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Dissolved organic matter in Arctic multi-year sea ice during winter: major components and relationship to ice characteristics

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Abstract Ice cores were collected between 10.03.93 and 15.03.93 along a 200 m profile on a large ice floe in Fram Strait. The ice was typical of Arctic multi-year ice, having a mean thickness along the profile of 2.56 ± 0.53 m. It consisted mostly of columnar ice (83%) grown through congelation of seawater at the ice bottom, and the salinity profiles were characterized by a linear increase from 0 psu at the top to values ranging between 3 and 5 psu at depth. Distributions of dissolved organic carbon (DOC) and nitrogen (DON) and major nutrients were compared with ice texture, salinity and chlorophyll *a*. DOC, DON, dissolved inorganic nitrogen (DIN), NH_4^+ and NO_2^- were present in concentrations in excess of that predicted by dilution curves derived from Arctic surface water values. Only NO_3^- was depleted, although not exhausted. High DOC and DON values in conjunction with high NH_4^+ levels indicated that a significant proportion of the dissolved organic matter (DOM) was a result of decomposition/grazing of ice algae and/or detritus. The combination of high NH_4^+ and NO_2^- points to regeneration of nitrogen compounds. There was no significant correlation between DOC and Chl *a* in contrast to DON, which had a positively significant correlation with both salinity and Chl *a*, and the distribution of DOM in the cores might best be described as a combination of both physical and biological processes. There was no correlation between DOC and DON suggesting an uncoupling of DOC and DON dynamics in multi year ice.

Introduction

There is currently substantial interest in the role of dissolved organic matter (DOM) and its influence on carbon and nitrogen cycling within the oceans. However, an aspect that has been largely overlooked is the amount and nature of DOM contained within the ice cover of polar seas. During sea ice formation plankton and detrital material are harvested from the upper few metres of the water column and incorporated into the ice matrix (Garrison et al. 1983, 1989). A sea ice community comprising diverse autotrophic and heterotrophic organisms often thrives within the ice, at times reaching high biomasses (Horner et al. 1992; Palmisano and Garrison 1993). Although few investigators have actually measured concentrations of DOM or any of its major components within sea ice, it is generally thought that there is a large pool of DOM within the ice as a result of ice algal exudation, and protozoan and/or metazoan grazing, as well as via the death and degradation of sea ice organisms. In the Arctic the dominant sea ice assemblages are the bottom ice communities, which can be highly productive, and at times even nutrient limited despite high nutrient fluxes (Cota et al. 1991; Smith et al. 1990, and references therein). It is quite plausible that in summer and autumn high quantities of DOM build up within these assemblages, as a result of both high biological activity and confinement. Following freezing in autumn and winter this may be retained (and modified) in the ice over winter, and released on ice melt, resulting in an inoculum of DOM into the surface waters at the time of the spring phytoplankton blooms. The influence of such an inoculum could be highly significant for heterotrophic organisms in the waters close to the melting ice

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surfaces, mostly on a scale of centimetres but possibly up to metres, depending on the DOM content of the mixed layer and the extent of vertical mixing.

Mel'nikov and Pavlov (1978), Apollonio (1980) and Bunch and Harland (1990) reported elevated DOM concentrations in Arctic sea ice. The first work reported only a slight enrichment of dissolved organic carbon (DOC) in various ice types, whereas the latter study measured a mean DOC concentration in the bottom layers of first-year sea ice 4 times greater than that of the underlying seawater (over a 2 year sampling campaign) in the Canadian subarctic. These workers showed that DOC values vary seasonally in conjunction with the development of the bottom ice community, and that there is a tight coupling between bacterial biomass and newly synthesised DOC. Gradinger et al. (1992) discussed the role of DOC production by ice algae in Arctic sea ice and its availability to heterotrophic organisms. They explained a spatial separation between bottom (diatom-dominated) and supra-bottom (heterotrophic organism-dominated) assemblages by a vertical transport of DOC within the ice. However, the scheme proposed by these authors remains speculative, since our knowledge about the DOM content of sea ice is so limited. Yet their study clearly highlighted the need for basic quantification and characterization of the nature of DOM for understanding carbon and nitrogen cycling within sea ice. For this reason we collected ice cores from a multi-year ice floe in Fram Strait, and sampled these in an attempt to correlate the distribution of dissolved organic carbon, nitrogen and major nutrients with ice texture, salinity and chlorophyll *a*.

Material and methods

A drift station was established on a multi-year ice floe in Fram Strait during the winter expedition ARK IX/1a of *RV "Polarstern"* (drifting from 81°27′ N, 7°22′ E on 8.03.93 to 80°30′ N, 3°52′ E on the 19.03.93). Further details about the floe, meteorological conditions and the drift during the sampling period are described by Eicken and Meincke (1994). The field programme consisted of drill-hole thickness measurements along a 200 m long transect, which had a mean ice thickness of 2.56 ± 0.53 m, and an average snow thickness of 0.25 ± 0.27 m. Three ice cores were obtained using a 10 cm diameter corer (fiberglass core barrel with stainless steel cutters) from a part of the profile in which the ice was level, core 7402 at 5 m, core 6901 at 20 m and core 6902 at 50 m from the beginning of the profile. Cores 6901 and 6902 were collected on 10.03.93 and core 7402 on 15.03.93. Between these two sampling dates surface ice temperatures did not rise above − 30°C.

During the coring and subsequent handling, care was taken not to contaminate the ice, and wherever possible plastic gloves were worn. Cores were transferred (in polyethylene bags) to a cold laboratory (< − 25°C) on board ship, where stratigraphic analysis of thick sections (cut with a stainless-steel band saw) under ordinary and polarized light was carried out. Based on textural stratigraphy, portions of the ice (between 5 and 20 cm in length) were melted in the dark at 4°C. Core 7402 was divided with a fine resolution (18 sections) especially towards the bottom of the core. The other two cores (6901 and 6902) were divided on a coarser scale (eight and six sections respectively) although where conspicuous stratigraphic features were present a finer resolution was employed.

The salinities of melted core sections were measured at room temperature with a conductivity salinometer (WTW, Weilheim, Germany), and samples then filtered through glass fibre filters (Whatman GF/C precombusted for 5 h at 450°C). The use of GF/C filters could result in a slight overestimation of DOM because they have a pore size of approximately 1.2 μm. However, previous investigations (Lara unpublished data) have shown that there are no significant differences in dissolved organic nitrogen (DON) measurements performed on surface seawater from the Greenland Sea filtered with GF/C and GF/F filters. The contribution of submicrometre (< 1 μm) particles to DOC has also been estimated to be only approximately 10% (Isao et al. 1990, and references therein).

Filters were retained for later Chl *a* determination and the filtrate divided: 30 ml was stored in precombusted (5 h at 450°C) glass ampoules for later DOC analysis, and 50 ml in alkali-washed polyethylene bottles for DON and nutrient analysis (NO_3^-, NO_2^-, NH_4^+). All filters and filtrates were frozen at − 30°C until analysis in the laboratory at Bremerhaven.

Chl *a* was determined fluorometrically (Evans et al. 1987) following homogenisation of filters and extraction in 90% acetone. DON was analysed following persulphate wet oxidation, and concentrations of nitrate, nitrite and ammonium were measured using standard autoanalyser methods (Kattner and Becker 1991). DOC was measured by high-temperature catalytic oxidation using a Shimadzu 5000 TOC analyser.

Results and discussion

The thickness of the ice floe, the textural stratigraphy and the salinity profiles (Fig. 1) are typical of Arctic multi-year ice (Weeks and Ackley 1986; Eicken unpublished data). Based on analysis of drifting buoys, Colony and Thorndike (1985) concluded that ice in the Fram Strait is between 3 and 4 years old, originating in the central Arctic and the shallow Eurasian shelves. The near linear increase in salinity from the top with depth is almost invariably found in Arctic multi-year ice, as a result of flushing with snow and ice meltwater produced at the ice surface during the ablation season (Untersteiner 1968; Eicken unpublished data). As evident in Fig. 1, there is a pronounced effect of meltwater flushing down to a depth of between 1.0 and 1.5 m, below which salinities are not so affected. This influence of meltwater has been confirmed by stable isotope analysis of these cores and others taken at the same time (Eicken unpublished data).

On average, the cores consisted of 83% columnar ice (core 6901, 96%; core 6902; 57%; core 7402, 95%) grown through congelation of seawater at the ice bottom (Fig. 1). The upper half of core 6902 was composed of sequences of granular and mixed columnar/granular ice, formed through dynamic growth processes and retexturing during previous ablation seasons (for details of ice nomenclature and growth processes, see Weeks and Ackley 1986). Despite differences in ice microstructure, the salinity profiles of the cores were similar (Fig. 1), being characterized by a linear increase in salinity from zero at the top to values ranging between 3 and 5 psu at depth. The

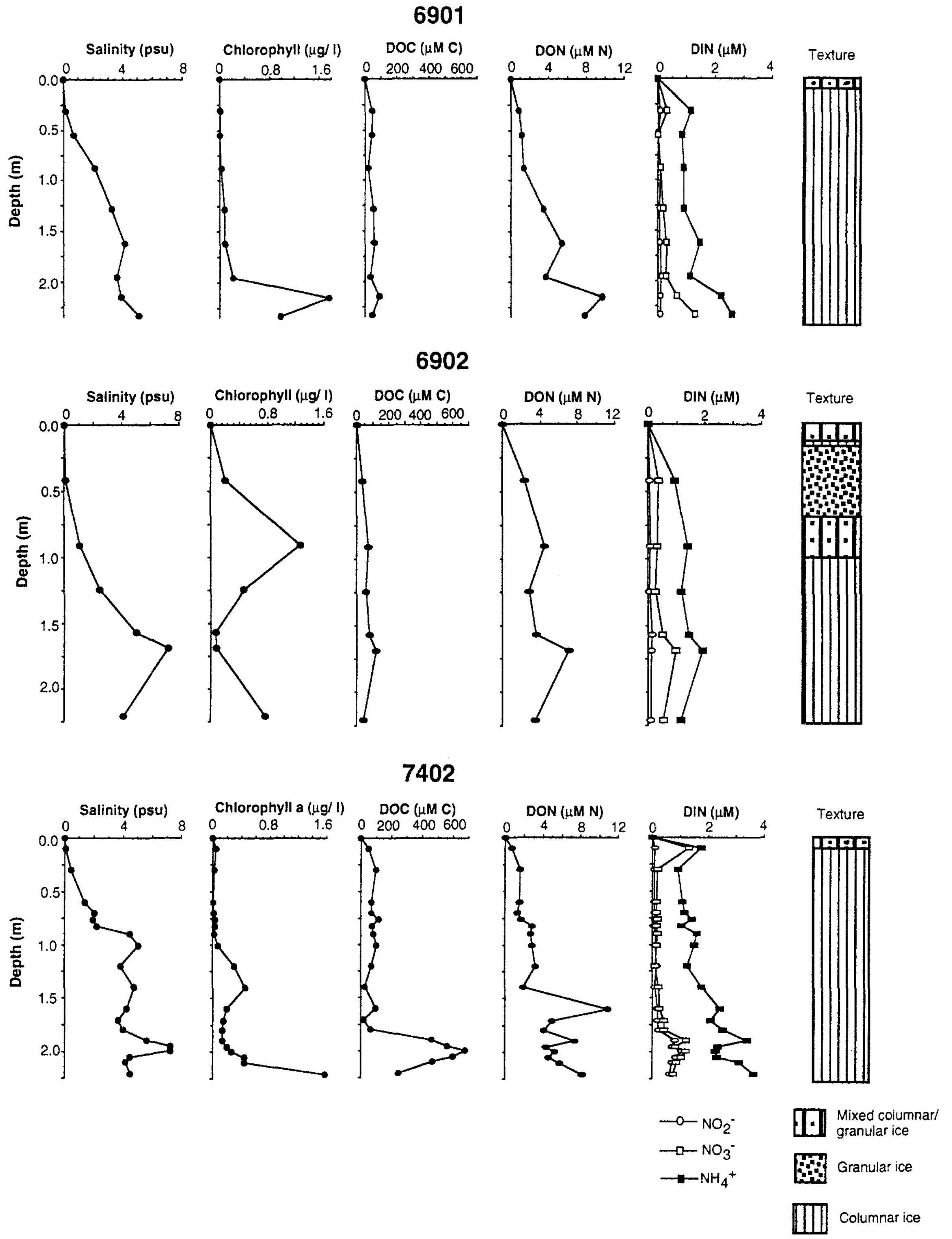

Fig. 1 Depth distribution of salinity, Chl *a*, dissolved organic carbon and nitrogen (DOC and DON respectively) and dissolved inorganic nitrogen (DIN) in three Arctic multi-year ice cores taken approximately 50 m apart on the same ice floe in Fram Strait (core numbers 7402, 6901, 6902). Stratigraphies are based on examination of thick sections

average core salinity was 3.0 psu ice (core 6901, 2.8 psu; core 6902, 2.8 psu; core 7402, 3.3 psu). This similarity indicates that the effects of previous ablation processes do not differ significantly between the coring sites, which is also confirmed by the low variation in thickness of this level ice.

Ice temperatures increased from − 30°C at the surface to − 19°C at 1.0 m and − 2.0°C just above the ice

Table 1 Correlation matrix of the chemical parameters of the combined ice cores (core nos. 7402, 6901, 6902) (with 32 samples, correlation coefficients $\geqslant 0.45$ are significant, $P \leqslant 0.01$)

	DON	DOC	Salinity	Chl *a*	NO_2^-	NO_3^-	NH_4^+
DOC	0.36	–					
Salinity	0.63	0.55	–				
Chl *a*	0.66	0.06	0.15	–			
NO_2^-	0.43	0.96	0.57	0.12	–		
NO_3^-	0.59	0.61	0.48	0.29	0.63	–	
NH_4^+	0.74	0.61	0.58	0.44	0.72	0.72	–
DIN	0.70	0.75	0.60	0.37	0.83	0.86	0.96

bottom. Due to the low temperatures, the volume fractions of liquid brine within the ice were very small and averaged less than 1% (Eicken et al. 1994). Brine loss during collection at temperatures greater than $-25°C$ is a hindrance to the precise study of ice chemistry. However, owing to the low brine volumes and cold ambient temperatures, loss of brine was minimized for this set of samples. The average salinities of these three ice cores were not much higher than those of summer multi-year ice (3 psu vs. 2–2.8 psu for summer ice), and the similarity in salinity profiles may help in interpreting brine loss problems encountered during summer campaigns.

The general trends in the concentrations of Chl *a*, DOC, DON and dissolved inorganic nitrogen DIN ($NO_3^- + NO_2^- + NH_4^+$) are shown in Fig. 1. Concentrations of DIN fall within the very broad range of values reported by various groups working with Arctic sea ice (Gosselin et al. 1985; Maestrini et al. 1986; Meese 1989; Cota et al. 1990, 1991; Smith et al. 1990). The same is true for the low Chl *a* values, which are typical for a winter community. Large numbers of amphipods were observed (using underwater video) to be highly active on the underside of the ice, and moving in the porous lowest layers (Poltermann 1994), presumably grazing on ice algae. The interior Chl *a* peaks in cores 7402 and 6902 are unusual for Arctic ice (Spindler 1994; Horner et al. 1992), internal ice assemblages as commonly described for Antarctic sea ice have not been often recorded (Smith et al. 1990; Palmisano and Garrison 1993). These, however, are probably relics from the summer bottom community of previous years. The fact that the Chl *a* peak occurs in the strata of mixed columnar-granular ice in core 6902 is curious. However, this cannot be attributed to any site-specific ablation effects, since the salinity profile of this core is so similar to those of the other cores, indicating that no anomalous melt processes have taken place at this location.

Core 7402 had a very different DOC loading to those of the other two cores. In the latter DOC levels were low and mostly below 100 µM C throughout the cores, whereas in core 7402 there was a large DOC peak that lay between the two Chl *a* peaks. This peak was enriched about 5 times compared to seawater values, and was significantly greater than that reported previously for Arctic sea ice (op. cit.). The DON profiles were similar in all three cores, gradually increasing with increasing core depth. The maximum DON concentrations in core 6902 were, however, slightly lower than in the other two cores. DIN showed a similar trend to that of DON both within and between cores. In all cores NH_4^+ was the dominant component of DIN, followed by NO_3^-, and then NO_2^-, which remained relatively low except at the bottom of core 7402, where it increased slightly. The high NO_3^- concentrations at the top of cores 6901 and, in particular 7402, appear anomalous. However, they could be derived from the overlying snow, since high NO_3^- contents (> 10 µM N) have been measured in snow samples (fresh and old) collected in Svalbard (Lara unpublished data). High nitrate concentrations have also been found in the uppermost few centimetres of Arctic ice from other regions (Eicken unpublished data).

The variability (in biology and chemistry) between cores taken in close proximity is a further example of sea ice spatial heterogeneity (cf. Eicken et al. 1991), although the stratigraphies and salinity profiles were remarkably similar. Formulation of general trends about the chemical status of sea ice is greatly hampered by the complex nature of sea ice development. This is especially true of Arctic multi-year sea ice, in which the complexity is compounded due to its longevity, and the influence of several seasonal cycles of freezing, melt and deformation. Therefore, in order to separate core specific trends from relationships between variables of more general validity, a correlation matrix between all measured parameters was made firstly for individual cores, and then for pooled data. In the following discussion mainly pooled data are described (Table 1).

As can be seen in Fig. 2, DOC, DON, DIN, NH_4^+ and NO_2^- were all present in concentrations in excess of those predicted by dilution curves derived using estimates for Arctic surface waters (salinity, 33 psu; DOC, 120 µM; DON, 5 µM; DIN, 7.5 µM; NO_3^-, 6 µM; NO_2^-, 0.02 µM; NH_4^+, 0.4 µM). Dilution curves such as these can only be a rough guide to the concentration processes that have occurred, since we do not know the exact concentrations in the water from which the ice was formed. However, they do help in distinguishing between physical and biological processes. Only in the case of NO_3^- were values below the dilution line,

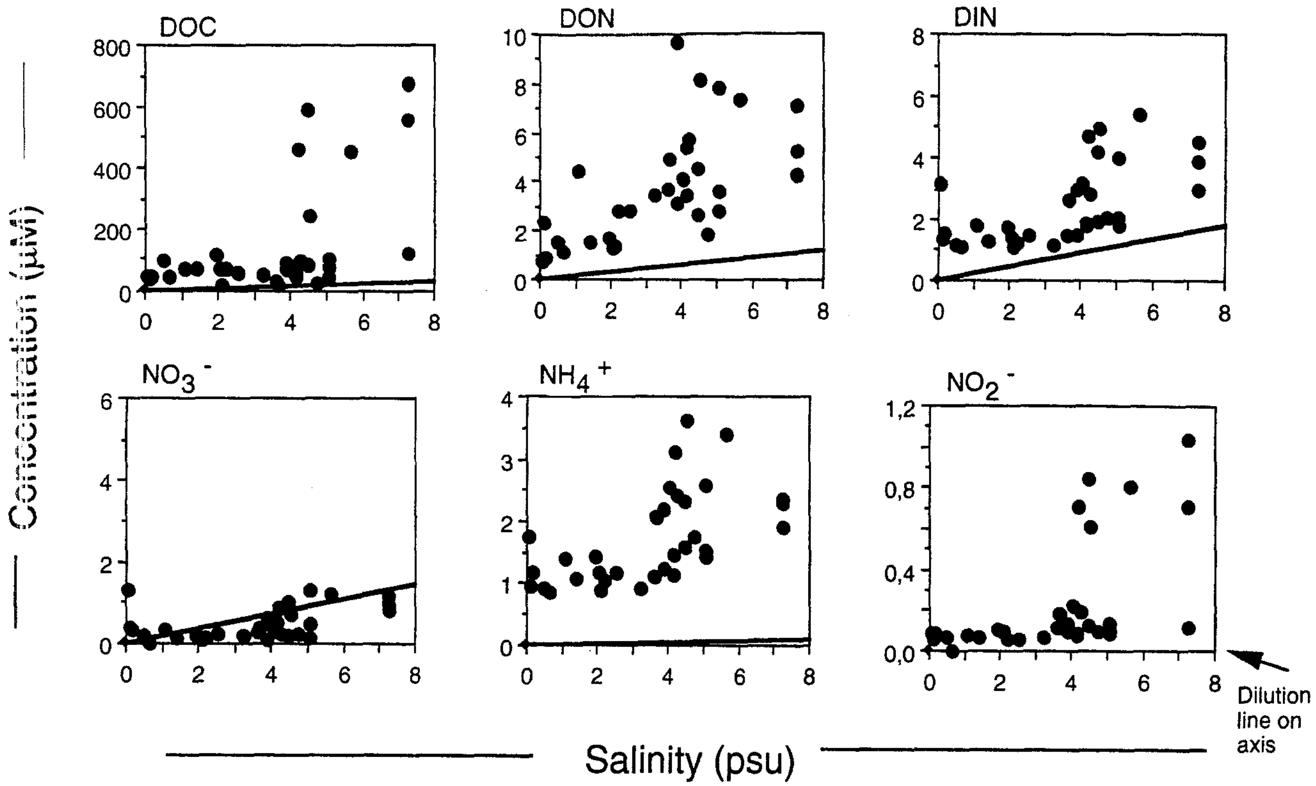

Fig. 2 Salinity vs. the concentration of dissolved organic carbon and nitrogen (DOC and DON respectively), dissolved inorganic nitrogen (DIN) and its components NO_3^-, NH_4^+ and NO_2^-. The data from ice cores 7402, 6901, and 6902 have been pooled. The indicated *dilution line* is extrapolated from estimates of Arctic surface seawater values (see text)

suggesting previous biological uptake, although this depletion was not excessive and only occasionally were values near zero measured. The enrichment of NH_4^+ and NO_2^- was particularly pronounced giving rise to the high DIN values. Such extremely high NH_4^+ values have frequently been measured in sea ice, and although heterotrophic activity is the most likely source, physical processes have also been suggested (Dieckmann et al. 1991, and references therein). The combination of high NH_4^+ and NO_2^- points to the regeneration of nitrogen compounds.

In the case of NO_2^- the pooled data showed a value of $r = 0.96$ for the correlation with DOC (Table 1). However, this was based on a somewhat unfortunate data distribution with only a few values at intermediate NO_2^- concentrations, between 0.2 and 0.6. It is difficult to assess the absolute meaning of such a correlation. However, since NO_2^- is an intermediate form in nitrogen remineralisation and, as observed, showed a significant correlation with salinity, its distribution is probably a result of the decomposition of organic matter and physical concentration processes. The high correlation with DOC suggests that NO_2^- may be a useful indicator for understanding DOM distribution in ice.

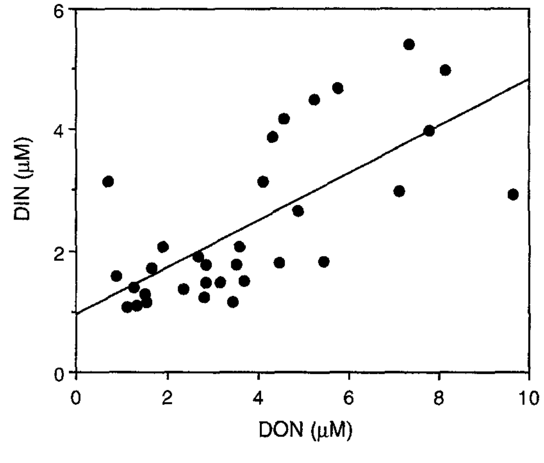

Fig. 3 The relationship between dissolved inorganic nitrogen (DIN) and dissolved organic nitrogen (DON) for the pooled data of ice cores 6901, 6902 and 7402

An interesting feature is the relationship between DON and DIN (Fig. 3). In seawater an inverse correlation between the two is usual, and is indicative of a coupling between inorganic nitrogen uptake and the release of organic nitrogen, or, if remineralisation occurs, the transformation of DON into DIN (Lara et al. 1993; Maita and Yanada 1990). In contrast, in all three cores and in the pooled data, there was a positive relationship between DON and DIN, and of all the DIN species, NH_4^+ showed the highest correlation with

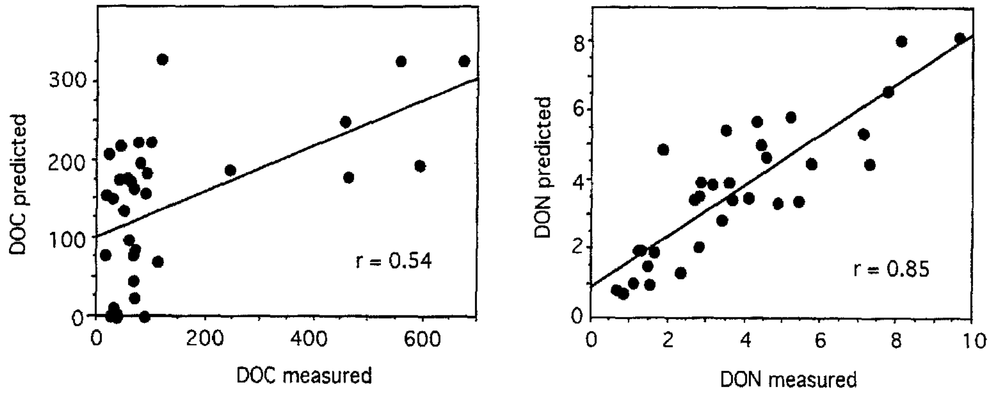

Fig. 4 Measured vs. predicted DON and DOC following multiple regression analysis taking both DOC and DON as dependent variables and Chl *a* and salinity as independent variables

DON (Table 1). Both these facts suggest that DON originates from decomposing material, and that this decomposition has not reached a high oxidation state, which is further supported by the much higher NH_4^+ values compared to those of NO_3^- (Figs. 2, 3). The average concentrations of nitrogen species are DON $> NH_4^+ > NO_3^- > NO_2^-$. If, as discussed previously, NO_3^- is a remnant from seawater, then the ice nutrient status is dominated by reduced nitrogen forms. The immature, perhaps labile, character of a part of this DON is supported by laboratory experiments that have demonstrated the bacterial uptake of the hydrophobic neutral fraction of DON derived from DOM-rich sea ice (Lara unpublished data). Antia and Kähler (1994) found indications that organic carbon in Antarctic sea ice consisted primarily of biologically degradable material.

The bottom community biomass of previous years was probably not extensive, as indicated by the low Chl *a* values, and only slightly depleted NO_3^- values. However, Chl *a* levels could be low as a result of flushing of algae or high grazing pressure, even if they have previously reached very high concentrations. Excretion of DOM by algae, bacteria, protozoa, metazoan grazing and faeces production, and subsequent death lysis and decomposition of organisms resulted in the net accumulation of DOC, DON, NH_4^+ and NO_2^-. The replete DIN concentrations and a positive correlation between Chl *a* and NO_3^- (Table 1) are evidence for the low ice algal activity, since if this had been high, a negative correlation between Chl *a* and NO_3^- would have been expected. The origin of the DOM in the ice is unclear, since it is not known what was incorporated in the surface waters during the ice formation. The DON concentrations suggest a much greater DON production than NO_3^- consumption, which may be an indication that at least some of the DON is derived from the decomposition of particulate organic matter present in the water on ice formation, and not all from the decomposition of new organic matter produced within the ice. However, the high DOC and DON values, in conjunction with high NH_4^+ levels, indicate that a significant proportion of the DOM was a result of decomposition/grazing of ice algae and/or detritus.

There was no significant correlation between DOC and Chl *a*, which is in contrast to DON that had a positively significant correlation with both salinity and Chl *a* (Table 1). In cores 6902 and 7402 DOC coincides with salinity maxima, and not with Chl *a*, suggesting that the distribution of DOM in the cores might be due to a combination of physical and biological processes. Salinity could be used as an indicator of the physical concentration of solutes, and Chl *a* as an indicator of biogenic sources of organic matter and consumption/remineralisation of nutrients. In order to test this possibility we performed multiple regression analysis, taking DOC and DON as dependent variables and Chl *a* and salinity as independent variables (Fig. 4). In the case of DOC this did not produce an improvement in the fit (*r*: 0.54) compared to salinity (*r*: 0.55) or Chl *a* (*r*: 0.06) alone. However, for DON, the multiple regression produced a highly significant correlation (*r*: 0.85), higher than with the individual parameters, salinity (*r*: 0.63) or Chl *a* (*r*: 0.66).

The lack of correlation between DOC and DON (Table 1), and the information from the multiple regression suggest that there are different processes determining the concentrations of DOC and DON in ice. Smith et al. (1992) have discussed different decomposition dynamics for DOC and DON in the ocean. The nitrogen-rich pool of particulate amino acids may be hydrolysed faster than carbon-rich pools such as polysaccharides, since protease activity can be 10–1000 times higher than the glucosidase activity. Similar uncoupling in carbon and nitrogen metabolism could also take place within the ice. This hypothesis is also supported by the calculated DOC/DON ratios. For example, in core 7402, the high DOC values were not accompanied by a proportional increase in DON, resulting in high C/N values (mean 54 ± SD 41), whereas cores 6901 and

6902 had mean values of 19 (± SD 17) and 17 (± SD 4), which are similar to the values of 10–25 determined for bulk seawater DOM (Williams and Druffel 1988). Another possibility for the uncoupling of DOC and DON is a differential adsorption onto the ice matrix. More detailed characterization of the nature of this DOM is needed to clarify this hypothesis.

The thickness, structure and morphology of the ice floe, as well as the salinity and chlorophyll profiles, indicate that this ice was typical of Arctic multi-year sea ice (3–4 years old) originating from the Eurasian shelves. The high correlations obtained with pooled data (all three cores) suggest that multiple regression techniques might be useful tools for understanding the driving forces governing the distribution of organic nitrogenated compounds in ice (cf. Gradinger et al. 1992). However, this does not seem to apply to DOC. Future work will further test the suitability of this approach for enabling general statements to be made about the DOM and nutrient status of sea ice.

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