



# Per- and polyfluoroalkyl substances (PFAS) in surface sediments of the North-east Atlantic Ocean: A non-natural PFAS background

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## ABSTRACT

The extreme persistence and environmental mobility of per- and polyfluoroalkyl substances (PFAS) make their presence ubiquitous in the marine environment. Target analysis of 20 most common PFAS revealed the presence of nine perfluoroalkyl acids at low levels in surface sediments from five Norwegian marine areas covering the vast region from the eastern North Sea in the south to the Arctic Ocean north of Svalbard in the north. After correcting for sediment characteristics, no substantial difference in the sum of the nine PFAS ( $\Sigma$ 9PFAS) between the five areas was found. Among separate compounds, PFOS, PFOA and PFNA dominate sample composition. Only two compounds, PFOS and PFUnDA, showed a statistically significant difference for one of the areas, the levels of these compounds being somewhat higher in the southernmost area than in the other areas. This may be due to local inputs in the fjords in this area. Open-sea and coastal sediments of the North-east Atlantic outside of locations with significant local sources seem to share a common, anthropogenic “PFAS background”, which may be part of a larger, global pattern.

## 1. Introduction

Synthetic organofluorine chemistry has had an explosive growth in the 20th century (Sandford, 2000). This led to abundant production and use of numerous substances that came to be classified as PFAS (per- and polyfluorinated alkylated substances) (Buck et al., 2011; Glüge et al., 2020), with the latest definition of this name including any molecule containing at least one fully fluorinated methyl or methylene carbon atom with no H, Cl, Br, I atoms attached to it (OECD, 2021.) It was only in early 2000-s that concerns about the global presence of PFAS in the environment were raised (Giesy and Kannan, 2001). After this, PFAS were found in all parts of the marine environment, including different types of biota, and significantly in the northern and arctic parts of the North Atlantic (e.g. Bossi et al., 2005; Smithwick et al., 2005; Yamashita et al., 2005; Kelly et al., 2009; Dassuncao et al., 2017; Valdernesnes et al., 2017; Skaar Solnes et al., 2019; Ali et al., 2021a; Hartz et al., 2023; Herzke et al., 2023). Significant local sources may be fire-fighting foams released during drills (Ahrens et al., 2015; Dubocq et al., 2020; Langberg et al., 2021; Ali et al., 2021a; Macorps et al., 2023), leachates from landfills (Knutsen et al., 2019, Ali et al., 2021a); wastewater treatment plant effluents (Ali et al., 2021b), and various types of industry

(Langberg et al., 2021). Once in the marine environment, PFAS may undergo sedimentation, with the sediments acting both as a sink for the contaminants, and as an important source of PFAS for benthic foodwebs (Lescord et al., 2015). While data on PFAS in benthic sediment feeders is scarce (Elvevoll et al., 2022), one study has described a rapid uptake and bioaccumulation of PFAS, particularly long-chain ones, by sea cucumbers (Martin et al., 2019). PFAS may then undergo further bioaccumulation and biomagnification (Kelly et al., 2009; Ren et al., 2022; Lewis et al., 2022; Khan et al., 2023), leading to adverse effects to animal life and humans (Ahrens and Bundschuh, 2014; Corsini et al., 2014; Chambers et al., 2021; Fenton et al., 2021).

A few specific, most common PFAS substances, such as PFOS and PFOA, have been included into the Stockholm Convention on banned chemicals (<http://chm.pops.int/>) as recently as 2019. In 2023, five European countries proposed a broader PFAS restriction to The European Chemicals Agency (ECHA, 2023). However, given their extreme environmental persistence and historical extensive use, PFAS are expected to remain for a long time after emissions cease. Further, various PFAS are mobile and undergo different types of long-range transport, via air and ocean currents and sea spray aerosols, affecting remote marine environments through diffuse contamination (Yamashita et al., 2005;

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Ahrens et al., 2009; Wong et al., 2018; Johansson et al., 2019; Evich et al., 2022; Sha et al., 2022; Xie and Kallenborn, 2023). Limited data exists on the background level of PFAS in marine environments impacted by diffuse contamination.

Here we report on PFAS levels and composition in surface sediments from a large part of the eastern North Atlantic open-sea and Norwegian fjord areas. Most parts of this region, including shelf and continental slope parts of the open sea, have not been studied for PFAS before. The

Norwegian MAREANO program of detailed geological, biological and geochemical mapping of the seabed (<http://www.mareano.no>) has since 2006 been assigned to study sediment chemical composition in the western Barents Sea, eastern Norwegian Sea and northeastern North Sea, and has delivered data on PFAS in surface sediments from 95 locations. The area mapped covers a vast marine region of 272 000 km<sup>2</sup> as of 2022. Few other studies have had the advantage of covering such large geographical areas with PFAS analyses of surface sediments using

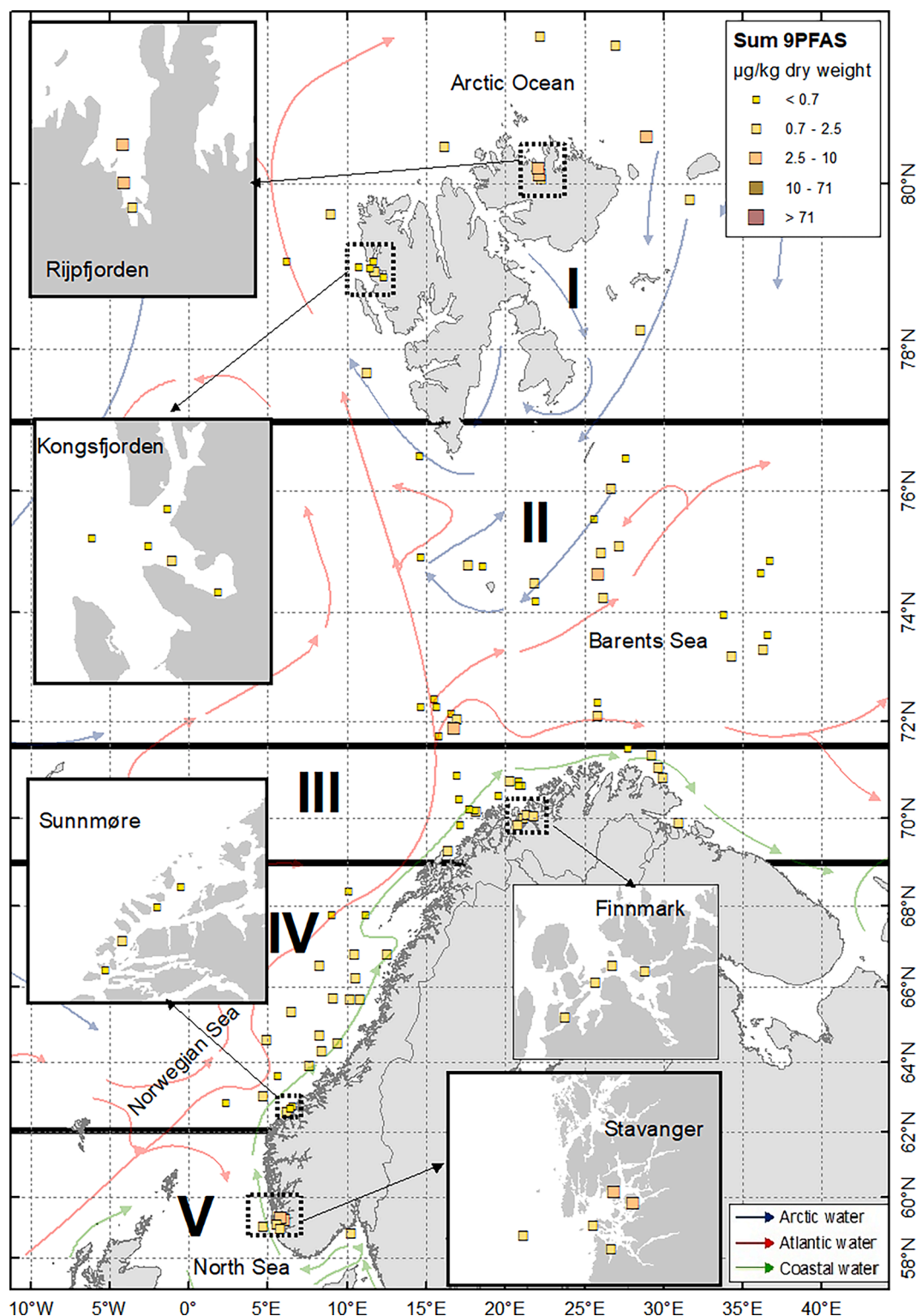


Fig. 1. Levels of 9 PFAS in surface sediments from five regions in Norwegian marine areas.

the same analytical method (e.g. Fiedler et al., 2022). Method inter-calibration uncertainty, inevitable in meta-studies and reviews, may be a significant source of error in this type of analyses (van der Veen et al., 2023). In this work, we discuss the absence of significant geographical variations in the extensive area studied and propose the existence of a non-natural “PFAS background” in marine surface sediments outside of locally exposed and contaminated areas.

## 2. Materials and methods

### 2.1. Sampling

This work is based on the analysis of surface sediment samples collected during 2007–2022 under the MAREANO program. All samples were collected during cruises with the Institute of Marine Research's research vessels. Sampling locations chosen for this study are shown in Fig. 1. The exact sampling positions, water depths and sampling years are given in Supplementary Data Table S1 together with sedimentation rates where those are available. Locations with fine-grained sediments were selected for sampling whenever possible. The specific locations were chosen based on detailed multibeam maps of the seabed provided in advance by the Norwegian Hydrographic Service as well as on seismic data available. The presence of fine-grain sediment material at each sampling station was further confirmed by high-resolution video recordings of the seabed in advance of the sampling. Most of the samples were from continental shelf areas (ca. 100–500 m water depth). Several samples were also taken from the continental slope, down to 1000–2000 m depth in the Norwegian Sea, see Table S1. A boxcore sampler was used for obtaining the samples. After opening the box, water was carefully removed by plastic hose with a glass tip and a sample of undisturbed surface (approximately 2 cm upper layer) was taken by a metal spade to pre-cleaned and baked glass jars, covered with aluminium foil and closed with lids. The samples were frozen onboard immediately after sampling and kept at -20 °C until analysis. Field blank samples were used with the samples gathered in 2018–2022. Field blank samples were prepared at the laboratory and kept sealed until the sampling, opened onboard at the same time as the boxcorer, sealed again when the samples were sealed and treated further in the same way as the samples. None of the 20 PFAS analysed in this work have been found in any of the field blank samples at any time. PFAS analyses were carried out in 2016–2023. The analyses of grain size and TOC % (see below) were carried out on surface sediments from multicore or boxcore samples taken at the same locations prior to the samples used for PFAS analyses.

### 2.2. Methods

#### 2.2.1. PFAS target analyses

The linear isomers of the target PFAS listed in Table S2 and their isotopically labeled standards listed below were obtained from Wellington Laboratories (Guelph, ON, Canada). Methanol (Lichrosolv from Meck) was checked for PFAS contamination before use. To a 50 mL polypropylene tube,  $2 \pm 0.2$  g sample (dried sediment) were added, spiked with 20 ng isotopically labelled PFAS internal standard mixture ( $^{13}\text{C}$  6:2 FTSA,  $^{13}\text{C}$  8:2 FTSA,  $^{13}\text{C}$  CPFBS,  $^{13}\text{C}$  PFHxS,  $^{13}\text{C}$  PFOS,  $^{13}\text{C}$  FOSA,  $^{13}\text{C}$  PFBA,  $^{13}\text{C}$  PFPEA,  $^{13}\text{C}$  PFHxA,  $^{13}\text{C}$  PFHpA,  $^{13}\text{C}$  PFOA,  $^{13}\text{C}$  PFNA,  $^{13}\text{C}$  PFDA,  $^{13}\text{C}$  PFUnDA,  $^{13}\text{D}$  PFDoDA,  $^{13}\text{C}$  PFTeDA, see Hanssen et al., 2013, for further details). Before extraction the sediment was soaked with 1 mL of 200 mM NaOH in methanol for 30 min, followed by 100  $\mu\text{L}$  2M HCl and 9 mL of methanol. The samples were sonicated three times for 10 min with vortexing in between. After extraction the samples were centrifuged before the supernatant was concentrated under vacuum to 2 mL, cleaned up by suspensive EnviCarb treatment under acidic conditions. After addition of the recovery standard (3,7-dimethyl PFOA), the samples were ready for analysis. The recovery standard is used to calculate the recovery of each internal standard in each sample, see results in table S3. Analyses were conducted by ultra-high pressure

liquid chromatography triple-quadrupole mass spectrometry (UHPLC-MS/MS), as previously described by Hanssen et al., 2013.

#### 2.2.2. Quality assurance and quality control

The quality and control of the extraction and analysis regarding repeatability and precision of the targeted PFAS was controlled by laboratory blank analysed with each sample batch, sediment spiking with native standards carried out to control the extraction efficiency, and extraction of replicates of samples (three replicates of one sample analysed with each sample batch). The calibration curve with a concentration range from 0.02 pg/ $\mu\text{L}$  to 10 pg/ $\mu\text{L}$  was analysed in every sequence, together with the samples. Calibration curves exhibited good linearity with correlation coefficients  $\geq 0.99$  for each analyte. To avoid PFAS contamination from the instrument, the LC system was equipped with a PFAS kit, which includes PEEK lines. In addition, a trapping column was installed before the injector to withhold the PFAS leaching from the system, avoiding it from eluting at the same time as the native PFAS in the sample.

Methanol injections were done between the calibration curve and the samples, and in between the samples to control for carry over. NILU participates yearly in a ring test program arranged by Arctic Monitoring and Assessment Program. The limit of quantification (LOQ) for separate analytes varied in different years between 0.003 and 0.18  $\mu\text{g}/\text{kg}$  dry weight. For specific LOQ values for each analyte see Supplementary Materials table S2.

#### 2.2.3. Sediment characteristics

The sediments collected for sediment characterization are obtained from either multicore or boxcore sampling. The sediments are sliced in 1 cm thick slices on-board and kept frozen at -20°C until freeze-drying and followed by grain size and total organic carbon (TOC) analyses.

One set of sub-samples was analysed for weight percentages (wt. %), TOC using a LECO CS 244 analyser (Leco Corporation, USA). For the TOC analyses, aliquots (~200 mg) of the samples were treated with 10 % (volume) hydrochloric acid (HCl) at 60°C to remove inorganic carbonate, and then washed with distilled water to remove HCl.

Grain size analyses of total sediment were obtained by laser diffraction techniques (Coulter LS 2000) (for details, see Xu, 2000, and references therein) and wet sieving. The Coulter LS 2000 measured the fractions from 0.4  $\mu\text{m}$  to 2 mm while particles larger than 2 mm were determined by wet sieving. Prior to each analysis, the samples were freeze-dried. The grain size distribution is determined with respect to volume percent and with the assumption of uniform density of the sample.

#### 2.2.4. Statistics and data treatment

To avoid the possibility of LOQ serving as a confounding variable, the highest value observed was uniformly applied in the statistical model used. Values falling below the LOQ were assigned a zero, yielding conservative estimates of concentrations. A log-link generalized linear model with Poisson-gamma distributed errors was used to analyze the zero-inflated data due to left-censoring by LOQ. The model incorporated the predictors area, TOC, and mud % and was implemented using the 'glmTMB' 1.1.8 package in 'R' 4.2.1 (Brooks et al., 2017). Data exploration showed mud to be collinear with TOC, and stepwise model selection based on Akaike's information criteria signified mud% to have highest predictive value. Thus, area and mud % were retained for the regression analysis. To test for differences between the areas, pairwise comparisons were performed using 'multcomp' 1.4.25 with single-step p-value adjustment (Hothorn et al., 2008). Simulation-based model validation was performed and is presented in the supplementary information.

### 3. Results and discussion

#### 3.1. PFAS concentrations

Out of twenty compounds analyzed (see Table S2 for the full list), only nine PFAS were found in the samples, with a maximum of seven, or less, in any given sample. Sum of 9 PFAS,  $\Sigma$ 9PFAS, is therefore used as the overall parameter for PFAS concentrations in the studied samples. The concentrations of the nine PFAS found above LOQ in the sediments are shown in Table 1 together with sediment characteristics (TOC and grain size, given as mud %, i.e. the percentage of particles below 63  $\mu$ m size). The complete results for all locations are given in Table S1, and the chemical structures of the nine PFAS are shown in Table S4 together with their partition coefficients.

Since the studied area covers a vast region of the ocean, stretching from the Skagerrak in the South to the Arctic Ocean in the North, it has been divided into five subregions to investigate possible geographical differences, as shown in Fig. 1. The subregions were defined based on natural geographical divisions adjusted to the sampling strategy of the MAREANO project and then rounded off to the nearest degree or half a degree North, resulting in the following: Area I (north of 77°N): Svalbard area and the Arctic Ocean; Area II (between 71.5°N and 77°N): Central Barents Sea; Area III (between 69°N and 71.5°N): Southern Barents Sea and coastal areas of Northern Norway; Area IV (between 62°N and 69°N): The Norwegian Sea coastal, shelf and continental slope areas;

Area V (south of 62°N): the North Sea and the Skagerrak.

The levels of PFAS are generally low, at 1.0  $\mu$ g/kg dry weight for  $\Sigma$ 9PFAS on average and up to 4.4  $\mu$ g/kg dry weight at its highest and are below LOQ for all compounds at nine out of 95 locations. These results are comparable to what was found in other studies of sediments in open-sea and remote freshwater areas (e.g. Lescord et al., 2015; Kahkashan et al., 2019; Léon et al., 2020; Lin et al., 2020; Fiedler et al., 2022; Shimizu et al., 2022; Chen et al., 2023), but are far below the levels found in areas affected by local sources (Lescord et al., 2015; Knutsen et al., 2019; Langberg et al., 2021; Macrops et al., 2023). The levels of PFAS presented here presumably represent the highest levels achieved for the studied areas, since sediment cores typically show increasing trends of PFAS in recently deposited layers (e.g. Theobald et al., 2012, Shen et al., 2018; Mussabek et al., 2019), with some studies of heavily impacted areas reporting downward trends in most recent sediments, similar to some other persistent organic pollutants (Logemann et al., 2022). Open sea areas of the North Atlantic typically have sedimentation rates of approximately 1 mm per year and below (e.g. Jensen et al., 2009). Sedimentation rates were measured in 31 out of 95 samples in this dataset, see Table S1, and ranged between 0.60 and 2.0 mm/year. This makes the upper layer of 2 cm representative of approximately the last 10–30 years, i.e. presumably containing the highest levels of PFAS.

**Table 1**

Concentrations of nine PFAS in  $\mu$ g/kg dry weight and sediment characteristics of surface sediments from Norwegian marine areas. Mud % is defined as percentage of particles below 63  $\mu$ m size. SD: standard deviation. All the measured results are shown in table S1. For full names and values of LOQ in different samples, see Supplementary Materials table S2.

|  | Mud %           | TOC%                        | PFOS               | PFHxA | PFHpA              | PFOA               | PFNA               | PFDA               | PFUnDA             | PFDoDA | PFTTrA             | $\Sigma$ 9PFAS     |
|--|-----------------|-----------------------------|--------------------|-------|--------------------|--------------------|--------------------|--------------------|--------------------|--------|--------------------|--------------------|
| <i>Overall (n=95)</i>  |                 |                             |                    |       |                    |                    |                    |                    |                    |        |                    |                    |
| Min  | 13              | 0.25                        | <LOQ               | <LOQ  | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | <LOQ               |
| Mean $\pm$ SD  | 71 $\pm$ 27     | 1.4 $\pm$ 0.99              | 0.28<br>$\pm$ 0.30 | <LOQ  | <LOQ               | 0.34<br>$\pm$ 0.30 | 0.21<br>$\pm$ 0.20 | <LOQ               | 0.09<br>$\pm$ 0.12 | <LOQ   | <LOQ               | 1.0 $\pm$ 0.93     |
| Median   | 82              | 1.2                         | 0.21               | <LOQ  | <LOQ               | 0.29               | 0.18               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | 0.80               |
| Max  | 98              | 5.5                         | 1.7                | 0.13  | 0.29               | 1.4                | 0.89               | 0.38               | 0.67               | 0.29   | 0.38               | 4.4                |
| n (above LOQ)  | 95              | 95                          | 76                 | 11    | 31                 | 86                 | 75                 | 38                 | 47                 | 1      | 1                  | 86                 |
| <i>I. Svalbard. Barents Sea northern parts and Arctic Ocean (n=18)</i> |                 |                             |                    |       |                    |                    |                    |                    |                    |        |                    |                    |
| Min  | 18              | 0.35                        | <LOQ               | <LOQ  | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | <LOQ               |
| Mean $\pm$ SD  | 89              | 1.4                         | 0.31               | <LOQ  | 0.06               | 0.46               | 0.32               | <LOQ               | 0.08               | <LOQ   | <LOQ               | 1.3                |
| Median   | 96              | 1.4                         | 0.17               | <LOQ  | <LOQ               | 0.34               | 0.21               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | 0.83               |
| Max  | 98              | 2.2                         | 1.1                | <LOQ  | 0.29               | 1.4                | 0.89               | 0.18               | 0.28               | <LOQ   | <LOQ               | 4.1                |
| n (above LOQ)  | 18              | 18                          | 17                 | 0     | 8                  | 17                 | 15                 | 7                  | 9                  | 0      | 0                  | 17                 |
| <i>II. Barents Sea central and western parts (n=27)</i>                |                 |                             |                    |       |                    |                    |                    |                    |                    |        |                    |                    |
| Min  | 22              | 0.25                        | <LOQ               | <LOQ  | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | <LOQ               |
| Mean $\pm$ SD  | 66 $\pm$ 23     | 1.6 $\pm$ 0.97              | 0.20<br>$\pm$ 0.22 | <LOQ  | <LOQ               | 0.36<br>$\pm$ 0.34 | 0.20<br>$\pm$ 0.16 | <LOQ               | 0.06<br>$\pm$ 0.09 | <LOQ   | <LOQ               | 0.89<br>$\pm$ 0.87 |
| Median   | 74              | 1.7                         | 0.16               | <LOQ  | <LOQ               | 0.28               | 0.15               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | 0.55               |
| Max  | 96              | 3.6                         | 0.87               | 0.13  | 0.19               | 1.3                | 0.53               | 0.15               | 0.35               | <LOQ   | <LOQ               | 3.0                |
| n (above LOQ)  | 27              | 27                          | 20                 | 6     | 6                  | 24                 | 21                 | 7                  | 11                 | 0      | 0                  | 24                 |
| <i>III. Barents Sea southern and coastal parts (n=21)</i>              |                 |                             |                    |       |                    |                    |                    |                    |                    |        |                    |                    |
| Min  | 13              | 0.28                        | <LOQ               | <LOQ  | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | <LOQ               |
| Mean $\pm$ SD  | 49 $\pm$ 31     | 0.94<br>$\pm$ 0.68          | 0.26<br>$\pm$ 0.23 | <LOQ  | <LOQ               | 0.26<br>$\pm$ 0.20 | 0.16<br>$\pm$ 0.13 | <LOQ               | 0.07<br>$\pm$ 0.11 | <LOQ   | <LOQ               | 0.80<br>$\pm$ 0.67 |
| Median   | 40              | 0.66                        | 0.23               | <LOQ  | <LOQ               | 0.26               | 0.16               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | 0.77               |
| Max  | 97              | 2.5                         | 0.67               | 0.13  | 0.11               | 0.77               | 0.42               | 0.17               | 0.31               | <LOQ   | <LOQ               | 2.3                |
| n (above LOQ)  | 21              | 21                          | 15                 | 1     | 1                  | 18                 | 15                 | 7                  | 7                  | 0      | 0                  | 18                 |
| <i>IV. Norwegian Sea shelf and continental slope areas (n=23)</i>      |                 |                             |                    |       |                    |                    |                    |                    |                    |        |                    |                    |
| Min  | 36              | 0.32                        | <LOQ               | <LOQ  | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ               | <LOQ   | <LOQ               | <LOQ               |
| Mean $\pm$ SD  | 76 $\pm$ 17     | 1.1 $\pm$ 1.1<br>$\pm$ 0.19 | 0.24<br>$\pm$ 0.19 | <LOQ  | 0.05<br>$\pm$ 0.05 | 0.27<br>$\pm$ 0.19 | 0.17<br>$\pm$ 0.14 | <LOQ               | 0.08<br>$\pm$ 0.08 | <LOQ   | <LOQ               | 0.87<br>$\pm$ 0.54 |
| Median   | 80              | 0.71                        | 0.25               | <LOQ  | 0.06               | 0.31               | 0.17               | 0.06               | 0.07               | <LOQ   | <LOQ               | 0.98               |
| Max  | 95              | 4.5                         | 0.73               | 0.13  | 0.17               | 0.57               | 0.44               | 0.14               | 0.23               | <LOQ   | <LOQ               | 1.7                |
| n (above LOQ)  | 23              | 23                          | 18                 | 2     | 13                 | 21                 | 18                 | 12                 | 14                 | 0      | 0                  | 21                 |
| <i>V. Southern areas (n=6)</i>   |                 |                             |                    |       |                    |                    |                    |                    |                    |        |                    |                    |
| Min  | 84              | 1.6                         | 0.28               | <LOQ  | <LOQ               | 0.13               | 0.05               | <LOQ               | 0.12               | <LOQ   | <LOQ               | 0.75               |
| Mean $\pm$ SD  | 93<br>$\pm$ 5.7 | 2.6 $\pm$ 1.4<br>$\pm$ 0.57 | 0.76<br>$\pm$ 0.57 | <LOQ  | 0.07<br>$\pm$ 0.08 | 0.40<br>$\pm$ 0.28 | 0.36<br>$\pm$ 0.27 | 0.15<br>$\pm$ 0.14 | 0.31<br>$\pm$ 0.22 | <LOQ   | 0.06<br>$\pm$ 0.16 | 2.2 $\pm$ 1.4      |
| Median   | 97              | 2.1                         | 0.52               | <LOQ  | <LOQ               | 0.36               | 0.31               | 0.10               | 0.25               | <LOQ   | <LOQ               | 2.0                |
| Max  | 97              | 5.5                         | 1.7                | 0.13  | 0.17               | 0.82               | 0.79               | 0.38               | 0.67               | 0.29   | 0.38               | 4.4                |
| n (above LOQ)  | 6               | 6                           | 6                  | 2     | 3                  | 6                  | 6                  | 5                  | 6                  | 1      | 1                  | 6                  |



### 3.2. Sediment characteristics

Sediment characteristics, such as TOC and grain size, may affect the amounts of PFAS found in sediments, since they play a significant role in PFAS sorption to sediments (Higgins and Luthy, 2006; Ahrens et al., 2011). A positive correlation between TOC and grain size (GS) is often found for fine-grained sediments (Secrieru and Oaie, 2009). The 95 surface sediment samples considered here had a significant positive correlation ( $p < 0.0001$ ) between the two parameters, see Figure S1.

Concentrations of  $\Sigma 9$ PFAS were positively correlated to mud % in the samples from the same locations (see Fig. 2 below and Table S5). Mud % seems to explain a considerable part of the variation in the concentrations of  $\Sigma 9$ PFAS found in the samples. In contrast, the correlation between these concentrations and total organic carbon (TOC) was much weaker and not significant at the  $p = 0.05$  level. The long-chain PFAS compounds PFOS, PFUnDA and to a lesser degree, PFDA, were positively correlated with TOC, though marginally or not significant at the  $p < 0.05$  level. This is in agreement with some other studies (Lin et al., 2020; Shimizu et al., 2022), although studies of more densely populated and exposed areas demonstrate that this relationship is not universal (e.g. Theobald et al., 2012, Shen et al., 2018). The effect of other characteristics not measured in this study, such as density, pH, and certain metal ion concentrations, on PFAS sorption is relevant mostly for short-chain compounds (Higgins and Luthy, 2006).

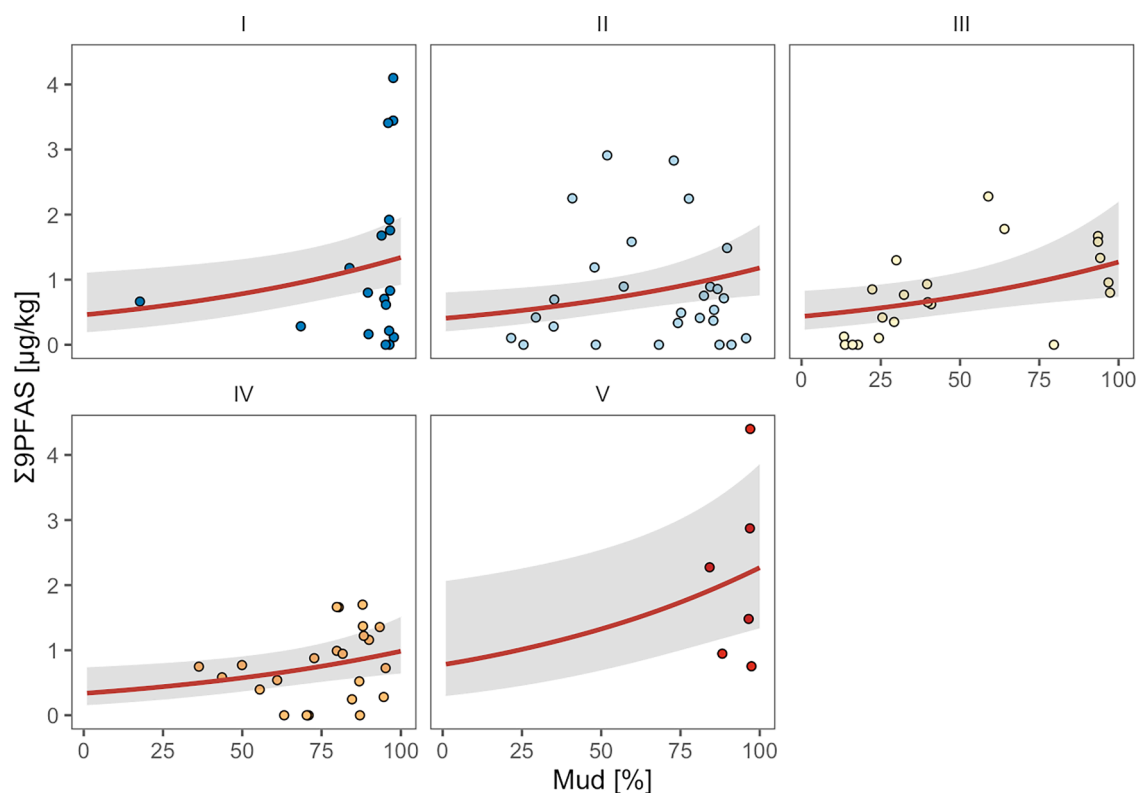
### 3.3. Spatial distribution

No clear geographical trends were immediately apparent in the low-to-moderate levels of  $\Sigma 9$ PFAS found in all areas, see the map in Fig. 1. Some of the highest concentrations (up to 4.4  $\mu\text{g}/\text{kg}$  dry weight for  $\Sigma 9$ PFAS) were found in area V close to the city of Stavanger, which may have local sources of pollution, such as the airport of Stavanger, local wastewater treatment plant, and more. These levels were, however, comparable to those found at some other locations, including Svalbard

(area I), where the concentrations of 9 PFAS were up to 4.1  $\mu\text{g}/\text{kg}$  dry weight. Ocean depths did not seem to affect the results significantly either, as the majority of the samples were from shelf locations below 500 m depth, see figure S2.

Possible geographical trends were further investigated by correlation analysis of the concentrations and geographical latitude of sampling locations. A south-to-north concentration gradient might be expected in the spatial distribution of surface sediment concentrations in accordance with the possible sources being located largely in the south, closer to densely populated and industrialized parts of Europe, and major ocean currents directed northwards along the coast of Norway and further to the Arctic Ocean (see map in Fig. 1). Since grain size explains some of the variation in the concentrations of  $\Sigma 9$ PFAS, the levels were corrected for mud % before checking the influence of the latitude. No significant differences between the five established areas (see Fig. 1) were found when correcting for mud % (Fig. 2). An earlier study has found significant geographical differences in PFAS levels in cod liver between eastern parts of the Norwegian coast (the Skagerrak) and its southwestern parts, but no differences between the southwestern and the northwestern parts of the coast (Valdersnes et al., 2017). Our dataset does not include coastal areas of the Skagerrak but confirms the absence of differences between the south and the north.

These results are in contrast with data on other types of organic contaminants found earlier in sediments from the same areas, which vary locally and on a larger geographical scale due to the influence of sea currents, marginal ice zone, “grasshopper effect”, inputs from natural sources etc. (e.g. Everaert et al., 2017; Boitsov et al., 2020). An absence of statistically significant geographical differences in PFAS levels throughout the enormous area mapped by MAREANO may point to an existence of a certain non-natural “PFAS background” in marine sediments outside of local hot spots. While this “background” may be subject to change in time as more PFAS are brought to the sediments, the studied sediments represent mostly overlapping time periods (Table S1). Our results suggest the levels to be similar for these time periods throughout



**Fig. 2.** Concentrations of  $\Sigma 9$ PFAS ( $\mu\text{g}/\text{kg}$  dry weight) in relation to mud % (grain size fraction  $< 63 \mu\text{m}$ ) by area, fitted a log-link generalized linear model with Poisson-gamma distributed errors. The ribbons denote the 95% confidence interval of the model estimate. The colour gradient blue to red represents north to south.

the enormous geographical area studied.

### 3.4. PFAS composition and source elucidation

The Norwegian Environmental Agency (NEA) has established a scale of five environmental quality classes for a number of pollutants, ranging from class I (“background”) to class V (“acute toxic effect”) and based in part on European environmental quality standards (EQS) for those compounds for which they are established (NEA, 2016). While class I is not established for pollutants such as PFAS which do not occur naturally, the upper limit of class II (“good condition”) for PFOS is established at 0.23 µg/kg d.w. and for PFOA it is much higher due to lower toxicity, at 71 µg/kg d.w. This limit normally corresponds to EQS annual average values for those compounds where those values exist, but for PFOS and PFOA EQS are not available. Out of 95 studied locations, 46 had PFOS values above this limit, corresponding to class III (“moderate contamination”), but were all far below class III upper limit, 72 µg/kg d.w. The concentrations of PFOA were far below class II upper limit in all the studied samples.

PFOS and PFOA dominate sample composition in all areas (Fig. 3), up to 66% PFOS and 100% PFOA at separate locations, PFNA being the third and PFUnDA the fourth most abundant PFAS compound in the samples. PFOA was found in the largest number of samples, all 86 in which any PFAS above the sample LOQ were found, while PFOS was found in 76 samples, PFNA was found in 75 samples and PFUnDA in 47 samples (see Table 1). The remaining compounds found above LOQ in this study, PFHxA, PFHpA, PFDA, PFDoDA and PFTrA, made up only a minor part of the total PFAS composition and were found at less than half of the locations, PFDoDA and PFTrA being found above LOQ at a single location in the vicinity of the city of Stavanger. Same as for Σ9PFAS, no significant differences between geographical areas were

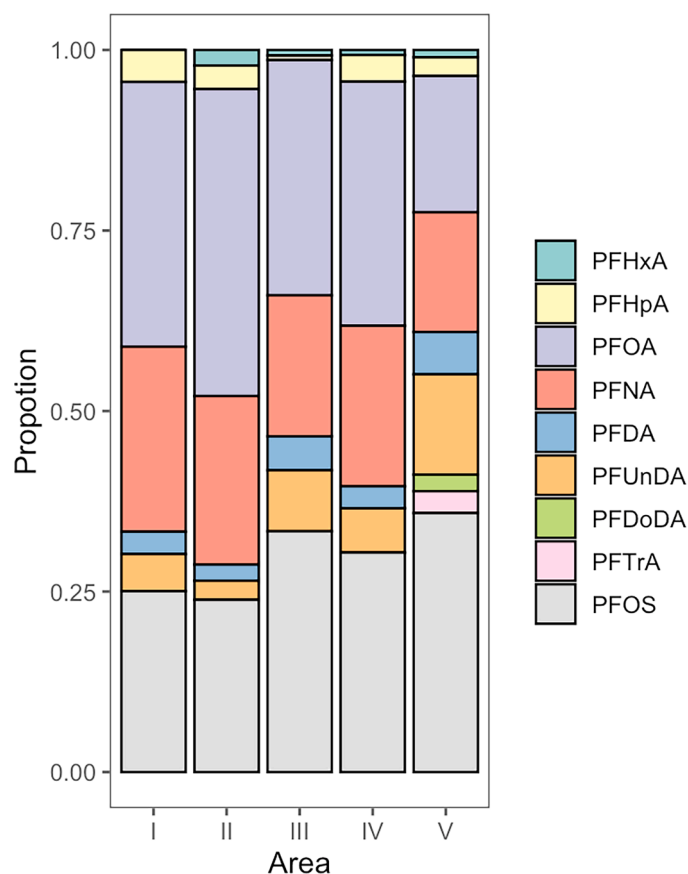


Fig. 3. PFAS composition of surface sediments from Norwegian marine areas, average concentrations by area.

found for separate compounds, except for PFOS and PFUnDA, which were significantly higher in area V (South) only (see Figures S3 and S4). This may be due to most of the sampling locations in this area being in the fjords and close to densely populated and industrial areas around the city of Stavanger. Earlier studies have shown that PFOS is strongly correlated to emissions from wastewater treatment plants, whereas PFOA has a weaker correlation with such sources (Pistocchi and Loos, 2009). However, there was a limited number of samples in this area (six) and the levels were not substantially higher than elsewhere. Based on that, our data seems to allow including Area V in our conclusions on the general PFAS background in the northeastern Atlantic, although this area may be at the borderline with the more exposed areas further south in Europe.

Unlike the PFOA, which was produced both by telomerization process, yielding pure linear isomers, and by electrochemical fluorination (ECF), which yields a complex mixture of linear and branched isomers with known percentages ( $70 \pm 1.1\%$  and  $30 \pm 0.8\%$ , respectively), PFOS and its precursors were exclusively produced by ECF (Jiang et al., 2015). Comparing the profile of PFOS isomers found in the sediments with ECF isomer products could be used to differentiate between the original ECF isomer mixture and PFOS resulting from the degradation of precursors, which usually results in higher branched isomers (Benskin et al., 2009) than PFOS produced by ECF. However, this is complicated by differences in the sedimentation and bioaccumulation between branched and linear isomers of PFOS due to their different physicochemical properties (Houde et al., 2008; Benskin et al., 2009). While isomer data on other PFAS is missing due to low concentrations of branched isomers, this data is available for PFOS. At 76 out of 95 locations where PFOS was found above LOQ, linear PFOS constituted on average 96% of total PFOS, varying between 85% and 100% of total PFOS at different locations. This agrees with earlier data on linear PFOS sorbing preferentially to sediments while branched PFOS being more likely to remain in the water phase (Schulz et al., 2020).

The long chain PFDA measured in these samples have a limited known commercial production (Prevedouros et al., 2006). Therefore, its widespread detection in these regions might indicate diffuse contamination through precursor degradation such as fluorotelomer alcohols and polyfluorinated sulfonamides-based chemicals (Martin et al., 2002; Young et al., 2007). Neutral PFAS precursors may be transported over long distances through the atmosphere and have the potential to degrade photochemically in the atmosphere to form PFDA (Ellis et al., 2004), which subsequently deposit to the ocean surface (Pickard et al., 2018). Therefore, degradation of these precursors contributes to the load of PFDA in the marine environment (Butt et al., 2010). Further, long-range transport through sea aerosol should not be ignored (Wong et al., 2018). Please note that known precursors investigated in these sediments, namely perfluorooctanesulfonamide (PFOSA) and three fluorotelomer sulfonates (4:2 FTS, 6:2 FTS and 8:2 FTS) were not detected.

The concentration ratio of PFOA (C8) to PFNA (C9) in an ice core from the Arctic which was assumed to receive input solely from the atmospheric degradation of precursors was found to be  $1.5 \pm 0.8$  (Young et al., 2007). Similarly, molar ratios of even-to-odd perfluoroalkyl carboxylic acid pairs (e.g., C8: C9) were typically less than 2 and greater than 0.5 in remote Arctic ice cores (MacInnis et al., 2017). Such ratios were not applied to sediments before. However, similar PFOA: PFNA ratios were found in the current study where regions I-IV showed average ratios of 1.6 or greater, and a slightly lower ratio was found in area V (1.0, see Fig. 4). This might indicate that regions I-IV are impacted by diffuse sources through long range transport of precursors, while area V (South) may receive a mixture of diffuse and direct local contamination, as discussed above. This relation was not observed for PFDA: PFUnDA.

PCA along two components explained 69.5% of the variation, see Fig. 5. It is apparent that the long-chain PFAS, C10, C11, as well as PFOS, correlate better with mud % and TOC than the relatively shorter ones (C6-C9). Several Stavanger locations are outliers, and one Arctic Ocean

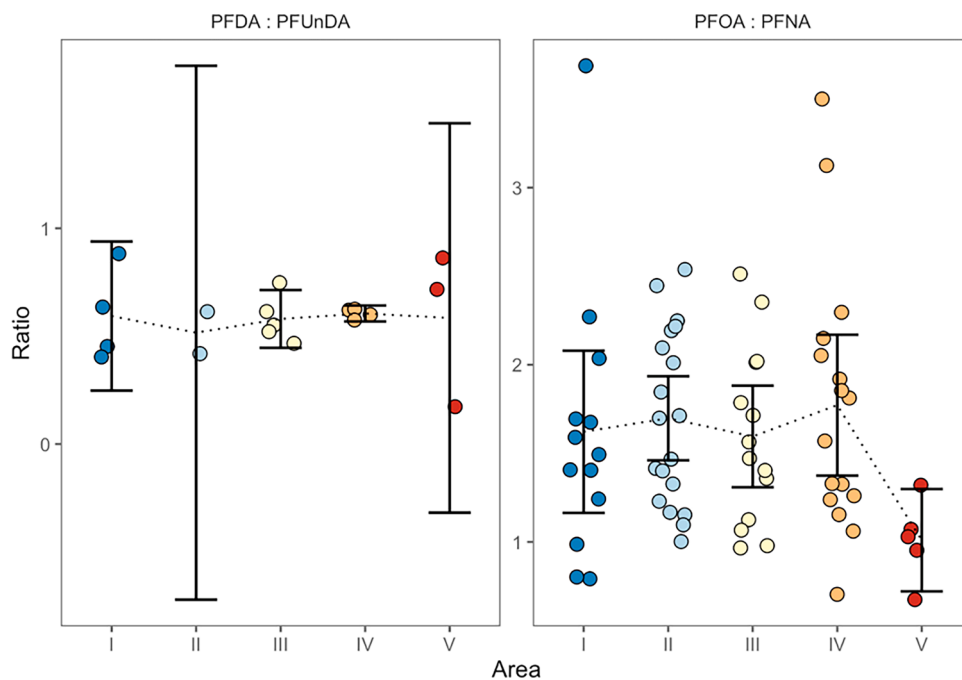


Fig. 4. PFDA:PFUnDA and PFOA:PFNA concentration ratios in the five areas of the North-east Atlantic, error bars denoting the 95% CI, assuming Gaussian errors.

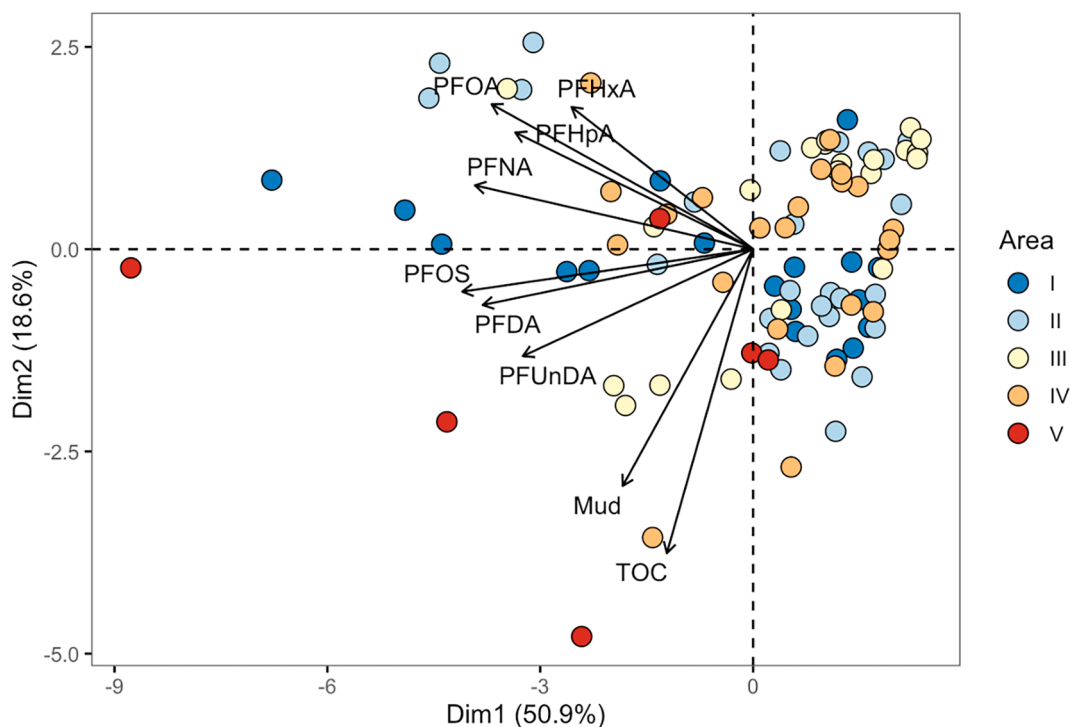


Fig. 5. PCA biplot of PFAS concentrations ( $\mu\text{g}/\text{kg}$  dry weight) and sediment characteristics (TOC %, Mud %) at 95 locations in the North-eastern Atlantic.

location. In Stavanger area, one location is strongly separated from the rest due to having the highest TOC value of 5.5%, but the levels of PFAS are not the highest here. The highest levels of PFAS found in Stavanger fjord area are from the deepest basins (down to 702 m depth and up to 97% mud content), relatively distant from the city of Stavanger.

4. Conclusions

Low-to-moderate levels of several PFAS (predominantly PFOS and

PFOA) are found in surface sediments from Norwegian marine areas unexposed to local sources, pointing to long-range transport as the main origin of this type of pollution. No statistically significant differences are found in total amounts of PFAS throughout the studied area from the Skagerrak in the south to the Arctic Ocean north of Svalbard in the north, if one corrects for sediment characteristics. Somewhat higher levels of two separate compounds, PFOS and PFUnDA, found in the south, may be due to local sources in the southern fjord areas, but do not affect the variations in the total amounts of PFAS significantly. The

extensiveness of the overall studied area suggests that PFAS are evenly supplied to and distributed in the sediments throughout the eastern North Atlantic, forming a non-natural “PFAS background” in areas without significant local sources.

### CRedit authorship contribution statement

**Stepan Boitsov:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Are Bruvold:** Writing – review & editing, Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Linda Hanssen:** Writing – review & editing, Writing – original draft, Validation, Methodology, Formal analysis, Data curation. **Henning K.B. Jensen:** Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation. **Aasim Ali:** Writing – review & editing, Writing – original draft, Validation, Investigation, Conceptualization.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Data availability

I have shared the link to my data at the attach file step. [The Marine Chemistry database for Norwegian waters \(Original data\)](#) (MarChem - The Marine Chemistry database for Norwegian waters).

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### Supplementary materials

Supplementary material associated with this article can be found, in the online version, at [doi:10.1016/j.envadv.2024.100545](https://doi.org/10.1016/j.envadv.2024.100545).

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