

## Mercury Fractionation in the Sediments of Kongsfjorden, an Arctic Fjord, Svalbard

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**Abstract** The presence of mercury (Hg) is increasing in Arctic region and the Arctic sea will take short time to reflect changes in atmospheric Hg levels. Hence, monitoring of mercury content in the environmental matrices of Arctic is highly significant. The present study assessed the total mercury (THg) as well as various Hg fractions in the sediments of Kongsfjorden, an Arctic Fjord in Svalbard. The Hg toxicity mainly depends on the form which occurs. Fractionation study will help to understand the different forms of Hg in sediment samples. The mean concentration of THg in sediment was 485 ng/gm dry weight. The results of fractionation indicated the mobility of Hg in the Kongsfjord sediments. The highest percentage of Hg was in fourth fraction (F4) followed by the fractions F2, F1, and F3 and final fraction (F5). The high Hg concentration in the initial fractions indicated the availability of Hg for chemical and biological transformations and transport in the Fjord. Hence potential toxic effects are possible in the system.

**Keywords** Pollution; Metal; Bioaccumulation; Sediment; Bioavailability

## Introduction

Mercury is considered as a global contaminant due to its persistence, transboundary nature and high bioaccumulation potential (Canário et al., 2007). Mercury in the environment has redistributed in air, water, snow, soil and sediment after the industrial revolution. Hence mercury contamination has been increasing in various ecosystems including polar ecosystems (Dietz et al., 2009; Rig á et al., 2011; S øndergaard, 2012). Arctic is considered as one of the pristine environment in the world; however climate change adversely affects its physical characteristics. In recent years, arctic become more vulnerable to the climatic changes which can influences the fate and transport of global environmental pollutants especially mercury (Rig á et al., 2011; S øndergaard, 2012).

Earlier studies indicated that mercury concentration is rising in the Arctic environment along with other heavy metals (Lamborg et al., 2002; Sprovieri et al., 2005; Xie et al., 2008; Liu et al., 2015). The presence of mercury was also noticed even in the fresh water lake sediments of Arctic (Jiang et al., 2011). The concentration of mercury is very high in the fish (Pacyna and Keeler, 1995; Pirrone et al., 1996) and thus in arctic fox and polar bears (AMAP, 1997; 1998).

The rise in temperature causes increased deposition of mercury in Arctic due to high rate of bromine emitted from refreezing (Ariya et al., 2004). Besides this, several factors like sunlight, presence of other halogens and ozone influence mercury deposition. Maximum deposition of mercury has been observed during spring time atmospheric mercury depletion events (AMDE) (Lindberg et al., 2002; Steffen et al., 2008).

According to AMAP (2005), no point source of mercury was present in Arctic and the deposited mercury might have transported from natural and anthropogenic sources outside the Arctic region. It is estimated that the global mercury emission was 1920 tonnes in the year 2005 (AMAP, 2011). Approximately 100 tonnes of mercury were deposited through atmosphere per year in the Arctic Ocean. Glacier melting, snow melting, water regeneration and primary production affects the distribution of mercury in the Arctic marine environment (Kirk and St.Louis, 2009; Dommergue et al., 2010; Stern et al., 2012).

Fjords can be considered as one of the most important ecosystems which can indicate the changes in Arctic environment. As the glacial melting and permafrost thawing increases, transboundary and highly persistent pollutants are deposited into the sedimentary environment of these fjords. The presence of mercury in the Arctic marine environment including fjords was already revealed (Gobeil et al., 1999; Sprovieri et al., 2005; Lu et al., 2012; Liu et al., 2015). The terrestrially deposited mercury as well as historic mercury from the ice reservoir and permafrost may finally reaches Fjords through streams/rivers (Amyot et al., 2009). Fjords are important link between the terrestrial environment and Arctic Ocean and most of the released mercury may retain by the fjords (Faganelli et al., 2003; Ruelas-Inzunza et al., 2009).

The environmental mobility and toxicological effects of mercury are high for organomercury species compared with inorganic mercury forms in aquatic ecosystems (Lacerda and Fitzgerald, 2001). Besides the physico-chemical conditions like pH, temperature, organic carbon etc., the formation of organic mercury are influenced by the availability of inorganic mercury which in turn depends on the strength of bonding between mercury and other elements (Hlodak et al., 2015). Hence fractionation and partitioning of mercury, become significant as the total mercury concentration alone does not help in understanding the risk to organisms (Fytianos and Lourantou, 2004; Rao et al., 2008; Sarica and Türker, 2012; Ramasamy et al., 2012; Frohne and Rinklebe, 2013). Selective extraction can therefore differentiate mercury compounds into fractions such as (a) water soluble (F1), (b) 'human stomach acid' soluble (F2), (c) organo-chelated (F3), (d) elemental Hg (F4), and (e) mercuric sulphide (F5) (Bloom et al., 2003; Kot, 2004; Boszke et al., 2006; Boszke et al., 2008). The present study assessed the bioavailability of mercury by fractionation and distribution in the sediments in an Arctic fjord.

## 1 Materials and Methods

### 1.1 Study area

Kongsfjorden, an Arctic fjord in the North West coast of Spitsbergen in the Svalbard archipelago was selected for the study. It is an established reference site for Arctic marine studies. The hydrography of Kongsfjorden is influenced by Atlantic/Arctic waters and is an important feeding ground for marine mammals and seabirds. The research activities are high in Kongsfjorden and have received a lot of research attention in the recent past as it is a suitable site for exploring the impacts of possible climate changes, with the Atlantic water influx and melting of tidal glaciers both linked to climate variability.

### 1.2 Sample collection and preservation

32 Sediment samples were collected from the Kongsfjorden using grab sampler (Figure 1). The sediments were stored in pre cleaned glass bottles at -20°C in the marine lab, Ny-Ålesund and then transferred to India by cold shipment for further analysis.



Figure 1 Sampling locations

### 1.3 Sample analysis

The total mercury in the sediment was detected by Cold Vapour Atomic Fluorescence Spectrophotometer (CVAFS, Brooks Rand, USA) following bromine monochloride (BrCl) oxidation (USEPA- method 1631, revision E, 2002). The sediment samples were oven dried at 35°C and are ground to fine powder using an agate mortar. Then it is sieved for <63 µm granulometric fraction with the help of ASTM standard sieves and digested with Suprapur Nitric acid before BrCl oxidation. Utmost care has been taken during the sieving. Among the 33 sediment samples, 14 samples were undergone fractionation studies followed by Bloom's five step sequential extraction scheme (Bloom et al., 2003). The various fractions were (a) water soluble (F1), (b) 'human stomach acid' soluble (F2), (c) organo-chelated (F3), (d) elemental Hg (F4), and (e) mercuric sulphide (F5) (Bloom et al., 2003; Boszke et al., 2003). The sequential extraction scheme was already explained in our earlier paper (Ramasamy et al., 2012) (Table 1). The mercury fractions F1 and F2 are weakly bound to sediments compared to other fractions. F3 fraction is the mercury bound to organic matter especially humic acids, fluvic acids mainly through sulphur bonding. Elemental form of mercury (F4) is mainly coming from the anthropogenic activities like gold mining. F5 fraction is the residual mercury and is not easily available for chemical or biological transformations. Fraction-1 was obtained by shaking with ultrapure water. The residue after centrifugation was subjected to shaking with 0.1 M CH<sub>3</sub>COOH + 0.01M HCl for the second fraction. Centrifuged and the residue was extracted for third fraction with 1 M KOH. The elemental mercury fraction (F4) was extracted with 12 M HCl. The final residue was treated with Conc. HCl and HNO<sub>3</sub> for the last fraction. All the extractants were made up with 0.2 M bromine monochloride.

Table 1 Bloom's (2003) five step sequential extraction procedure (Ramasamy et al., 2012)

	Fraction of Hg	Chemical Extraction method
Step 1	Water –soluble	1g of sediment was mixed with 25 mL of ultra pure water in a centrifuging tube and shaken for 18±2 hour in an end-over-end shaker at 30 rpm. Separate the extract from the solid residue by centrifugation at 3,000 rpm for 20 min. Centrifuged extracts were then filtered and 1 mL of 0.2 M BrCl was added. As a rinse step, the extraction vials containing the sediment residue were refilled with 20 mL of the same extractant, shaken vigorously to resuspend the sediment, re-centrifuged and filtered. The rinse was then added to the extract from the same sample and the combined sample diluted to 100 mL with ultrapure water.
Step 2	Human stomach acid soluble	To Step 1-residue add 25 mL 0.1 M CH <sub>3</sub> COOH + 0.01M HCl (pH= 2). Shake for 18±2h. Centrifuge extract as per Step 1. Again rinse with 20 mL of the same extractant. The detailed operation conditions were the same as those of step 1.
Step 3	Organo-chelated	The residue from the Step 2 was added with 25 mL of 1M KOH and centrifuged. Because the solution (extract) has high acid neutralizing capacity, 10 mL of 0.2 M BrCl was added. The detailed operation conditions were the same as those of step 1.
Step 4	Elemental mercury	The Step 3 residue was extracted with 25 mL of 12 M HNO <sub>3</sub> solution and then rinsed with another 20 mL HNO <sub>3</sub> solution. The detailed operation conditions were the same as those of step 1. But no filtration step was employed since the solution can destroy the filter paper.
Step 5	Mercuric sulfide	10 mL of conc. HCl was added to the sediment residue remaining in the vial. After swirling the sample to dislodge the sediment, 3 mL of conc. HNO <sub>3</sub> was added. Then the vials were loosely capped and kept in room temperature for 12 hours. Separate the extract from the solid residue by centrifugation at 3,000 rpm for 20 min and the final volume was made up to 50 ml using ultrapure water.

### 1.4 Quality assurance

High quality acids and ultrapure water was used for the analysis. Blank was determined for every run. Precision was checked in between the runs with different concentration of mercury. The certified reference material 'Estuarine sediment' (ERMCC 580) was used for validating the total mercury determination. The percentage of recovery was 97.07 ±0.21%. The method detection limit was 5.6 ng/L.

## 1.5 Geochemical Parameters

Oven dried (80 °C) and powdered sediment samples were analysed for pH, total nitrogen (N) and organic carbon (OC) (Maiti, 2003). Fe was detected with ICP-MS (Thermo).

## 2 Results and Discussion

### 2.1 THg content in the Fjord

The mean concentration of THg in the sediment was 485 ng/g dry weight (Table 2). The maximum concentration was observed at the site 7 (1850 ng/g). The result of sediment mercury was discussed with respect to different zones such as outer, middle, transitional and inner (Figure 2). Sediment from the middle zone showed higher concentration of mercury followed by outer zone. The lowest content was noticed for transitional and inner zone which is more influenced by the fresh water from surging glaciers. The middle and outer zone received glacial water through small rivers which were fed with various glaciers, glaciated terrains, permafrost etc. and were carrying high load of suspended matter. Lu et al. (2012) suggested that the high concentration may also be due to the supply of mercury rich sediments from low and middle latitude to the Arctic by the West Spitsbergen Currents. The mean concentration of THg in the sediments of Kongsfjorden was higher than that in any other Arctic region (Table 3). The pattern of mercury contamination was same in the fjord and the sediment mercury content observed during the year 2009 was 8-65 ng/g (Lu et al., 2012). Compared with the results of Lu et al. (2012), the Hg concentration is higher in the present study. This might be due to the fine sediment fraction used in this study. Unlike earlier study (Lu et al., 2012), the present study has shown high mercury content in the inner zone of Kongsfjorden which is close to the port and glaciers. This can be owed to two things, one is the various activities occurred in and around the the Ny-Alesund port and the other one is the deposition due to increased melting. This can be an indicator for the influence of other sources like atmospheric mercury depletion events, glacial melting etc. than the oceanic transportation.

Table 2 Hg concentration in the Kongsfjorden

Sample No.	Hg (ng/g)
1	101
2	178
3	1250
4	130
5	950
6	86
7	1850
8	107
9	125
10	034
11	1530
12	177
13	259
14	14
Mean	485

### 2.2 Mercury fractionation in the Kongsfjorden sediment

The percentage of various fractions of mercury obtained from the sequential extraction is given in the Figure 3. The water soluble Hg fraction (F1) of Kongsfjorden sediments varied from 6.65% to 41.47% with a mean of 15.74%. The F2 fraction varied from 6.91% to 49.47% with a mean of 20.47%. The organic matter bound mercury in the sediments of Kongsfjorden was varied from 4.43% - 84.72% where as the elemental mercury fraction (F4) showed a high range of variation (2.4% to 93.74%). The mean contribution was 22.09% and 29.31% respectively for F3 and F4 respectively. The fifth fraction, sulphide-bound mercury, was varied from 0.82% - 59.89%. The mean value noticed for F5 was 12.38%. The average distribution of mercury in different chemical fractions followed the order F4>F2>F1>F3>F5 (Figure 4).

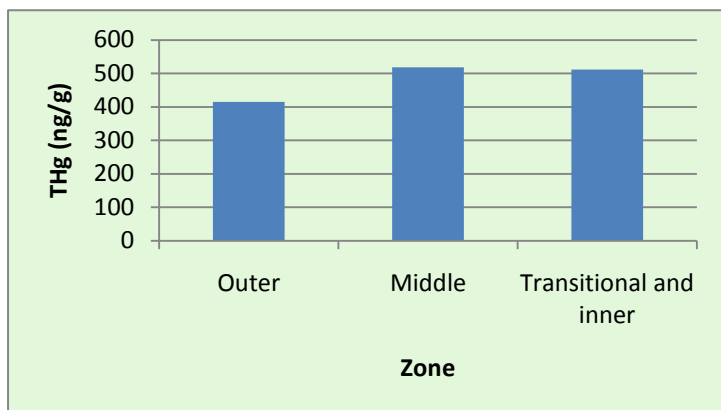


Figure 2 Mean mercury content in the sediments of different zones

Table 3 Comparison of THg content in Kongsfjorden with different regions of Arctic

THg (ng/g dry wt.)	Site	Reference
485 (14-185)	Kongsfjorden, NyAlesund	Present study
2-250	Greenland	Lindeberg et al., 2006
10 - 159	Canada	Lockhart et al.,
20-100	Sweden	Rydberg et al.,
8	Arctic	Muir et al.,
13	Sub-Arctic	Muir et al.,
21-48	NyAlesund	Jiang et al., 2011
9 - 86	Kongsfjorden, NyAlesund	Liu et al., 2015
22	Kongsfjorden, NyAlesund	Lu et al., 2012
6	Alaska	AMAP, 2002
2	East Siberia Sea	Presley, 1997
28	Kara Sea	Esnough, 1996

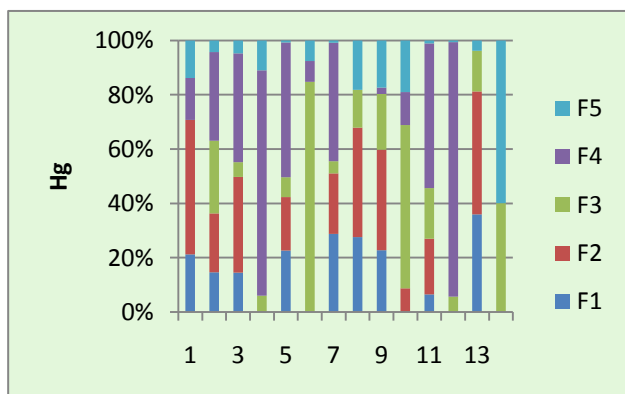


Figure 3 The percentage of mercury in various fractions of Kongsfjorden sediments

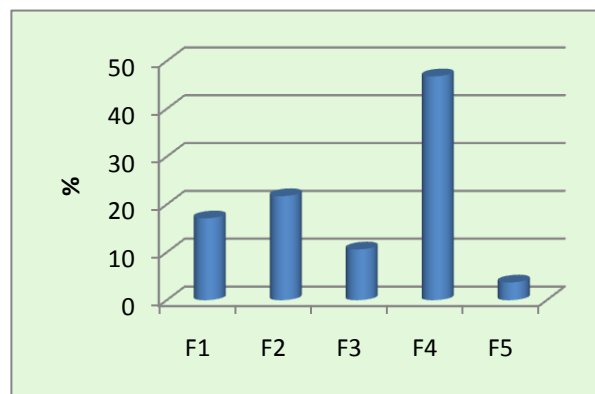


Figure 4 Mean percentage of mercury in fractions

The percentage of elemental mercury was high when compared with other fractions. The high content of elemental form of mercury has indicated that the sediment was contaminated (Lechler et al., 1997; Pestana et al., 2000; Ramsamy et al., 2012). This form of mercury is highly persistent and it remains available for chemical and biological transformations to highly toxic organic forms (Biester et al., 2002; Wang et al., 2004).

The fraction-1 and 2 are easily available as these fractions are weakly bound to sediments. The former one is bound to dissolved organic matter without a Hg-C bond or suspended matter in pore waters whereas the later one is bound to iron monosulfides, iron and manganese hydroxides and carbonates (Biester and Scholz, 1997; Wallaschlager et al., 1998; Renneberg and Dudas, 2001; Kot, 2004). Even though the F1 is water soluble form, it may not be in form of water soluble ionic species. The F2 is easily susceptible to pH variations and changes in

environmental conditions could cause release of loosely bound mercury from the iron monosulfides, carbonates of iron and manganese (Lechler et al., 1997; Filgueiras et al., 2002; Bloom et al., 2003). Hence F1 and F2 are more mobile and bioavailable than any other forms and can be transported by natural processes later made available for methylation (Ullrich et al., 2001; Boszke et al., 2003). Studies on mercury fractionation was meagre in polar ecosystems, hence comparison is very difficult. High percentage (7.8%) of F2 was noticed from mining areas (Miller et al., 1995) whereas the mean value of the present study was high (20.47%). The mean value of F1 was also comparably higher than the reported values (Kot and Matyushkina, 2003; Ramasamy et al., 2012).

Even though the total concentration was low, the bioavailable mercury bound to first and second fraction was slightly high in the system. The cumulative percentage of these two fractions of Hg in the sediments was plotted (Figure 5) and assessed based on the different risk assessment code (RAC) classes (Ramasamy et al., 2012; Navya et al., 2015). Among the samples analysed, four samples (26% of the samples) are in very high risk (>50%) and more than 52% of the samples are under high to very high risk class. This indicated the potential availability of mercury for the biota as well as for the chemical and biological transformations like methylation in the system.

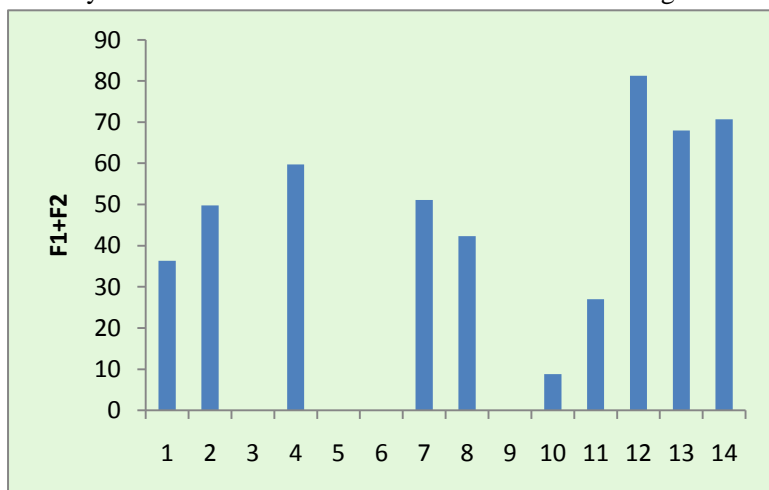


Figure 5 Percentage of first two easily available fractions of mercury in the sediments

Organic chelated mercury (F3) fraction was the second smallest among the five fractions. This fraction is mainly influenced by the presence of organic matter. The mercury bound to organic ligands like humic and fulvic acids, amino acids etc. through reduced sulphur species (Xia et al., 1999). Oxygen and nitrogen atoms can also act as binding sites for mercury (Hesterberg et al., 2001). Apart from this a very small amount of mercury associated with living and dead biota and methyl mercury can also found in this fraction (Bloom et al., 2003; Ramasamy et al., 2012; Navya et al., 2015). The methylation potential is high for F3 compared with F4 and F5 (Frohne et al., 2012). The sediments of Kongsfjorden have low F3 content and such transformations may comparatively less. However at the same time bioavailable fractions are high.

Mercury bound to sulphide (F5) fraction is normally not available for methylation. The F5 fractions showed a very low concentration in Kongsfjorden sediments. However, if conditions become aerobic ionic mercury could be released and which in turn can undergo methylation (Ullrich et al., 2001; Boszke et al., 2003). The reducing conditions favours the mercury bound to sulphide fraction in aquatic sediments (Lechler et al., 1997; Beldowski and Pempkowiak, 2003). This fraction generally represents the residual Hg and the presence of Hg sulphide in sediments is normally related to natural occurrences of the metal (Lechler et al., 1997). Hence it is evident that the mercury present in the sediments was of anthropogenic origin.

The fractionation results showed that more mobile fractions are there in the outer fjord, however a few samples from inner fjord also showed the presence of mobile fractions. This might be due to the deposition of mercury through melted water from glaciers. Grain size may also play a vital role in the adsorption of mercury as it is evident from the sites 12 (very near to glacier) and 14 (very near to the stream mouth) where coarse sediments

present. The locations 7, 8, 9 and 13 are away from these points and having fine grained sediments which can adsorb more mobile mercury and it was confirmed by the high concentration observed at site 7.

The various fractions indicated that the source of mercury in Kongsfjorden system was anthropogenic and very recent in nature. It is substantiated by the presence of high Hg content in the bioavailable fractions (F1 and F2). Based on the potential to induce toxic effects in marine organism, the effects range low (ERL) and effects range median (ERM) for THg are 150 and 710 ng/g respectively (Long et al., 1995). In the present study, the mean concentration (228.5 ng/g) of Hg in the bio accessible fractions (F1 to F3) was between the ERL–ERM, therefore biotic effects are expected and the mercury concentration in the sediments of Kongsfjorden is not negligible.

The minimum, maximum and mean values of geochemical parameters were given in Table 4. The sediment was slightly alkaline and the mean OC was 1.2%. The mean concentration of Fe was 28785.58 mg/kg.

Table 4 Mean values of geochemical parameters

	Min	Max	Mean
Fe (mg/kg)	23120.18	38351.36	28785.58
pH	7.21	8.13	7.77
OC (%)	0.36	2.17	1.2
N (mg/kg)	0.014	0.056	0.029

The correlation analysis showed that different fractions were positively correlated each other except between F3 and F5 (Table 5). F5 has not showed any significant correlation with other fractions. F1, F2 and F3 have a significant positive relation with F4 ( $p=.01$ ) indicating common source of origin. These results indicate a significant influence of elemental mercury on the bioavailable fractions. There was no significant correlation observed between OC and various mercury fractions including F3 fraction. This result can be interpreted in two ways; 1) the low mercury concentration nullify the significance of organic carbon, and 2) the mercury entering into the system is not forming any bond with organic ligands and mainly prefer to be in F1, F2 and F4 fraction.

Table 5 Correlation between geochemical parameters and various mercury fractions

	F1	F2	F3	F4	F5	Fe	pH	OC	N
F1	1								
F2	.841**	1							
F3	.242	.549	1						
F4	.809**	.924**	.637*	1					
F5	.135	.520	-.059	.067	1				
Fe	.031	.506	.175	.386	.498	1			
pH	-.150	-.602	-.267	-.273	-.344	-.614*	1		
OC	.016	.048	-.219	-.021	.041	.057	.192	1	
N	-.294	-.435	-.403	-.314	-.083	-.193	.560*	.717**	1

Note: \*\*: Correlation is significant at the 0.01 level (2-tailed); \*: Correlation is significant at the 0.05 level (2-tailed)

The insignificant correlation of F3 with carbon indicates that the mercury present in this fraction is not directly linked to the permafrost thawing. The thawing of permafrost is a major source of carbon in the form of humic and fulvic acids along with other elements, hence there should be relation with F3 fraction. However there could be other sources for F3-Hg like glacier melting and historical release at Ny-Alesund.

The results suggest that biogeochemical reactions of mercury in the sediments of Kongsfjorden are mainly involved between non-cinnabar mercury forms (F1–F4). Iron has not showed any significant correlation with other parameters. However it was positively related with mercury fractions especially with F2. No significant correlation was observed for other parameters with mercury fractions.

As mentioned earlier mercury reaches Arctic by air as well as ocean currents and a significant increase in the mercury deposition in the Arctic region has been observed during last decades (Sprovieri et al., 2005). The atmospheric depletion events influenced by the factors such as sunlight, presence of halogens and ozone are being depositing mercury to the land or water especially during the spring time (Lindberg et al., 2002; Berg et al., 2003; Ariya et al., 2004). The ionic mercury form may evolve from the oxidation of stable mercury compounds liberated during the rock weathering. The increase in permafrost melting might have also caused fast releasing of historically accumulated mercury (Walker, 2007; Oberman, 2008; Stern et al., 2012). Studies done by Givelet et al. (2004) showed that mercury concentration in the permafrost peat was about 20 to 50 ng/g and in the Beaufort sea coast, permafrost peat cores showed higher mercury concentration, varied from 20 to 100 ng/g (Leitch, 2006). More studies are needed to address the release of mercury from permafrost as the flux of mercury depends on the mercury concentration in the permafrost, the thawing rate and the erosion rate (Klaminder et al., 2008; Stern et al., 2012). The fractionation study indicate that even though we succeeded in reducing mercury emission worldwide, the already deposited mercury will be a significant threat to Arctic biota as the major portion was weakly bound fractions.

### 3 Conclusion

The sediments of Kongsjorden have low concentration of total mercury except for a few sites in outer zone. However the high concentration observed at inner site indicates the influence of increased glacial melting. The fractionation study showed that the major portion of mercury found in these sediments was in elemental form followed by easily available forms and this may enhance the methylation potential of the system. Very low mercury content was noticed in sulphide fraction (mercury of natural origin) which indicated that the mercury present in the Kongsfjorden sediment was of anthropogenic origin and very recent, most probably deposited through long range transport or from glacial melting. More studies are to be conducted to finalise the role of AMDE, permafrost melting, oceanic currents in the transport of mercury to the Kongsfjorden.

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