Nordic Council of Ministers

Screening of compounds in tire wear road run off

Contents

Preface 4

Summary 5

1 Introduction 6

2 Methods 7

2.1 Screening strategy 7

2.2 Sampling methods 9

2.3 Sample preparation & chemical analysis 15

2.3.1 Water samples 15

2.3.2 Sediment samples 15

2.3.3 Biota samples 15

2.3.4 LC/MS analysis 15

2.3.5 Quality assurance 18

3 Results 19

3.1 Water samples 21

3.2 Sediment samples 27

3.3 Biota samples 28

4 Discussion 29

5 Acknowledgements 31

References 32

Appendix A - Results summary table 34

Appendix B - Sampling manual for road run-off 43

General sampling strategy 43
Appendix C - List of substances 46
About this publication 49

This publication is also available online in a web-accessible version at:
https://pub.norden.org/temanord2024-525

Preface

The climate and environmental research institute NILU, acting on behalf of the Joint Nordic screening group, organized the sampling and carried out subsequent analysis for tire related additive chemicals in the "Screening of tire wear road run off" project. The project was initiated in 2022, with field work and chemical analysis carried out in 2023. Linda Hanssen was the project leader, whilst the steering group was comprised of the members of the Joint Nordic screening group: Eivind Farmen, Linda Linderholm, Maria Linderoth, Iben Boutrup Kongsfelt, Bård Nordbø, Katrin Hoydal, Emmi Vähä and Bergdís Björk Bæringsdóttir. The sampling efforts were a collaborative effort, where each partner country provided relevant samples to NILU for analysis. Sample preparation was conducted by Gabriele Piattoli, Ådne Hotvedt and Linda Hanssen. Method development and sample analysis was performed by Vladimir Nikiforov, Linda Hanssen and Natascha Schmidt. This report has been collaboratively written by Linda Hanssen, Natascha Schmidt and Vladimir Nikiforov.

(Tromsø, 10.12.2023)
Linda Hanssen
Project Leader
NILU

Summary

Road run-off and associated snow, water recipient and sediment/soil samples were collected in six Nordic countries in spring/summer 2023 and analysed for the presence of 15 tire related chemicals including antioxidants/antiozonants, transformation products and a crosslinking agent. Additionally, 20 blue mussel samples were analysed, summing up to a total of 87 samples. Tire related chemicals were found in 98% of samples, with one compound (TPPD; N-Phenyl-N'-tolyl-p-phenylenediamine) being predominant in all sample matrices. Direct road run-off samples exhibited highest concentrations, while recipient waters were generally less contaminated. A high variability in chemical concentrations and relative abundance of individual compounds was however observed depending on sample type and location. This variability might be driven by the use of different additives in tire formulations, environmental factors (UV irradiation, rainfall) or population density, car use and traffic conditions.

The Nordic countries might be particularly prone to contamination by tire wear and associated chemicals due to a prolonged period in which softer winter tires are used, the use of studded tires and potentially bad road conditions due to harsh climatic conditions. However, while the amount of scientific literature on tire related chemicals is generally increasing, data on the environmental occurrence of TPPD is currently lacking.

The aim of this study was to provide a first assessment of the presence of tire related chemicals in road run-off and associated samples in the Nordic countries. Future studies might monitor the occurrence of these compounds in locations identified as potential hotspots or increase the spatial and/or temporal sampling coverage.

1 Introduction

Tire and road wear particles (TRWP) are created through the friction between tires and the road surface. These particles are considered a major source of microplastics (MPs) to the environment. For example, an annual release of 19,000 tonnes of MPs from land-based sources is estimated for Norway, 40% of which are considered to be TRWP (Sundt et al., 2021). While the application of speed limits and an optimal tire pressure can reduce the production of TRWP, the use of studded tires and a heavy car weight can increase the tire wear (Rødland et al., 2022).

As for other plastic polymers, additive chemicals are used during the production of tires and can leach out and enter the environment. On average, over 400 organic chemicals are identifiable in a tire (EmissionsAnalytics, 2023). Examples of such chemicals include the antioxidant and antiozonant 6PPD (N-(1,3-dimethylbutyl)-N′-phenyl-p-phenylenediamine), one of its transformation products 6PPD-Q (6PPD-Quinone) and the crosslinking agent HMMM (Hexa(methoxymethyl)melamin). Naturally, concentrations of these chemicals are particularly high in compartments strongly influenced by road traffic, such as road run-off and recipient waters. The presence of tire related additive chemicals has been reported in urban streams (Johannessen et al., 2022), wastewater treatment plant effluents (Seiwert et al., 2020), road run-off (Challis et al., 2021) and marine sediments (Zeng et al., 2023), among others. Recently, 6PPD and 6PPD-Q have also been detected in three fish species, indicating their occurrence in wildlife for the first time (Ji et al., 2022).

Cold-climate regions have been identified as being particularly prone to contamination by tire related additive chemicals due to several factors: the common use of softer winter tires and the use of studded tires, both increasing the production of TRWP, bad road conditions due to the harsh climatic conditions and the use of road salts (Challis et al., 2021). Furthermore, it has been shown that the presence of road salts can enhance the toxic effects of 6PPD, as is the case for the rotifer *Brachionus calyciflorus* (Klauschies & Isanta-Navarro, 2022). Therefore, screening for tire related additive chemicals in these regions will offer valuable information on the current state and potential actions that might need to be taken to reduce the risks for the environment. The Nordic countries have stated their willingness to actively investigate and manage this challenge, e.g. by assessing the different possibilities to reduce the release of MPs from tire wear (Furuseth & Rødland, 2020) and by initiating the study that is presented here.

2 Methods

2.1 Screening strategy

Samples originated from six Nordic countries, namely Iceland, the Faroe Islands, Norway, Denmark, Sweden and Finland (Figure 1). The locations of the individual sampling stations are indicated in Figure 2. To screen for tire related additive chemicals in a variety of sample types and environmental contexts, the sampling locations chosen in this study include urban areas with high and low road traffic and background sites. Sites, where specific measures are taken to stock or treat road run-off or snow from roads, were also included. These comprise snow dumping sites, a snow treatment facility in Oslo (Norway) and a stormwater treatment system in Finland. Some examples of sampling stations are shown in Figure 3.

Whenever available, information concerning rainfall or the application of road salts during the sampling period was collected.

Due to challenges, such as a lack of rain during long periods in spring in the Nordic countries, not all planned samples could be collected, and some sample bottles broke during shipment. To compensate, five samples of atmospheric deposition and 20 blue mussel samples were included for analysis.

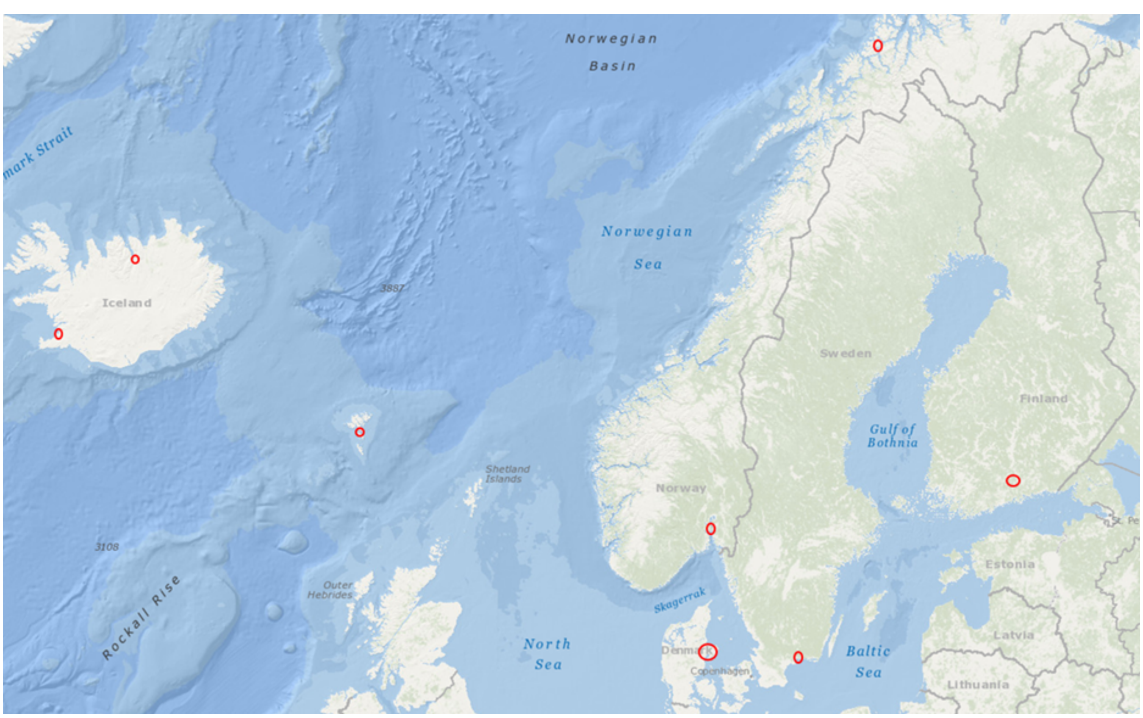
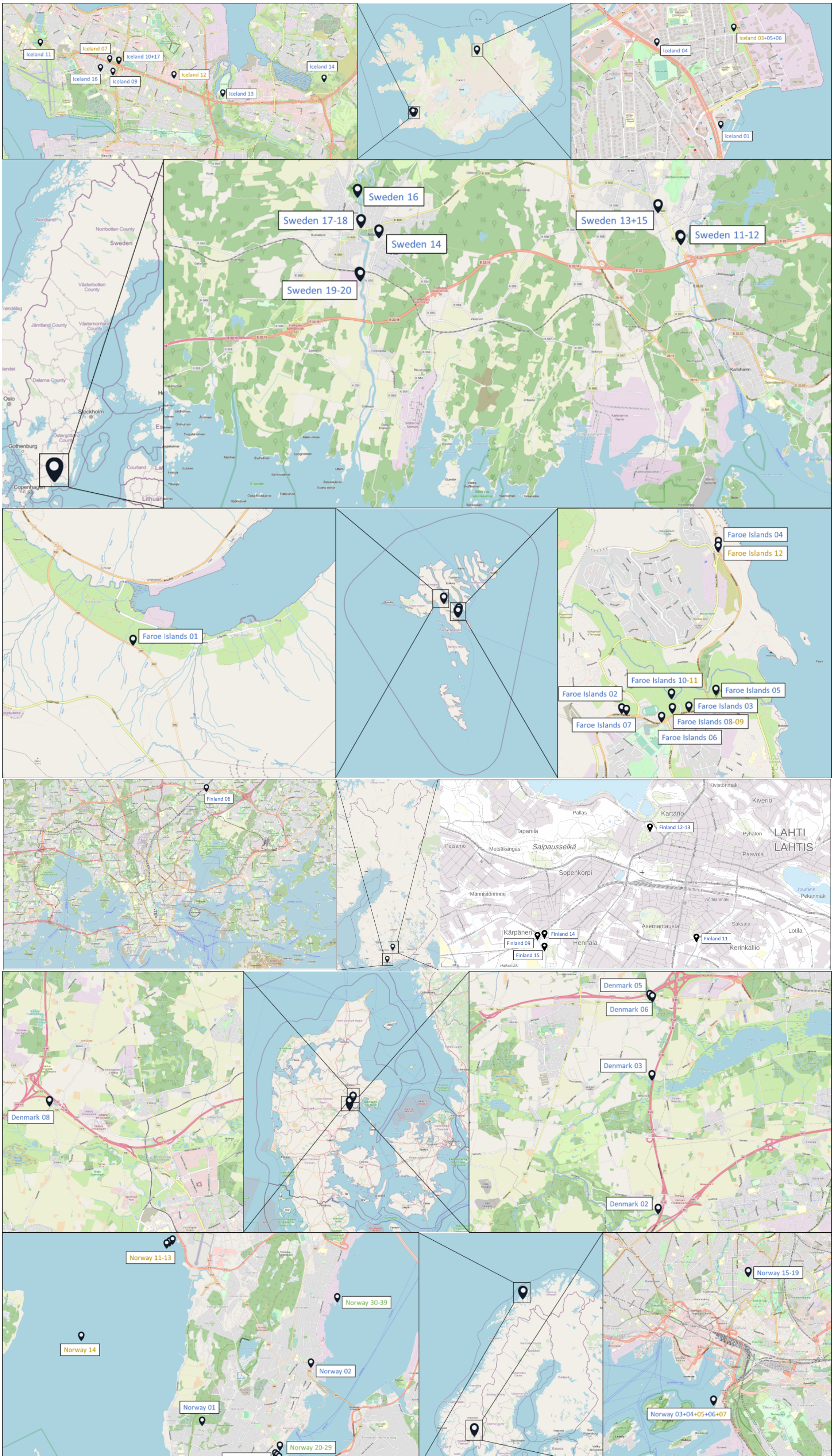


Figure 1: Sampling locations in Iceland, the Faroe Islands, Norway, Denmark, Sweden and Finland. Map source: NOAA.



Iceland 11
Iceland 07
Iceland 10+17
Iceland 16
Iceland 09
Iceland 12
Iceland 13
Iceland 14
Iceland 04
Iceland 03+05+06
Iceland 01
Sweden 16
Sweden 17-18
Sweden 14
Sweden 13+15
Sweden 11-12
Sweden 19-20
Faroe Islands 01
Faroe Islands 04
Faroe Islands 12
Faroe Islands 10-11
Faroe Islands 02
Faroe Islands 05
Faroe Islands 03
Faroe Islands 07
Faroe Islands 08-09
Faroe Islands 06
Finland 06
Finland 12-13
LAHTI
LAHTIS
Finland 14
Finland 09
Finland 15
Finland 11
Denmark 08
Denmark 05
Denmark 06
Denmark 03
Denmark 02
Norway 11-13
Norway 30-39
Norway 14
Norway 02
Norway 01
Norway 20-29
Norway 08-10
Norway 15-19
Norway 03+04+05+06+07

Figure 2 (previous page)**:** Location of the sampling stations in (from top to bottom) Iceland, Sweden, the Faroe Islands, Finland, Denmark and Norway. The font colour of the sample IDs indicates the sample types: water [including snow and precipitation] (blue), sediment (brown) or biota (green).
Map source: NOAA.

Figure 3: Examples of sampling location. Top left: Iceland 10, top right: Iceland 17, bottom left: Norway 02, bottom right: Norway 02 sample in glass bottle. © Photos top left and top right: Bergdís Björk Bæringsdóttir. © Photos bottom left and bottom right: Natascha Schmidt.

2.2 Sampling methods

Samples were collected from late winter to early summer 2023. Road run-off was collected during or following rain events. All samples were collected in 500 mL or 1 L pre-burnt (450°C, 8 h) glass bottles covered with aluminum foil. If possible, samples were sent frozen to the NILU laboratory and stored in the freezer upon arrival. In total, 87 samples were analyzed. Sample details are listed in Table 1.

Table 1: Details on sample ID, sample type, location, date, rainfall within the past 24 hours before sampling (when relevant) and additional information.

Sample ID	Sample type	Location	Date	Rainfall (last 24 h)	Details
Iceland 01	Water	65.682726, -18.087351	30.06.2023	0.2 mm	Road run-off recipient (ocean)
Iceland 03	Sediment	65.688057, -18.085776	04.04.2023	0 mm	Soil from snow dumping site
Iceland 04	Water	65.687221, -18.095865	30.06.2023	0.2 mm	Road run-off water
Iceland 05	Snow	65.688057, -18.085776	04.04.2023	0 mm	Snow dumping site
Iceland 06	Snow	65.688057, -18.085776	04.04.2023	0 mm	Snow dumping site
Iceland 07	Sediment	64.133775, -21.903341	19.05.2023	2 mm	Soil close to road
Iceland 09	Water	64.130688, -21.901615	12.05.2023	9.9 mm	Road run-off
Iceland 10	Water	64.133450, -21.898427	19.05.2023	2 mm	Road with heavy traffic
Iceland 11	Water	64.137682, -21.941952	12.05.2023	9.9 mm	Road run-off recipient, stream
Iceland 12	Sediment	64.129918, -21.867733	12.05.2023	9.9 mm	Soil from big parking lot
Iceland 13	Water	64.125484, -21.840630	12.05.2023	9.9 mm	Road run-off recipient, retention pond
Iceland 14	Water	64.129029, -21.784306	12.05.2023	9.9 mm	Road run-off recipient, stream
Iceland 16	Snow	64.131593, -21.908594	27.04.2023	0.5 mm	Snow/melting snow (little traffic)
Iceland 17	Snow	64.133388, -21.898252	27.04.2023	0.5 mm	Snow/melting snow (heavy traffic)
Sweden 11	Water	56.1911647, 14.8451090	19.06.2023	20 mm	Stormwater tube
Sweden 12	Water	56.1911749, 14.8450171	19.06.2023	20 mm	Recipient
Sweden 13	Water	56.1964898, 14.8383000	19.06.2023	20 mm	Stormwater tube

Sweden 14	Water	56.1921212, 14.7545221	19.06.2023	20 mm	Stormwater tube/ very small stream
Sweden 15	Water	56.1964898, 14.8383000	19.06.2023	20 mm	Stormwater well
Sweden 16	Water	56.1989597, 14.7478497	19.06.2023	20 mm	Recipient
Sweden 17	Water	56.1938351, 14.7491084	19.06.2023	20 mm	Stormwater tube
Sweden 18	Water	56.1938552, 14.7489291	19.06.2023	20 mm	Recipient
Sweden 19	Water	56.1849898, 14.7484205	19.06.2023	20 mm	Stormwater tube
Sweden 20	Water	56.1849851, 14.7486089	19.06.2023	20 mm	Recipient
Faroe Islands 01	Snow	62.095833, -6.944167	14.03.2023	1.4 mm	Snow (side of the road)
Faroe Islands 02	Snow	62.021744, -6.785019	16.03.2023	7.2 mm	Snow (side of the road)
Faroe Islands 03	Water	62.021908, -6.771858	23.03.2023	12 mm	Road run-off water
Faroe Islands 04	Snow	62.036944, -6.766111	14.03.2023	1.4 mm	Snow
Faroe Islands 05	Water	62.023364, -6.766742	23.03.2023	12 mm	Road run-off water
Faroe Islands 06	Water	62.020944, -6.777294	23.03.2023	12 mm	Road run-off water
Faroe Islands 07	Water	62.021617, -6.783922	23.03.2023	12 mm	Road run-off water
Faroe Islands 08	Water	62.021731, -6.775153	23.03.2023	12 mm	Recipient
Faroe Islands 09	Sediment	62.021731, -6.775153	23.03.2023	12 mm	Recipient
Faroe Islands 10	Water	62.022981, -6.775047	23.03.2023	12 mm	Recipient
Faroe Islands 11	Sediment	62.022981, -6.767547	23.03.2023	12 mm	Recipient
Faroe Islands 12	Sediment	62.036617, -6.766206	11.04.2023	8.4 mm	Soil from side of the road

Finland 06	Water	60.240463, 24.997300	18.04.2023	0 mm	Stream
Finland 09	Water	60.967123, 25.110517	06.04.2023	0 mm	Stormwater after filtration pond
Finland 11	Water	60.965572, 25.660345	06.04.2023	0 mm	Urban stream
Finland 12	Water	60.985325, 25.646433	06.04.2023	0 mm	Storm/melt water
Finland 13	Water	60.985325, 25.646433	06.04.2023	0 mm	Storm/melt water
Finland 14	Water	60.967934, 25.610367	06.04.2023	0 mm	Storm/melt water
Finland 15	Water	60.965782, 23.445733	06.04.2023	0 mm	Stormwater after filtration ponds
Denmark 02	Water	56.104740, 10.037850	10.07.2023	8 mm	Recipient
Denmark 03	Water	56.134560, 10.035190	10.07.2023	11 mm	Road run-off
Denmark 05	Water	56.152270, 10.034550	10.07.2023	14 mm	Road run-off
Denmark 06	Water	56.151970, 10.035350	10.07.2023	14 mm	Recipient
Denmark 08	Water	56.22020, 10.126170	10.07.2023	15 mm	Recipient
Norway 01	Snow	69.647226, 18.914239	14.04.2023	0 mm	Residential area, background site
Norway 02	Snow	69.656328, 18.963571	14.04.2023	0 mm	Road with heavy traffic
Norway 03	Water	59.896573, 10.749784	10.02.2023	-	Outlet of snow treatment facility
Norway 04	Water	59.896573, 10.749784	10.02.2023	-	Last sedimentation basin before filtration step, snow treatment facility
Norway 05	Sediment	59.896573, 10.749784	10.02.2023	-	First sedimentation basin, snow treatment facility
Norway 06	Water	59.896573, 10.749784	10.02.2023	-	First sedimentation basin, snow treatment facility

Norway 07	Sediment	59.896573, 10.749784	10.02.2023	-	Sludge from membrane filter, snow treatment facility
Norway 08	Sediment	69.641946, 18.947395	06.03.2023	-	Transect 1 snow dumping site (marine)
Norway 09	Sediment	69.641627, 18.947928	06.03.2023	-	Transect 1 snow dumping site (marine)
Norway 10	Sediment	69.641102, 18.948792	06.03.2023	-	Transect 1 snow dumping site (marine)
Norway 11	Sediment	69.675692, 18.901104	06.03.2023	-	Transect 2 snow dumping site (marine)
Norway 12	Sediment	69.675371, 18.899716	06.03.2023	-	Transect 2 snow dumping site (marine)
Norway 13	Sediment	69.675051, 18.898106	06.03.2023	-	Transect 2 snow dumping site (marine)
Norway 14	Sediment	69.662416, 18.864808	06.03.2023	-	Reference/background site (marine)
Norway 15	Water	59.923048, 10.765784	16.08.2023–30.08.2023	208 mm*	Atmospheric deposition (dry & wet)
Norway 16	Water	59.923048, 10.765784	30.08.2023–14.09.2023	3.3 mm*	Atmospheric deposition (dry & wet)
Norway 17	Water	59.923048, 10.765784	14.09.2023–27.09.2023	89 mm*	Atmospheric deposition (dry & wet)
Norway 18	Water	59.923048, 10.765784	27.09.2023–11.10.2023	17 mm*	Atmospheric deposition (dry & wet)
Norway 19	Water	59.923048, 10.765784	11.10.2023–25.10.2023	5 mm*	Atmospheric deposition (dry & wet)
Norway 20	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 21	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 22	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 23	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel

Norway 24	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 25	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 26	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 27	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 28	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 29	Biota	69.643241, 18.949529	13.06.2023	-	Blue mussel
Norway 30	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 31	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 32	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 33	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 34	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 35	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 36	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 37	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 38	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel
Norway 39	Biota	69.666653, 18.975562	12.06.2023	-	Blue mussel

2.3 Sample preparation & chemical analysis

2.3.1 Water samples

Water (n = 43) and snow (n = 9) samples were left to de-freeze at room temperature and filtered through pre-burnt (450°C, 8 h) GF/F filters (pore size 0.7 µm) using a glass filtration unit. A sub-sample of 300 mL or less was taken, dependent on the sample amount available. Internal standards (50 µL, 0.4 ng/µL 6PPD-D_5 & 6PPD-Q-D_5) were added and the samples were left to equilibrate for 30 minutes. Extraction was performed in batches using Solid Phase Extraction (SPE). The samples were passed through pre-conditioned SPE cartridges (HLB Oasis, 200 mg sorbent, Waters®) using PTFE liners. The cartridges were left to vacuum-dry for 1–2 hours. Then, the target chemicals were eluted with 9 mL of methanol in 3x3 mL aliquots. Finally, the extracts were concentrated to 1 mL using nitrogen and a 50 µL aliquot was taken out for analysis and mixed with 25 µL buffer.

2.3.2 Sediment samples

Sediment samples (n = 15) were transferred to glass petri dishes and dried at 50°C for 48 h. If necessary, larger stones were removed through sieving (2 mm mesh size). Sub-samples of 200 mg were weighed out and spiked with internal standards (50 µL, 0.4 ng/µL, 6PPD-D_5 & 6PPD-Q-D_5). After adding 1 mL of acetonitrile, the sample was vortexed and extracted through ultrasonication for 15 minutes. After centrifugation, the extract was collected and the extraction was repeated with 1 mL of acetonitrile. Next, the extracts were combined, dried to dryness and redissolved in 100 µL of acetonitrile. Finally, the sample was filtered through press filters before taking out 50 µL sample for analysis and mixed with 25 µL buffer.

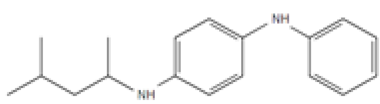
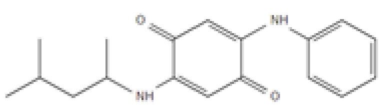
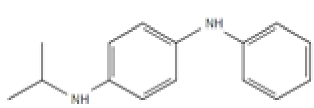
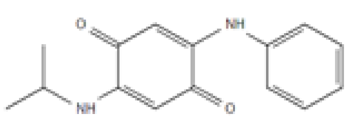
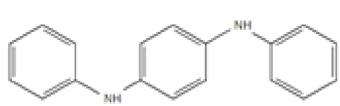
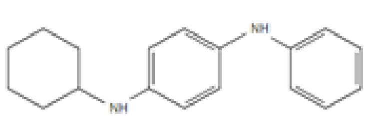
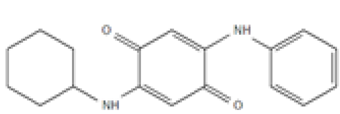
2.3.3 Biota samples

Blue mussel (*Mytilus edulis*; 1 g each) samples (n = 20) were homogenized, internal standards (50 µL, 0.4 ng/µL, 6PPD-D_5 & 6PPD-Q-D_5), ceramic beads and 0.5 g of Na_2SO_4 were added. Next, 1.5 mL of acetonitrile were added, the samples were vortexed and extracted through ultrasonication for 15 minutes. The vortex and ultrasonication steps were repeated once. The samples were further placed on a horizontal shaker for 25 minutes and then centrifuged for 10 minutes. The acetonitrile was transferred into a glass vial and a 50 µL aliquot was taken out for analysis and mixed with 25 µL buffer.

2.3.4 LC/MS analysis

The samples were analyzed by ultrahigh pressure liquid chromatography triple–quadrupole mass-spectrometry (UHPLC-MS/MS). Analysis was performed on a Thermo Scientific quaternary Accela 1250 pump (Thermo Fisher Scientific Inc., Waltham, MA, USA) with a PAL Sample Manager (Thermo Fisher Scientific Inc., Waltham, MA, USA) coupled to a Thermo Scientific Vantage MS/MS (Vantage TSQ) (Thermo Fisher Scientific Inc., Waltham, MA, USA); 10 µL were injected on a Waters Acquity UPLC HSS 3 T column (2.1×100 mm, 1,8 µm) (Waters Corporation, Milford, MA, USA) equipped with a Waters Van guard HSS T3 guard column (2.1×5 mm, 1.8 µm) (Waters Corporation, Milford, MA, USA). Separation was achieved using 2 mM NH_4OAc in 90:10 methanol/water (A) and 2 mM NH_4OAc in methanol (B) as the mobile phases. Ionization was conducted in the positive electrospray ionization mode (ESI+). The results were not blank corrected. LODs were calculated based on signals in the blank samples.

Samples were screened for the presence of the antioxidants/antiozonants 6PPD, IPPD, DPPD, CPPD, TPPD, 44PD, 77PD, DNPD and DTPD, the transformation products 6PPD-Q, IPPD-Q and CPPD-Q, the crosslinking agent HMMM, Solvent red 52, 4-amino-*N,N*-diethylaniline. Chemicals structures and full names of the target analytes are given in Figure 4.

	**6PPD**: N-(1,3-dimethylbutyl)-N′-phenyl-p-phenylenediamine
	**6PPD-Q**: 6PPD-Quinone
	**IPPD**: N-Isopropyl-N′-phenyl-1,4-phenylenediamine
	**IPPD-Q**: IPPD-Quinone
	**DPPD**: N,N′-Diphenyl-p-phenylendiamin
	**CPPD**: N-Cyclohexyl-N′-phenyl-p-phenylenediamine
	**CPPD-Q**: CPPD-Quinone

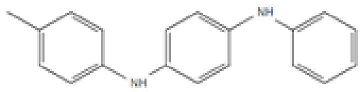
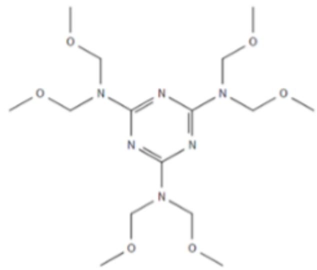
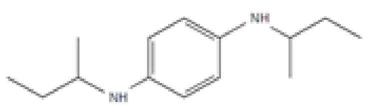
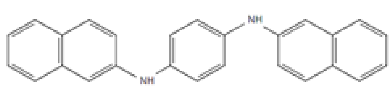
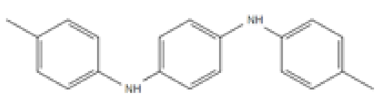
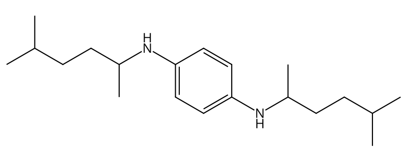
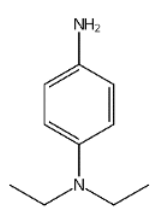
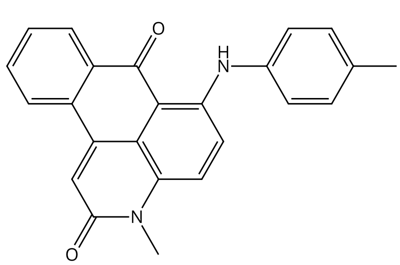
	*** TPPD:** N-Phenyl-N′-tolyl-p-phenylenediamine
	**HMMM**: Hexa(methoxymethyl) melamin
	**44PD:** N,N′-Di-2-butyl-1,4-phenylenediamine
	**DNPD:** N,N′-Di-2-naphthyl-p-phenylenediamine
	*** DTPD:** N,N′-Di-(p-tolyl)-p-phenylenediamine
	**77PD:** N,N′-Bis(1,4-dimethylpentyl)-p-phenylenediamine
	**Solvent red 52**
	**Et2N-PD:** N,N-Diethyl-p-phenylenediamine

Figure 4: Chemical structure, acronym and full name of the chemicals analysed in this study.

** Provisional structures for TPPD and DTPD. TPPD and DTPD were quantified using a commercial antioxidant mixture (Vulcanox®).*

2.3.5 Quality assurance

A laboratory blank was processed with every 10th sample. There are no standard methods for these compounds. For water and sediment samples, we adapted methods that were published in peer-review journals (Rauert et al., 2022; Zeng et al., 2023). For biota samples we used an extraction method previously developed in-house (Galtung, 2023).

We spiked all three sample matrices with a mix of the targeted compounds to verify the extraction method.

2.3.5.1 Sample conservation and storage

The target substances include compounds which can be readily transformed by UV irradiation, which is why special care needs to be taken during sample handling and storage. Rubber materials can be alternative sources for some of these compounds and should therefore be avoided. Studies have shown that the 6-PPD half-life is about 8 h at environmental pH, because both hydrolysis and oxidation are occurring (ECHA) increasing the HDPA and 6-PPDQ concentrations in the sample. To reduce transformation effects, it is recommended to freeze the sample until analysis.

Guidelines concerning the sampling, sample handling and shipment of samples for analysis of substances found in relation to tire wear particles (TWP) were developed, summarized in a sampling manual (see Appendix B) and sent to the partner countries. These guidelines aim to ensure easy, fast and reliable sampling of road run-off, snow, sediments and soils.

3 Results

Tire related additive chemicals were found in all samples analysed, except for two sediment samples from the Faroe Islands. TPPD was the predominant compound in all sample matrices, while CPPD, 77PD, Solvent red 52 and Et2N-PD were not detected in any sample (see Table 2 detection frequencies). All results are listed in Table A1 in Appendix A.

Table 2: Detection Frequencies (%) of tire related chemicals in environmental samples. Only concentrations > 1 ng/L or 1ng/g were included as detected.

Function	Compound/ Matrix	Water				Sediments/Soil		Biota
		Road run-off/ stormwater *n=20*	Recipient *n=15*	Snow *n=9*	Other* *n=8*	Snow dumping site/Recipient *n=12*	Urban *n=3*	Blue mussels *n=20*
Antioxidants/ Antiozonants	6PPD	40	20	11	75	58	0	10
	IPPD	0	0	0	0	8	0	0
	77PD	0	0	0	0	0	0	0
	DPPD	35	7	44	63	50	67	0
	CPPD	0	0	0	0	0	0	0
	TPPD	60	73	44	100	83	67	100
	44PD	5	7	0	0	0	0	0
	DNPD	5	0	0	0	0	0	0
	DTPD	40	13	33	50	17	67	0
	Et2N-PD	0	0	0	0	0	0	0
Transformation products	6PPD-Q	80	67	89	63	33	67	0
	IPPD-Q	15	13	33	0	0	0	10
	CPPD-Q	15	0	11	0	8	0	95
Crosslinking agent	HMMM	100	100	89	88	0	0	5
Dye	Solvent red 52	0	0	0	0	0	0	0

**water from precipitation (n=5) or from sedimentation basins from a snow treatment facility (n=3).*

3.1 Water samples

TPPD was generally the predominant compound in water samples, with a maximum concentration of 449,023 ng/L detected in a road run-off sample. For easier interpretation of the results, the data is being presented separately for each country (Figures 5–10). Hereby, the chemicals which were the most abundant in the samples from each country were selected for the graphical representation.

In Iceland sample number 10, collected at a road with heavy traffic, exhibited highest TPPD (18,325 ng/L), HMMM (11,548 ng/L), 6PPD (109 ng/L) and 6PPD-Q (354 ng/L) concentrations (Figure 5). Sample Iceland 17 was collected at the same location, but consisted of snow/melting snow and was collected approximately 3 weeks earlier, which could explain the still high (TPPD 7,610 ng/L; HMMM 2,769 ng/L; 6PPD n.d.; 6PPD-Q 83.4 ng/L), but significantly lower concentrations compared to sample Iceland 10. Samples collected in Akureyri (Iceland 01, 04-06; right map in Figure 5) generally exhibited lower additive concentrations compared to samples collected in Reykjavik (left map in Figure 5).

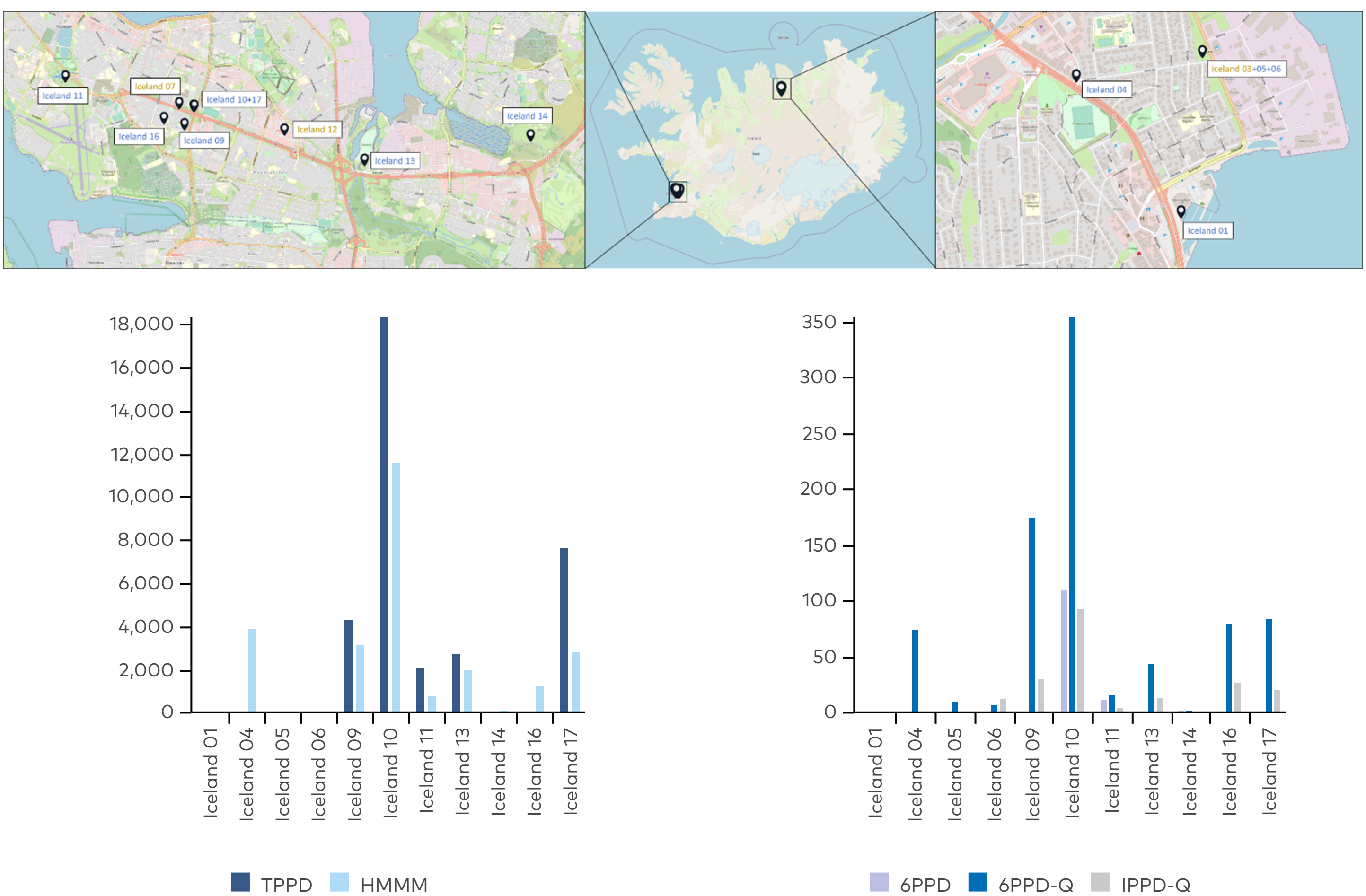


Figure 5: Concentrations in ng/L of selected tire related additive chemicals detected in water samples from Iceland.

In samples from Sweden, TPPD concentrations reached a maximum of 1,026 ng/L (Sweden 11) and HMMM a maximum of 97.5 ng/L (Sweden 17). This example shows that which TPPD and HMMM are the two predominant compounds in water samples, they do not necessarily follow the same trends. Sample Sweden 19 exhibited the lowest additive concentrations, with a ∑ concentration of 4.1 ng/L. The examples of samples Sweden 11 + 12, Sweden 17 + 18 and Sweden 19 + 20 indicate that the chemical pattern observed in stormwater tubes and their corresponding recipients can be very different (Figure 6). This information can be of high relevance for stakeholders, e.g. for the selection of relevant sites for the monitoring of tire related additive chemicals.

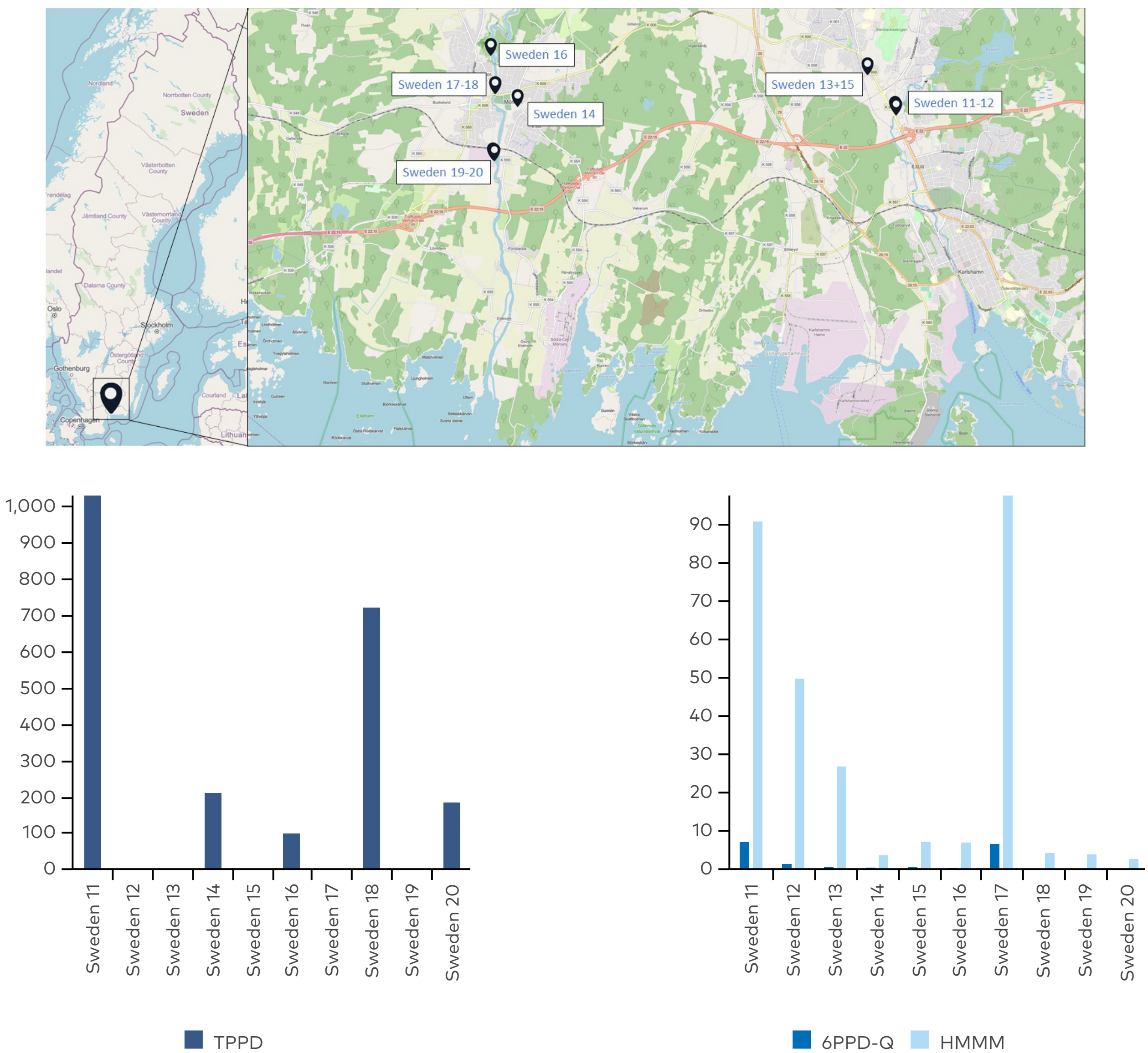


Figure 6: Concentrations in ng/L of selected tire related additive chemicals detected in water samples from Sweden.

In samples from the Faroe Islands a high heterogeneity was observed, e.g. in the TPPD concentrations which ranged from 4,185 ng/L (Faroe Islands 10) to 449,023 ng/L (Faroe Islands 07), the highest TPPD concentration detected in this study. Generally, the road run-off samples exhibited highest concentrations of tire related additive chemicals (Faroe Islands 03, 05-07), while samples from recipients (Faroe Islands 08 & 10) exhibited lowest concentrations (Figure 7).

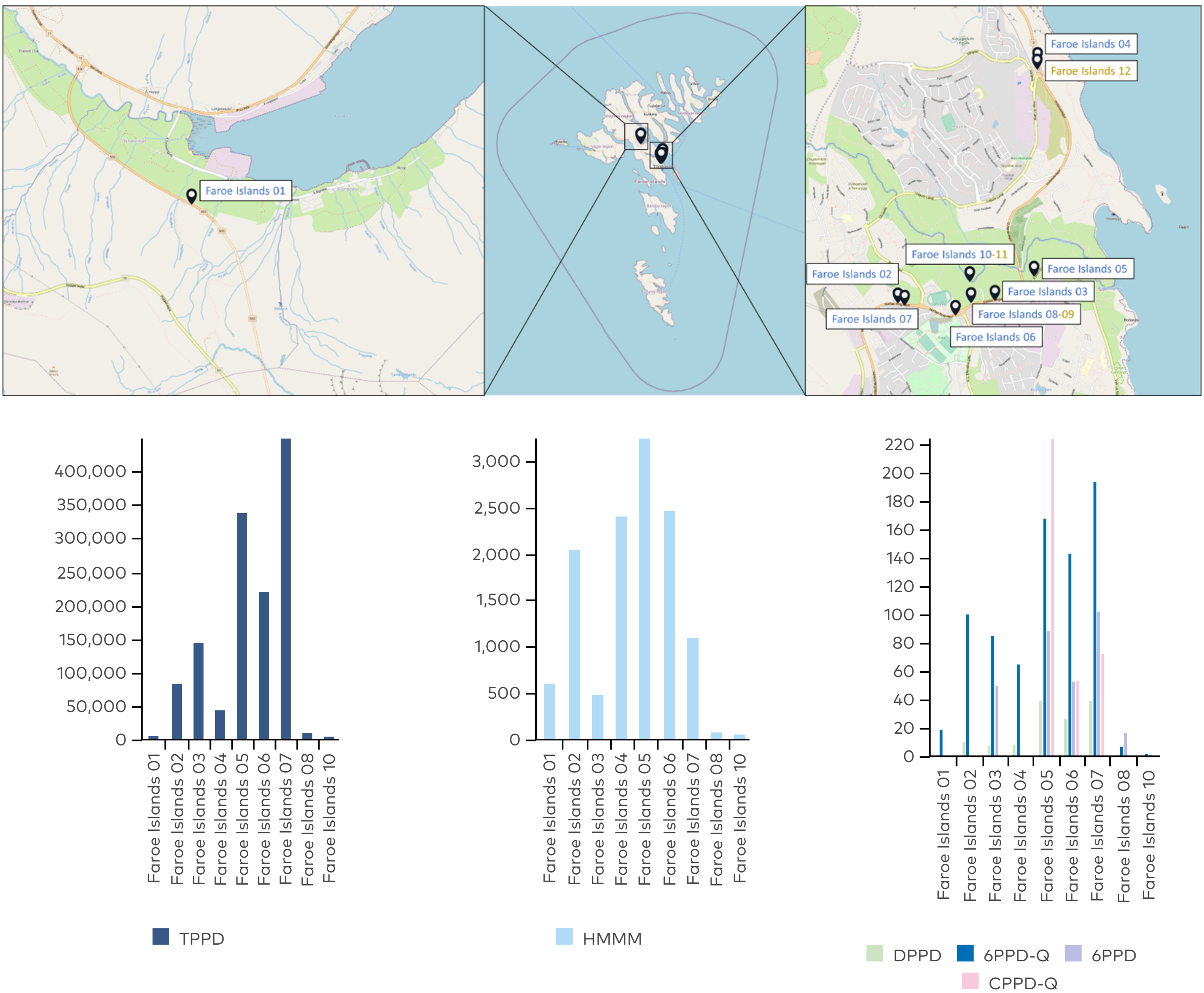


Figure 7: Concentrations in ng/L of selected tire related additive chemicals detected in water samples from the Faroe Islands.

In water samples from Finland, the two storm-/meltwater samples Finland 12 and 13, collected at the same location, exhibited highest TPPD, HMMM and 6PPD-Q concentrations (Figure 8). Sample Finland 11, collected at an urban stream, showed lowest concentrations, with a ∑ concentration of 235 ng/L. The stormwater samples collected after filtration ponds (Finland 09 & 15) show that after the natural filtration system (based on vegetation), tire related additive chemicals can still be detected in the water, though at varying concentrations.

Figure 8: Concentrations in ng/L of selected tire related additive chemicals detected in water samples from Finland.

Water samples from Denmark consisted of road run-off (Denmark 03 & 05) and recipient waters (Denmark 02, 06 & 08) from/near a major road (Figure 9). Interestingly, TPPD was not detected in the two road run-off samples, but they exhibited the highest HMMM concentrations (1,674 and 1,295 ng/L, respectively). 6PPD-Q showed highest concentrations in sample Denmark 03 (35.3 ng/L), while its parent compound 6PPD was absent in this sample.

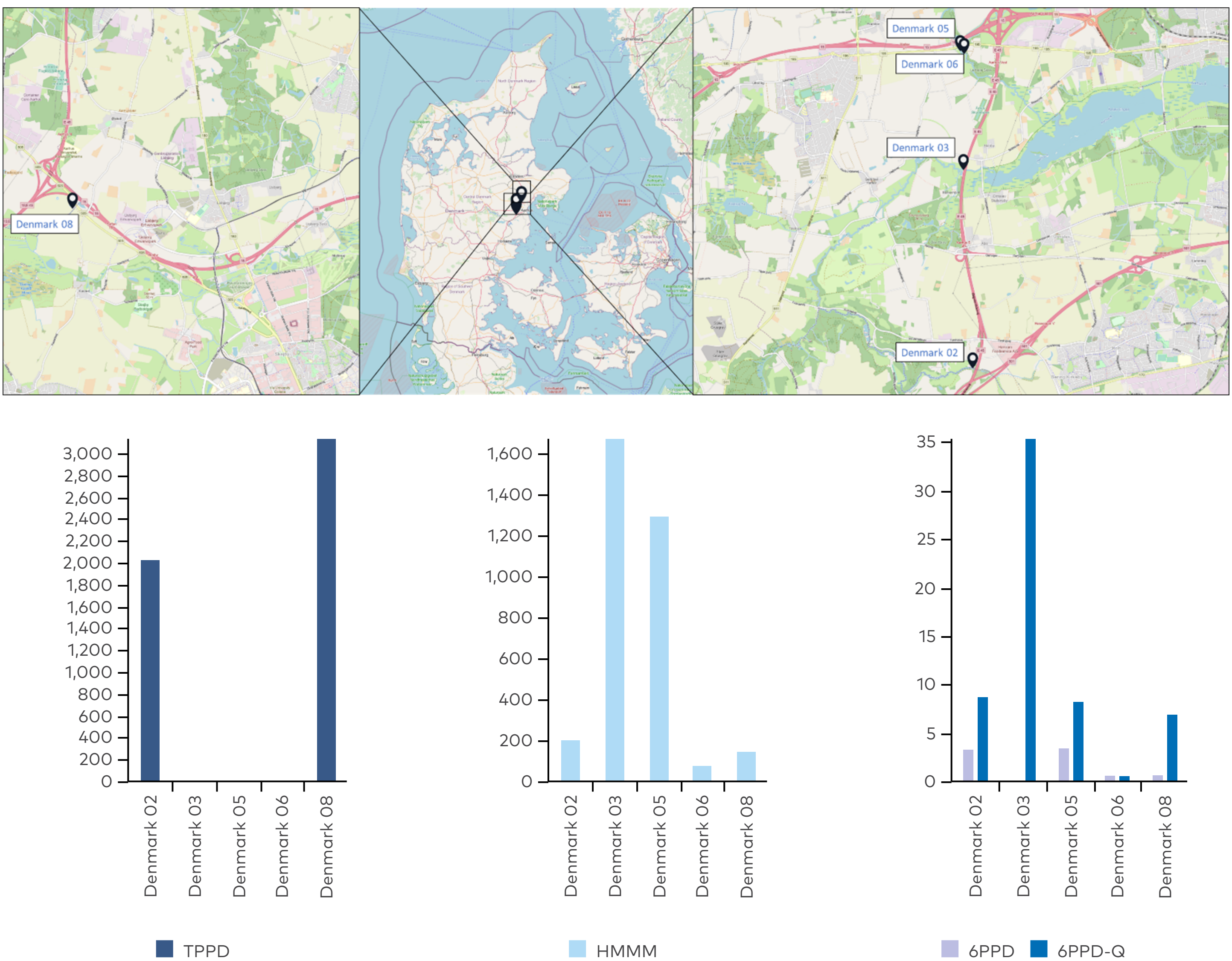


Figure 9: Concentrations in ng/L of selected tire related additive chemicals detected in water samples from Denmark.

In Norway, a snow sample from a residential background site (Norway 01) exhibited lowest additive concentrations (∑ 4.0 ng/L), while samples with highest concentrations all originated from a snow treatment facility (Norway 03, 04 & 06) (Figure 10). Samples Norway 15-19 represent dry and wet atmospheric deposition collected during a 14-day period each. The amount of tire related additive chemicals in these samples is not correlated with the amount of precipitation during the sampling periods (see Table 1). This suggests that the presence of these chemicals in these samples might rather be related to the presence of small airborne tire wear particles. The increasing concentrations towards the later sampling timepoints (e.g. Norway 18: 27.09.2023-11.10.2023 and Norway 19: 11.10.2023-25.10.2023) might be correlated with a switch from summer tires to winter tires.

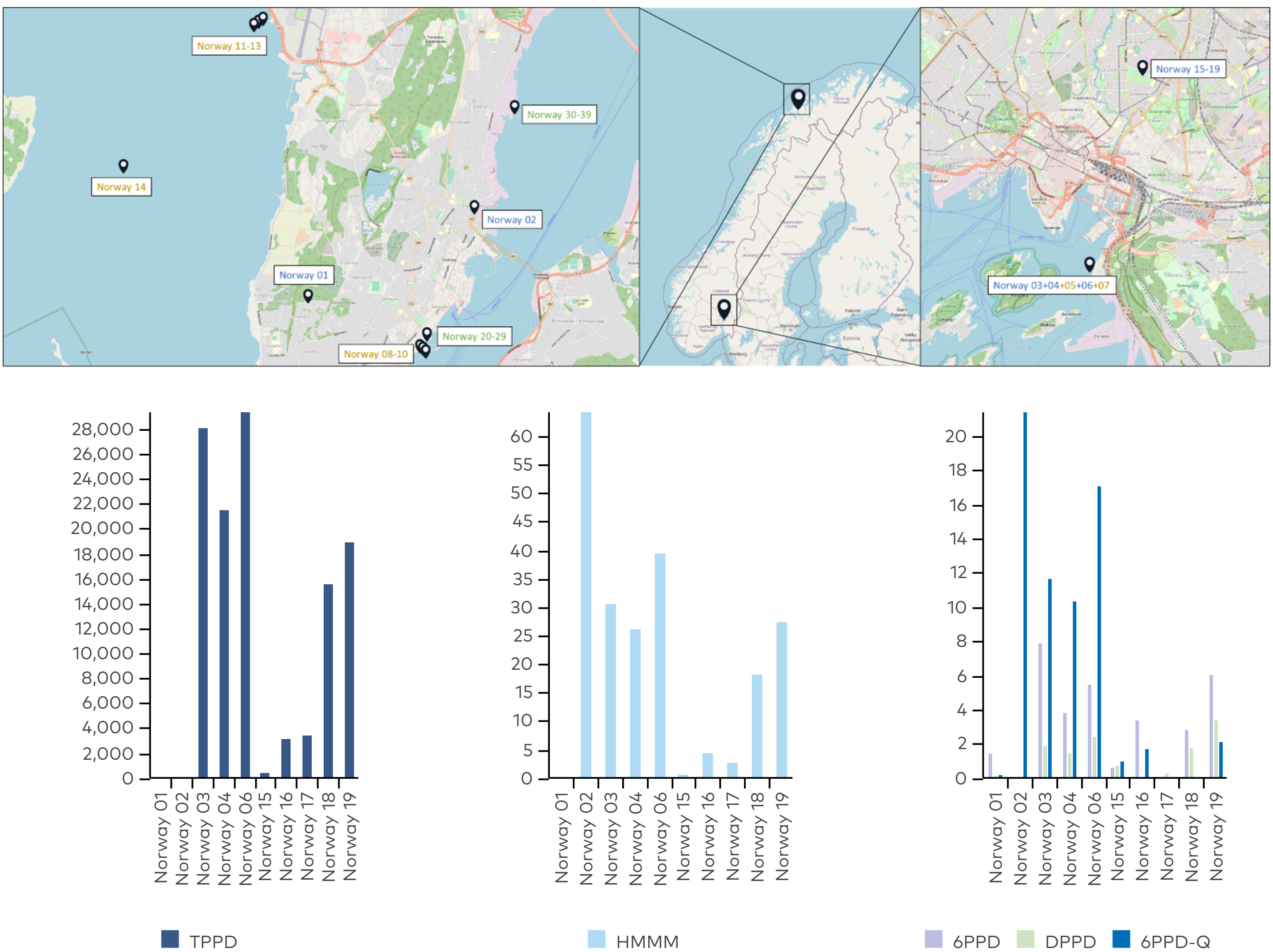


Figure 10: Concentrations in ng/L of selected tire related additive chemicals detected in water samples from Norway.

3.2 Sediment samples

In sediment samples, the highest chemical concentrations were observed in the two samples from the snow treatment facility in Oslo (Norway 05 and 07). Here, TPPD reached concentrations of 83,746-131,064 ng/g (Figure 11). The maximum 6PPD concentration was 278 ng/g, while its transformation product 6PPD-Q reached 13.7 ng/g.

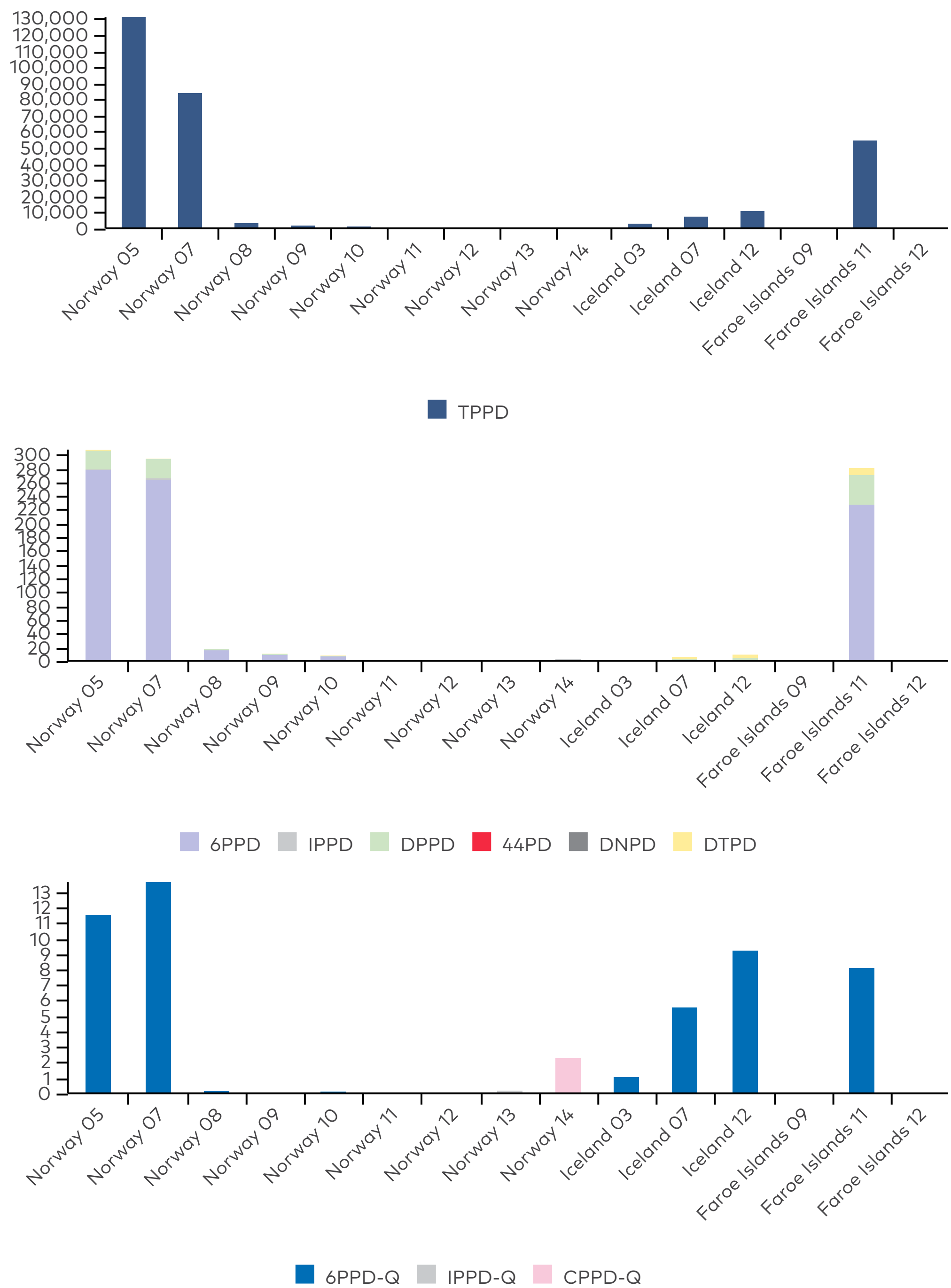


Figure 11: Concentrations in ng/g (dry weight) of tire related additive chemicals detected in sediment samples. Only chemicals detected at concentrations >LOQ in at least one sample are included. Please note the different scales on the y-axes.

3.3 Biota samples

As for water and sediment samples, TPPD was the predominant compound in blue mussel samples, with a maximum concentration of 737 ng/g and a high variability in concentrations among individual mussels (Figure 12). Of the three antioxidant transformation products measured in this study, CPPD-Q was the most abundant compound in biota samples, detected in all samples except one with values ranging from 1.2 to 9.6 ng/g.

Figure 12: Concentrations in ng/g (wet weight) of tire related additive chemicals detected in blue mussel samples. Only chemicals that were detected in at least one sample at concentrations >LOQ are included. Please note the different scales on the y-axes.

4 Discussion

Tire related additive chemicals were present in road run-off from Nordic countries as well as in associated compartments/sites such as snow dumping sites, stormwater recipients and soils/sediments. They were further detected in blue mussel samples collected in the wild, indicating that a transfer into biota is taking place. Target chemicals were present in 98% of samples and only one target chemical (CPPD) was not detected in any sample. This suggests a ubiquitous contamination by tire related additive chemicals, though at varying degrees depending on the sample type and location. For example, concentrations in marine sediments near the snow dumping sites in Tromsø (Norway 08-13) were relatively low, while sediments collected at a snow treatment facility in Oslo (Norway 05 & 07) exhibited concentrations 30–40 times higher. This is probably due to heavier traffic in Oslo and/or because the water mixing in marine waters can lead to a rapid dilution of chemicals, thus limiting their accumulation in sediments.

TPPD, an antioxidant, was the most abundant compound in all sample matrices. Literature on the occurrence of TPPD is almost nonexistent, but a laboratory exposure study has shown that ingestion of rubber particles by lumpfish can lead to a transfer of TPPD into the fishes' blood (Hägg et al., 2023). The high concentrations of TPPD detected in this study and the lack of comparable scientific literature indicate that more data is urgently needed.

The crosslinking agent HMMM is highly water soluble, explaining its presence in sometimes very high amounts in water samples, but its near absence in sediment and biota samples.

While road run-off water samples from the Faroe Islands and Iceland exhibited highest tire chemical concentrations, clear differences between the two countries could be seen concerning the relative abundance of individual chemicals: in samples from the Faroe Islands TPPD is highly predominant, while in samples from Iceland HMMM is very abundant. Different tire brands with different formulations could be used more commonly in certain countries than in others, but a detailed analysis of the tire market in Nordic countries would be needed to draw any conclusions. In general, variations in chemical concentrations between sampling sites can for example depend on the sample type, the amount of precipitation and UV irradiation before/during sampling or the frequency with which the roads are being cleaned and tire wear particles are hence being removed from the roads. The sampling timepoint can also be relevant when comparing tire additive concentrations in environmental samples: due to a lack of rain in many locations during spring 2023, the sampling campaign spanned from March until July (October for opportunistic atmospheric deposition samples), i.e., a period where transitions from winter tires, including studded tires, to all-year tires or summer tires are taking place.

The antioxidant and antiozonant 6PPD and its transformation product 6PPD-Q have recently been reported in three different fish species (Ji et al., 2022). However, this report is, to our knowledge, the first report on the occurrence of other tire related additives such as TPPD and CPPD-Q in wildlife. No data on toxicological effects of these compounds is known to us.

5 Acknowledgements

Thanks to Unni Mette Nordang (NILU) for packaging and shipping of the sample bottles. Fredrik Andreasson (Länsstyrelsen i Blekinge län, Sverige) and Kristin Galtung (NIVA) for sampling, in addition to all the local participants in the Nordic countries. We thank the City of Lahti for providing assistance in sampling and Halla W. Reinert for helping with the sampling on the Faroe Islands.

References

Challis, J.K., Popick, H., Prajapati, S., Harder, P., Giesy, J.P., McPhedran, K., Brinkmann, M., 2021. Occurrences of Tire Rubber-Derived Contaminants in Cold-Climate Urban Runoff. Environmental Science & Technology Letters, 8, 961-967.

EmissionsAnalytics, 2023. https://www.emissionsanalytics.com/news/fishy (accessed 29.11.2023).

Furuseth, I.S., Rødland, E.S., 2020. Reducing the Release of Microplastic from Tire Wear: Nordic Efforts. https://norden.diva-portal.org/smash/get/diva2:1515089/FULLTEXT01.pdf

Galtung, K. 2023, Master thesis, Exposure of marine invertebrates to car tire rubber: Uptake of rubber particles and related organic chemicals, Faculty of Bioscience, Fisheries and Economics, Department of Arctic and Marine Biology, University of Tromsø, Norway.

Hägg, F., Herzke, D., Nikiforov, V.A., Booth, A.M., Sperre, K.H., Sørensen, L., Cresse, M.E., Halsband, C., 2023. Ingestion of car tire crumb rubber and uptake of associated chemicals by lumpfish (*Cyclopterus lumpus*). Frontiers in Environmental Science. doi.org/10.3389/fenvs.2023.1219248

Ji, J., Li, C., Zhang, B., Wu, W., Wang, J., Zhu, J., Liu, D., Gao, R., Ma, Y., Pang, S., Li, X., 2022. Exploration of emerging environmental pollutants 6PPD and 6PPDQ in honey and fish samples. Food Chemistry, 396.

Johannessen, C., Helm, P., Lashuk, B., Yargeau, V., Metcalfe, C.D., 2022. The Tire Wear Compounds 6PPD-Quinone and 1,3-Diphenylguanidine in an Urban Watershed. Archives of Environmental Contamination and Toxicology, 82:171-179.

Klauschies, T., Isanta-Navarro, J., 2022. The joint effects of salt and 6PPD contamination on a freshwater herbivore. Science of the Total Environment, 829.

Rauert, C., Charlton, N., Okoffo, E.D., Stanton, R.S., Agua, A.R., Pirrung, M.C., Thomas, K.V., 2022. Concentrations of Tire Additive Chemicals and Tire Road Wear Particles in an Australian Urban Tributary. Environmental Science & Technology, 56, 2421-2431.

Rødland, E. S., Lind, O. C., Reid, M. J., Heier, L. S., Okoffo, E. D., Rauert, C., Thomas, K. V., Meland, S., 2022. Occurrence of tire and road wear particles in urban and peri-urban snowbanks, and their potential environmental implications. Science of the Total Environment, 824.

Seiwert, B., Klöckner, P., Wagner, S., Reemtsma, T., 2020. Source-related smart suspect screening in the aqueous environment: search for tire-derived persistent and mobile trace organic contaminants in surface waters. Analytical and Bioanalytical Chemistry, 412:4909-4919.

Sundt, P. S. R., Haugedal, T. R., Schulze, P.-E.,2021. Norske land baserte kilder til mikroplast (Norwegian Land-based Sources to Microplastics). MEPEX, Oslo, p.88.

Zeng, L., Li, Y., Sun, Y., Liu, L.-Y., Shen, M., Du, B., 2023. Widespread Occurrence and Transport of p-Phenylenediamines and Their Quinones in Sediments across Urban Rivers, Estuaries, Coasts, and Deep-Sea Regions. Environmental Science & Technology, 57 (6), 2393-2403.

Appendix A - Results summary table

Table A1: Information on sample type and concentrations of tire related additive chemicals detected in water (ng/L), sediment (ng/g, dry weight) and biota (ng/g, wet weight) samples.

Sample ID	Samp type	Details	6PPD	IPPD	DPPD	CPPD	TPPD	44PD	DNPD	DTPD	6PPD-Q	IPPD-Q	CPPD-Q	HMMM	77PD	Sol Red 52	Et2N-PD
Iceland 01	Water	Road run-off recipient (ocean)	<0.1	<0.1	<0.1	<1	<1	4.2	<0.1	<0.1	0.44	<0.5	<0.5	4.2	<1	<1	<5
Iceland 03	Sedi-ment	Soil from snow dumping site	<0.1	<0.1	1.7	<1	2842	<1	<0.1	<0.1	1.1	<0.5	<0.5	0.07	<1	<1	<5
Iceland 04	Water	Road run-off water	<0.1	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	73.4	<0.5	<0.5	3880	<1	<1	<5
Iceland 05	Snow	Snow dumping site	<0.1	<0.1	0.47	<1	<1	<1	<0.1	<0.1	9.4	<0.5	<0.5	25.4	<1	<1	<5
Iceland 06	Snow	Snow dumping site	<0.1	<0.1	<0.1	<1	<1	<1	0.18	<0.1	6.7	11.90	<0.5	21.3	<1	<1	<5
Iceland 07	Sedi-ment	Soil close to road	<0.1	<0.1	3.3	<1	7034	<1	<0.1	2.3	5.5	<0.5	<0.5	0.52	<1	<1	<5
Iceland 09	Water	Water from road	<0.1	<0.1	1.6	<1	4266	<1	0.10	1.7	173	29.6	<0.5	3113	<1	<1	<5

Iceland 10	Water	Road with heavy traffic	109	<0.1	4.7	<1	18325	<1	<0.1	<0.1	354	92.0	<0.5	11548	<1	<1	<5
Iceland 11	Water	Road run-off recipient. stream	10.8	<0.1	<0.1	<1	2081	<1	0.11	0.79	15.3	3.6	<0.5	756	<1	<1	<5
Iceland 12	Sedi-ment	Soil from big parking lot	<0.1	<0.1	4.8	<1	10545	<1	<0.1	4.2	9.2	<0.5	<0.5	0.25	<1	<1	<5
Iceland 13	Water	Road run-off recipient. retention pond	<0.1	<0.1	<0.1	<1	2699	<1	0.22	1.7	42.8	12.7	<0.5	1970	<1	<1	<5
Iceland 14	Water	Road run-off recipient. stream	0.89	<0.1	<0.1	<1	<1	<1	<0.1	0.43	1.2	<0.5	<0.5	48.1	<1	<1	<5
Iceland 16	Snow	Snow/ melting snow (little traffic)	<0.1	<0.1	1.3	<1	<1	<1	<0.1	1.8	78.9	25.9	<0.5	1195	<1	<1	<5
Iceland 17	Snow	Snow/ melting snow (heavy traffic)	<0.1	<0.1	2.5	<1	7610	<1	<0.1	<0.1	83.4	20.2	<0.5	2769	<1	<1	<5
Sweden 11	Water	Stormwater tube	<0.1	<0.1	<0.1	<1	1026	<1	0.25	0.68	7.0	<0.5	<0.5	90.7	<1	<1	<5
Sweden 12	Water	Recipient	0.46	<0.1	0.32	<1	<1	<1	<0.1	<0.1	1.3	<0.5	<0.5	49.7	<1	<1	<5
Sweden 13	Water	Stormwater tube	064	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	0.44	<0.5	<0.5	26.7	<1	<1	<5

Sweden 14	Water	Stormwater tube/very small stream	<0.1	<0.1	0.12	<1	209	<1	<0.1	<0.1	0.29	<0.5	<0.5	3.6	<1	<1	<5
Sweden 15	Water	Stormwater well	<0.1	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	0.61	<0.5	<0.5	7.1	<1	<1	<5
Sweden 16	Water	Recipient	<0.1	<0.1	<0.1	<1	96.9	<1	<0.1	<0.1	0.22	<0.5	<0.5	6.9	<1	<1	<5
Sweden 17	Water	Stormwater tube	<0.1	<0.1	<0.1	<1	<1	<1	0.11	<0.1	6.5	<0.5	<0.5	97.5	<1	<1	<5
Sweden 18	Water	Recipient	<0.1	<0.1	<0.1	<1	718	<1	<0.1	<0.1	<0.1	<0.5	<0.5	4.1	<1	<1	<5
Sweden 19	Water	Stormwater tube	0.15	<0.1	0.12	<1	<1	<1	<0.1	<0.1	<0.1	<0.5	<0.5	3.8	<1	<1	<5
Sweden 20	Water	Recipient	0.22	<0.1	<0.1	<1	182	<1	<0.1	<0.1	<0.1	<0.5	<0.5	2.6	<1	<1	<5
Faroe Islands 01	Snow	Snow (side of the road)	<0.1	<0.1	0.71	<1	5655	<1	0.30	0.76	18.6	<0.5	<0.5	596	<1	<1	<5
Faroe Islands 02	Snow	Snow (side of the road)	<0.1	<0.1	10.0	<1	83110	<1	0.12	1.6	99.7	<0.5	<0.5	2037	<1	<1	<5
Faroe Islands 03	Water	Road run-off water	49.4	<0.1	7.3	<1	144332	8.1	<0.1	3.7	84.9	<0.5	<0.5	481	<1	<1	<5
Faroe Islands 04	Snow	Snow	<0.1	<0.1	7.8	<1	43235	<1	0.43	2.1	64.5	<0.5	<0.5	2401	<1	<1	<5

Faroe Islands 05	Water	Road run-off water	88.4	<0.1	38.9	<1	337583	<1	<0.1	5.5	167	<0.5	224	3241	<1	<1	<5
Faroe Islands 06	Water	Road run-off water	52.6	<0.1	26.4	<1	219973	<1	1.0	7.0	143	<0.5	53.0	2460	<1	<1	<5
Faroe Islands 07	Water	Road run-off water	102	<0.1	39.0	<1	449023	<1	0.16	5.6	193	<0.5	72.2	1091	<1	<1	<5
Faroe Islands 08	Water	Recipient	16.1	<0.1	0.91	<1	9751	<1	<0.1	1.4	6.6	<0.5	<0.5	77.5	<1	<1	<5
Faroe Islands 09	Sedi-ment	Recipient	<0.1	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	<0.1	<0.5	<0.5	nd	<1	<1	<5
Faroe Islands 10	Water	Recipient	1.2	<0.1	0.7	<1	4185	<1	<0.1	<0.1	1.8	<0.5	<0.5	54.4	<1	<1	<5
Faroe Islands 11	Sedi-ment	Recipient	227	0.61	43.3	<1	54456	<1	<0.1	9.6	8.1	<0.5	<0.5	nd	<1	<1	<5
Faroe Islands 12	Sedi-ment	Soil from side of the road	<0.1	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	<0.1	<0.5	<0.5	nd	<1	<1	<5
Finland 06	Water	Stream	1.5	<0.1	<0.1	<1	1029	<1	<0.1	0.20	2.9	<0.5	<0.5	31.0	<1	<1	<5
Finland 09	Water	Stormwater after filtration	5.0	<0.1	0.78	<1	4127	<1	<0.1	<0.1	20.6	<0.5	<0.5	264	<1	<1	<5

Finland 11	Water	Urban stream	0.74	<0.1	<0.1	<1	208	<1	<0.1	<0.1	3.2	<0.5	<0.5	23.9	<1	<1	<5
Finland 12	Water	Storm/melt water	<0.1	<0.1	1.7	<1	6754	<1	<0.1	3.4	68.9	<0.5	<0.5	3672	<1	<1	<5
Finland 13	Water	Storm/melt water	<0.1	<0.1	<0.1	<1	12903	<1	<0.1	1.4	87.4	<0.5	<0.5	3091	<1	<1	<5
Finland 14	Water	Storm/melt water	3.5	<0.1	0.15	<1	1314	<1	<0.1	0.22	9.0	<0.5	<0.5	470	<1	<1	<5
Finland 15	Water	Stormwater after filtration ponds	<0.1	<0.1	<0.1	<1	<1	<1	<0.1	1.6	42.3	<0.5	<0.5	1737	<1	<1	<5
Denmark 02	Water	Recipient	3.3	<0.1	0.58	<1	2022	<1	<0.1	0.11	8.7	<0.5	<0.5	200	<1	<1	<5
Denmark 03	Water	Road run-off	<0.1	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	35.3	12.2	<0.5	1674	<1	<1	<5
Denmark 05	Water	Road run-off	3.4	<0.1	<0.1	<1	<1	<1	<0.1	0.18	8.2	<0.5	<0.5	1295	<1	<1	<5
Denmark 06	Water	Recipient	0.58	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	0.54	<0.5	<0.5	75.5	<1	<1	<5
Denmark 08	Water	Recipient	0.64	<0.1	2.6	<1	3134	<1	<0.1	<0.1	6.9	<0.5	<0.5	144	<1	<1	<5
Norway 01	Snow	Residential area. background site	1.4	<0.1	<0.1	<1	<1	<1	<0.1	<0.1	0.14	<0.5	2.3	nd	<1	<1	<5

Norway 02	Snow	Road with heavy traffic	<0.1	<0.1	<0.1	<1	<1	<1	0.26	<0.1	21.4	<0.5	<0.5	64.2	<1	<1	<5
Norway 03	Water	Outlet of snow treatment facility	7.9	<0.1	1.8	<1	28072	<1	<0.1	1.3	11.6	<0.5	<0.5	30.5	<1	<1	<5
Norway 04	Water	Last sedimentation basin before filtration step. snow treatment facility	3.8	0.12	1.4	<1	21471	<1	<0.1	1.7	10.3	<0.5	<0.5	26.1	<1	<1	<5
Norway 05	Sedi-ment	First sedimentation basin. snow treatment facility	278	0.59	27.9	<1	131064	<1	<0.1	1.1	11.5	<0.5	<0.5	0.22	<1	<1	<5
Norway 06	Water	First sedimentation basin. snow treatment facility	5.4	<0.1	2.4	<1	29347	<1	<0.1	1.0	17.0	<0.5	<0.5	39.4	<1	<1	<5
Norway 07	Sedi-ment	Sludge from membrane filter. snow treatment facility	264	1.8	28.6	<1	83746	<1	<0.1	0.43	13.7	<0.5	<0.5	0.24	<1	<1	<5
Norway 08	Sedi-ment	Transect 1 snow dumping site (marine)	15.7	0.13	1.4	<1	2961	<1	<0.1	<0.1	0.13	<0.5	<0.5	<0.1	<1	<1	<5

Norway 09	Sedi-ment	Transect 1 snow dumping site (marine)	8.2	0.56	1.3	<1	1696	<1	<0.1	<0.1	<0.1	<0.5	<0.5	<0.1	<1	<1	<5
Norway 10	Sedi-ment	Transect 1 snow dumping site (marine)	6.4	0.11	0.80	<1	1074	<1	<0.1	0.35	0.12	<0.5	<0.5	<0.1	<1	<1	<5
Norway 11	Sedi-ment	Transect 2 snow dumping site (marine)	0.19	<0.1	<0.1	<1	60.6	<1	<0.1	<0.1	<0.1	<0.5	<0.5	<0.1	<1	<1	<5
Norway 12	Sedi-ment	Transect 2 snow dumping site (marine)	<0.1	<0.1	0.10	<1	117	<1	<0.1	<0.1	<0.1	<0.5	<0.5	<0.1	<1	<1	<5
Norway 13	Sedi-ment	Transect 2 snow dumping site (marine)	<0.1	<0.1	0.19	<1	<1	<1	<0.1	<0.1	<0.1	<0.5	<0.5	<0.1	<1	<1	<5
Norway 14	Sedi-ment	Reference/ background site (marine)	1.5	0.97	0.21	<1	212	<1	<0.1	0.16	<0.1	<0.5	2.2	<0.1	<1	<1	<5
Norway 15	Water	Atmospheric deposition (dry & wet)	0.59	0.19	0.71	<1	378	<1	<0.1	1.2	0.96	<0.5	<0.5	0.54	<1	<1	<5
Norway 16	Water	Atmospheric deposition (dry & wet)	3.4	<0.1	<0.1	<1	3125	<1	<0.1	0.25	1.7	<0.5	<0.5	4.3	<1	<1	<5

Norway 17	Water	Atmospheric deposition (dry & wet)	<0.1	<0.1	0.26	<1	3383	<1	<0.1	0.60	<0.1	<0.5	<0.5	2.6	<1	<1	<5
Norway 18	Water	Atmospheric deposition (dry & wet)	2.8	<0.1	1.7	<1	15516	<1	<0.1	0.87	<0.1	<0.5	<0.5	18.1	<1	<1	<5
Norway 19	Water	Atmospheric deposition (dry & wet)	6.0	<0.1	3.3	<1	18882	<1	<0.1	0.95	2.1	<0.5	<0.5	27.3	<1	<1	<5
Norway 20	Biota	Blue mussel	0.56	<0.1	0.16	<1	291	<1	<0.1	0.18	<0.1	<0.5	2.5	<0.1	<1	<1	<5
Norway 21	Biota	Blue mussel	0.29	<0.1	0.12	<1	65.1	<1	<0.1	<0.1	<0.1	2.3	4.1	0.26	<1	<1	<5
Norway 22	Biota	Blue mussel	0.40	0.13	<0.1	<1	62.6	<1	<0.1	<0.1	<0.1	1.1	1.9	0.23	<1	<1	<5
Norway 23	Biota	Blue mussel	0.35	<0.1	<0.1	<1	105	<1	<0.1	<0.1	<0.1	<0.5	2.2	0.18	<1	<1	<5
Norway 24	Biota	Blue mussel	<0.1	<0.1	0.10	<1	410	<1	<0.1	<0.1	<0.1	<0.5	3.7	0.38	<1	<1	<5
Norway 25	Biota	Blue mussel	0.20	<0.1	0.25	<1	522	<1	<0.1	<0.1	<0.1	<0.5	9.6	0.37	<1	<1	<5
Norway 26	Biota	Blue mussel	1.8	<0.1	<0.1	<1	313	<1	<0.1	<0.1	0.11	<0.5	1.2	0.34	<1	<1	<5
Norway 27	Biota	Blue mussel	0.40	0.11	<0.1	<1	138	<1	<0.1	<0.1	<0.1	<0.5	4.2	1.2	<1	<1	<5

Norway 28	Biota	Blue mussel	0.89	0.10	0.17	<1	56.3	<1	<0.1	<0.1	<0.1	<0.5	2.7	0.18	<1	<1	<5
Norway 29	Biota	Blue mussel	0.86	0.10	<0.1	<1	154	<1	<0.1	<0.1	<0.1	<0.5	2.0	0.23	<1	<1	<5
Norway 30	Biota	Blue mussel	0.31	<0.1	0.19	<1	320	<1	<0.1	<0.1	<0.1	<0.5	5.1	0.20	<1	<1	<5
Norway 31	Biota	Blue mussel	0.16	<0.1	0.21	<1	186	<1	<0.1	0.11	<0.1	<0.5	6.0	0.24	<1	<1	<5
Norway 32	Biota	Blue mussel	0.23	<0.1	0.17	<1	737	<1	<0.1	<0.1	<0.1	<0.5	4.8	0.23	<1	<1	<5
Norway 33	Biota	Blue mussel	<0.1	<0.1	0.13	<1	415	<1	<0.1	<0.1	0.12	0.98	8.6	0.37	<1	<1	<5
Norway 34	Biota	Blue mussel	0.16	<0.1	0.13	<1	253	<1	<0.1	<0.1	<0.1	0.92	3.0	<0.1	<1	<1	<5
Norway 35	Biota	Blue mussel	1.5	<0.1	<0.1	<1	191	<1	<0.1	<0.1	<0.1	<0.5	2.1	0.16	<1	<1	<5
Norway 36	Biota	Blue mussel	0.42	<0.1	0.14	<1	692	<1	<0.1	<0.1	<0.1	0.99	1.7	0.26	<1	<1	<5
Norway 37	Biota	Blue mussel	<0.1	<0.1	0.14	<1	283	<1	<0.1	0.17	0.11	<0.5	<0.5	0.46	<1	<1	<5
Norway 38	Biota	Blue mussel	0.76	<0.1	0.10	<1	515	<1	<0.1	<0.1	<0.1	<0.5	3.2	0.35	<1	<1	<5
Norway 39	Biota	Blue mussel	0.91	<0.1	0.16	<1	383	<1	<0.1	<0.1	<0.1	<0.5	6.2	0.24	<1	<1	<5

Appendix B - Sampling manual for road run-off

General sampling strategy

Sampling site selection

The Joint Nordic Screening group have selected the sites relevant for their countries.

Homogeneity of samples

The sample represents a snapshot. and due to different sites and possible weather condition during sampling the homogeneity of the samples will vary.

Documentation

The sample protocol must be filled in and sent to NILU. together with the sample and/or by e-mail. If the documentation is filled out in the field. we recommend taking a photo of it as backup. An explanation should be given if variables are left blank. There must be a clear connection between the labels on the sample containers and the documentation.

If possible. add responsible person/persons in the form.

Sampling equipment

NILU will ship precleaned bottles to the participants together with aluminum foil to put under the cap. To transfer solid samples (snow. sediments or soils) into the bottles use precleaned spoons or spatulas.

How to sample

Contact between the sample and rubber materials (including rubber gloves) should be avoided at all times.

Road run-off water should be sampled close to the road. If possible. road run-off should be sampled during heavy rain events.

Do not fill the bottle more than ¾ full to avoid bursting of the bottles during freezing.

Snow samples should be taken from the bottom layer of the snow. i.e.. close to the road surface.

Sample storage

After sampling the bottles should be kept in a dark place and. if possible. frozen to limit transformation of compounds.

Sample shipment

The samples should be shipped to NILU as soon as possible.

If the street address is required for shipment. please use:

NILU
Framsenteret
Hjalmar Johansens gate 14.
9007 Tromsø
Attn: Linda Hanssen. Phone: + 47 92 88 72 91

Also. do not forget to add a proforma invoice with the shipment.

We would advise you to mark the shipment with:

Sampling information for NMR Screening. tire wear particles

NILU ID on bottle:		Customer ID:	
Sample material:			

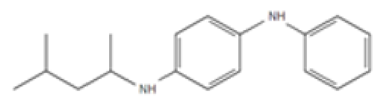
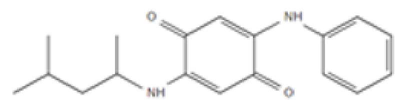
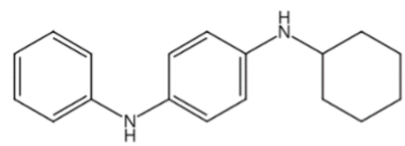
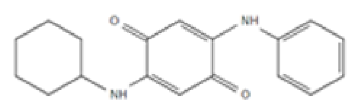
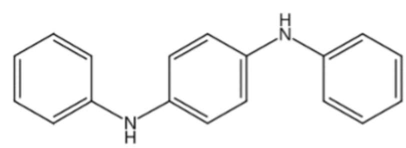
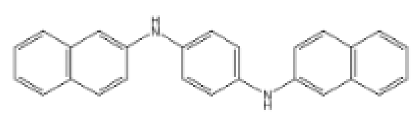
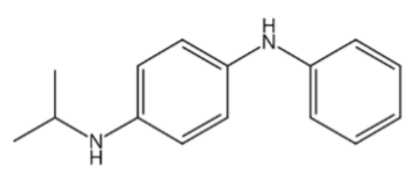
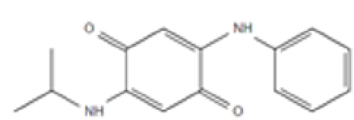
Sampling:		**Comments:**
Date and time:		
Site (description. GPS coordinates)		
Sample volume:		
Water temperature:		
Road salts used in the area?		
Precipitation amount in the last 24 h:		
Transport temperature after sampling:		
Observations/deviations from sampling procedure:		

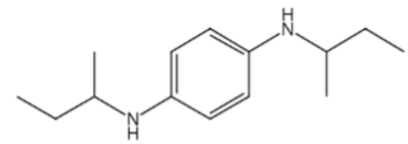
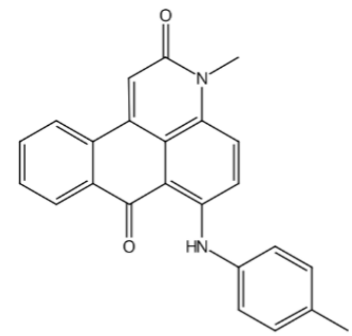
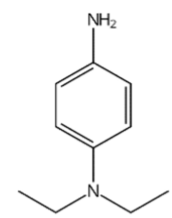
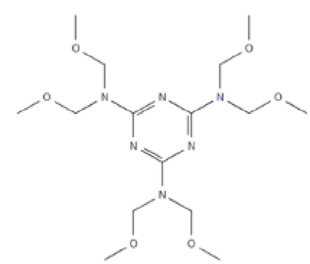
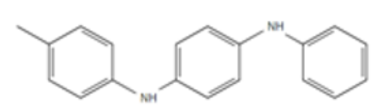
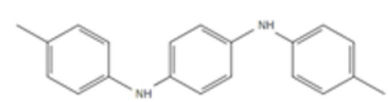
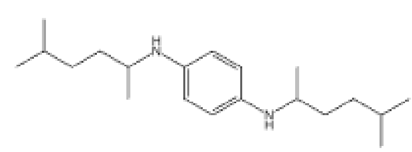
Storage:		**Comments:**
Storage date:		
Storage temperature:		
Special observations:		

Shipping:		**Comments:**
Date sample sent:		
Shipping temperature:		
Date sample received*:		
Condition upon arrival*:		
Special observations*:		

Appendix C - List of substances

Table C1: Overview of the substances included in the study.

CAS	NAME	Short name	Structure
793-24-8	N-(1.3-dimethylbutyl)-N′-phenyl-p-phenylenediamine	6-PPD	
2754428-18-5	N-(1.3-dimethylbutyl)-N′-phenyl-p-phenylenediamine-quinone	6-PPD-Q	
101-87-1	N1-Cyclohexyl-N4-phenyl-1.4-benzenediamine	CPPD	
68054-78-4	N1-Cyclohexyl-N4-phenyl-1.4-benzenediamine-quinone	CPDD-Q	
74-31-7	N.N′-Diphenyl-p-phenylenediamine	DPPD	
93-46-9	N.N′-Di(2-naphthyl)-p-phenylenediamine	DNPD	
101-72-4	N-Isopropyl-N'-phenyl-1.4-phenylenediamine Antioxidant 4010NA	IPPD	
	IPPD-Quinone	IPPD-Q	

101-96-2	*N.N*′-Di-*sec*-butyl-*p*-phenylenediamine	44PD	
81-39-0	3-Methyl-6-[(4-methylphenyl)amino]-3H-naphtho[1.2.3-de]quinoline-2.7-dione (ACI)	Solvent Red 52	
93-05-0	N.N-Diethyl-p-phenylenediamine 4-amino-N,N-diethylaniline	Et2N-PD	
68002-20-0	Hexa(methoxymethyl)melamin	HMMM	
101-54-2	N-Phenyl-N′-tolyl-p-phenylenediamine	TPDD*	
620-91-7	N,N′-Di-(p-tolyl)-p-phenylenediamine	DTPD*	
3081-14-9	N,N′-Bis(1,4-dimethylpentyl)-p-phenylenediamine	77PD	

** Provisional structures for TPPD and DTPD.*

NILU

The climate and environmental research institute NILU is an independent. non-profit research institution established in 1969. Through its research NILU increases the understanding of atmospheric composition. climate change. air quality. environmental contaminants. health effects. sustainable systems. circular economy. and digitalisation. Based on its research. NILU markets integrated services and products within analysing. monitoring and consulting. NILU is concerned with increasing public awareness about climate change and environmental pollution.

NILU's values:	Integrity - Competence - Benefit to society
NILU's vision:	Create sustainable development through internationally leading climate and environmental research

NILU
P.O. Box 100. NO-2027 KJELLER. Norway

E-mail: nilu@nilu.no
http://www.nilu.no
Enterprise no.: 941 705 561

ISBN: 978-82-425-
ISSN: 2464-3327

About this publication

Screening of compounds in tire wear road run off

Linda Hanssen, Natascha Schmidt, Vladimir Nikiforov

TemaNord 2024:525

ISBN 978-92-893-7861-1 (PDF)
ISBN 978-92-893-7862-8 (ONLINE)
http://dx.doi.org/10.6027/temanord2024-525

Cover photo: shawnanggg/Unsplash
Published: 28/5/2024

Disclaimer

This publication was funded by the Nordic Council of Ministers. However, the content does not necessarily reflect the Nordic Council of Ministers' views, opinions, attitudes or recommendations.

Rights and permissions

Nordic co-operation

Nordic co-operation is one of the world's most extensive forms of regional collaboration, involving Denmark, Finland, Iceland, Norway, Sweden, and the Faroe Islands, Greenland and Åland.

Nordic co-operation has firm traditions in politics, economics and culture and plays an important role in European and international forums. The Nordic community strives for a strong Nordic Region in a strong Europe.

Nordic co-operation promotes regional interests and values in a global world. The values shared by the Nordic countries help make the region one of the most innovative and competitive in the world.

The Nordic Council of Ministers
Nordens Hus
Ved Stranden 18
DK-1061 Copenhagen
pub@norden.org

Read more Nordic publications on www.norden.org/publications