The assessment factor depends on duration of the test (acute or chronic) and the number of trophic levels. Higher assessment factors are used when only acute data have been used. Also, a higher assessment factor is typically used to encompass for higher uncertainty related to the PNEC value. The minimum requirement for deriving a PNEC is acute toxicity for algae, daphnids, and fish. When no experimental derived PNECs are available, QSAR based prediction models can be used for prioritization purposes (Aalizadeh et al., 2017). It is obvious that modelling PNECs include many simplifications and uncertainties. In addition, most of these lowest PNEC data have not been fully reviewed and verified by experts. This is particularly applicable to some of the PNECs from NORMAN, as demonstrated by Welch et al. (2023).

The PNECs used herein were mainly collected from the two databases, by priority, of ECHA (European chemicals agency: https://echa.europa.eu/) and the NORMAN Ecotoxicology Database (https://www.norman-network.com/nds/ecotox/lowestPnecsIndex.php). The environmental compartments of *freshwater, freshwater sediment,* and *biota fish* freshwater has been used. When no PNEC was available for a given substance in a given environmental compartments, PNECS have been derived based on available literature. The PNECs were collected as is, and without any further quality assurance.

3 Results and Discussion

In this study 154 samples were collected and analysed for 59 substances. Not all substances were determined in all samples, but the total number of analytical results exceeds 1300. Here, highlights from the results are presented. The complete dataset is available for download from the database, Vannmiljø (https://vannmiljo.miljodirektoratet.no/).

3.1 Detection frequencies and average concentrations

Results are presented here by substance group and with the aim of showcasing those substances that were detected most frequently in multiple environmental compartments. For those substances with a detection frequency above 30% the average measured concentration is presented, together with the detection frequency. For the complete list of substances, see B. Appendix – List of substances. Further, the average concentration was evaluated against environmental predicted no-effect concentrations (PNEC) where available. These are ecotoxicological threshold values. For reasons discussed in Chapter 2.5, the following comparison tables should only be used for prioritization and cannot replace more detailed risk assessment. For more information on this, see e.g. Welch et al. (2023).

3.1.1 Bisphenols

The group of bisphenols analysed in this study consisted of five substances. These were found in relatively low concentrations (Figure 28 and 29). OH-BPA (CAS 142648-65-5) is a persistent product of transformation of bisphenol A (Drzewiecka et al., 2021). It was found in samples from the tyre collection site, private home, urban park, and in samples from the wastewater treatment plant. TMBP (CAS 57244-54-9) is an impurity of industrial grade bisphenol A (Poskrobko et al., 2000; Terasaki et al., 2005). It was found in samples from the tyre collection site, tunnel environment, the urban park, and in some of the sample from private homes. Bisphenol TMC (CAS 83558-87-6) is a bisphenol derivative used in the preparation of polycarbonate, polyester, and epoxy resins. It was found in relatively small amounts in sludge and dust from the tyre collection site, and in air from the urban park. 44-BPS2 (CAS 93589-69-6) is a colour developer of the leuco-type thermosensitive papers. In this study it was found in low amounts in sludge from the tyre collection site, dust in private homes, and in air from the urban park. Trans-4,4'-azodiphenol (4,4’-ADP) (CAS 51437-66-2) is an azo dye and a reagent. Only three bisphenols had available PNEC in databases (Table 12). None of the concentrations measured in this study exceeded these values.

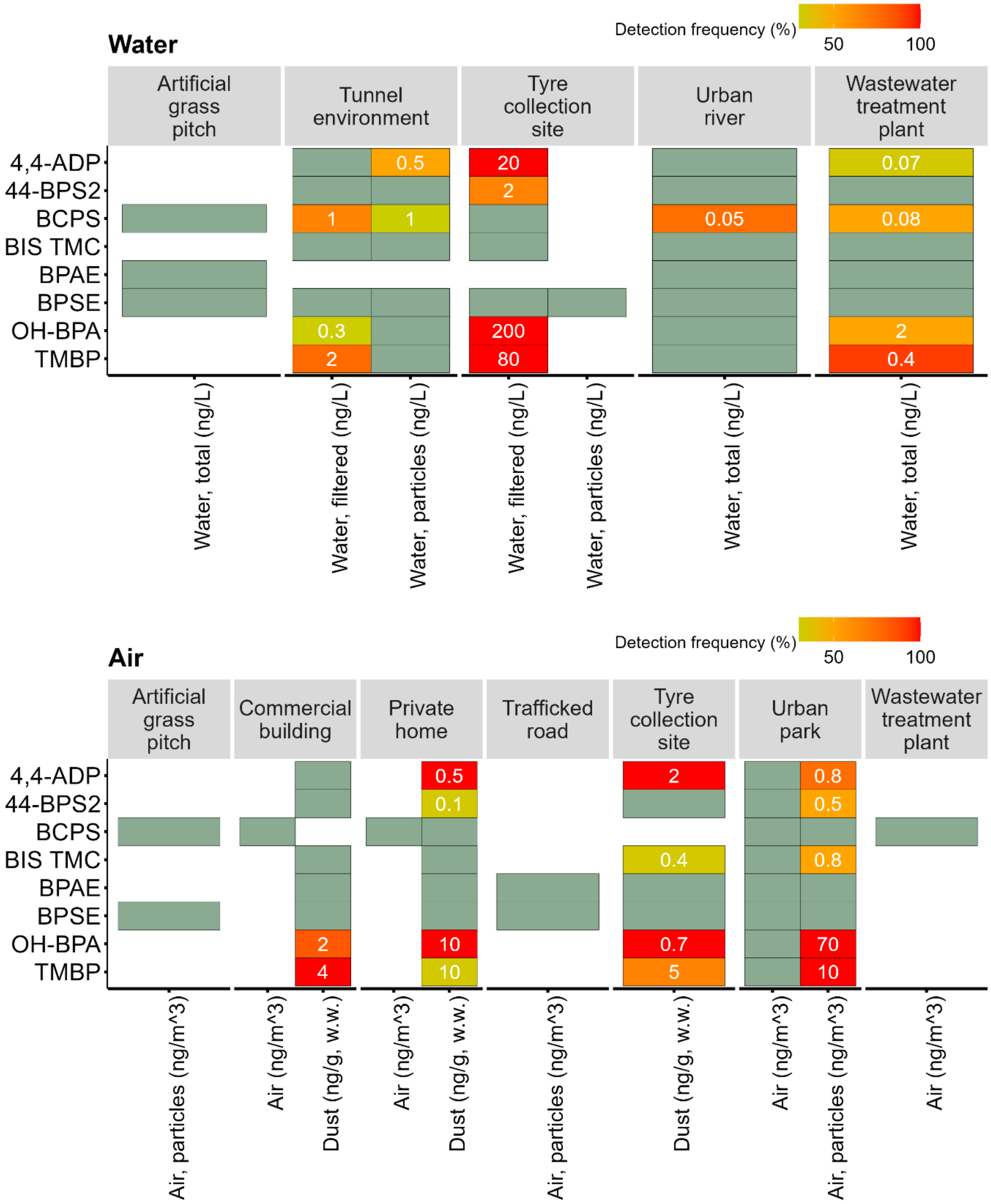


Figure 28: Average concentration in water and air of bisphenols with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

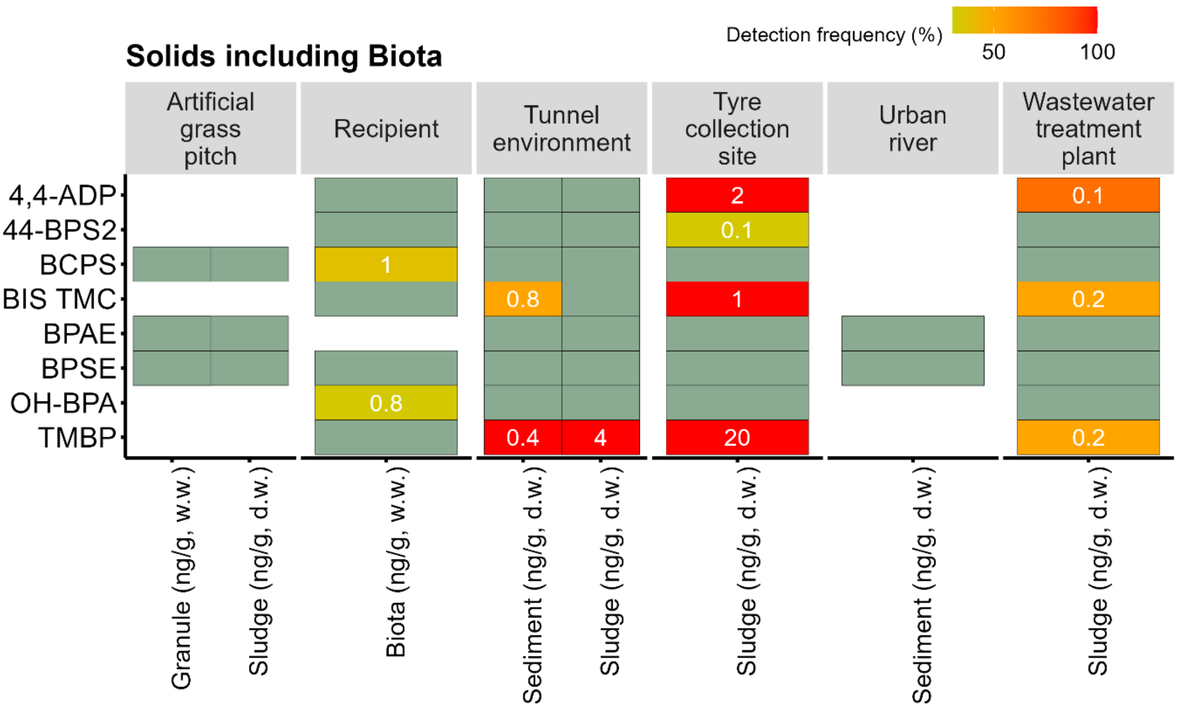


Figure 29: Average concentration in solids including biota of bisphenols with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

Table 12: Predicted no-effect concentrations (PNEC) for bisphenols. None of the average measured concentrations in samples exceeded the PNEC.

Short name	**CAS**	**Fresh water, ng/L**	**Sediment, ng/g**	**Biota (fish), ng/g**	**Data Source**	**Assessment factor**
44-BPS2	93589-69-6	940	254	18.7	NORMAN	1 000
BIS TMC	83558-87-6	1 570	46.6	6.24	NORMAN	1 000
BCPS	80-07-9	6 000	120	802	Freshwater: ECHA Sediment: ECHA Biota: NORMAN	50 100 1 000

3.1.2 Disinfectants and solvents

Among the disinfectant and solvent substances (Figure 30), TCM (CAS 67-66-3) was the only one found in air samples from the wastewater treatment plant. Together with PCE and DCM it was also found in river water. None of the substances were found at levels exceeding their respective PNECs (Table 13).

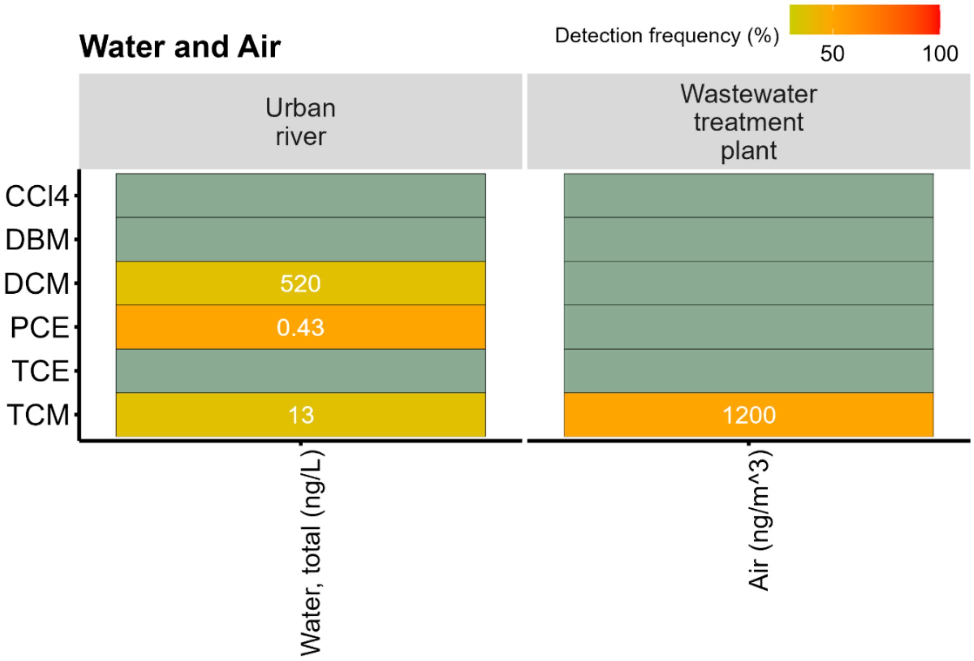


Figure 30: Average concentration in water and air of disinfectants and solvents with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

Table 13: Predicted no-effect concentrations (PNEC) for disinfectants and solvents. None of the average measured concentrations in samples exceeded the PNEC.

Short name	CAS	Freshwater, ng/L	Sediment, ng/g	Biota (fish), ng/g	Data Source	Assessment factor
TCM	67-66-3	146 000	450	32.1	NORMAN	10
DCM	75-09-2	310 000	2 570	455	Freshwater: ECHA Sediment: ECHA Biota: NORMAN	20 Partitioning* n.a.
TCE	79-01-6	576 000	10 200	158	Freshwater: ECHA Sediment: ECHA PNEC Biota: NORMAN	10 Partitioning* n.a.
PCE	127-18-4	51 000	903	524	Freshwater: ECHA Sediment: ECHA PNEC Biota: NORMAN	10 Partitioning* n.a.
CCl4	56-23-5	220 000	61.1	89.4	Freshwater: ECHA Sediment, Biota: NORMAN	10 n.a.
DBM	75-27-4	51	0.23	0.39	NORMAN	n.a.

*a different method from assessment factor has been used.

3.1.3 Long-chain chlorinated paraffins (LCCP)

Long chain chlorinated paraffins were analysed in water, solids, and biota from various locations. They were detected in most of the samples analysed (Figure 31). The highest average concentrations were found in the dust from private and commercial buildings and followed by the tyre collection site. The concentrations detected in the ambient air were lower than average concentrations measured in the Nordic countries (Schlabach et al., 2022). CAS 63449-39-8 (Cereclor) is a plastic additive. Average concentration in water did not exceed the PNEC value, but individual measurements were found to exceed the PNEC (see Chapter 3.3 for overall evaluation of environmental risk).

Table 14: Predicted no-effect concentrations (PNEC) for long-chain chlorinated paraffins (LCCP). Red cells indicate that average measured concentrations in samples exceeded the PNEC.

Short name	**CAS**	**Freshwater (ng/L)**	**Sediment (ng/g)**	**Biota (fish) ng/g**	**Data Source**	**Assessment factor**
LCCP	63449-39-8	3 000	5 710 000	3300 (C18-20)	Freshwater, Sediment: ECHA	10
				580 (C20-30)	Biota: Derived from Freshwater **§**	

§ Biota (Fish) – PNECfw*BCF
- C18-20 LCCP, BCF = 1.096 L/g (Source ECHA)
- C20-30 LCCP, BCF = 0.192 L/g (Source ECHA)

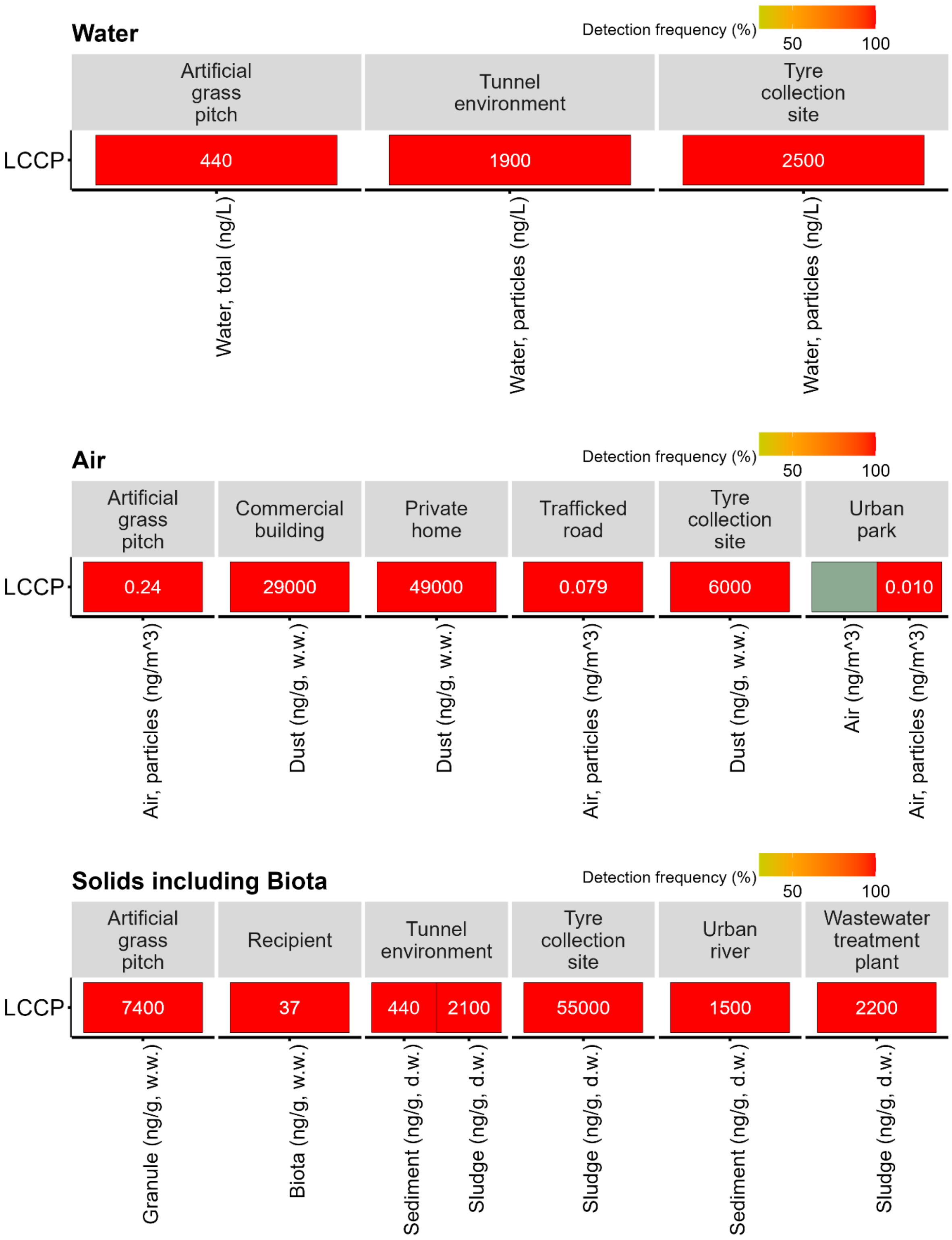


Figure 31: Average concentration in water, air, and solids including biota of long-chain chlorinated praffins (LCCP) with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

3.1.4 Pesticides and pharmaceuticals

The group of pesticides and pharmaceuticals included six substances. These were analysed in samples of treated water and sludge from the wastewater treatment plant. Analysis was also carried out for the fungicide CTC/CTA (CAS42074-68-0) on samples of water and sediment collected from the Alna River. Dust samples from residentials and samples associated with car-tyres were also investigated.

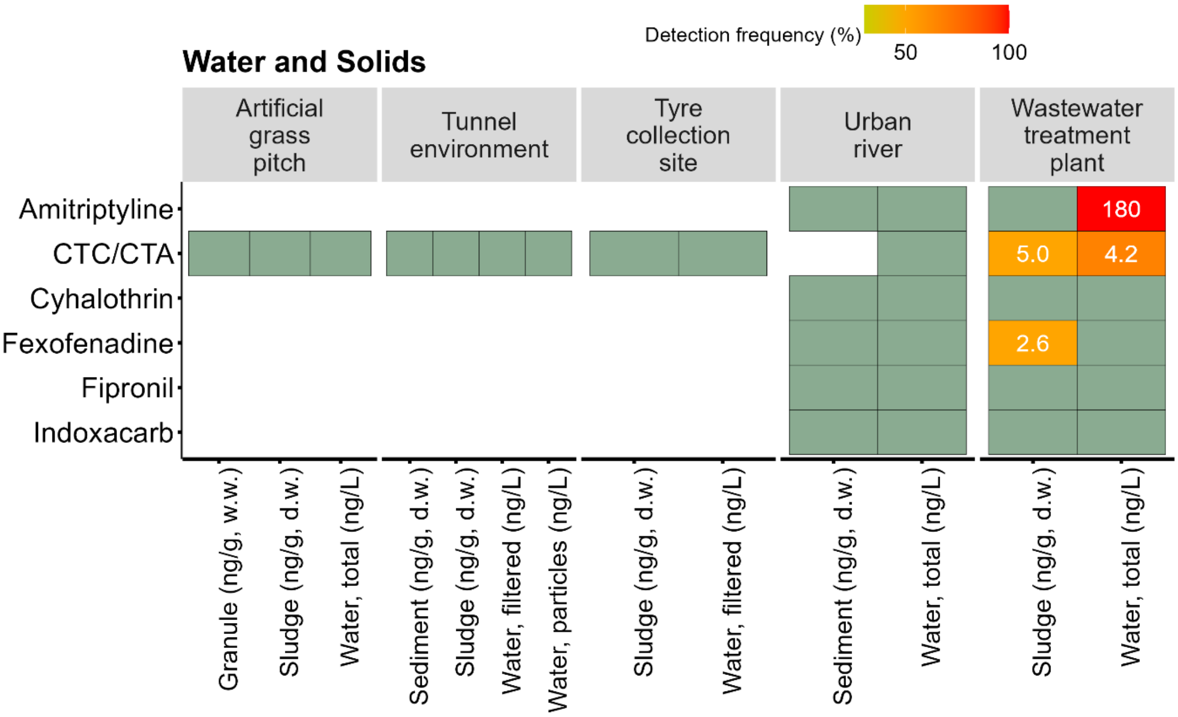
Amitriptyline (CAS 549-18-8) is a tricyclic antidepressant which is among the most prescribed antidepressant in Norway. It was detected in all wastewater effluent samples (Figure 32). There was no seasonal variation observed (data not shown), but large differences were observed between the two different degrees of treatment (see Chapter 3.2.2). The measured concentrations of amitriptyline in treated wastewater effluent were more than those shown to have an impact on aquatic organisms (Table 15) (Schmieg et al., 2022; Ziarrusta et al., 2019). These results indicate potential risks where dilution of treated wastewater in the receiving waters may be limited. This may be of particular concern where treatment discharge is to freshwater rivers, for example. There are only a few studies available that assesses the potential for amitriptyline to bioaccumulate. In a recent study by Magnuson et al. (2022), amitriptyline was found to bioaccumulate in marine systems. Amitriptyline is considered to be stable in the environment, see e.g. (Choi et al., 2018). The combination of bioaccumulation and high stability may warrant risk of effects considering the continuous supply potentially presented by a wastewater treatment plant.

Fexofenadine (CAS 83799-24-0) was also detected at the municipal wastewater treatment plant, albeit at concentrations lower than the PNEC and those previously shown to have an impact on aquatic environments (Sundelin, 2015). This antihistamine is used for the treatment of various allergic symptoms. Approximately 80% of the dose of this pharmaceutical is excreted in faeces. Fexofenadine is a lipophilic drug (LogK$_{ow}$ >5). This is in accordance with detection of this substance in only sludge from the wastewater treatment plant (Figure 32). Fexofenadine was by Malnes et al. (2023) identified as potentially qualifying for the category of persistent, mobile, and toxic (PMT). More studies are warranted.

The fungicide CTC/CTA (CAS 42074-68-0) was detected at low concentrations (below PNECs) in samples from the wastewater treatment plant only.

Table 15: Predicted no-effect concentrations (PNEC) for pesticides and pharmaceuticals. Red cells indicate that average measured concentrations in samples exceeded the PNEC.

Short Name	**CAS**	**Freshwater (ng/L)**	**Sediment (ng/g)**	**Biota (fish) (ng/g)**	**Data Source**	**Assessment factor**
CTC/CTA	42074-68-0	100	60.8	107	Freshwater, Sediment, Biota: NORMAN	1 000
Amitriptyline	549-18-8	55	18	29v	Freshwater: NORMAN Sediment, Biota: derived from freshwater by factors below **§**	1 000
Fexofenadine	83799-24-0	200 000	1 582 098	2529	Freshwater, Sediment, Biota: NORMAN	n.a.
§ Other Assessment Factors: 1. Sediments – Lowest (P)NECfw*2.6*(0.615+0.019*Koc) 2. Biota (Fish) – (P)NECfw*BCF						



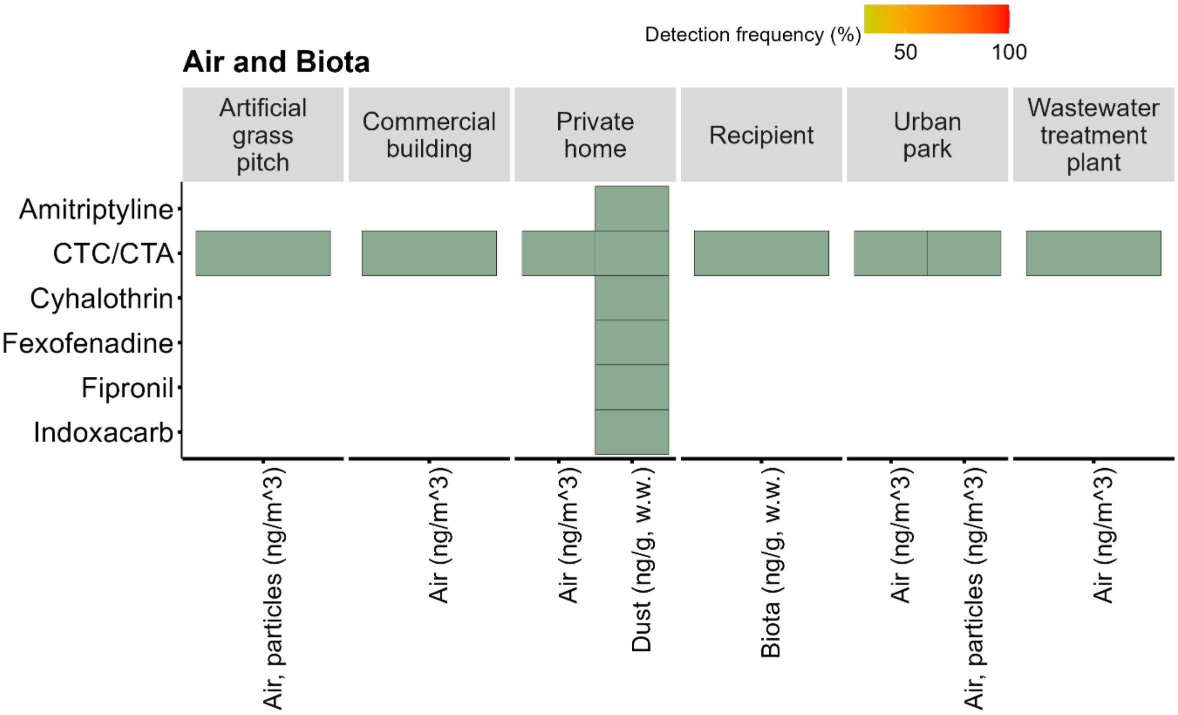


Figure 32: Average concentration in water and solids, and in air and biota of pesticides and pharmaceuticals with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green indicating the substance was not detected.

3.1.5 Rubber related substances

A total of 25 substances were included in the group associated with rubber. These substances were analysed in all samples except marine biota (Figures 33-35).

High concentrations were observed in all samples associated with vehicle tyres. This included all environmental compartments near busy roads, where tyres are recycled, and artificial football fields (both indoor and outdoor). The highest concentrations were observed for CAS 941-57-1 (Benzothiazole-2-sulfonic acid, BTSA), CAS 934-34-9 (2-hydroxybenzothiazole, OHBT), CAS 793-24-8 (6PPD) and its oxidized form 6PPD-Quinone, CAS 74-31-7 (DPPD), CAS 101-72-4 (IPPD), CAS 26780-96-1 (Antioxidant TMQ), CAS 3089-11-0 (Hexamethoxy methyl melamine, TMMAT) and CAS 102-06-7 (1,3-Diphenylguanidine, DPG).

Most of these substances are added in rubber tyre manufacturing at ratios of parts per hundred (%) to the polymer or resin. They are integrated in the polymer particles so even very small amounts of solid, even as fine dust, can therefore lead to very high levels of these substances being found in the sample. The total amount bound within the particles is expected to be of the order of 0.5 – 5.0 % by weight of polymer, as formulated. In this study, exhaustive extraction from particles was not possible. Therefore, the results herein presented should be considered as conservative. For the substances bound to particles, levels in the environment can be reduced through efforts to remove particles, suspended solid and dust.

However, several of the substances have been documented to leach from the car tyre material. In a recent study by Müller et al. (2022), the chemical complexity of new commercial tyres was investigated and the leachability the substances was assessed. Among the more than 200 different organic substances identified, 145 were classified as leachables. Some of the leachables were identified here, for example DPG (CAS 102-06-7), MBT (CAS 149-30-4), and 6PPD (CAS 793-24-8). Another study found additional relevant substances leachable, such as TMQ (Stack et al., 2023). Our results show, that for these substances, there is a tendency of higher concentrations in the filtrated water samples compared to the particulate material (Figure 33).

Note that several of the tyre-related substances in question are transformation products from the substances originally present in the tyre. These may have different physiochemical properties and toxicity than their parent substances. One example is the 6PPD-Quinone which is the more toxic and persistent transformation product of 6PPD. See e.g., Tian et al. (2021); Zhang, R. et al. (2023).

For several of the substances, the average measured levels exceeded PNECs for freshwater and sediment, see Table 16. There are limited data on effects to organisms, so further investigation is needed given the scale of measured concentrations observed here.

CAS 26780-96-1 (Antioxidant TMQ) is used in combination with the p-phenylenediamine 6PPD (CAS 793-24-8) to form a complementary protection system for rubber. Antioxidant TMQ is typically added to rubber at a ratio of 0.7 – 2.0 % (by weight of the rubber) (Nocil, 2023; Zhedongauxiliary, 2018). It should also be noted that the exact composition of Antioxidant TMQ varies from supplier to supplier as it is a polymerized trimethyl quinoline. The main component is however 2,2,4-trimethyl-1,2-dihydroquinoline as analysed. The results for Antioxidant TMQ (CAS 26780-96-1) should therefore be considered as conservative, and indicative of the substance at or near the surface of particles only.

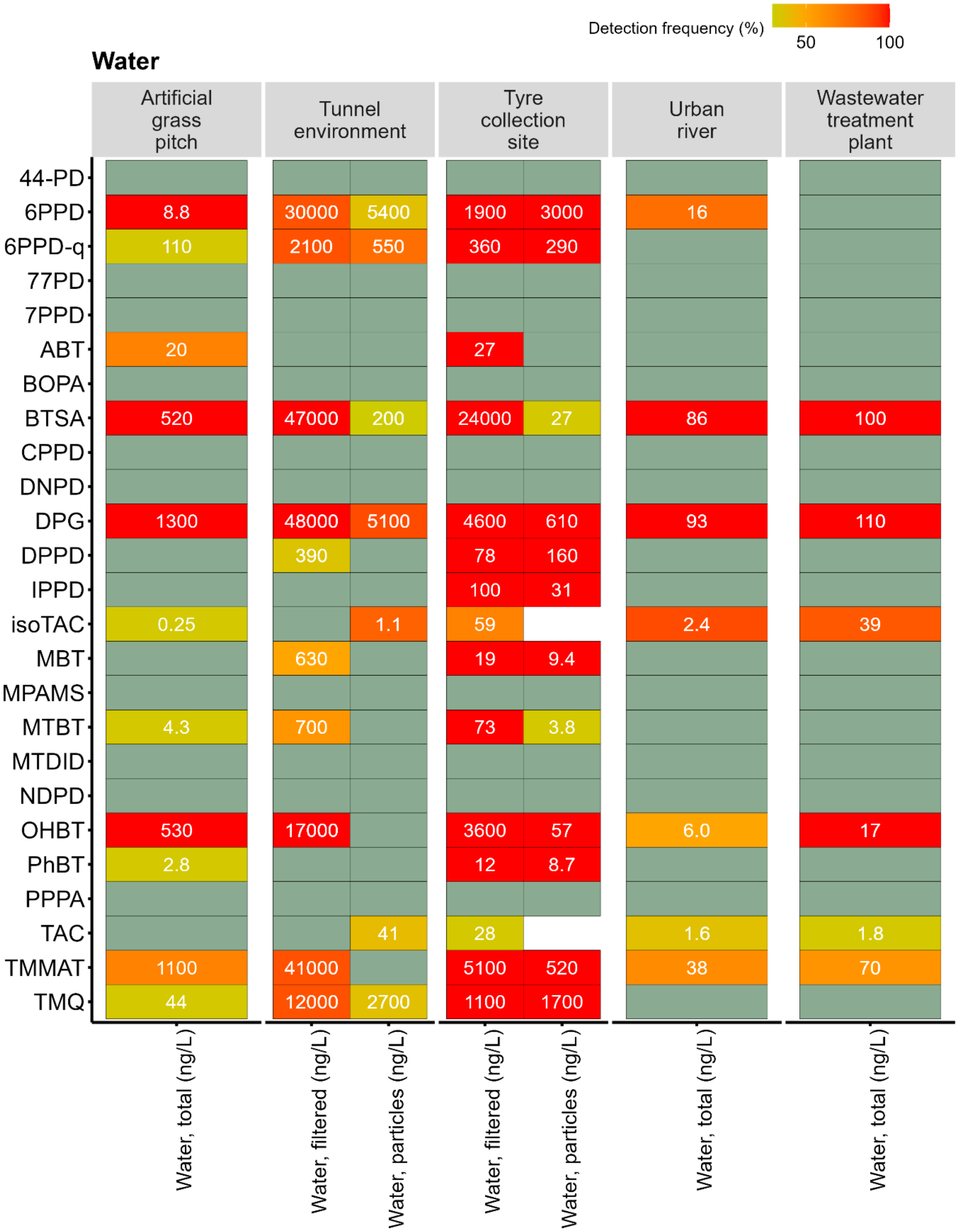
CAS 74-31-7 (DPPD) and CAS 101-72-4 (IPPD) are antioxidants and antiozonates with similar functional use to 6PPD. Their recommended dosage levels in rubber are approximately 0.2-1.5% by weight of polymer.

CAS 3089-11-0 (TMMAT) is a crosslinking agent designed to improve solidity, elasticity, and impermeability to gases. This class of rubber related substances can improve a rubber's resistance to chemicals, heat and abrasion. This additive is typically formulated to 0.5 – 5% by weight of polymer.

CAS 102-06-7 (1,3-Diphenylguanidine) is a secondary vulcanization accelerator that activates thiazoles (such as MTBT and MTBS which were also determined here), thiurams or sulphonamides. It is used in natural rubber (formulated to 0.1-0.5 % by polymer weight), and in styrene-butadiene rubber (SBR) such as in car tyres (formulated to 0.15 - 0.75% by polymer weight).

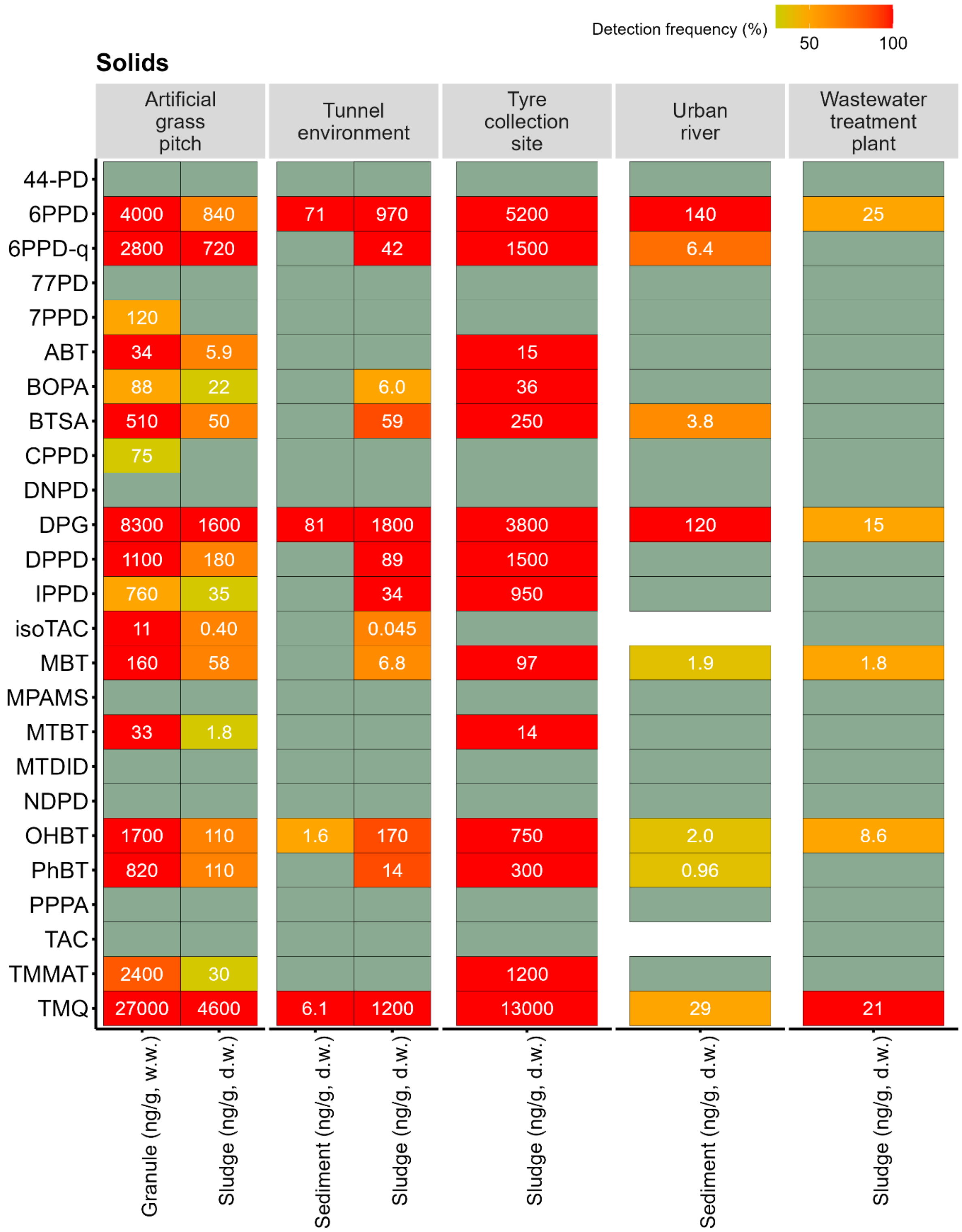
CAS 941-57-1 (BTSA) and CAS 934-34-9 (OHBT) are intermediates formed during biodegradation of MBT (CAS 149-30-4) in industrial and municipal treatment plants and in the environment (EU Risk assessment, 2008). MBT is a vulcanization accelerator with typical dosage rate as primary accelerator for rubber of 1.0-2.0% relative to polymer weight.

CAS 1025-15-6 (Triallyl isocyanurate (isoTAC)) and its isomeric derivative TAC is used as a vulcanisation and crosslinking agents (Lanxess). From the 2020 Screening Programme high concentrations of these substance were identified in tunnel wash, surface water, and sewage effluent (Schlabach et al., 2021). In the current study relatively low concentrations of isoTAC were found in samples of water from all sites sampled, granule and sludge from artificial grass pitch and tunnel environment, and in air samples from commercial and private buildings. TAC was found in water from all sites except artificial grass pitch.



	Artificial grass pitch	Tunnel environment		Tyre collection site		Urban river	Wastewater treatment plant
	Water, total (ng/L)	Water, filtered (ng/L)	Water, particles (ng/L)	Water, filtered (ng/L)	Water, particles (ng/L)	Water, total (ng/L)	Water, total (ng/L)
44-PD							
6PPD	8.8	30000	5400	1900	3000	16	
6PPD-q	110	2100	550	360	290		
77PD							
7PPD							
ABT	20			27			
BOPA							
BTSA	520	47000	200	24000	27	86	100
CPPD							
DNPD							
DPG	1300	48000	5100	4600	610	93	110
DPPD		390		78	160		
IPPD				100	31		
isoTAC	0.25		1.1	59		2.4	39
MBT		630		19	9.4		
MPAMS							
MTBT	4.3	700		73	3.8		
MTDID							
NDPD							
OHBT	530	17000		3600	57	6.0	17
PhBT	2.8			12	8.7		
PPPA							
TAC			41	28		1.6	1.8
TMMAT	1100	41000		5100	520	38	70
TMQ	44	12000	2700	1100	1700		

Figure 33: Average concentration in water of rubber related with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.



	Artificial grass pitch		Tunnel environment		Tyre collection site	Urban river	Wastewater treatment plant
	Granule (ng/g, w.w.)	Sludge (ng/g, d.w.)	Sediment (ng/g, d.w.)	Sludge (ng/g, d.w.)	Sludge (ng/g, d.w.)	Sediment (ng/g, d.w.)	Sludge (ng/g, d.w.)
44-PD							
6PPD	4000	840	71	970	5200	140	25
6PPD-q	2800	720		42	1500	6.4	
77PD							
7PPD	120						
ABT	34	5.9			15		
BOPA	88	22		6.0	36		
BTSA	510	50		59	250	3.8	
CPPD	75						
DNPD							
DPG	8300	1600	81	1800	3800	120	15
DPPD	1100	180		89	1500		
IPPD	760	35		34	950		
isoTAC	11	0.40		0.045			
MBT	160	58		6.8	97	1.9	1.8
MPAMS							
MTBT	33	1.8			14		
MTDID							
NDPD							
OHBT	1700	110	1.6	170	750	2.0	8.6
PhBT	820	110		14	300	0.96	
PPPA							
TAC							
TMMAT	2400	30			1200		
TMQ	27000	4600	6.1	1200	13000	29	21

Figure 34: Average concentration in solids of rubber related with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

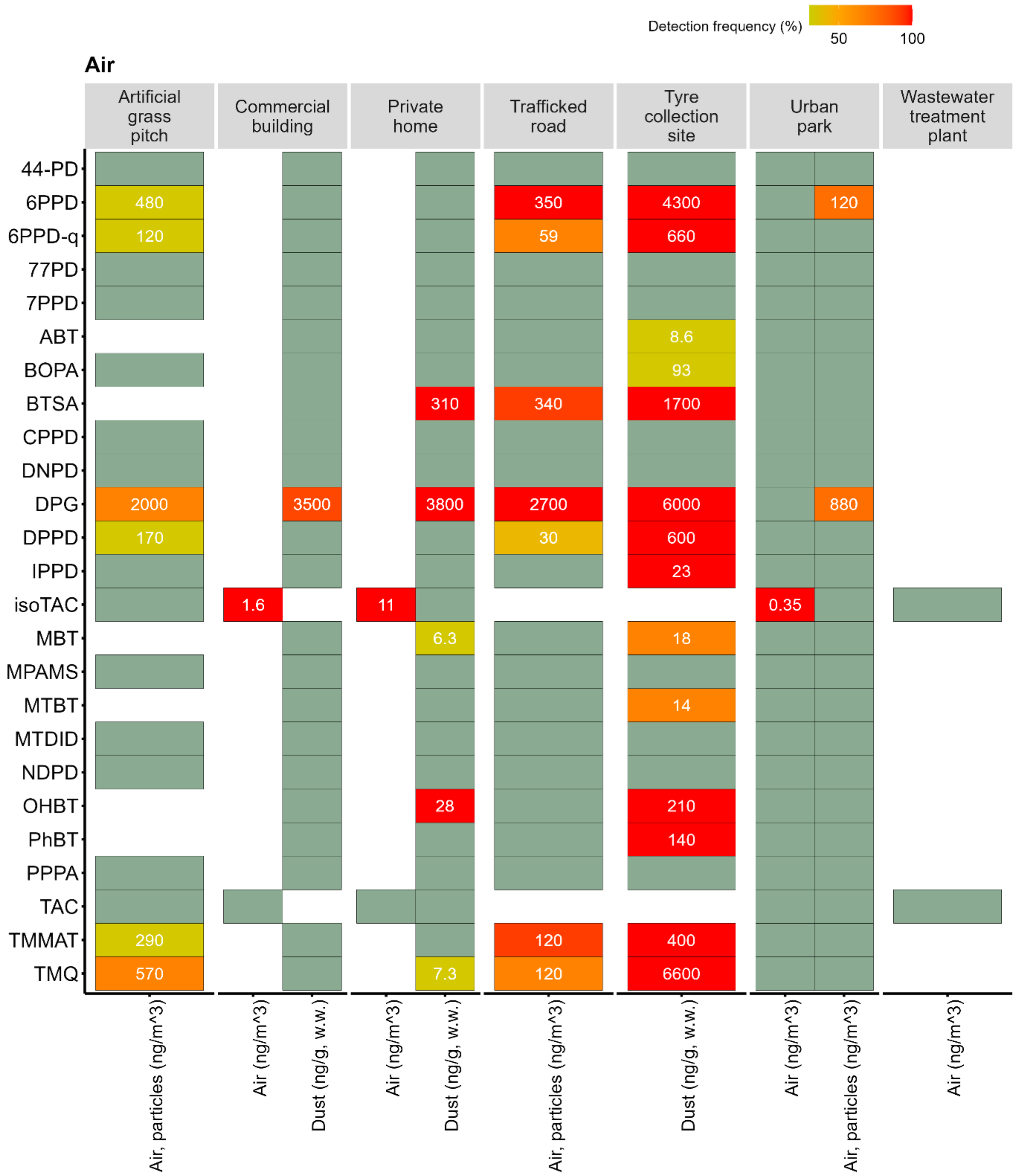


Figure 35: Average concentration in air of rubber related with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

Table 16: Predicted no-effect concentrations (PNEC) for rubber related. Red cells indicate that average measured concentrations in samples exceeded the PNEC.

Short name	CAS	Freshwater (ng/L)	Sediment (ng/g)	Biota (fish) (ng/g)	Data Source	Assessment factor
BTSA	941-57-1	10 000	33	53	Freshwater, Sediment, Biota: NORMAN	1000
OHBT	934-34-9	14 000	323	63	Freshwater, Sediment, Biota: NORMAN	n.a.
6PPD	793-24-8	370	110	27¤	Freshwater, Sediment: ECHA Biota: NORMAN	10 1000
6-PPD-q	6PPD-Q~	0.095¤ ‡	0.011	0.002	¤LC50 (acute) in Coho salmon (Tian et al., 2022), remaining derived from freshwater by factors below §	
DPPD	74-31-7	1 000	36	38	Freshwater, Sediment: ECHA Biota: NORMAN	1 000
IPPD	101-72-4	280	1.0	3.8¤	Freshwater, Sediment: ECHA ¤Biota: NORMAN	100 1 000
TMQ	26780-96-1	56 000	21 000	6 000	Freshwater, Sediment: ECHA ¤Biota, derived from freshwater by factor below **§**	1 000
TMMAT	3089-11-0	17	0.25	0.06	Freshwater: NORMAN Sediment, Biota: derived from freshwater by factors below **§**	1 000
DPG	102-06-7	30 000	2 500	21¤	Freshwater, Sediment: ECHA ¤ Biota: derived from freshwater by factor below **§**	10
isoTAC	1025-15-6	100 000	3 000	17	Freshwater, Sediment: ECHA Biota: NORMAN	1 000 1 000
TAC	101-37-1	7 000	173	106	Freshwater, Sediment: ECHA Biota: NORMAN	1 000 1 000

~ No CAS number has been assigned to this biproduct.

§ Other Assessment Factors:

1. Sediments – Lowest (P)NECfw*2.6*(0.615+0.019*Koc)
2. Biota (Fish) – (P)NECfw*BCF

‡ Freshwater – LC50 (biota)/ 1000 (Zhang, H.-Y. et al., 2023)

3.1.6 Siloxanes

The group of siloxanes included in this study consisted of the linear siloxanes L6-L10. Due to the lack of available analytical standards, substances L9-L10 were analysed by suspect screening only, and so the results for these are semi-quantitative. All siloxanes were detected in samples of freshwater, sediment, biota, air, and dust (Figure 36 and 37). The siloxanes were quantified in samples of wastewater and sewage-sludge, as well as recipient biota, at concentrations exceeding their PNEC values (Table 17).

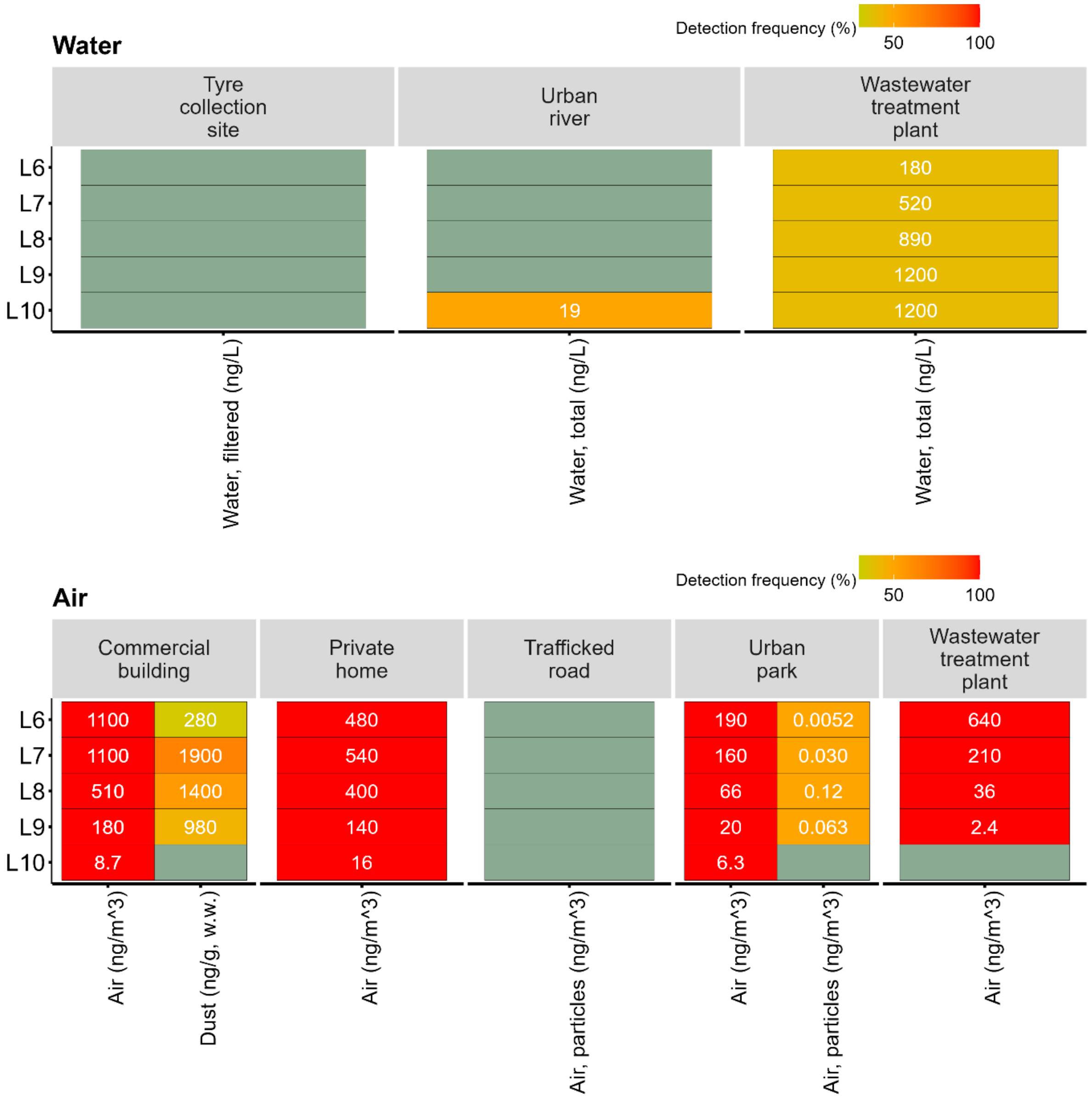


Figure 36: Average concentration in water and air of siloxanes with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%. Substances marked with "S" were a subject of a suspect screening without concentrations being presented.

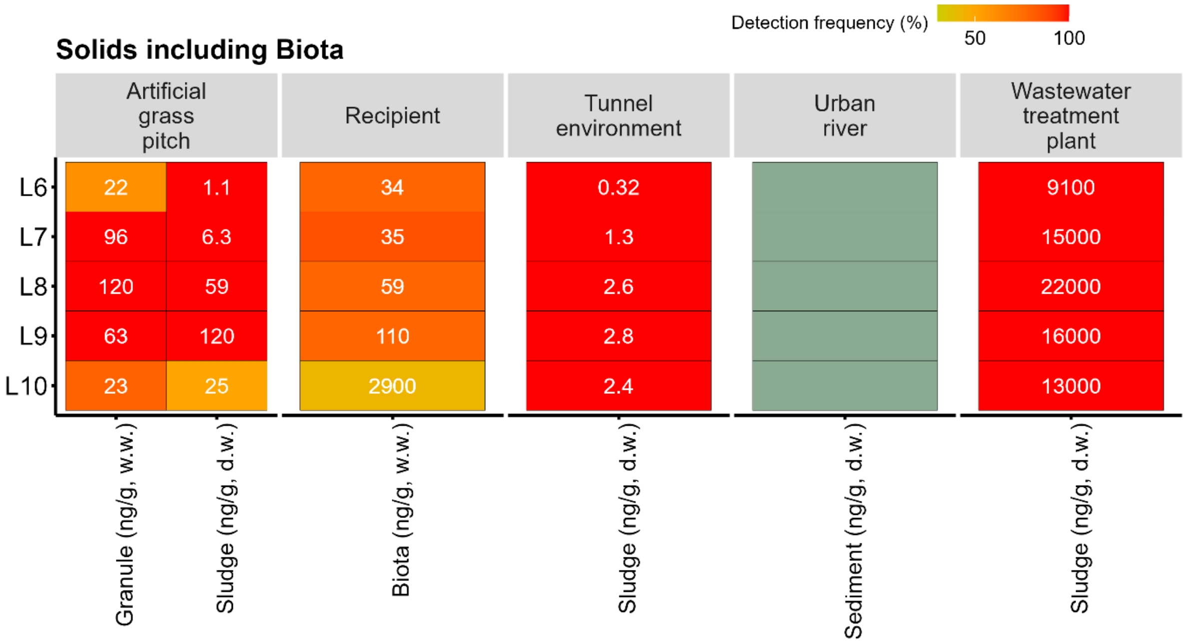


Figure 37: Average concentration in solids including biota of siloxanes with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%. Substances marked with "S" were a subject of a suspect screening without concentrations being presented.

Table 17: Predicted no-effect concentrations (PNEC) for siloxanes. Red cells indicate that average measured concentrations in samples exceeded the PNEC.

Short name	CAS	Freshwater, ng/L	Sediment, ng/g	Biota (fish), ng/g	Data Source	Assessment factor
L6	107-52-8	630	1 304	258	Freshwater, Sediment, Biota: NORMAN	1 000
L7	541-01-5	300	1 262	3.81	Freshwater, Sediment, Biota: NORMAN	1 000
L8	556-69-4	180	587	2.28	Freshwater, Sediment, Biota: NORMAN	1 000

3.1.7 Surfactants including PFAS

None of the PFAS substances included herein were detected above their methods LODs (Figure 38 and 39 and Appendix Table A5). However, the substance OP (CAS 2687-94-7) was found in multiple samples covering air from busy roads and an urban park, in sludge from the tyre collection site, in tunnel environment, and in a limited number of samples from artificial grass pitch. None of the measured concentrations exceeded the PNEC for this substance (Table 18). OP enhances clarity and transparency in products. It has a wide range of use, including vinyl flooring, carpet backing, roofing membrane, coated textiles, cable sheathing, refrigerator as well as window gaskets, hoses, pond liners, tarpaulins, sheets, bathmats, shower curtains, in artificial leather, shoe soles and automotive underbody coatings. OP is also registered for use as a surfactant (Surfadone LP-100 among others) which includes use as a degreaser in dishwashing detergents, automotive degreasers, and textile treatments (SpecialChem, 2023).

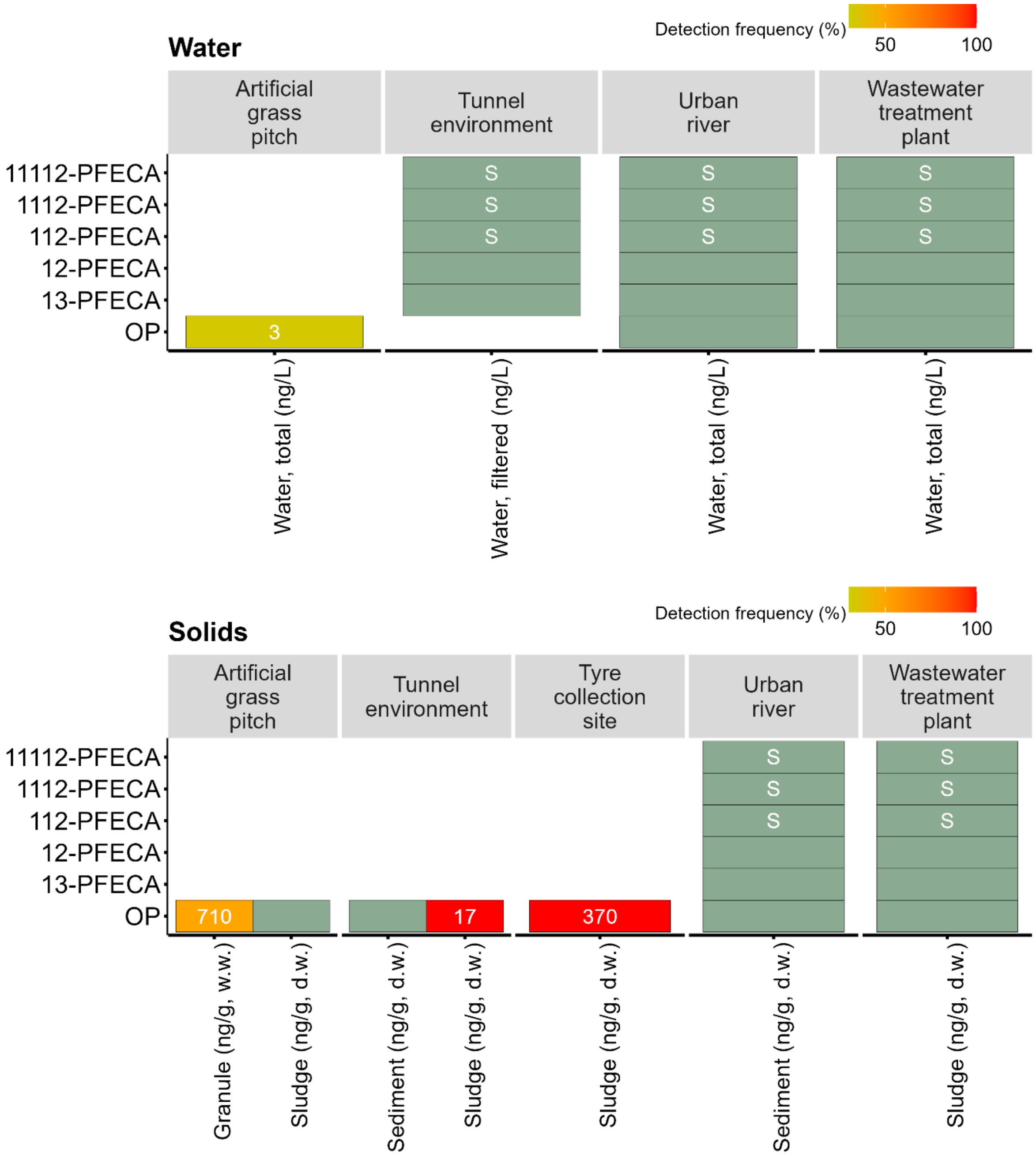


Figure 38: Average concentration in water and solids of the surfactants with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%. Substances marked with "S" were a subject of a suspect screening without concentrations being presented.

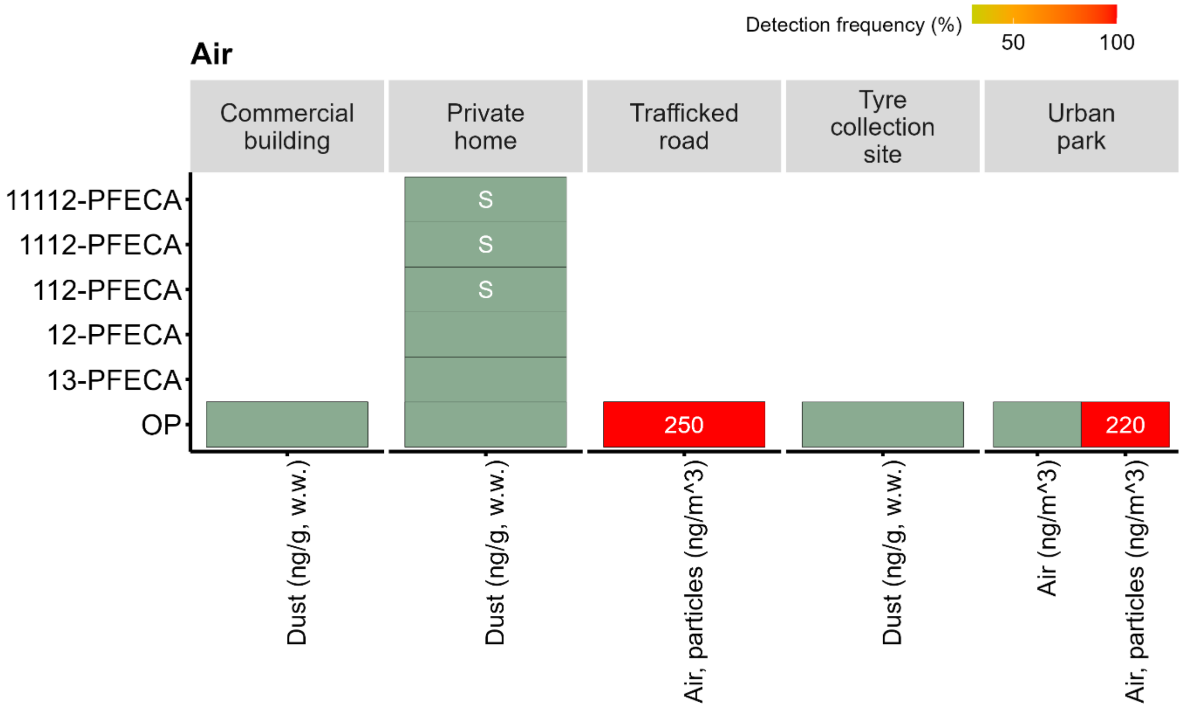


Figure 39: Average concentration in air of the surfactants with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%. Substances marked with "S" were a subject of a suspect screening without concentrations being presented.

Table 18: Predicted no-effect concentrations (PNEC) for surfactant OP. None of the average measured concentrations in samples exceeded the PNEC.

Short name	**CAS**	**Freshwater, ng/L**	**Sediment, ng/g**	**Biota (fish), ng/g**	**Data Source**	**Assessment factor**
OP	2687-94-7	91 000	3140	45.9	Freshwater, Sediment: ECHA Biota: NORMAN	10 1 000

3.1.8 UV-additives

Two substances of UV-additives were found in different types of samples (Figure 40). CAS 1137-42-4 (4-Hydroxybenzophenone, 4-HBP) is used as an industrial UV-filter and used as an intermediate in the manufacture of the medication Clomifene. It was detected in samples from all sampling sites except for those from an urban river. CAS 131-53-3 (benzophenone 8, BP-8) was detected less frequently and at lower levels. Neither substance was detected at concentrations exceeding PNECs (Table 19).

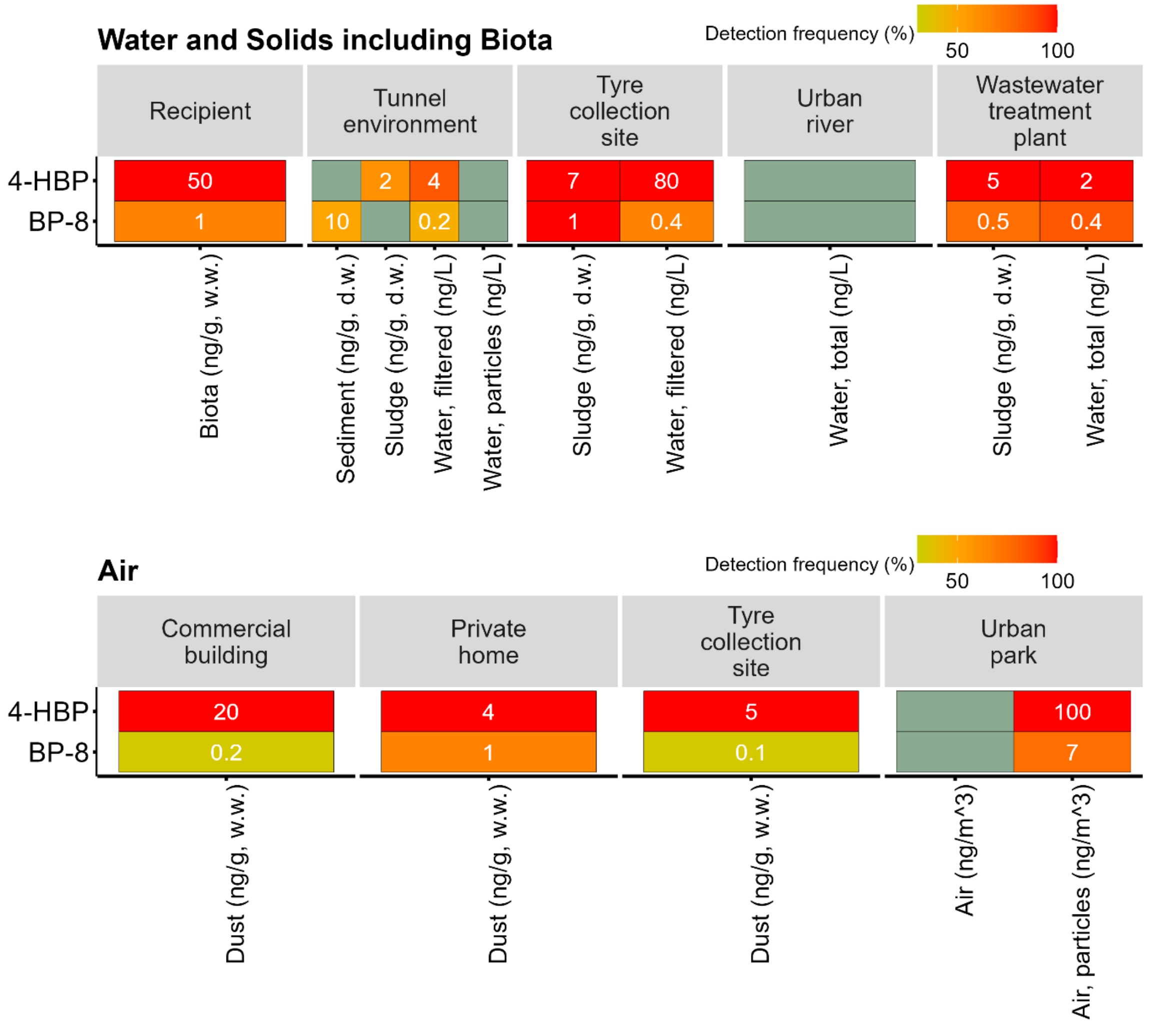


Figure 40: Average concentration in water and solids including biota and in air of UV-additives with detection frequency > 30% (in at least two different types of sample matrices and/or sampling site). Observations below the method LOD is included in the averages as 0.5x the LOD value. Colour indicates detection frequency, and with green ≤ 30%.

Table 19: Predicted no-effect concentrations (PNEC) for UV-additives. Red cells indicate that average measured concentrations in samples exceeded the PNEC.

Short name	**CAS**	**Freshwater, ng/L**	**Sediment, ng/g**	**Biota (fish), ng/g**	**Data Source**	**Assessment Factor (NORMAN)**
BP-8	131-53-3	860	18.8	55.1	Freshwater, Sediment, Biota: NORMAN	1 000
4-HBP	1137-42-4	11 000	172	79.4	Freshwater: ECHA Sediment: ECHA Biota: NORMAN	1 000 Partitioning* 1 000

*a different method from assessment factor has been used.

3.2 Site specific contamination patterns

Here follows some highlights on site-specific contamination patterns looking at all substance groups together. The aim is to identify potential sources of release to the environment, and to describe levels found in various environmental compartments. Levels in different sample types are compared and the effect from various parameters explored. Note that the number of samples from each site is generally limited, and so there is not a foundation for stating exact differences between samples with a high degree of certainty. Results for substances analysed “suspect” have not been included here (n=5).

3.2.1 Artificial grass pitch - substances from granule

Figure 41 displays average concentrations measured in the various matrices from artificial grass pitches. Interestingly, the “fingerprint” of substances found in the granulates is more or less directly reflected in the different samples collected from the sites. The sample type where the different types of compounds are found is largely determined by the physiochemical properties of the compounds. For example, LCCP is determined in the granulate and in water samples, but not at significant level in samples of sludge and air. Contrary, the high levels of TMQ found in the granulates is reflected in the sludge samples while not at the same extent in samples of water and air. This indicates that the antioxidant TMQ is effectively retained in sludge.

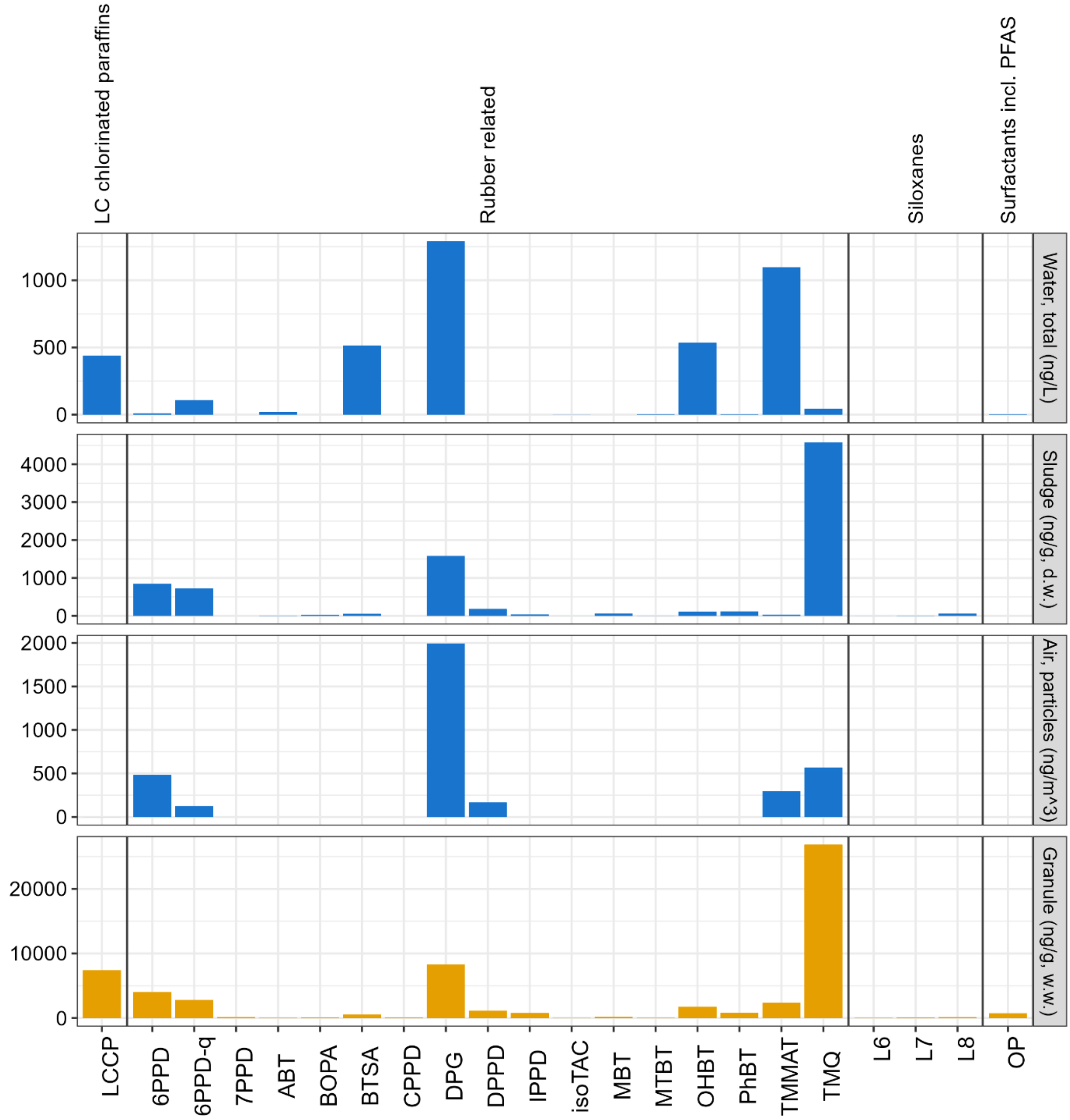


Figure 41: Average concentrations measured in various sample types from artificial grass pitches. Colour yellow indicate source material and blue are environmental matrices. LC is short for Long-chain.

3.2.2 Tunnel wash – will substances reach the nearby pond?

From the tunnel environment, one interesting question is whether the substances are efficiently retained by the sedimentation basin, and if it is possible to find the same substances in the pond downstream of a tunnel discharge. Figure 42 shows the average levels of the substances in sludge from the sedimentation basin and in sediment from the downstream pond, Smestaddammen. Note that the sedimentation basin is from the Vålerenga tunnel while the pond is located downstream of the Smestad tunnel. Despite the different tunnels, clear similarities can be seen in the types of substances found in the sedimentation basin and in the pond. This could indicate that the sedimentation basin does not have sufficient removal rates to perform a complete purification of the discharged tunnel wash water. Note that runoff from nearby roads is also a likely contributing source. At the Smestad tunnel, an additional purification step with a rain bed is applied after the sedimentation basin. The effect from such rain beds has not been thoroughly investigated.

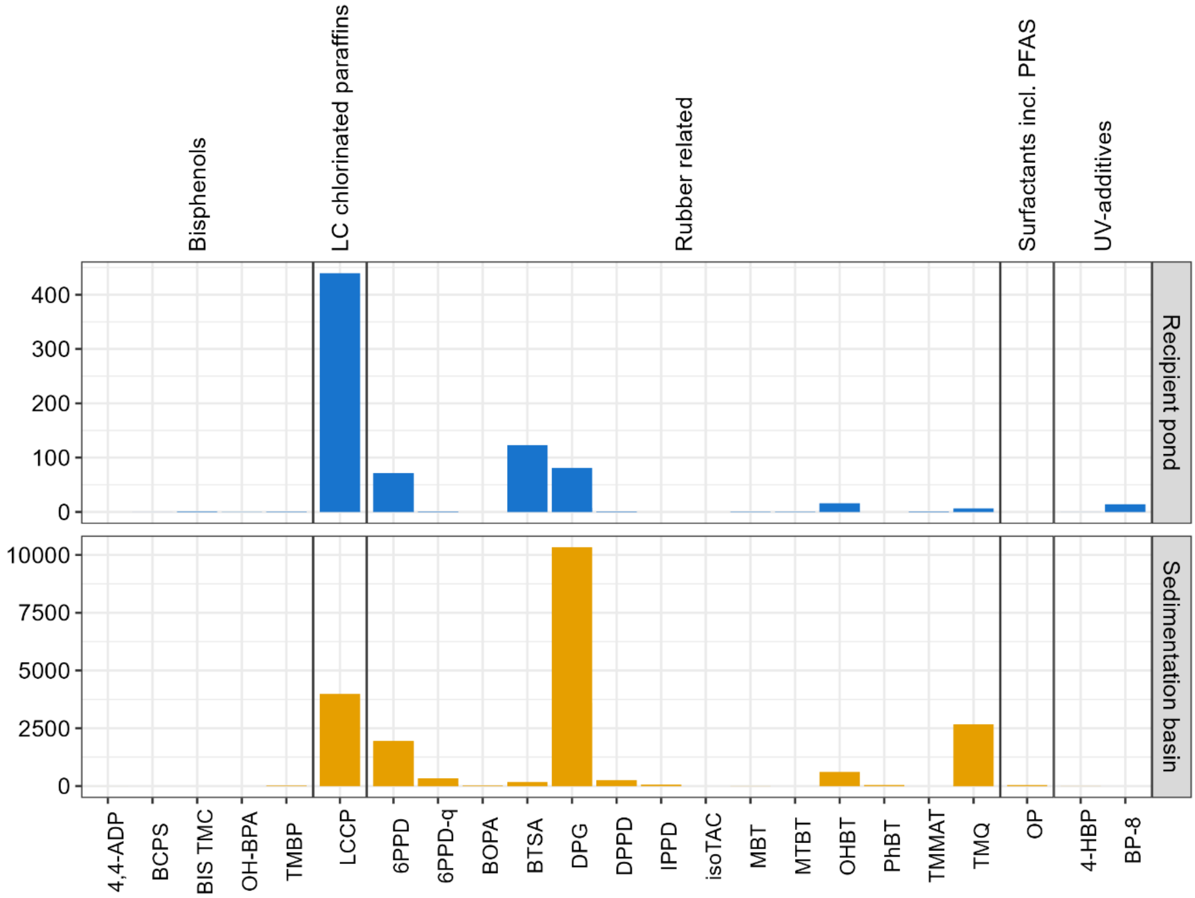


Figure 42: Average concentrations (ng/g, d.w.) in samples of sediment and sludge from the recipient pond (Smestaddammen) and the tunnel sedimentation basin, respectively. Colour yellow indicate source material and blue are environmental matrices. LC is short for Long-chain.

3.2.1 Outdoor air – trafficked road and urban air

While there were similarities in the range of substances determined in air- particles from busy roads and the urban park, there were also differences (Figure 43). Several of the car tyre substances were found at both sites (e.g., 6PPD, DPG, and OP) although with a lower concentration in samples from the park. However, at the park, some substances, including bisphenols, were detected and these were not seen in the samples from trafficked sites. Note that UV-additives were only determined in samples from the urban park. Overall, there were no clear trends in the levels between winter and summer (Figure 37). Interestingly, levels of the rubber related substance, DPG was much higher during summer than winter, both at the trafficked roads and the urban park. One hypothesis for this is that it is the biodegradation product of MBT and the higher level during summer is explained by the overall higher biological activity during this season (Foscari et al., 2023). In addition, this can be related to a different chemical composition of winter and summer tyres. Levels were generally lower during weekends than weekdays (data not shown).

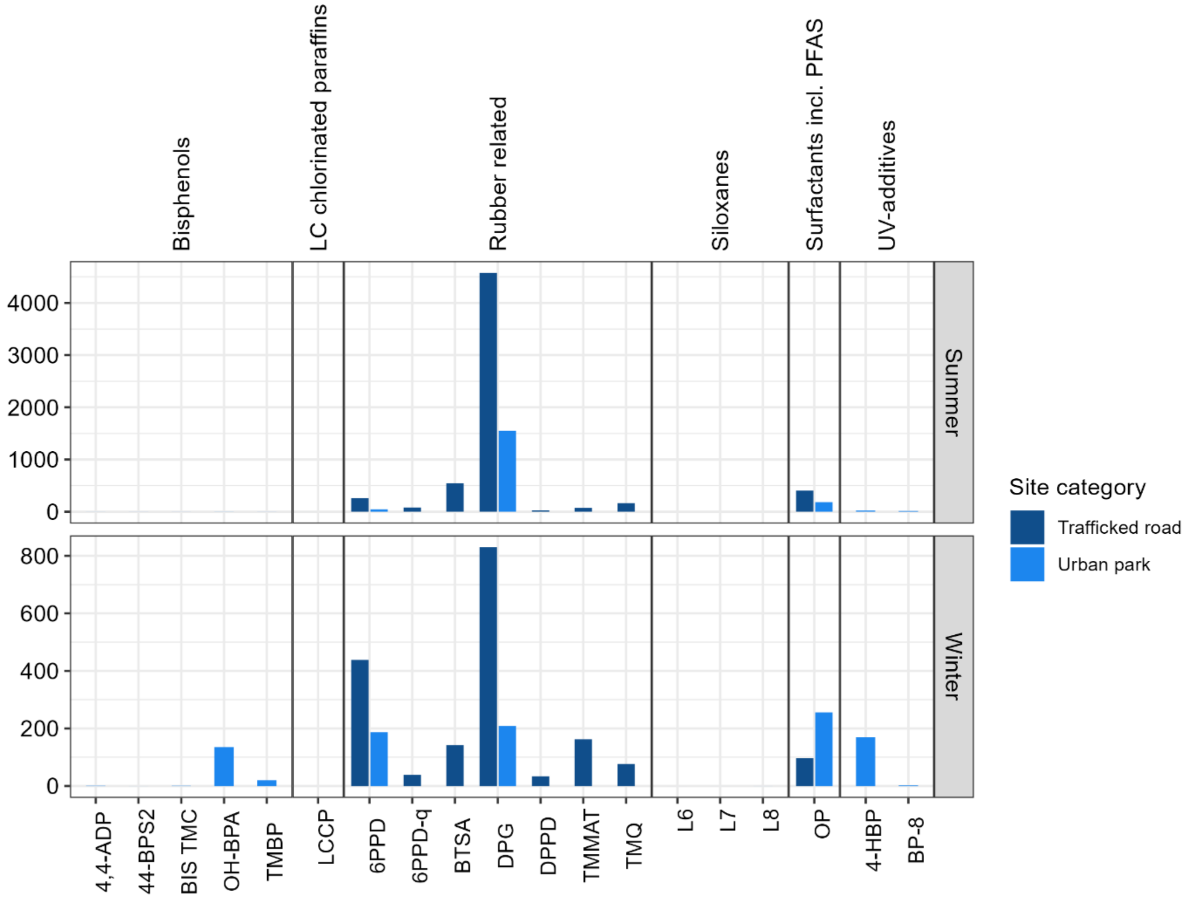


Figure 43: Average concentrations measured in air, particles (ng/m^3) from trafficked roads (dark blue) and the reference urban park site (light blue) during summer and winter. Note the different y-axis ranges.

3.2.2 Effect from high and low wastewater treatment

The pattern and distribution of substances detected at the municipal wastewater treatment plant were strongly linked to their physiochemical properties. More polar substances are generally found in water, while lipophilic substances and polymers are retained to a greater degree in treated sludge.

The measured concentrations of amitriptyline and BTSA increased in higher grade wastewater treatment compared to the lower level of treatment. See Figure 44. OHBT was found at equivalent concentrations irrespective of treatment. Most of the dose of amitriptyline is excreted in human urine as glucuronide or sulphate conjugates. Enzyme activity in the biological treatment step is expected to remove the conjugate thereby back transforming the parent pharmaceutical. This phenomenon of back-transforming metabolites of pharmaceuticals is often seen in wastewater treatment plants. BTSA and OHBT are intermediates formed during biodegradation of MBT (CAS 149-30-4). MBT was not detected in high treatment grade wastewater. Siloxanes (L6, L7, L8) were found in samples collected during summer, while in samples from autumn the levels were below the LODs. In the summer samples, high levels were found in the low grade treated water and with lower levels in the high-grade treated water. This indicates that the high degree purification is more efficient at removing these siloxanes from the water phase. The siloxanes are likely removed with the sludge, which was depicted by the higher concentrations in sludge from the high-degree treatment compared to the low degree of purification. This was further supported by simple mass-balance calculations (data not shown).

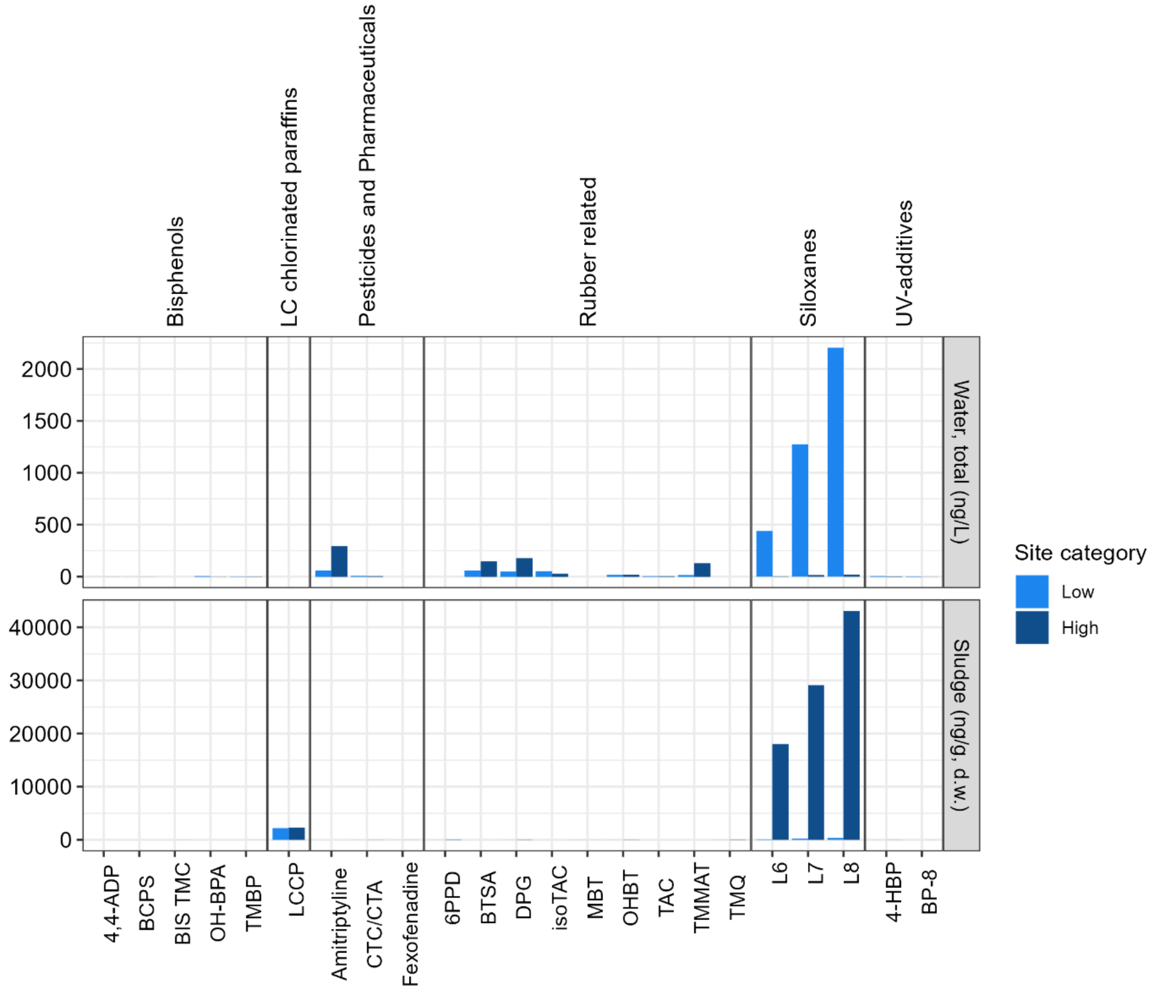


Figure 44: Average concentrations in samples of water (top) and sludge (bottom) from the wastewater treatment plant with low (light blue) and high (dark blue) treatment.

3.2.3 Tyre collection site – distribution to the nearby environment

In Figure 45, the type and level of substances measured in samples of dust and water from the site for collection and reuse of scrapped tyres are presented. At the site, both two sample types represent a pathway for direct release and emission to the nearby environment. The site was regularly flushed with seawater to avoid car tyre particles from being distributed with the wind to the surrounding neighbourhood, from which residents had previously complained.

The distribution of substances observed in the dust overlap with the expected formulation in car tyres. TMQ, DPG, and 6PPD are for example formulated in tyres at part per hundred levels relative to polymer. The non-soluble polymeric additive TMQ was observed in the water samples, which is not surprising given its high loading in the tyres. TMQ has been found in laboratory studies to be leachable (Stack et al., 2023). In water, it is however the relatively polar biodegradation product, BTSA that was observed with the highest concentrations. Moreover, concentrations were found to be higher in the filtrated samples than in the particles (Figure 33). This reflects the physiochemical properties of the substances. BTSA was classified as a potential leachable substance in the study by Müller et al. (2022), which was the study that also unequivocally classified DPG and 6PPD as leachable.

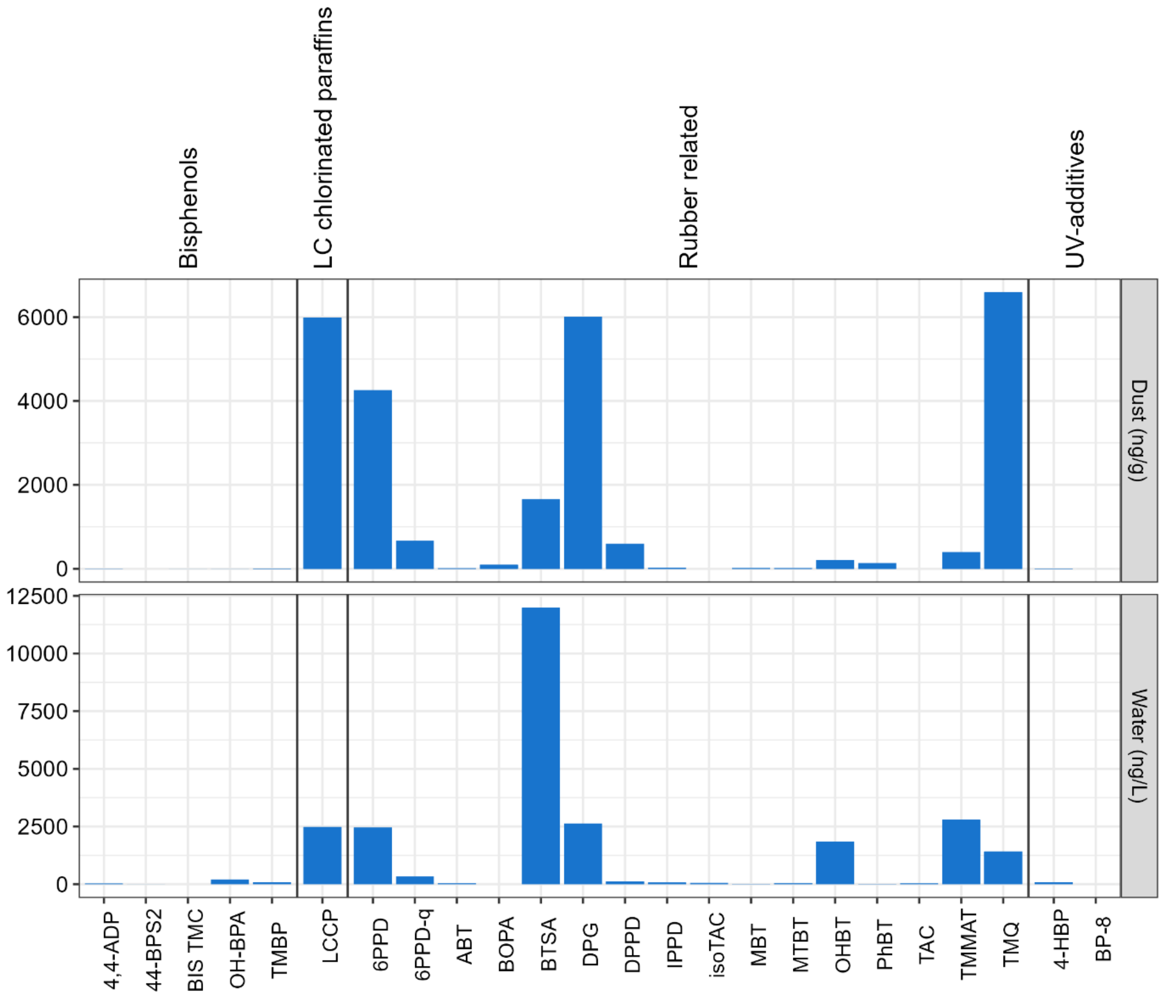


Figure 45: Average concentrations measured in dust and water from the site of care tyre collection and recycling.

3.2.4 Variations along an urban river

The urban river Alna flows through different regions of various anthropogenic activities, which can potentially contribute with a range of different substances. Not surprisingly, the lowest number and levels of substances was prevalent at the most upstream site, Kalbakken (Figure 46). Only the three widespread rubber related substances, BTSA, DPG, and TMMAT were found there at high levels. From the next downstream station Brubak, relatively high levels of the solvent, DCM were found. High levels of DCM were also found in the 2020 Screening Programme, though at even higher concentrations in river Alna. The concentration of DCM was similar at the two downstream stations, Breivoll and Kværner. This could indicate a source of DCM release somewhere between station Kalbakken and Brubak, or perhaps more likely from the upstream Fossumbekken. This stream has been associated with contaminated soils, partly originating from an old landfill located nearby. Station Breivoll is located downstream the industry-dominated site at Alna. However, this did not seem to influence the distribution and concentration of the relevant substances to a noteworthy extent. During the 2020 Screening Programme, high levels of the rubber related substances isoTAC and TAC were found in water from River Alna. The levels were comparable to those found in samples from tunnel wash. While in this year’s assessment, both isoTAC and TAC were detected in the Alna River water, the concentrations were thousands of times lower than what was found in the 2020 programme.

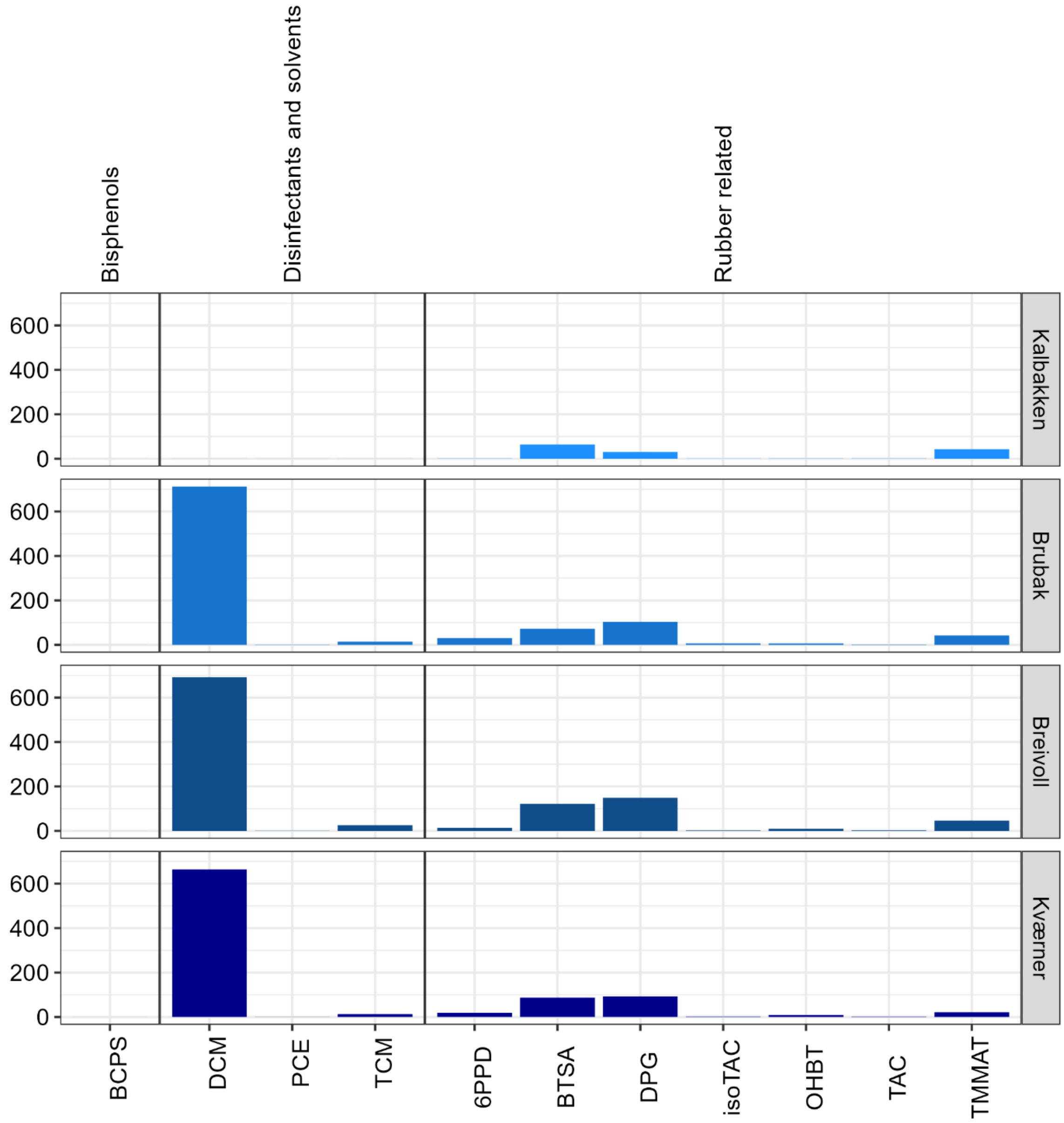


Figure 46: Average concentrations in water (ng/L) from the urban River Alna at the stations, from upstream to downstream, Kalbakken, Brubak, Breivoll, and Kværner.

3.2.5 Indoor air – commercial building versus private homes

Similar patterns in the type of substances detected were found in samples of indoor air and dust from commercial buildings (furniture stores, dental clinics, and 3D-printing facilities) and private homes (Figure 47). In samples of air, the rubber-related substance isoTAC was found in addition to the three siloxanes (L6, L7, L8). For the latter, a higher concentration was found in the commercial buildings compared to the private homes. In samples of dust, the most abundant substance was the long-chain chlorinated paraffines (LCCP), both in commercial buildings and private homes. Other findings in dust were a few rubber related substances as well as siloxanes.

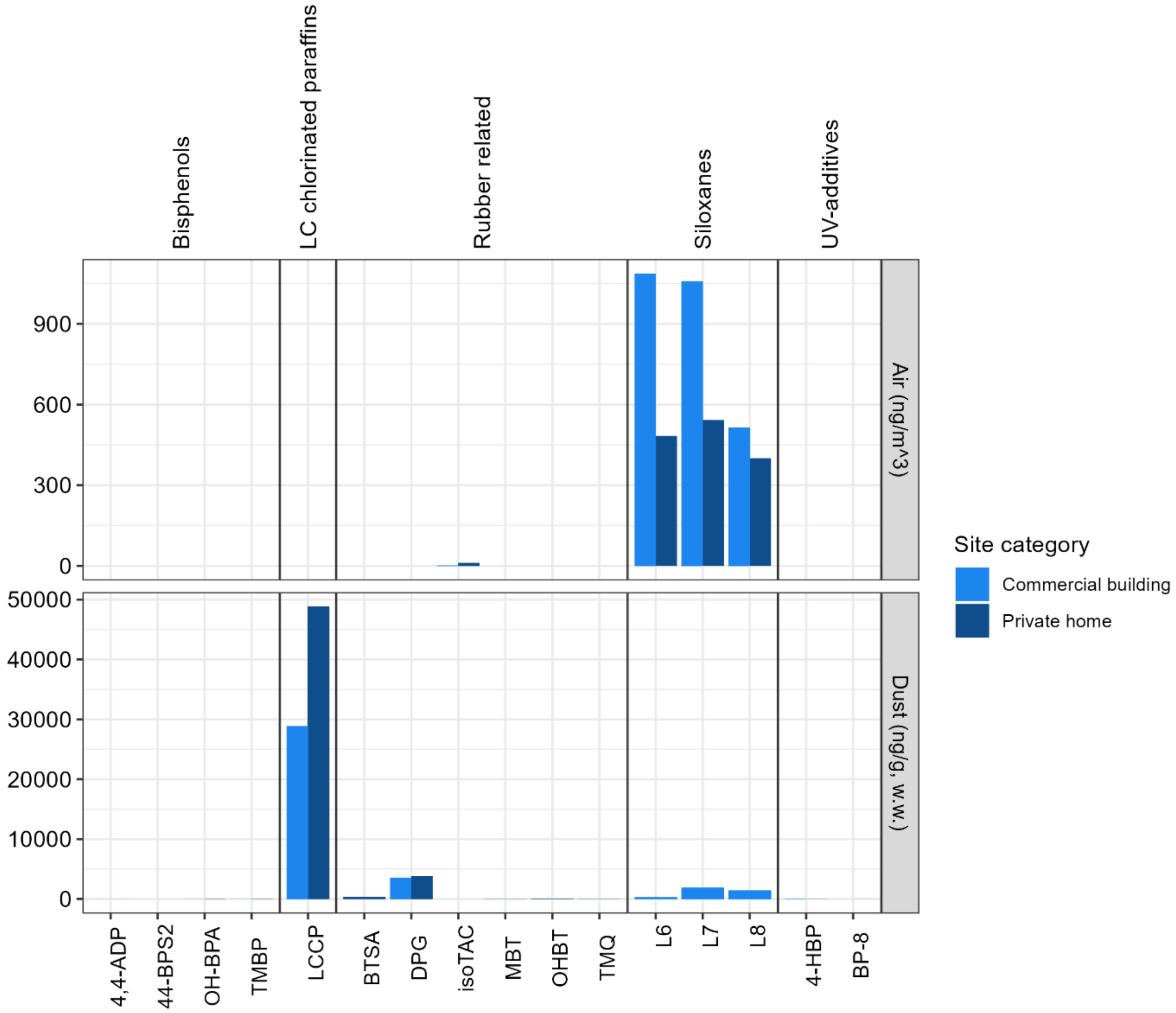


Figure 47: Average concentrations in samples of air and dust from indoor commercial buildings (light blue: furniture stores, dental clinics, and 3-D printing facilities) and private homes (dark blue).

3.2.6 Recipient biota

Several of the substances were found in different tissues of recipient biota (Figure 48). Long-chain chlorinated paraffins and siloxanes were distributed in both freshwater and marine species. In freshwater, additional substances of one bisphenol (OH-BPA) and the two UV-additives (4-HPB and BP-8) were present. While in the Oslo fjord, another bisphenol (BCPS) and one rubber related substance was found (isoTAC).

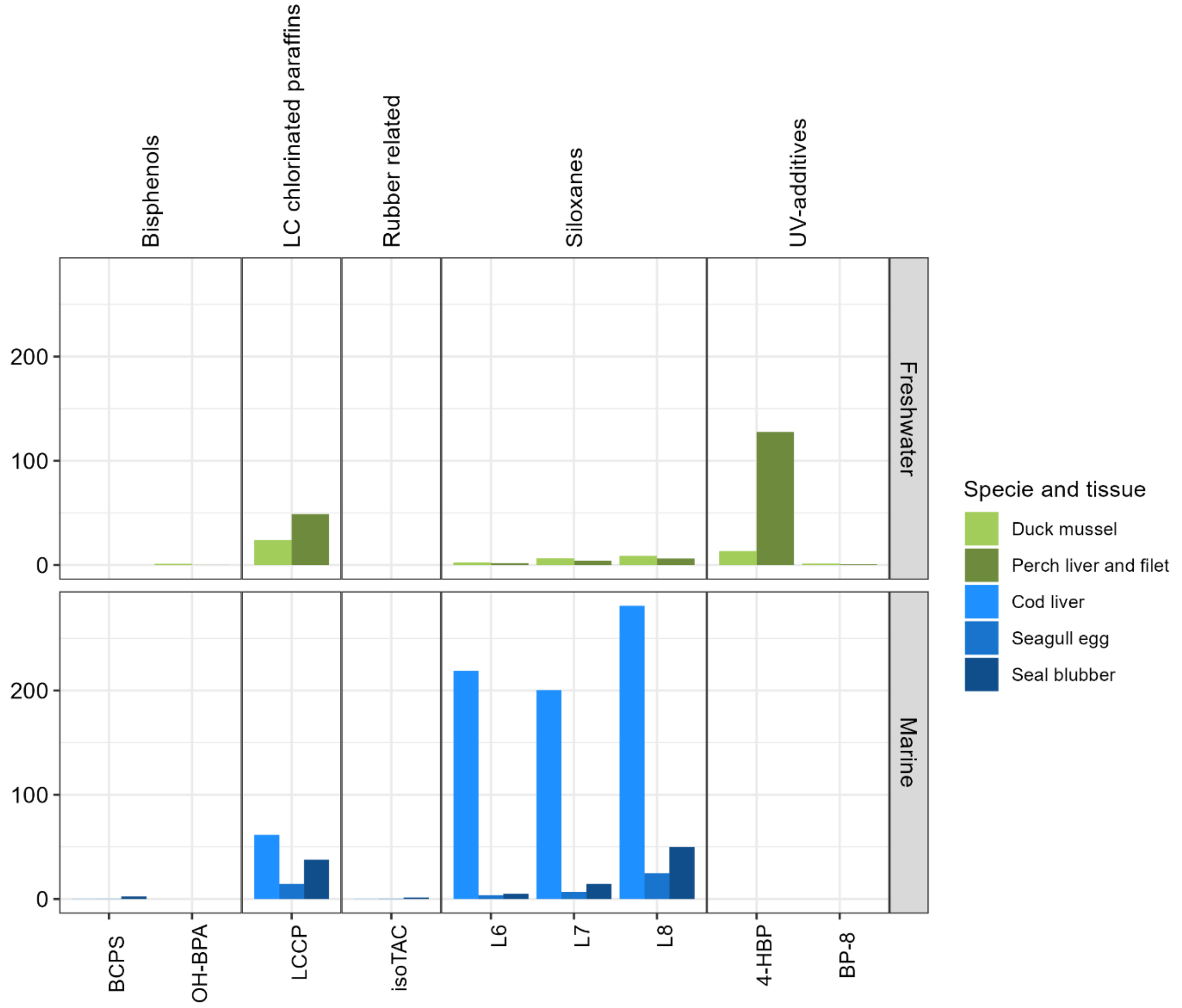


Figure 48: Average concentrations in various species and tissue of biota (ng/g, w.w.) from freshwater (top) and marine water (bottom). Freshwater samples from lake Padderudvannet and marine samples from Oslo fjord.

3.3 An overall evaluation of environmental risk

Figure 49 illustrates the lowest PNEC for freshwater, sediment, and biota together with the range of concentrations measured in this study (minimum to maximum for substances with detection frequency > 30%).

For several of the substances, levels of environmental concern (based on the lowest PNEC) have been found. This applies to several of the rubber additives, the pharmaceutical amitriptyline, the LCCP, and siloxanes. These results indicate the potential for environmental concern based on the available PNEC values and further studies on occurrence of these substances is recommended. It is important to recognise that several of the PNECs are associated with relatively high uncertainty and that this assessment should be used for prioritization only and cannot replace more detailed risk assessment.

No exceedance was observed for substances within the groups of bisphenols, surfactants including PFAS, and disinfectants and solvents.

Note that substances either not found above the method LOD or determined by “suspect screening” have not been included. Based on the analyses here it cannot be ruled out that those specific substances are not present in the environment at levels that pose a risk. For method concentration LODs, see Appendix A.3 Table A5.

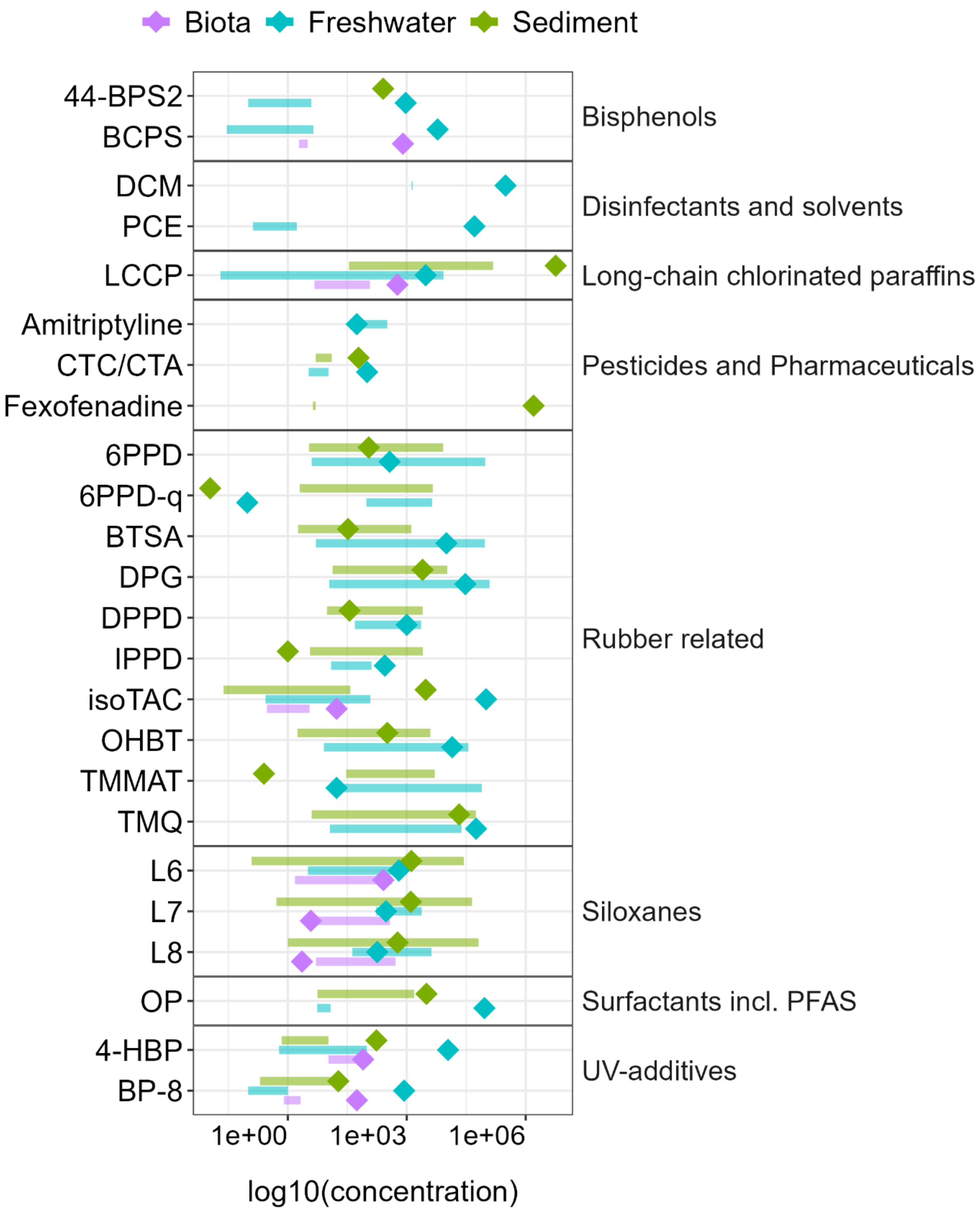


Figure 49: The lowest PNEC for the substances in biota (purple ◆), freshwater (blue ◆), and sediment (green ◆) together with the measured concentration range, minimum to maximum, in biota (purple ▬), freshwater (blue ▬), and sediment (green ▬) of the substances with detection frequency > 30%. Note that the concentrations are in $\log_{10}$ scale.

4 Conclusion

In this study 154 environmental samples were analysed for 59 different substances. Functional use of the substances and their physiochemical properties were critical factors guiding the design of the sampling campaign. Most of the substances are components in various products such as car-tyre rubber, plastics, dyes, and textiles. Priority was therefore given to sampling locations associated with car tyre material such as artificial grass pitches and heavily trafficked roads. Sample types included stormwater, particles in air and water, the environment at sedimentation basin, and possible absorption in biota.

High concentrations, exceeding respective PNECs, were observed for rubber additives in all samples associated with vehicle tyres. The highest concentrations were observed for CAS 941-57-1 (Benzothiazole-2-sulfonic acid, BTSA), CAS 934-34-9 (2-hydroxybenzothiazole, OHBT), CAS 793-24-8 (6PPD) and its ozone bi-product 6PPD-Quinone, CAS 74-31-7 (DPPD), CAS 101-72-4 (IPPD), CAS 26780-96-1 (Antioxidant TMQ), CAS 3089-11-0 (Hexamethoxy methyl melamine, TMMAT) and CAS 102-06-7 (1,3-Diphenylguanidine, DPG). Most of these substances are added polymer/resin during manufacture of rubber, at ratios of parts per hundred (%). The substances are integrated into the polymer particles so even very small amounts of solid, even as fine dust, can therefore show very high levels of these substances. However, several of these are also known to leach from the particles to the water phase.

The surfactant CAS 2687-94-7 (OP) is registered for use as an automotive degreaser, and it was also detected in samples associated with vehicle traffic and car-tyre recycling.

The siloxanes were detected frequently, and levels in wastewater and sewage-sludge were above PNEC values. Long-chain chlorinated paraffins (LCCP) were also detected very frequently, but at levels below the respective PNECs.

Of the pharmaceuticals and pesticides included in this study, only amitriptyline (CAS 549-18-8) was detected in all wastewater effluent at concentrations exceeding those shown to have an impact on aquatic organisms. Enzyme activity in the treatment plant also resulted in increased concentrations of the parent drug as the conjugation from human metabolites was transformed back during wastewater treatment.

The objectives of the 2022 Screening Programme were to:

- investigate whether the substances were found at hot spot locations
- investigate whether the substances were introduced to- and found in nature
- assess whether the levels found may cause environmental damage

Results from this study confirm that all three conditions were met. Elevated concentrations of pollutants were observed, particularly in areas associated with vehicle traffic or where car-tyres are recycled.

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A. Appendix - Methods

A.1 Methods for sampling

All sampling has been conducted with safety in focus, and in accordance with applicable laws, regulations, standards, and guidelines.

All equipment used for sampling were washed and sterilised prior to use. Sample containers were washed, and either burned at 550 °C, rinsed with solvent, or washed with acid solution before use according to the properties of the container and the contaminants. Special considerations were taken when collecting samples to be analysed for siloxanes.

A.1.1 Surface water

Water samples were collected either as *i)* grab samples; *ii)* using an automatic sampler; or *iii)* using the special sampler illustrated in Figure A1. The latter is a storm water sampler (Nalgene^{TM}) that can be mounted in a manhole to collect the first flush of a precipitation event. A drawback for this technique, as well as the use of an automatic sampler, is related to the substances with shorter lifetime which was the case in this study. Therefore, the use of grab samples was more widespread than originally planned for.

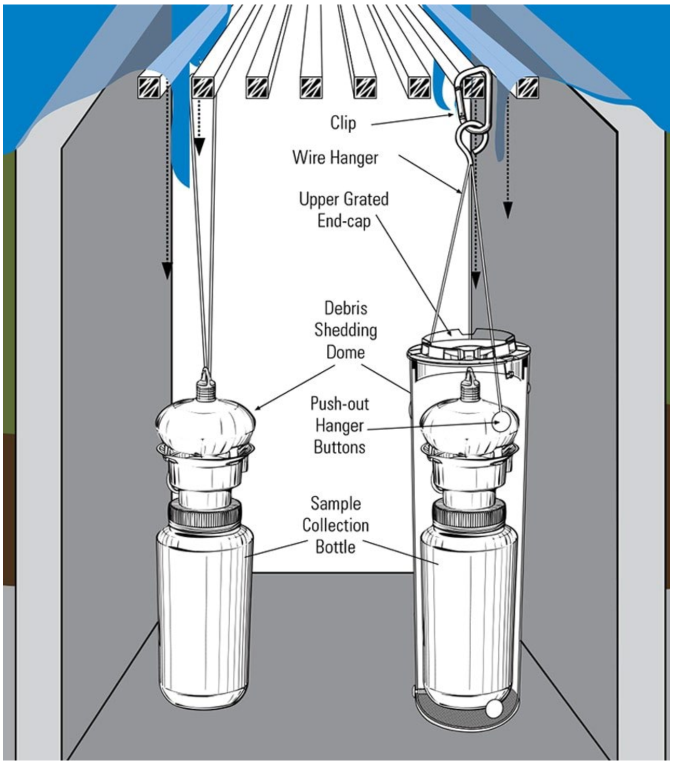

Figure A11: Illustration of the sampling equipment used to sample the first flush of surface water runoff during a high precipitation event. Nalgene^{TM} water sampler and mounting kit.

A.1.2 Sediments, sludge, and granule

The sludge samples were collected using a mini-grab sampler (Figure A.2). Subsequently, the sample is transferred onto aluminium trays from which sub-samples were collected using a metal spoon and pooled in a clean glass jar (pretreated in an oven at 480 °C) which was covered with aluminium foil and capped.

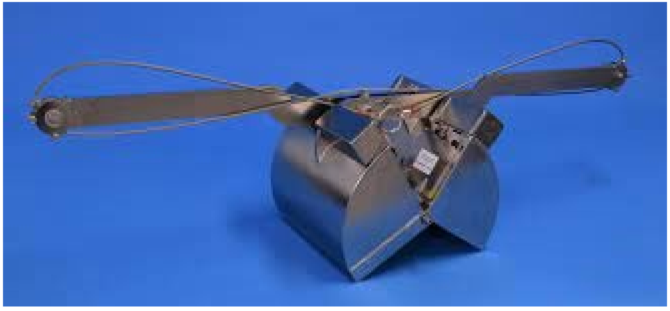

Figure A2: Photo of a generic van-Veen grab sampler.

A.1.3 Dust

The dust samples were collected on a cellulose filter using an industrial vacuum cleaner (Nilfisk GM 80P) equipped with a special forensic nozzle with a one-way filter housing (KTM AB, Bålsta, Sweden) which was placed in the front of the vacuum cleaner tube (Figure A3). After sampling a lid was put on the filter housing, and the whole sampling compartment was wrapped in double layers of alumina foil, placed in two sealed plastic bags and stored at -20̊ C until sample preparation.

Figure A3: Pictures of the industrial vacuum cleaner used for sampling dust. Photo: NILU.

A.1.4 Air

Air samples for VOCs were collected as grab samples in stainless steel canisters. Air samples for volatile to semi volatile chemicals were collected on ABN adsorbents using low-volume active air samples (flow rate of ~0.7 m^3/hr, sampling times ~24 hrs). Low volume air samplers were utilized in indoor football fields, placed at a height of 2-3 meters, approximately 3-4 meters back from the field of play. These samples were collected over a period of 48 hours each.

Figure A4: Photo of the equipment used to sample air. Photo: NILU.

A.2 Details of sampling

A.2.1 Urban River

Time of sampling and amount of rainfall 24 hours (and during sampling at Brubak during the first sampling campaign) at the four different sampling locations along Alna are shown in Table A1. The water flow was at each station acquired from different sources.

The water flow at Kalbakken was too low to measure properly but was estimated to be <<1 L/s. The water flow at Brubak was measured using an ISCO 2150 flow meter with the sensor mounted on a metal plate fixed to the river floor. However, during the last sampling period (12.-13.9) the river current was so strong that the plate loosened. The flow was then therefore estimated based on the measured velocity (m/s) and calculated transection (m^2) (estimated from the water depth and width of the river). See Table A1. The reported flow at Kværner in was provided by the Oslo Municipality, from their continuous measurements at their monitoring station at Kværner ("Kværnerristen"). Figure A2 shows the water flow measured at station Kværner during the two sampling. The water flow at Breivoll was assumed to be 75% of the flow at Kværner based on the relative size of the catchment area of the side stream Østensjøbekken, which enters Alna between Breivoll and Kværner, and the size of Alna's catchment above Breivoll.

Table A1. Location and rainfall 24 hours before and during sampling (only for composite sampling), type and time of sampling at the four locations along the river Alna.

Location		**Rainfall**		**Flow**	**Sampling**	**Time of sampling**
No.	**Station name**	**24 h before**	**During**	**L/s**		
1	Kalbakken	23 mm	-	<<1	Grab	
		1.3 mm	-	<<1	Grab	
2	Brubak	5 mm	17 mm	580	Time-comp.	Kl: 23:00-03:00
		0.6 mm	-	141	Grab	
3	Breivoll	25 mm	-	3 500	Grab	
		0.6 mm	-	1 130	Grab	
4	Kværner	25 mm	-	4 400	Grab	
		0.1 mm	-	1 510	Grab	

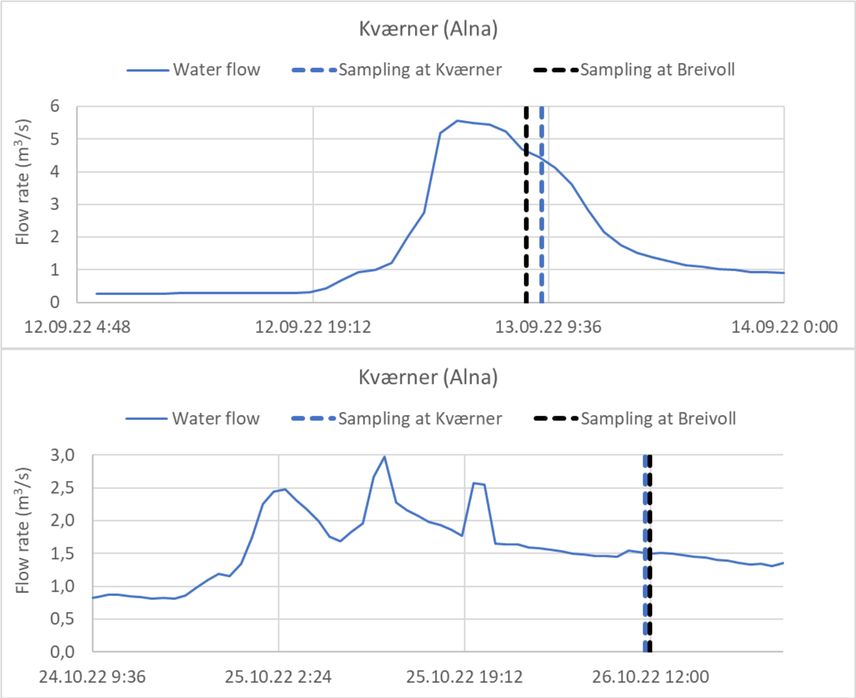

Figure A.5: Water flow measured at Kværner in the two periods when samples were collected.

A.2.2 Wastewater treatment plant

Here follows supporting data for the sampling at the wastewater treatment plant.

Table A3. Period for sampling of primary treated and final effluent at Bekkelaget WWTP. The samples were taken as time-proportional composite samples. The volume of wastewater (m^3) that was treated at the plant during each sampling campaign is also provided.

Sample No.	**Start** -	**End** -	**Treated volume** m^3
Primary treatment			
1	26.9.22 11:00	27.9.22 15:00	20 315
2	28.9.22 07:00	28.9.22 10:57	18 753
3	29.9.22 08:00	29.9.22 11:00	14 789
4	3.10.22 08:17	3.10.22 12:02	17 894
5	4.10.22 08:12	4.10.22 12:03	17 325
6	5.10.22 08:00	5.10.22 12:00	18 637
Advanced treatment			
7	26.9.22 11:00	27.9.22 15:00	21 059
8	28.9.22 07:00	28.9.22 10:57	19 626
9	29.9.22 08:00	29.9.22 11:00	15 083
10	3.10.22 08:17	3.10.22 12:02	18 400
11	4.10.22 08:12	4.10.22 12:03	16 819
12	5.10.22 08:00	5.10.22 12:00	17 967

Table A4. The period for sampling of final treated sewage sludge at Bekkelaget WWTP. The total amount (ton) of sludge that was produced during the period of sampling, as well as the dry matter content (TS) of the sludge is presented (plants' own measurements).

Start -	End -	# subsamples	Sludge production ton TS
Primary treatment			
27.9.22	29.9.22	12	61
3.10.22	5.10.22	12	64
Advanced treatment			
27.9.22	29.9.22	5	122
3.10.22	5.10.22	6	102

A.3 Details of analytical methods

VOC (NILU)

For this analysis, water was sampled in designated plastic bottles (200 mL, high density polyethylene). When sampling, care was taken to ensure there was no air left inside the bottles when sealed for shipment. In the lab, a screwcap with an inlet for synthetic air and an outlet with a Tenax sampling tube was attached on top of the sample bottle. Synthetic air was bubbled through the water to evacuate the VOCs. The air and the evacuated VOCs were then pushed out of the bottle, through the Tenax tube. The VOCs were retained on the Tenax material while the synthetic air was just passing through. A critical orifice was attached between the Tenax tube and the bottle to ensure a steady flow of 80 mL/min. This allowed for accurate control of the total amount of air passing through the Tenax tube.

After evacuating the VOCs from the water, the Tenax tubes were analysed using high resolution "Thermal Desorption Gas Chromatography Time-of-Flight Mass Spectrometry" (TD-GC-ToF-MS). A calibration curve for each of the individual target compounds was prepared simultaneously using the multicomponent solution VOC Mixture 230 from LGC Standards. The recorded signals were then processed using proprietary software (Unknowns Analysis) from Agilent and finally quantified in Microsoft Excel.

SVOC (NILU)

Before extraction, a mixture of internal standards was added. Biota, sediment, and sludge samples were homogenised and then extraction was carried out with mixture of acetonitrile and hexane. Hexane layer was separated and concentrated, if necessary. ABN cartridges (for air samples) were extracted with ethyl acetate and concentrated by evaporation in a gentle stream pf nitrogen. Granulate, particles (with filters) and dust samples were extracted with acetone. Extracts were concentrated, if necessary. Aqueous samples were extracted with a mixture of dichloromethane and ethyl acetate. Analysis is performed with full-scan GC-HRMS. The instrument used was Thermo Q-EXactive GC-Orbitrap-HRAM-MS operated at mass-resolution 120000 or 60000. Mass-range was adjusted to optimize sensitivity, depending on the sample type. Quantification was made with help of calibration solutions (authentic specimens available for all compounds).

Bisphenols, BIS (NILU)

Extraction of water samples

An aliquot was measured out of the preserved and filtered sample before addition of a mixture of isotopically labeled internal standards. The procedural blanks consisted of MilliQ-water treated as real samples. The samples were extracted using Affinisep® HLB disks and eluted with distilled Methanol. Extracts were concentrated with miVac to <0.5 mL and then either sent to further cleanup or evaporated to approx. 100 µL, filtrated using centrifugal filter and added recovery standards before LC-HRMS analysis.

Extraction of solid samples

Solid samples (soil, sludge, sediments, indoor dust, and air particles) were extracted using accelerated solvent extraction (ASE). An aliquot of the sample was weighed and added to a stainless teel cell together with a dispersant (diatomaceous earth or Ottawa sand) and added a mixture of isotopically labeled internal standards. Procedural blanks consisted of cells packed with dispersant spiked with internal standards and otherwise treated the same as samples. Each cell was extracted with acetone/hexane 1:1 and distilled methanol. After extraction some samples were further purified with SPE, that (together with cleaner samples) after addition of recovery standards were subjected to instrumental analyses.

The samples were analyzed using Thermo Scientific Q Exactive Plus Orbitrap high resolution mass spectrometry. Methanol and water were used as mobile phase. Separation was achieved in 20min.

LCCP (NILU)

Solid samples (soil, sludge, sediments, indoor dust, and air particles) were extracted using ASE. An aliquot of the sample was weighed and added to a stainless-steel cell together with a dispersant (diatomaceous earth or Ottawa sand) and added a mixture of isotopically labeled internal standards. Each cell was extracted with two cycles using acetone/hexane 1:1. The extracts were treated with sulfuric acid and cleaned with activated silica. Finally, extracts were concentrated and added recovery standards before instrumental analyses.

LCCPs were analyzed with Agilent UHPLC-HR-QTOF-MS operated in negative electrospray mode with acetonitrile and water mixed with TMAC (tetramethyl ammonium chloride).

SIL (NILU)

Before extraction, a mixture of internal standards was added. Sediment, sludge, water, ABN cartridges for air analysis, water and effluent from treatment plants and biota samples were extracted with organic solvents that ensure a good yield of the analytes. All concentrated extracts were analysed with help of a GC-HRMS (Q Exactive GC, Orbitrap) in full-scan mode at mass-resolution 120,000 or 60,000. Quantification of the three shortest siloxanes (L6-L8) was made with calibration solutions (authentic specimens available). For the two longest siloxanes, L9 and L10, (authentic samples not available), quantitation was done with an assumption of equal response to that of the longest available congener. Retention times for the congeners without authentic specimen were calculated from RIs for available congeners by simple additive scheme.

PFAS (NILU)

Before extraction, a mixture of isotopically labelled PFAS was added, which followed both extraction and processing, and which was used for quantification of the analytes. Sediment, sludge, effluent from treatment plants and biota samples were extracted with organic solvents that ensure a good yield of the analytes. Buffers were used for pH control; this is particularly important for the extraction of the sediment samples. All the extracts were concentrated under nitrogen before they were analysed. Concentrated extract was analysed with UHPLC-HRMS (Orbitrap, Exploris 120) for new PFAS in full-scan mode. At least one blank sample/series of samples was analysed. At least one spiked sample (and one non-spiked) was analysed for each type of material. Quantification of the three shortest acid was made with help of calibration solutions (authentic specimens available). For the two longest acids (authentic samples not available), quantitation was done in an assumption of equal response to that of the longest available congener. Retention times for the congeners without authentic specimen were calculated from RIs for available congeners by simple additive scheme.

SDPA (NIVA)

Biota samples were homogenised and then extraction was carried out with two sequential volumes of acetonitrile. Excess sodium chloride was added for salting-out and then the upper acetonitrile phase was removed for analysis. Analysis was performed via LC-HRMS.

Sediment, sludge, granulate and dust samples were extracted with a 9:1 mixture of methanol and ethyl acetate. Extracts were evaporated and reconstituted in methanol for analysis. Quantitative analysis was performed by LC-HRMS.

Aqueous samples were extracted via solid phase extraction (SPE) on Waters HLB columns. Due to known instability via hydrolysis of many of the analytes, this procedure was carried out in the field at the time of sampling, or immediately on return to the laboratory. Elution from the SPE columns was carried out without delay and without excess drying to reduce airflow and ozone interaction with the analytes. SPE elution was done with a 1:1:1 mixture of methanol, dichloromethane, and ethyl acetate. Analysis was performed by LC-HRMS.

BTZ (NIVA)

Biota samples were homogenised and then extraction was carried out with two sequential volumes of acetonitrile. Excess sodium chloride was added for salting-out and then the upper acetonitrile phase was removed for analysis. Analysis was performed by LC-MS.

Sediment, sludge, granulate and dust samples were extracted with a 9:1 mixture of methanol and ethyl acetate. Extracts were evaporated and reconstituted to give 50% methanol in water for analysis. Quantitative analysis was performed by LC-MS.

Aqueous samples were extracted via solid phase extraction (SPE) on Waters HLB columns. Elution from the SPE columns was with a 1:1:1 mixture of methanol, dichloromethane and ethyl acetate. Eluents were evaporated and extracts reconstituted in 50% methanol for analysis by LC-MS.

LEGE (NIVA)

Sediment and sludge samples were extracted with a 9:1 mixture of methanol and ethyl acetate. Extracts were evaporated and reconstituted to give 50% methanol in water for analysis. Quantitative analysis was performed by LC-HRMS.

Aqueous samples were extracted via solid phase extraction (SPE) on Waters HLB columns. Elution from the SPE columns was with a 1:1:1 mixture of methanol, dichloromethane and ethyl acetate. Eluents were evaporated and extracts reconstituted in 50% methanol for analysis by LC-HRMS.

PEST (NIVA)

For lambda-Cyhalothrin (CAS 91465-08-6) extraction of sediment, sludge and dusts samples was done with a 50:40:10 mixture of ethyl acetate, cyclohexane and acetonitrile. Extracts were cleaned-up and interferants removed via gel-permeation chromatography (GPC). Analysis of cleaned extracts was doe by GC-MS/MS.

Analysis of CAS 91465-08-6 in aqueous samples followed liquid-liquid extraction with dichloromethane. Extracts were cleaned via GPC before analysis on GC-MS/MS.

For the remaining substances in this group, sediment, sludge and dusts samples were extracted with a 9:1 mixture of methanol and ethyl acetate. Extracts were evaporated and reconstituted to give 50% methanol in water for analysis. Quantitative analysis was performed by LC-HRMS.

Aqueous samples were extracted via solid phase extraction (SPE) on Waters HLB columns. Elution from the SPE columns was done with a 1:1:1 mixture of methanol, dichloromethane and ethyl acetate. Eluents were evaporated and extracts reconstituted in 50% methanol for analysis by LC-HRMS.

SURF (NIVA)

Sediment, sludge, and dusts samples were extracted with a 9:1 mixture of methanol and ethyl acetate. Extracts were evaporated and reconstituted to give 50% methanol in water for analysis. Quantitative analysis was performed by LC-MS.

Aqueous samples were extracted by solid phase extraction (SPE) on Waters HLB columns. Elution from the SPE columns was with a 1:1:1 mixture of methanol, dichloromethane, and ethyl acetate. Eluents were evaporated and extracts reconstituted in 50% methanol for analysis by LC-MS.

Concentration limit of detection (LOD)

The concentration LOD for the substances in the different types of sample matrices are provided in Table A5. Note that for some substances the LOD can vary also for the same sample type. The reason for this is most often the varying amount of sample collected from the various sampling types. A high sample volume/mass can typically provide a lower LOD. Furthermore, samples with a high particle load, and/or containing many other substances can lead to a lower LOD compared to a clean water sample.

Table A5: concentration LOD for the various substances in the different types of samples matrices.

CAS	Method ID	Short name*	Water (ng/L)	Sludge, sediment (ng/g)	Granule (ng/g)	Biota (ng/g, w.w.)	Air and Air, particle (ng/m^3)	Dust (ng/g)
51437-66-2	BIS	4_4-ADP	0.1/ 0.2	0.1/ 0.2/ 0.3	NA	0.2	0.2/ 2.9	0.2/ 0.3
57244-54-9	BIS	TMBP	0.2/ 0.3/ 0.4/ 0.5/ 0.6/ 0.7/ 0.8	0.1	NA	0.2/ 0.9	0.2/	0.2/ 0.3/ 0.4/ 0.6
142648-65-5	BIS	OH-BPA	0.1/ 0.2/ 0.3/ 0.4/ 0.6/ 0.7/ 0.8/ 0.9/ 1.0	0.1/ 0.2/ 0.4/ 0.8	NA	0.2/ 1.1	0.2/	1.7
93589-69-6	BIS	44-BPS2	0.1/ 0.2/ 0.3/ 0.4/ 1.0	0.1/ 0.2/ 0.5	NA	0.1/ 0.2	0.2/ 0.7/ 2.6	0.1/0.2/ 0.4/ 0.5
83558-87-6	BIS	BIS TMC	0.1/ 0.2/ 0.3/ 0.4/ 0.5/ 0.7/ 1.6/ 2.7/ 3.4/ 3.9	0.1/ 0.2/ 0.3	NA	0.3/ 0.6/ 0.9	0.2/ 2.1/ 3.1	0.1/ 0.3/ 0.4 /0.5/ 0.6/ 0.7/ 0.9/ 1.5/ 2.2
131-53-3	BIS	BP-8	0.1/ 0.2/ 0.3/ 0.4/ 0.5/ 1.0	0.2/ 0.5/ 189/ 507	NA	0.6	0.2/ 2.1	0.1/ 0.2/ 0.3/ 0.4/ 1.0/ 2.6/
1137-42-4	BIS	4-HBP	0.1	0.1/ 0.3	NA	0.2	0.2	0.2
63449-39-8	LCCP	LCCP					100	
67-66-3	VOC	TCM	0.30	NA	NA	NA	300	Na
75-09-2	VOC	DCM	0.30	NA	NA	NA	300	Na
79-01-6	VOC	TCE	0.21	NA	NA	NA	210	Na
127-18-4	VOC	PCE	0.13	NA	NA	NA	130	Na
56-23-5	VOC	CCl4	0.6	NA	NA	NA	600	Na
75-27-4	VOC	DBM	0.33	NA	NA	NA	330	Na

549-18-8	LEGE	Amitriptyline	1	1	NA	NA	NA	10
83799-24-0	LEGE	Fexofenadine	1	1	NA	NA	NA	10
173584-44-6	PEST	Indoxacarb	1	1	NA	NA	NA	10
120068-37-3	PEST	Fipronil	0.1	1	NA	NA	NA	10
91465-08-6	PEST	Cyhalothrin	10	1	NA	NA	NA	10
136-95-8	BTZ	ABT	1/ 50	1	1	0.5	10	10
941-57-1	BTZ	BTSA	1/ 50	1	1	0.5	10	10
615-22-5	BTZ	MTBT	1/ 50	1	1	0.5	10	10
883-93-2	BTZ	PhBT	1/ 50	1	1	0.5	10	10
934-34-9	BTZ	OHBT	1/ 50	1	1	0.5	10	10
149-30-4	BTZ	MBT	1/ 50	1	1	0.5	10	10
101-87-1	SDPA	CPPD	1/ 50	1	1	0.5	10	10
NO CAS#	SDPA	6PPDq	1/ 50	1	1	0.5	10	10
74-31-7	SDPA	DPPD	1/ 50	1	1	0.5	10	10
93-46-9	SDPA	DNPD	1/ 50	1	1	0.5	10	10
101-72-4	SDPA	IPPD	1/ 50	1	1	0.5	10	10
3081-14-9	SDPA	77PD	1/ 50	1	1	0.5	10	10
793-24-8	SDPA	6PPD	1/ 50	1	1	0.5	10	10
3081-01-4	SDPA	7PPD	1/ 50	1	1	0.5	10	10
101-96-2	SDPA	44PD	1/ 50	1	1	0.5	10	10
81-39-0	SDPA	MTDID	1/ 50	1	1	0.5	10	10
93-05-0	SDPA	NDPD	2/ 10/ 50	10	10	2	10	10
92-09-1	SDPA	MPAMS	1/ 50	1	1	0.5	10	10
10081-67-1	SDPA	PPPA	1/ 50	1	1	0.5	10	10
15721-78-5	SDPA	BOPA	1/ 50	1	1	0.5	10	10
3089-11-0	SDPA	TMMAT	1/ 50	1	1	0.5	10	10
102-06-7	SDPA	DPG	1/ 50	1	1	0.5	10	10
26780-96-1	SDPA	TMQ	1/ 50	1	1	0.5	10	10
68015-60-1	SDPA	BPSE	1 / 50	1	1	5	10	10
1025-15-6	SURF	TAI	1	1	1	NA	10	10
101-37-1	SURF	TAOT	1	1	1	NA	10	10
2687-94-7	SURF	OP	1	1	1	NA	10	10
901-44-0	SURF	BPAE	10	10	10	NA	30	30
1025-15-6	SVOC	isoTAC	0.02/ 0.25/ 0.5	0.02/ 0.2	0.2	0.25/ 0.5	0.001/ 0.025/ 20/ 1000	10
101-37-1	SVOC	TAC	0.2/ 2/2.5/ 5/	0.2/ 2	2.5/ 5	2.5/ 5	0.001/ 5/20/ 1000	10
42074-68-0	SVOC	CTC/CTA	0.5/ 1/2	0.5/ 2	5	NA	0.005/ 0.025/ 0.05/ /0.1 /0.120 /20/ 1000	10

80-07-9	SVOC	BCPS	1 / 0.2/ 0.25/ 0.025	0.2	2	NA	0.001/ 0.05 / 0.1/ 0.2/ 20 / 1000	10
107-52-8	SIL	L6	2.5	2.5	1	10	0.003	90
541-01-5	SIL	L7	30	30	30	20	0.01	300
556-69-4	SIL	L8	40	40	40	100	0.04	700
2652-13-3	SIL	L9	30	30	30	200	0.06	1000
556-70-7	SIL	L10	20	5/ 20	4	1000	0.05/ 0.27	1000
674-13-5	PFAS	12-PFECA	0.5	0.1	NA	NA	NA	2
39492-88-1	PFAS	112-PFECA	0.5	0.1	NA	NA	NA	2
39492-89-2	PFAS	1112-PFECA	0.5	0.1	NA	NA	NA	2
39492-90-5	PFAS	11112-PFECA	0.5	0.1	NA	NA	NA	2
377-73-1	PFAS	13-PFECA	0.5	0.1	NA	NA	NA	2

B. Appendix – List of substances

Table B1: Overview of the substances included in the study.

CAS	NAME	Short name*	Substance Group	Method ID
68015-60-1	Isopropylidenedi-1,4-phenylene bis(2-aminobenzenesulphonate)	BPSE	Bisphenol	SDPA
901-44-0	Ethanol, 2,2'-[(1-methylethylidene)bis(4,1-phenyleneoxy)]bis-	BPAE	Bisphenol	SURF
80-07-9	4,4'-Dichlorodiphenyl sulfone	BCPS	Bisphenol	SVOC
51437-66-2	trans-4,4'-Azodiphenol	4_4-ADP	Bisphenol	BIS
57244-54-9	Phenol, 4,4'-(1,3,3-trimethyl-1-propene-1,3-diyl)bis-	TMBP	Bisphenol	BIS
142648-65-5	2,2-Bis(4-hydroxyphenyl)-1-propanol	OH-BPA	Bisphenol	BIS
93589-69-6	Phenol, 4,4'-[methylenebis(oxy-2,1-ethanediylthio)]bis-	44-BPS2	Bisphenol	BIS
83558-87-6	2,2-Bis(3-amino-4-hydroxyphenyl)-hexafluoropropane	BIS TMC	Bisphenol	BIS
63449-39-8	Chlorinated paraffins	LCCP	Chlorinated paraffins	LCCP
67-66-3	Chloroform	TCM	Disinfectant/ Solvent	VOC
75-09-2	Dichloromethane	DCM	Disinfectant/ Solvent	VOC
79-01-6	Trichloroethylene	TCE	Disinfectant/ Solvent	VOC
127-18-4	Tetrachloroethylene	PCE	Disinfectant/ Solvent	VOC
56-23-5	Carbon tetrachloride	CCl4	Disinfectant/ Solvent	VOC
75-27-4	Bromodichloromethane	DBM	Disinfectant/ Solvent	VOC
549-18-8	Amitriptyline hydrochloride	Amitriptyline	Pharmaceutical / Pesticide	LEGE
83799-24-0	Fexofenadine	Fexofenadine	Pharmaceutical / Pesticide	LEGE
173584-44-6	Indoxacarb	Indoxacarb	Pharmaceutical / Pesticide	PEST
120068-37-3	Fipronil	Fipronil	Pharmaceutical / Pesticide	PEST
91465-08-6	lambda-Cyhalothrin	Cyhalothrin	Pharmaceutical / Pesticide	PEST
42074-68-0	2-Chlorotrityl chloride	CTC/CTA	Pharmaceutical / Pesticide	SVOC
136-95-8	2-Aminobenzothiazole	ABT	Rubber Production or Stabilization	BTZ
941-57-1	Benzothiazole-2-sulfonic acid	BTSA	Rubber Production or Stabilization	BTZ
615-22-5	2-(Methylthio)benzothiazole	MTBT	Rubber Production or Stabilization	BTZ
883-93-2	2-Phenylbenzothiazole	PhBT	Rubber Production or Stabilization	BTZ
934-34-9	Benzothiazolone	OHBT	Rubber Production or Stabilization	BTZ
149-30-4	2-Mercaptobenzothiazole	MBT	Rubber Production or Stabilization	BTZ
101-87-1	N-Cyclohexyl-N'-phenyl-4-phenylenediamine	CPPD	Rubber Production or Stabilization	SDPA

NO CAS#	2-anilino-5-[(4-methylpentan-2-yl)amino]cyclohexa-2,5-diene-1,4-dione	6PPDq	Rubber Production or Stabilization	SDPA
74-31-7	N,N'-Diphenyl-p-phenylenediamine	DPPD	Rubber Production or Stabilization	SDPA
93-46-9	N,N'-Di-2-naphthyl-p-phenylenediamine	DNPD	Rubber Production or Stabilization	SDPA
101-72-4	N-Isopropyl-N'-phenyl-p-phenylenediamine	IPPD	Rubber Production or Stabilization	SDPA
3081-14-9	N,N'-Bis(1,4-dimethylpentyl)-4-phenylenediamine	77PD	Rubber Production or Stabilization	SDPA
793-24-8	6PPD	6PPD	Rubber Production or Stabilization	SDPA
3081-01-4	N-(5-Methyl-2-hexyl)-N'-phenyl-p-phenylenediamine	7PPD	Rubber Production or Stabilization	SDPA
101-96-2	N,N'-Bis(1-methylpropyl)-1,4-benzenediamine	44PD	Rubber Production or Stabilization	SDPA
81-39-0	3-Methyl-6-(p-toluidino)-3H-dibenz(f,ij)isoquinoline-2,7-dione	MTDID	Rubber Production or Stabilization	SDPA
93-05-0	N,N-Diethyl-4-aminoaniline	NDPD	Rubber Production or Stabilization	SDPA
92-09-1	N-{2-[(4-amino-3-methylphenyl)(ethyl)amino]ethyl} methanesulfonamide	MPAMS	Rubber Production or Stabilization	SDPA
10081-67-1	4-(2-Phenylpropan-2-yl)-N-[4-(2-phenylpropan-2-yl)phenyl]aniline	PPPA	Rubber Production or Stabilization	SDPA
15721-78-5	N,N-Bis(4-tert-octylphenyl)amine	BOPA	Rubber Production or Stabilization	SDPA
3089-11-0	Hexa(methoxymethyl)melamine	TMMAT**	Rubber Production or Stabilization	SDPA
102-06-7	1,3-Diphenylguanidine	DPG	Rubber Production or Stabilization	SDPA
26780-96-1	1,2-Dihydro-2,2,4-trimethylquinoline, polymer	TMQ	Rubber Production or Stabilization	SDPA
1025-15-6	Triallyl isocyanurate	isoTAC	Rubber Production or Stabilization	SVOC
101-37-1	2,4,6-Tris(allyloxy)-1,3,5-triazine	TAC	Rubber Production or Stabilization	SVOC
107-52-8	Tetradecamethylhexasiloxane	L6	Siloxane	SIL
541-01-5	Hexadecamethylheptasiloxane	L7	Siloxane	SIL
556-69-4	Octadecamethyloctasiloxane	L8	Siloxane	SIL
2652-13-3	Eicosamethylnonasiloxane	L9*	Siloxane	SIL
556-70-7	Decasiloxane, docosamethyl-	L10*	Siloxane	SIL
2687-94-7	1-Octyl-2-pyrrolidone	OP	Surfactants incl. Perfluoronated Substances	SURF
674-13-5	Perfluoro-2-methoxyaceticacid	12-PFECA	Surfactants incl. Perfluoronated Substances	PFAS
39492-88-1	Perfluoro-3,5-dioxahexanoic acid	112-PFECA*	Surfactants incl. Perfluoronated Substances	PFAS
39492-89-2	Perfluoro-3,5,7-trioxaoctanoic acid	1112-PFECA*	Surfactants incl. Perfluoronated Substances	PFAS
39492-90-5	Perfluoro-3,5,7,9-butaoxadecanoic acid	11112-PFECA*	Surfactants incl. Perfluoronated Substances	PFAS

377-73-1	Perfluoro-3-methoxypropanoic acid	13-PFECA	Surfactants incl. Perfluoronated Substances	PFAS
131-53-3	2,2'-Dihydroxy-4-methoxybenzophenone	BP-8	UV-Additive	BIS
1137-42-4	4-Hydroxybenzophenone	4-HBP	UV-Additive	BIS

*Substances analysed by “suspect screening” (n=5), meaning that the concentration cannot be quantified. Only qualitative analyses.

**Also commonly known by HMMM.

C. Appendix – List of sampling sites

Table C1: Overview over the sites sampled including coordinates and dates of sampling.

Site name	**Coordinates (UTM32)**	**Date 1**	**Date 2**	**Date 3**	**DATE 4**	**DATE 5**
Apalløkka	603920.36, 6648116.03	23.05.2022	03.11.2022			
Valle Hovin, north	600713.35, 6644256.21	07.11.2022				
Valle Hovin, east	600860.36, 6644164.96	07.11.2022				
Obos	603879.10, 6648151.25	28.11.2022				
LSK	615636.66, 6648938.15	28.11.2022				
Smestadtunnelen	593952.71, 6645584.36	03.10.2022				
Vålerengatunnelen	599859.58, 6642229.39	18.11.2022	02.12.2022	08.12.2022		
Smestaddammen (upper)	258631.00, 6652072.00	29.08.2022				
Smestaddammen (lower)	258666.00, 6651802.00	29.08.2022				
Alnabru	603735.01, 6644759.43	20.09.2022	23.09.2022	12.12.2022	02.01.2023	
Hjortneskaja	595595.20, 6642492.65	12.09.2022	09.09.2022	13.12.2022	02.01.2023	
Smestad	594180.77, 6645711.49	24.09.2022	26.09.2022	02.01.2022	02.01.2023	
Sofienbergparken	598581.85, 6644165.16	06.09.2022	09.09.2022	13.09.2022	10.12.2022	16.12.2022
Bekkelaget	599091.92, 6639673.98	26-29.09.2022	03-05.10.2022			
Skjerkøya	536609.80, 6546879.61	08.09.2022	29.09.2022			
Kalbakken	59.954285, 10.865691	13.09.2022	26.10.2022	27.02.2023	03.11.2022	27.02.2023
Brubak	604962.51, 6646841.52	10.05.2022	12.09.2022	13.09.2022	26.10.2022	27.02.2023
Breivoll	602600.10, 6644278.52	10.05.2022	12.09.2022	13.09.2022	26.10.2022	27.02.2023
Kværner	600216.99, 6642145.02	25.05.2022	12.09.2022	13.09.2022	26.10.2022	27.02.2023
Padderudvannet	576160.53, 6632576.720	30.08.2022				
Private home 1	NN	22.06.2022				
Private home 2	NN	27.06.2022				
Private home 3	NN	29.06.2022				
Private home 4	NN	14.07.2022				
Private home 5	NN	08.09.2022				
Private home 6	NN	16.08.2022				
Dental clinic 1	NN	13.10.2022				

Dental clinic 2	NN	01.11.2022				
Dental clinic 3	NN	11.11.2022				
3-D printing 1	NN	14.09.2022	14.09.2022			
3-D printing 2	NN	08.11.2022				
3-D printing 3	NN	03.11.2022				
Furniture store 1	NN	24.08.2022				
Furniture store 2	NN	24.08.2022				
Furniture store 3	NN	26.10.2022				
Søndre Skjælholmen, Raudskjæra and Husbergøya	Multiple	23.05-21.06.2022				
Midtmeie, southwest of Steilene	Multiple	17.08.2022				
Torbjørnskjær, Søndre Missingen, Singleøya and Garnholmen	Multiple	09.01-11.01.2021				

Norges ledende kompetansesenter på vannmiljø

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