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Monitoring of microplastics in the Norwegian environment (MIKRONOR)

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Monitoring of microplastics in the Norwegian environment (MIKRONOR)
Overvåking av mikroplast i det norske miljø (MIKRONOR)

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Abstract

In 2021 The Norwegian Environment Agency (Miljødirektoratet) assigned the first analyses of microplastics within a national monitoring program "Microplastics in Norwegian coastal areas, rivers, lakes and air (MIKRONOR)" to NIVA. The aim of the program was to build knowledge about the background levels of microplastics in Norwegian environment, as well as identify potential sources and sinks. This is the second annual report, which presents the results from samples of 1) marine and lake/river sediments, biota and water, 2) air and deposition at two sites, including one at Svalbard, and 3) potential sources: urban runoff and effluent of wastewater treatment plants (WWTP) in two cities (Oslo and Hamar). The samples were analysed for microplastics, including tyre wear particles (TWP) from cars. The concentrations of plastic particles (mass of polymers per volume/weight unit) were calculated, using a novel formula for estimating volume of particles from the numerical analysis by spectroscopic (FTIR) analysis. The air samples were analysed for mass concentrations by mass spectrometric analysis. The main findings were the large number and concentrations of particles found in the inner Oslofjord. This included large numbers of microplastic particles resulting in high mass concentrations (µg/g dw) of plastic polymers. Particularly high mass concentrations of TWP were found in the sediments of the inner Oslofjord. TWP were also found at considerably high concentrations in blue mussels from the same area (Akershuskaia). Additionally, the urban runoff samples from both Oslo and Hamar showed high concentrations of TWP. High concentrations of TWP were also found in freshwater sediments near Hamar.

Keywords: Microplastic pollution, Environmental contaminants, monitoring, tyre wear particles
Emneord: Mikroplast, forurensning, overvåking, dekkpartikler

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Preface

The Norwegian Institute for Water Research (NIVA), acting on behalf of the Norwegian Environment Agency (Miljødirektoratet, NEA), organised the sampling and carried out subsequent analysis for microplastics in the Norwegian environments for the second round/year of the national microplastic monitoring program "Microplastics in Norwegian coastal areas, rivers, lakes and air (MIKRONOR), whilst Eivind Farmen coordinated the project at the NEA. The project was initiated in 2021, and Vanja Alling has been project manager at NIVA since May 2023. The sampling efforts were a collaborative effort, incorporating seven ongoing national monitoring programs run by NEA. Sverre Hjelset managed the coordinating of sampling equipment and logistics. Sample preparation and microplastic analysis was conducted by Sverre Hjelset, Cecilie Singdahl-Larsen, Chiara Consolaro, Svetlana Pakhomova, Madeline Jefroy, Elena Martinez-Frances and Vilde Kloster Snekkevik. Elisabeth Rødland analysed for tyre wear particles with Pyrolysis-GCMS, while air sampling was coordinated and samples were analysed by Dorte Herzke and Natascha Schmidt from the Climate and Environmental Research Institute NILU. The development of the formulas for calculation of polymer masses from FTIR data was performed by Svetlana Pakhomova, Rachel Hurley and Vanja Alling. Data analyses and development of the interactive database and visualisation tool "SUPERSET" was performed by Espen Lund, Vanja Alling and Jemmima Knight.

The scientific quality assurance was provided by Amy Lusher, Bert van Bavel and Marianne Olsen. This report, focusing on the second phase of MIKRONOR, has been collaboratively written by Vanja Alling, Espen Lund, Amy Lusher, Vilde Kloster Snekkevik, Elisabeth Rødland, Svetlana Pakhomova, Natascha Schmidt and Dorte Herzke.

Oslo, 20. December 2023

Vanja Alling

Project Manager

NIVA

Sammendrag

På oppdrag fra Miljødirektoratet har Norsk institutt for vannforskning (NIVA) organisert og analysert prøver for mikroplast i norske akvatiske miljøer og luft gjennom det nasjonale overvåkningsprogrammet «Mikroplast i kystområder, elver og innsjøer (MIKRONOR)». Programmet ble initiert i 2021 og har som hensikt å kartlegge mikroplastnivåer, undersøke potensielle kilder til mikroplast og danne et grunnlag for videre overvåking av mikroplastnivåer i Norge.

For å dekke et mangfold av prøvetyper og geografiske områder har andre overvåkningsprogrammer bidratt i prøveinnsamlingen, i tillegg til prøvetakingen innenfor MIKRONOR-prosjektet. Det ble tatt prøver fra norske innsjøer, kystområder, åpent hav, elver, luft, nedbør, renseanlegg, urban overflateavrenning, sedimenter og biota, samt blankprøver fra felt og laboratorium. Prøvetakingsstasjonene var hovedsakelig de samme som i de deltagende overvåkningsprogrammene, og prøvetakingsmetodene var etablerte metoder i programmene, med tilpasninger for mikroplast.

Prøvene ble behandlet og analysert i et kontrollert laboratorium, der anbefalte internasjonale protokoller ble fulgt og metodevalideringer utført. Analytisk kvantifisering og karakterisering av plastpartikler (antall, form, størrelse, polymertype) ble utført ved hjelp av mikro-Fourier-transform infrarød spektroskopi (μFTIR), i tillegg til kvantifisering av bildekk-partikler (TWP) ved bruk av pyrolyse-gasskromatografi-massespektrometri (Pyrolyse GC-MS). Analysene av bildekk ble introdusert i årets analyseprogram for MIKRONOR. Denne rapporten inneholder analyser og diskusjoner av totalt 374 prøver med tilhørende blankprøver. Rapporten har også inkludert forrige års data (for noen prøvetyper som også ble rapportert i 2022), for å gi et større grunnlag for diskusjonen.

I prøver av vann, sedimenter og biota fra områder uten kjente, nærliggende kilder viste analysene generelt lave nivåer av mikroplast, ofte under deteksjonsgrensene. I prøver fra urbane områder derimot (Oslo og Hamar, med tilhørende resipienter), ble det funnet betydelig større mengder mikroplast og dekkpartikler. Feltblankene som ble tatt parallelt med miljøprøvene viste lave nivåer av mikroplast, og dermed liten kontaminasjon i felt, for alle prøvetyper unntatt prøver tatt med planktonnett. Det var fremfor alt fibrer som var problematisk i planktonprøvene, og alle planktonnett-prøver har derfor blitt rapportert uten fibrer.

Bildekkpartikler ble funnet i alle analyserte prøvetyper, med høyest konsentrasjoner målt i blåskjell og marint sediment i indre Oslofjord utenfor Akershuskaia. Like høye konsentrasjoner av bildekkpartikler ble også påvist i ferskvannssedimenter nær Hamar (Mjøsa). Disse resultatene kan kobles til analyser av overflateavrenning fra Hamar og Oslo by, der prøvene viste høye konsentrasjoner av bildekkpartikler.

Mengde mikroplast for ni polymertyper ble målt i aktive luftprøver og nedbørsprøver fra Birkenes (Agder) og Zeppelin-stasjonen på Svalbard. Mengde mikroplast i aktive luftprøver fra Birkenes var høyere enn ved Zeppelin (omtrentlig 2 ganger mer), trolig forårsaket av større påvirkning fra kontinentale luftmasser. I nedbørsprøver kunne vi ikke se en lignende forskjell mellom de to stasjonene når alle prøvene sees samlet. Når vi sammenligner variasjon av mikroplast i luft over tidsperioden, kan vi se en økning av mikroplast-konsentrasjoner i Birkenes mot slutten av høsten. Det ble funnet betydelig mer mikroplast i nedbørsprøver enn i aktive luftprøver.

Av de polymertyper som defineres i AMAP (2021), og som ble målt med FTIR, ble det totalt funnet flest partikler av polypropylen, når summert for alle prøvetyper unntatt luft og deposisjon. Polypropylen var også polymeren med høyest antall partikler i prøvene fra urban avrenning, men ikke i prøvene fra avløpsvannsanleggene (WWTP). Polypropylen er blant de mest produserte og brukte plasttypene globalt (Lusher & Pettersen, 2021). Når man ser på sammensetning av type plast funnet i luftprøvene fra

Birkenes og Zeppelin, dominerer polyvinylklorid (PVC) og polyetylen (PE) i de fleste prøvene. Et unntak er aktive prøver fra Zeppelin, hvor ingen PVC ble funnet. Disse er vanlige polymere, både med høyt produksjonsvolum og mange bruksområder. PVC og PE forekom, men var kun dominerende i noen prøver, fra andre prøvematriks enn luft.

I årets rapport har vi hatt ekstra fokus på prøvene fra indre Oslofjorden. I tillegg til høye nivåer av mikroplastpartikler generelt, høye konsentrasjoner basert på masse, og høye nivåer av bildekksrelaterte partikler (opp til 2 % av sedimentene bestod av bildekkspartikler), skilte sedimentprøvene fra Akershuskaia seg ut på grunn av sine store mengder malingspartikler. I motsetning til dette, viste verken prøvene fra overvann eller prøvene fra Bekkelaget renseanlegg høye konsentrasjoner av malingspartikler.

Alle årets resultater og beskrivelse av metoder finnes på NIVAS interaktive hjemmeside «SUPERSET».

Basert på erfaringene fra MIKRONOR, anbefales det å fortsette å inkludere dekkpartikler i overvåking av mikroplast, samt å forbedre etablerte overvåkningsprogrammer (f.eks. Screeningprogrammet, MILFERSK, MILKYS og Urban fjord) ved å inkludere analyser av miljøgifter relatert til bildekkpartikler, som for eksempel 6-PPD-quinone. Dette bør spesielt gjøres i prøvetyper som sediment og blåskjell. Videre anbefales det å redusere antall prøvetyper i mikroplastovervåkingen, samtidig som det bør legges økt vekt på utvalgte prøvetyper og øke antall prøver. Dette vil bidra til å øke kvaliteten og representativiteten av prøvene, og føre til tydeligere og bedre resultater. En ytterligere fordel vil være å styrke metodikken rundt prøvetaking og analyse av mikroplastprøver. Det anbefales også å prioritere utvikling av databehandling og statistisk analyse innen mikroplast, slik at det lages gode og sammenlignbare datasett fra år til år innenfor MIKRONOR.

1 Introduction to the monitoring program MIKRONOR

Norwegian Institute for Water Research (NIVA), on behalf of the Norwegian Environment Agency (NEA), is responsible for Norway's first national microplastic monitoring program, with the climate and environmental research institute NILU as subcontractors for air sample analyses. The program, Microplastics in Norwegian coastal areas, rivers, lakes and air (MIKRONOR) started in 2021 with organization of sampling for different environmental matrices from other running national monitoring programs, and the sampling continued until 2023 (van Bavel et al., 2022). This is the second report in the program.

MIKRONOR aims to establish a baseline for future microplastics monitoring programs and to investigate potential high-impact areas and sources to microplastics. The ultimate, long-term goal is to create a robust knowledge base on microplastics pollution for policymaking and to ensure the public is well-informed about the state of the environment.

As microplastics are ubiquitous in the environment, MIKRONOR has investigated several matrices to establish baseline levels and to understand trends of microplastics in the Norwegian environment. The samples have been analysed at NIVA, with the exception for the air samples, that have been analysed at NILU.

The definition of microplastics (MP) which has been used within MIKRONOR is in accordance with EU DIRECTIVE 2019/904: Synthetic material (primarily oil-based polymers) identified in the environment in the size range 50 µm to 5 mm, with a few exceptions (see Table 1). The lower cut-off of 50 µm, is in accordance with the equipment and instrumentation limitations employed in the program. The samples analysed at NIVA were fractionated by size of the particles, using different identification methods for particle smaller or bigger than 300 µm. Microplastic particles have been characterised and identified using Fourier Transform Infrared (FTIR) spectroscopy, identifying the categories and polymer types defined by AMAP (2021). Additionally, the mass of tyre wear particles (TWP), particles resulting from tear and wear of vehicles tyres on the roads, as well as mass of particles in air samples, have been determined by Pyrolysis Gas Chromatography/Mass Spectrometry (Pyrolysis GCMS). The mass of microplastic particles analysed with FTIR have been calculated based on the volume and density of the polymer types of each particle. A summary of the samples analysed, methods and size fractions analysed is presented in Table 1. All methods are described in detail in the appendices of the report.

2 Overview of sample types, sampling programs and analyses

To achieve a wide distribution of samples, both spatial and by matrices, the samples were collected by other already well-established national monitoring programs, such as the Ocean Acidification Monitoring in Norwegian Waters responsible for open ocean water samples through the ferrybox sampling system (Water, Ferrybox). The program Pollutants in an Urban Fjord has collected high-volume water samples from the Oslofjord (Water, pump), while the programs Ecosystem Monitoring in Coastal Waters (ØKOKYST), and in lakes (ØKOSTOR and ØKOFERSK), have conducted plankton sampling from coastal areas and freshwaters (Water, vertical plankton nets). These programs have also collected sediment samples in marine and freshwater environments (Marine sediment & Freshwater sediment), with ØKOKYST additionally contributing with invertebrates (mainly polychaeta). Blue mussels have been supplied by the Contaminants in coastal waters (MILKYS), and duck mussels by the Monitoring of environmental pollutants in freshwater (MILFERSK). River samples (Water, manta trawl) of surface waters have been collected through the Norwegian River Monitoring Program, and both Urban Fjord and MILFERSK have provided samples from wastewater treatments plants (effluent) and stormwater samples of urban runoff. Sampling in Svalbard has been performed using both high-volume pump (Water, pump, Svalbard) and neuston net trawls of surface waters (Water, neuston trawl, Svalbard) by a separate research project at Svalbard and air sampling have been conducted by NILU under the monitoring program for long-range transported atmospheric contaminants.

An overview of sample types, including methods and analyses per sample type are listed in Table 1, together with the total number of samples analysed since the last annual report. Some samples were taken in replicates, some are not. Field blanks are taken in parallel with all sample types except for mussels (as they are not exposed to air between sampling and laboratory procedures). In all, 260 atmospheric blanks and 68 other field blanks were analysed in parallel with the environmental samples. This report includes data also reported in the 2022 annual report. Maps of sampling stations are given in Figures 1-3, with station names in Tables 2-4.

Table 1. Sample types in different environments, field methods, number of samples and field blanks, size fractions within each sample type and methods for lab analyses, including both Fourier-Transform Infrared Spectroscopy (FTIR) and Pyrolysis GCMS (Pyr-GCMS), for 2023 report. Pyr-GCMS was only used for tyre wear particles (TWP) and 9 polymers in air samples.

Environment	Sample Type	Sampling Method	Samples	Field blanks	Size fractions analysed	FTIR for MP analysis	Pyr-GCMS for TWP
Coastal	Water, Ferrybox	Ferrybox system collecting seawater at 'M/S Color Fantasy'	20	10	100 µm	✓	
					200 µm	✓	
	Water, pump	High volume pump of surface seawater	9	12	50 µm	✓	✓
					250 µm	✓	
	Water, vertical plankton nets	Vertical plankton net haul	45	137	200 µm	✓	
	Marine sediment	Sediment grab collecting sediments	66	55	50 µm	✓	✓
					300 µm	✓	
	Blue Mussel	Collection of live mussels	18		50 µm	✓	✓
					300 µm	✓	
	Invertebrates (polychaeta)	Collection of invertebrates in sediments	42	46	50 µm	✓	✓
					300 µm	✓	
Svalbard	Water, neuston trawl, Svalbard	Trawling of seawater using a neuston net	18	4	1-5 mm	✓	
	Water, pump, Svalbard	High volume pumping of surface seawater	18	4	100-500 µm	✓	
Lake	Water, vertical plankton nets	Vertical plankton net haul	30	10	200 µm	✓	
	Freshwater sediment	Sediment grab collecting sediment	16	27	50 µm	✓	✓
					300 µm	✓	
	Duck mussel	Collection of live duck mussels	20		50 µm	✓	✓
					300 µm	✓	
River	Water, manta trawl	Trawl of river surface water using manta net	21	7	200 µm	✓	
Wastewater Effluent	Wastewater treatment plants (effluent)	Subsample of effluent from wastewater treatment plants	15	9	50 µm	✓	✓
					300 µm	✓	
Urban	Water, urban run-off	Sample of urban run-off storm water	12		50 µm	✓	✓
					250 µm	✓	
Air	Active air samples	Full-metal filter holder	12	12	20 µm		*✓
	Deposition	Full-metal bulk precipitation sampler	12	12	0 µm		*✓
Total number of samples			**374**	**345**			

*Pyr-GCMS for air samples conducted for 9 polymers (see Appendix 5.5), TWP not included.

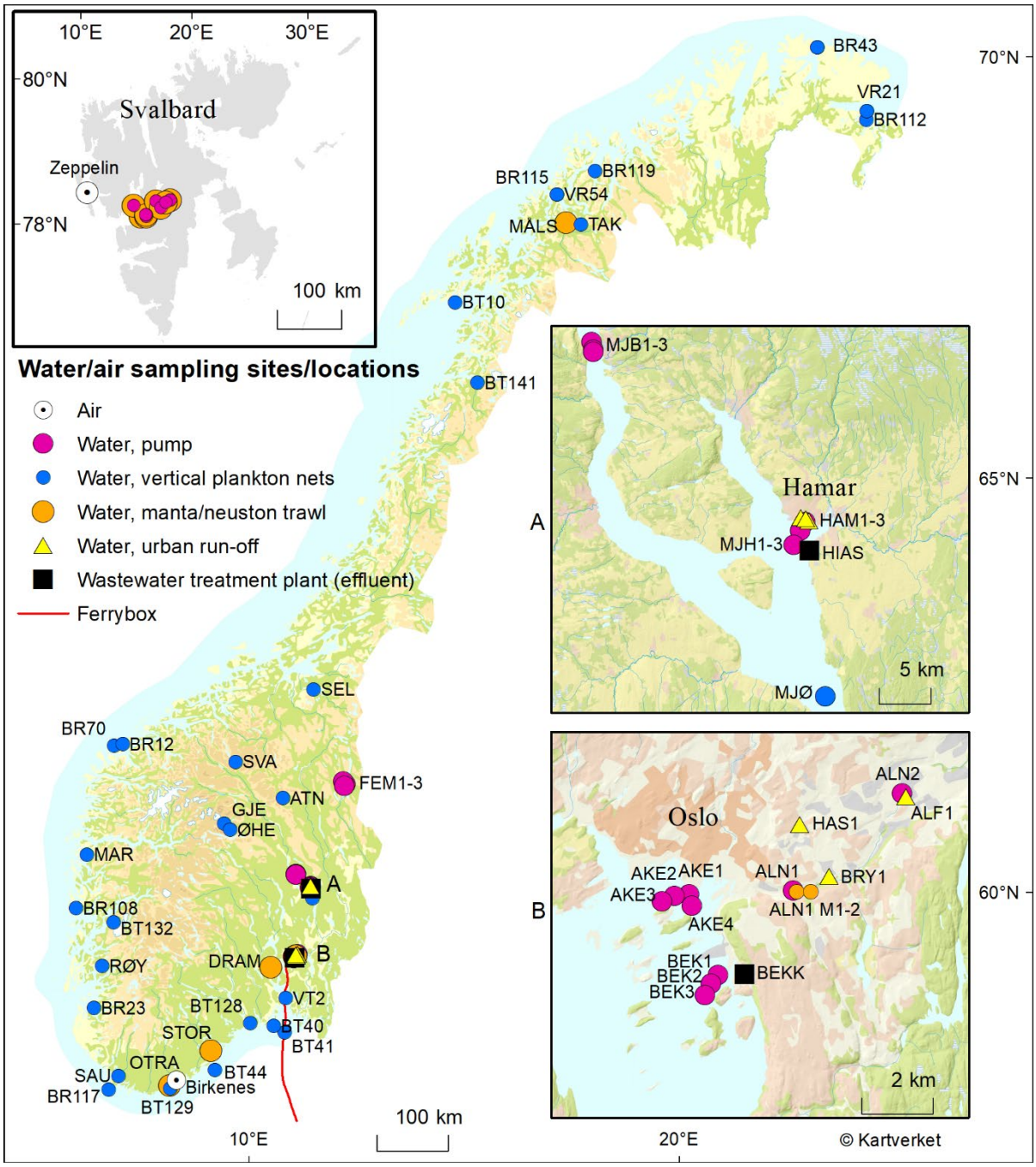

Figure 1. Stations for the analysis of microplastics in water/air samples 2023; stations codes in Table 2.

Table 2. Station names and codes of water samples collected for analysis of microplastics in 2023.

Code	Name	Code	Name	Code	Name	Code	Name
AKE1-3	Akerhuskaia 1-1	VT2	VT2 Skagerak	RØY	Røyravatnet	BR108	Klokkarvik, Sotra
BEK1-3	Bekkelaget 1-3	BT128	BT128 Skagerak	GJE	Gjende	BR70	Herøyfjorden
ALN1	Alnaelva, Kværner 1	BT129	BT129 Skagerak	MJØ	Mjøsa	BR12	Skinnbrokleia
ALN2	Alnaelva, Alnabru	BT41	BT41 Skagerak	MAR	Markhusdalsv.	VR54	Straumsfjorden
MJH1-3	Mjøsa, Hamar 1-1	BR117	BR117 Nordsjøen	ØHE	Ø. Heimdalsv.	BR119	Ullsfjorden/ Fugløyfj.
MJB1-3	Mjøsa, Mjøsbrua 1-3	BR23	BR23 Nordsjøen	ATN	Atnsjøen	HIAS	Hamar, HIAS
FEM1-3	Femunden 1-3	BT141	BT141 Norskehavet N	SVA	Svartdalsv.	BEKK	I. Oslofjord, Bekkelaget
ALN-M1-2	Alnaelva, Kværner 1-2	BR115	BR115 Norskehavet N	SEL	Selbusjøen	ALF1	Alfaset
DRAM	Drammenselva	VR21	Bugøynes/Barentsh.	TAK	Takvatnet	BRY1	Brynseng
STOR	Storelva	BR43	Tanafjorden/Barentsh	BT40	Færder, Y. Oslofj.	HAS1	Hasle
OTRA	Otra	BR112	BR112 Barentshavet	BT44	Arendal	HAM1-3	Hamar by 1-3
MÅLS	Målselva	SAU	Saudlandsvannet	BT132	Maurangsfj.		

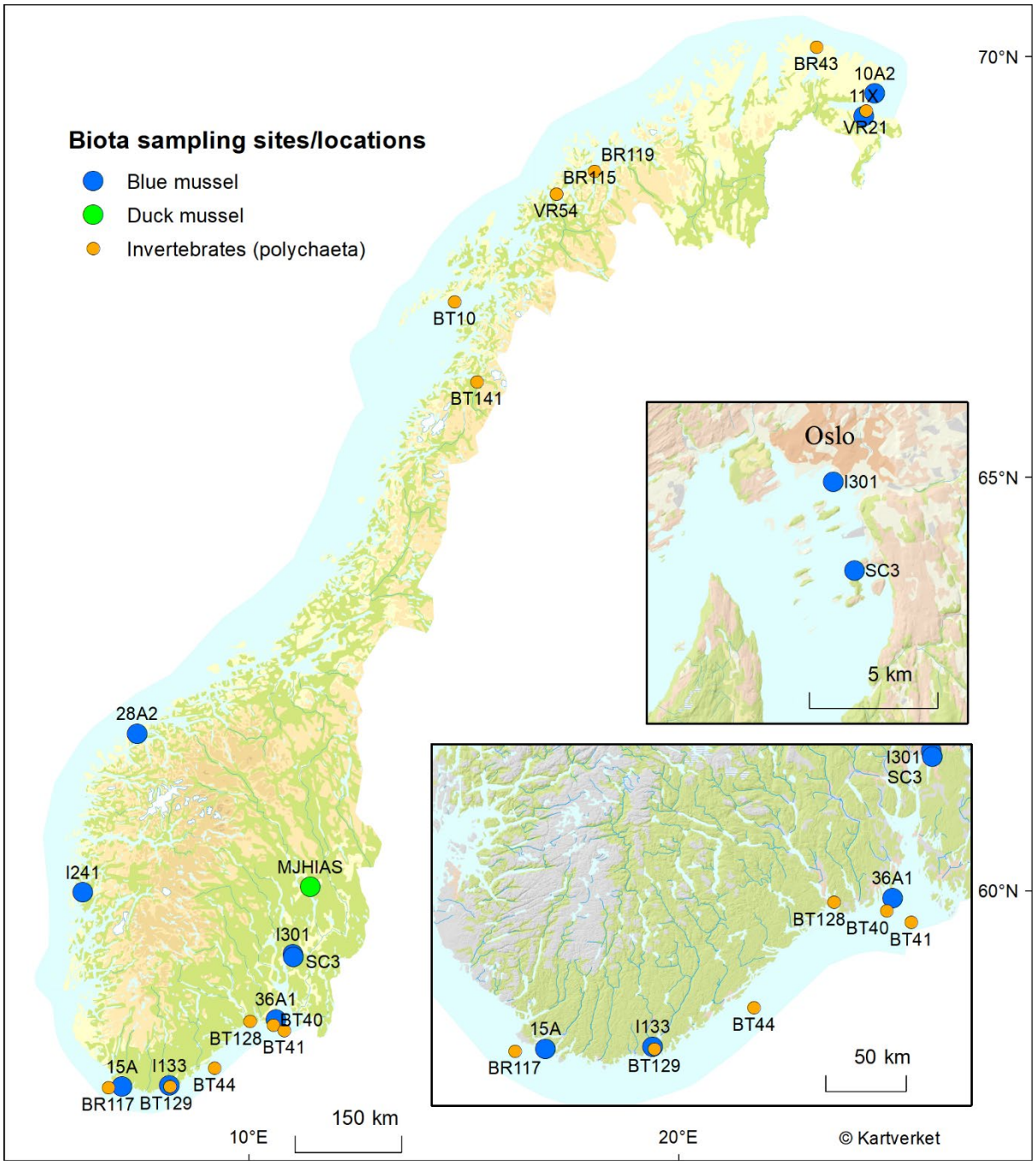

Figure 2. Stations for the analysis of microplastics biota samples 2023; station codes in Table 3.

Table 3. Station names and codes of biota samples collected for analysis of microplastics in 2023.

Code	Name	Code	Name
I301	Akershuskaia, I. Oslofjord	BT128	BT128 Skagerrak
SC3	Bekkelaget, I. Oslofjord	BT44	Arendal, Arendal-Tromøy
36A1	Tjøme, Y. Oslofjord	BT129	BT129 Skagerrak
I133	Kristiansand havn	BR117	BR117 Nordsjøen
15A	Farsund	BT141	BT141 Norskehavet Nord
I241	Bergen havn	BT10	BT10 Norskehavet Nord
28A2	Ålesund havn	VR54	Straumsfjorden
10A2	Skallneset, Varangerfjorden	BR115	BR115 Norskehavet Nord
11X	Brashavn, Varangerfjorden	BR119	Ullsfjorden/Fugløyfjorden
MJHIAS	Mjøsa, Hamar	BR43	Tanafjorden
BT41	BT41 Skagerrak	VR21	Bugøynes, Varangerfjorden
BT40	Færder, Ytre Oslofjord		

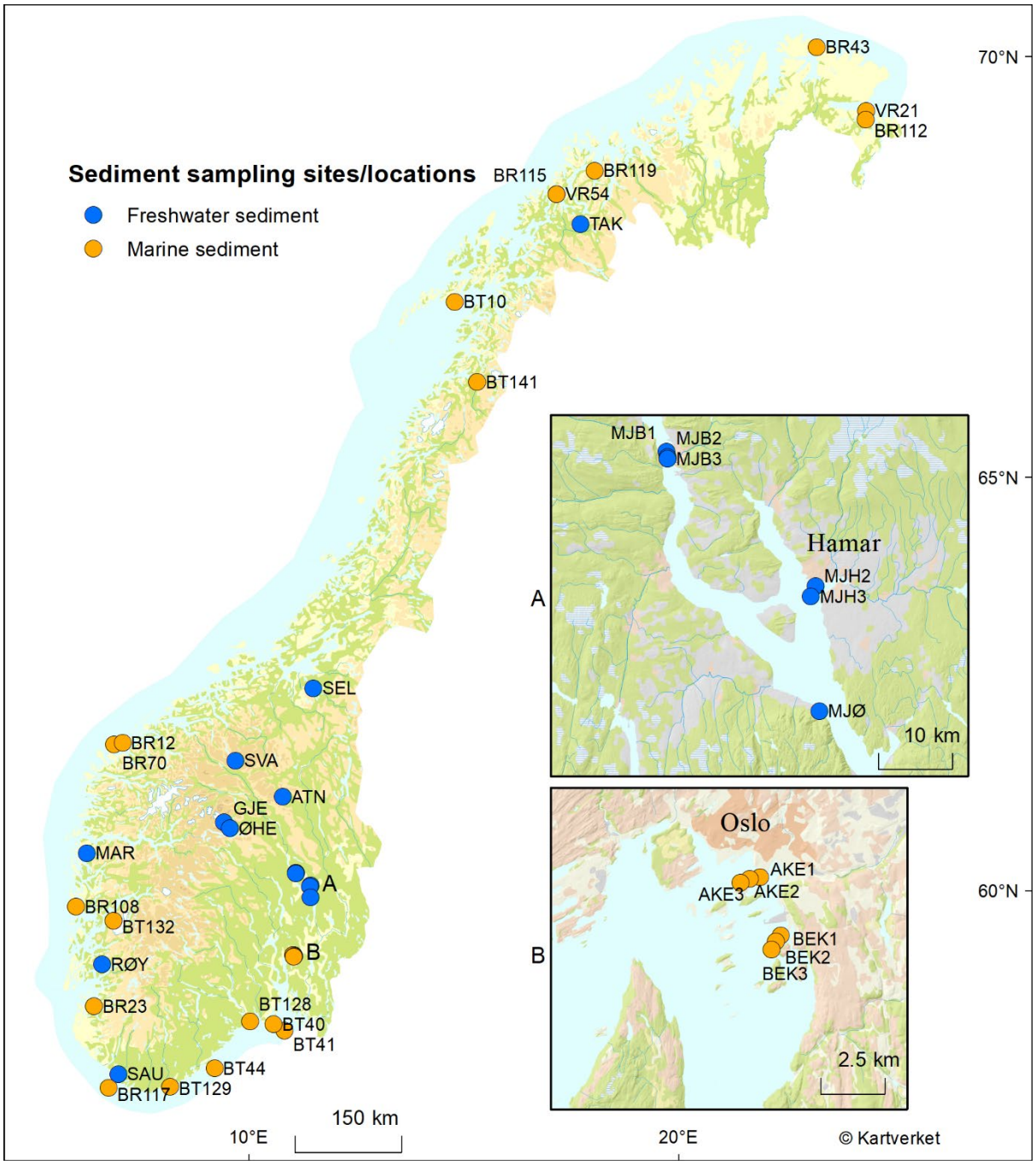

Figure 3. Stations for the analysis of microplastics in sediments 2023; stations codes in Table 4.

Table 4. Station names and codes of sediment samples collected for analysis of microplastics in 2023.

Code	Name	Code	Name	Code	Name
AKE1-3	Akerhuskaia 1-3	BR70	Herøyfjorden	MJB1-3	Mjøsa, Mjøsbrua 1-3
BEK1-3	Bekkelaget 1-3	BR12	Skinnbrokleia	MJØ	Mjøsa
BT41	BT41 Skagerrak	BT141	BT141 Norskehavet N	SAU	Saudlandsvannet
BT40	Færder, Y. Oslofjord	BT10	BT10 Norskehavet N	RØY	Røyravatnet
BT128	BT128 Skagerrak	BR115	BR115 Norskehavet N	MAR	Markhusdalsvatn
BT44	Arendal	VR54	Straumsfjorden	GJE	Gjende
BT129	BT129 Skagerrak	BR119	Ullsfjorden/Fugløyfjorden	ØHE	Ø.Heimdalsvatnet
BR117	BR117 Nordsjøen	BR43	Tanafjorden	ATN	Atnsjøen
BR23	BR23 Nordsjøen	VR21	Bugøynes, Varangerfjorden	SVA	Svartdalsvatnet
BT132	Maurangsfjorden	BR112	BR112 Barentshavet	SEL	Selbusjøen
BR108	Klokkarvik, Sotra	MJH2-3	Mjøsa, Hamar 2-3	TAK	Takvatnet

3 Key findings

In the current year of MIKRONOR, one of the main focus areas has been on analysing tyre wear particles (TWP) to provide initial estimates for various locations and matrices, offering an overview of contamination resulting from car tyre wear in the environment. Furthermore, we have compared TWP with the number of other microplastic particles in the samples and the calculated mass concentrations of other polymers. The results are presented in the following summary.

3.1 Concentrations of TWP in different areas and matrices

Tyre wear particles are a significant source of microplastic pollution in the environment. It has been estimated that between 5,000 to 11,000 tons of road particles, with at least 80% being TWP, are released into the environment each year in Norway (Mepex, 2021). This estimate suggests that TWP could be the most substantial land-based source of microplastics in the Norwegian environment. The distribution and fate of these particles in the Norwegian environment have not been monitored previously and are included in MIKRONOR for the first time.

We have measured TWP with a method developed at NIVA (Rødland et al., 2022), and the method is described in detail in Appendix 5.3. In Figure 4, we present an overview of TWP concentrations in different sample types. Even though the data for TWP in solid samples (Figure 4A) and TWP in water samples (Figure 4B) cannot be compared due to different units and matrices, it clearly demonstrates that TWPs were more prominent in certain matrices than others.

Urban runoff exhibited the highest concentrations of TWP among the analysed water samples (Figure 4B). These were samples taken of stormwater in direct vicinity to roads and traffic areas. TWP concentrations were also high in the sediments from inner Oslofjord, as well as from lake Mjøsa close to Hamar, but low in most of the sediment samples from other locations. Blue mussels from inner Oslofjord, and Skallneset outside Kirkenes showed high TWP values. The WWTP effluent samples as well as the pump samples from surface water in inner Oslofjord, showed low TWP concentrations.

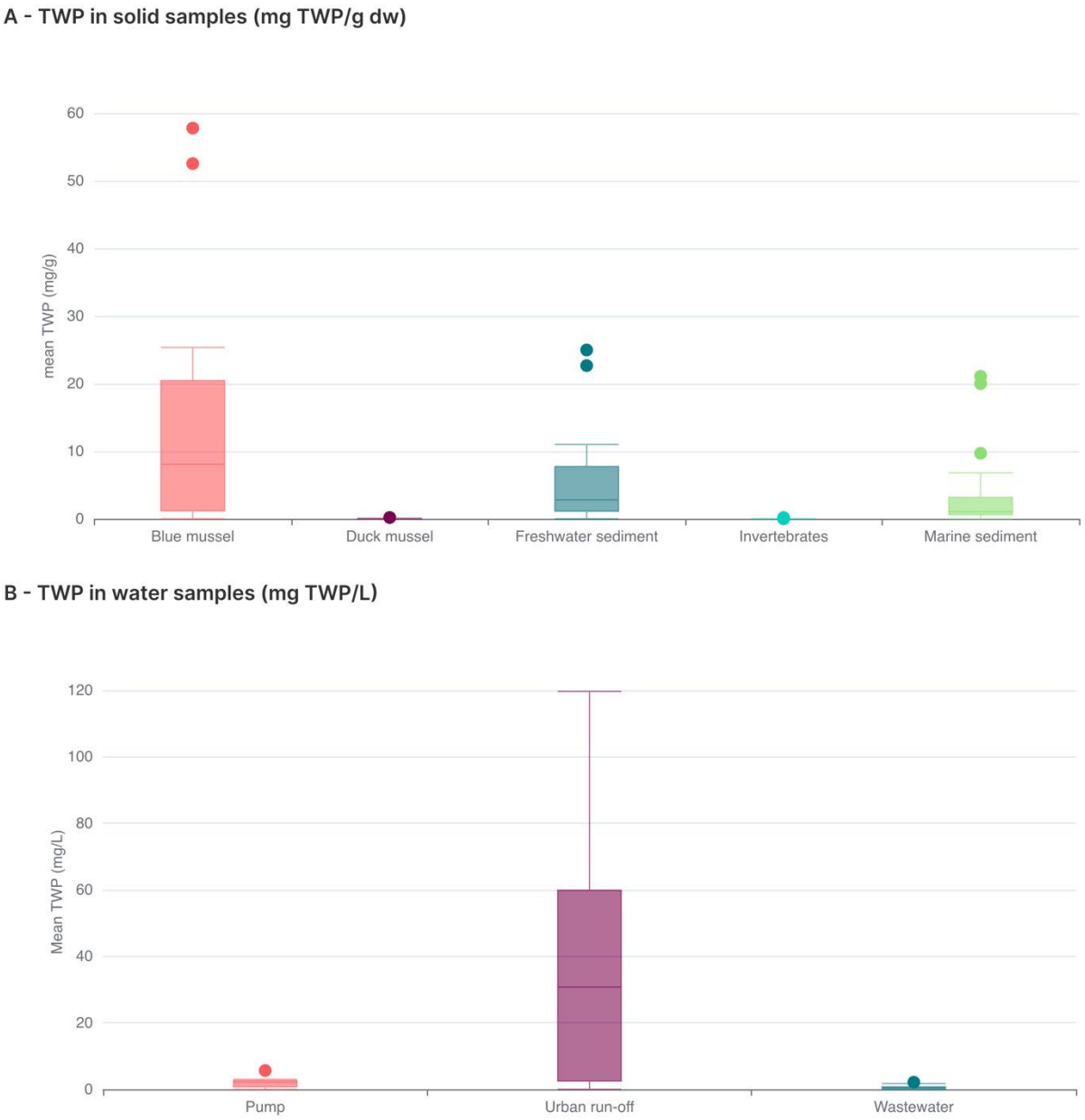

Figure 4. Summary figure of TWP concentrations in different sample types. The results are presented as boxplots of all datapoints of each sample type, with boxes marked with median. Whiskers are showing the 1.5 interquartile range, and the samples that fall outside of this range are marked as points. Panel A shows solid samples (mg TWP per gram dry weight): Blue mussels n = 18, Duck mussels (freshwater) n = 20, Invertebrates (from marine sediments, mainly Polychaetes) n = 47, Marine sediments stations, measured in replicates of three (26 stations, n = 78), and freshwater sediments, measured in replicates of three (16 stations, n = 48). Panel B shows water samples (mg/L): Pump samples (marine water) n = 9, urban runoff samples from Oslo and Hamar n = 12, wastewater treatment plant effluent samples n = 15. Colours for visual effect only.

Sediments

The sediments revealed high spatial variation in distribution of TWP, going from high concentrations close to land, and particularly in the inner Oslofjord, to very low concentrations further away from the coast (Figure 4A and Figure 8). The concentrations of TWP in the sediments of Akershuskaia are similar to previous measurements in environmental recipients (soils) to roads using the same analytical method (Rødland et al., 2023) . In Rødland's study, which investigated the presence of TWP in soils along low-traffic and high-traffic roads in Norway, TWP concentrations were found to range from 2 to 26.4 mg/g

dry weight (dw) (Rødland et al., 2023). Notably, the highest concentrations observed in the inner Oslofjord, around 20 mg/g dw, align with the highest values from Rødland's study (2023), indicating the accumulation of TWP in high concentrations in the fjord sediments. The levels reported in this study is also comparable to TWP levels previously reported for river sediments using PYR-GC/MS, but following a different method (TWP 0.04-7.4 mg/g dw, Unice et al. 2013).

To the best of our knowledge, there are no published data on TWP concentrations in marine sediments, although there are studies investigating the potential sources to the marine environment such as WWTP effluents, surface runoff and atmospheric fallout (Parker-Jurd et al., 2021). For freshwater sediments, on the other hand, there are several studies reporting mass concentrations of TWPs, with levels ranging from 0.05 - 155 mg/g in river sediments and 0.7 mg/g in one study of lake sediments (Goßmann et al., 2021; Klöckner et al., 2019; Kumata et al., 2000; Rauert et al., 2022; Spies et al., 1987; Unice et al., 2013; Zakaria et al., 2002) . These findings collectively highlight the significant presence of TWP in various environments, with implications for pollution and potential environmental impact. Studies of TWP employ a wide range of different analytical approaches, so comparisons between studies should be performed with caution (Rødland et al., 2023)

These patterns with higher concentrations of TWP close to shore likely reflect that the rather heavy TWP are sinking quickly and accumulating close to the shore. Low concentrations in surface water samples confirms the relevance of sinking and sedimentation for the distribution of TWP. In the sediments from inner Oslofjord, the highest concentrations were found in the sediment samples at Akershuskaia. The second-highest concentrations in sediments were found outside Bekkelaget WWTP, though the low concentrations of TWP in WWTP effluent samples from Bekkelaget indicates that there may be other sources. The Bekkelaget sampling station is both close to the outlet from the WWTP and the outlet of runoff from several roads, stormwater pipes, and tunnels in Oslo's inner city. Similar concentrations in urban runoff and sediments to those observed in the inner Oslofjord were also found in the urban runoff in Hamar and in the sediments outside Hamar, for example, at station Mjøsbrua (see Figure 8).

Blue mussels and other biota

The blue mussel samples showed high TWP concentrations in some stations. The Akershuskaia blue mussel samples, with concentrations up to 60 mg TWP/g dw, corresponded to stations where TWP concentrations were also high in sediments (Figure 5-6). Notable, though high concentrations in the blue mussels were found from highly impacted areas (Akershuskaia, inner Oslofjord), concentrations were also high in an area in the Varanger fjord (Skallneset), with unknown sources of contamination (Figure 5). At three stations, however, the concentrations of TWP in blue mussels were low or close to LOQ (Tjøme, Kristiansand havn, Bergen havn). For the benthic invertebrates collected with marine sediments in Skagerrak, the North Sea, Norwegian Sea and Barents Sea, as well as in the duck mussels from lake Mjøsa, the concentrations of TWP were below or close to LOQ of the analysis (here applied as the lowest point on the calibration curve for analysis), which was 0.1 µg TWP/sample for biota and freshwater sediments and 1 mg TWP/sample for marine sediments and water samples. The LOD and LOQ values for biota, water and sediment samples, is described in Appendix 5.1.

Previously, black rubber particles have been observed through microscopic analysis in blue mussels from the Oslofjord, as reported by Bråte et al. in (2018). Black rubber particles have also been identified in mussels from sites of high urban impact in a large Nordic study with samples from Sweden, Denmark, Iceland, as well as Norway (Bråte et al., 2020). However, prior to this study, the concentrations of TWP in blue mussels from Norwegian samples have not been quantified using Pyr-GC/MS. Hence, the specific concentrations of these particles in the mussels from Oslofjord, as well as at other Norwegian sites, were previously not measured.

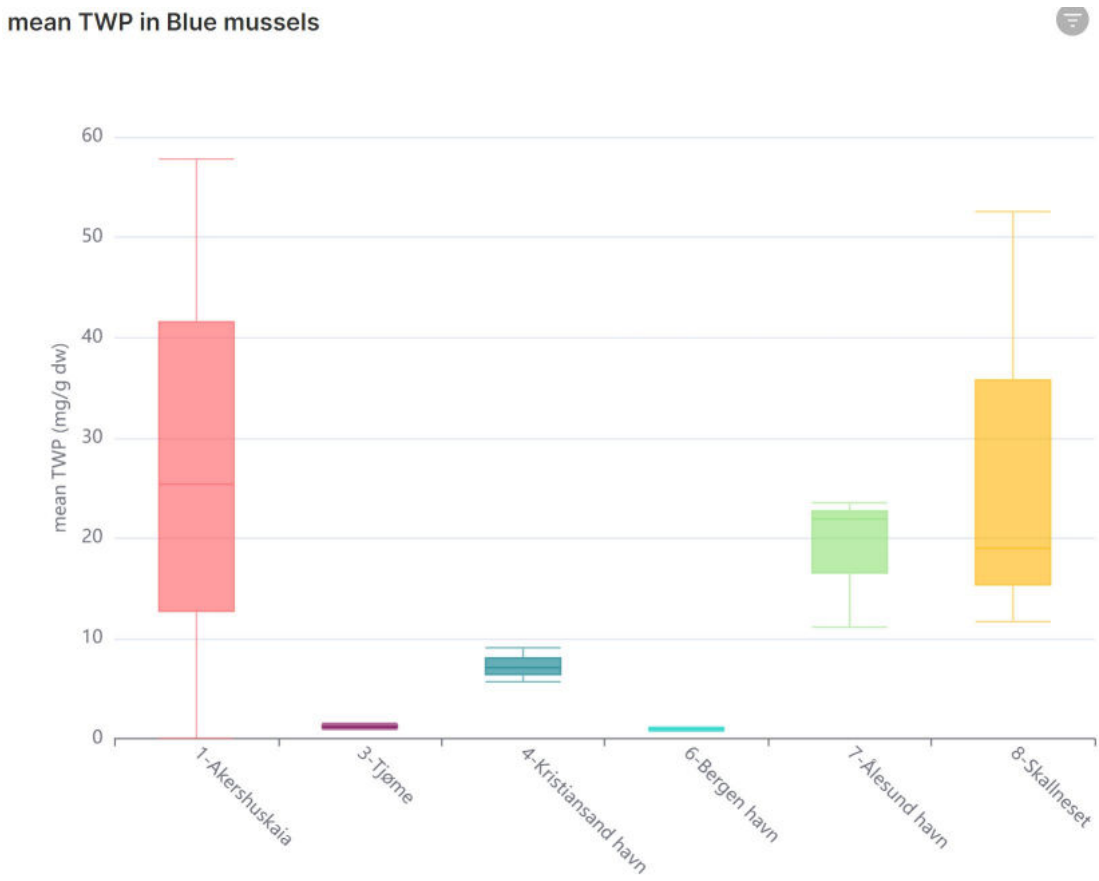

Figure 5. Mean concentrations of TWP (mg/g dw) in blue mussels. n= 3 for all stations.

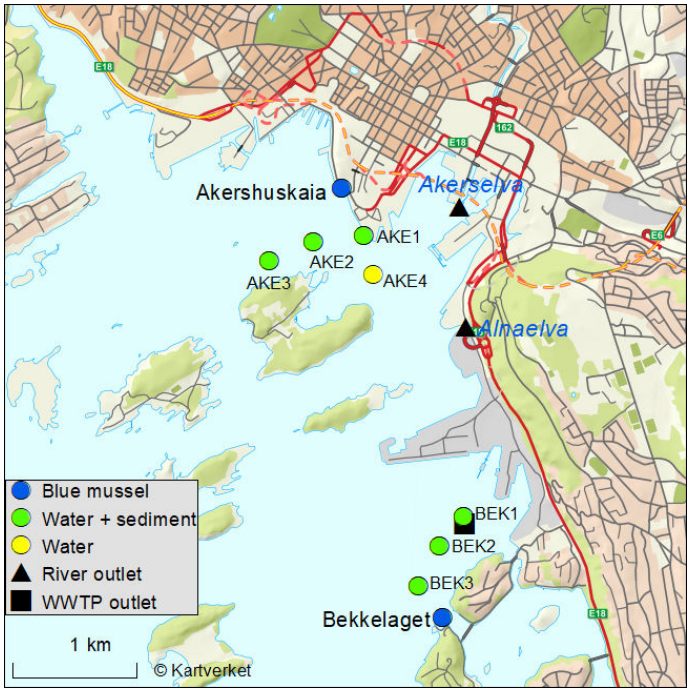

Figure 6. Map showing the biota (blue mussel), sediment and surface water stations for sampling during 2021 and 2022 in the inner Oslofjord. The outlets from WWTP Bekkelaget and rivers Akerselva and Alnaelva are marked in the map.

It is important to note that the biota, water, and sediment samples do not represent the exact same size fractions:

- Sediments underwent sieving through a 500 µm sieve before subsamples were taken for the PYR-GC/MS analysis of TWP. The size fraction analysed for TWP in sediments was, therefore, 0-500 µm.

- Blue mussels were sieved using both a 50 µm and a 300 µm sieve. The size fraction analysed for TWP in blue mussels was, therefore, 50-300 µm.

- Water samples, which include WWTP and urban runoff, were sieved using both a 50 µm and a 300 µm sieve. The size fraction analysed for TWP in water samples was 50-300 µm.

For more detailed method descriptions, please refer to Appendix 5.5 and the information provided on superset.

Reflections on environmental impact of TWP

Considering the high levels of TWPs found in sediments and in blue mussels in this study, potential negative impacts on organisms should be further investigated as car tyre particles have been shown to have a number of negative impacts on the environment. Rubber particles, like other microplastic particles can be ingested by wildlife, which can lead to blockages in the digestive system and other negative effects (Gomes et al., 2022). Tyres also contain a wide range of different chemicals, including PAHs, metals such as zinc and a range of organic compounds. Some of these compounds are added to give the tyres various attributes and for protection against degradation. One of these compounds, 6-PPD, is added as an antioxidant (protection against oxygenation) and antiozonant (protection against ozonation). Other compounds come from the production phase of the tyres, such as Zn used for the vulcanisation process. Studies have demonstrated that many of the compounds added to the tyre can leach from tyre particles into the environment (Müller et al., 2022), thus detectable levels of tyre-related chemicals have been found in water, air and soil samples (Cao et al., 2022; Rauert et al., 2022). Several of the tyre-related compounds, including 6-PPD-quinone (a transformation product coming from the 6-PPD added to tyres) have recently been linked to acute toxicity for several aquatic species (Hiki & Yamamoto, 2022; Tian et al., 2021), including negative impact on sediment dwelling organisms (Garrard et al., 2022).

3.2 Microplastics in sediments

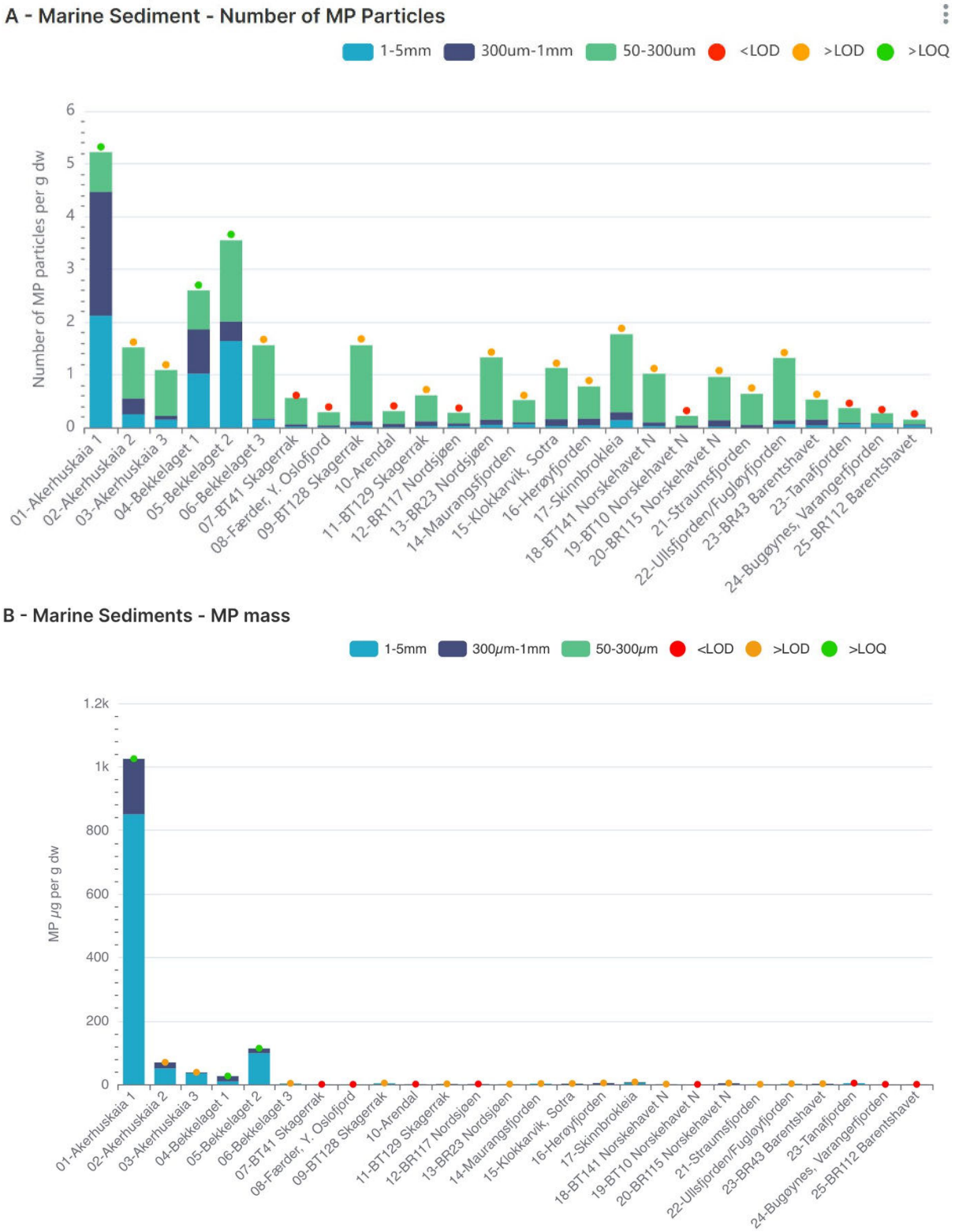

Figure 7. A. The number of microplastic particles (number of MP per g dw), **B.** The mass of the same particles (µg/g dw) The polymer types included. are the 20 polymer types defined by AMAP (2021). In A. and B. the proportion of particles in each size fraction are marked in different colours. The samples are marked with green, orange or red dots, to show whether they are under LOD (red), over LOD but under LOQ (orange), or above LOQ (green).

The results for marine sediments revealed distinct patterns in terms of the number of microplastic particles, their masses, and in TWP concentrations. Figures 7 and 8 provide a comparison across sampling stations of numbers and mass of MP particles and of TWP, respectively. All field blanks (atmospheric) showed consistently low number of particles, well below LOD (see Appendix 5.1)

It is notable that larger microplastic particles were more prevalent in stations closer to the shore and in urbanised areas. This trend became even more pronounced when examining their respective masses, expressed as µg/g dry weight. Additionally, TWP concentrations were highest in the samples collected from the areas around Akershuskaia and Bekkelaget in the inner Oslofjord.

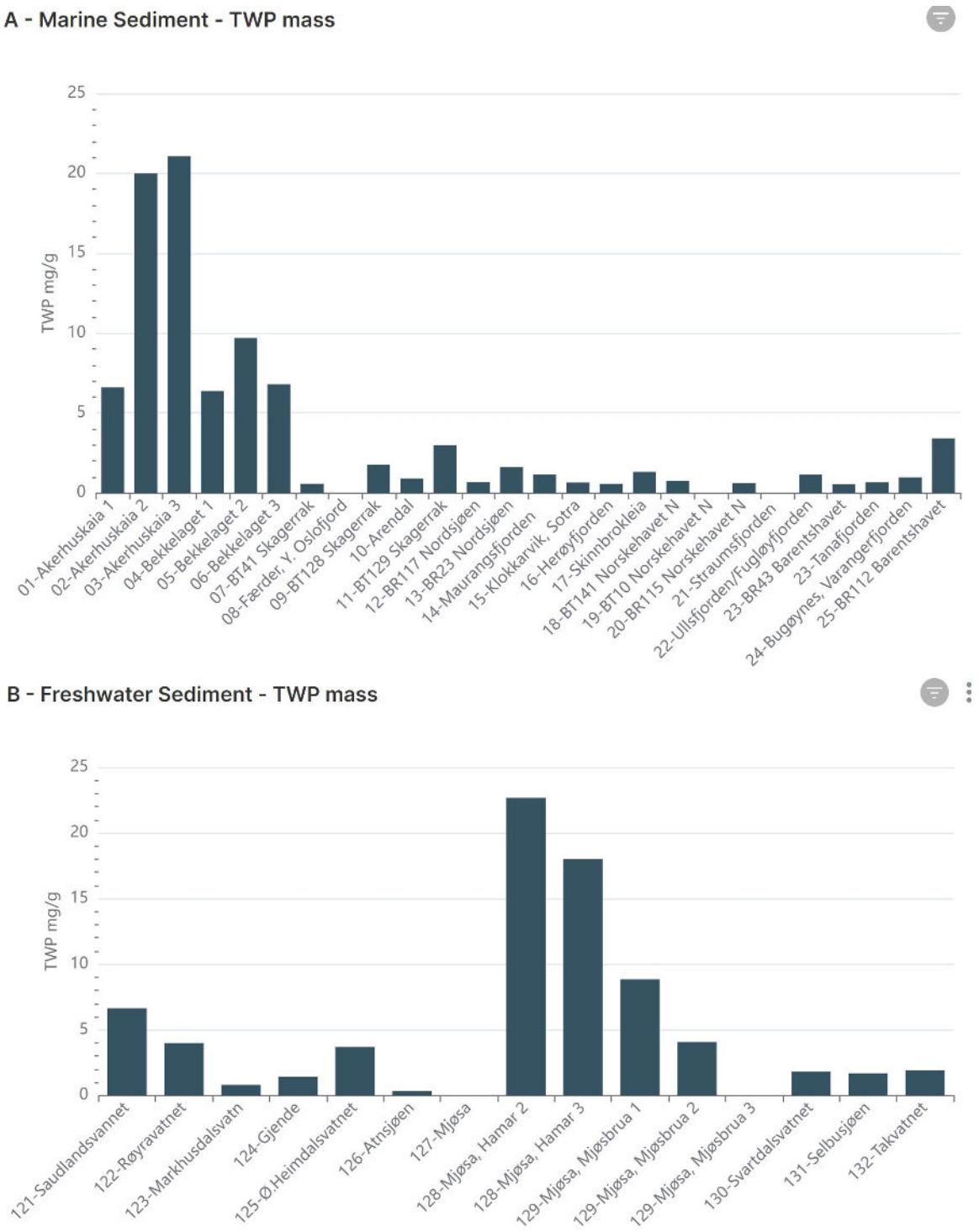

Figure 8. Concentrations of tyre wear particles (TWP) in (mg/g dw) in **A.** marine sediments (corresponding to the same samples as in Figure 7), and **B.** freshwater sediments. In all TWP analyses of sediments, particles between 0-500 µm were included, and only results over LOQ are shown.

Previously published concentration levels of numbers of microplastic particles in sediments displayed similar patterns to our samples. For example, a study by Haave et al., (2019) reported microplastic concentrations ranging from 12 to 200 particles per gram of dry sediment in Bergen Harbour. It is worth noting that Haave et al. measured particles down to 10 µm, with most particles found in the smallest fractions. Even though these numbers are higher than those we found in the sediment samples collected for MIKRONOR, where the number of particles ranged from 0.2 to 5 particles per gram of dry

sediment, 95 % of the particles in Haave et al., were smaller than 100 µm, and 56–70 % smaller than 25 µm. Actual numbers of particles >50 µm were in the same range in Bergen and Oslo harbours. Microplastic particles (not including TWP) have previously been measured with similar detection limits and size fractions in the western Oslofjord outside the WWTP VEAS, showing numbers of microplastics of 0.02 to 1.71 microplastic particles per g dw (Bronzo et al., 2021). These numbers are somewhat lower than measured in this year's study for MIKRONOR. This could be a sign that the Akershuskaia sediment area has a higher input of rapidly sinking microplastic particles than the area outside VEAS WWTP, possibly due to higher input of urban runoff.

MIKRONOR provides data from remote areas. These sediment stations, revealed few particles and even lower mass concentrations of microplastic particles, including TWP. Many of the results are either under or close to the limit of detection (LOD), as shown in Figure 7. From this, sediment samples far from the coast and from potential sources seem to have low levels of microplastics, including TWP.

The calculated masses, based on the size of the microplastic particles, are largely influenced by the larger particles. This can be observed when comparing panel A and B in Figure 7. Notably, the only stations with significant masses of microplastics are those that also contain particles larger than 300 µm. Despite their lower number, the particles exceeding 300 µm, and especially those bigger than 1 mm, constitute the majority of the microplastic mass in the sediments. We estimated mass concentrations by calculating the mass of each individual particle. Therefore, the limits of detection (LOD) and quantification (LOQ) for the masses depended on determining whether a particle was an environmental microplastic (and not contamination). Thus, the LOD and LOQ for the masses were based on whether the number of particles exceeded the LOD/LOQ, respectively. If the samples instead had been analysed with pyrolysis, the results for the mass concentrations would probably have been below detection limits of the analysis for the majority of the samples. If pyrolysis had been the *only* analytical method used, samples containing only small particles (< 300 µm) would be characterised as clean samples. The potential risk that small particles could be posing to the environment would then not have been identified.

3.3 Two potential sources of microplastics in the environment

To explore the differences in the contribution from WWTPs and stormwater from urban runoff, MIKRONOR has included samples from two WWTPs and urban runoff from two cities: Hamar close to lake Mjøsa, and Oslo, with the inner Oslofjord as recipient. Both wastewater effluent and urban runoff samples exhibited the highest value (mass and number) of microplastics in all our samples, with 1000-10 000 times higher concentration of particles than the samples in the water of their respective recipient (see Figure 9 and 10, for water samples in lake Mjøsa, and for inner Oslofjord see Appendix 5.5).

The WWTP HIAS in Hamar showed slightly higher numbers of microplastic particles, as well as mass concentrations, compared to Bekkelaget WWTP in Oslo, with mean number of particles being 2.6 MP/L at HIAS, and 0.9 MP/L at Bekkelaget. This difference is mainly due to one high measurement at HIAS (sample T7, Figure 9). Note that the concentrations are in particles per litre, not m^3 as for coastal and freshwater samples. The concentrations of TWP were low, close to LOQ for many samples. However, slightly higher concentrations of TWP were found in the HIAS samples compared to the samples from Bekkelaget. An earlier study carried out at Bekkelaget (Vogelsang et al., 2020), investigated the presence of microplastics in influent and effluent waters over a year. The study found that a variety of particles and polymers entered the WWTP and that the discharge to the fjord could amount to 1.1 ± 2.3 fibres (>300 µm*excluding TWP) / m^3, when considering the size fraction 20-300 µm discharge was

estimated as 5.1 ± 7.2 µg / m^3. It should be noted that there were methodological differences and challenges encountered in this project limiting the comparison to our present data.

Wastewater effluent seems to be an important source of microplastic particles to the environment, apart from tyre wear particles. However, the levels of microplastics (numbers and mass) in this study varied considerably between the samples. A more comprehensive study should be conducted before strong conclusions are drawn, or yearly fluxes could be calculated with reasonable uncertainties. A comprehensive study should include estimates of how much microplastics are removed from the raw wastewater, and what fractions most microplastics end up in (sludge, filters etc), compared to what is released in the effluent waters. An example of this approach is presented in Vogelsang et al., (2020).

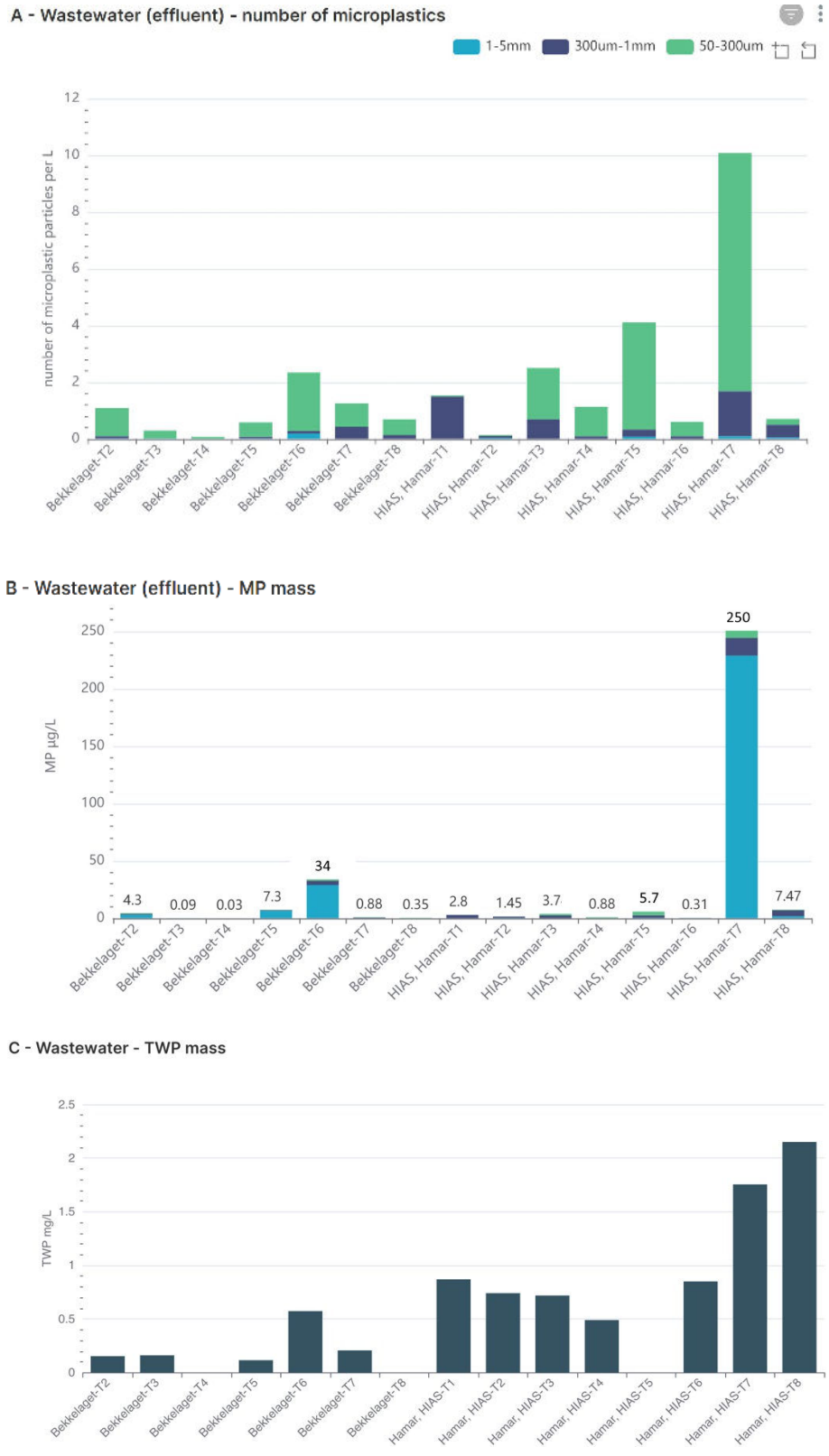

Figure 9. Results from WWTP effluent samples at Bekkelaget WWTP, Oslo, and HIAS WWTP, Hamar. Samples are taken as subsamples of the effluent and named as T1-8. **A.** The number of microplastic particles (number of MP per L), **B.** The mass of those particles (µg/L). In A. and B. the proportion of particles in each size fraction are marked in different colours. In **C**. the concentrations of TWP (mg/L) are shown. All samples marked in the figure are above LOQ. Field blanks exhibited considerably lower numbers of particles than the samples.

Figure 10. Results from stormwater samples of urban runoff from Oslo (Alfaset, Brynseng and Hasle) and Hamar (1-3). **A.** The number of microplastic particles (number of MP per L), **B.** The mass of those particles (µg/L). In A. and B. the proportion of particles in each size fraction are marked in different colours. In **C**. the concentrations of TWP (mg/L) are shown. All samples marked in the figure are above LOQ. Field blanks exhibited considerably lower numbers of particles than the samples.

The urban runoff samples exhibited the highest numbers of microplastic particles (Figure 10A), among the highest mass concentrations (Figure 9B) and by far the highest TWP concentrations (Figure 10C). Urban runoff, here sampled in stormwater drain pits, were expected to have high concentrations of car tyre related particles. Comparing the levels of both TWP and other microplastics in urban runoff to the high levels of microplastic particles and TWP concentrations in the inner Oslofjord, the urban runoff to the fjord stands out as an important source of the microplastics that we found in the fjord. In Hamar, the

sediments exhibited high concentrations of TWP in some of the stations close to Hamar city, consistent with the results found in the urban runoff from Hamar.

3.4 Microplastics and UV compounds in air and deposition– comparison between Svalbard and mainland samples

Microplastic particles were present with concentrations > LOD in 80% of samples from Birkenes and Zeppelin station in 2022. Hereby, average fluxes in deposition samples reached 73.5 µg/m^2/d in Birkenes (median 63.8 µg/m^2/d) and 99.6 µg/m^2/d in Zeppelin (median 18.4 µg/m^2/d, while the corresponding average microplastic concentrations in active air samples were 6.85 ng/m^3 (median 8.14 ng/m^3) and 1.86 ng/m^3 (median 1.98 ng/m^3), respectively. The slightly higher average of MP deposition at Zeppelin was primarily caused by a high polypropylene concentration at one sampling period (426 µg/m^2/d during sampling period 10.11. – 24.11.2022). No indication for contamination in the lab contributing to this elevated datapoint was found. When comparing air trajectories of the relevant sampling periods, we observed that air masses coming from the Norwegian mainland and close to Svalbard resulted in low microplastic concentrations in deposition samples. Samples from periods with airmasses coming from the European mainland on the other hand showed elevated microplastic concentrations (Figure 11).

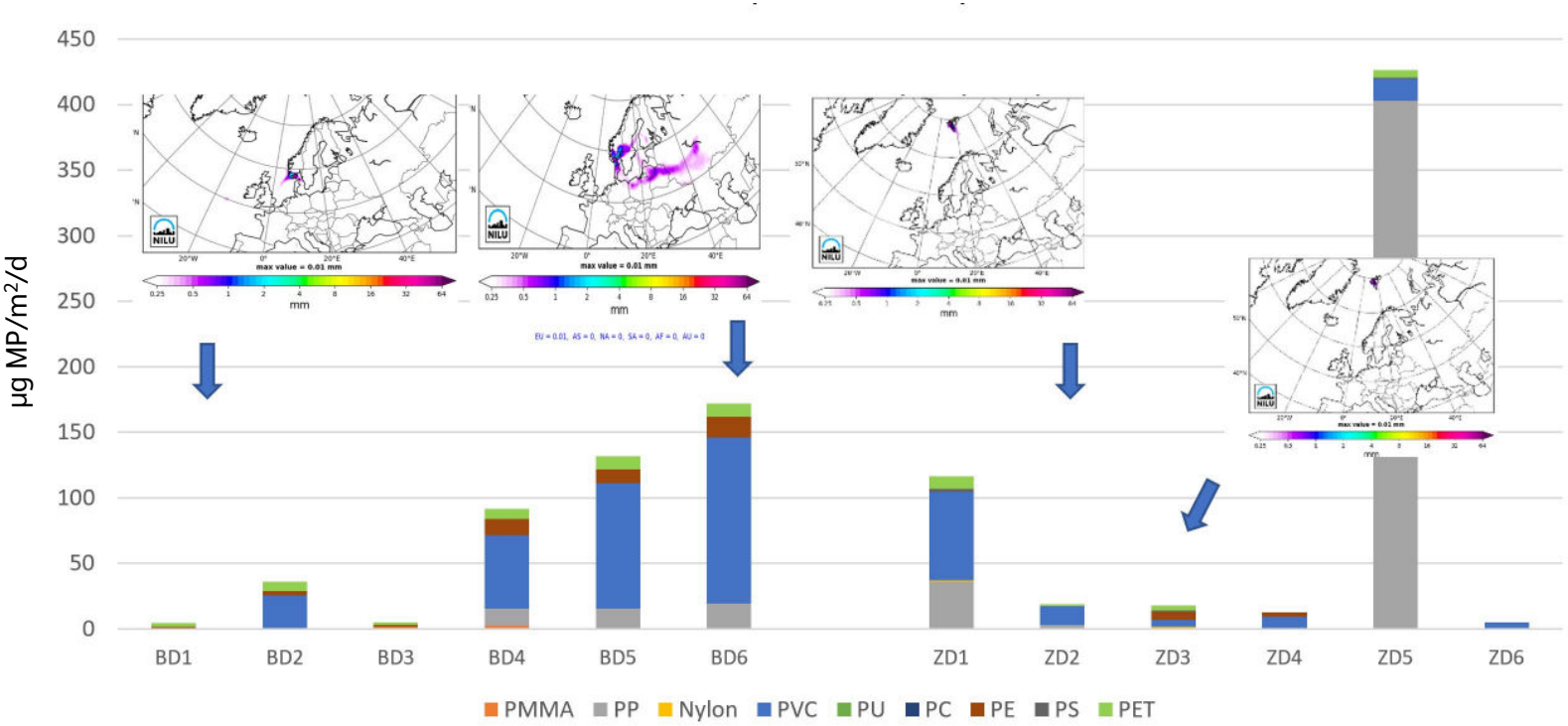

Figure 11. Air trajectories compared to MP deposition in samples from Birkenes station (Agder) and Zeppelin station (Svalbard) at six time periods from August to November 2022 in µg/m²/d.

In deposition samples, PVC (Polyvinyl chloride) and PP (Polypropylene) were the predominant polymers, followed by PE (polyethylene) and PET (Polyethylene terephthalate, polyester) (Figure 11). In contrast, the polymer contribution in active air samples varied considerably between the stations. While PVC, PP and PE dominated at Birkenes, PMMA (Poly(methyl methacrylate)), PET and PP were amongst the most detected polymers at Zeppelin station (Figure 12). Samples exhibiting high microplastic concentrations generally also showed high concentrations of UV compounds. The mean concentrations of UV compounds (including UV-320, UV-326, UV-327, UV-328 and UV-329) reached 0.15 and 0.01 ng/m^3 in active air samples and 20.6 and 20.9 ng/m^2/d in deposition samples from Birkenes and Zeppelin, respectively.

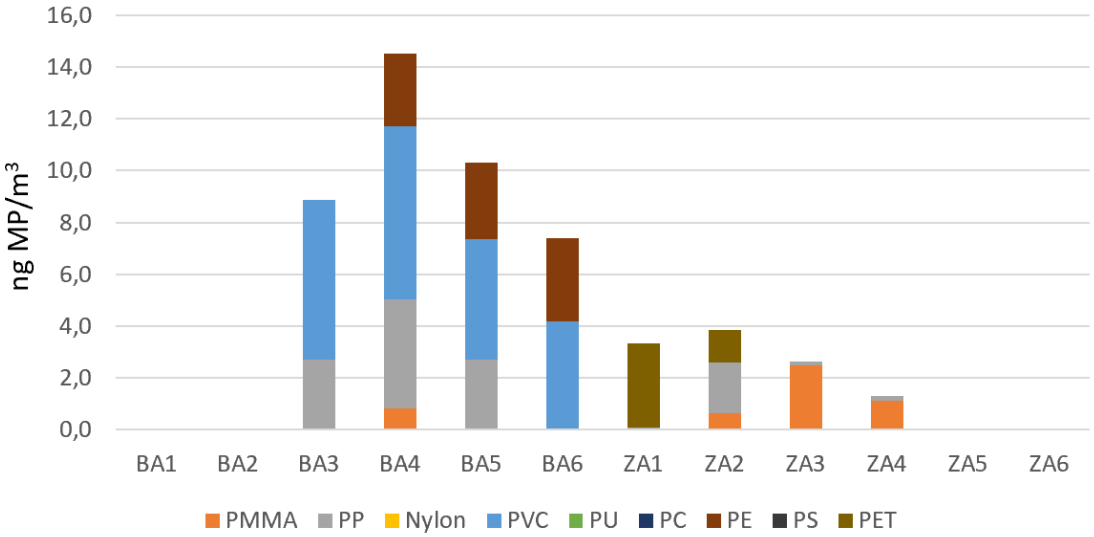

Figure 12. Concentrations of 9 polymer types in microplastic particles sampled with active air samplers at Birkenes (samples BA 1-6) and at Zeppelin station (samples ZA 1-6) at six time periods from August to November 2022.

For comparison with the sparse available literature data, the average concentrations of microplastics in deposition samples from Birkenes and Zeppelin (73.5 and 99.6 µg/m^2/d), were significantly lower than the minimum and maximum concentrations detected in Krakov (0.002-0.01 g/m^2/d) in (Jarosz et al., 2022), where the high concentrations were suspected to be linked to high touristic activity as well as intense construction and maintenance work. Deposition samples were also analysed in Auckland (New Zealand) in 2020, where an average MP deposition rate of 334 ± 81 µg/m^2/d was observed, which is closer to the observed fluxes in our study, with PE, PC and PET being the predominant polymers (Fan et al., 2022). Mizuguchi et al., (2023) detected PP, PS and SBR in the lower ng/m^3 range in active air samples from Tokushima (Japan).

These examples show that while the presence of microplastic particles in atmospheric samples from remote Norwegian areas is concerning, the detected concentrations are clearly lower than those measured in areas with higher anthropogenic pressure.

UV compounds have not been reported in air samples before, besides in MIKRONOR (2022), where like this report, UV-326 and -328 were mostly found. In other matrices, UV 328 and other UV compounds were detected in several samples from the inner Oslofjord marine food web in 2020 as well as the riverine food web in Oslo (M2073, M1509). The concentrations found in this study are comparable to ionic PFAS, but lower than particle bound PAH measured in air at the same stations in 2020 (M-2060, 2021). As for PFAS, we cannot distinguish if the concentrations of UV compounds detected here, are caused by MP vectoring UV compounds to the Arctic or if these compounds are transported adsorbed to other atmospheric particles.

3.5 Polymer types in different matrices

The MIKRONOR samples were analysed for 20 plastic polymer categories described by AMAP (AMAP, 2021) except for the air samples that were analysed with PYR-GCMS for 9 polymer types, as listed in Appendix 5.5 (table 13 and 24). A wide variety of polymer categories were identified in the MIKRONOR samples, and they are presented as relative contributions of different polymer types found in samples in the following figures (Figure 13-16). The figures do not include TWP. TWP was measured as concentrations of total mass content of tyre wear derived particles, and can not be combined or directly compared with the number of particles of certain other plastic polymer types. Polymer types found in two potential sources of microplastics to the aquatic environment, urban runoff and wastewater effluent, are presented in Figure 13. Figure 13 also shows the polymer types found in atmospheric blanks, (260 atmospheric blank samples). The pie charts are based on the count of particles of respective polymer type (not based on mass). The particles found in wastewater effluent and urban runoff samples had slightly different relative contribution of polymers, where effluent waters had higher proportion of polyethylene-based particles, while urban runoff had the highest proportion of polypropylene-based particles. The relatively high contribution of black rubber particles in the urban runoff is also notable. It is consistent with the high concentrations of TWP found in the samples. Very few particles were found in the atmospheric blanks (mean 0.7 ± 0.9, n= 260 samples). The polymers found in the atmospheric blanks included polyethylene (a common plastic type used in packaging), polyester and cellulose acetate commonly used in clothing.

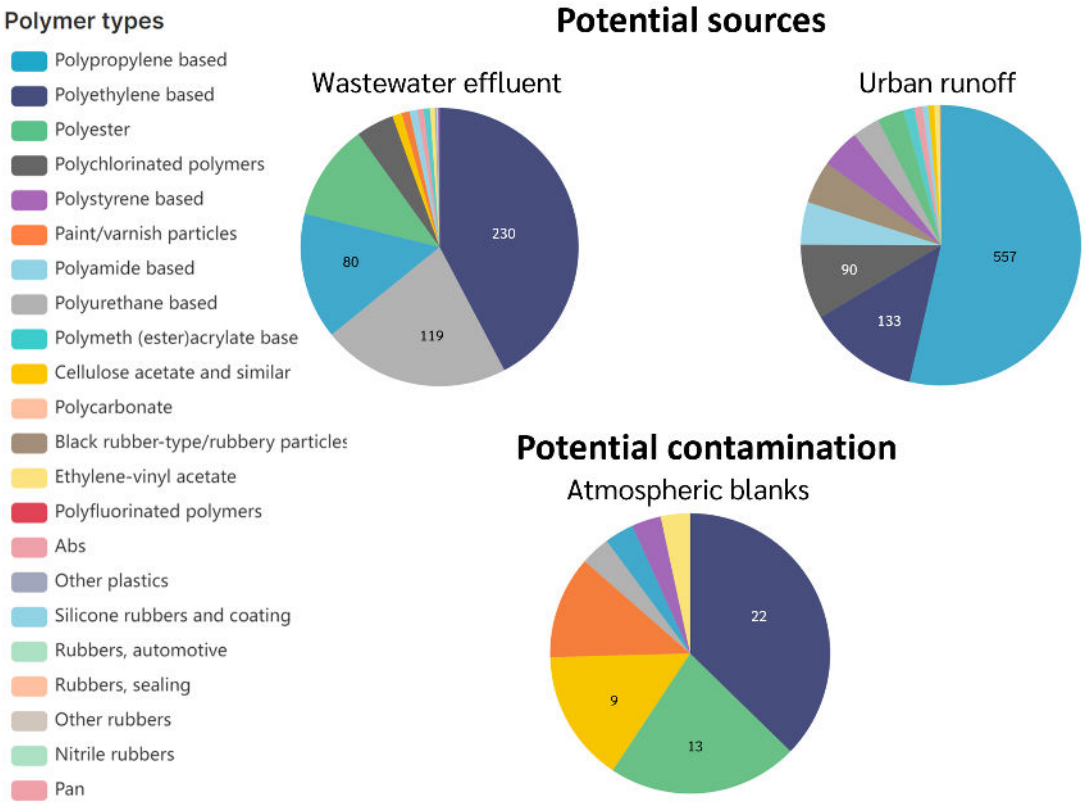

Figure 13. Polymer composition of particles found in samples from the two potential sources (wastewater effluent, urban runoff) of microplastics to the aquatic environment. As comparison, the potential contamination from air during sampling is shown as the results of field blank samples combined (atmospheric blanks). Numbers in the pie charts represent the number of particles of respective polymer type. Number of samples included in each figure is: Wastewater = 15 samples, Urban runoff = 12 samples Atmospheric blanks (n=260). Numbers in the pie charts represent the number of particles of respective polymer type found.

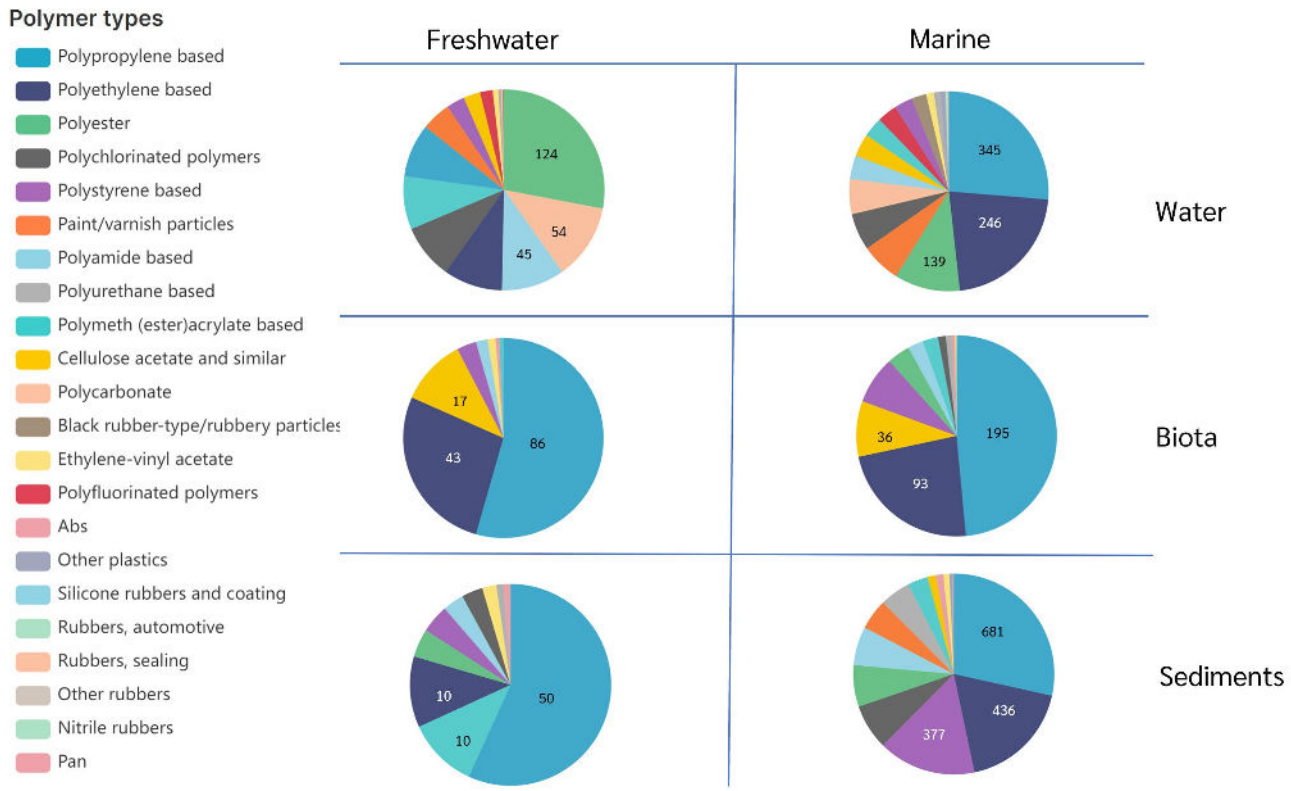

Figure 14. Number and polymer composition of particles found in samples from different matrices (water, sediment, biota) and environments (marine and freshwater). Numbers in the pie charts represent the number of particles of respective polymer type. Number of samples included in each figure is: freshwater water samples = 93, Marine water samples = 164, freshwater biota samples = 20, marine biota samples = 136, freshwater sediments = 16 samples and marine sediments = 66 samples.

Freshwater samples contained more polyester particles compared to marine water samples, which in turn were richer in polypropylene particles. It should be noted that all fibres were excluded from plankton net samples, before analysing the relative contribution of different polymers, due to contamination of fibres in most of the water samples taken with plankton nets. In contrast, the biota samples, including samples from both marine waters and freshwater (blue mussels, invertebrates from sediments, and duck mussels), predominantly contained polypropylene particles.

The air samples showed a higher relative contribution of a few polymer types (Figure 15), compared to the other MIKRONOR samples. One explanation to this might lie in the difference between analysing for found polymer types by mass (as in the air samples) and by number (as in the other MIKRONOR samples), and conclusions based on other variables should be performed with caution. The samples from Birkenes station (Agder) were dominated by polychlorinated polymers in both active air samples and deposition samples. In contrast, the samples from Zeppelin station showed different contribution of polymer types in active air and deposition samples, with the active air samples dominated by polyester (also known as PET) and microplastics in deposition samples dominated by polypropylene. However, polychlorinated polymers relative contribution in both sample types from Birkenes, and also second highest contribution to deposition samples at Zeppelin, were not a polymer type with a high relative contribution in any other MIKRONOR sample types.

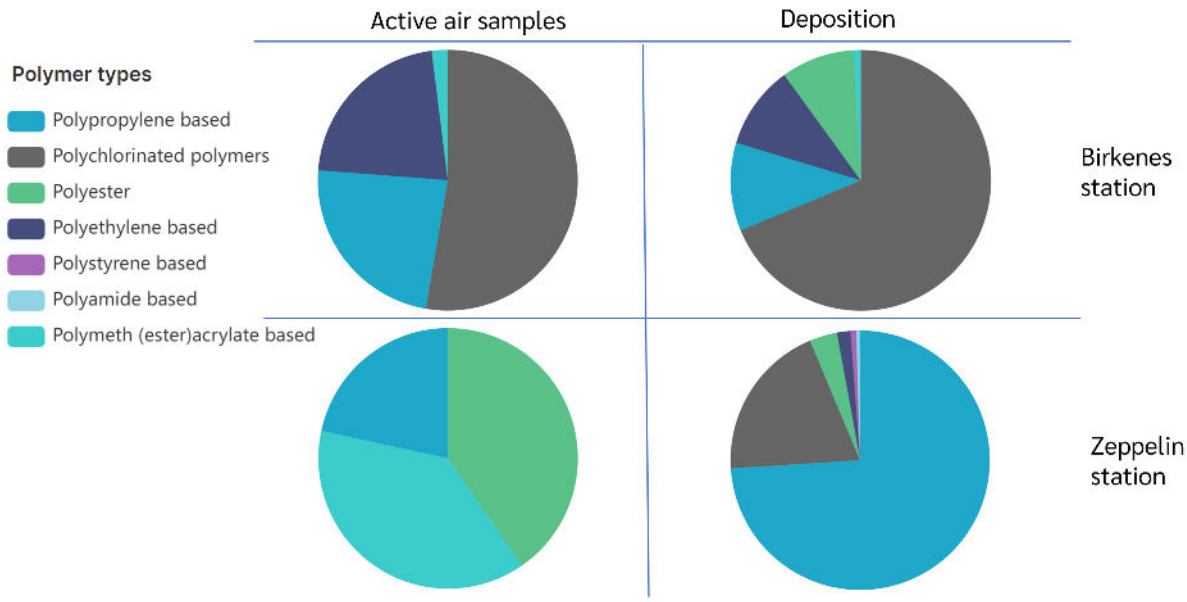

Figure 15. Polymer composition in the air samples (active sampling and deposition) at Birkenes station (Agder) and Zeppelin station (Svalbard). Note that the samples are analysed with PYR-GCMS and the figure represent the mass percentage.

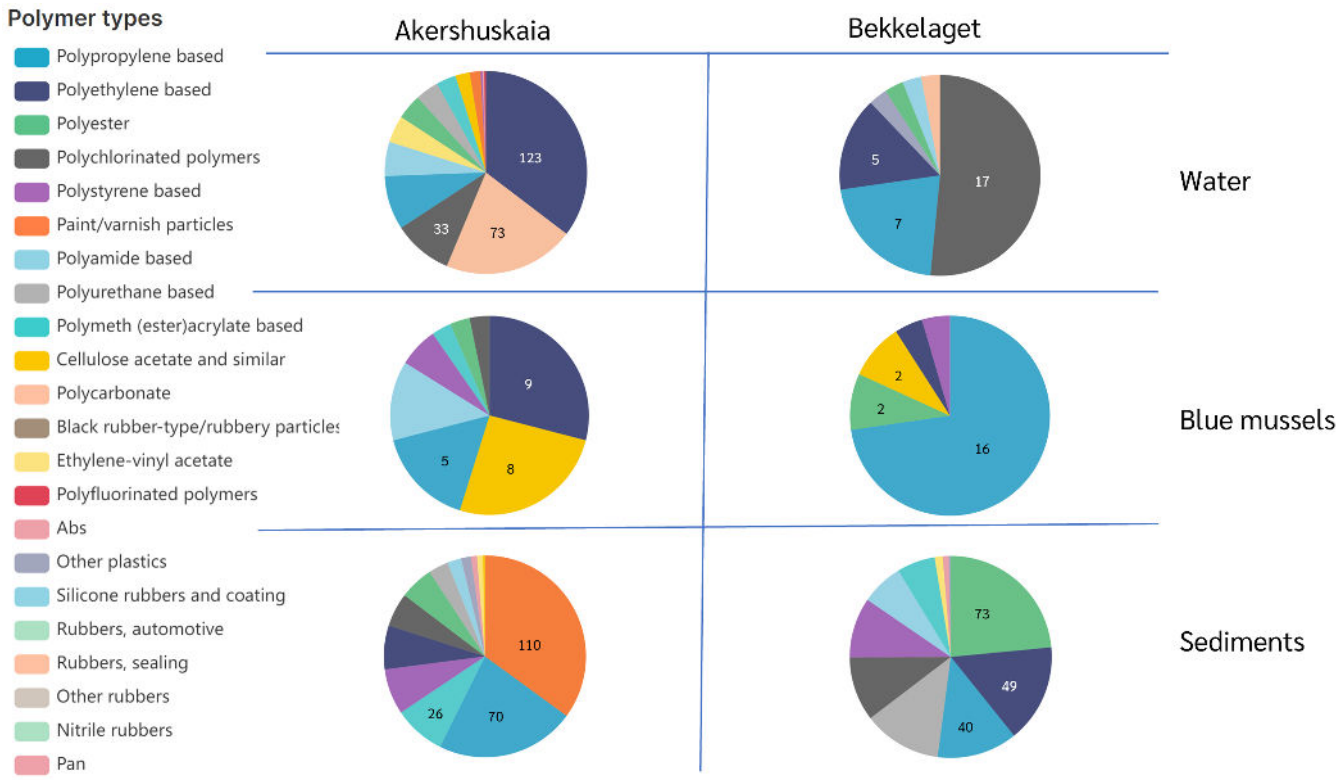

Figure 16. Polymer composition of particles found in samples from Akershuskaia and outside Bekkelaget WWTP. Results are presented for surface water pump samples (Akershuskaia sample n =18, Bekkelaget n = 9), sediments (Akershuskaia sample n = 3 Bekkelaget sample n = 3), and blue mussels (sample n = 13). The stations included are marked as Akershuskaia and AKE 1-4, and BEK 1-3 in the map in Figure 6.

Short summary on polymer types

Overall, polypropylene was the most common polymer type in MIKRONOR samples. Notably, it also predominates in the samples collected from urban runoff. However, polypropylene is not the most

common polymer type in the wastewater treatment plant (WWTP) effluent samples. Polypropylene is among the most widely produced and utilised plastics on a global scale (Lusher & Pettersen, 2021).

It is worth highlighting that the sediment samples from Akershuskaia stood out due to their elevated quantities of paint particles, likely originating from local sources in the harbour area. In contrast, neither the urban runoff samples nor any samples from the Bekkelaget transect exhibit high concentrations of paint particles.

It is essential to exercise caution when interpreting the comparison of different polymer types across various matrices. The number of particles found in sediment samples is higher than the number found in blue mussels, as illustrated in Figure 14. These variations in sample sizes and particle counts should be considered when drawing conclusions from the data. Nevertheless, the data presented here clearly show that different polymers distribute differently in the environment and across matrices.

Polymer types found in water and sediments from inner Oslofjord

We conducted the same analyses exclusively for the samples within the inner Oslofjord, focusing on Akershuskaia and the area close to the outlet of Bekkelaget WWTTP (Figure 16). When compared the two areas, both situated in the inner part of Oslofjord (stations in Figure 3 and 6), there are pronounced differences between the polymer composition in samples from Akershuskaia and outside Bekkelaget.

The sediments from Akershuskaia had considerable numbers of paint particles. Paint particles were not found in any considerable numbers in the sediment samples from outside Bekkelaget, indicating a local source of paint particles in the sediments from Akershuskaia. Polyester was the most common polymer in the sediments from outside Bekkelaget, polyester is often associated with synthetic textiles. However, polyester was not one of the three most common polymers in the wastewater effluent samples (Figure 13). Polyethylene was the most common polymer type in the water samples from Akershuskaia, as well as the second and third most common polymer type in the sediment and water samples from Bekkelaget. However, polyethylene did not have a high relative contribution to the sediment samples from Akershuskaia. The WWTP effluent samples (Figure 13) were dominated by polyethylene-based and polyurethane. Polyurethane was a common polymer type in the sediment samples from Bekkelaget, indicating the WWTP as a potential source to these particles. Polyurethane was not a common polymer type in any other samples from neither Bekkelaget nor Akershuskaia, nor was it a common polymer in the overall analyses of the samples.

Polymer types found in blue mussels from inner Oslofjord

While polypropylene dominated as the overall most common polymer type in blue mussels from Bekkelaget, blue mussels from Akershuskaia displayed higher numbers of polyethylene and cellulose acetate particles. Note that, across all biota samples, polypropylene remained the most prevalent polymer type (Figure 14).

Polypropylene also emerged as the most common polymer type in the urban runoff samples, but only the third most common type in the WWTP effluent samples. This suggests that the composition of microplastic particles in blue mussels does not align with the hypothesis that the Bekkelaget station is influenced by WWTP effluent water while Akershuskaia better reflects urban runoff. However, it's essential to acknowledge that the MIKRONOR dataset is not exhaustive, and small local variations in currents and land-based releases may not be fully represented by just these two potential sources. Moreover, the findings are consistent with those of Bråte et al. in (2018), who also identified polypropylene as the most common polymer type in blue mussels.

3.6 Reflections on monitoring program approach and data quality versus international guidelines

Microplastic research is still very much in the development phase of the research field (Aliani et al., 2023). However, this cannot be seen to hinder the establishment of a sustained national monitoring program to try to create baselines and produce data for future assessments and government mitigation measures (Lusher & Primpke, 2023), as well as data input for international guidelines and conventions such as the global plastic treaty (Aliani et al., 2023; Rognerud et al., 2023) and the Basel convention (Basel Convention, n.d.).

After three years of sampling and currently two annual reports from MIKRONOR, there is now a basis established for adjustments and improvements. Considerable effort has been directed into method development. Method optimisation is still needed for both sampling and for analysis, to ensure targeted monitoring and cost-efficient implementation. Most importantly, the monitoring program will benefit from improved design to align with a more targeted approach (baseline, trends, source tracking, environmental impact etc.), building upon the results and experience from the first years of sampling and analysis.

In addition to the overarching adjustments needed to ensure a sampling design fitting to the aim of the monitoring, as well as keeping the program's data outputs aligned with international guidelines and reporting, the following recommendations for future microplastic monitoring are highlighted:

1. **Continue analysing TWP in sediment and blue mussel samples.** It's essential to maintain a focus on tyre wear particles and related contaminants, such as 6-PPD-quinone, in MIKRONOR as well as other monitoring programs, like Urban Fjord.

2. **Prioritise taking a representative and statistically valid number of samples, rather than covering too many areas or sample types.** Prioritizing the quality and representativeness of samples can lead to more certain and meaningful results.

3. **Microplastic particle analysis will require further refinement to ensure reproducible data outputs.** Any changes in methods along the analytical chain are likely to interfere with data analysis, both between sample types and matrices, as well as compromise spatial and temporal trends. Continued assessments should consider implications for the program.

4. **Data architecture and statistical analyses.** It is important to recognise that microplastic datasets are three-dimensional due to the unique characteristics of each particle. This necessitates a different approach to data handling compared to traditional monitoring data. Allocating adequate resources to build robust and comparable datasets between monitoring years is crucial. There are several ongoing international efforts and MIKRONOR can both learn from and contribute to shared goals.

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5 Appendix

5.1 QA/QC

This chapter includes a description of quality assurance and quality control (QA/QC) applied throughout the project, except for tyre wear particle analysis (TWP) using pyro-GCMS, where the QA/QC is described in Appendix 5.3. Two types of blanks are included in this chapter: field blanks and laboratory blanks. This chapter also contains information about how we have calculated LOD and LOQ, and the results from new recovery tests.

Field blanks

Atmospheric blanks

Atmospheric blanks were used during field sampling to account for any microplastic contamination that may have occurred during the sampling process due to deposits from the surrounding air (Figure 17). The air at the sampling site could have been contaminated with particles from the clothing and skin of the sampler, as well as from other sources (boats, equipment onboard etc.) at the sampling site.

Atmospheric blanks were taken together with the following sample types:

- Manta nets
- Plankton nets
- Sediment samples
- Invertebrates[1]
- Ferry box samples

Net blanks

To mitigate contamination arising from the nets used for sampling (plankton nets, newton nets and manta trawls), a net blank was conducted following net cleaning. This net blank was taken by attaching a freshly cleaned cod-end (the cup collecting the sample at the end of the net) and flushing the net multiple times (a minimum of four) from the outside with a seawater hose to transfer its content into the cod-end. Subsequently, the material from the cod-end was moved to a sample glass using RO-water.

During the laboratory analysis, the net blanks were analysed before the samples to get an indication of potential net-related contamination. In cases where a net blank contained more than 50 fibres, based on experience with MIKRONOR net samples, we know that this is a clear sign of contamination, and the number of fibres in the sample will not be of any scientific value. The fibre counts for all samples connected to that net blank were recorded but not included into further analysis. However, any fragments present in the sample were still counted and analysed. When a net blank contained less than 50 fibres, a comprehensive count and analysis of all fibres and fragments were conducted.

The next stage in quality control involved comparing the fibre count (and any other microplastics if necessary) in the net blank with the count in the environmental samples. Following international recommendations, if the net blanks displayed a higher or equivalent fibre count compared to the environmental samples, fibres in the environmental samples were not reported and considered contamination from the net (Montoto-Martínez et al., 2022; Ryan et al., 2020). It is worth noting that most of the plankton net samples collected in 2022 had a higher fibre count in the net blanks than in the

[1] *Sometimes atmospheric blanks for invertebrates were the same as the sediment atmospheric blanks – taken as parallel samples*

environmental samples. Consequently, all fibres in plankton nets, as well as Neuston nets from Svalbard were excluded from the reporting of the 2022 sampling. This action was taken to ensure an accurate representation of the quantity of fibres present in the environment, thus avoiding both underestimation and overestimation.

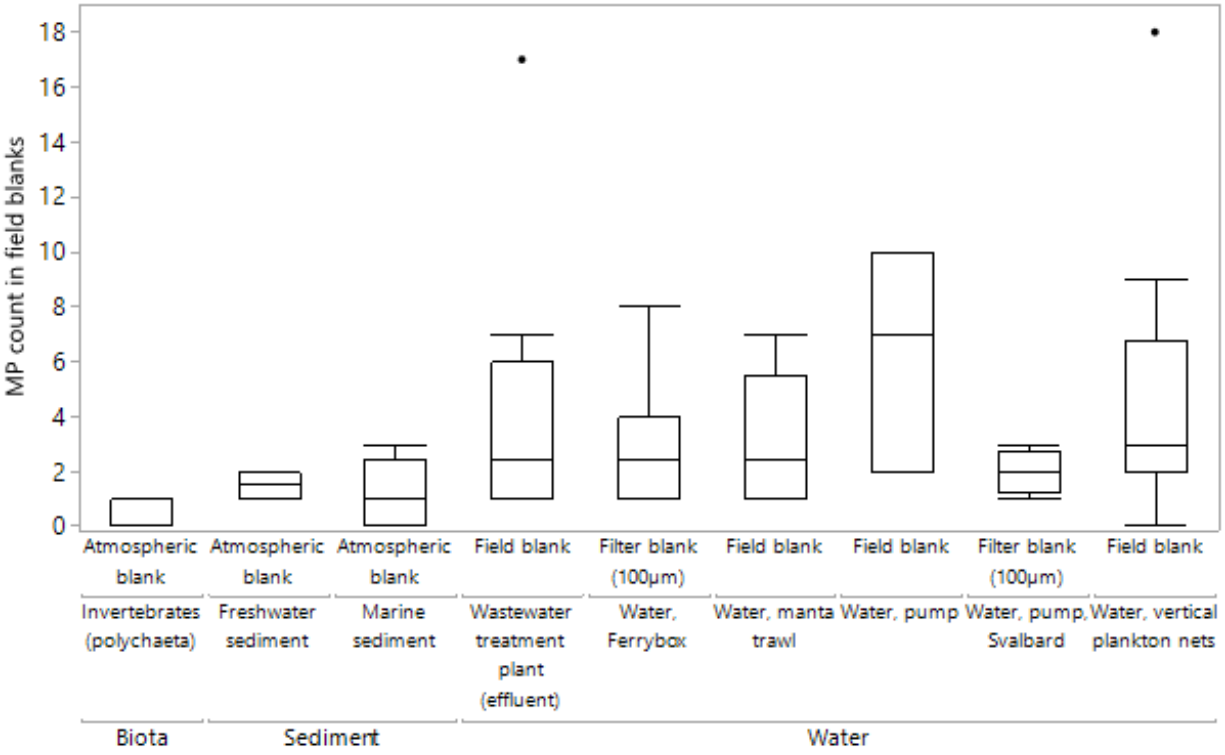


Figure 17. Box plot of number of microplastics particles in the field blanks for each sample type.

Laboratory (Lab) procedural blanks

Laboratory (lab) procedural blanks monitor potential contamination that may occur during processing and analysing the samples in the laboratory. Particles in the lab blanks might come from airborne contamination (such as ventilation and clothes), equipment and chemicals used to process the samples. In all our methods, each batch of samples was accompanied by ca. three lab blanks, consisting of 200 ml RO water that were treated in the same way as the environmental samples . One batch is defined as samples that are processed on the same day(s). The number of environmental samples analysed within a single batch differ between sample types as the methods for sample processing differ in complexity and time used. Number of samples in one batch may also differ within each sample type.

We have in total analysed 58 lab blanks, collected from the analysis of the different sample types (Table 5).

[2] *In a few batches, a lab blank has been lost in the procedure.*

Table 5. Number of lab blanks per sample type.

Sample type	Number of lab blanks
Blue mussel	3
Duck mussel	3
Invertebrates (polychaeta)	9
Water, pump, Svalbard + neuston, Svalbard	2
Water, Ferrybox	10
Water, pump	2
Freshwater sediment	3
Marine sediment	5
Water, urban run-off	3
Water, vertical plankton nets + manta trawl	15
Wastewater treatment plant (effluent)	3

Summary of sample analysis quality control using blank analyses

1. **The field blanks** were analysed as part of QA/QC to check that the sampling situation had not caused any significant contamination of the samples. Procedure:
    a. The number of particles and the particle/polymer type in the field blank were compared to those in the environmental samples taken in parallel. If the number of particles in the field blank for a station were higher or equal to the number of particles in the samples, the sample must be regarded as contaminated and not to be further analysed.
        i. If the number of fibres in the field blank were higher or equal to the numbers of fibres in the sample, fibres were excluded from further analyses. Fibres are a well-known contamination problem in most net samples, where international recommendations are to exclude fibres when analysing net samples (OSPAR protocol or MSFD guidelines in progress).
        ii. If there were microplastic particles present in both field blanks and samples that were matching in all characteristics: shape, approximate size, colour and polymer type, these particles could be excluded from the samples[3].
        iii. If potential sources to contamination of samples were identified, such as textiles, ropes etc, those were analysed and compared with particles in the samples and blanks. If there was a match, the similar particles in the samples were excluded[4].
2. **The laboratory blanks** (three per batch of samples processed) were analysed and used to:
    a. QA/QC to check that the treatment used in the laboratory had not caused any unusually high contamination of the samples.
    b. Calculate LOD/LOQ for sample type as described in next chapter (as number of microplastic particles in the analysed sample, not normalised against volume or weight).
3. The number of microplastics particles in each analyse were compared to LOD/LOQ established for each sample type (MP/sample figures 19-25 in this appendix)
    a. Samples over LOQ are marked with a green dot in the main report figures.
    b. Samples over LOD but under LOQ are marked with an orange dot in the main report figures.
    c. Samples under LOD are marked with a red dot in the main report figures.

[3] *This was never the situation in the MIKRONOR samples analysed in 2023. It did occur in one ferrybox sample analysed in 2022 (see MIKRONOR report, 2022)*

[4] *Not the case in any MIKRONOR samples so far*

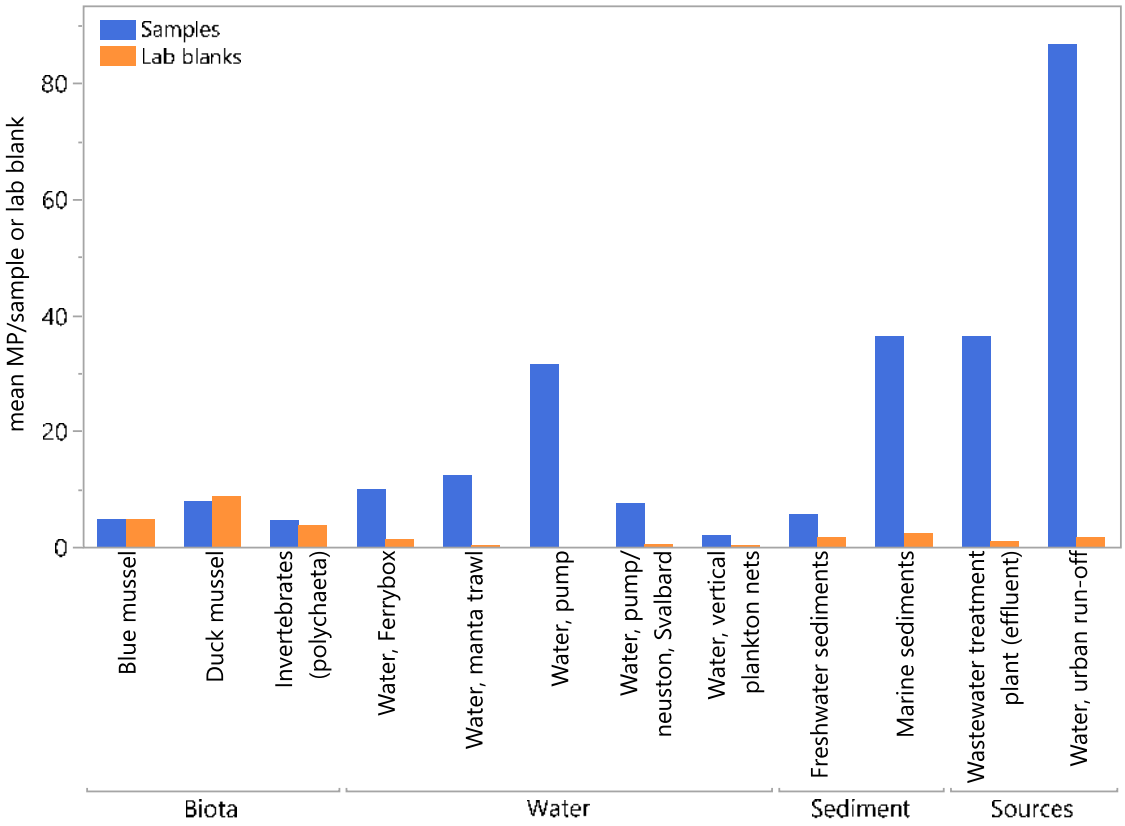


Figure 18. Mean count of microplastic particles in various sample types vs. mean count in respective lab blanks.

The difference in number of particles in lab blanks between sample types reflects mainly the time and complexity of processing different types of samples (Figure 18). For example, the biota (blue mussels, duck mussels and invertebrates) samples were exposed to possible contamination for longer time at the lab bench, since they had to go through more steps of cleaning and processing than many of the water samples, such as the pump samples and the plankton net samples. Compared to the sediment samples, the count in each biota sample was lower, and so closer to the laboratory blanks for that sample type. This is a well-known situation as described in Noonan et al. (2023).

Calculation of LOD/LOQ from lab blanks

The lab blanks in MIKRONOR were used to calculate the LOD and LOQ for the different sample types, as follows:

- LOD: the mean number of microplastics in the lab blanks for that sample type + 3 x SD
- LOQ: the mean number of microplastics in the lab blanks for that sample type + 10 x SD

Biota and some of the sediment samples were found to have number of particles below/in the same range as LOD and/or LOQ. The lab blanks for the water samples had very low number of microplastics, resulting in low LOD/LOQ. The results for all environmental sample types are plotted and shown together with the calculated LOD and LOQ (as dotted lines) in Figures 19-25.

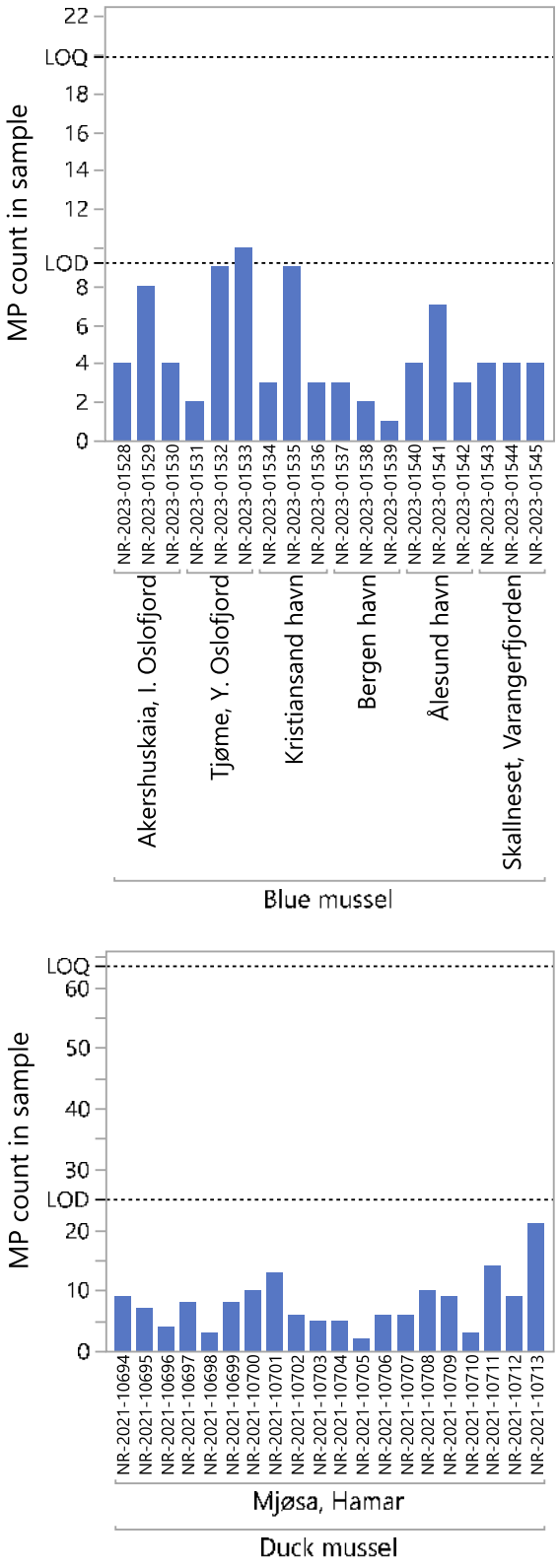


Figure 19. MP count in samples of blue mussel (top) and duck mussel (bottom). LOD and LOQ indicated by dotted lines.

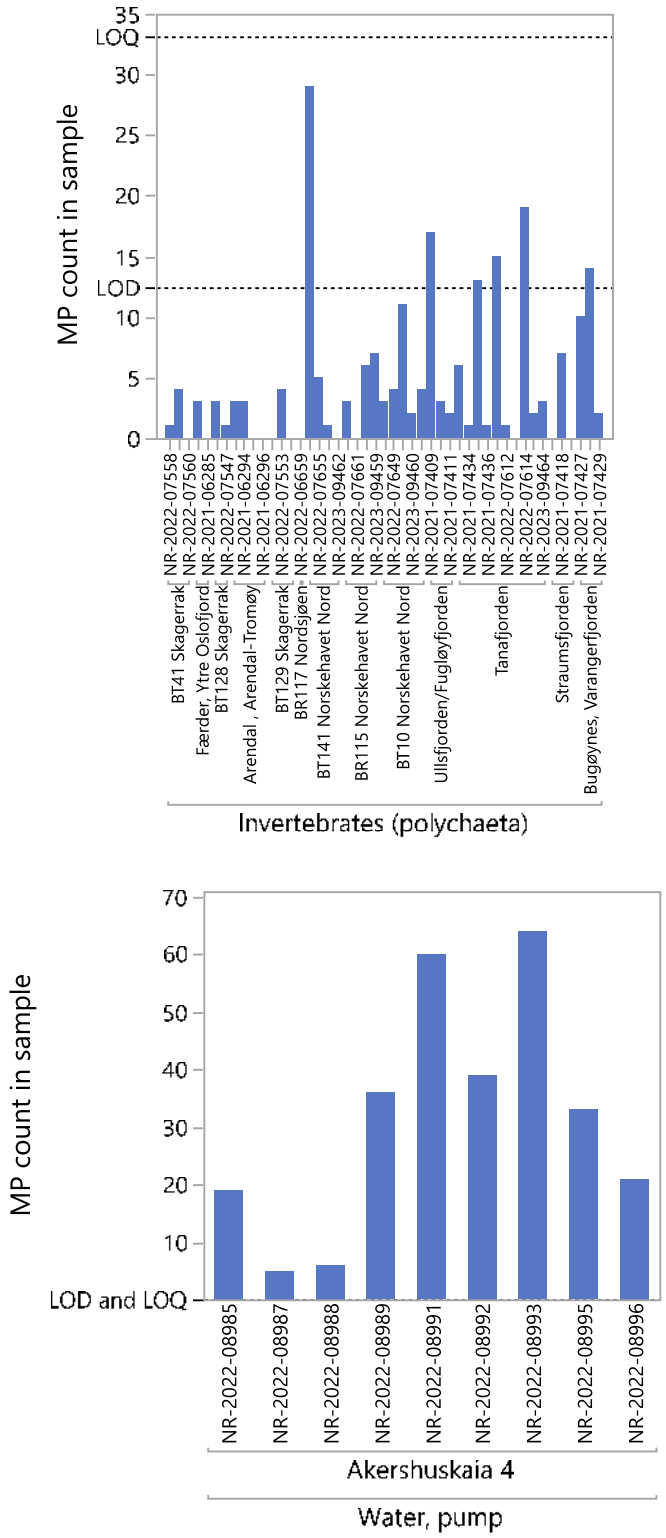


Figure 20. MP count in samples of invertebrates (top) and water, pump (bottom). LOD and LOQ indicated by dotted lines.

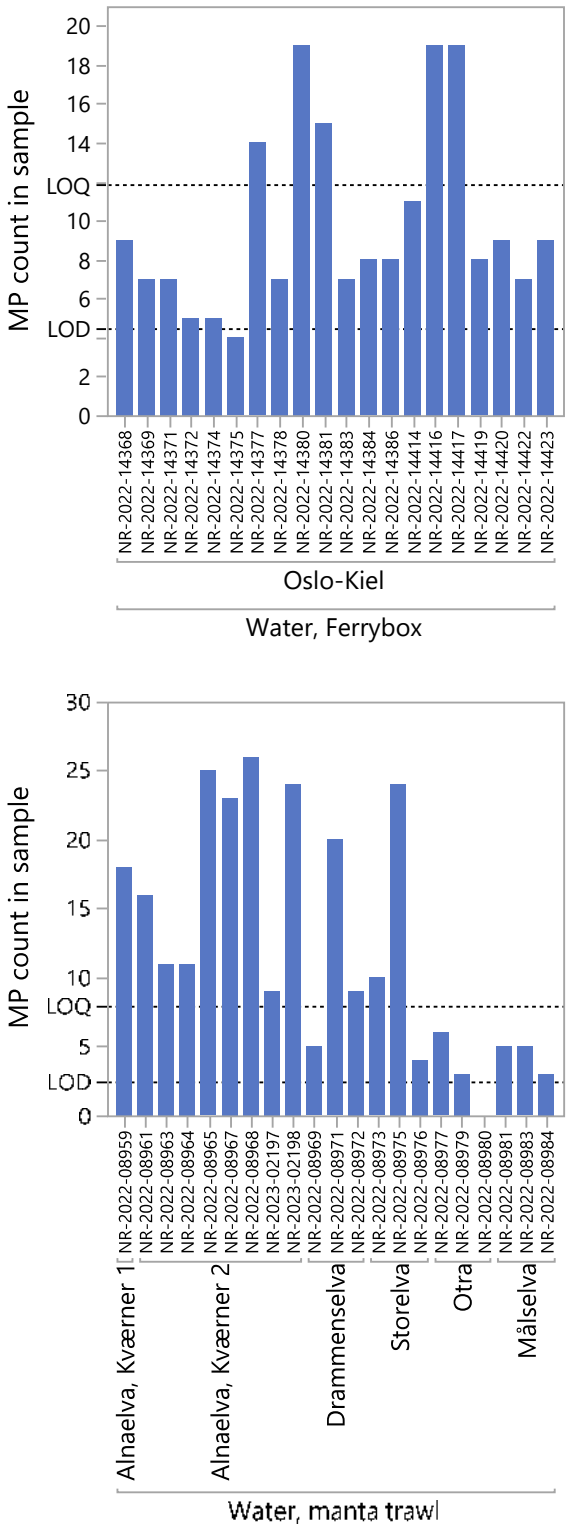


Figure 21. MP count in samples of water: Ferrybox (top) and manta trawl (bottom). LOD and LOQ indicated by dotted lines.

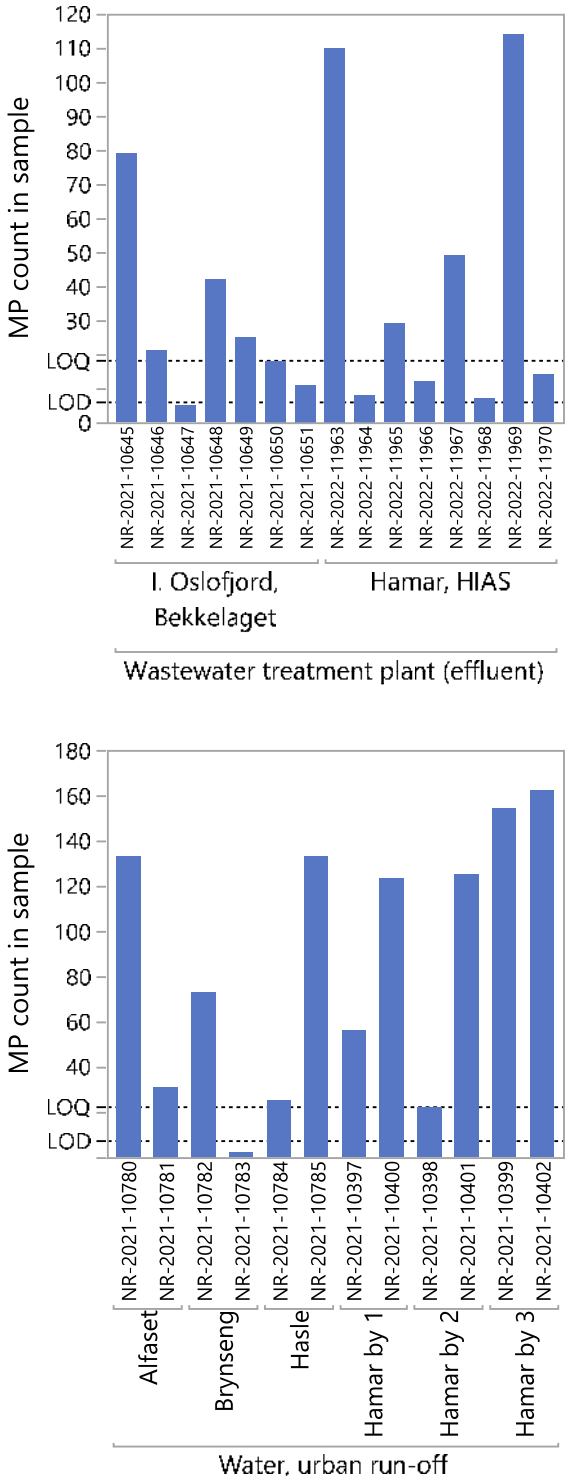


Figure 22. MP count in samples of water: Wastewater treatment plant (top) and urban run-off (bottom). LOD and LOQ indicated by dotted lines.

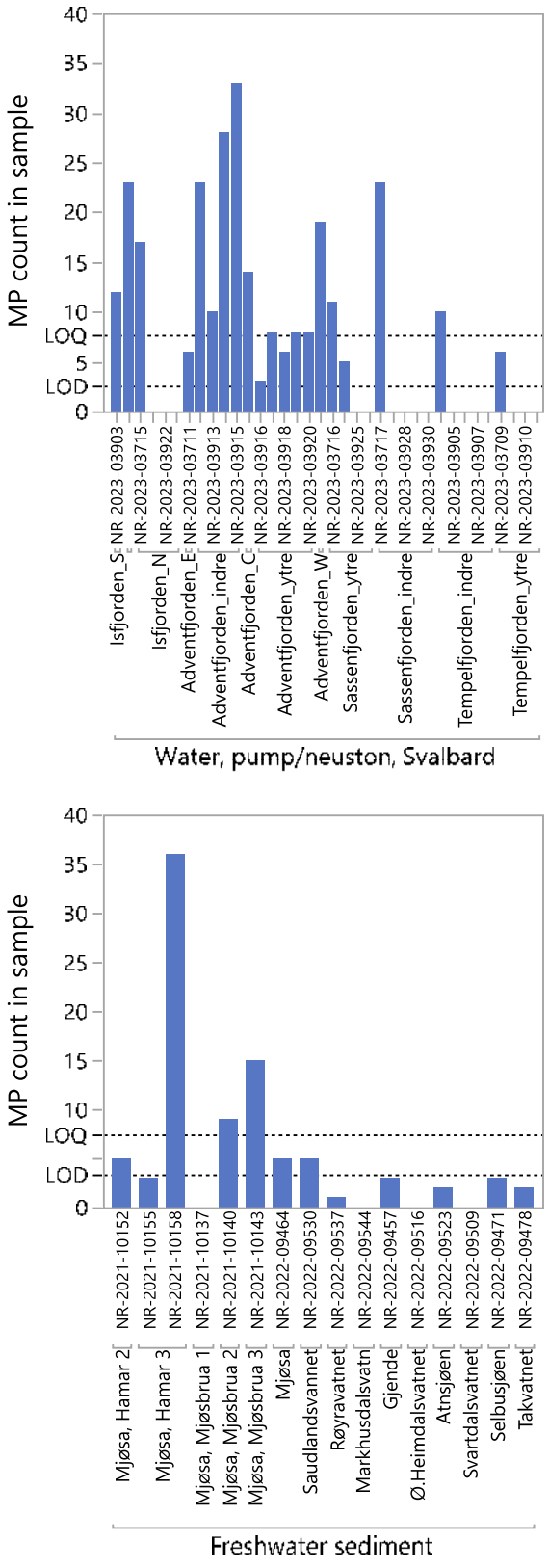


Figure 23. MP count in samples of water: pump/neuston, Svalbard (top) and freshwater sediments (bottom). LOD and LOQ indicated by dotted lines.

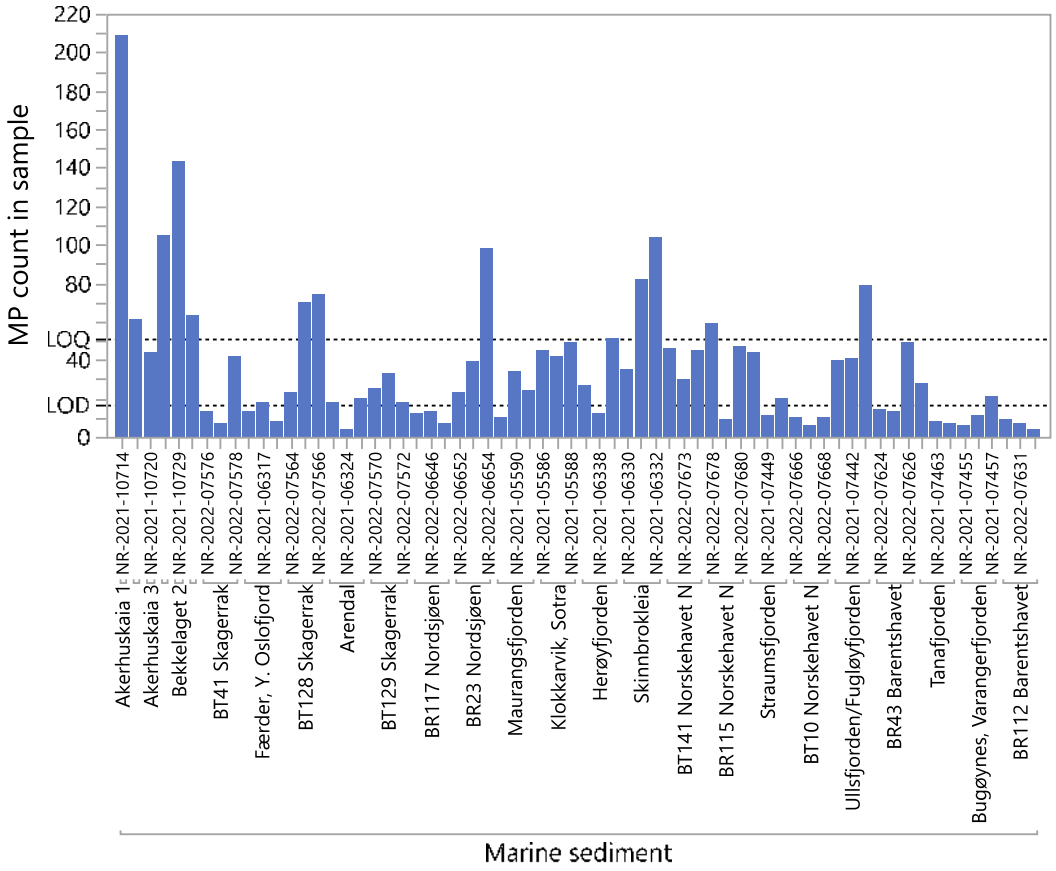


Figure 24. MP count in samples of marine sediments. LOD and LOQ indicated by dotted lines.

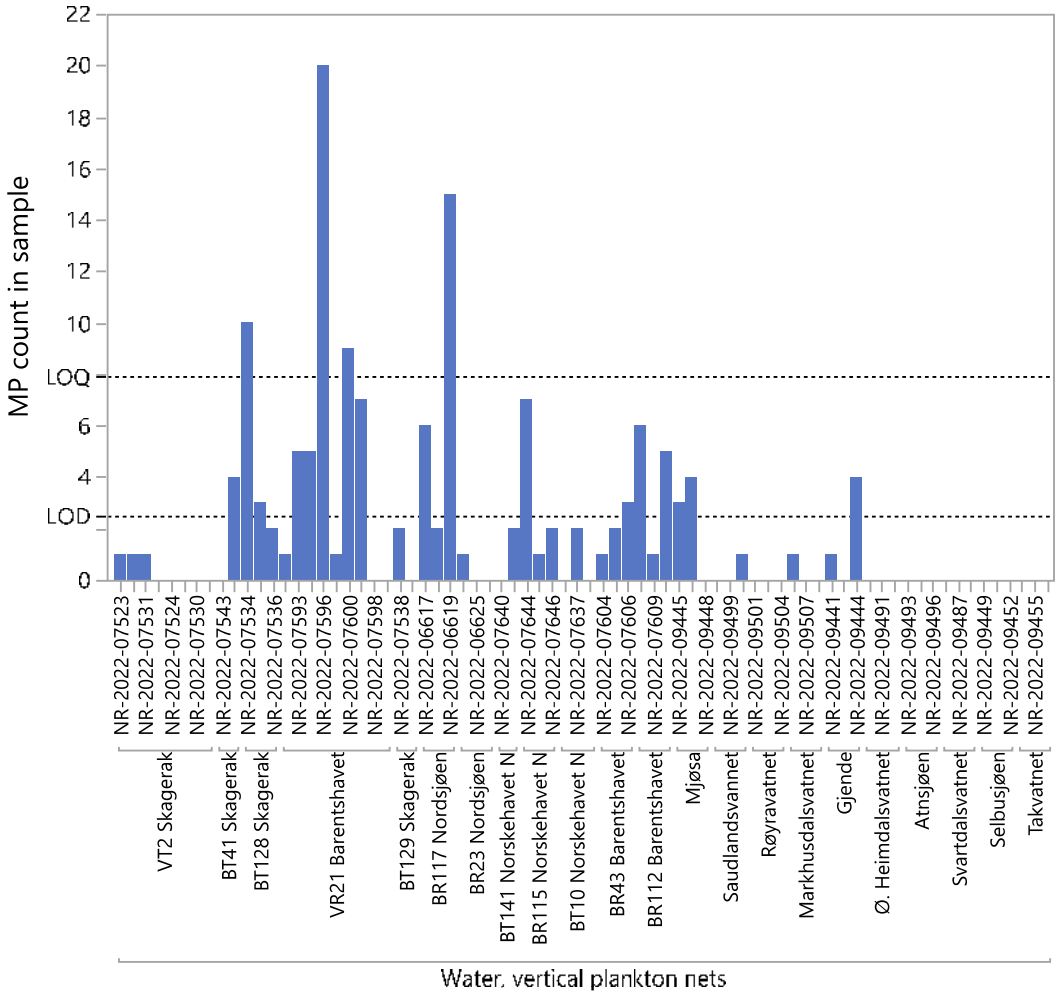


Figure 25. MP count in samples of water: vertical plankton nets. LOD and LOQ indicated by dotted lines.

Table 6. The mean MP per sample, LOD and LOQ for each sample type.

Matrix	Sample type	Mean MP/sample	LOD	LOQ
Biota	Blue mussel	4.7	9.2	19.9
	Duck mussel	7.9	25.2	63.7
	Invertebrates (polychaeta)	4.5	12.5	33.2
Water	Water, pump	31.4	0.0	0.0
	Water, ferrybox	9.9	4.5	11.9
	Water, manta trawl	12.2	2.5	7.9
	Water, pump/neuston, Svalbard	7.6	2.6	7.6
	Water, vertical plankton nets	1.9	2.5	7.9
Sediments	Marine sediments	36.2	16.9	50.7
	Freshwater sediments	5.6	3.4	7.4
Sources	Wastewater treatment plant (eff.)	36.3	6.2	18.3
	Water, urban runoff	86.6	7.9	22.5

The environmental samples that are over LOQ are marked with a green point and samples over LOD, but under LOQ are marked with an orange point, where samples are reported throughout the report.

Samples under LOD are marked with a red point. All particles in the environmental samples are included in the mean values for each sample matrices in Table 6.

About blank corrections

Within microplastics research, there has been an ongoing discussion whether to blank correct microplastic samples or not. As pointed out by Hermsen et al., (2018), who assessed potential airborne contamination in the laboratory; *blank correcting by particle count can often lead to an incorrect final sample number*. This was after determining that no particles were of a similar appearance to particles in environmental samples in that study.

Munno et al., (2023), however, recommended a complex correction by specific characteristics using combined methods. While this is more precise, and the total corrected value subtracts the lowest number of particles, it is time consuming and uncertain if it is applicable to a lab where lots of samples are handled in a large monitoring program.

Our approach has been to align microplastic monitoring with procedures that are common among other environmental monitoring analyses, to establish LOD and LOQ for methods and matrices, and to adjust number of replicates and amount of sample material to the established LOD/LOQ. This is also in line with the latest updates of international guidelines, e.g., EU Marine Strategy Framework Directive (MSFD)

In samples from areas without known, nearby sources, the analyses generally revealed low levels of microplastics, often below the detection limits. However, in samples close to high impacted areas the levels were higher and above detection limits (see figures above). For some sample types, it was challenging to obtain sample values which were over the LOD and LOQ. It was therefore necessary to modify our methodological approach. Firstly, we increased the number of individuals in the blue mussel samples to increase the number of microplastic particles in each processed sample. This year however, the blue mussels provided to MIKRONOR were much smaller than previous year. Also, several stations did not have enough blue mussels to provide 30 individuals to MIKRONOR, and so the samples did not necessary become much larger than previous year despite more individuals.

In two cases, we have been conducting subtractions of microplastic particles:

- If the contamination source was known, and particles with certainty came from that source, we have been subtracting those particles from the sample. However, this was not the case in this year's analyses.
- Fibres are a known problem in all net samples, and we have been excluding fibres from the plankton net samples and the Neuston net samples. Half of the net samples had fibre counts in the net blanks that were higher than in the samples. We are unsure whether the remaining samples have lower fibre content due to correct handling of the nets, or whether the net blanks were taken after the samples. Therefore, we have excluded fibres from all our net samples. This is also in line with international recommendations (Guidelines for MSFD, 2023, Michida et al., 2019).

Validation of the methods

Here we describe the validation of our methods for biota (performed on blue mussels) and sediments, as those were new (for sediments) or slightly changed (for biota) since the last report. Both methods include both fraction > 300 µm, where the analyses are performed on GFA filters, and small fraction 50-300 µm which are filtered onto silver filters.

Blue mussels

Recovery tests and method validation for blue mussel samples were carried out by using 10 blue mussels per spiked sample bought in the local grocery store (purchased from Coop Hasle). Recovery tests were handled separately for the two size fractions; >300 mm (Table 7) and (50-300 µm (Table 8). The samples were prepared and manually spiked with 30 transparent polyethylene (PE) beads (125-150 µm), spheric and 15 transparent polystyrene (PS) beads (500 µm). In addition to the spiked samples, three laboratory blanks without particles, and three spiked RO-water samples with added particles (spiked blanks) were included.

Table 7. Recovery tests: Blue mussel microscope analyses (>300 µm).

Sample type	Number of PS particles added (500 µm)	Average number of PS particles recovered (500 µm)	Recovery (%)
Blank (n=3)	0	0	n.a.
Spiked blank (n=3)	15	15	98 ± 4
Spiked sample (n=6)	15	14	90 ± 17

Table 8. Recovery tests: Blue mussel silver filter (50-300 µm).

Sample type	Number of PE particles added (125 µm)	Average number of PE particles recovered (125 µm)	Recovery (%)
Blank (n=3)	0	0	n.a.
Spiked blank (n=3)	30	19	63 ± 20
Spiked sample (n=6)	30	16	54 ± 11

Sediments

Recovery tests and method validation for sediment samples were carried out by using field collected sediments to have a representative material with the same organic and inorganic content. Six marine sediment samples were prepared and manually spiked with 30 transparent PE beads (125-150 µm) and 10 transparent PS beads (500 µm). In addition to the spiked samples, three laboratory blanks without particles, and three spiked RO-water samples with added particles (spiked blanks) were included (Table 9 and 10).

The spiked sediment samples followed the same procedure as the sediment samples. The spiked material had a characteristic colour, shape and size and they were therefore only analysed visually.

Table 9. Recovery tests: Sediments microscope analyses (big fraction).

Sample type	Number of PS particles added (500 µm)	Average number of PS particles recovered (500 µm)	Recovery (%)
Blank (n=3)	0	0	n.a.
Spiked blank (n=3)	10	10	97 ± 6
Spiked sample (n=6)	10	10	97 ± 6

Table 10. Recovery tests: Sediments silver filter (small fraction).

Sample type	Number of PE particles added (125 µm)	Average number of PE particles recovered (125 µm)	Recovery (%)
Blank (n=3)	0	0	n.a.
Spiked blank (n=3)	30	18	59 ± 5
Spiked sample (n=6)	30	12	49 ± 6

For both sample types, both the big and the small fraction was analysed visually under the microscope in agreement with the upcoming ISO/NP 16094-2 'MP in water -Vibrational spectroscopy. The same principle applies for other matrices where the final detection techniques might differ from the detection method used for the samples. We observed that there was an incompatibility between the scanning software used for the smaller fraction (Purency), the silver filters and the particles (which were reflective). The beads used as small reference materials contained a reflective additive which resulted in bad quality FTIR spectra which did not pass QA/QC criteria using current settings used in the scanning and analyse programs. Therefore, the validation was performed using the visual assessment only. The reference materials were chosen to allow a distinction between any background contamination present in the field sample since these types of beads are extremely rare in environmental samples. We are currently working on more relevant reference material for the smaller fraction.

The recovery range for smaller microplastic of the blue mussel samples is also in agreement with the minimum recovery rate proposed in the ISO standard for water samples. This recovery for the smaller fraction is in line with the current 'state of the art' as shown in several interlaboratory intercomparison studies (Van Mourik et al., 2021). The uncertainties in all smaller fractions must be regarded as high and highlight that studies of this fraction in environmental samples are still in the research development phase and will not easily be used in quantitative comparisons, neither national nor international. This is evidenced by the fact that the currently available international bodies only recommend reporting of microplastics > 300 µm (AMAP, 2021; Martin et al., 2022).

For validation of our water samples, we refer to MIKRONOR annual report 2022.

QA/QC for air samples

Procedural and field blanks were in most cases showing similar contributions by contamination, and LODs were calculated by using field blank data, since they incorporate both the contribution in the field and the procedure used in the laboratory (Table 11).

Table 11. LODs for the determination of various polymer types in the air samples. BD = Birkenes deposition, ZD = Zeppelin deposition, BA = Birkenes active air and ZA = Zeppelin active air.

	Unit	PMMA	PP	Nylon	PVC	PU	PC	PE	PS	PET
LOD BD	ng/ L	0.87	1.35	0.02	4.69	0.28	0.004	0.61	0.07	0.17
LOD ZD	ng/L	2.59	0.05	0.37	1.74	4.16	0.005	6.38	0.18	0.57
LOD BA	ng/m^3	0.55	1.46	0.05	2.51	0.04	0.008	2.01	0.07	5.51
LOD ZA	ng/m^3	0.001	0.01	0.04	0.27	0.01	0.004	0.11	0.001	0.001

5.2 Calculations of masses from FTIR data

To estimate the mass of each plastic polymer type per sample, the volume of each particle and data from FTIR (polymer type) were used. The estimation was carried out since the methods used for FTIR did not allow for mass quantification, whilst mass was only obtained for TWP, limiting the comparability between the two data sets.

Measuring TWP using pyrolysis-GCMS required a higher temperature compared to other plastic particles (for specific temperature details, see Appendix 5.3). To report both TWP and other plastics by mass concentrations, NIVA received a special assignment from NEA in 2023 to develop formulas for calculating plastic masses from the size of each particle and FTIR data. Here we briefly describe how this was conducted and the resulting formulas that have been used to calculate the mass concentrations of polymers in all the samples, including samples from last year's report.

NIVA has two datasets with environmentally sampled microplastics particles that we used for developing the most accurate formula, and to validate the calculated weights:

1. Dataset_1. An extended dataset containing about 3600 particles in the size range 50-5000 µm, which sizes were measured in three dimensions (L, w, *and* h).
2. Dataset_2. A dataset with 142 particles in the size range 1000-5000 µm, which were weighed individually using a micro-balance (mg, Sartorius, lim=0.001 mg) and the longest, the shortest dimensions of particles were measured together with particle squares from top view (using Infinity 1–3C/INFINITY 1 Lumenera camera and INFINITY ANALYZE and CAPTURE software).

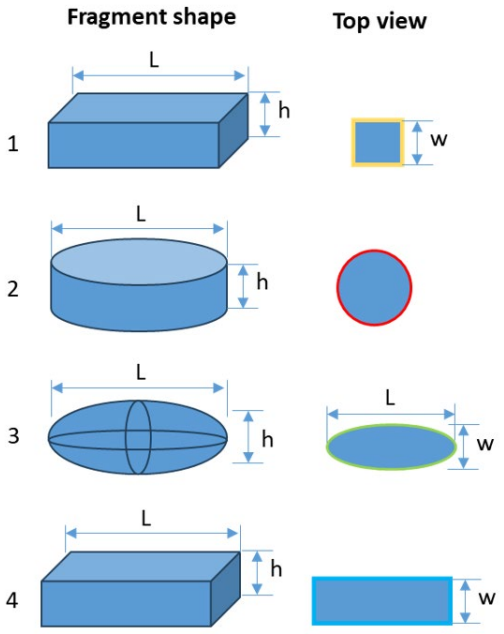
These two datasets were used to develop formulas for calculating the volume of each particle, and to validate the tested formulas for the volume/mass calculation.

We acknowledge that the most critical step in such calculation is the estimation of the volume of each particle. The calculation of the volume of a 3D particle is connected to a large uncertainty, since only two dimensions of the particles were measured in the MIKRONOR programme (the longest and the shortest dimension of the particle seen from a top view under the microscope).

Evaluation of existing literature on volume estimation per microplastic particle

We have examined four published formulas to calculate the volume of the microplastic particles using measured longest and shortest dimensions of the particles.

1. Cózar et al., (2014): assumes particles are rectangular prisms with a face that is a square of the length and a height of 0.1 times the length.
2. Isobe et al., (2021): assumes particles are cylindrical prisms with a face that has the length as the diameter and a height of 0.4 times the length.
3. Simon et al., (2018): assumes particles are ellipsoids using the measured length and width as the length and width of the ellipsoid and a height that is 0.67 times the width.
4. Kim et al., (2018): assumes particles are rectangular parallelepipeds with a common dimension of one third of the length.



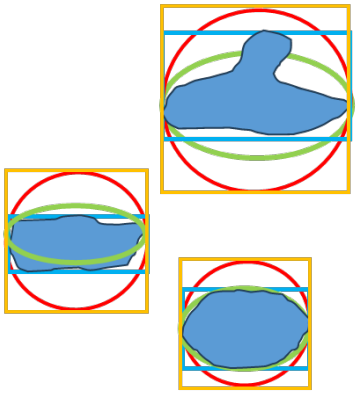
$h = 0.1 \times L$ $V = L^2 \times h$

$h = L \times 0.4$ $V = \pi r^2$

$h = w \times 0.67$ $V = \frac{4}{3}\pi\frac{Lwh}{8}$

$h = w = L \times 0.33$ $V = L \times w \times h$

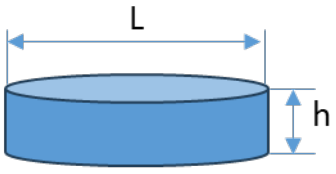
In most of cases all five formulas will overestimate the particle areas seen from top view:

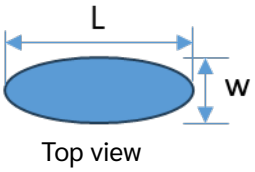


The overestimation of the areas could be compensated with an underestimation of the height estimation. However, without any way to validate this, the error will remain unknown.

In addition to formulas 1-4, we tested a model based on an elliptical cylinder using the measured longest and shortest dimensions of the particle as the length (L) and width (w) of the ellipsoid respectively.

5



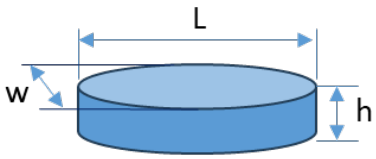


$$V = \pi \frac{Lwh}{4}$$

We employed five formulas with Dataset 1 to compare estimated and measured heights. Our findings revealed that the height relative to the width of particles depended on the size fraction. With the exception of Formula 3 (Simon et al., 2018) and our own Formula 5, all other formulas significantly overestimated the volume of particles in Dataset 1.

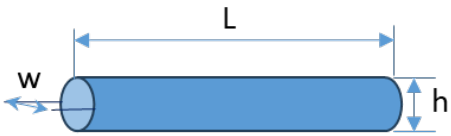
Moreover, our elliptical cylinder formula (Formula 5) yielded the same results for both fragments and fibres. This characteristic proved advantageous in Mikronor, especially for the scanning applied to size fraction 50-300 µm, where the particle shapes are not discerned.

Fragments



$$V = \pi \frac{Lwh}{4}$$

Fibres



$$V = \pi \frac{h}{2} \times \frac{w}{2} \times L = \pi \frac{Lwh}{4}$$

h = *k* w

Validation of formulas

For Formula 3 (Simon et al., 2018), we utilized the published coefficient k of 0.67. Concerning Formula 5, we employed Dataset_1 to derive the height as a function of the width of the particles in the following manner:

It was assumed that the coefficient k could vary for different size fractions. Consequently, Dataset_1 was divided into four subsets based on size fractions: 50-300 µm, 300-1000 µm, 1000-2000 µm, and 2000-5000 µm. Typically, films are very thin with a depth of less than approximately 25 µm, regardless of L and w. Hence, the ratio between depth and width for films may differ from that for fragments and fibres.

The median ratio of depth to width (k = h/w) was calculated for the entire dataset and separately after removing all film-like particles from the dataset (particles with a depth of 10-25 µm), see Table 12.

Table 12. Height as a function of width (h =*k* w) dependent on size fraction and whether films could be excluded.

	50-300 µm	300-1000 µm	1000-2000 µm	2000-5000 µm
k_1 = h/w, all particles	0.55	0.48	0.36	0.28
Std	0.29	0.28	0.29	0.39
k_2 = h/w, without films	0.58	0.50	0.49	0.41
Std	0.25	0.27	0.29	0.26

The results showed that the bigger particles, the smaller ratio between height and width of particles (*k*). The uncertainty in the volume calculations was reduced if the shape of the particle was known and the coefficient k_2 from the table was used (without film). In the MIKRONOR project, this could only be applied for particles that were analysed manually, mainly particles >300 µm. For data obtained with scanning analysis the shape was unknown. This was mainly particles <300 µm, and here the average coefficient k_1 for all particles (0.55) was used.

To calculate the volume of films, the formula for elliptical cylinders with a constant height of 15 µm fitted best with the heights for films measured in dataset 1.

$$V = \pi \left(\frac{L}{2}\right)\left(\frac{w}{2}\right) h \quad = \pi \left(\frac{Lw}{4}\right) 0.15$$

The mass of each particle was calculated by multiplying the volume of particle by its density.

$$m = V \times \rho$$

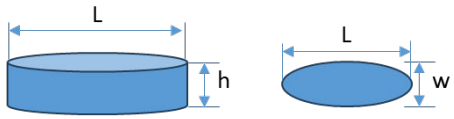
Densities for the identified polymer types were obtained from product datasheets which are widely available on the internet. The most common value found for each polymer was used for the calculations, and the highest and lowest densities found were used to estimate the uncertainties.

Table 13. Densities (g/cm^3) as used in the calculation of mass for each particle.

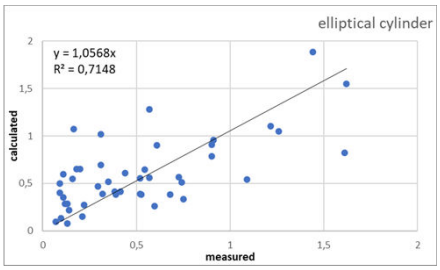
Mikronor polymer categories	Plastic	Density	Density, min	Density, max
mixed plastic (low confidence)	false	N/A	N/A	N/A
natural rubber derivatives	false	N/A	N/A	N/A
non-anthropogenic	false	N/A	N/A	N/A
other microlitter materials	false	N/A	N/A	N/A
unknown	false	N/A	N/A	N/A
paint/varnish particles	true	1.3	N/A	N/A
other plastics	true	1.317	N/A	N/A
abs	true	1.05	1	1.25
polyamide based	true	1.44	1.38	1.44
polycarbonate	true	1.2	1.2	1.22
black rubber-type/rubbery particles	true	1.7	1.2	2.1
other rubbers	true	1.7	1.2	2.1
rubbers, automotive	true	1.7	1.2	2.1
rubbers, sealing	true	1.7	1.2	2.1
ethylene-vinyl acetate	true	0.94	0.926	0.95
cellulose acetate and similar	true	1.28	1.28	1.3
polymeth (ester)acrylate based	true	1.22	1.22	1.22
silicone rubbers and coating	true	1.25	1.1	2.3
polychlorinated polymers	true	1.36	1.1	1.45
polystyrene based	true	1.05	1.04	1.06
polyurethane based	true	1.15	0.871	1.42
pan	true	1.18	1.17	1.2
nitrile rubbers	true	1	0.93	1.35
polyfluorinated polymers	true	2.2	2.1	2.2
polyethylene based	true	0.94	0.9	0.98
polypropylene based	true	0.91	0.9	0.92
polyester	true	1.39	1.3	1.4

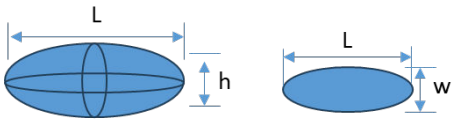
Results from the validation

We validated the calculations of the particle mass of different polymer types with the suggested formulas for the ellipsoid (Formula 3, (Simon et al., 2018), and the elliptical cylinder (Formula 5) using the obtained coefficients k_1 and k_2, by comparing calculated masses with the measured mass of particles from Dataset_2. The results showed that Formula 5, for elliptical cylinders coincided better with the measured weights (m_{calc}=1.06 x m_{meas}). Formula 3, for ellipsoid underestimates particles mass values with 30 % (m_{calc}=0.71 x m_{meas}).

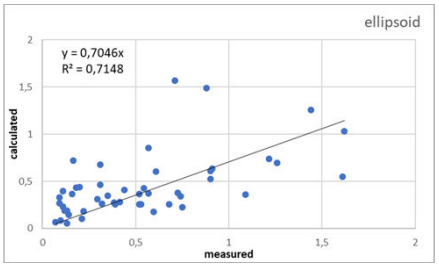


m_{calc}=1.06*m_{meas}
Std=0.097





m_{calc}=0.71*m_{meas}
Std=0.065



Conclusions

We have used formula 5, elliptical cylinders with a variable coefficient for the height as follows:

$$m = V \times \rho \qquad (1)$$

where
ρ = specific density for each polymer type

and

$$V = \pi \frac{Lwh}{4} \qquad (2)$$

where
$h = kw$
where k is dependent on whether the shape was known, and what size fraction the particle sorted under

Scanning analysis:
Shape was unknown, mainly for L <300 µm:
h = 0.55 w *(4)*

Manual Analysis:
Shape was known, mainly for L >300 µm
Fragments and fibres:

for L <300 µm:
$h = 0.58\ w$ (5)

for L = 300-1000 µm:
$h = 0.50\ w$ (6)

for L = 1000-2000 µm:
$h = 0.49\ w$ (7)

for L>2000 µm:
$h = 0.41\ w$ (8)

All films in the dataset:
$h = 15\ \mu m$

5.3 Analysis of tyre wear particles (TWP) with pyrolysis gas chromatography mass spectrometry

Method description

Pyrolysis gas chromatography mass spectrometry

Quantification of tyre wear particles with pyrolysis gas chromatography mass spectrometry (pyr-GC/MS) is performed by heating the sample rapidly (12 sec) at high temperature (700C). This enables organic components in the sample to be vaporised to gas phase and can then be separated on a GC-column. After being separated, each compound elutes from the column at different times based on their boiling point and polarity. After leaving the GC-column, the components are ionised by the mass spectrometer (electron ionization) and the ionised molecules are then accelerated through a mass analyser and separated based on their mass-to-charge ratio (m/z). Description of pyr-GC/MS settings are provided in Tables 14 and 15. For biota samples, the TWP levels were expected to be low and to improve quantification, a lower split ratio (25:1) was used for these samples compared to sediment and water samples (50:1 split). This refers to the amount of material from the pyrolysis that is injected to the GC. Lower split ratio, allowing more of the sample to the GC/MS, can help detection of the analytes, however, if the sample has a lot of other organic compounds, all signals potentially be equally amplified, and the outcome would not be improved. It is therefore only used in the biota samples in this study, for which several steps of treatments to remove organic material have been performed to dissolve the biota samples.

Table 14. PYR-GC/MS settings for all sediment samples and water samples. 50:1 split

Apparatus	Parameters	Settings
Micro-furnace Pyrolyzer Frontier EGA/PY-3030D (Single-Shot analysis)	Pyrolyzer furnace/oven temperature	700 °C
	Pyrolyzer interface temperature	300 °C
	Pyrolysis time	0.20 min (12 seconds)
Gas chromatogram (GC)	Column	Ultra-Alloy® 5 capillary column (30 m, 0.25 mm I.D., 0.25 µm film thickness) (Frontier Lab)
	Injector port temperature	300 °C
	Column oven temperature program	50 °C (2 min) → (5 °C /min) → 190 °C (30 min) → (20 °C /min) → 300 °C (37.5 min)
	Injector mode	Split (split 50:1)
	Carrier gas	Helium, 1.0 mL/min, constant linear velocity
Mass spectrometer (MS)	Ion source temperature	230 °C
	Ionization energy	Electron ionization (EI); 70 eV
	Scan mode/range	Total ion chromatogram (TIC) mode, 50 to 350 m/z

Table 15. PYR-GC/MS settings for all biota samples, 25:1 split

Apparatus	Parameters	Settings
Micro-furnace Pyrolyzer Frontier EGA/PY-3030D (Single-Shot analysis)	Pyrolyzer furnace/oven temperature	700 °C
	Pyrolyzer interface temperature	300 °C
	Pyrolysis time	0.20 min (12 seconds)
Gas chromatogram (GC)	Column	Ultra-Alloy® 5 capillary column (30 m, 0.25 mm I.D., 0.25 µm film thickness) (Frontier Lab)
	Injector port temperature	300 °C
	Column oven temperature program	50 °C (2 min) → (5 °C /min) → 195 °C (31 min) → (20 °C /min) → 300 °C (38.25 min)
	Injector mode	Split (split 25:1)
	Carrier gas	Helium, 1.0 mL/min, constant linear velocity
Mass spectrometer (MS)	Ion source temperature	230 °C
	Ionization energy	Electron ionization (EI); 70 eV
	Scan mode/range	Total ion chromatogram (TIC) mode, 50 to 350 m/z

Markers for quantification

For the identification and quantification of tyre wear particles, 5 specific marker compounds (Table 16) for synthetic rubber (styrene butadiene rubber and butadiene rubber, SBR+BR) are monitored. In (Rødland et al., 2022) the variation of these markers in tyre material was evaluated across 31 new tyres (including both personal vehicles and truck tyres, summer, winter and winter with studs) representing the Norwegian car park. In this study it was found that the markers that gave the lowest standard deviation (std) in the tyres included in the database, e.g., presents the most stable marker for tyre wear across the variation of tyres, were the four-marker combination M4 (Table 16), with an average mass concentration of 319 µg/mg SBR+BR and 40% std. The marker 4-VCH, which is described in the current ISO Technical specification (ISO, 2017) gave a lower mass concentration of rubbers in the tyres, 116 µg/mg SBR+BR and higher standard deviation, up to 62% std. A few studies have also used the SB and SBB markers (Eisentraut et al., 2018; Goßmann et al., 2021) for quantification, however, in the study of Rødland et al., 2022 they were both found to give very high standard deviations in the tyre material tested. SB was reported to give an average mass concentrations of 352 µg/mg SBR+BR with 77% std., whereas SBB gave a concentration of 226 µg/mg SBR+BR with 85% std.

For these reasons, the marker combination of M4 was chosen for further use and spike-recovery tests validated that the method was suitable for road-related samples (83-92% recovery for tyre tread; 88-104% recovery for road dust) (Rødland et al., 2022). Method M4 has been used in several recent publications of tyre wear particles in road tunnels (Rødland et al., 2022), road-side snow (Rødland et al., 2022) and road-side soils (Rødland et al., 2023).

However, the variation of marker compounds in commercial tyres is not the only challenge to quantification of tyre wear particles in environmental samples. Although the M4 marker combination has been found to be the most reliable marker in the Norwegian tyres tested and validated with good recoveries in environmental samples close to the road, the inclusion of *benzene* as a marker has proven to be challenging when working with more complex matrices, such as terrestrial soil (Rødland et al., 2023). Because of this, the signal ratio of the four markers is monitored and remove *benzene* from the combination if it is higher than what is expected from the average tyre material. This approach has been used in this project and we found that the ratios of markers M4 was impacted by an additional *benzene* source in some of the matrices (marine sediments, freshwater sediments, duck mussels) (Table 17), and the TWP concentrations in these samples have therefore been quantified using M3. In addition to the SBR+BR values and TWP values using M4/M3, the SBR+BR values using the 4-VCH marker are included in the results (Table 18) for comparison with other studies published.

Currently, many studies use an average SBR+BR level of 50% synthetic rubber in tyre tread when calculating TWP from the measured SBR+BR concentrations, as is described in the ISO Technical specification (ISO, 2017). However, the use of these fixed rubber values may lead to over- or underestimation of TWP present in environmental samples due to the highly variable SBR+BR concentration found in different types of tyres (Rødland et al., 2022). To reduce this uncertainty, Rødland et al., (2022) developed a method using Monte Carlo prediction modelling to predict the TWP concentration in a sample based on the measured SBR+BR concentration in the sample and the measured SBR+BR concentrations in relevant reference tyres. The model is adapted to include tyres for the season that is relevant for the sample. For example, a sample of surface water runoff collected during winter is likely to have mainly TWPs from winter tyres present in the sample. Thus, the SBR+BR levels from winter tyres is used for the prediction model. Likewise, sediment samples are likely to contain TWPs from several years and seasons, thus the prediction model is performed with SBR+BR levels from all tyres present in the database. The presented result for the project contains the average, median and standard deviation of the predicted TWP concentrations (Table 18).

Calibration curve, limit of detection and limit of quantification

Two calibration curves were made, one for water and sediment samples using 50:1 split (Figure 26) and one for the biota samples using 25:1 split (Figure 27). The 50:1 curve was established by adding 0.1, 1, 3, 5, 10, 20, 40, 60 µg SBR/cup of SBR (SBR1500 standard, Polymer Source) into separate pyrolysis cups and spiked with 10µg/cup d6-PB as internal standard. The 25:1 curve was established by adding 0.1, 0.5, 1, 3, 5, 10 SBR/cup and 10µg d6-PB/cup for all levels. The normalised sum peak of all marker compounds is plotted against the mass of SBR at each calibration level to form the calibration curve (R = 0.995) (Figure 26 and 27). The signal to noise ratio (S/N) is determined by the Agilent Masshunter software for each of the selected markers and then summarised to represent the sum of markers. The S/N level was plotted against the concentration level of SBR to determine the S/N vs concentration relationship following the method by Donovan (2016), using power of regression. The calculated LOD (3 x S/N) using the sum of markers were 0.043µg SBR and LOQ (10 x S/N) were 0.188 µg SBR for calibration curve 50:1. For the 25:1 curve, the LOD was 0.015µg SBR and LOQ was 0.083.

Table 16. Marker compounds used to quantify styrene butadiene rubber (SBR+BR). Italics and bold values used for calibration and quantification. Internal standard Poly(1,4-butadiene-$_d$6) is used for normalization of all samples.

Marker combination	Pyrolysis product	Indicator ions (m/z)
M4/M3	Benzene	51, 67, **78**
M4/M3	α-methylstyrene	78, 91, **118**
M4/M3	Ethylstyrene	77, 91, **117**
M4/M3	Butadiene trimer A	65, **91**, 146
4-VCH	4-Vinylcyclohexane	**54**, 79, 108
Internal standard	Poly(1,4-butadiene-$_d$6)	***60,*** 120, 42, 86

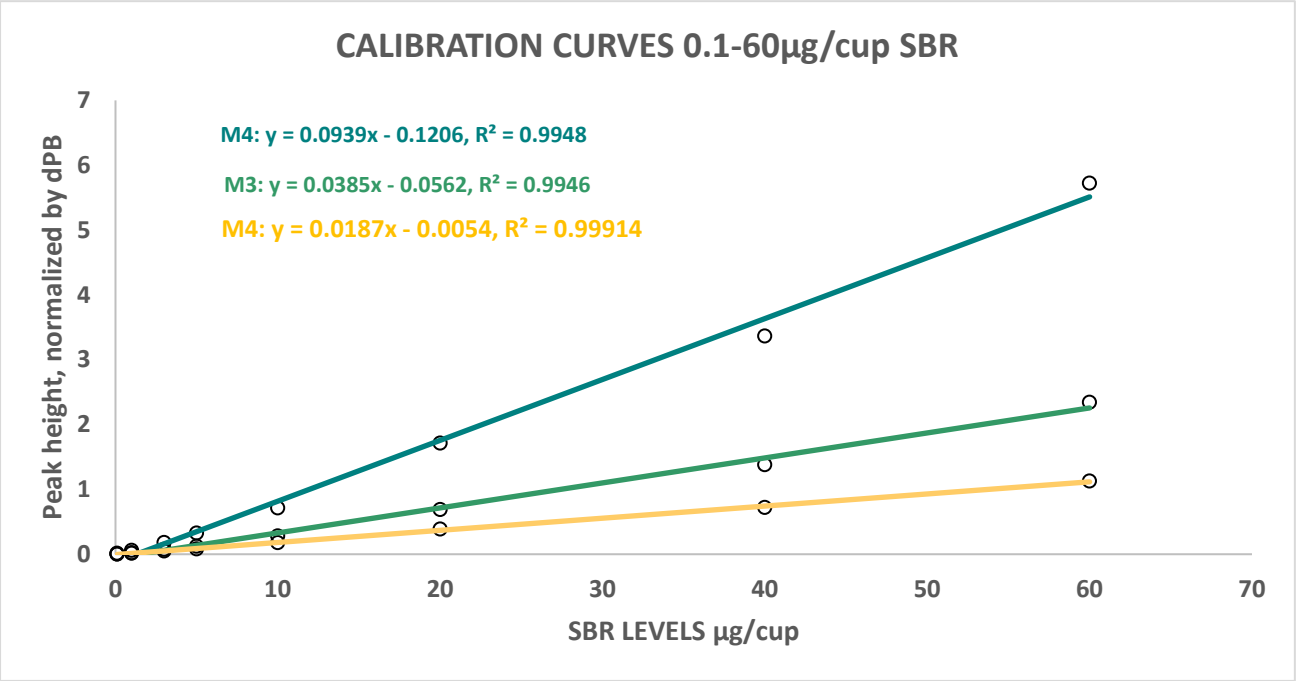


Figure 26. Calibration curves for all biota samples in 50:1 split. Curves made using 4 levels of SBR1500 (0.1, 1, 3, 5, 10, 20, 40, 60 µg SBR/cup). Calibration levels shown are for methods M4, M3 and 4-VCH.

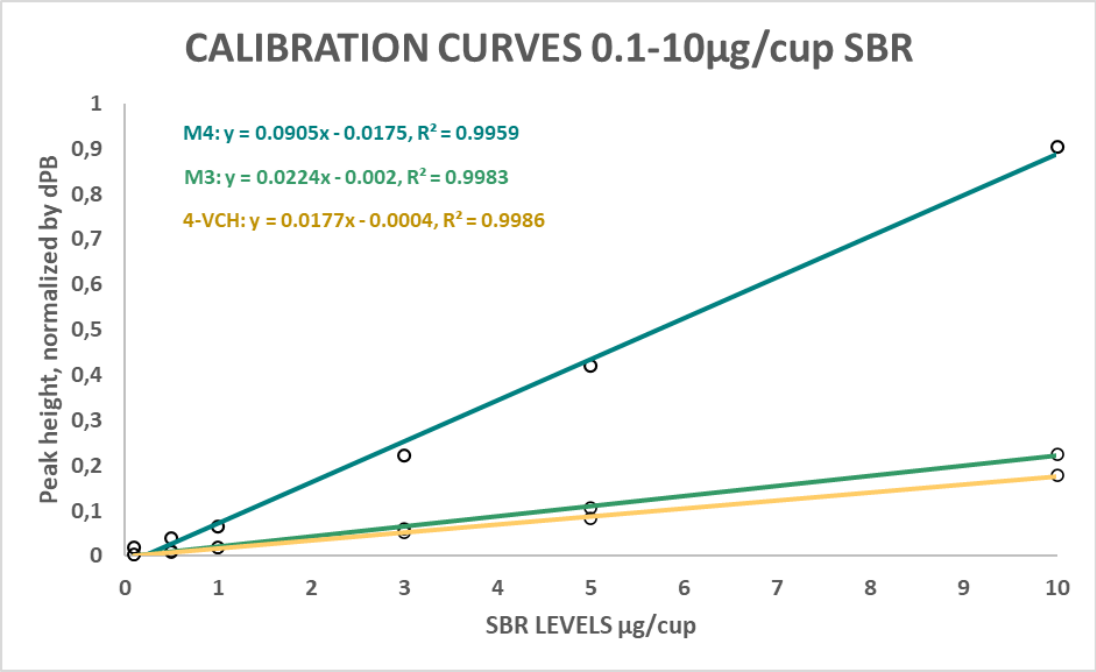


Figure 27. Calibration curves for all biota samples in 25:1 split. Curves made using 4 levels of SBR1500 (0.1, 0.5, 1, 3, 5, 10 µg SBR/cup). Calibration levels shown are for methods M4, M3 and 4-VCH.

Testing pretreatment for freshwater sediments

In this project, extensive pretreatment before analysis with PYR-GC/MS was not possible due to time and cost limitations. All solid samples (freshwater and marine sediments) were analysed directly, with no other preparations than freeze-drying and sieving (<500µm, metal mesh sieves). As described in section A1, in samples with high levels of organic matter and potentially also low concentrations of tyre wear particles, the marker ratios of method M4 is influenced, especially by *benzene*. To investigate which methods could have been most useful to perform to improve the TWP analysis in these complex matrices, a spike and recovery test using freshwater sediment was performed. The chosen sediment sample were from Heimdalsvatn (freshwater lake) and the total organic content (TOC) in this sample prior to treatment was 69mg/g. The sample was freeze dried, sieved at 500µm and homogenised prior to treatment.

Four different treatments were tested and compared to untreated samples, all from one site (Heimdalsvatnet). The treatments tested were 1) Sodium dodecyl sulphate (SDS)+Fentons reagent (30% H_2O_2 + $FeSO_4$*$7H_2O$), 2) SDS+Fentons reagent +enzymes (Pectinase, Cellulase, Viscozyme), 3) SDS+Fentons reagent+KOH (potassium hydroxide), 4) SDS+Fentons reagent+KOH+enzymes (Table 17).

In addition, both single shot pyrolysis and double shot pyrolysis were tested. In this project, all samples have been analysed in single shot flash pyrolysis, as described in section A1-1. However, the microfurnace PYR-GC/MS set-up also has the ability to perform a thermal desorption step before the flash pyrolysis, enabling a double shot feature. This first shot (thermal desorption step), slowly heats the sample up from 100 to 300C with a 50C/min increase and then holds 300C for 1min. In this step, the volatile fraction of the sample is thermally desorbed and can be chromatographically separated, allowing for this fraction to be analysed as well as removing interference from the sample before pyrolysis. For this test, only the second shot was utilised for comparisons.

All samples were analysed in triplicates, in which one of the triplicates contained a 20µg SBR (SBR1500) spike. This lets us first establish the background level of SBR+BR in the sample based on the first two replicates and then calculate the recovery level of SBR with the third replicate. All the results and the recovery percentages for all treatments are listed in Table 19 and 20.

The results from this test demonstrated method M4 did not give good recoveries for any treatment when using single shot flash pyrolysis, however, when using double shot and SDS+Fentons+enzymes, recovery was within acceptable range (108%). The same treatment was found to give the best recovery also for M3 using double shot. As all samples in the project were analysed untreated in single shot, it should be noticed that for the Heimdalsvatnet samples, the recovery using M3 was within an acceptable range (80%), although better recoveries were found with treatments. The treatment with SDS+Fentons+enzymes also gave good recoveries using the 4-VCH method for both single shot (111.3%) and double shot (107.8%), as well as SDS+Fentons+KOH+enzymes for double shot (107.7%). For the 4-VCH method, also analysis of the untreated samples had acceptable recoveries for both single shot (92.8%) and double shot analysis (92.0). In conclusion, using double shot pyrolysis and pretreatment with SDS+Fentons+enzymes give the best results for M4, M3 and 4-VCH markers combined.

Table 17. Treatments tested for freshwater sediments. 1) Sodium dodecyl sulphate (SDS)+Fentons reagent, 2) SDS+Fentons reagent+enzymes, 3) SDS+Fentons reagent+KOH, 4) SDS+Fentons reagent+KOH+enzymes

Sample nr	Sample mass g	Treatment
1	8.04	20mL of 10% SDS and 40mL prefiltered RO-water added to a flask with the sample. Incubated at 50C, 100rpm for 48hours. Then the sample was retained in a 50µm metal sieve and rinsed with RO water to remove SFS. Sample were transferred back to flask to start Fentons reagent; 10mL 30% H_2O_2 and 10mL $FeSO_4*7H_2O$. The procedure was repeated 5-7 times. Then the sample was again transferred to 50µm sieve and rinsed with RO water and then transferred to a glass jar, frozen, freeze dried and analysed with PYR-GC/MS.
2	8.07	20mL of 10% SDS and 40mL prefiltered RO-water added to a flask with the sample. Incubated at 50C, 100rpm for 48hours. Then the sample was retained in a 50µm metal sieve and rinsed with RO water to remove SFS. Sample were transferred back to flask to start Fentons reagent; 10mL 30% H_2O_2 and 10mL $FeSO_4*7H_2O$. The procedure was repeated 5-7 times. Then the sample was again transferred to 50µm sieve and rinsed with RO water. The sample was then transferred to a flask using a sodium acetate buffer solution (0.1 M NaAc, pH=5, 75mL). Then 5mL Pectinase, 5 mL Cellulase and 1 mL Viscozyme was added to the sample and put in the incubator at 50C, 100 rpm for 48 hours. Then the sample rinsed through a 50µm sieve with RO water and transferred to a glass jar, frozen, freeze dried and analysed with PYR-GC/MS.
3	8.01	20mL of 10% SDS and 40mL prefiltered RO-water added to a flask with the sample. Incubated at 50C, 100rpm for 48hours. Then the sample was retained in a 50µm metal sieve and rinsed with RO water to remove SFS. Sample were transferred back to flask to start Fentons reagent; 10mL 30% H_2O_2 and 10mL $FeSO_4*7H_2O$. The procedure was repeated 5-7 times. Then the sample was again transferred to 50µm sieve and rinsed with RO water and transferred back to the flask with a metal spoon and the rest using a10% KOH to rinse off the sieve and into the flask. After this, the flask was filled up to 100mL with 10% KOH and put in incubator at 50C, 100 rpm for 24 hours. Then the sample was rinsed with RO water through the 50µm sieve and transferred to a glass jar, frozen, freeze dried and analysed with PYR-GC/MS.
4	8.04	20mL of 10% SDS and 40mL prefiltered RO-water added to a flask with the sample. Incubated at 50C, 100rpm for 48hours. Then the sample was retained in a 50µm metal sieve and rinsed with RO water to remove SFS. Sample were transferred back to flask to start Fentons reagent; 10mL 30% H_2O_2 and 10mL $FeSO_4*7H_2O$. The procedure was repeated 5-7 times. Then the sample was again transferred to 50µm sieve and rinsed with RO water and transferred back to the flask with a metal spoon and the rest using a10% KOH to rinse off the sieve and into the flask. After this, the flask was filled up to 100mL with 10% KOH and put in incubator at 50C, 100 rpm for 24 hours. Then the sample was again transferred to 50µm sieve and rinsed with RO water. The sample was then transferred to a flask using a sodium acetate buffer solution (0.1 M NaAc, pH=5, 75mL). Then 5mL Pectinase, 5 mL Cellulase and 1 mL Viscozyme was added to the sample and put in the incubator at 50C, 100 rpm for 48 hours. Then the sample was rinsed with RO water through the 50µm sieve and transferred to a glass jar, frozen, freeze dried and analysed with PYR-GC/MS.

Raw data tables

Table 18. Concentration of all samples (presented by LIMS nr) SBR+BR with marker method M4 (*), M3(**) and 4-VCH (***), and the predicted TWP concentration based on the measured SBR+BR concentration with the chosen method. Predicted TWP concentrations are presented as the average (mean), median and standard deviation (St.d.) value across 100 000 predictions (Monte Carlo simulation, Crystal ball), following the methods of (Rødland et al., 2022).

LIMS nr	SBR µg/unit M4/M3	SBR µg/unit vch	UNIT	TWP Mean	TWP Median	TWP St.d.	UNIT	Method
BM_ProcedureBl	<1	<1	µg/sampl	<1				*
BM_ProcedureBl	<1	<1		<1				*
BM_ProcedureBl	<1	<1		<1				*
Proc.blank 1	<1	<1		<1				*
Proc.blank 2	<1	<1		<1				*
Proc.blank 3	<1	<1		<1				*
Proc.blank 1	<1	<1		<1				*
Proc.blank 2	<1	<1		<1				*
Proc.blank 1	<1	<1		<1				*
Proc.blank 2	<1	<1		<1				*
Proc.blank 3	<1	<1		<1				*
Procedure blank	<1	<1		<1				*
Procedure blank	<1	<1		<1				*
Procedure blank	<1	<1		<1				*
Procedure blank	<1	<1		<1				*
Procedure blank	<1	<1		<1				*
NR-2023-01528	8.37	<1	µg/g	25.36	25.26	2.28	mg/g	*
NR-2023-01529	<1	<1		<1				*
NR-2023-01530	19.07	<1		57.79	57.58	5.20		*
NR-2023-01534	2.34	2.49		7.08	7.05	0.64		*
NR-2023-01535	2.99	2.74		9.06	9.02	0.81		*
NR-2023-01536	1.87	2.08		5.67	5.65	0.51		*
NR-2023-01537	0.38	0.19		1.15	1.15	0.10		*
NR-2023-01538	0.25	<1		0.74	0.74	0.07		*
NR-2023-01539	0.32	0.29		0.98	0.97	0.09		*
NR-2023-01543	6.27	<1		18.99	18.92	1.71		*
NR-2023-01544	3.85	<1		11.67	11.63	1.05		*
NR-2023-01545	17.34	<1		52.54	52.35	4.73		*
NR-2023-01540	7.76	<1		23.52	23.43	2.12		*
NR-2023-01541	7.23	0.52		21.90	21.82	1.97		*
NR-2023-01542	3.67	0.34		11.12	11.08	1.00		*
NR-2023-01532	0.40	<1		1.20	1.20	0.11		*
NR-2023-01533	0.51	<1		1.53	1.53	0.14		*
NR-2023-01531	0.31	<1		0.93	0.92	0.08		*
NR-2022-07518	4.30	<1	µg/L	1.30	1.30	0.12	mg/L	*
NR-2022-08994	3.23	<1		0.98	0.98	0.09		*
NR-2022-08990	1.48	<1		0.45	0.45	0.04		*
NR-2022-07517	<1	<1		<1				*
NR-2022-07519	0.81	0.08		2.29	2.16	0.62		*
NR-2022-07520	0.28	0.16		0.79	0.74	0.21		*
NR-2022-08989	0.59	<1		1.68	1.59	0.46		*
NR-2022-08991	0.76	<1		2.16	2.05	0.59		*
NR-2022-08992	0.22	<1		0.64	0.60	0.17		*
NR-2022-08993	2.00	0.10		5.68	5.37	1.55		*
NR-2022-08995	1.07	0.11		3.03	2.87	0.83		*
NR-2022-08996	0.96	0.15		2.73	2.59	0.75		*
NR-2022-11963	0.31	<1		0.87	0.82	0.24		*
NR-2022-11964	0.26	0.03		0.74	0.70	0.20		*
NR-2022-11965	0.25	0.14		0.72	0.68	0.20		*

NR-2022-11966	0.17	<1		0.49	0.46	0.13		*
NR-2022-11967	<1	<1		<1				*
NR-2022-11968	0.30	<1		0.85	0.81	0.23		*
NR-2022-11969	0.62	0.11		1.75	1.66	0.48		*
NR-2022-11970	0.76	0.06		2.15	2.04	0.59		*
NR-2021-10645	0.05	0.02		0.15	0.15	0.04		*
NR-2021-10646	0.06	<1		0.16	0.15	0.04		*
NR-2021-10647	<1	<1		<1				*
NR-2021-10648	0.04	0.02		0.12	0.11	0.03		*
NR-2021-10649	0.20	<1		0.57	0.54	0.16		*
NR-2021-10650	0.07	<1		0.21	0.20	0.06		*
NR-2021-10651	<1	<1		<1				*
NR-2021-10397	<1	<1		<1				*
NR-2021-10398	0.82	<1		2.75	2.72	0.31		*
NR-2021-10399	22.95	17.00		77.27	76.36	8.59		*
NR-2021-10402	35.54	16.62		119.64	118.23	13.31		*
NR-2021-10400	0.68	0.25		2.28	2.25	0.25		*
NR-2021-10401	6.22	2.38		20.93	20.68	2.33		*
NR-2021-10780	12.05	5.08		40.58	40.10	4.51		*
NR-2021-10781	0.87	<1		2.92	2.88	0.32		*
NR-2021-10782	22.63	14.93		76.20	75.30	8.48		*
NR-2021-10783	0.59	0.09		2.00	1.97	0.22		*
NR-2021-10784	12.93	<1		43.54	43.03	4.84		*
NR-2021-10785	12.90	4.18		43.44	42.93	4.83		*
NR-2021-07455	0.25	<1	µg/mg	0.84	0.83	0.09	mg/g	*
NR-2021-07455	0.26	<1		0.89	0.88	0.10		*
NR-2021-07455	0.35	<1		1.17	1.15	0.13		*
NR-2021-06316	<1	<1		<1				*
NR-2021-06316	<1	<1		<1				*
NR-2021-06316	<1	<1		<1				*
NR-2022-07672	0.21	<1		0.71	0.70	0.08		*
NR-2022-07672	0.24	<1		0.82	0.81	0.09		*
NR-2022-07672	0.22	<1		0.75	0.74	0.08		*
NR-2022-07678	0.13	<1		0.43	0.43	0.05		*
NR-2022-07678	0.20	<1		0.67	0.67	0.07		*
NR-2022-07678	0.16	<1		0.55	0.54	0.06		*
NR-2022-06645	0.20	<1		0.69	0.68	0.08		*
NR-2022-06645	0.16	<1		0.54	0.54	0.06		*
NR-2022-06645	0.24	<1		0.80	0.79	0.09		*
NR-2022-07666	<1	<1		<1				*
NR-2022-07666	<1	<1		<1				*
NR-2022-07666	<1	<1		<1				*
NR-2021-05586	0.16	<1		0.53	0.52	0.06		*
NR-2021-05586	0.17	<1		0.59	0.58	0.07		*
NR-2021-05586	0.24	<1		0.81	0.80	0.09		*
NR-2021-06337	0.18	<1		0.60	0.59	0.07		*
NR-2021-06337	0.15	<1		0.50	0.49	0.06		*
NR-2021-06337	0.15	<1		0.51	0.50	0.06		*
NR-2022-07624	0.14	<1		0.48	0.47	0.05		*
NR-2022-07624	0.16	<1		0.52	0.52	0.06		*
NR-2022-07624	0.18	<1		0.60	0.59	0.07		*
NR-2021-05589	0.35	<1		1.17	1.15	0.13		*
NR-2021-05589	0.35	<1		1.19	1.18	0.13		*
NR-2021-05589	0.33	<1		1.11	1.10	0.12		*
NR-2021-07455	0.25	<1		0.84	0.83	0.09		*
NR-2021-07455	0.26	<1		0.89	0.88	0.10		*
NR-2021-07455	0.35	<1		1.17	1.15	0.13		*
NR-2021-06316	<1	<1		<1				*
NR-2021-06316	<1	<1		<1				*
NR-2021-06316	<1	<1		<1				*
NR-2022-07672	0.21	<1		0.71	0.70	0.08		*

NR-2022-07672	0.24	<1		0.82	0.81	0.09		*
NR-2022-07672	0.22	<1		0.75	0.74	0.08		*
NR-2022-07678	<1	<1		<1				*
NR-2022-07678	0.20	<1		0.67	0.67	0.07		*
NR-2022-07678	0.16	<1		0.55	0.54	0.06		*
NR-2022-06645	0.20	<1		0.69	0.68	0.08		*
NR-2022-06645	0.16	<1		0.54	0.54	0.06		*
NR-2022-06645	0.24	<1		0.80	0.79	0.09		*
NR-2022-07666	<1	<1		<1				*
NR-2022-07666	<1	<1		<1				*
NR-2022-07666	<1	<1		<1				*
NR-2021-05586	0.16	<1		0.53	0.52	0.06		*
NR-2021-05586	0.17	<1		0.59	0.58	0.07		*
NR-2021-05586	0.24	<1		0.81	0.80	0.09		*
NR-2021-06337	0.18	<1		0.60	0.59	0.07		*
NR-2021-06337	<1	<1		<1				*
NR-2021-06337	0.15	<1		0.51	0.50	0.06		*
NR-2022-07624	0.14	<1		0.48	0.47	0.05		*
NR-2022-07624	0.16	<1		0.52	0.52	0.06		*
NR-2022-07624	0.18	<1		0.60	0.59	0.07		*
NR-2021-05589	0.35	<1		1.17	1.15	0.13		*
NR-2021-05589	0.35	<1		1.19	1.18	0.13		*
NR-2021-05589	0.33	<1		1.11	1.10	0.12		*
NR-2021-07462	0.17	<1		0.57	0.56	0.06		*
NR-2021-07462	0.17	<1		0.56	0.55	0.06		*
NR-2021-07462	0.24	<1		0.82	0.81	0.09		*
NR-2021-06330	0.44	<1		1.48	1.46	0.16		*
NR-2021-06330	0.37	<1		1.25	1.23	0.14		*
NR-2021-06330	0.36	<1		1.22	1.20	0.14		*
NR-2021-06323	0.30	<1		1.03	1.01	0.11		*
NR-2021-06323	0.29	<1		0.96	0.95	0.11		*
NR-2021-06323	0.21	<1		0.71	0.70	0.08		*
NR-2022-07570	n.d	<1		n.d.				*
NR-2022-07570	1.16	0.13		3.92	3.87	0.44		*
NR-2022-07570	0.60	<1		2.03	2.01	0.23		*
NR-2022-07576	0.17	<1		0.56	0.55	0.06		*
NR-2022-07576	n.d	<1		n.d.				*
NR-2022-07576	n.d	<1		n.d.				*
NR-2021-07448	n.d	<1		n.d.				*
NR-2021-07448	n.d	<1		n.d.				*
NR-2021-07448	n.d	<1		n.d.				*
NR-2022-07630	0.66	<1		2.21	2.18	0.25		*
NR-2022-07630	0.81	<1		2.74	2.70	0.30		*
NR-2022-07630	1.57	0.18		5.29	5.22	0.59		*
NR-2021-10717	21.39	1.04		72.00	71.15	8.01		*
NR-2021-10717	15.24	1.06		51.30	50.69	5.71		*
NR-2021-10717	15.93	0.93		53.63	53.00	5.97		*
NR-2021-10720	14.48	0.92		48.76	48.19	5.42		*
NR-2021-10720	12.47	0.82		41.99	41.49	4.67		*
NR-2021-10720	17.64	1.14		59.40	58.70	6.61		*
NR-2022-06652	0.49	<1		1.65	1.63	0.18		*
NR-2022-06652	0.47	<1		1.58	1.56	0.18		*
NR-2021-10714	1.32	0.22		4.45	4.40	0.49		*
NR-2021-10714	1.08	0.15		3.64	3.60	0.41		*
NR-2021-10714	1.60	0.21		5.39	5.33	0.60		*
NR-2021-10726	1.81	0.15		6.11	6.04	0.68		*
NR-2021-10726	2.07	0.18		6.96	6.88	0.77		*
NR-2021-10726	1.80	0.15		6.07	5.99	0.67		*
NR-2021-10732	1.54	<1		5.17	5.11	0.58		*
NR-2021-10732	2.12	0.20		7.14	7.06	0.79		*
NR-2021-10732	2.39	0.20		8.05	7.96	0.90		*

NR-2022-07564	0.57	<1		1.91	1.89	0.21		*
NR-2022-07564	0.42	<1		1.43	1.41	0.16		*
NR-2022-07564	0.59	0.24		1.98	1.96	0.22		*
NR-2021-10729	2.50	0.21		8.40	8.30	0.93		*
NR-2021-10729	2.90	0.22		9.76	9.64	1.09		*
NR-2021-10729	3.24	0.27		10.92	10.79	1.21		*
NR-2021-07441	0.31	<1		1.04	1.03	0.12		*
NR-2021-07441	0.32	<1		1.07	1.06	0.12		*
NR-2021-07441	0.40	<1		1.33	1.32	0.15		*
MJØSBRU3-1	n.d	<1	µg/mg	n.d.			mg/g	*
MJØSBRU3-2	n.d	<1		n.d.				*
MJØSBRU3-3	n.d	<1		n.d.				*
NR-2021-09545-	Ratios off	0.24		0.77	0.72	0.24	mg/g	***
NR-2021-09545-	Ratios off	0.28		0.90	0.84	0.28		***
NR-2021-09545-	Ratios off	0.24		0.76	0.72	0.24		***
HEIMDALSVAT	0.971	n.d.		3.12	2.94	0.95		**
HEIMDALSVAT	1.325	0.11		4.26	4.01	1.30		**
NR-2021-09509-	0.539	<LOD		1.73	1.63	0.53		**
NR-2021-09509-	0.472	<LOD		1.52	1.43	0.46		**
NR-2021-09509-	0.675	<LOD		2.17	2.04	0.66		**
NR-2021-09478-	0.670	<LOD		2.15	2.03	0.66		**
NR-2021-09478-	0.576	<LOD		1.85	1.74	0.56		**
NR-2021-09478-	0.531	<LOD		1.71	1.61	0.52		**
NR-2021-09471-	0.417	<LOD		1.34	1.26	0.41		**
NR-2021-09471-	0.581	<LOD		1.87	1.76	0.57		**
NR-2021-09471-	0.574	<LOD		1.84	1.74	0.56		**
NR-2021-09530-	2.046	0.21		6.58	6.19	2.00		**
NR-2021-09530-	2.101	0.21		6.76	6.35	2.06		**
NR-2021-09530-	2.047	0.26		6.58	6.19	2.01		**
NR-2021-09538-	1.393	0.11		4.48	4.21	1.36		**
NR-2021-09538-	1.173	0.09		3.77	3.55	1.15		**
NR-2021-09538-	1.157	0.10		3.72	3.50	1.13		**
NR-2021-09457-	0.594	0.10		1.91	1.80	0.58		**
NR-2021-09457-	0.302	<LOD		0.97	0.92	0.30		**
NR-2021-09457-	0.434	<LOD		1.40	1.31	0.43		**
NR-2021-09523-	<LOD	<LOD		<LOD				**
NR-2021-09523-	0.114	<LOD		0.37	0.34	0.11		**
NR-2021-09523-	0.096	<LOD		0.31	0.29	0.09		**
NR-2021-09464-	<LOD	<LOD		<LOD				**
NR-2021-09464-	<LOD	<LOD		<LOD				**
NR-2021-09464-	<LOD	n.d.		<LOD				**
NR-2021-10137-	2.740	0.22		8.81	8.29	2.69		**
NR-2021-10137-	2.761	0.25		8.88	8.35	2.71		**
NR-2021-10137-	2.752	0.24		8.85	8.32	2.70		**
NR-2021-10143-	0.931	0.10		2.99	2.82	0.91		**
NR-2021-10143-	1.863	0.19		5.99	5.64	1.83		**
NR-2021-10143-	1.011	0.13		3.25	3.06	0.99		**
NR-2021-10714	1.97	0.11		6.32	5.95	1.93		*
NR-2021-10714	1.96	0.13		6.31	5.94	1.92		*
NR-2021-10714	2.23	0.11		7.18	6.75	2.19		*
NR-2021-10717	6.516	0.32		20.96	19.71	6.39		**
NR-2021-10717	5.497	0.32		17.68	16.63	5.39		**
NR-2021-10717	6.638	0.28		21.35	20.08	6.51		**
NR-2021-10720	8.469	0.46		27.24	25.62	8.30		**
NR-2021-10720	5.803	0.34		18.66	17.55	5.69		**
NR-2021-10720	5.381	0.40		17.31	16.28	5.27		**
NR-2021-10152	5.928	0.28		19.07	17.93	5.81		**
NR-2021-10152	7.371	0.35		23.70	22.30	7.22		**
NR-2021-10152	7.845	0.35		25.23	23.73	7.69		**
NR-2021-10155	6.400	0.30		20.58	19.36	6.27		**
NR-2021-10155	8.304	0.34		26.70	25.12	8.14		**

NR-2021-10155	8.596	0.32		27.64	26.00	8.42		**
MJØHAVN-4-1	3.744	0.27		12.04	11.33	3.67		**
MJØHAVN-4-2	2.983	0.23		9.59	9.02	2.92		**
MJØHAVN-4-3	3.540	0.33		11.38	10.71	3.47		**
DUCKM-	n.d	0.49	µg/sampl	n.d			mg/g dw	**
DUCKM-	n.d	0.20		n.d				**
DUCKM-	n.d	0.12		n.d				**
NR-2021-10694	21.513	0.70	µg/g	0.06921	0.06503	0.0260	mg/g	**
NR-2021-10695	5.848	0.63		0.01882	0.01768	0.0070		**
NR-2021-10696	6.845	0.47		0.02202	0.02069	0.0082		**
NR-2021-10697	3.903	0.24		0.01256	0.0118	0.0047		**
NR-2021-10698	6.592	1.16		0.02121	0.01993	0.0079		**
NR-2021-10699	1.789	0.48		0.00576	0.00541	0.0021		**
NR-2021-1070	6.017	0.59		0.01936	0.01819	0.0072		**
NR-2021-10701	9.185	0.62		0.02955	0.02776	0.0111		**
NR-2021-10702	11.523	0.56		0.03707	0.03483	0.0139		**
NR-2021-10703	7.458	0.55		0.024	0.02254	0.0090		**
NR-2021-10704	3.435	0.27		0.01105	0.01038	0.0041		**
NR-2021-10705	8.302	0.53		0.02671	0.02509	0.0100		**
NR-2021-10706	19.566	0.70		0.06295	0.05914	0.0237		**
NR-2021-10707	23.864	0.78		0.07678	0.07213	0.0289		**
NR-2021-10708	61.560	2.62		0.19806	0.18608	0.0745		**
NR-2021-10709	20.389	0.91		0.0656	0.06163	0.0247		**
NR-2021-10710	7.689	0.42		0.02474	0.02324	0.0093		**
NR-2021-10711	9.757	0.90		0.03139	0.02949	0.0118		**
NR-2021-10712	4.609	0.28		0.01483	0.01393	0.0055		**
NR-2021-10713	9.736	0.46		0.03132	0.02943	0.0117		**
EVERTBR-	n.d	0.17	µg/prøve	n.d			mg/prøv	**
EVERTBR-	n.d	0.18		n.d				**
EVERTBR-	n.d	0.14		n.d				**
EVERTBR-	n.d	0.26		n.d				**
EVERTBR-	n.d	0.13		n.d				**
EVERTBR-	n.d	<LOD		n.d				**
EVERTBR-	n.d	0.21		n.d				**
EVERTBR-	n.d	<LOD		n.d				**
EVERTBR-	n.d	0.16		n.d				**
NR-2021-06284	3.425	0.36	µg/prøve	0.01102	0.01035	0.0041	mg/prøv	**
NR-2021-06285	4.869	<LOD		0.01567	0.01472	0.0059		**
NR-2021-06293	5.919	0.48		0.01904	0.01789	0.0071		**
NR-2021-06294	0.960	0.23		0.00309	0.0029	0.0011		**
NR-2021-06295	1.062	0.22		0.00342	0.00321	0.0012		**
NR-2021-06296	3.759	0.34		0.0121	0.01136	0.0045		**
NR-2021-07409	n.d	2.16		n.d				**
NR-2021-07410	n.d	0.48		n.d				**
NR-2021-07411	n.d	0.14		n.d				**
NR-2021-07417	n.d	0.24		n.d				**
NR-2021-07418	n.d	0.41		n.d				**
NR-2021-07421	n.d	0.26		n.d				**
NR-2021-07427	n.d	0.21		n.d				**
NR-2021-07428	n.d	2.88		n.d				**
NR-2021-07429	n.d	0.10		n.d				**
NR-2021-07433	n.d	0.20		n.d				**
NR-2021-07434	n.d	0.15		n.d				**
NR-2021-07435	n.d	0.60		n.d				**
NR-2021-07436	n.d	0.21		n.d				**
NR-2021-07437	n.d	0.38		n.d				**
NR-2022-06659	8.061	0.87		0.02594	0.02437	0.0097		**
NR-2022-07546	n.d	0.44		n.d				**
NR-2022-07547	11.367	0.43		0.03657	0.03436	0.0137		**
NR-2022-07552	20.483	1.39		0.0659	0.06191	0.0248		**
NR-2022-07553	n.d	0.22		n.d				**

NR-2022-07554	n.d	0.15		n.d				**
NR-2022-07558	7.267	0.48		0.02338	0.02197	0.0088		**
NR-2022-07559	1.699	0.20		0.00547	0.00514	0.0020		**
NR-2022-07560	4.379	0.22		0.01409	0.01324	0.0053		**
NR-2022-07612	n.d	0.23		n.d				**
NR-2022-07613	3.906	1.25		0.0126	0.0118	0.0047		**
NR-2022-07614	n.d	2.01		n.d				**
NR-2022-07648	n.d	0.19		n.d				**
NR-2022-07649	n.d	0.17		n.d				**
NR-2022-07650	n.d	0.25		n.d				**
NR-2022-07654	n.d	4.16		n.d				**
NR-2022-07655	2.805	0.20		0.00902	0.00848	0.0034		**
NR-2022-07656	n.d	0.18		n.d				**
NR-2022-07660	n.d	0.24		n.d				**
NR-2022-07661	n.d	0.85		n.d				**
NR-2022-07662	n.d	0.23		n.d				**
NR-2023-09459	n.d	0.22		n.d				**
NR-2023-09460	n.d	0.21		n.d				**
NR-2023-09462	n.d	0.75		n.d				**
NR-2023-09463	2.613	0.71		0.00841	0.0079	0.0031		**
NR-2023-09464	n.d	0.22		n.d				**
NR-2023-09461	57.681	1.87		0.18558	0.17435	0.0698		**

Table 19. Results of the spike and recovery tests for Heimdalsvatnet using single shot (SS) analysis, comparing between the treatments 1) Sodium dodecyl sulphate (SDS)+Fentons reagent, 2) SDS+Fentons reagent+enzymes, S) DS+Fentons reagent+KOH, 4)SDS+Fentons reagent+KOH+enzymes and no treatment. Recovery rates 90-110% is depicted in green, 80-120% in yellow and <80%, >120% in red.

M4	SBR µg/cup	SBR µg/mg	weight	Spike	Mean background	% recovery	Treatment
HEIMT5-4	4.01	0.46	8.7791	no			
HEIMT5-5	2.56	0.31	8.1322	no	0.39		
HEIMT5-6-SPIKE	14.38	1.79	8.0301	20		56.38	SDS+FENTONs
HEIMT6-4	2.52	0.31	8.175	no			
HEIMT6-5	2.10	0.26	8.0394	no	0.29		
HEIMT6-6-SPIKE	13.86	1.68	8.2661	20		57.51	SDS+FENTONS +ENZYMES
HEIMT7-4	5.63	0.64	8.7843	no			
HEIMT7-5	1.95	0.24	8.1941	no	0.44		
HEIMT7-6-SPIKE	16.91	2.03	8.3124	20		66.28	SDS+FENTONS +KOH
HEIMT8-4	2.94	0.33	8.8772	no			
HEIMT8-5	1.42	0.17	8.1845	no	0.25		
HEIMT8-6-SPIKE	14.50	1.77	8.1904	20		62.15	SDS+FENTONS +KOH+ENZYMES
HEIM-4	72.43	9.01	8.0401	no			
HEIM-5	55.82	6.42	8.6934	no	7.71		
HEIM-6-SPIKE	45.57	5.23	8.7085	20		-108.09	UNTREATED
M3	**SBR µg/cup**	**SBR µg/mg**	**weight**	**Spike**	**Mean background**	**% recovery**	**Treatment**
HEIMT5-4	1.30	0.15	8.7791	no			
HEIMT5-5	1.62	0.20	8.1322	no	0.17		SDS+FENTONs

HEIMT5-6-SPIKE	11.13	1.39	8.0301	20		48.66	
HEIMT6-4	0.12	0.01	8.175	no			
HEIMT6-5	0.13	0.02	8.0394	no	0.02		SDS+FENTONS
HEIMT6-6-SPIKE	10.98	1.33	8.2661	20		54.29	+ENZYMES
HEIMT7-4	0.16	0.02	8.7843	no			
HEIMT7-5	0.12	0.02	8.1941	no	0.02		
HEIMT7-6-SPIKE	12.93	1.56	8.3124	20		63.96	SDS+FENTONS+KOH
HEIMT8-4	0.14	0.02	8.8772	no			
HEIMT8-5	0.10	0.01	8.1845	no	0.01		SDS+FENTONS
HEIMT8-6-SPIKE	10.81	1.32	8.1904	20		53.46	+KOH+ENZYMES
HEIM-4	7.81	0.97	8.0401	no			
HEIM-5	11.52	1.33	8.6934	no	1.15		
HEIM-6-SPIKE	26.17	3.00	8.7085	20		80.83	UNTREATED
4-VCH	**SBR µg/cup**	**SBR µg/mg**	**weight**	**Spike**	**Mean background**	**% recovery**	**Treatment**
HEIMT5-4	0.00	0.00	8.7791	no			
HEIMT5-5	0.00	0.00	8.1322	no	0.00		
HEIMT5-6-SPIKE	22.81	2.84	8.0301	20		114.07	SDS+FENTONs
HEIMT6-4	0.00	0.00	8.175	no			
HEIMT6-5	0.00	0.00	8.0394	no	0.00		SDS+FENTONS
HEIMT6-6-SPIKE	22.27	2.69	8.2661	20		111.35	+ENZYMES
HEIMT7-4	0.00	0.00	8.7843	no			
HEIMT7-5	0.00	0.00	8.1941	no	0.00		SDS+FENTONS
HEIMT7-6-SPIKE	23.77	2.86	8.3124	20		118.85	+KOH
HEIMT8-4	0.00	0.00	8.8772	no			
HEIMT8-5	0.00	0.00	8.1845	no	0.00		SDS+FENTONS
HEIMT8-6-SPIKE	22.06	2.69	8.1904	20		110.29	+KOH+ENZYMES
HEIM-4	0.00	0.00	8.0401	no			
HEIM-5	0.92	0.11	8.6934	no	0.05		
HEIM-6-SPIKE	19.01	2.18	8.7085	20		92.75	UNTREATED

Table 20. Results of the spike and recovery tests for Heimdalsvatnet using double shot (DS) analysis, comparing between the treatments 1) Sodium dodecyl sulphate (SDS)+Fentons reagent, 2) SDS+Fentons reagent+enzymes, S) DS+Fentons reagent+KOH, 4) SDS+Fentons reagent+KOH+enzymes and no treatment. Recovery rates 90-110% is depicted in green, 80-120% in yellow and <80%, >120% in red.

M4	SBR µg/cup	SBR µg/mg	weight	Spike	Mean background	% recovery	Treatment
HEIMT5-1	1.18	0.14	8.2691	no			
HEIMT5-2	2.40	0.28	8.4752	no	0.21		
HEIMT5-3-SPIKE	18.28	2.24	8.1682	20		82.73	SDS+FENTONs
HEIMT6-1	2.24	0.27	8.3314	no			
HEIMT6-2	2.70	0.30	8.8912	no	0.29		
HEIMT6-3-SPIKE	24.21	2.71	8.9265	20		108.24	SDS+FENTONS+ENZYMES
HEIMT7-1	3.98	0.47	8.5142	no			
HEIMT7-2	2.16	0.25	8.7728	no	0.36		
HEIMT7-3-SPIKE	18.57	2.16	8.5831	20		77.55	SDS+FENTONS+KOH
HEIMT8-1	3.45	0.41	8.4123	no			
HEIMT8-2	3.23	0.40	8.1006	no	0.40		
HEIMT8-3-SPIKE	18.15	2.12	8.5783	20		73.39	SDS+FENTONS+KOH+ENZYMES
HEIMT1-1	####	####	8.713	no			
HEIMT1-2	63.23	7.58	8.3437	no	10.10		
HEIMT1-3-SPIKE	60.83	6.89	8.8261	20		-141.42	UNTREATED
M3	**SBR µg/cup**	**SBR µg/mg**	**weight**	**Spike**	**Mean background**	**% recovery**	**Treatment**
HEIMT5-1	1.18	0.14	8.2691	no			
HEIMT5-2	2.46	0.29	8.4752	no	0.22		
HEIMT5-3-SPIKE	18.97	2.32	8.1682	20		86.01	SDS+FENTONs
HEIMT6-1	1.89	0.23	8.3314	no			
HEIMT6-2	2.18	0.25	8.8912	no	0.24		
HEIMT6-3-SPIKE	22.50	2.52	8.9265	20		101.98	SDS+FENTONS+ENZYMES
HEIMT7-1	2.94	0.35	8.5142	no			
HEIMT7-2	1.65	0.19	8.7728	no	0.27		
HEIMT7-3-SPIKE	18.29	2.13	8.5831	20		80.00	SDS+FENTONS+KOH
HEIMT8-1	2.58	0.31	8.4123	no			
HEIMT8-2	2.52	0.31	8.1006	no	0.31		
HEIMT8-3-SPIKE	16.28	1.90	8.5783	20		68.15	SDS+FENTONS+KOH+ENZYMES
HEIMT1-1	32.66	3.75	8.713	no			
HEIMT1-2	23.12	2.77	8.3437	no	3.26		
HEIMT1-3-SPIKE	32.62	3.70	8.8261	20		19.27	UNTREATED
4-VCH	**SBR µg/cup**	**SBR µg/mg**	**weight**	**Spike**	**Mean background**	**% recovery**	**Treatment**
HEIMT5-1	0.00	0.00	8.2691	no			
HEIMT5-2	0.00	0.00	8.4752	no	0.00		
HEIMT5-3-SPIKE	24.11	2.95	8.1682	20		120.56	SDS+FENTONs
HEIMT6-1	0.00	0.00	8.3314	no			
HEIMT6-2	0.00	0.00	8.8912	no	0.00		
HEIMT6-3-SPIKE	21.55	2.41	8.9265	20		107.77	SDS+FENTONS+ENZYMES

HEIMT7-1	0.00	0.00	8.5142	no			
HEIMT7-2	0.00	0.00	8.7728	no	0.00		
HEIMT7-3-SPIKE	23.62	2.75	8.5831	20		118.11	SDS+FENTONS+KOH
HEIMT8-1	0.00	0.00	8.4123	no			
HEIMT8-2	0.00	0.00	8.1006	no	0.00		
HEIMT8-3-SPIKE	21.54	2.51	8.5783	20		107.71	SDS+FENTONS+KOH+ENZYMES
HEIMT1-1	0.01	0.00	8.713	no			
HEIMT1-2	0.01	0.00	8.3437	no	0.00		
HEIMT1-3-SPIKE	18.40	2.08	8.8261	20		91.95	UNTREATED

5.4 Results

All results are presented at NIVAs interactive website superset, where data can be downloaded, and the results can be filtered and explored by the viewer. In this report we present a highlight of the most interesting findings. In this chapter, we present microplastic counts, their calculated masses and for those samples where it has been analysed, and the concentrations of tyre wear particles and pyrolysis data for UV compounds and polymers in air. The raw data results from the air samples are included as well.

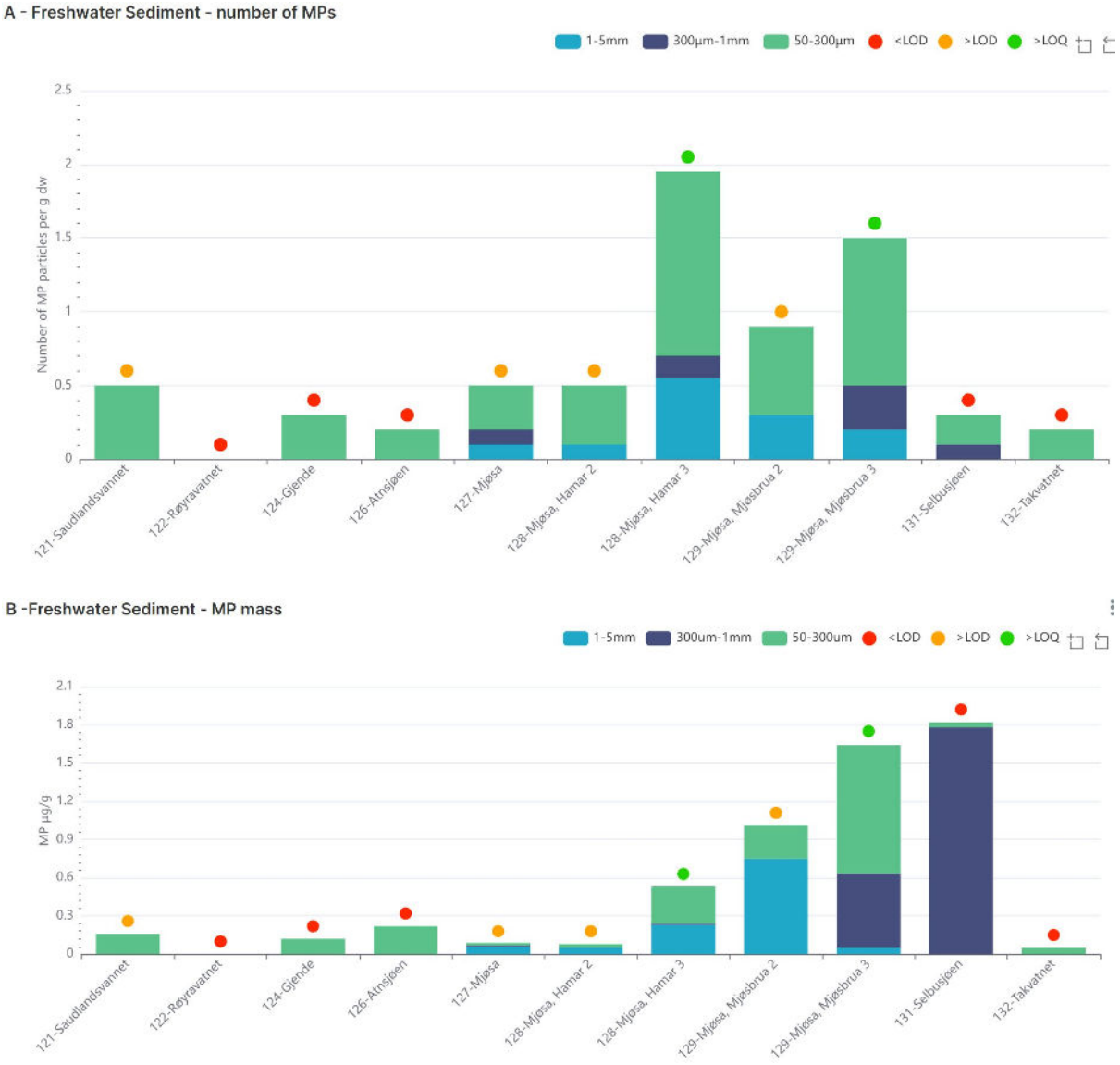


Figure 28. Freshwater sediments. **A** showing number of microplastic particles per g dw, **B** showing their respective masses, as calculated from the volume and density for each particle. Each station is marked with a coloured point, describing whether the results are under LOD (red), over LOD but under LOQ (orange), and over LOQ (green).

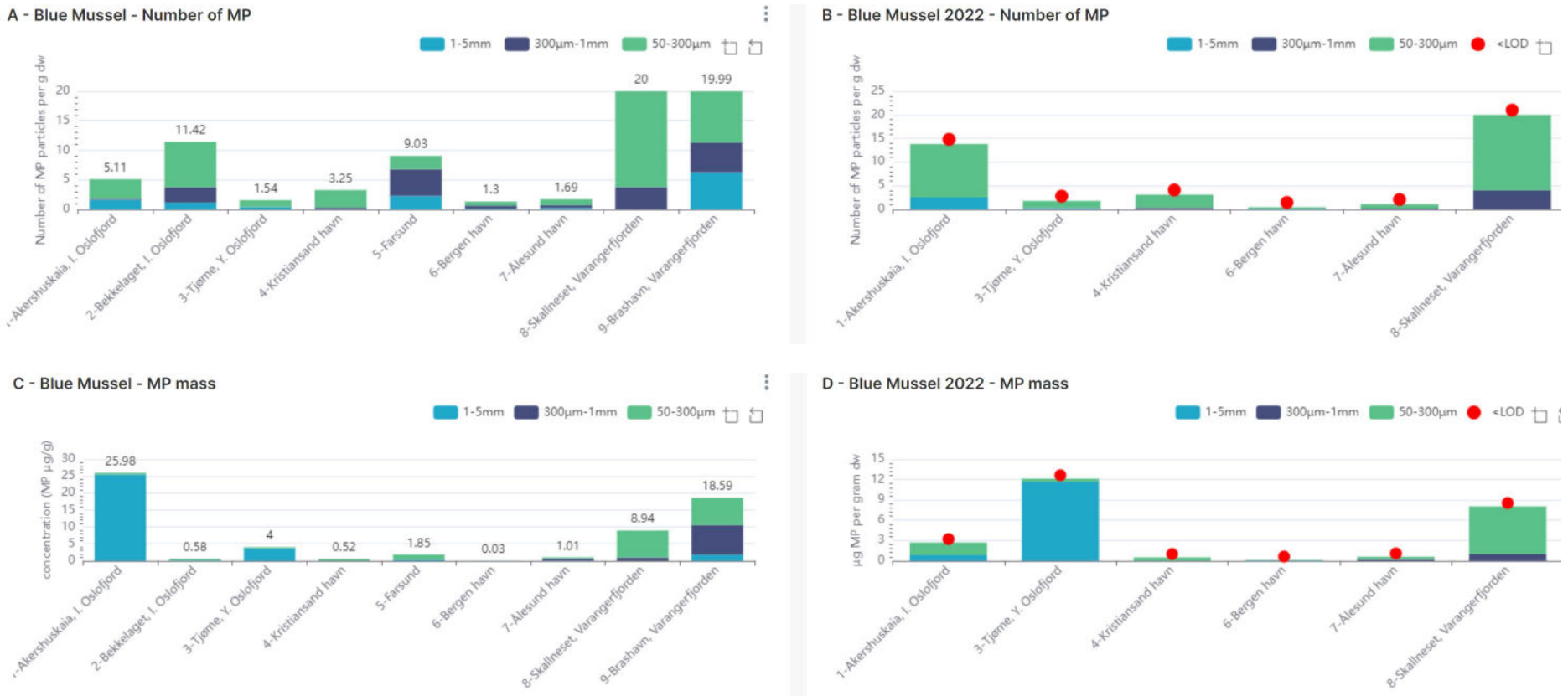


Figure 29. Blue mussels (LOD/LOQ available for 2022 samples only) **A** showing number of microplastic particles per g dw, for 2021 and 2022 results combined, and for **B** 2022 data separately. **C** showing their respective masses, as calculated from the volume and density for each particle, for both year's results combined, and **D** for 2022 data separately. Each station for 2022 year's data (**B** and **D**) is marked with a coloured point, describing whether the results are under LOD (red), over LOD but under LOQ (orange), and over LOQ (green). Note that all results are under LOD. For TWP, see key findings.

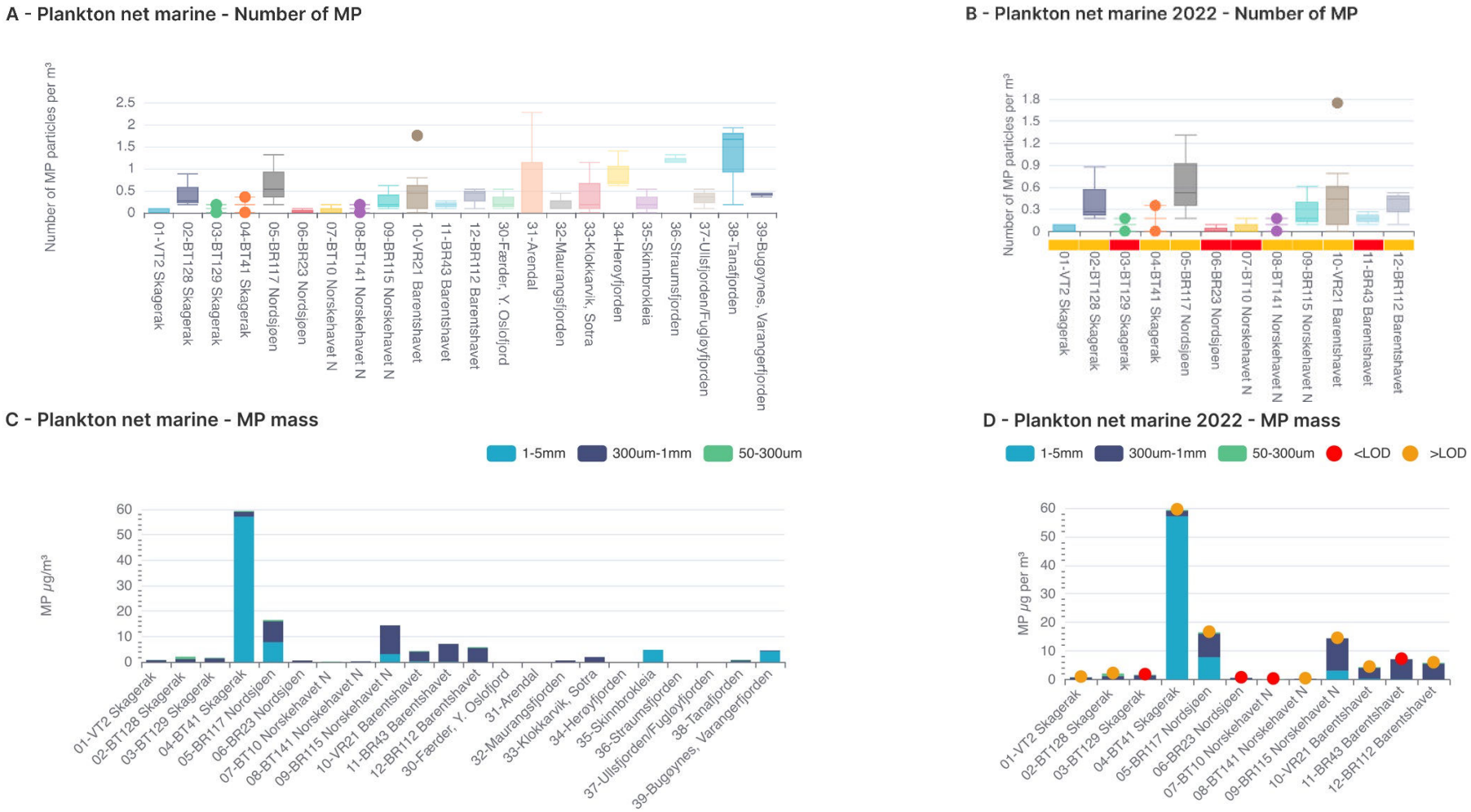


Figure 30. Water samples from marine plankton net hauls (LOD/LOQ available for 2022 samples only)**. A** showing number of microplastic particles per m^3, for 2021 and 2022 results combined, and **B** 2022 data separately. **C** showing their respective masses, as calculated from the volume and density for each particle, for both year's results combined, and **D** for 2022 data separately. Each station for 2022 year's data (**B** and **D**) is marked with a coloured x-axes annotation (line or dots), describing whether the results are under LOD (red), over LOD but under LOQ (orange), and over LOQ (green). The results from the field blanks showed contamination results in line with the numbers in the environmental samples. Fibres are excluded.

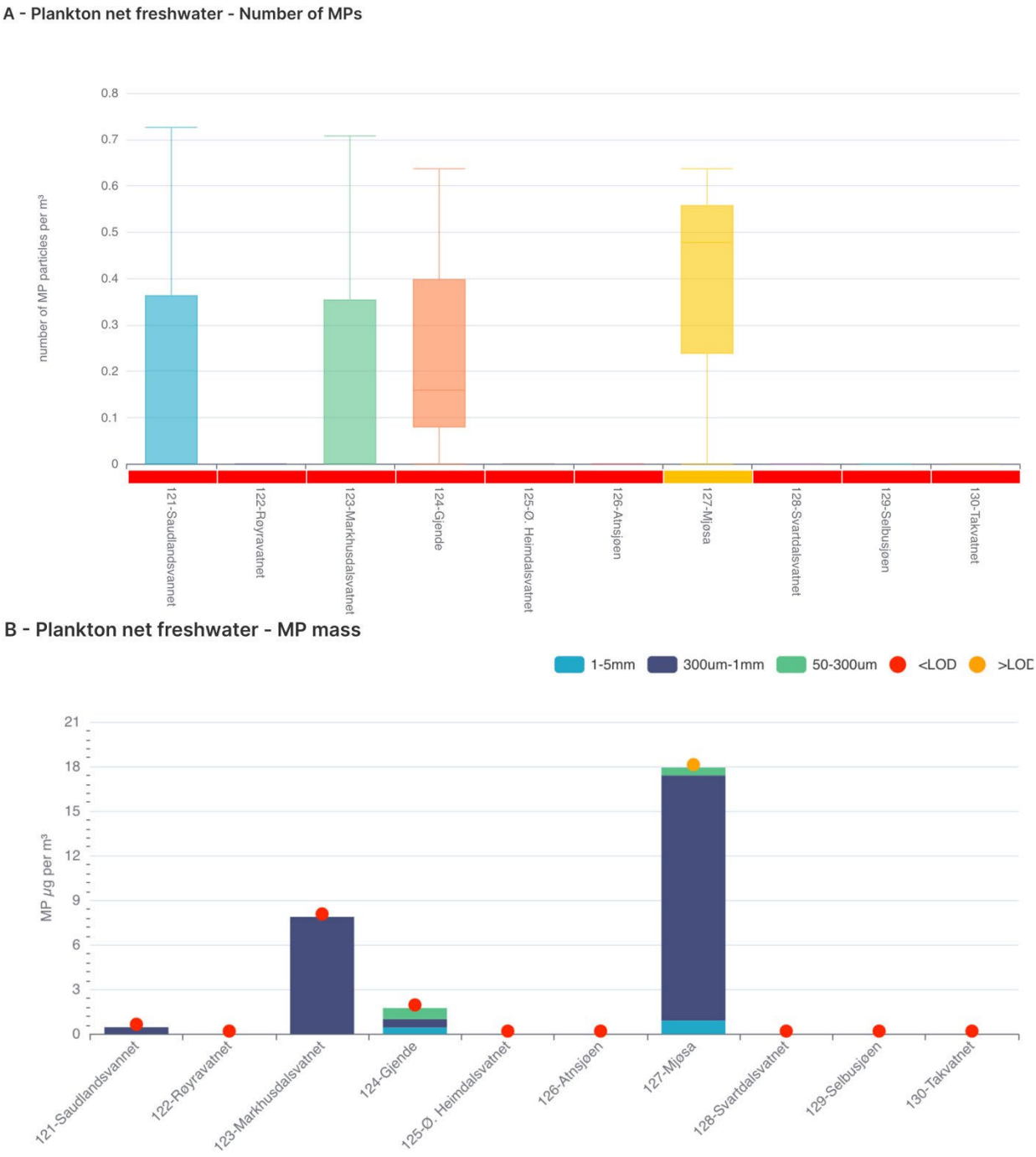


Figure 31. Water samples from freshwater plankton net hauls**. A** showing number of microplastic particles per m^3, **B** showing their respective masses, as calculated from the volume and density for each particle. All results are marked with a coloured x-axes annotation (line or dots), describing whether the results are under LOD (red), over LOD but under LOQ (orange), and over LOQ (green). The results from the field blanks showed contamination results in line with the numbers in the environmental samples. Fibres are excluded.

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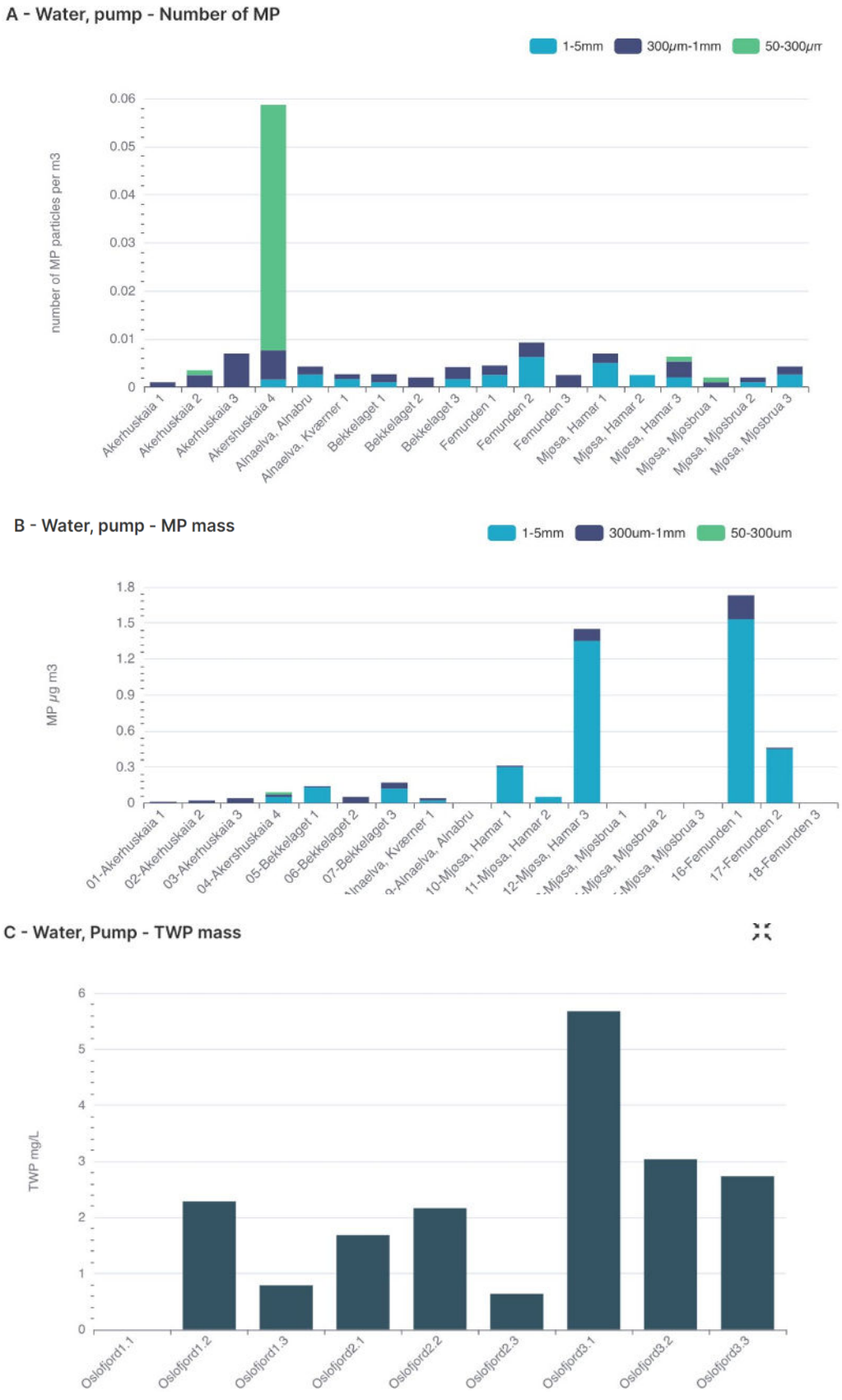


Figure 32. Water pump samples **A.** showing number of microplastic particles per m^3, **B** showing their respective masses, as calculated from the volume and density for each particle (µg/m^3). LOD/LOQ were available for 2022 year's sample (Akershuskaia 4). This sample was above LOQ. **C**. showing the TWP (mg/L) for the Akershuskaia 4 samples (n=3) from 2022.

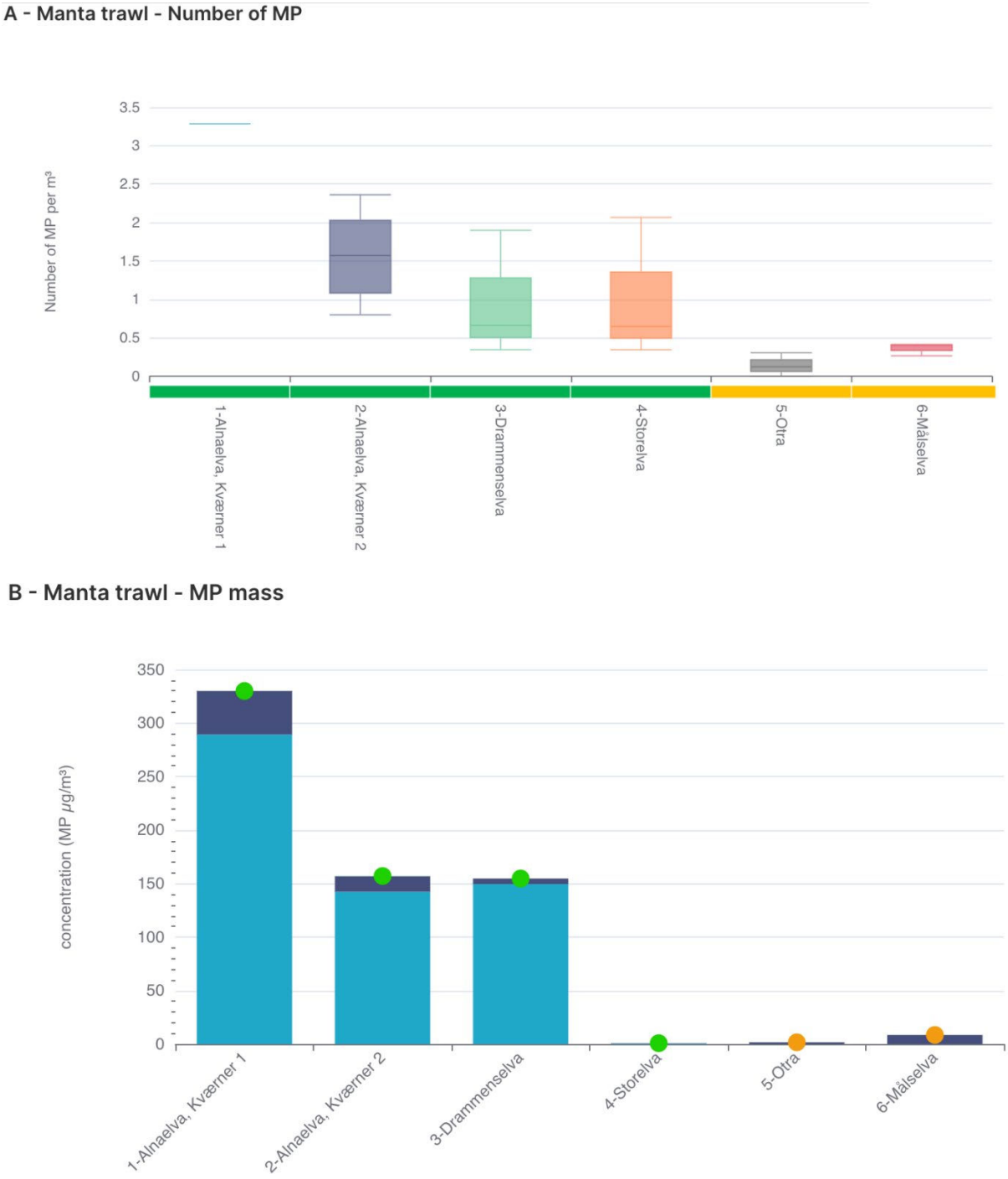


Figure 33. Manta trawl samples A. showing number of microplastic particles per m^3, **B** showing their respective masses, as calculated from the volume and density for each particle. All results are marked with a coloured x-axes annotation (line or dots), describing whether the results are under LOD (red), over LOD but under LOQ (orange), and over LOQ (green). Field blanks exhibited lower numbers of microplastics than the samples for the samples from Alnaelva and Storelva, but were in line with the results from Otra and Målselva.

UV compounds in air samples

Among the 5 UV compounds analysed, UV 326 and UV 328 were among the most abundant (Figure 35). Active air samples contained a larger variety of UV compounds, collecting suspended atmospheric particles, at concentrations ranging between <LOD and 0.14 ng/m^3 for UV 326 and <LOD and 0.06 ng/m^3 for UV 328.

As Figure 34 illustrates, are UV compound concentrations on average much lower in active samples from Zeppelin than in samples from Birkenes. In contrast, deposition samples do not show such a clear difference (Figure 34).

The composition of sumUV varies within stations and sampling method (see appendix 5.5).

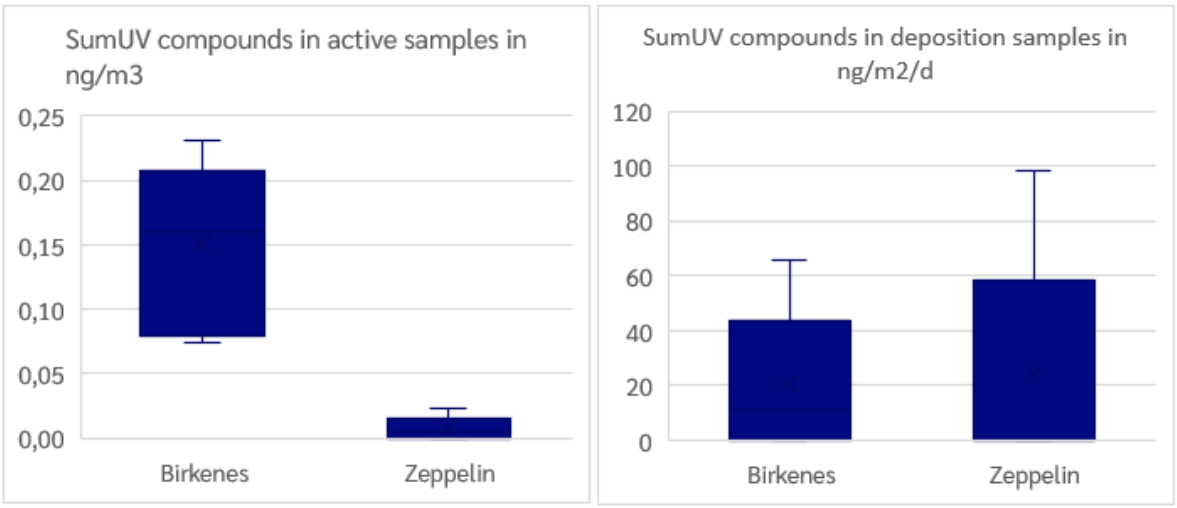


Figure 34. SumUV compounds concentrations in active (left) and deposition samples (right) at both sites.

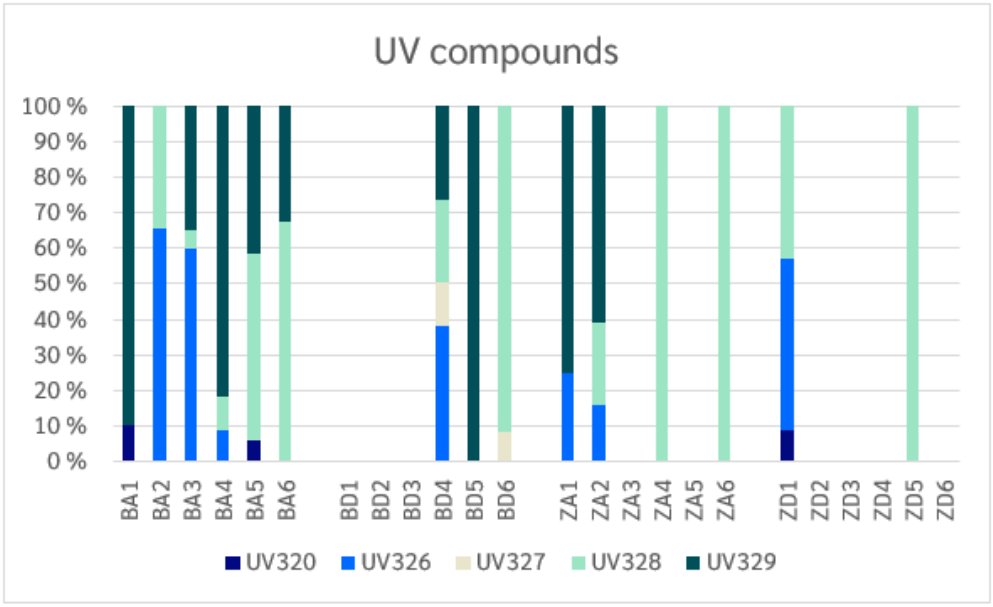


Figure 35. Relative composition of UV compounds in active and deposition samples.

5.5 Sample collection, laboratory treatment and analytical methods

Plankton net samples

Sample Collection

Vertical plankton samples were provided to MIKRONOR through the national monitoring programs ØKOKYST, ØKOSTOR and ØKOFERSK. The samples collected with the plankton net indicate the amount of microplastic per cubic meter in the water column to a given depth. A Standard Operating Procedure (SOP) was developed to ensure synergies within the sampling activities already being performed. The methods used for plankton net sampling are based on available international guidelines (AMAP, 2021; GESAMP, 2019; Michida et al., 2019), in addition to contamination and quality control procedures (Brander et al., 2020). Read more under field blanks and contamination control.

For each sampling, the vessel was stationed at a fixed position and a plankton net was deployed down to the decided depth (50 m) and slowly hauled to the surface at a speed of 0.5 m/s. For marine samples, the net diameter was 0.6 m, with a mesh size of 200 µm, resulting in a theoretical volume of 14.14 m^3. A flow meter was used to control and to calculate the volumes of water which passed through the net. The volumes measured by the flow meter agreed almost with the theoretical volumes. An average measured volume of 11.45 m^3 was defined for all samples, as it was considered more accurate than the theoretical volume based on net opening and approximate depth. For freshwater samples, nets with diameters of 0.2 and 0.3 m and 90 µm mesh size were used.

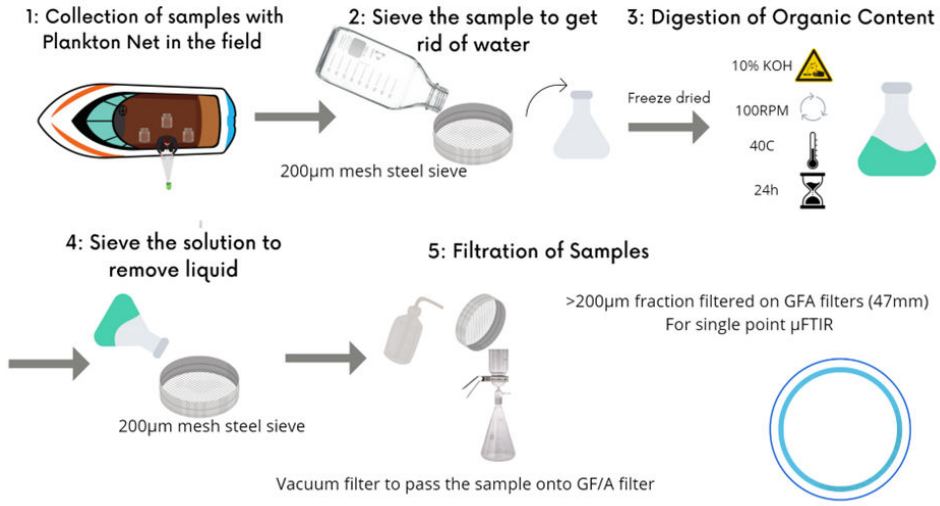
When the net was returned to the surface, the outside of the net was flushed to ensure all particles moved to the cod-end. The concentrated sample in the cod-end was rinsed into a 1L glass jar with RO-water. All samples were kept in the dark and refrigerated after collection, before being sent to NIVA to carry out analyses.

To minimise the risk of contaminating the samples with microplastics, the net was carefully cleaned before collecting a net blank. This was done by deploying the net without the cod-end attached and flushing it from the outside of the net with seawater. This was followed by the collection of the net field blank: a cleaned cod-end was attached, and the net was flushed again several times (at least four times on all sides) to transfer the contents into the cod end, from where it was transferred to a sample bottle using RO-water. In addition to net blanks, atmospheric field blanks were set up around the area where the samples were collected. Atmospheric field blanks give an overview of potential contamination from airborne microplastic particles during sampling, and to further quality check the microplastic we find in our samples.

To best control potential contamination from the net used for the plankton haul, the net blank samples were visually analysed first. If the sample had more than 50 fibres the number of fibres were counted, but not further analysed. The fragments in the sample were still counted and analysed. If the sample had less than 50 fibres, all fibres and fragments were counted and analysed further. Next step for quality control was to compare number of fibres (and other microplastics if needed) in the net blank with the number of fibres in the samples. If number of fibres in the net blank was higher or similar to number in the samples, the fibres in the samples are not reported but regarded as contamination. In the majority of the samples from 2022, the fibres in the net blanks exceeded the numbers in the samples, we have decided to exclude all fibres from reporting this year. Hopefully the more rigorous cleaning procedures and contamination control implemented in 2023 sampling, will result in less fibre contamination. This process was done to ensure that the values of microplastic found in the sample gives a realistic indication of the number of microplastics found in the environment.

For results of field blanks, se Appendix 5.1

Laboratory Procedure



To prepare the samples for microplastic analysis, the following procedure was followed:

1. Sieve the whole sample with a 200 µm pore sized mesh and remove big plant materials if needed but ensure to rinse properly off before.
2. Rinse the material into glass jars (200mL) and put into the freezer on -21°C overnight.
3. Freeze dry the samples until the material is dried.
4. Add 10% KOH to reach between 50 – 100mL to completely cover the sample.
5. Place the glass jar in the incubator (100 rpm, at 40 °C for 24 hours).

After the processing steps, the samples were filtered onto GFA filters using a glass vacuum pump before being analysed. The analysis steps include microscopy to identify the microplastics found in the samples, followed by characterizing of the microplastic particles (type of polymer) using Fourier-transform infrared spectroscopy (FTIR). Read more about analytical methods here

Laboratory Blanks and LOD/LOQ

To test for laboratory contamination, analysis of procedural blanks in the lab were carried out. These blanks follow the same preparatory steps as the samples and were included every day of the sample processing. One blank was done in the beginning of the process, one during the process and one at the end of the process. Any microplastic particles detected in the blanks was characterised.

For results of laboratory blanks, see Appendix 5.1

Recovery/Method Validation

Recovery tests were carried out for water samples in 2022 and reported in the annual report from 2022. Standard microplastic reference material (SRM) in form of soda tablets were used. These SRM contains plastic particles made of three polymer types: PS, PVC and PE in size range 125-350 µm (Table 1). For plankton net samples, 10 PE particles with size 450 µm were manually added in addition, as particles < 180 µm from SRM (PVC and PE) will pass through sieves under processing. SRMs were added to the water samples before processing and to the biota samples at the first step under processing. Since the particles in SRM have a specific shape/size, only visual analysis under a microscope was needed to identify SRM particles. Processing large volume water samples had for plankton nets a 100%, 95% and 92% recovery rate.

High-volume water pump system (200 and 50 µm mesh)

Sample Collection

High-volume water samples (HW200 and HW50) were provided to MIKRONOR through the programmes Urban Fjord and Elveovervåkingsprogrammet. A SOP was developed to ensure synergies with the sampling activities already being performed in each programme, and comparison between the programmes. The methods used for high-volume sampling are based on available international guidelines (AMAP, 2021; GESAMP, 2019; Michida et al., 2019), in addition to contamination and quality control procedures (Brander et al., 2020).

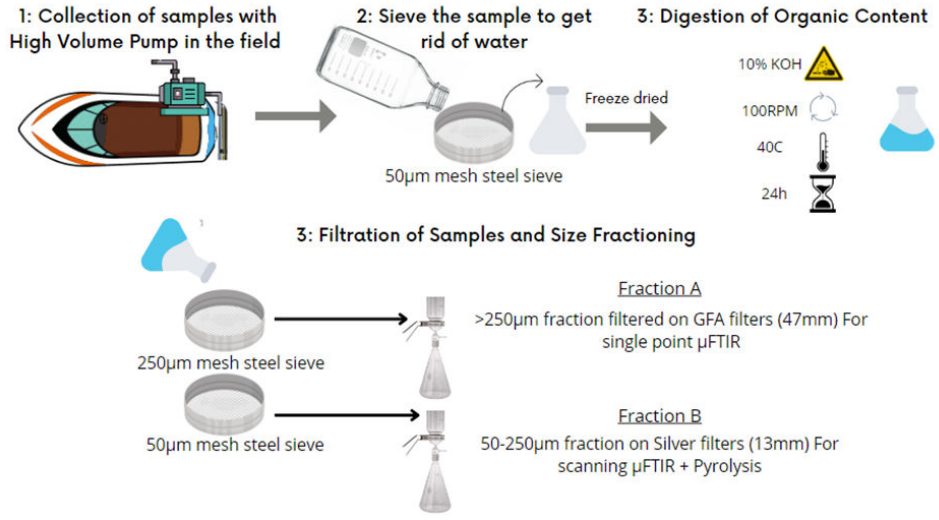
The high-volume pump system was adapted using an existing pump system (KCDenmark, Silkeborg, Denmark) which was redesigned to allow the collection of water samples (freshwater and marine) at a maximum speed of 200 L/min. The high-volume water samples of the surface water, approximately 0.5 m below the surface were taken at one point in 2022 (3 replicates), and at three points in 2021 (1 replicate per station). A metal filter (Axium, Swansea, UK) were used with 50 µm mesh size. A filter was also fitted at the water intake so that particles larger than 5 mm do not enter the system. A volume of 1000 L was filtered for each sample. A flow meter was in place to ensure the correct measurement of the water volume was collected.

Field Blanks

The blank sample was taken to check for any potential contamination from the pump-system. This was done by flushing the system with filtered water (RO-water), and take a sample, but without having the system in the water.

For results, see Appendix 5.1.

Laboratory Procedure



The 50 µm fraction was analysed with µFTIR + pyrolysis, and the 200 µm fraction was analysed with FTIR.

To prepare the samples for microplastic analysis, the following procedure was followed:

1. The whole sample was sieved with a 50 µm pore sized mesh. Then the material was rinsed into glass jars and put into a freezer on -21°C overnight. Following day, the samples were freeze dried until the material was dry.

2. Between 50 – 100 mL 10% KOH was to the samples, before the samples were kept in an incubator at 100 rpm, at 40 °C for 24 hours.
3. The samples were then split into 50 µm pore sized mesh and the 250 µm pore sized mesh for filtrating onto filter.

After the processing steps, the samples were filtred onto GFA filters for 250 µm fraction, and onto silver filters for the 50-250 µm fraction using a glass vacuum pump before being analysed. The analysis steps include firstly microscopy to identify the microplastics found in the samples, then followed by characterizing the microplastics (type of polymer) using Fourier-transform infrared spectroscopy (FTIR).

Laboratory Blanks and LOD/LOQ

To test for laboratory contamination, analysis of procedural blanks in the lab were carried out. These blanks follow the same steps as the samples and are included each day of the sample processing. One blank was done in the beginning of the process, one during the process and one at the end of the process.

For results of laboratory blanks, see Appendix 5.1. For recovery tests, we refer to MIKRONOR 1 report (2022).

Ferrybox Samples collected from Oslo- Kiel transect:

Sample Collection

Ferrybox samples were provided to MIKRONOR through the Ocean Acidification monitoring program. The SOP used follows previously published methods (Lusher et al., 2021; van Bavel et al., 2020).

The Ferrybox system is set up to collect water from a seawater intake situated at 5 m depth on the starboard side of 'M/S Color Fantasy'. The system is remotely operated to start sampling and to stop at designated positions along the vessels transect (see maps at superset). The NIVA microplastic sampling module connected to the Ferrybox enables the sampling of relatively large volumes of sea water (5000-15000 L), thus improving the limit of detection (LOD, numbers of microplastic particles/L). The system also accurately measures the volume of seawater improving the accuracy of the microplastic concentration (flow precision < 0.2%).

Number of samples of per station/travelled distance:

- 1 x 100-200 µm sieve
- 1 x 200-500 µm sieve
- 1 >500 µm
- 1 x atmospheric blank (on 100 µm sieve)

Each sample was collected over the south bound (Oslo-Kiel) and the north bound (Kiel-Oslo) resulting in two time periods and volumes (Table 1) collected on the same standard system set-up with three filters: 500 µm, 200 µm and 100 µm. These are stacked sequentially for size fractionation and subsequent analysis in the laboratory. The volume of water filtered was measured by the built-in flow meter allowing all samples to be standardised to "per cubic meter filtered (m^3)". Following each sample period, the filters were removed from the Ferrybox and the samples were packed in aluminium foil, frozen and transported to the lab as soon as possible.

Field Blanks

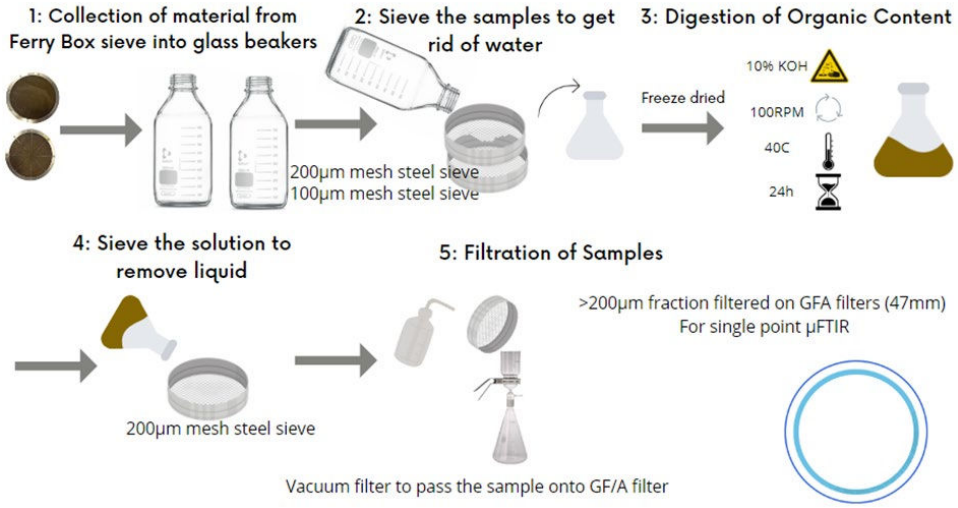
To control contamination, atmospheric blanks were carried out when collecting the ferry box samples. Therefore, before opening the Ferry Box (FB) system and taking out the sieves with the samples, a box

with a clean 100 µm sieve was opened and put on the working bench. When closing the sieves with the samples in their own box, the sieve with the atmospheric blank was put in its own box.

For results from field blanks, see Appendix 5.1.

Laboratory Procedure

The biological material in the filters needs to be broken down to leave only non-digestible material, including plastics before analyses. KOH is a recommended method commonly used to remove organic material from a sample, without harming any plastic particles present in the matrix. KOH is highly basic, forming strongly alkaline solutions in water and other polar solvents. This method has been recommended and included in international guidelines e.g., GESAMP, 2019 and AMAP, 2021. Accordingly, NIVA has optimised the method for use in monitoring programs for the different water samples, including Ferrybox samples (Lusher et al., 2021).



To prepare the samples for microplastic analysis, following procedure was followed:

1. The material from the Ferrybox filters was rinsed into 3 L glass beakers (one glass beaker for the 100 µm sieve, one for the 200 µm sieve, and one for the 100 µm field blank sieve)
2. To reduce volume, samples were sieved again with respective mesh size (100 or 200 µm) and transferred through thoroughly rinsing with distilled water, into 250 mL Erlenmeyer flasks
3. Transfer the material from the sieve into a 250 ml Erlenmeyer flask using a squirt bottle with RO-water and rinsing thoroughly (at least 3 times)
4. Concentrated KOH was added to the samples to reach 10% concentration and the samples were incubated at 40 °C and 100 RPM overnight.
5. The following day, the samples were sieved (100 or 200 µm, respectively), and filtered onto GF/A filters for further analyses (see Visual analyses, Single point FTIR and µFTIR scanning)

After the processing steps, the samples were filtered onto GFA filters using a glass vacuum pump before being analysed. The analysis steps include firstly microscopy to identify the microplastics found in the samples, then followed by characterizing the microplastics (type of polymer) using Fourier-transform infrared spectroscopy (FTIR).

Laboratory Blanks and LOD/LOQ

To test for laboratory contamination, and to ensure that we don't contaminate our samples in the preparation process, procedural blanks in the lab were carried out. These blanks follow the same steps as the samples and are included each day of the sample processing. One blank is done in the beginning of the process, one during the process and one at the end of the process.

For results, see Appendix 5.1

Recovery/Method Validation

Recovery tests were carried out for Ferrybox in 2022. These three have different size ranges and processing procedures. Therefore, recovery tests were made for four different cases. For samples with lower size limit 50-100 µm standard microplastic reference material (SRM) in form of soda tablets were used. These SRM contains plastic particles made of three polymer types: PS, PVC, and PE in size range 125-350 µm (Martínez-Francés et al., 2023). For plankton net samples, 10 PE particles with size 450 µm were manually added in addition, as particles < 180 µm from SRM (PVC and PE) will pass through sieves under processing. SRMs were added to the water samples before processing. Since the particles in SRM have a specific shape/size, only visual analysis under a microscope was needed to identify SRM particles.

For results, see Appendix 5.1

Samples from Svalbard:

Sample Collection

Sampling for microplastics was conducted in four fjords at Svalbard between June 28–July 2, 2022. Sampling was carried out using two methods: with a neuston net, which catches microplastics from the surface, 0-20 cm depth, and with on-board pump taking water from 1-1.5 m depth. Two sampling sites were identified in Isfjorden, Sassenfjorden and Tempelfjorden for both methods while Adventfjorden was divided into two and three sampling sites for net and pump sampling respectively (Figure 36). Sampling was conducted aboard a Polar cirkel boat equipped with a manually operated winch. The boat was made of PE-100 plastic, 8.5 m length and 2.7 wide; 6 scientists were working on board. Totally it was taken 9 pump samples and 29 net samples. One of net samples was lost (in Isfjorden, IF1.2).

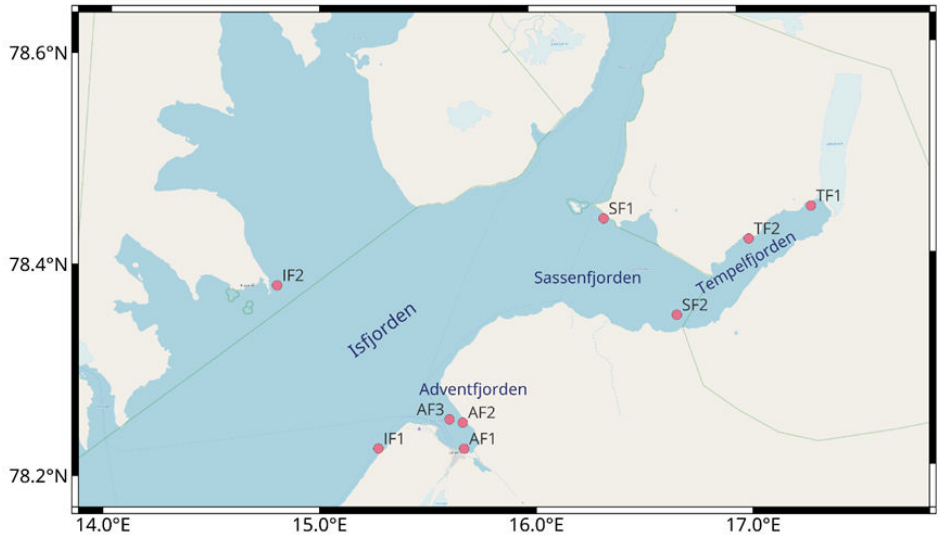


Figure 36. Microplastic sampling sites (start coordinates) at Svalbard 2022

Sample collection with Neuston net: The collection of microplastics floating on the sea surface was carried out by neuston net (40 x 60 cm opening, nylon material, 0.333 mm mesh size) trawling over the sea surface at low vessel speed (2-3 knots) for average 20-30 min. Each sampling site consisted of three parallel transects. After trawling, the net was washed from the outside with sea water and the content of cod end was washed off with filtered water (0.45 µm pore size filter) into a clean glass jar with a lid. The jars with suspended matter were transferred to a clean room in the NIVA laboratory for further analysis. Flowmeter was used to calculate the exact filtered water volume, which varied between 79-346 m^3.

Sample collection with pump/Ferrybox system: Microplastics were collected by a filtration of the subsurface seawater using ship-board underway pump-through system with an intake located at a depth of about 1-1.5 m on the left side of the boat. Filtration system consists of first step water appliance protective systems with stainless steel mesh filters (100 µm pore size) and food grade PVC pipes. A flow meter Decast Metronic BKCM-15/Valtec VLF-U integrated into the system provided accurate registration of water volume for each sample, which varied from 0.127 to 0.463 m3 per sample. After every sampling period, stainless steel mesh filter with collected material was removed from the system, placed into clean jar and transferred to the NIVA clean lab for further analysis. The filtration system (filter holder) was rinsed with pre-filtered water between the samples.

Field Blanks

Neuston net.

To control and minimise external contamination, the following steps were performed: Only distilled and filtered water was used to wash the equipment and working surfaces. The neuston net was rinsed from outside with seawater before each sampling. The composition of all plastic materials used during sampling and analysis was identified and taken into account during analysis (material of the net, boat, laboratory equipment).

Pump/Ferrybox

To monitor the potential introduction of contamination during the sampling and analysis procedure, field and procedural blanks were conducted. Specifically, sampling field blanks were performed alongside the subsurface sampling procedure (1 field blanks each sampling day, 4 blanks totally) using the same procedures as for subsurface sampling excluding seawater pumping when filters were in the filtration system.

Laboratory Procedure

Neuston net: No treatment was carried out prior to the analysis. Collected suspended matter was washed off with filtered water from the jars into a plastic basin for primary visual analysis(Lusher et al., 2020) for the presence of microplastics (>500µm) in the sample. After the end of visual sorting, the sample was washed onto a cascade of metal sieves with a mesh of 1 mm and 0.3 mm for final verification. During the analysis, a lamp with an illuminated magnifying glass was used. Potential plastic particles were stored in Eppendorf tubes before analysis in the laboratory.

Pump/Ferrybox: The same procedure as for other Ferrybox samples was used: in the laboratory, suspended matter was washed from the stainless-steel mesh filters and volume was reduced using 90 µm sieves. Organic matter was digested using a protocol with 10% KOH (24hr incubation at 40 degrees). The processed samples were filtered onto 47 mm GF/A papers with 1.6µm pore size. The filter with material was immediately transferred to a petri dish and covered for drying and further analysis.

Laboratory blanks and LOD/LOQ

Neuston net: For control of airborne contamination, a wetted GF/A filter was exposed in an open glass Petri dish close to the sample during processing and analysed for foreign plastic particles after sample processing.

Pump/ferry box samples: In the laboratory, procedural blanks (2 blanks, 10% KOH in glass jars) were run simultaneously with processing of samples. All field and procedural blanks were analysed for microplastics in the same way as the samples using a dissecting microscope Nikon SMZ745 after filtering onto GF/A paper and μ-FTIR.

Blank correction and limitations. Given that no contamination was observed within the target size range we did not perform any corrections of our data, apart from removing particles which were not shown to be plastic after FTIR. Furthermore, fibres were excluded from the analysis due to the challenges of controlling for this contamination during field sampling and limitations of used method.

Recovery test for *Neuston net:* To control the quality of the analysis a recovery test was performed in the laboratory. Standard material of three polymers (polyethylene, polypropylene and polystyrene) were added to the cod ends containing some marine suspended matter. Specifically, 21 particles (0.5 – 4 mm) were added to the samples and processed in the same way as the samples. This was performed in 3 replicates to ensure method validity. All analysis steps were followed according to the field samples, including visual sorting and FTIR measurements. The method validation showed a recovery rate of 100%.

See also Appendix 5.1.

Blue mussels

Sample collection

Mussels were collected in August 2021 and August 2022 (see Table 21). The sampling procedure follows previously published methods (Bråte et al., 2018). The samples were collected for MIKRONOR by the MILKYS and Screening programs, and 10-30 individuals per site were collected from natural substrate (i.e. avoiding those individuals growing on nylon ropes, plastic buoys etc.). Only living individuals (3-6 cm in size) with no visible signs of damage were collected and wrapped in aluminum foil before storage. Individuals were frozen (- 20 °C) whole (in their shell) as soon as possible after collection until sample processing and analyses. Neither atmospheric nor other field blanks were collected, as the blue mussels closed when taken out of the water and frozen as soon as possible.

Table 21. Summary of locations and metadata for mussels collected for the MIKRONOR project in 2021. All data for length and weight are presented as averages.

Station name	Station code	n	Length (mm)	Weight (g, w.w)	Weight (g, d.w)
Akershuskaia, Inner Oslofjord	I301	10	45.23	1.59	0.28
Bekkelaget, Inner Oslofjord	SC3	10	39.06	0.68	0.13
Tjøme, Outer Oslofjord	36A1	10	52.45	1.84	0.30
Gåsøya-Ullerøya, Farsund	15A	10	40.13	0.59	0.07
Nordnes, Bergen harbour	I241	10	61.40	4.58	0.75
Ålesund harbour	28A2	10	57.65	3.86	0.67
Brashavn, Outer Varangerfjord	11X	11	35.03	0.78	0.12
Total		**71**	**47.10**	**1.99**	**0.33**

Table 22. Summary of locations and metadata for mussels collected for the MIKRONOR project in 2022. All data for length and weight are presented as averages.

Station name	Station code	n	Sub-samples n	Length (mm)	Weight (g, w.w)	Weight (g, d.w)
Akershuskaia, Inner Oslofjord	I301	21	3	24.14	1.94	0.31
Tjøme, Outer Oslofjord	36A1	18	3	50.49	30.65	4.76
Odderøya, Kristiansand harbour	I133	30	3	43.42	9.76	1.44
Nordnes, Bergen harbour	I241	18	3	49.08	20.46	4.53
Ålesund harbour	28A2	30	3	53.28	28.71	4.97
Skallneset, Ytre Varangerfjord	10A2	18	3	24.78	1.47	0.22
Total		**135**		**40.87**	**15.50**	**2.71**

Laboratory Procedure

The samples from 2021 showed low numbers of microplastic in each individual (range: 0 – 10 MP/individual). To increase the precision and lower the detection limit in our analyses, it was decided to pool individuals from a station into three replicates for the 2022 analysis. Therefore in 2022, up to 30 individuals were collected per site and pooled into three subsamples of up to 10 individuals. (for number of individuals see table under sampling strategy).

Conforming to previous reporting, each individual was measured for their length (mm) with calipers before rinsing with RO water. Shells were opened and the soft tissue was dissected out, weighed (g, w.w.), and placed in a pre-rinsed, clean glass beaker.

Dry weight

Each blue mussel was measured for length and weighted (w.w.) and pooled into subsamples according to table under sampling strategy. Subsamples were freeze dried minimum overnight (until completely dried) and each were weighted, to get the dry weight per subsample. As the method for 2021 was slightly different samples were not freeze dried before dissolution) the dry weight measurements were slightly different.

Digestion of organic tissue with KOH and further dissolution of CaCO3

The biological material of the mussels needs to be broken down to leave only non-digestible material, including plastics before analyses. KOH is a recommended method commonly used to remove soft tissue of invertebrates or fish organs such as liver or small stomach, without harming any plastic particles present in the matrix. KOH is highly basic, forming strongly alkaline solutions in water and other polar solvents. This method has been recommended and included in international guidelines e.g. GESAMP, (2019) and AMAP, (2021).Accordingly, NIVA has optimised the method for use in monitoring programs for blue mussels (Bråte et al., 2020).

When working with only KOH, it was observed that residues of shells and pearls remained. This was seen to have an impact on the efficiency of visual inspection and µFTIR analysis. Considering that mussel shells are not affected by soft tissue reagents (e.g., KOH), an additional treatment was required. Acetic acid was therefore applied to dissolve the calcium carbonate ($CaCO_3$) that the shells are made of. Acetic acid is a weak acid and will dissolve components consisting of calcium. Digestion of $CaCO_3$ occurs at pH <6 and will be faster digested when exposed to lower pH. 3).

The method applied therefore followed a two-step approach:

First, a pre-filtered solution of 10% KOH was added to each beaker in a ration of 1:10 (biota: KOH, v/v) in order to digest the soft tissues. Beakers were sealed with aluminum foil and placed in an incubator for 48 h at 40 °C with continuous agitation (100 rpm). The samples were thereafter sieved through 300 µm and 50 µm sieve mesh, one on the top of each other. The two fractions (50-300 µm, and >300 µm) were collected into two new 250 ml Erlenmeyer flasks. In some instances, a second KOH step was needed to to remove all the organic tissue. This was performed on the size fractions separately.

Next, a pre-filtered solution of 10% acetic acid was then added to the samples in a 1:1 ratio (10% acetic acid: 10% KOH) for a final concentration of 5% acetic acid, in order to dissolve CaCO3 shell residues, and then placed in an incubator overnight with continuous agitation (100 rpm). The samples were then size fractionated through 250 µm and 50 µm sieves. The larger fraction (>250 µm) was filtered onto glass microfibre filter papers (GF/A, 47 mm diameter, 1.6µm pore size) and allowed to dry in a covered petri dish. Filters were visually inspected for the presence of suspected microplastics, they were confirmed as plastic using by single-point- µFTIR. The smaller fraction (50-250 µm) was filtered onto a silver membrane filters (13 mm in diameter, pore size of 3 µm) for analysis with µFTIR scanning imaging mode followed by pyrolysis- GC/MS.

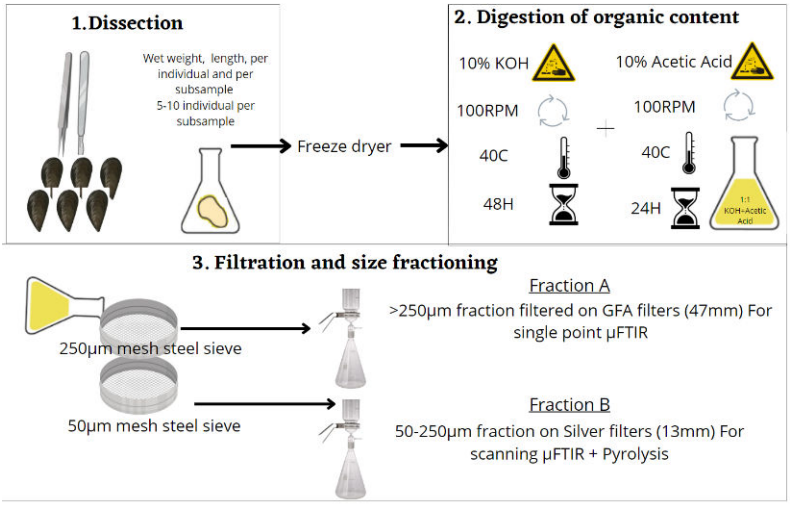


Figure 37. The sample pre-treatment is outlined in the figure

Laboratory blanks and LOD/LOQ

Three laboratory procedural blanks were included per set of samples analysed. Laboratory blanks only contain RO water and added chemicals but were treated in the same way as the actual samples. Any microplastic particles detected in the blanks was characterised and the results were used to calculate LOD and LOQs, and to correct for background contamination.

For results of laboratory blanks and recovery tests, see Appendix 5.1.

Manta Trawl Samples

Sample collection

Manta trawl samples were provided to MIKRONOR through the programme Elveovervåkingsprogrammet. The manta trawl is used to sample the surface layer of water (0-15cm depth). The mechanical flow meter attached to the manta trawl records the filtered water volume. The manta trawl consists of a metal framed opening (0.30m x0.15m) and a catchment net with 300µm mesh size and will therefore collect microplastic particles from 300µm and above. The samples are collected by lowering the manta trawl from a bridge or another height compared to the river. The trawl collects the sample for around 10-30min, depending on the speed of the water in the river.

Field Blanks

To get a better understanding of possible contamination coming from the manta net and trawl, a net blank is collected by lowering the net in the water but stopping it before water enters the opening. The trawl is kept still for 5-10 min before the trawl is lifted and the net-blank is collected. In addition to the net blank, atmospheric field blanks have been collected by opening glass jars with RO-water around the area of where the samples are collected while sampling.

For results from field blanks, see Appendix 5.1

Laboratory Procedure

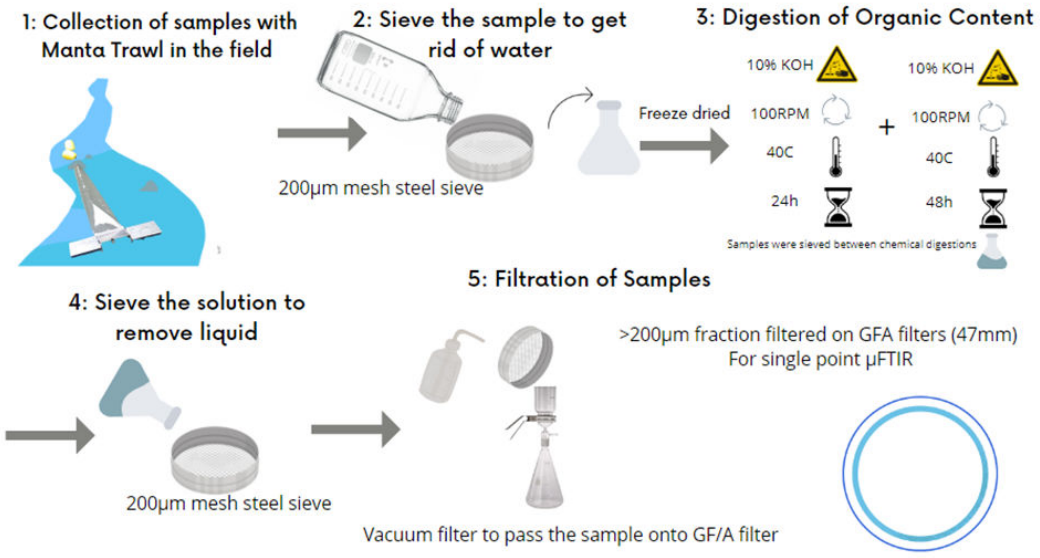


Figure 38. Laboratory procedures

To prepare the samples for microplastic analysis, following procedure was followed:

1. The whole samples were sieved through a 200µm mesh before frozen in -21°C overnight before being freeze dried.
2. When samples were dried 10% KOH was added to reach between 50-100mL.
3. Samples were digested in an incubator for 24 hours at 100 rpm and 40°C. Then samples were sieved through a 200µm mesh.
4. Due to the high amount of biotic material in the samples, the process included two steps of digestion with KOH in an incubator.

After the processing steps, the samples were filtered onto GFA filters for 250µm fraction, and onto silver filters for the 50-250µm fraction using a glass vacuum pump before being analysed. The analysis steps include firstly microscopy to identify the microplastics found in the samples, then followed by characterising the microplastics (type of polymer) using Fourier-transform infrared spectroscopy (FTIR).

Laboratory blanks and LOD/LOQ

To test for laboratory contamination, and to ensure that we don't contaminate our samples in the preparation process, procedural blanks in the lab were carried out. These blanks follow the same steps as the samples and are included each day of the sample processing. One blank is done in the beginning of the process, one during the process and one at the end of the process.

For results from laboratory blanks see Appendix 5.1. For recovery tests, we refer to MIKRONOR report 2022.

Sediments

Sample collection

Marine and lake sediment samples were provided to MIKRONOR through the programs ØKOKYST, Urban Fjord, ØKOSTOR, ØKOFERSK and MILFERSK. The sampling of both coastal and lake sediment was carried out using a grab. The grab was rinsed with water from the sea or the lake, to minimise contamination, before it was lowered into the water. The water on top of the sediment, in the grab, was removed and three pre-rinsed and clean glass jars (200 ml) were filled with the upper 0-2 cm sediment layer using a metal spoon. Three samples, three glass jars with sediment, were collected from each sampling location. The samples were collected from many sample locations that were already established in the other monitoring programs.

Field Blanks

Atmospheric field blanks were set up around the area where the samples were collected. Atmospheric field blanks give an overview of potential contamination airborne microplastic particles during sampling, and to further quality check the microplastic we find in our samples. The atmospheric field blanks were open during the same time the grab was open and the glass jars were filled with sediment.

For results, see Appendix 5.1.

Laboratory Procedure

Marine and lake sediments were expected to have different contents of organic and inorganic material and the sample processing was therefore a little different. The pre-treatment was however the same. They were both freeze dried, homogenised, and split in two where one part (8-10 g dw) where used for pyrolysis GC/MS and the other part was used for microplastic analysis. The amount of sediment used for

microplastic analysis was different for the marine and lake sediment. The amount of the marine sediment was 40-50 g dw while the amount of lake sediment was 10 g dw.

Marine sediment:

The first step, after pre-treatment, was addition of 10% acetic acid to dissolve calcareous ($CaCO_3$) shells, followed by wet sieving to size fraction the sample and treatment with 10% potassium hydroxide (KOH), and density separation to separate the plastic particles from sediment by using the difference in density. At this step, the sample has been split in two size fractions and the big size fraction (>300 µm) was filtered on to a GF/A filter (Ø 47 mm, pore size 1.6 µm) after density separation, but the small size fraction (50-300 µm) needed more treatment before analysis. The small size fraction got a second treatment with 10% KOH and a second density separation before it was filtered on to a silver filter (Ø 13 mm, pore size 3 µm).

Freshwater sediment:

The first step, after pre-treatment, was addition of 10% sodium dodecyl sulphate (SDS), followed by Fenton's reagent (30% H_2O_2 + $FeSO_4$*$7H_2O$), treatment with 10% KOH and wet sieving to size fraction the sample, and density separation to separate the plastic particles from sediment by using the difference in density. The big size fraction (>300 µm) was filtered on to a GF/A filter (Ø 47 mm, pore size 1.6 µm) after density separation, but the small size fraction (50-300 µm) needed more treatment before analysis. The small size fraction got a second treatment with 10% KOH before it was filtered on to a silver filter (Ø 13 mm, pore size 3 µm).

Laboratory blanks and LOD/LOQ

To test for laboratory contamination, and to ensure that we don't contaminate our samples in the preparation process, procedural blanks in the lab were carried out. These blanks follow the same steps as the samples, one blank is included for each batch of sediment samples.

For results from laboratory blanks and LOD/LOQ, as well as for method validation, see Appendix 5.1.

General contamination control

In the field:

Microplastic occur wherever there is plastic are plastic products, which can in turn potentially affect the samples. It is therefore very important to assess possible sources of microplastic contamination from clothing, equipment, air, boat paint etc. during the entire sampling. In addition to this are field blanks, which give an additional control over where potential contamination might be sourced from.

A net blank is taken to ensure that the microplastic found in samples are not of origin in the net or equipment used for sampling. It is important to thoroughly clean the net from the outside before conducting the field blank, as the cleaning procedure will impact the contamination of samples (Michida et al., 2019).

Atmospheric field blanks are used to get an overview of contamination from airborne microplastic particles during sampling, and to further quality check the microplastic we find in our samples. These samples consist of glass jars half filled with RO-water only open during the sampling time. If for example certain types of microplastics are found in the sample, and in the atmospheric blank, it can be concluded that it is from a potential contamination source.

General guidelines to avoid contamination during field sampling

Below are the general guidelines provided for collection of environmental samples for microplastic analysis. These are NIVA's interpretation of the guidelines recommended from GESAMP (2019), Michida et al., (2019), Brander et al., (2020) and AMAP, (2021).

- Avoid synthetic clothing, etc. fleece of polyester or another type of plastic polymers. Preferably wear clothes made from natural fabrics such as cellulose, cotton or wool. If synthetic clothing is used, the type and colour should be filled into the overview of contamination to be able to
- Avoid the use of plastic equipment. All the sampling equipment should be either metal or glass. If plastic equipment is used it needs to be documented, this includes plastic gloves, plastic containers, plastic shovel etc.
- All samples should be handled in the shortest possible time to avoid unnecessary exposure to air.
- Including field blanks. To be able to correct airborne microplastic contamination, as well as contamination from equipment such as nets etc.
- In addition, when sampling mussels: avoid picking mussels from ropes or other areas in close contact with potential plastic sources.
- Make sure all equipment is thoroughly cleaned and flushed with RO-water before sampling.

Analyses and data reporting

Visual Inspection

The microplastic particles of the larger size fractions (250 or 200 µm) were first identified following methods and standards presented in Lusher et al., (2020) using a stereomicroscope (Nikon SMZ745T, 20× magnification). When identified, the particles were photographed using Infinity 1-3C/INFINITY 1 Lumenera camera and INFINITY ANALYZE and CAPTURE software and shape, colour and size (longest, length and shortest, width, µm).

Microplastic Characterization

Fraction >300 µm

To further characterise the particles found, µFTIR (Spotlight 400, PerkinElmer) was used to determine the type of plastic. This was done by transferring the particles onto a diamond compression cell (DCC). By squeezing the particles, the surface becomes thinner and more even, which improves the spectra analysis. The analysis was performed in transmission mode with a resolution of 4 cm-1 and wavelength 4000 to 600 cm-1. Background scanning was performed before each analysis. The instrument is used together with the Spectrum 10 software (v. 10.6.2), and each spectrum is compared to several different libraries: PerkinElmer ATR Polymers library, STJapan Polymers ATR library, BASEMAN library (Primpke et al., 2018), and in-house libraries including reference material, various textiles, and potential sources of contamination from the lab. All spectra were manually inspected to ensure that each library match is acceptable. Particles were accepted as plastics if they fell into the categories as assigned by AMAP.

Fraction 50-200 µm

To be able to analyse the smaller sized particles, the fractions from 50-200 µm were analysed by using the automatic scanning mode on the silver membrane filters by using the µFTIR imaging reflectance mode (Spotlight 400, PerkinElmer). This method is more sensitive in that it is possible to analyse smaller sized particles, and human bias linked to subjectivity in the visual preselection step is avoided. The method also limits particle loss linked to the physical handling of particles.

In short, µFTIR imaging involves analysing spectra for each defined pixel within a defined area, and building a chemical map of the entire sample, i.e., all identified particles. Automatic scanning of particles >50 µm was done using a Perkin Elmer Spotlight 400. The resulting FTIR spectra were further analysed using Purency Microplastic Finder (Version 4.17) where after loading the data and reference spectra, the spectral were fit a Pearson correlation for the untreated data, the first derivative and the second derivative, resulting in several correlation factors. Purency Microplastic Finder identifies the recorded spectra based on the results of the Pearson correlation factors. In short, the sample is collected on a silver membrane filter, scanned using the µFTIR with a heat map generated during the identification process, and final identification completed after applying a threshold value.

Air samples (NILU)

To document the long-range transport of MP, the monitoring stations/observatories in this report are located, as far as possible, in areas that are not influenced by local sources for the regulated and long-term monitored contaminants. However, for microplastic and related chemicals such as the UV 328, it is important to continuously evaluate possible influences of local sources in comparison to long-range transport, since these particles can be emitted by the personal handling the samples and the surfaces of the surrounding facilities. Consequently, measures to limit the impact of contamination have been put in place and continuously monitored.

The two observatories, Zeppelin and Birkenes, were used to represent different parts of Norway, and areas that receive air from different source regions globally. The two observatories used for the monitoring of MPs and additives in air; Birkenes in southern Norway, and Zeppelin, located on Svalbard, an archipelago in the Arctic Ocean (Figure 1, Table 23). Further information of the sampling sites is available at: http://www.nilu.no/projects/ccc/sitedescriptions/. The observatories included in this monitoring programme are, in most cases, coordinated and correspond to those within "*the national measurement programme of long-range transported air pollutants for main components in air and precipitation*", coordinated by NILU on behalf of the NEA, and the Ministry of Climate and Environment (Aas et al., 2019).

Sample collection

Two sampling strategies were used: i) active sampling for suspended atmospheric MP and ii) deposition sampling for wet and dry deposition of MP.

Table 23. Information about the monitoring stations and list of measured contaminants at each station in 2022. **Six samples per campaign, 14 days of sampling.

Monitoring station	Birkenes**	Zeppelin**
Station code (EBAS)	NO0001R, NO0002R	NO0042G
Lat	58 23 N	78 54 N
Long	8 15 N	11 53 E
m.a.s.l.	190/219	475
Sample matrix	Air & Precipitation	Air & Precipitation
Sampling periods for MP in Deposition	14.09.-28.09. 28.09.-12.10. 12.10.-26.10. 26.10-09.11 09.11-23.11 23.11-07.12	15.09.-29.09. 29.09.-13.10. 13.10.-27.10. 27.10.-10.11. 10.11.-24.11. 24.11.-08.12.
Sampling periods for suspended MP in Air	03.08.-17.08. 17.08.-31.08. 31.08.-14.09. 14.09.-28.09. 28.09.-12.20. 12.10.-26.10.	01.09.-15.09. 15.09.-29.09. 29.09.-13.10. 13.10.-27.10. 10.11.-24.11 24.11.-08.12.

Sampling

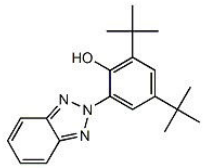
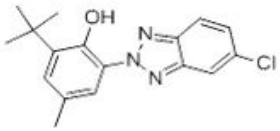
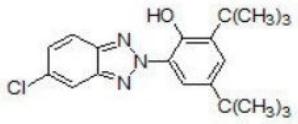
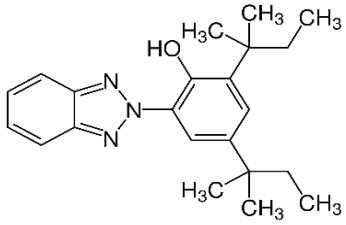
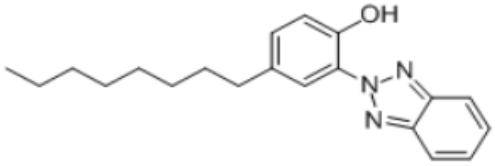
I) Suspended atmospheric MP and related additives were collected using active air samplers at both sites. The active air samples were collected on a bi-weekly basis throughout the fall using full-metal filter holders, equipped with 10 µm steel filters (Table 23). They collect TSP (total suspended particles) down to 10 µm size with a sampling rate of 3 m^3/hour MP.

II) The deposition samples are collected on bi-weekly basis using full metal bulk precipitation samplers (Innovation Nilu's Atmospheric Microplastic Collector) with no MP size limitation, for a period of 14 days per period at both stations.

Active air samples and precipitation samples were extracted, analysed and quantified at NILU under strict quality control (Clean room) using the isotopic dilution method and the specific method details can be found in Goßmann et al., (2023).

Figure 39. Illustration of deposition sampler at Zeppelin station, photo by Dorte Herzke.

Methods to analyse Microplastic and UV compounds
In this study we measured the following additives:

UV 320		2-benzotriazol-2-yl-4,6-di-tert-butylphenol
UV 326		2-(2′-Hydroxy-3′-tert-butyl-5′-methylphenyl)-5-chlorobenzortriazole
UV 327		2-(2′-Hydroxy-3′,5′-di-tert-butylphenyl)-5-chlorobenzotriazole
UV 328		2-(2*H*-1,2,3-Benzotriazol-2-yl)-4,6-bis(2-methylbutan-2-yl)phenol
UV 329		2-(2′-hydroxy-5′-(1,1,3,3-tetramethylbutyl)phenyl)benzotriazole

Sample preparation consisted of extraction of resuspended MPs in ethanol (prefiltered), followed by up-concentration and GC/OrbitrapMS analyses. The isotopic dilution method was applied. Field- and method blanks were applied throughout the project. All laboratory work was carried out either in a Clean Room or in a laminar flow cabinet. All used equipment was plastic-free and thoroughly cleaned prior use. All concentrations are blank corrected by the Average + 2x STDEV of the field blanks belonging to the sample batch processed.

Atmospheric microplastic particles
We measured 9 polymer types within this study, see Table 24:

Table 24. Polymer types in air samples

- Poly (methyl 2-methylpropenoate) : PMMA
- Polypropylene : PP
- Polyvinylchloride : PVC
- Polyamide/ Nylon : Nylon
- Polyurethane : PU
- Polystyrene : PS
- Polyethylene : PE
- Polyethylene terephthalate : PET
- Polycarbonate : PC

Sample preparation for deposition samples consisted of filtering on a 10 µm steel filter, followed by resuspension in ethanol and a filter change onto a GF/F filter. Internal standard was added and the sample was analysed on a Frontier pyrolysis/Thermo GC/MS/MS. Field- and method blanks were applied throughout the project. All laboratory work was carried out either in a Clean Room or in a laminar flow cabinet. All used equipment was plastic-free and thoroughly cleaned prior use.

The Norwegian Institute for Water Research

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