

Field experiment on thin-layer capping in
Ormefjorden and Eidangerfjorden, Telemark
Functional response and bioavailability of
dioxins 2009-2011



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Abstract

As part of a research project to examine the effects of thin capping, test fields were established at 30 and 100 m depth in the outer Grenlandfjord in September 2009. One field was treated with crushed limestone, one field was treated with clay dredged from a nearby location and two fields were treated with a mixture of dredged clay and activated carbon (AC). Biogeochemical fluxes, release of chlororganic compounds to the overlying water and uptake in two sediment-living organisms were investigated in box-core samples transferred from test fields and reference locations to the mesocosm laboratory at Solbergstrand in October 2009 and November 2010. Oxygen and nitrogen nutrient fluxes were not significantly affected by capping. Anomalous fluxes of phosphate and silicate were observed in the first investigation and primarily in sediments treated with the limestone material. In all boxes transferred in 2009 and in boxes from clay and AC treatments transferred in 2010, bioaccumulation and leakage of dioxins (PCDD/F-TEQ) was 67% - 91% lower than at the uncapped reference fields. At the two fields treated with limestone gravel and dredged clay without activated carbon, however, cap efficiencies had decreased to less than 46%. It was concluded that for the cap thicknesses of <5 cm applied in the Grenland field experiment, AC-amendment was required to obtain satisfactory cap performance.

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Preface

The present report is the result of a joint effort of the two research projects *THINC* (NIVA) and *OPTICAP* (Norwegian Geotechnical Institute (NGI), NIVA and project partners Norsk Avfallshåndtering (NOAH), Agder Marine, Secora, Hustadmarmor). Both projects were funded by Hydro, Climate and Pollution Agency (Klif) and the Norwegian Research Council (NFR) and *OPTICAP* project partners. The projects have common objectives in developing a method to prevent or reduce risk of spreading of pollutants from historically contaminated sediments to marine water and biota. Within both projects, field experiments were important supplements to smaller scale laboratory and mesocosm experiments, and the joint effort have been mutually beneficial from financial as well as scientific point of view. Espen Eek, NGI, had a superior responsibility for the capping operation in September 2009 supported by Agder Marine, Secora, NOAH and Hydro. NIVA was primarily engaged in the field investigations, which among other activities, included six surveys in the Grenlandfjord area with FF Trygve Braarud during the period October 2008 - May 2011. During two of these surveys, box-core samples were transferred from the capped and un-capped test fields to the soft bottom mesocosm at Solbergstrand Marine Research Station for further measurements as described in this report. The chemical analyses of chlororganic contaminants were performed at the University of Umeå, Miljökemiska Laboratoriet. Analyses of mercury and micronutrient species were performed by Sarah Josefsson under surveillance of staff from the NIVA-laboratory in Oslo.

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Summary

In order to reduce transfer of dioxins from historically contaminated sediments to water and biota, thin layer capping is tested at four different sites in the Grenlandfjord area. In September 2009, three fields were established at 30 m depth in Ormefjorden and one field at 100 m depth in Eidangerfjorden. The applied test materials are gravel (<5 mm fraction) from the limestone quarry at Langøya in the Oslofjord and a marine, silty clay with or without added activated carbon (AC). The clay was dredged from a nearby fjord location and AC was added before deposition at the test fields (Eek et al., 2011).

The main objective of the work presented in this report was to determine the efficiency of thin caps to retain dioxins (PCDD/F), hexachlorobenzene (HCB) and octachlorostyrene (OCS) in the sediments and reduce bioaccumulation in sediment-living organisms. A secondary objective of the field experiment was to investigate potential negative impacts on the benthic ecosystem. Results on the benthic habitat and community structure were reported in Schaanning et al., 2011. In this report ecosystem functions are addressed by determination of the exchange of oxygen and nutrients between the sediments and the overlying water.

The investigations were performed in box-core samples transferred from the test fields to the mesocosm laboratory at NIVAs' Marine Research Station Solbergstrand in October 2009 and in November 2010. During the first 1-2 weeks in the mesocosm, biogeochemical fluxes were measured by determination of the concentration change in the seawater flowing through the boxes. In the second phase of the experiment, passive samplers (Low Density Polyethylene (LDPE) membranes) were installed in the overlying water to measure the release of chlororganic compounds from the sediments and two different sediment living species were added to determine bioaccumulation. These were the deep burrowing polychaete *Nereis (Hediste) diversicolor* and the surface dwelling gastropod *Hinia reticulata*. Exposure periods of 2-3 months were allowed to ensure detectable dioxin levels in most of the samples.

Oxygen and nitrogen nutrient (i.e. ammonium and nitrate) fluxes were not significantly affected by capping. This was consistent with previously reported electrode measurements, which showed little effects of capping on the vertical distribution of oxygen and redox potentials within the sediments. Thus, no evidence was found for any severe disturbance of the major metabolic processes involved in the turnover of organic matter.

Anomalous uptake of phosphate was observed in the boxes transferred from the limestone field in 2009, but not in 2010. It was concluded that the uptake in 2009 was a short-term effect resulting from adsorption to the freshly exposed limestone surfaces. Silicate was released to the overlying water from all sediments. The largest release occurred from uncapped reference sediments and the lowest fluxes occurred from sediments capped with the limestone material which is known to be depleted with regard to soluble silicates. As a result of natural processes restoring normal ecosystem functions, the retention of silicate at the limestone field decreased from 74% in 2009 to 42% in 2010.

In order to compare the three end-points for dioxin bioavailability, the cap efficiency was described as the ratio (R) between the accumulation of dioxins in passive samplers and the two sediment-living species exposed in boxes from capped fields (C_{cap}) and reference fields (C_{ref}) so that $R=C_{cap}/C_{ref}$ (low ratios correspond to high cap performance). In 2009, low cap-efficiency-ratios of 0.09-0.33 showed that bioaccumulation of dioxins (PCDD/F-TEQ) in *Hinia reticulata*, was low at all capped fields compared to uncapped reference fields. In 2010, the cap-efficiency-ratio had increased to 0.54 at the limestone and 0.79 at the dredged clay field without activated carbon. At the two fields treated with

activated carbon, however, the ratios had remained low or decreased to 0.17 at the clay/AC field in Eidangerfjorden and 0.14 at the clay/AC field in Ormefjorden.

Uptake in LDPE membranes in the overlying water and bioaccumulation in the polychaete *Nereis diversicolor* was only measured in 2010. These analyses confirmed the poor performance ($R \geq 0.93$) of the caps without activated carbon, as well as the good performance of the clay-AC caps ($0.17 \leq R \leq 0.28$).

Thus, compared to uncapped reference fields the two fields with clay/AC caps, the observed cap efficiency ratios for dioxin toxicity (PCDD/F WHO2005-TEQ) corresponded to

- 72 and 78% reduced leakage to the overlying water
- 75 and 83% reduced bioaccumulation in *Hinia reticulata* and
- 83 and 86 % reduced bioaccumulation in *Nereis diversicolor*.

Reduced efficiency of the caps without AC added, may have resulted from cap recontamination from particles settling from above or old sediments mixed upwards from below, or from upwards migration of the front of concentration gradients maintained by release of dioxins from the old contaminated sediment below the cap. The general correlation between Hg and chlororganic contaminants in this area, and the observed increase of concentrations of Hg within the cap layers, indicated substantial recontamination of the cap layers between the surveys in 2009 and 2010. Thus the use of activated carbon appeared not only to reduce the bioavailability of dioxins present below the cap, but also to reduce bioaccumulation and leakage of dioxins entering the cap after cap placement.

Reduced pore water concentrations resulting from adsorption to carbon particles was a suitable mechanism for explaining observed differences between the three bioavailability indicators with regard to accumulation of chlororganic compounds.

Repeated investigations of cap performance in the Grenland field experiment have been suggested, and hereby recommended, for verification of cap performance in a longer time perspective.

Sammendrag

For å redusere biotilgjengelighet av dioksiner i sedimentene ble det i september 2009 gjennomført en prøveutlegging på fire områder i de ytre Grenlandsfjordene (Eek et al., 2011). Tre felt på 100x100 m ble etablert på 30 m dyp i Ormefjorden og dekket med hhv grus (<5mm) fra kalksteinsbruddet på Langøya i Oslofjorden, leirsediment mudret fra en nærliggende lokalitet i Ormefjorden og samme leirsediment iblandet aktivt kull (AC). I tillegg ble et større felt på 200x200 m etablert på 100 m dyp i Eidangerfjorden tildekket med mudret leirsediment og AC.

Hovedmålet med undersøkelsene beskrevet i denne rapporten var å bestemme effektiviteten av tynnsjikt i forhold til reduksjon av utlekking av dioxiner (PCDD/F), hexaklorbensen (HCB) og oktaklorstyren (OCS) fra sediment til vannmasse og bioakkumulering i sedimentlevende organismer. Et av målene med felteksperimentet var å undersøke eventuelle negative effekter på sediment-økosystemet. I forhold til dette målet er effekter på habitat og samfunnsstruktur presentert i en tidligere rapport (Schaanning et al., 2011). I denne rapporten adresseres sekundært økosystemfunksjoner som utveksling av oksygen og næringssalter mellom sedimenter og vannmasse.

Alle målinger ble utført i box-core prøver overført fra testfeltene i Grenland til bløtbunnslaboratoriet ved Marin Forskningsstasjon Solbergstrand i oktober 2009 og november 2010. I løpet av de første 1-2 ukene i laboratoriet ble flukser av oksygen og næringssalter bestemt på grunnlag av konsentrasjonsendringer i sjøvann som ble ledet kontinuerlig gjennom boksene. I den påfølgende fasen ble det installert passive prøvetakere (LDPE) i vannet i boksene for å måle utlekkingen av klororganiske forbindelser og dyr ble tilsatt sedimentene for å måle bioakkumulering. To arter ble benyttet; den dypt-gravende børstemarken *Nereis (Hediste) diversicolor* og sneglen *Hinia reticulata* som oppholder seg nærmere sedimentoverflaten. Eksponeringstider på 2-3 måneder ble benyttet for å sikre detekterbare mengder i de fleste prøvene.

Flukser av oksygen og nitrogen-næringssalter (ammonium og nitrat) var ikke signifikant påvirket av tildekkingen på noen av feltene. Heller ikke vertikal-fordelingen av oksygen og redokspotensialer beskrevet i foregående rapport, viste vesentlige effekter av tildekkingen, og undersøkelsene har således ikke gitt noen indikasjoner på at tynnsjikt-tildekkingen hadde vesentlige effekter på prosesser involvert i omsetningen av organisk materiale i sedimentene.

Fosfat ble tilført vannmassen fra sedimentene i de fleste boksene. Et klart unntak var opptak av fosfat til sedimentet i boksene overført i 2009 fra feltet behandlet med kalkstein. Silikat ble alltid tilført vannmassen fra sedimentene, men utlekkingen var noe lavere fra tildekkete sedimenter og lavest i boksene behandlet med kalkstein. Grusen fra kalksteinsbruddet på Langøya inneholder lite silikat og har trolig en viss evne til å adsorbere fosfat. Naturlige prosesser vil medvirke til å gjenopprette normale økosystemfunksjoner. I 2010 ble det således ikke observert adsorpsjon av fosfat på kalkfeltet og retensjonen av silikat var redusert til 42 % sammenlignet med 74 % i 2009. (Tallene viser her utlekking av silikat fra kalkfeltet i prosent av utlekking av silikat fra referensefeltet.)

I 2009, viste effektivitetsforhold ($R = C_{\text{cap}}/C_{\text{ref}}$) på 0,09-0,33 at bioakkumulering av dioksiner (PCDD/F-TEQ) i *Hinia reticulata* var lav på alle tildekkingsfeltene sammenlignet med ikke-tildekkete referensefelt. Året etter var forholdet økt til 0,54 på kalkfeltet og 0,79 på feltet dekket med leirsediment uten aktivt kull. På begge feltene dekket med leirsediment iblandet aktivt kull, var effektivitetsforholdene fortsatt lave, dvs 0,17 på leire/AC feltet i Eidangerfjorden og 0,14 på leire/AC feltet i Ormefjorden.

Akkumuleringen på LDPE membranene i vannet over sedimentet og i børstemarken *Nereis diversicolor* ble bare målt i 2010. Disse analysene bekreftet den dårlige effektiviteten ($R \geq 0.93$) på feltene uten aktivt karbon og den gode effekten ($0.17 \leq R \leq 0.28$) på feltene med aktivt karbon.

Sammenlignet med referensfeltene var

- utlekking redusert med 72 - 78 %
- bioakkumulering i *Hinia reticulata* redusert med 75 - 83% og
- bioakkumulering i *Nereis diversicolor* redusert med 83 - 86%.

Redusert effektivitet fra 2009 til 2010 av tynnsjiktene uten AC-tilsetning kan ha resultert fra rekontaminering av tynnsjikt ved sedimentasjon av forurenset materiale eller bioturbasjon og oppblanding med gammelt, forurenset sediment, eller fra diffusiv eller advektiv transport av løste forbindelser via porevannet. Den generelle korrelasjonen mellom kvikksølv og klororganisk forurensing i dette området, og den observerte økningen av konsentrasjonen av kvikksølv indikerte betydelig rekontaminering av tynnsjiktene i løpet av det første året etter utlegging. Bruken av aktivt kull viste seg altså også effektivt i forhold til å binde dioksiner tilført tynnsjiktene etter utlegging.

Redusert konsentrasjon i porevannet som følge av binding til karbon partikler var egnet mekanisme til å forklare observerte forskjeller mellom de tre biotilgjengelighets-indikatorerne med hensyn til akkumulering av klororganiske forbindelser.

Oppfølgende undersøkelser av testfeltene i Grenland er blitt foreslått og anbefales herved, for å få dokumentasjon om hvordan tynnsjiktene evne til å holde forurensingen tilbake i sedimentene utvikler seg over et lengre tidsrom enn et år etter utlegging.

1. Introduction

1.1 Objectives

The primary objective of thin capping is to develop a method to reduce the release of contaminants from sediments to fjord water and biota. As a supplement to theoretical modeling and small scale laboratory and mesocosm experiments, a field experiment was conducted to test the technical and engineering challenges of cap placement and cap performance on a real seabed.

Because the thin cap method is intended for remediation of large areas with potentially high ecological status, a secondary objective of the field and mesocosm experiments was to assess the changes imposed by thin caps on benthic community composition and important functions such as the exchange of oxygen and nutrient species between the sediments and fjord water.

The benthic habitat and macrofauna analyses from the field experiment were reported in Schaanning et al., 2011. This report address the investigations performed in box-cores transferred from the test fields in Eidangerfjorden and Ornefjorden in October 2009 and November 2011. The objectives of the box-core investigations were to assess the effects of the caps applied in the field experiment on biogeochemical fluxes and bioavailability of dioxins.

1.2 The field experiment

The test fields were established in September 2009. Field codes and brief description of treatments and obtained cap thickness are given in **Table 1**. Locations are shown in **Figure 1**. A complete description of the capping operation is given in Eek et al., 2011.

In Ornefjorden, 3 fields of 10 000 m² at 24-32 m depth were treated with 1) gravel supplied from the limestone quarry operated by Norsk Avfallshåndtering (NOAH) at Langøya, 2) silty-clay sediments suction-dredged at 10-20 m depth in a nearby location and 3) sediments dredged from the same location amended with 2 kg m⁻² activated carbon. A fourth field was left untreated for control purposes. At the dredging site, the moderately contaminated top layer (ca 1 m) was suctioned off and shipped to a land-deposit before dredging sediments for the capping operation (Eek et al., 2011).

In Eidangerfjorden, one field (FE5) of 40 000 m² at 92-96 m depth was capped with suction-dredged silty-clay sediments amended with 2 kg m⁻² activated carbon. In Eidangerfjorden the reference location was situated at 85 m depth to the north of the test field. Trawling is a regular activity in Eidangerfjorden. In understanding with the local fishermen, FE5 is not trawled during the field experiment and the reference field is beyond reach of the trawling gear due to topographic restrictions.

Table 1. Field names and treatments. Mean cap thickness \pm 1 standard deviation as given in Eek et al., 2011.

Fjord	Field	Treatments	Acronym	Cap thickness (cm)	Typical depth (m)	Field Area (m ²)
Ornefjorden	FO 1	Gravel of limestone	NOAH 30	2.1 \pm 1.2	30	10 000
Ornefjorden	FO 2	Clay (dredged)	Clay 30	3.7 \pm 1.1	30	10 000
Ornefjorden	FO 3	Active Carbon (in clay)	Clay AC 30	1.1 \pm 0.6	26	10 000
Ornefjorden	FO 4	Reference	REF 30	-	30	-
Eidangerfjorden	FE 5	Active Carbon (in clay)	Clay AC 10	1.2 \pm 0.3	95	40 000
Eidangerfjorden	FE 6	Reference	REF100	-	85	-

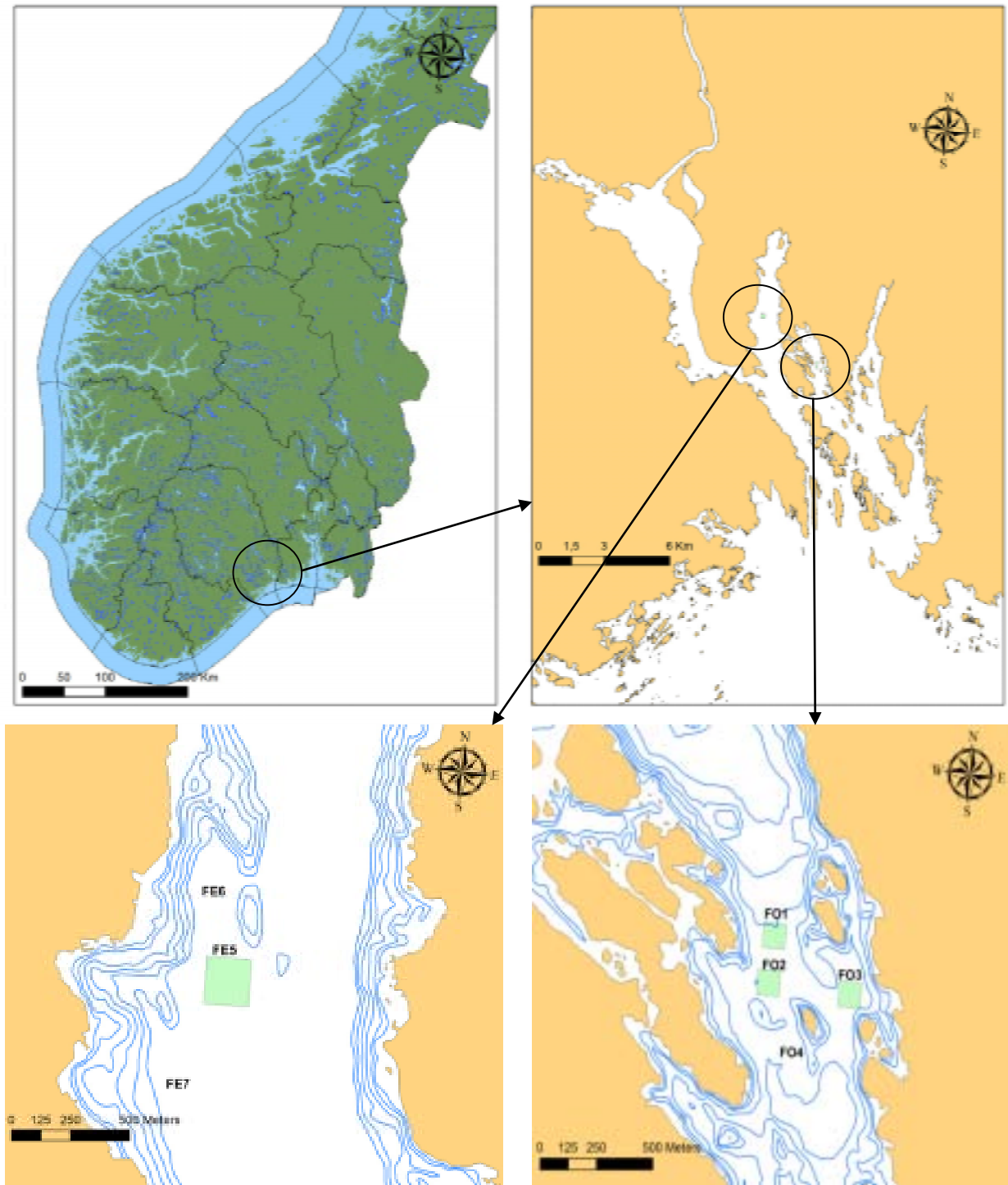


Figure 1. Map showing the Grenlandfjord area (top) and test plots in Eidangerfjorden (low left) and Ormefjorden (low right).

2. Methods

2.1 Box-core experiments

2.1.1 Sampling and transfer to mesocosm

Box-core samples were transplanted from the test plots to the Solbergstrand mesocosm 15.-16.10.2009 and 10.-11.11.2010. The samples were collected with a 0.1 m² KC-Denmark™ box corer with transparent polycarbonate liners attached inside the steel box. On deck, a steel sheet was inserted at the base of the liner to provide the bottom of the box core sample. The liner with apparently undisturbed cap and approximately 30 cm of layered sediment with inherent organisms and niches, was then released from the steel box and placed on deck. The overlying water was removed through a siphon to reduce erosion of the sediment surface during transportation and handling. A lid was placed on top of the liner and the boxes were stored on deck overnight before transport to the research station at Solbergstrand where they were placed in a large water bath and supplied with deep seawater less than 32 hours after sampling.

In the mesocosm (Berge et al., 1986), artificial light and a continuous supply of fjord water from 60 m depth (Skagerrak/Outer Oslofjord) was used to maintain an experimental environment resembling the conditions at the fjord sampling locations, i.e. dim light, temperatures of 8-10°C, salinities of 34-35 PSU and 7-9 mg O₂ L⁻¹. The water overlying the sediments in each box was continuously exchanged with the fjord water to maintain high (>60%) degree of saturation with O₂.

During the biogeochemical measurements a flow of about 10 ml min⁻¹ was maintained using peristaltic pumps with one channel/box. Stirring was performed using aquarium pumps submersed in each box and adjusted to ensure a well-mixed water column without visible resuspension of sediments. After completion of the biogeochemical flux measurements, the flow-through was reduced to about 0.5-1 ml min⁻¹ and the aquarium pumps were replaced with an airlift system as described in Schaanning et al., (2006) to ensure a well-mixed and oxygen saturated overlying water.

2.1.2 Biogeochemical measurements

Fluxes (F) of oxygen (O₂) and nutrient species (SiO₄, PO₄, NH₄ and NO₃ incl. NO₂) were determined from the concentration difference between the inlet water (C_i) and the overlying water (C_o) in each box core: $F = (C_i - C_o)Q/A$, in which A is the sediment area in the box core and Q is the flow rate of seawater passing through each box core.

The O₂ difference was measured with a precision of <0.05 mg O₂ l⁻¹ using a Clark-type oxygen electrode with an internal reference electrode. Nutrient concentrations were determined in water samples collected from the header tank and overlying water in each box using a 50 ml syringe. All samples were preserved during sampling in accordance with laboratory prescriptions and stored in the dark at -20°C until analyses at the NIVA-laboratory using automated spectrophotometric methods for nutrient analyses in sea water.

In 2009 the boxes were set up with flow-through of seawater from 60 m depth in the Oslofjord and fluxes of oxygen and nutrient species were done as soon as steady state concentrations were obtained in the overlying water in each box. To ensure that steady state was obtained, oxygen concentrations

were recorded daily during the period 20.-23.10.09. Water samples from the header tank (common supply water) and each box were collected for nutrient analyses on 23.10.2009.

A similar procedure was followed for the boxes installed in the mesocosm 11.11. 2010. Oxygen fluxes were calculated from the mean of three concentrations measured during the period 17.-22.11. Nutrient fluxes were calculated from concentrations measured in one sample collected from each box on 22.11. and 3 samples collected in the header tank during the period 17.-22.11. The variation in water quality analyses, as indicated by the standard deviations given in **Table 2** is a major source of error in flux determination.

Table 2. Mean concentrations and standard deviation of oxygen and nutrient species in the source water (header tank) and overlying water in each box sampled three times during the period 17.-22.11.2011.

	Header tank (n=3)		Overlying water (n=54)	
	mean	stdev	mean	stdev
O ₂ (mg L ⁻¹)	6.97	± 0.12	5.72	± 0.28
PO ₄ (µM)	0.98	± 0.18	1.17	± 0.24
SiO ₄ (µM)	9.6	± 2.5	17.0	± 3.3
NO ₃ (µM)	9.3	± 1.7	8.0	± 0.4
NH ₄ (µM)	0.7	± 0.1	4.7	± 4.1

2.1.3 Fluxes of dioxins

Dioxin fluxes were measured in 2010 only. This was done by deployment of LDPE membranes in the overlying water in each box. The membranes were deployed from 16.12.2010 to 29.03.2011. During deployment, water flow was reduced to 0.8-0.9 mg min⁻¹ which corresponded to a turnover of about two weeks for the overlying water. Aeration was applied to avoid oxygen deficits and to maintain a well-mixed watercolumn. LDPEs exposed in one of the three replicate boxes only, were sent to the laboratory in Umeå for dioxin analyses. Fluxes were calculated in accordance with Josefsson et al., 2011.

2.1.4 Uptake of dioxins (and Hg) in benthic organisms

Uptake of dioxins were determined in two species the gastropod *Hinia reticulata* and the polychaete worm *Nereis diversicolor* (**Figure 2**). The gastropod lives mostly within the top 1-2 cm of the sediment, while the polychaete dwells in burrows down to 10-20 cm. The polychaetes are therefore assumed to be more exposed to contaminated sediments and pore water than the gastropods. Both species were collected from wild populations in Outer Oslofjord and added to the boxes. After 8-12 weeks of exposure, the organisms from each box were collected and allowed to depurate their gut of sediment overnight in glass beakers with 500 ml of clean seawater. Samples from replicate boxes were pooled before analyses.

In 2009 thirty individuals of the gastropod were added to each box during the first week of November and sampled two months later on 06.01.2010. Mercury analyses were performed on homogenised soft tissue shortly after depuration on a Lumex Hg-analyser set up in the mesocosm laboratory. Six samples with remaining soft-tissues pooled from each field were sent to the laboratory in Umeå for dioxin analyses.

In 2010 twenty individuals of the polychaete was added to each box at the end of November 2010. Unfortunately, probably due to an early winter, no snails could be found in November 2010, and the

exposure had to be postponed. Twenty individuals/box of the gastropod were collected and added to the boxes on 13.05.2012. All organisms were retrieved from the sediments 7.-9.07.2012 and allowed to depurate overnight in clean seawater, before homogenisation of soft-tissues pooled to provide one sample per specie per field. The samples were frozen and sent to the laboratory in Umeå for dioxin analyses.

2.1.1 Chemical analyses

Analytical procedures and calculation of dioxins are given in appendix Appendix A. -Appendix C.



Figure 2. Concentration of dioxins was measured in two different species. Left: the netted dogwhelk *Hinia reticulata* (Gastropoda) or *nettsnegl* (in Norwegian). Right: the polychaete *Nereis* sp (Annelida) or *børstemark* (in Norwegian).

3. Biogeochemical fluxes

The fluxes of oxygen and nutrient species are sensitive indicators of geochemical, biochemical and biological processes in the sediment. The macrobenthic community affects fluxes by their own metabolism and, maybe more important, by sediment mixing, pore water irrigation and creation and maintenance of niches such as ventilated burrows which are thought to be important sites for microbiological and geochemical processes. Long-term changes of these processes may affect the nutrient balance in the fjord water and hence primary production, composition of plankton communities and oxygen conditions in deeper strata. In the present project, oxygen and nutrient fluxes were measured in order to assess the extent and permanence of thin cap disturbance of sediment-water exchange of oxygen and nutrients.

The fluxes at each field measured in October 2009, about one month after cap placement, and in November 2010, about 14 months after cap placement, are shown in Figure 3- **Figure 7**. The fluxes measured at each field was compared statistically (ANOVA, Tukey, $\alpha=0.05$). Tukey's test compares all fields and the results are presented by letters assigned to each field in accordance with the following rule: "fields not connected with the same level are significantly different".

3.1 Fluxes of oxygen, nitrate and ammonium

None of the fields showed significantly different fluxes of O₂, nitrate and ammonium neither in 2009 nor in 2010.

Uptake of nitrate and nitrite are normally driven by microbial processes in the sediment and frequently correlated with the uptake of O₂. Uptake of nitrate from water to sediment was observed in all boxes (**Figure 4**). The highest uptake occurred in 2009 in sediments treated with NOAH limestone and the lowest uptake occurred also in 2009 in the clay AC treatment in Ornefjorden. The differences between fields were smaller in 2010 than in 2009 indicating normalisation of nitrate fluxes after cap placement.

Ammonium is generally low in oxic in sea water due to nitrification (biological oxidation of NH₄ to NO₃) and assimilation in bacteria and algae, and high in anoxic pore water due to degradation of organic matter. Therefore ammonium is generally released from the sediment to the overlying water. In 2009, very low release of NH₄ was observed from the NOAH limestone and dredged clay fields in Ornefjorden (Figure 5). This may be explained by the fact that the cap layers were thicker than on the other fields, contained little organic matter and possibly sufficiently oxic to allow nitrification. In 2010, the differences between the fields were smaller and as for nitrate, this may be interpreted as normalisation of minor anomalies introduced by cap placement.

Oxygen may be supplied at the water surface by uptake from the atmosphere or production by algae. Oxygen is consumed in sub-surface water and in sediments by degradation of organic matter and oxidation of reduced substances such as H₂S, NH₄, Fe²⁺ and Mn²⁺. In 2009, the sediment oxygen consumption was somewhat lower in clay-AC fields (240-276 $\mu\text{mol m}^{-2} \text{h}^{-1}$) than in the reference fields in both fjords (343-395 $\mu\text{mol m}^{-2} \text{h}^{-1}$). In 2010, a similar difference was observed in Ornefjorden but not in Eidangerfjorden. In both years the NOAH limestone treatment showed lower oxygen consumption (248 and 288 $\mu\text{mol m}^{-2} \text{h}^{-1}$) than the reference field (311 and 343 $\mu\text{mol m}^{-2} \text{h}^{-1}$). These differences were consistent with the concentrations of organic carbon of 2,2% TOC in Eidangerfjorden as compared to 1,2-1,5% in the dredged sediments (Eek et al., 2011) and the fact that

the NOAH gravel can be assumed low in TOC (not measured by Eek et al., 2011, due to high conc. of inorganic carbon). In addition, any organic carbon present in the NOAH limestone or in the clay sediment dredged after removal of the top layer will be older and more refractory than the organic carbon present on the reference fields.

The absence of significant effects on these fluxes was consistent with the small differences between the fields revealed by the electrode measurements of O₂ and redox potentials reported in Schaanning et al., 2011. It appears that carbon mineralisation had been marginally affected by the thin caps applied in this experiment.

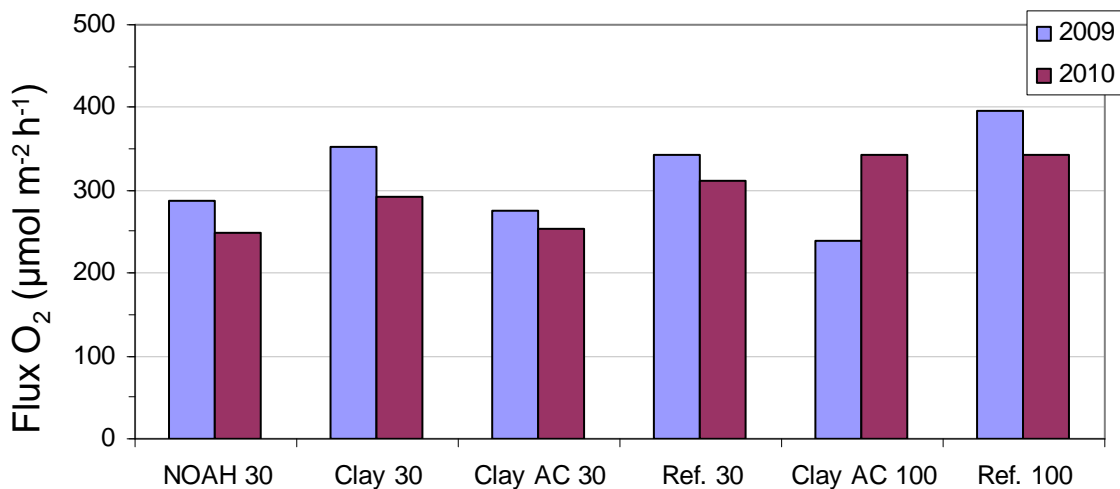


Figure 3. Oxygen uptake from water to sediment 1 and 14 months after cap placement. Statistical comparison (ANOVA, Tukey) showed no difference between fields neither in 2009 nor in 2010.

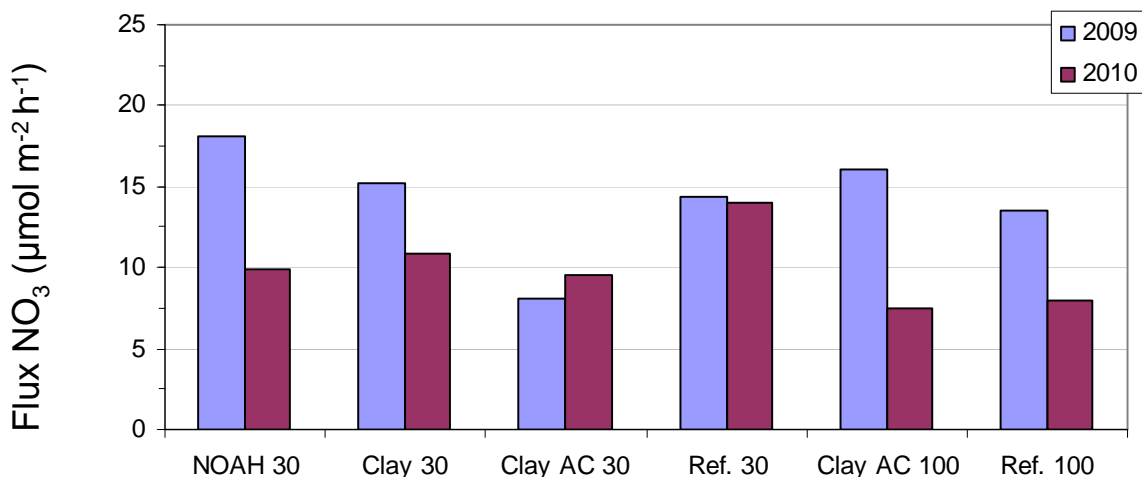


Figure 4. Uptake of nitrate (included nitrite) from water to sediment 1 and 14 months after cap placement. Statistical comparison (ANOVA, Tukey) showed no difference between fields neither in 2009 nor in 2010.

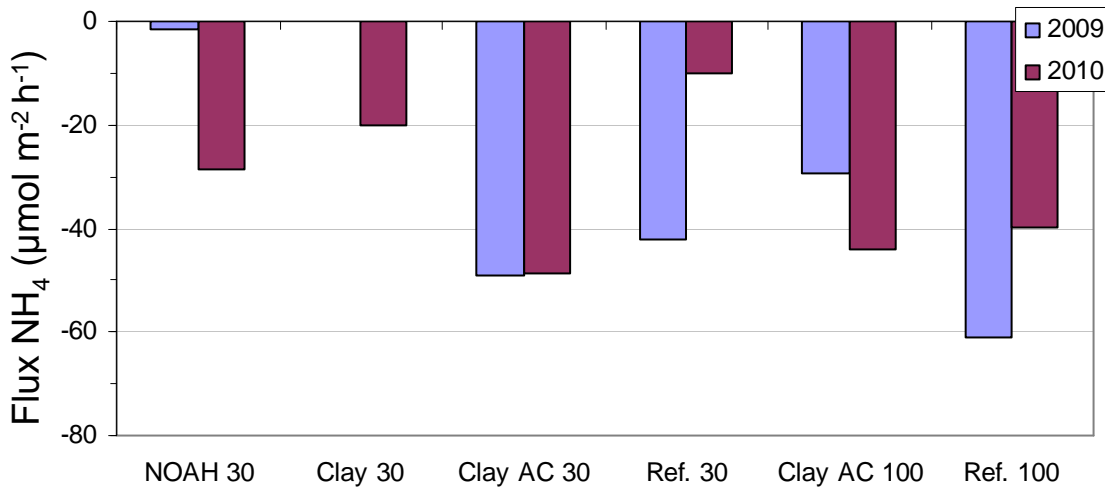


Figure 5. Release of ammonium from water to sediment 1 and 14 months after cap placement. Statistical comparison (ANOVA, Tukey) showed no difference between fields neither in 2009 nor in 2010.

3.2 Fluxes of phosphate

Fluxes of PO_4 are frequently small and variable and may be controlled by both biotic and abiotic processes. When buried sediments are dredged and re-exposed to oxic water on the sediment surface precipitation of ferric oxides (or hydroxides) and co-precipitation of PO_4 may occur. The contribution of iron minerals in the added limestone is not known, but phosphate is known to be adsorbed onto other mineral phases than ferric oxides.

Phosphate was released from sediment to water in both reference fields and most of the capped fields (**Figure 6**). This may partly result from desorption and diffusion from subsurface anoxic layers, and partly from degradation of organic matter at the sediment surface. A considerable uptake of PO_4 was observed in the NOAH limestone treatment in 2009, and this uptake was significantly different from the maximum release of PO_4 observed in the untreated Eidangerfjord field. In 2010, small and similar release of PO_4 was observed from all fields in Ormefjorden. The uptake observed shortly after cap placement was most likely chemical adsorption to the freshly mined material, but the second year results showed that this was not a long-term effect.

The relatively high release of PO_4 in Eidangerfjorden compared to Ormefjorden is probably related to the higher TOC-concentrations cited above and possibly an impact from a near-by discharge from a local sewage treatment plant (M.Olsen, pers.com.). In 2010, the release of PO_4 from the Clay-AC field in this area was significantly higher than the release of PO_4 from all fields in Ormefjorden (**Figure 6**), but there is little rationale to ascribe this high release to the placement of the clay-AC cap.

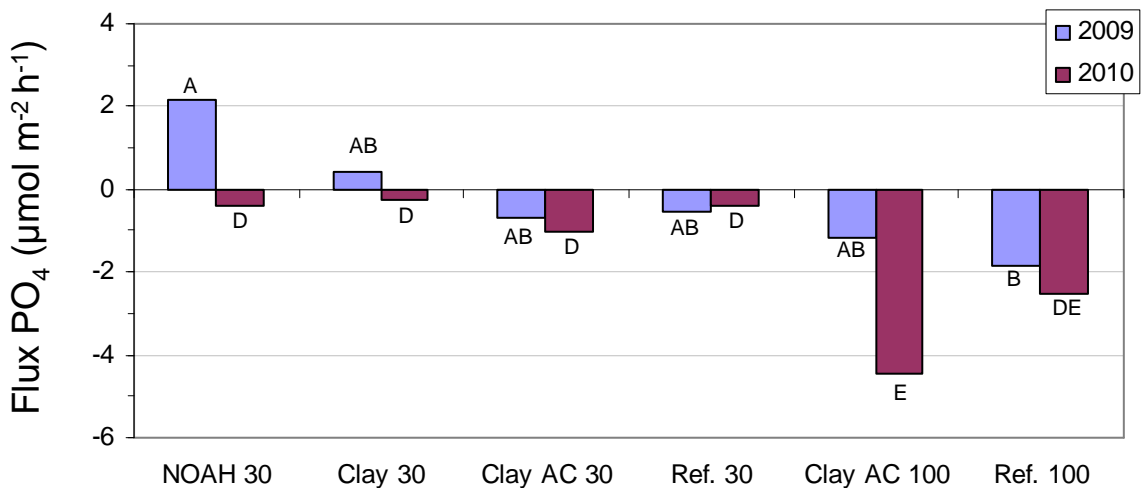


Figure 6. Flux of phosphate between sediment and water 1 and 14 months after cap placement. Negative flux means release from sediment to overlying water. Letters show results of statistical comparison (ANOVA, Tukey) of all fields for 2009 (A, B) and 2010 (D, E) (see text).

3.3 Fluxes of silicate

Silicate is utilised primarily by planktonic algae (diatoms) which produce silica skeletons in the surface fjord water. Silicate is supplied to the fjord water from river runoff and from dissolution of silica minerals and degradation of diatoms in deep water and sediments.

Silicate was released from the sediment to the overlying water in all boxes. In 2009, the highest release occurred from the two reference sediments (136 and $176 \mu\text{mol m}^{-2} \text{h}^{-1}$) and the lowest release occurred from the sediment treated with NOAH limestone ($30 \mu\text{mol m}^{-2} \text{h}^{-1}$ or 22% of the reference field). In the dredged clay and dredged clay AC treatments the release of silicate was 66-77% of the release from the respective reference fields.

In the mesocosm experiment (Näslund et al., 2011) the release of silicate from sediments treated with pre-glacial clay was 61-64% of control sediments, and 12-22% in sediments treated with mineral materials of mostly CaCO_3 (from Hustadmarmor) or CaSO_4 (from NOAH). These minerals are not likely to contain significant amounts of biogenic silica or silicate minerals and the low release of silicate observed shortly after capping both in mesocosm and field experiment was as expected and showed that the caps act as lids to reduce the flux of silica from the sediments below the cap. The lid effect may have contributed to the fact that the two lowest fluxes occurred from the two fields with the thickest cap layers (NOAH limestone and dredged clay in Ornefjorden).

Because of the higher age and solubility of silica minerals, the dredged clay, as well as the pre-glacial clay used in the mesocosm experiment, might be expected to be more or less depleted with regard to both biogenic and mineralogenic silica compared to the sediment surface layers at the reference fields. Therefore, the fluxes of silicate from these old clays were, as expected, intermediate between the reference fields or control boxes and the mineral materials dominated by calcium carbonates and calcium sulphates.

The statistical analyses showed that in 2009 all capped fields in Ormefjorden released significantly less silicate than the reference field in Eidangerfjorden, and that the low release from the NOAH limestone field was significantly less than 4 of the 5 other fields.

With time, sedimentation of new material will imply restoration of the rate of production of dissolved silicate in the cap layers and the diffusive flux from the sediments below the caps will penetrate the cap layers. Both mechanisms will contribute to an expected increase of the release to the overlying water. 14 months after the capping operation, the major decrease of silicate fluxes from both reference fields may indicate large seasonal or annual variations, but the difference between the fields had become clearly smaller, and in spite of the general decrease, the flux had increased slightly from NOAH 30, which was no longer significantly different from the reference field.

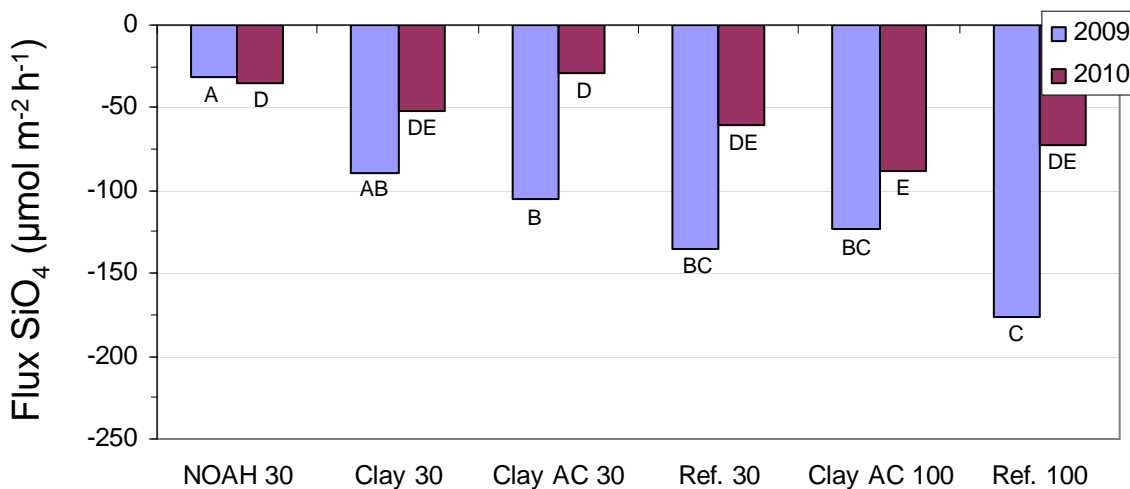


Figure 7. Release of silicate from sediment to overlying water 1 and 14 months after cap placement. Letters show results of statistical comparison (ANOVA, Tukey) of all fields for 2009 (A, B, C) and 2010 (D, E) (see text).

4. Cap recontamination

4.1 Dioxins and mercury in sediment and cap materials

Data from previous NIVA projects (**Figure 8**) shows that the dioxin concentrations in the 0-2 cm top layer of sediments sampled in the deepest parts of Eidangerfjorden in 2005 were approximately 2x the upper limit of class V “very bad” (Klif, 2007). Furthermore the figure shows that concentrations tend to increase with depth and verifies the expected decrease due to emission reductions. The sediments of the Grenland fjords are also contaminated with mercury and Josefsson et al. (2012) found a close correlation between dioxins (SumPCDD/F) and mercury in mixtures of clean and heavily

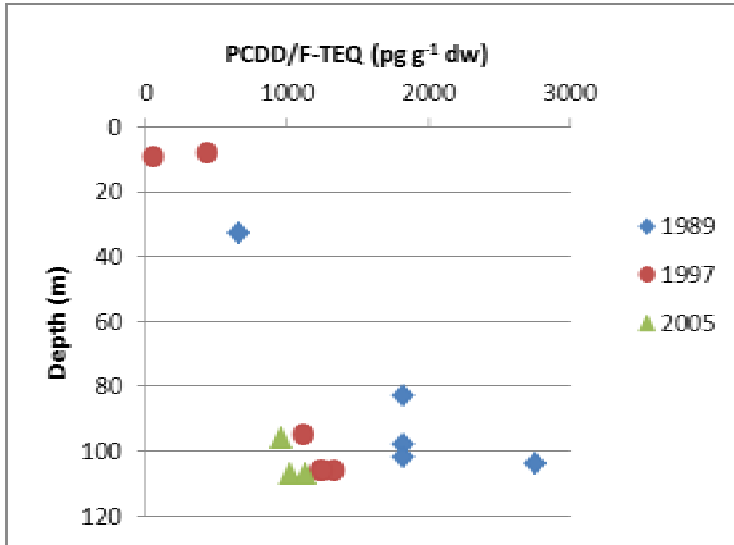


Figure 8. Dioxin analyses in top 0-2cm of sediments in Eidangerfjorden sampled during the period 1989-2005. Norwegian EQS for class V “very bad” is 500 pg g⁻¹ d.w.

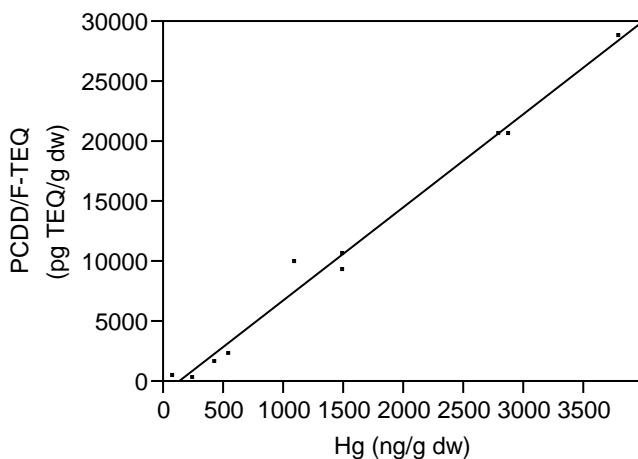


Figure 9. Correlation between concentrations of dioxins (PCDD/F-TEQ) and mercury in sediment samples representing various mixtures of sediments from the Grenlandfjord area (from Josefsson et al., 2012).

Table 3. Concentrations of mercury in sediment top layer shortly after cap placement on experimental fields in Ornefjorden and Eidangerfjorden. Analyses were performed on 0-1 cm sections of cores subsampled in January 2010 from 12 box cores transferred from field locations to the Solbergstrand mesocosm in October 2009. Last column shows classification according to Norwegian Guidelines (Klif, 2007).

Field	Sample type	ng Hg g ⁻¹ d.w.	Classification
FO1	NOAH limestone	12-27	I Background
FO2	Dredged clay	106-174	I Background
FO3	Dredged clay/AC	195-247	II Good
FO4	Ornefjorden ref.	318-371	II Good
FE5	Dredged clay/AC	133-392	II Good
FE6	Eidangerfjorden ref.	703-850	III Moderate

polluted sediments from Frierfjorden (**Figure 9**). Dioxins were not analyzed in sediment samples collected during this investigation. In this investigation, frequent analyses of Hg were used to indicate mixing between sediments and cap materials.

The mercury concentrations determined in the top 0-1 cm of the sediments (**Table 3**) showed lowest concentrations (12-27 ng Hg g⁻¹ d.w.) in the NOAH limestone field (FO1) and 106-174 ng Hg g⁻¹ d.w. in the dredged clay field (FO2). Because of the relatively thick cap layers applied on these fields (**Table 1**), these samples were considered the most representative for the added cap materials. The difference between the two materials was assumed to result from a higher background level in the dredged clay compared to the NOAH limestone. The correlation in **Figure 9** shows that 140 ng Hg g⁻¹ d.w. correspond to 1000-2000 pg g⁻¹ TEQ, but the correlation is uncertain at these low contaminant levels. This is due to the source history which implicates that the Hg:dioxin ratio change with sediment depth and is likely to be very low at the depth at which the clay used for capping was dredged (Eek et al., 2011).

At the other two fields treated with dredged clay and AC, the thickness of the cap layers were only 1.1 and 1.2 cm (**Table 1**) and hardly exceeded sampling depth. Therefore, the higher concentrations of Hg at these two fields probably resulted from co-sampling of original sediments present in the lower part of the sampled sediment section. Thus, the analyses gave no evidence for cap contamination 1-2 months after cap placement. Simultaneously, this confirmed that mixing between cap materials and underlying sediments during box core sampling, transfer and 2-3 months maintenance in the mesocosm had not been severe.

4.2 Recontamination of cap layers

The inherent concentration of Hg in the cap materials (C_{cap0}) was assumed to be 12 ng Hg g⁻¹ d.w. for the NOAH limestone and 106 ng Hg g⁻¹ d.w. for the dredged clay. This corresponded to the minimum concentrations of the range given in **Table 3**. A cap contamination factor (F_{cap}) could then be calculated as:

$$F_{cap} = (C_{cap} - C_{cap0})/C_{ref}$$

in which C_{cap} was the concentration of Hg in the 0-1cm layer of the capped sediment and C_{ref} was the concentration of Hg in the 0-1 cm layer at the corresponding reference field. **Figure 10** shows that in 2009 the cap was more contaminated at FO3 and FE5 than at FO1 and FO2. This was consistent with cap thickness of only 1.1±0.6 cm at FE5 and 1.2 ±0.3 cm at FO3 (**Table 1**), which makes the probability small for sampling cap only in all replicate samples of the 0-1cm section.

With time, F_{cap} is expected to increase due to recontamination of the cap with Hg mixed in from below by bioturbation or from above by sedimentation of new material which is likely to contain less contaminants than the sediment below the cap but more than the cap material. Even though the major sources in the area has been eliminated, the new material entering the sediment in the deep fjord bottoms may be contaminated due to resuspension and lateral transport from other and more shallow areas of the fjord. Except for FO3, at which a relatively high factor was determined already in 2009, the ratios increased from 2009 to 2010 indicating a relatively rapid process of recontamination (**Figure 10**). The standard deviations revealed large data scatter, but the mean factor of 0.85 in Eidangerfjorden in 2010 showed negligible reduction of the Hg level one year after the capping operation at this field. The cap layer was equally thin at FO3 and neither total abundance nor biomass of macrobenthic organisms (Schaanning et al., 2011) indicated any major difference with regard to bioturbation. However, suspended particles collected at various depths in the Grenland fjords in November 2010 showed that the concentrations of dioxins in suspended matter increased with depth (Allan et al.,

2011). This tended to support the hypotheses that sedimentation of resuspended material is an important factor contributing to recontamination of the cap at the deep FE5 field. Another factor which may be important is the frequent observation of increasing Hg input from diffuse sources rather than decreasing in many Norwegian fjords. This might imply that the use of Hg as tracer tend to overestimate recontamination with regard to dioxins. If the sewer discharge in the vicinity of FE5 adds to such overestimation is not known.

Thus, it is important not to disregard the limitations with regard to the use of Hg as tracer for dioxin contamination. If, however, recontamination occurs as indicated in **Figure 10**, the cap efficiency discussed in the next chapter might be expected to decrease correspondingly between 2009 and 2010, i.e. major decrease at FO1, FO2 and FE5.

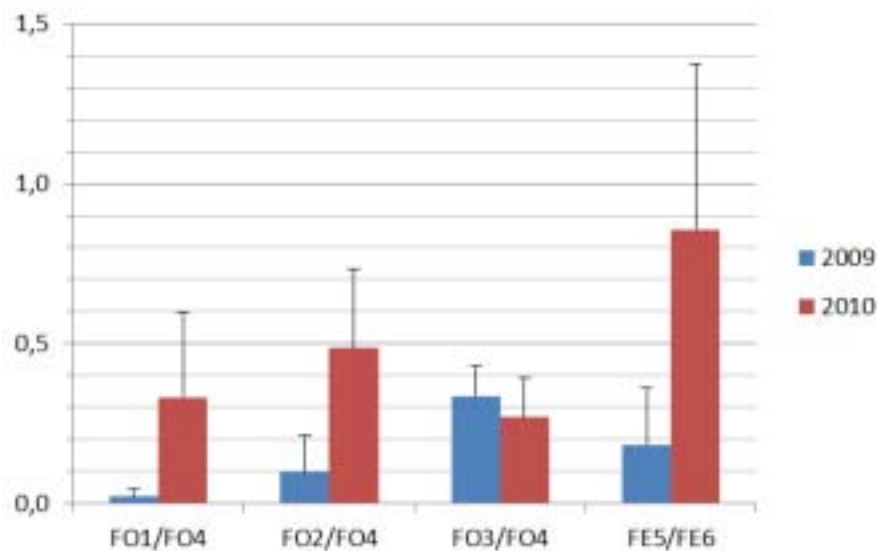


Figure 10. Cap contamination factors (F_{cap}) (see text) determined for the 0-1 cm depth interval at the test fields in Ornefjorden (FO) and Eidangerfjorden (FE) in 2009 and 2010. (Mean value + 1 std. dev.).

5. Bioavailability of dioxins

5.1 Cap efficiency

Bioavailability was measured as the accumulation of chlororganic compounds in two organisms exposed in the sediments and on LDPE membranes exposed in the water overlying the sediments in the box-core samples. The gastropod *Hinia reticulata* is a mobile organism residing at or just below the sediment surface with its siphon extending into the overlying water. The polychaete *Nereis diversicolor* are facultative filter feeders and reside in ventilated, U-shaped tubes which can extend downwards to >20 cm depth into the sediment (Esselink and Schwartz, 1989, Nilsen et.al., 1995). During our sampling for dioxin-analyses, most of the polychaetes were found within the 5-15 cm layer of the sediment.

Because of the costs of dioxin analyses, analyses of replicate samples were not performed. However, by expressing the accumulation in the various sample matrixes (LDPE, Nereis, Hinia) as the ratio between capped (C_{cap}) and uncapped reference sediments (C_{ref}), the three different matrixes could be used to estimate 3 equivalent, numeric values. These ratios

$$R=C_{cap}/C_{ref}$$

describe cap efficiency with regard to preventing chlororganic compounds to be taken up in sediment-living organisms and leakage of water-soluble fractions to the overlying water.

Throughout this report, FO4 was used as reference for FO1, FO2 and FO3 and FE6 was used as reference for FE5.

5.2 Results

5.2.1 Bioaccumulation in gastropods

Bioaccumulation in *Hinia reticulata* was the only cap efficiency parameter determined both in 2009 and 2010. At the reference fields FO4 and FE6, the concentrations of dioxins (PCDD/F-TEQ) in this organism ranged from 3.7-6.1 pg/g wet wght. (**Figure 11**). Low concentrations from 0.4 to 1.4 pg/g wet wght. were observed at all capped fields in 2009 and at the clay/AC fields FO3 and FE5 in 2010. Thus, during the first year after cap placement, the bioaccumulation in the gastropod increased 4-5x in caps without AC amendment, whereas no clear change was observed neither in AC-amended caps nor in uncapped reference sediments. Thus, the performance of the limestone gravel and clay without AC caps declined during the first year after cap placement.

The PCDD/F-TEQ cap efficiency ratios in 2010 was 0.54 and 0.83 for limestone gravel and dredged clay, respectively, as compared to 0.19 at the clay/AC field in Ornefjorden (FO3) and 0.24 at the clay/AC field in Eidangerfjorden (**Table 4**). As shown in the table, the ratios varied among individual congeners, but the general shift towards higher ratios at the two fields without AC (FO1 and FO2) and towards lower ratios for the two fields with AC amendment (FO3 and FE5), is clearly seen when comparing the columns in **Table 4**.

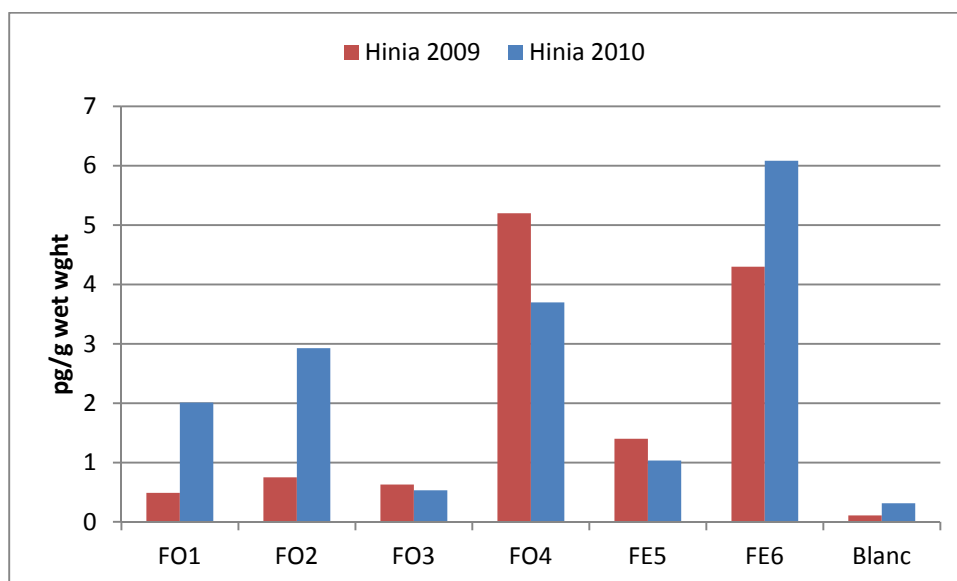


Figure 11. Dioxins (Sum PCDD/F-TEQ) in laboratory blank and in *Hinia reticulata* (snails) exposed in box-core samples from experimental fields capped with 1-4 cm thick layers of limestone gravel (FO1), dredged clay (FO2), dredged clay/AC (FO3) and reference field (FO4) in Ornefjorden and dredged clay/AC (FE5) and reference field (FE6) in Eidangerfjorden.

5.2.2 Bioaccumulation in polychaetes

At the reference fields, the concentration of dioxins (PCDD/F-TEQ) in *Nereis diversicolor* (**Figure 12**) was generally higher than the concentration in *Hinia reticulata* (**Figure 11**). This difference is probably characteristic for the species and believed to result primarily from different feeding strategies and exposure to pore water. Both organisms showed clearly higher concentrations in Eidangerfjorden (FE6) compared to Ornefjorden (FO4), most likely as a result of different concentrations in the sediments. A higher level of dioxin concentrations in the sediments in Eidangerfjorden compared to Ornefjorden would be consistent both with the depth gradient of dioxin concentrations shown in **Figure 8** and the difference in concentrations of Hg determined in this work (**Table 3**). Also the LDPE's (see below) showed a similar difference between the two locations, whereas the gastropod showed no consistent difference between the two locations (**Figure 11**).

At the limestone field (FO1), bioaccumulation ratios for *Nereis diversicolor* were close to 1.0 for HCB and OCS and slightly higher (typically 1.8-2.7) for dioxins and furans (**Figure 14, Table 4**). At the dredged clay (FO2) field the ratios were close to 1.0 for most compounds (**Table 4**). At the clay/AC fields FO3 and FE5, the ratios were frequently less than 0.5. This showed that the caps without AC had little or no effect on the dioxin levels in the polychaetes, whereas caps with AC clearly reduced the levels of chlororganic compounds in this organism.

For both organisms the PCDD/F-TEQ ratios at the clay/AC fields ranged 0.14-0.25, which correspond to 75-86% reduced bioaccumulation of total dioxin toxicity.

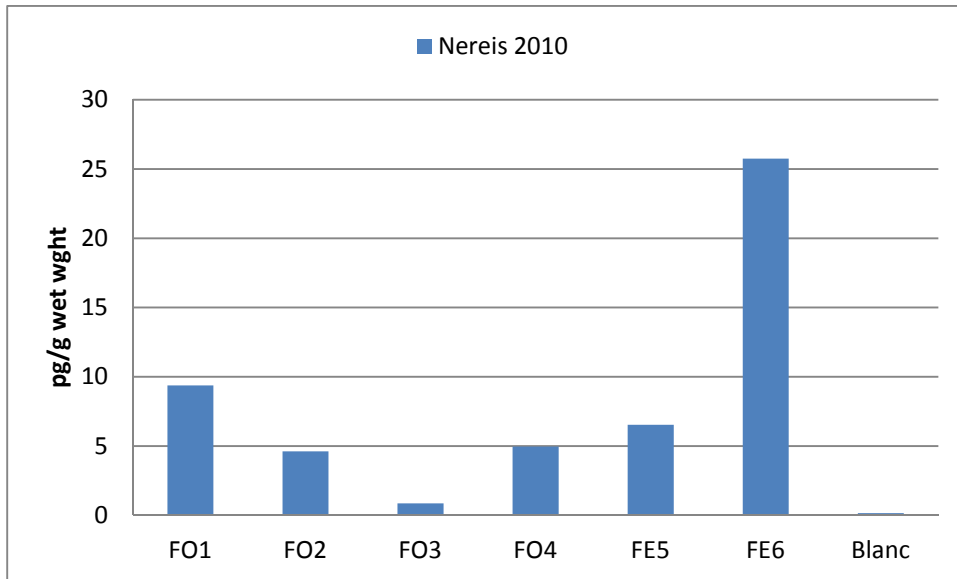


Figure 12. Dioxins (Sum PCDD/F-TEQ) in laboratory blank and *Nereis diversicolor* (polychaete) exposed in box-core samples from experimental fields capped with 1-4 cm thick layers of limestone gravel (FO1), dredged clay (FO2), dredged clay/AC (FO3) and reference field (FO4) in Ormefjorden and dredged clay/AC (FE5) and reference field (FE6) in Eidangerfjorden.

5.2.3 Accumulation in LDPE membranes

Using identical membrane size, stirring rates and water exchange rates in all boxes, the accumulation of dioxins on the membranes should be proportional to the leakage from the sediments to the overlying water. If the sampling rate is low compared to the water exchange rate, some of the dioxins released from the sediments may be lost in the outflowing water. The calculated fluxes should therefore be considered to represent minimum fluxes.

By division of the total accumulation of dioxins with 103 days of exposure and 0.1 m² box area, a flux from sediment of 0.7 pg m⁻² d⁻¹ PCDD/F-TEQ was calculated for the reference field in Ormefjorden and 5 pg m⁻² d⁻¹ for Eidangerfjorden. This was within the order of magnitude expected from the leakage of 7.8 pg m⁻² d⁻¹ previously measured in uncapped sediment from Frierfjorden (Josefsson et al., 2012) using a similar set-up but a different type of passive samplers (SPMD).

The fluxes of 3.1-12.8 pg m⁻² d⁻¹ from the capped fields without AC/amendment were, however, unexpectedly high, yielding 4x higher leakage of PCDD/F-TEQ at the limestone gravel and 18x higher at the clay field, compared to control field.

For the two fields with AC-amended caps, the cap efficiency ratios for PCDD/F-TEQ were 0.22 in Eidangerfjorden and 0.28 in Ormefjorden. This compared well the bioaccumulation data and confirmed the good effect of activated carbon. Again ratios close to 1.0 for HCB (**Table 4**) appeared to indicate different behavior of this compound.

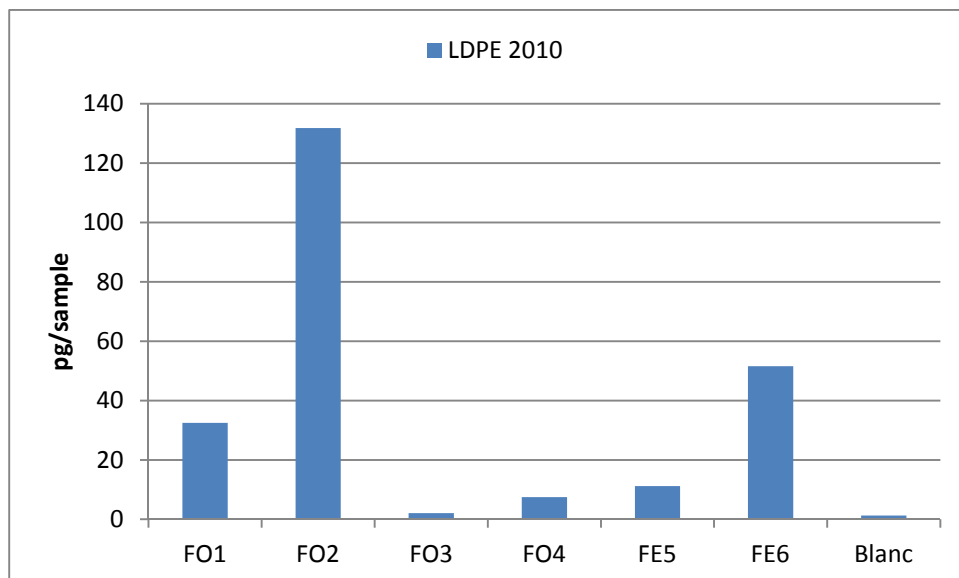


Figure 13. Dioxins (Sum PCDD/F- TEQ) extracted from LDPE membranes exposed in overlying water in box-core samples from experimental fields capped with 1-4 cm thick layers of limestone gravel (FO1), dredged clay (FO2), dredged clay/AC (FO3) and reference field (FO4) in Ormefjorden and dredged clay/AC (FE5) and reference field (FE6) in Eidangerfjorden.

Table 4. Concentration ratios for dioxins, furans, HCB and OCS determined in *Hinia reticulata*, *Nereis diversicolor* and LDPE membranes exposed in box cores transferred from field in October 2009 and November 2010. Ratios are conditionally coloured to highlight good (green) vs. poor (red) cap efficiency. *nd* means not detectable in both fields or reference field. (*value*) means not detectable in capped field.

	Limestone gravel (FO1/FO4)						Dredged clay (FO2/FO4)						Dredged clay/AC (FO3/FO4)						Dredged clay/AC (FE5/FE6)					
	Hinia		Nereis		LDPE		Hinia		Nereis		LDPE		Hinia		Nereis		LDPE		Hinia		Nereis		LDPE	
	2009	2010	2010	2010	2010	2010	2009	2010	2010	2010	2010	2010	2009	2010	2010	2010	2010	2010	2009	2010	2010	2010	2010	2010
2378 TeCDD	0,73	0,48	<i>nd</i>	<i>nd</i>	<i>nd</i>	<i>nd</i>	0,80	0,90	0,91	<i>nd</i>	<i>nd</i>	0,67	(0,15)	<i>nd</i>	<i>nd</i>	0,80	0,28	<i>nd</i>	0,80	0,28	<i>nd</i>	<i>nd</i>	<i>nd</i>	<i>nd</i>
12378 PeCDD	0,09	0,59	(1,34)	<i>nd</i>	<i>nd</i>	<i>nd</i>	0,07	0,86	0,91	<i>nd</i>	<i>nd</i>	0,10	(0,18)	(0,20)	<i>nd</i>	0,19	(0,08)	(0,14)	0,19	(0,08)	(0,14)	(0,14)	(0,00)	(0,00)
123478 HxCDD	0,15	0,55	<i>nd</i>	<i>nd</i>	<i>nd</i>	<i>nd</i>	0,10	0,80	<i>nd</i>	<i>nd</i>	<i>nd</i>	0,13	(0,14)	<i>nd</i>	<i>nd</i>	0,50	0,13	(0,29)	0,50	0,13	(0,29)	(0,29)	0,22	0,22
123678 HxCDD	0,10	0,63	3,38	2,54	<i>nd</i>	<i>nd</i>	0,07	0,90	1,10	9,26	9,26	0,09	(0,10)	(0,36)	(0,29)	0,39	0,19	(0,21)	0,39	0,19	(0,21)	(0,21)	0,18	0,18
123789 HxCDD	0,30	0,73	<i>nd</i>	<i>nd</i>	<i>nd</i>	<i>nd</i>	0,22	(0,58)	<i>nd</i>	<i>nd</i>	<i>nd</i>	0,27	(0,32)	<i>nd</i>	<i>nd</i>	0,35	(0,28)	<i>nd</i>	0,35	(0,28)	<i>nd</i>	<i>nd</i>	0,44	0,44
1234678 HpCDD	0,52	0,82	3,01	2,88	<i>nd</i>	<i>nd</i>	0,48	0,86	1,06	7,18	7,18	0,56	0,30	0,44	0,64	0,52	0,32	0,67	0,52	0,32	0,67	0,67	0,40	0,40
OCDD	0,53	0,91	2,36	2,84	<i>nd</i>	<i>nd</i>	0,39	0,93	0,83	5,99	5,99	0,59	0,39	0,55	1,26	0,48	0,44	0,91	0,48	0,44	0,91	0,91	1,92	1,92
Sum dioxins	0,32	0,82	2,51	2,96	<i>nd</i>	<i>nd</i>	0,26	0,89	0,92	8,53	8,53	0,34	0,32	0,51	0,87	0,45	0,35	0,60	0,45	0,35	0,60	0,60	0,81	0,81
2378 TeCDF	0,06	0,34	0,91	1,55	<i>nd</i>	<i>nd</i>	0,18	0,73	1,03	10,35	10,35	0,08	0,16	0,09	0,09	0,33	0,18	0,42	0,33	0,18	0,42	0,42	0,61	0,61
12378 PeCDF	0,06	0,38	1,41	3,08	<i>nd</i>	<i>nd</i>	0,24	0,65	0,88	24,40	24,40	0,15	0,09	0,10	0,04	0,31	0,12	0,29	0,31	0,12	0,29	0,29	0,25	0,25
23478 PeCDF	0,04	0,38	1,56	5,26	<i>nd</i>	<i>nd</i>	0,10	0,68	0,88	28,18	28,18	0,05	0,10	0,05	0,10	0,26	0,12	0,20	0,26	0,12	0,20	0,20	0,27	0,27
123478 HxCDF	0,09	0,62	2,35	5,03	<i>nd</i>	<i>nd</i>	0,13	0,83	0,91	16,19	16,19	0,13	0,13	0,20	0,29	0,40	0,19	0,26	0,40	0,19	0,26	0,26	0,21	0,21
123678 HxCDF	0,31	0,67	2,26	4,39	<i>nd</i>	<i>nd</i>	0,40	0,87	0,82	15,44	15,44	0,44	0,15	0,22	0,15	0,36	0,19	0,23	0,36	0,19	0,23	0,23	0,17	0,17
234678 HxCDF	0,08	0,65	3,17	4,02	<i>nd</i>	<i>nd</i>	0,19	0,85	0,94	11,69	11,69	0,19	0,15	0,28	0,11	0,39	0,19	0,25	0,39	0,19	0,25	0,25	0,18	0,18
123789 HxCDF	0,15	0,66	2,59	4,39	<i>nd</i>	<i>nd</i>	0,17	0,86	0,98	11,90	11,90	0,17	0,17	0,25	0,48	0,44	0,22	0,34	0,44	0,22	0,34	0,34	0,21	0,21
1234678 HpCDF	0,38	0,83	3,35	6,20	<i>nd</i>	<i>nd</i>	0,35	0,87	1,08	13,47	13,47	0,49	0,23	0,36	0,34	0,49	0,31	0,53	0,49	0,31	0,53	0,53	0,17	0,17
1234789 HpCDF	0,53	0,79	3,10	4,71	<i>nd</i>	<i>nd</i>	0,51	0,89	0,96	14,98	14,98	0,67	0,21	0,35	0,40	0,47	0,29	0,65	0,47	0,29	0,65	0,65	0,17	0,17
OCDF	0,46	0,92	3,09	5,95	<i>nd</i>	<i>nd</i>	0,40	0,92	1,01	13,13	13,13	0,61	0,27	0,40	0,04	0,50	0,39	0,90	0,50	0,39	0,90	0,90	0,23	0,23
Sum furans	0,25	0,78	2,64	4,93	<i>nd</i>	<i>nd</i>	0,27	0,87	0,98	15,06	15,06	0,34	0,22	0,29	0,18	0,43	0,31	0,47	0,43	0,31	0,47	0,47	0,22	0,22
Sum PCDD/F	0,26	0,78	2,63	4,69	<i>nd</i>	<i>nd</i>	0,27	0,87	0,97	14,27	14,27	0,34	0,23	0,31	0,27	0,44	0,31	0,48	0,44	0,31	0,48	0,48	0,27	0,27
Sum WHO 2005 TEQ	0,09	0,54	1,89	4,34	<i>nd</i>	<i>nd</i>	0,14	0,79	0,93	17,62	17,62	0,12	0,14	0,17	0,28	0,33	0,17	0,25	0,33	0,17	0,25	0,25	0,22	0,22
Hexachlorobenzene (HCB)	0,31	0,48	0,84	0,34	<i>nd</i>	<i>nd</i>	0,49	0,79	0,92	0,93	0,93	0,18	0,31	0,16	1,07	0,25	0,34	1,41	0,25	0,34	1,41	1,41	0,88	0,88
Octachlorostyrene (OCS)	0,32	0,45	1,10	1,55	<i>nd</i>	<i>nd</i>	0,68	0,86	1,13	6,14	6,14	0,32	0,17	0,21	0,45	0,38	0,25	0,37	0,38	0,25	0,37	0,37	0,14	0,14

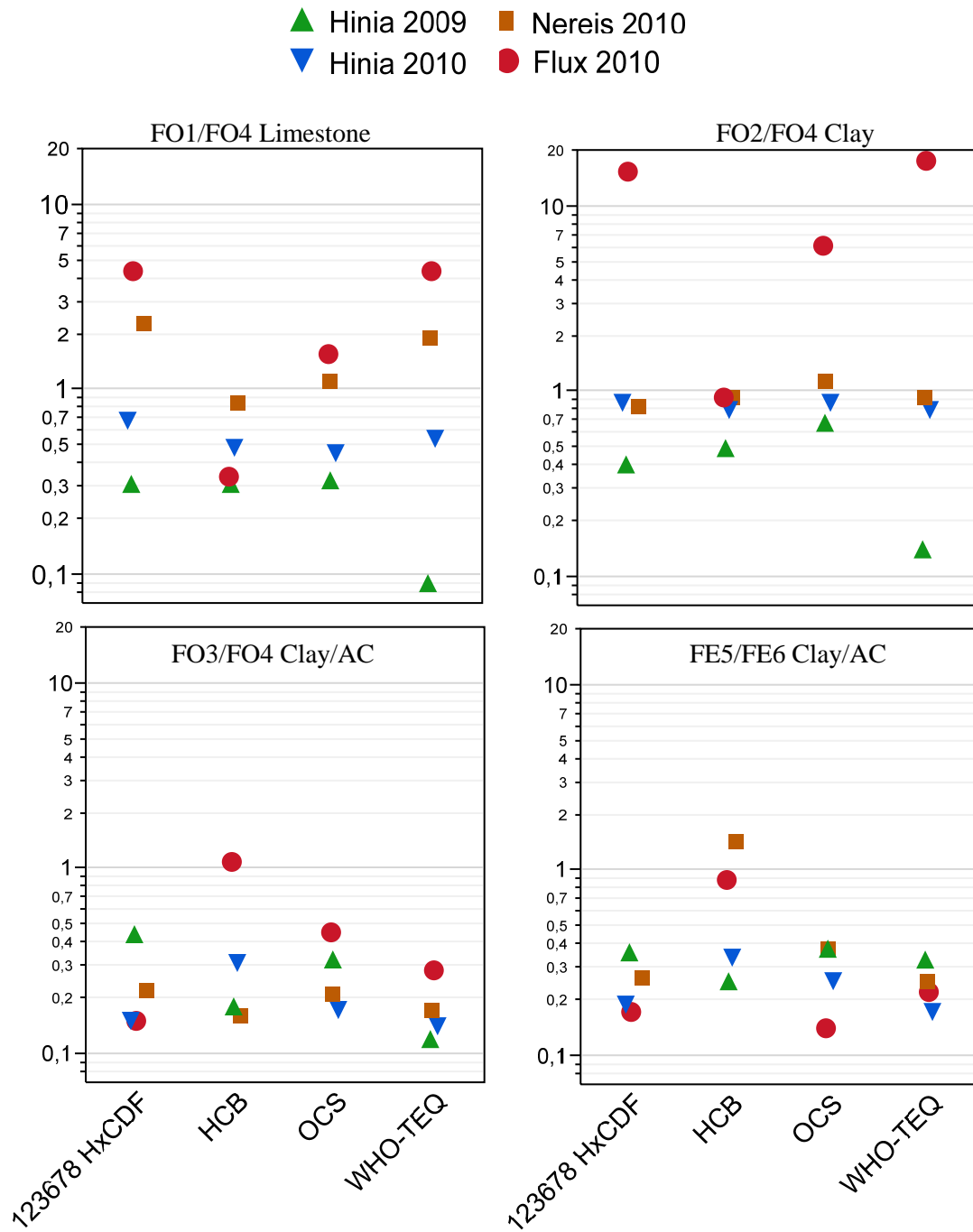


Figure 14. Cap efficiency ratios for HCB, OCS, 123678HxCDF and dioxin toxicity (WHO-TEQ) for all bioavailability indicators determined in box core samples from the Grenland field experiment in 2009 and 2010. Median ratios are given in the table and conditionally coloured to highlight good (green) vs. poor (red) cap efficiency.

5.2.4 ANOVA analyses of cap efficiency

Using JMP v.9 statistical software, a one-way ANOVA analyses was run for log-transformed cap efficiency ratios for all samples for a) dioxin congeners (n=112), b) furan congeners (n=160), c) OCS (n=16), d) HCB (n=16) and PCDD/F-WHO2005-TEQ (n=16). Comparison of mean ratios using Tukey's method showed that for dioxins and furans the cap efficiency ratios at both clay/AC fields were significantly lower than the ratios at both fields without AC (**Figure 15**). For OCS the cap efficiency ratios at the clay/AC fields were significantly lower than the ratios at FO1 but not significantly different from the ratios at FO2. Neither HCB nor PCDD/F-TEQ showed significant differences. The figure shows, however, a big difference between the mean ratios at the clay/AC vs. no-AC fields for PCDD/F-TEQ, but not for HCB. It is important not to confuse the back-transformed mean log ratios with the actual efficiency ratios, but the figure provides an overall view of the main trends observed for the bioavailability of the various groups of compounds.

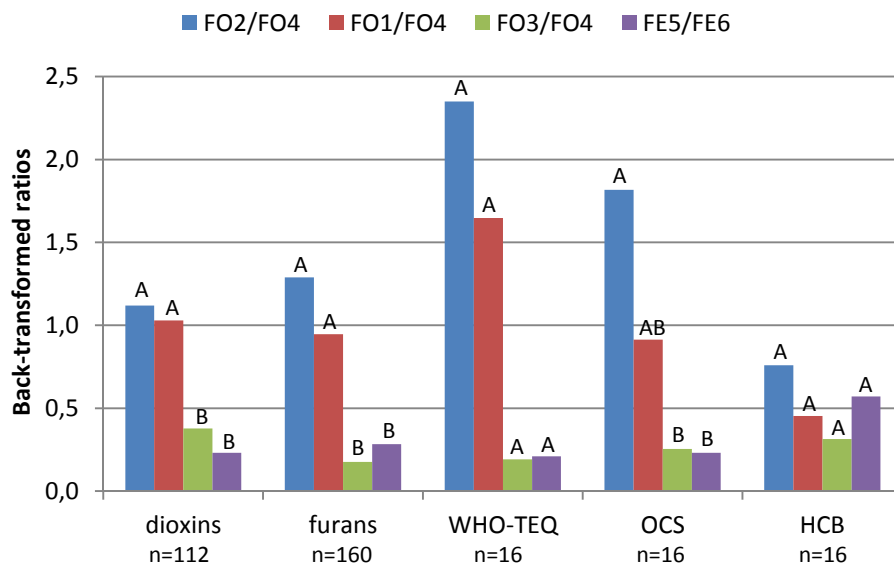


Figure 15. Results of one-way ANOVA analyses with Tukey's comparison of log-transformed cap efficiency ratios. The columns represent the back-transformed mean log ratio ($10^{\text{mean}(\log R)}$). Within each group of compounds, different letters show fields with significantly different ratios. FO1 was capped with limestone gravel, FO2 was capped with dredged clay, FO3 and FE5 were capped with dredged clay and AC, and FO4 and FE6 were uncapped reference fields in Ornefjorden and Eidangerfjorden, respectively.

5.3 Discussion

5.3.1 Unexpectedly high fluxes from FO1 and FO2

Because of the lack of replication and high complexity of the measurements, care should be taken in interpretation of unexpected results such as the high fluxes observed at FO1 and FO2. The polychaete ratios of about 1 indicated that the pore water concentrations were not so much different at FO1, FO2 and the reference field FO4. If the flux is proportional to the product between pore water concentration and the rate of pore water exchange, different rates of pore water exchange appears to be the only possible explanation for large difference in the fluxes. Larger grain size might favour the rate of diffusion through the cap at the limestone gravel field, but small particles will rapidly fill in and

replace pore water in the course cap. Grain size differences are certainly not suitable to explain the high fluxes at the clay field. Compaction and pore water advection generated by the weight of the cap materials is a possible explanation, but not very likely considering the magnitude of the increase (ratios up to 17 at FO2) and the time elapsed between cap placement and these flux measurements. Bioirrigation is a third possible explanation. The fauna report (Schaanning et al., 2011) showed low biomass and number of individuals at FO4 compared to FO1 and FO2, but again the question is whether moderately different macrofauna communities can result in this large difference in fluxes. The silicate and other biogeochemical fluxes (Figure 3-Figure 7) gave no support for a lower rate of pore water exchange at FO4 compared to FO1 and FO2. Thus, until further evidence is brought forward we do not recommend any clear interpretations based on these two measurements.

5.3.2 Deviating behavior of HCB and OCS

When most of the chlororganic compounds showed consistent patterns of accumulation, HCB and to a less extent OCS, was frequently found to deviate. Thus, compared to the highly variable leakage ratios of dioxins and furans, leakage ratios for HCB varied about 1.0 (**Table 4**). Also, the accumulation ratios for HCB and OCS in the surface-dwelling gastropods were occasionally higher than the ratios for dioxins and furans whereas the polychaetes revealed little difference between the various compounds. Differences may result from different environmental behavior due to differences in molecular size, abundances and input pathways. Allan et al. (2011), found indications that the suspended matter (SPM) flowing across the Brevik sill from Frierfjorden to Eidangerfjorden was enriched with HCB, but not PCDD/F, compared to the SPM collected from inflowing water below, and the possibility should not be excluded that sedimentation of new organic material onto the test fields may act as a more important source of HCB and OCS than PCDD/F, for uptake in surface dwelling organisms and leakage to the overlying water.

It may be added that if new input of particulate matter from the watercolumn is generally more important than old sediments and pore water as source of chlororganic compounds for the gastropods, the large difference in leakage (**Figure 13**) and polychaetes (**Figure 12**) between Eidangerfjorden (FE6) and Ornefjorden (FO4), which was most likely explained by different concentrations in sediment and pore water, should not apply to the gastropods, as shown in **Figure 11**.

5.3.3 The importance of AC-amendment

Undoubtedly, the most important finding in this investigation was the importance of activated carbon and the loss of efficiency of the caps without activated carbon. In the mesocosm experiments the cap efficiency ratio could be generally described by

$$R = e^{-b\sqrt{x}}$$

(Josefsson et al. (2012) in which x was cap thickness and b was the efficiency coefficient determined experimentally (e is the base of the natural logarithm). For caps with activated carbon, b was typically about 0.5 for bioaccumulation in gastropods exposed in sediments with AC-amended caps and about 0.3 in caps without AC amendments. This indicated that cap efficiencies of 80% ($R=0.2$) might be obtained either in a 10 mm cap with activated carbon or in a 28 mm cap without activated carbon. This result was very similar to the results obtained for the gastropods exposed in the boxes transferred from the field experiment in 2009. Thus, from the mesocosm experiment and first year field investigation an interesting option might be to replace AC amendment with increased cap thickness.

However, the field experiment clearly demonstrated that AC is required to maintain appropriate cap efficiencies for time periods longer than the 3-6 months perspective of the mesocosm and first year field investigations. In fact, the gastropods showed slightly lower bioaccumulation of most compounds

in 2010 compared to 2009 (**Table 4**). This showed that immobilization of chlororganic compounds is a slow process and might suggest that concentrations of dioxins will continue to decrease as long as binding sites are available on the AC particles.

The loss of cap efficiency at the limestone gravel (FO1) and clay (FO2) fields was most strongly shown by the polychaetes and the passive samplers in the overlying water. Although these samplers were not applied in 2009, the mesocosm experiment showed that within six months after cap placement, caps without AC or any other active substances clearly reduced accumulation in these samplers as well as in the gastropods. If the loss of cap efficiency was a result of recontamination of the cap layers, the gastropods would not be expected to perform different from the polychaetes. If, however, the loss of cap efficiency is the result of increasing concentrations in the pore water, the gastropod, which lives near the sediment surface, may feed selectively on fresh detritus and has a low body exposure to the pore water compared to the polychaetes, the two species might be expected to respond differently. As shown in **Table 4**, frequent gastropod ratios of 0.5-0.9 showed that this organism still benefitted from the caps without AC-amendment, but the polychaete ratio close to 1.0 at FO2 and up to more than 3 at FO1, indicated that the pore water concentrations at these fields were no longer different from the pore water concentrations at the reference field FO4.

The contaminated sediments will continue to release dioxins to the pore water below the caps. Both compaction and diffusion processes may contribute to an upwards migration of the front of pore water gradients and increased leakage when this front breaks through to the overlying water. Essentially similar results would be expected if the flux was driven primarily by pore water exchange through ventilated burrows, as suggested above. In both cases the chemical adsorption of dioxins on activated carbon reduce pore water concentrations within the cap layer and/or within ventilated burrows preventing uptake of dioxins in polychaetes and leakage via pore water exchange to the overlying water.

Continued monitoring of the experimental fields is strongly recommended for documentation of cap performance over longer time periods of 3-5 years.

6. Conclusions

- The applied thin caps had little effect on carbon metabolism in the sediments as indicated by vertical profiles of oxygen and redox potentials in the sediments and fluxes of oxygen, ammonium and nitrate between the sediment and the overlying water.
- Capping with limestone gave a temporary uptake of phosphate (released from all other treatments) and reduced release of silicate nutrients from the sediment.
- Capping with dredged clay had little effects on any of the biogeochemical parameters.
- All applied thin caps effectively reduced bioaccumulation of dioxins (PCDDF-TEQ) in *Hinia reticulata* exposed in box core samples collected in 2009.
- In 2010, only thin caps with activated carbon maintained good performance.
- At the clay/AC fields cap efficiency ratios for dioxin toxicity (PCDD/F WHO2005-TEQ) ranged 0.14-0.28, which correspond to
 - 72 - 78% reduced leakage to the overlying water
 - 75 - 83% reduced bioaccumulation in *Hinia reticulata* and
 - 83 - 86 % reduced bioaccumulation in *Nereis diversicolor*.

7. References

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Appendix A. Dioxin analyses 2009

See samples 3394:4-15 only.

Appendix B. Dioxin analyses LDPE membranes 2010

Appendix C. Dioxin analyses biota 2010

Appendix A



RAPPORT
utförd av ackrediterat laboratorium
REPORT issued by an Accredited Laboratory

ISO/IEC 17025

Umeå 2010-06-24

Mpr 3394
Sida 1(22)

UMEÅ UNIVERSITET

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Resultat från analys av polyklorerade dibenso-*p*-dioxiner, polyklorerade dibensofuraner (PCDD/F), hexaklorbensen (HCB) och oktaklorstyren (OCS) i biologiska prov (*Hinia reticulata*) och SPMD.

Laboratorier ackrediteras av Styrelsen för ackreditering och teknisk kontroll (SWEDAC) enligt svensk lag. Den ackrediterade verksamheten vid laboratorierna uppfyller kraven i SS-EN ISO/IEC 17 025 (2005).

Förkortningar och definitioner i analysrapporten

TrC-	Triklor (3 klor)	-DD	Dibenso- <i>p</i> -dioxin(er)
TeC-	Tetraklor (4 klor)	-DF	Dibensofuran(er)
PeC-	Pentaklor (5 klor)	-B	Bifenyl(er)
HxC-	Hexaklor (6 klor)		
HpC-	Heptaklor (7 klor)		
OC-	Oktaklor (8 klor)		
ng	nanogram (10 ⁻⁹ gram)		
pg	pikogram (10 ⁻¹² gram)		
fg	femtogram (10 ⁻¹⁵ gram)		
IS	Internstandard		
fw	Färskvikt (fresh weight)		
lw	Fettvikt (lipid weight)		
ND	Icke-detekterad (Not Detected)		
LOD	Detektionsgräns (Limit-of-Detection)		
TEF	Toxisk ekvivalentfaktor (Toxic Equivalency Factor)		
TEQ	Total koncentration i TCDD-ekvivalenter (TCDD toxic equivalent concentration)		

Metodbeskrivning

Använda uppberedningsmetoder är väl validerade genom ett flertal internationella inter-kalibreringar och GC-MS analyserna utförs enligt Svensk standard SS-EN 1948:1-3. De genomförda analyserna uppfyller även kvalitetskraven för analys av dioxiner och dioxinlika PCB, EU-direktiv 2002/69/EC, som publicerades i EUs officiella tidskrift (Official journal of the European Communities) den 30 juli 2002. En sammanfattning av analysmetoderna följer nedan.

Extraktion och fettviktsbestämning

Före extraktionen tillsattes internstandard bestående av ^{13}C -anrikade isotoper av merparten av de ämnen (kongener) som skall bestämmas. Proven extraherades med organiska lösningsmedel. Lösningsmedlet avlägsnades genom indunstning och mängden fett bestämdes genom vägning.

Upprening

Uppreningen av polyklorerade dibenso-p-dioxiner (PCDD), polyklorerade dibensofuraner (PCDF), hexaklorbensen (HCB) och oktaklorstyren (OCS) utfördes med två vätskekromatografikolonner: en flerskiktsskolonn bestående av kiselgel, svavelsyra- och kaliumhydroxidimpregnerad kiselgel samt en kolonn med aktivt kol. På den sistnämnda separeras provet i två fraktioner innehållande 1) OCS, 2) PCDD/F och HCB. Innan den slutliga analysen tillsattes ytterligare ^{13}C -kongener, sk. återfinningsstandarder.

Analys

Isomerspecifik analys har skett med gaskromatografi (GC) kopplat till masspektrometri (MS). Separationen av ämnena sker på GC:n och detektionen med masspektrometern. Vid MS-analysen detekterades ämnen med olika masstal selektivt vilket möjliggjorde utnyttjandet av syntetiska ^{13}C -isotopanrikade ämnen (^{13}C -kongener) vilka användes som interna standarder med så kallad isotoputspädningsmetodik. Härvidlag jämfördes responskvoten mellan naturliga kongener och ^{13}C -kongener i provet med motsvarande kvot i en kvantifieringsstandard innehållande kända mängder av naturliga och tillsatta ^{13}C -kongener. Detta förfarande medförde att resultaten automatiskt blev kompenserade för uppberedningsförluster. En MS (Waters Autospec Ultima) med hög massupplösning (~10000) har använts. Den använde elektronstötjonisering (EI) där sedan utvalda joner registrerades (SIR). Koncentrationsbestämningen har utförts enligt ovan nämnda norm, SS-EN 1948:3 och återfinningsgraden av de internstandarder (IS) som tillsatts proven beräknas och uttrycks i procent av ursprunglig mängd.

När en kongen ej kan detekteras räknas detektionsgränsen ut (LOD – limit of detection). Den motsvarar en signal från analysinstrumentet som är tre gånger högre än brusnivån och anges som ett mindre än-värde. LOD beror av ett antal faktorer och varierar därför något från prov till prov, mellan olika kongener och från ett

analystillfälle till ett annat. Ackrediterade resultat kan endast fås ner till kvantifieringsgränsen (LOQ – limit of quantification) som definieras av signaler som överstiger tio gånger brusnivån. Det framgår av analysrapporten för vilka kongener detta kriterium inte är uppfyllt. I området mellan tre och tio gånger brusnivån är mätosäkerheten förhöjd men ger ändå ett värdefullt bidrag till resultaten och TEQ-beräkningen.

Laboratorieblankens koncentration har redovisats separat, ingen subtraktion har gjorts från de verkliga provens koncentrationer. Normalisering har gjorts till samma enhet som för proven med medelvärdet av de provmängder som använts till dessa.

Beräkning av TCDD-ekvivalenter (TEQ)

Utifrån de enskilda kongenernas koncentration har s.k. TCDD-ekvivalenter (TEQ) beräknats. TCDD-ekvivalenterna relaterar de toxiska kongenerna till den mest toxiska, 2,3,7,8-TeCDD.

$$\text{TEQ} = \text{koncentration} \times \text{TEF}$$

Det finns ett antal olika TEF-skalar som använts genom åren. Idag är WHO-TEF-skalan den vedertagna men resultaten kan omräknas enligt den skala som önskas (tabell 1, sid. 5).

När en kongen ej kan detekteras räknas detektionsgränsen ut. Den motsvarar en signal från analysinstrumentet som är tre gånger högre än brusnivån. Detektionsgränsen beror av ett antal faktorer och varierar därför något från prov till prov, mellan olika kongener och från ett analystillfälle till ett annat.

TEQ beräknas på tre nivåer. En nedre koncentrationsgräns där koncentrationerna av icke detekterade ämnen satts till noll, en övre koncentrationsgräns där koncentrationerna av icke detekterade ämnen ersatts med detektionsgränsen samt en medelkoncentration (medelvärdet av de båda).

En sammanfattning av analysrapporten finns på sidan 6-8 med WHO-TEQ-värden för samtliga prov och ev kommentarer.

Umeå som ovan,

Maria Hjelt,
Laboratorieingenjör

Tabell 1. Skalor för beräkning av toxiska ekvivalenter (TEQ):

Kongen	TEF			
	WHO ₂₀₀₅	WHO ₁₉₉₈	Internationella	Nordic
2378-TeCDD	1	1	1	1
12378-PeCDD	1	1	0,5	0,5
123478-HxCDD	0,1	0,1	0,1	0,1
123678-HxCDD	0,1	0,1	0,1	0,1
123789-HxCDD	0,1	0,1	0,1	0,1
1234678-HpCDD	0,01	0,01	0,01	0,01
OCDD	0,0003	0,0001	0,001	0,001
2378-TeCDF	0,1	0,1	0,1	0,1
12378-PeCDF	0,03	0,05	0,05	0,01
23478-PeCDF	0,3	0,5	0,5	0,5
123478-HxCDF	0,1	0,1	0,1	0,1
123678-HxCDF	0,1	0,1	0,1	0,1
123789-HxCDF	0,1	0,1	0,1	0,1
234678-HxCDF	0,1	0,1	0,1	0,1
1234678-HpCDF	0,01	0,01	0,01	0,01
1234789-HpCDF	0,01	0,01	0,01	0,01
OCDF	0,0003	0,0001	0,001	0,001

Resultatsammanställning

Vår provmärkning:	3394:1	3394:2	3394:3	3394:18
Er provmärkning:	Boks 10 18/5-25/5	Boks 8 5/4-12/4	Boks 9 18/5-25/5	Boks 8 4/6-2/7
Provtyp:	SPMD	SPMD	SPMD	SPMD
Mängd analyserat prov färskvikt:	-	-	-	-
Enhet:	pg/prov	pg/prov	pg/prov	pg/prov
PCDD och PCDF:				
Σ WHO 2005 TEQ _{diox}				
Övre koncentration	492	7.6	176	8.8
Medel koncentration	492	3.9	176	6.2
Nedre koncentration	492	0.26	176	3.6
HCB:	414000	1720	264000	6320
Återfunnen ¹³C IS(%)	77	61	80	66
OCS:	92800	59	56300	171
Återfunnen ¹³C IS(%)	91	99	94	101
PRC (kvot):				
PCB 8	15	16	17	15
PCB 37	14	16	14	17
PCB 54	38	40	42	41

Vår provmärkning:	3394:19	3394:20	3394:4+5	3394:6+7
Er provmärkning:	Boks 9 4/6-2/7	Boks 10 4/6-2/7	FO1A+B	FO2A+B
Provtyp:	SPMD	SPMD	Hinia	Hinia
Mängd analyserat prov färskvikt:	-	-	17.177	17.982
Enhet:	pg/prov	pg/prov	pg/g	pg/g
PCDD och PCDF:				
Σ WHO 2005 TEQ _{diox}				
Övre koncentration	343	818	0.65	0.88
Medel koncentration	343	818	0.49	0.75
Nedre koncentration	343	818	0.33	0.61
HCB:	294000	401000	19	30
Återfunnen ¹³C IS(%)	72	79	77	64
OCS:	89300	77200	1.4	3.0
Återfunnen ¹³C IS(%)	95	101	110	111
PRC (kvot):				
PCB 8	17	18	-	-
PCB 37	17	16	-	-
PCB 54	49	45	-	-

Vår provmärkning:	3394:8+9	3394:10+11	3394:12+13	3394:14+15
Er provmärkning:	FO3A+B	FO4A+B	FE5A+B	FE6A+B
Provtyp:	Hinia	Hinia	Hinia	Hinia
Mängd analyserat prov färskvikt:	18.313	22.727	13.057	25.278
Enhet:	pg/g	pg/g	pg/g	pg/g
PCDD och PCDF:				
Σ WHO 2005 TEQ _{diox}				
Övre koncentration	0.78	5.2	1.6	4.3
Medel koncentration	0.63	5.2	1.4	4.3
Nedre koncentration	0.48	5.1	1.2	4.3
HCB:	11	61	29	117
Återfunnen ¹³C IS(%)	64	62	77	68
OCS:	1.4	4.4	18	48
Återfunnen ¹³C IS(%)	105	125	119	127
PRC (kvot):				
PCB 8	-	-	-	-
PCB 37	-	-	-	-
PCB 54	-	-	-	-

Kommentar:

HCB har hittats i båda fraktionerna för proven 3394: 1-3, 18-20 (SPMD).

Laboratorieblank 3394:B1 är upparbetad med proven 3394: 4+5, 6+7, 8+9, 10+11, 12+13 och 14+15.

Laboratorieblank 3394:B2 är upparbetad med proven 3394: 1-3, 18-20.

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:1
 Er provmärkning : Boks 10, 18/5-25/5
 Provtyp : SPMD
 Mängd analyserat prov (g) : -
 Fettvikt (g) : -
 Provsort : pg/prov
 Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/prov)	Återfunnen ¹³ C IS (%)
2378 TeCDD	15	94%
12378 PeCDD	57	101%
123478 HxCDD	30	100%
123678 HxCDD	48	97%
123789 HxCDD	25	107%
1234678 HpCDD	85	95%
OCDD	78	92%
2378 TeCDF	705	92%
12378 PeCDF	527	98%
23478 PeCDF ^a	473	87%
123478 HxCDF	803	91%
123678 HxCDF	466	92%
234678 HxCDF	234	85%
123789 HxCDF ^b	179	85%
1234678 HpCDF	916	86%
1234789 HpCDF	291	88%
OCDF	1580	90%
Sum	övre konc.	492
WHO-	medelkonc.	492
TEQ₂₀₀₅	nedre konc.	492

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-06-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:2
Er provmärkning : Boks 8, 5/4-12/4
Provtyp : SPMD
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(2.0)	94%
12378 PeCDD	ND(2.5)	97%
123478 HxCDD	ND(3.5)	99%
123678 HxCDD	ND(3.6)	98%
123789 HxCDD	ND(3.6)	108%
1234678 HpCDD	4.8	91%
OCDD	11	88%
2378 TeCDF	1.7	98%
12378 PeCDF	ND(2.4)	96%
23478 PeCDF ^a	ND(2.7)	89%
123478 HxCDF	ND(2.4)	94%
123678 HxCDF	ND(2.4)	94%
234678 HxCDF	ND(1.6)	84%
123789 HxCDF ^b	ND(1.6)	85%
1234678 HpCDF	3.8	85%
1234789 HpCDF	ND(1.8)	89%
OCDF	7.9	86%
Sum	övre konc.	7.6
WHO-TEQ₂₀₀₅	medelkonc.	3.9
	nedre konc.	0.26

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppabetning:	2010-06-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:3
Er provmärkning : Boks 9, 18/5-25/5
Provtyp : SPMD
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)	Återfunnen ¹³ C IS (%)
2378 TeCDD	3.7	94%
12378 PeCDD	18	100%
123478 HxCDD	14	94%
123678 HxCDD	20	97%
123789 HxCDD	10	102%
1234678 HpCDD	46	90%
OCDD	54	86%
2378 TeCDF	194	89%
12378 PeCDF	165	101%
23478 PeCDF ^a	165	91%
123478 HxCDF	331	91%
123678 HxCDF	194	92%
234678 HxCDF	97	87%
123789 HxCDF ^b	71	85%
1234678 HpCDF	484	84%
1234789 HpCDF	133	87%
OCDF	938	83%
Sum	övre konc.	176
WHO-	medelkonc.	176
TEQ₂₀₀₅	nedre konc.	176

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-06-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:4+5
Er provmärkning : FO1A+B
Provtyp : Biota
Mängd analyserat prov (g) : 17.177
Fettvikt (g) : 0.1332
Provsort : pg/g
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.11)		78%
12378 PeCDD	ND(0.13)		92%
123478 HxCDD	ND(0.20)		89%
123678 HxCDD	ND(0.19)		92%
123789 HxCDD	ND(0.19)		91%
1234678 HpCDD	0.44		92%
OCDD	0.79		88%
2378 TeCDF	0.32		89%
12378 PeCDF	0.24		96%
23478 PeCDF ^a	0.21		93%
123478 HxCDF	1.0		92%
123678 HxCDF	0.47		96%
234678 HxCDF	ND(0.30)		92%
123789 HxCDF ^b	0.28		92%
1234678 HpCDF	2.7		93%
1234789 HpCDF	0.80		88%
OCDF	8.3		83%
Sum	övre konc.	0.65	
WHO-	medelkonc.	0.49	
TEQ₂₀₀₅	nedre konc.	0.33	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppärbetning:	2010-05-07	Uppärbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:6+7
Er provmärkning : FO2A+B
Provtyp : Biota
Mängd analyserat prov (g) : 17.982
Fettvikt (g) : 0.1071
Provsort : pg/g
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.12)	69%
12378 PeCDD	ND(0.11)	87%
123478 HxCDD	ND(0.14)	88%
123678 HxCDD	ND(0.14)	84%
123789 HxCDD	ND(0.14)	85%
1234678 HpCDD	0.40	86%
OCDD	0.59	81%
2378 TeCDF	0.96	87%
12378 PeCDF	0.89	92%
23478 PeCDF ^a	0.61	89%
123478 HxCDF	1.4	90%
123678 HxCDF	<i>0.60</i>	90%
234678 HxCDF	<i>0.36</i>	90%
123789 HxCDF ^b	<i>0.33</i>	89%
1234678 HpCDF	2.5	88%
1234789 HpCDF	0.77	84%
OCDF	7.2	78%
Sum	övre konc.	0.88
WHO-	medelconc.	0.75
TEQ₂₀₀₅	nedre konc.	0.61

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-05-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:8+9
Er provmärkning : FO3A+B
Provtyp : Biota
Mängd analyserat prov (g) : 18.313
Fettvikt (g) : 0.1267
Provsort : pg/g
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.10)	63%
12378 PeCDD	ND(0.15)	68%
123478 HxCDD	ND(0.18)	69%
123678 HxCDD	ND(0.17)	70%
123789 HxCDD	ND(0.17)	70%
1234678 HpCDD	0.47	71%
OCDD	0.88	71%
2378 TeCDF	0.40	74%
12378 PeCDF	0.54	75%
23478 PeCDF ^a	0.31	77%
123478 HxCDF	1.4	74%
123678 HxCDF	0.66	75%
234678 HxCDF	0.36	74%
123789 HxCDF ^b	0.33	71%
1234678 HpCDF	3.5	74%
1234789 HpCDF	1.0	73%
OCDF	11	71%
Sum	övre konc.	0.78
WHO-	medelkonc.	0.63
TEQ₂₀₀₅	nedre konc.	0.48

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-05-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:10+11
 Er provmärkning : FO4A+B
 Provtyp : Biota
 Mängd analyserat prov (g) : 22.727
 Fettvikt (g) : 0.1247
 Provsort : pg/g
 Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.15)	77%
12378 PeCDD	0.75	82%
123478 HxCDD	0.67	83%
123678 HxCDD	1.0	82%
123789 HxCDD	0.32	82%
1234678 HpCDD	0.84	84%
OCDD	1.5	82%
2378 TeCDF	5.3	90%
12378 PeCDF	3.7	89%
23478 PeCDF ^a	5.9	92%
123478 HxCDF	11	89%
123678 HxCDF	1.5	88%
234678 HxCDF	1.9	88%
123789 HxCDF ^b	1.9	87%
1234678 HpCDF	7.1	89%
1234789 HpCDF	1.5	85%
OCDF	18	81%
Sum	övre konc.	5.2
WHO-	medelkonc.	5.2
TEQ₂₀₀₅	nedre konc.	5.1

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-05-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:12+13
Er provmärkning : FE5A+B
Provtyp : Biota
Mängd analyserat prov (g) : 13.057
Fettvikt (g) : 0.0866
Provsort : pg/g
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.16)		74%
12378 PeCDD	ND(0.20)		85%
123478 HxCDD	0.16		83%
123678 HxCDD	0.23		82%
123789 HxCDD	ND(0.16)		81%
1234678 HpCDD	0.68		87%
OCDD	1.0		86%
2378 TeCDF	1.5		88%
12378 PeCDF	1.6		91%
23478 PeCDF ^a	1.1		91%
123478 HxCDF	2.8		89%
123678 HxCDF	1.5		90%
234678 HxCDF	0.78		92%
123789 HxCDF ^b	0.62		88%
1234678 HpCDF	4.9		93%
1234789 HpCDF	1.6		87%
OCDF	14		86%
Sum	övre konc.	1.6	
WHO-	medelkonc.	1.4	
TEQ₂₀₀₅	nedre konc.	1.2	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppärbetning:	2010-05-07	Uppärbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:14+15
Er provmärkning : FE6A+B
Provtyp : Biota
Mängd analyserat prov (g) : 25.278
Fettvikt (g) : 0.1494
Provsort : pg/g
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)		Återfunnen ¹³ C IS (%)
2378 TeCDD	0.10		76%
12378 PeCDD	0.54		81%
123478 HxCDD	<i>0.32</i>		83%
123678 HxCDD	<i>0.59</i>		79%
123789 HxCDD	<i>0.23</i>		80%
1234678 HpCDD	1.3		81%
OCDD	2.1		80%
2378 TeCDF	4.5		87%
12378 PeCDF	5.2		87%
23478 PeCDF ^a	4.3		88%
123478 HxCDF	7.0		88%
123678 HxCDF	4.2		87%
234678 HxCDF	2.0		84%
123789 HxCDF ^b	1.4		82%
1234678 HpCDF	10		85%
1234789 HpCDF	3.4		83%
OCDF	28		82%
Sum	övre konc.	4.3	
WHO-	medelkonc.	4.3	
TEQ₂₀₀₅	nedre konc.	4.3	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppärbetning:	2010-05-07	Uppärbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:18
Er provmärkning : Boks 8, 4/6-2/7
Provtyp : SPMD
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(2.6)	92%
12378 PeCDD	ND(1.3)	90%
123478 HxCDD	ND(4.1)	89%
123678 HxCDD	ND(4.1)	94%
123789 HxCDD	ND(4.1)	94%
1234678 HpCDD	6.3	86%
OCDD	7.6	71%
2378 TeCDF	3.1	97%
12378 PeCDF	3.9	96%
23478 PeCDF ^a	3.4	89%
123478 HxCDF	8.4	89%
123678 HxCDF	4.4	86%
234678 HxCDF	3.1	93%
123789 HxCDF ^b	3.1	85%
1234678 HpCDF	15	77%
1234789 HpCDF	4.6	79%
OCDF	29	53%
Sum	övre konc.	8.8
WHO- TEQ₂₀₀₅	medelkonc.	6.2
	nedre konc.	3.6

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppärbetning:	2010-06-07	Uppärbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:19
 Er provmärkning : Boks 9, 4/6-2/7
 Provtyp : SPMD
 Mängd analyserat prov (g) : -
 Fettvikt (g) : -
 Provsort : pg/prov
 Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)	Återfunnen ¹³ C IS (%)
2378 TeCDD	7.0	88%
12378 PeCDD	37	87%
123478 HxCDD	24	87%
123678 HxCDD	41	89%
123789 HxCDD	21	90%
1234678 HpCDD	84	89%
OCDD	86	71%
2378 TeCDF	390	83%
12378 PeCDF	336	96%
23478 PeCDF ^a	315	87%
123478 HxCDF	632	88%
123678 HxCDF	378	87%
234678 HxCDF	182	96%
123789 HxCDF ^b	150	89%
1234678 HpCDF	885	83%
1234789 HpCDF	263	86%
OCDF	1663	62%
Sum	övre konc.	343
WHO-	medelkonc.	343
TEQ₂₀₀₅	nedre konc.	343

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-06-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:20
Er provmärkning : Boks 10, 4/6-2/7
Provtyp : SPMD
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	24		94%
12378 PeCDD	88		94%
123478 HxCDD	50		89%
123678 HxCDD	80		93%
123789 HxCDD	44		97%
1234678 HpCDD	165		94%
OCDD	160		79%
2378 TeCDF	1178		88%
12378 PeCDF	836		102%
23478 PeCDF ^a	751		95%
123478 HxCDF	1393		89%
123678 HxCDF	798		93%
234678 HxCDF	409		101%
123789 HxCDF ^b	341		95%
1234678 HpCDF	1809		90%
1234789 HpCDF	587		90%
OCDF	3427		82%
Sum	övre konc.	818	
WHO-	medelkonc.	818	
TEQ₂₀₀₅	nedre konc.	818	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2010-10-21	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-06-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:B1
 Er provmärkning : -
 Provtyp : Laboratorieblank
 Mängd analyserat prov (g) : "19.089"
 Fettvikt (g) : -
 Provsort : pg/g
 Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)		Återfunnen ¹³ C IS (%)	
2378 TeCDD	ND(0.049)		79%	
12378 PeCDD	ND(0.095)		87%	
123478 HxCDD	ND(0.12)		89%	
123678 HxCDD	ND(0.057)		91%	
123789 HxCDD	ND(0.057)		91%	
1234678 HpCDD	ND(0.082)		90%	
OCDD	0.046		85%	
2378 TeCDF	ND(0.024)		85%	
12378 PeCDF	ND(0.060)		91%	
23478 PeCDF ^a	ND(0.065)		91%	
123478 HxCDF	ND(0.067)		91%	
123678 HxCDF	ND(0.067)		93%	
234678 HxCDF	ND(0.048)		91%	
123789 HxCDF ^b	0.058		92%	
1234678 HpCDF	ND(0.082)		94%	
1234789 HpCDF	ND(0.082)		89%	
OCDF	0.062		81%	
Sum	övre konc.	0.22	HCB: 1.6	75%
WHO- TEQ₂₀₀₅	medelkonc.	0.11	OCS: 0.094	99%
	nedre konc.	0.006		

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:		Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-05-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3394:B2
Er provmärkning : -
Provtyp : Laboratorieblank
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : $\pm 26\%$ (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)	
2378 TeCDD	ND(1.1)		93%	
12378 PeCDD	ND(1.3)		98%	
123478 HxCDD	ND(2.3)		99%	
123678 HxCDD	ND(2.3)		96%	
123789 HxCDD	ND(2.3)		97%	
1234678 HpCDD	<i>1.8</i>		98%	
OCDD	<i>0.83</i>		92%	
2378 TeCDF	ND(0.59)		94%	
12378 PeCDF	ND(0.71)		100%	
23478 PeCDF ^a	ND(0.35)		100%	
123478 HxCDF	ND(0.89)		98%	
123678 HxCDF	ND(0.81)		100%	
234678 HxCDF	ND(1.1)		96%	
123789 HxCDF ^b	<i>1.1</i>		97%	
1234678 HpCDF	ND(1.6)		99%	
1234789 HpCDF	ND(1.6)		94%	
OCDF	ND(0.28)		88%	
Sum	övre konc.	3.7	HCB: 28	51%
WHO- TEQ₂₀₀₅	medelkonc.	1.9	OCS: 4.4	98%
	nedre konc.	0.12		

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:		Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2010-06-07	Upparbetning:	Maria Hjelt
Startdatum för analys:	2010-06-19	Analys:	Maria Hjelt

Appendix B



RAPPORT

utfärdad av ackrediterat laboratorium

REPORT issued by an Accredited Laboratory

ISO/IEC 17025

Umeå 2011-09-29

Mpr 3469

Sida 1(14)

UMEÅ UNIVERSITET

Kemiska institutionen,
Miljökemiska Laboratoriet
Anna Kitti-Sjöström
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Norwegian Institute for Water Research - NIVA
Morten Schaanning
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Resultat från analys av polyklorerade dibenso-*p*-dioxiner, polyklorerade dibensofuraner (PCDD/F), hexaklorbensen (HCB) och oktaklorstyren (OCS) i LDPE membran.

Laboratorier ackrediteras av Styrelsen för ackreditering och teknisk kontroll (SWEDAC) enligt svensk lag. Den ackrediterade verksamheten vid laboratorierna uppfyller kraven i SS-EN ISO/IEC 17 025 (2005).

Förkortningar och definitioner i analysrapporten

TrC-	Triklor (3 klor)	-DD	Dibenso- <i>p</i> -dioxin(er)
TeC-	Tetraklor (4 klor)	-DF	Dibensofuran(er)
PeC-	Pentaklor (5 klor)	-B	Bifenyl(er)
HxC-	Hexaklor (6 klor)		
HpC-	Heptaklor(7 klor)		
OC-	Oktaklor (8 klor)		

ng nanogram(10^{-9} gram)pg pikogram (10^{-12} gram)fg femtogram (10^{-15}
gram)

IS Internstandard

fw Färskvikt (fresh weight)

lw Fettvikt (lipid weight)

ND Icke-detekterad (Not Detected)

LOD Detektionsgräns (Limit-of-Detection)

TEF Toxisk ekvivalentfaktor (Toxic Equivalency Factor)

TEQ Total koncentration i TCDD-ekvivalenter (TCDD toxic equivalent concentration)

Metodbeskrivning

Använda uppberedningsmetoder är väl validerade genom ett flertal internationella inter-kalibreringar och GC-MS analyserna utförs enligt Svensk standard SS-EN 1948:1-3. De genomförda analyserna uppfyller även kvalitetskraven för analys av dioxiner och dioxinlika PCB, EU-direktiv 2002/69/EC, som publicerades i EUs officiella tidskrift (Official journal of the European Communities) den 30 juli 2002. En sammanfattning av analysmetoderna följer nedan.

Extraktion och fettviktsbestämning

Före extraktionen tillsattes internstandard bestående av ^{13}C -anrikade isotoper av merparten av de ämnen (kongener) som skall bestämmas. Proven extraherades med organiska lösningsmedel. Lösningsmedlet avlägsnades genom indunstning.

Upprening

Uppreningen av polyklorerade dibenso-p-dioxiner (PCDD), polyklorerade dibensofuraner (PCDF), hexaklorbensen (HCB) och oktaklorstyren (OCS) utfördes med två vätskekromatografikolonner: en flerskiktsskolonn bestående av kiselgel, svavelsyra- och kaliumhydroxidimpregnerad kiselgel samt en kolonn med aktivt kol. På den sistnämnda separeras provet i två fraktioner innehållande 1) OCS och HCB, 2) PCDD/F. Innan den slutliga analysen tillsattes ytterligare ^{13}C -kongener, sk. återfinningsstandarder.

Analys

Isomerspecifik analys har skett med gaskromatografi (GC) kopplat till masspektrometri (MS). Separationen av ämnena sker på GC:n och detektionen med masspektrometern. Vid MS-analysen detekterades ämnen med olika masstal selektivt vilket möjliggjorde utnyttjandet av syntetiska ^{13}C -isotopanrikade ämnen (^{13}C -kongener) vilka användes som interna standarder med så kallad isotoputspädningsmetodik. Härvidlag jämfördes responskvoten mellan naturliga kongener och ^{13}C -kongener i provet med motsvarande kvot i en kvantifieringsstandard innehållande kända mängder av naturliga och tillsatta ^{13}C -kongener. Detta förfarande medförde att resultaten automatiskt blev kompenserade för uppberedningsförluster. En MS (Waters Autospec Ultima) med hög massupplösning (~10000) har använts. Den använde elektronstötjonisering (EI) där sedan utvalda joner registrerades (SIR).

Koncentrationsbestämningen har utförts enligt ovan nämnda norm, SS-EN 1948:3 och återfinningsgraden av de internstandarder (IS) som tillsatts proven beräknas och uttrycks i procent av ursprunglig mängd.

När en kongen ej kan detekteras räknas detektionsgränsen ut (LOD – limit of detection). Den motsvarar en signal från analysinstrumentet som är tre gånger högre än brusnivån och anges som ett mindre än-värde. LOD beror av ett antal faktorer och varierar därför något från prov till prov, mellan olika kongener och från ett analystillfälle till ett annat. Ackrediterade resultat kan endast fås ner till

kvantifieringsgränsen (LOQ – limit of quantification) som defineras av signaler som överstiger tio gånger brusnivån. Det framgår av analysrapporten för vilka kongener detta kriterium inte är uppfyllt. I området mellan tre och tio gånger brusnivån är mätosäkerheten förhöjd men ger ändå ett värdefullt bidrag till resultaten och TEQ-beräkningen.

Laboratorieblankens koncentration har redovisats separat, ingen subtraktion har gjorts från de verkliga provens koncentrationer. Normalisering har gjorts till samma enhet som för proven med medelvärdet av de provmängder som använts till dessa.

Beräkning av TCDD-ekvivalenter (TEQ)

Utifrån de enskilda kongenernas koncentration har s.k. TCDD-ekvivalenter (TEQ) beräknats. TCDD-ekvivalenterna relaterar de toxiska kongenerna till den mest toxiska, 2,3,7,8-TeCDD.

$$\text{TEQ} = \text{koncentration} \times \text{TEF}$$

Det finns ett antal olika TEF-skalar som använts genom åren. Idag är WHO-TEF-skalan den vedertagna men resultaten kan omräknas enligt den skala som önskas (tabell 1, sid. 5).

När en kongen ej kan detekteras räknas detektionsgränsen ut. Den motsvarar en signal från analysinstrumentet som är tre gånger högre än brusnivån. Detektionsgränsen beror av ett antal faktorer och varierar därför något från prov till prov, mellan olika kongener och från ett analystillfälle till ett annat.

TEQ beräknas på tre nivåer. En nedre koncentrationsgräns där koncentrationerna av icke detekterade ämnen satts till noll, en övre koncentrationsgräns där koncentrationerna av icke detekterade ämnen ersatts med detektionsgränsen samt en medelkoncentration (medelvärdet av de båda).

En sammanfattning av analysrapporten finns på sidan 6-8 med WHO-TEQ-värden för samtliga prov och ev kommentarer.

Umeå som ovan,



Anna Kitti-Sjöström,
Forskningsingenjör

Tabell 1. Skalar för beräkning av toxiska ekvivalenter (TEQ):

Kongen	TEF			
	WHO ₂₀₀₅	WHO ₁₉₉₈	Internationella	Nordic
2378-TeCDD	1	1	1	1
12378-PeCDD	1	1	0,5	0,5
123478-HxCDD	0,1	0,1	0,1	0,1
123678-HxCDD	0,1	0,1	0,1	0,1
123789-HxCDD	0,1	0,1	0,1	0,1
1234678-HpCDD	0,01	0,01	0,01	0,01
OCDD	0,0003	0,0001	0,001	0,001
2378-TeCDF	0,1	0,1	0,1	0,1
12378-PeCDF	0,03	0,05	0,05	0,01
23478-PeCDF	0,3	0,5	0,5	0,5
123478-HxCDF	0,1	0,1	0,1	0,1
123678-HxCDF	0,1	0,1	0,1	0,1
123789-HxCDF	0,1	0,1	0,1	0,1
234678-HxCDF	0,1	0,1	0,1	0,1
1234678-HpCDF	0,01	0,01	0,01	0,01
1234789-HpCDF	0,01	0,01	0,01	0,01
OCDF	0,0003	0,0001	0,001	0,001

Resultatsammanställning

Vår provmärkning:	3469:1	3469:2	3469:3	3469:4
Er provmärkning:	A5=FO2b	A4=FE6a	B3=FE5b	B4=FO1b
Provtyp:	LDPE	LDPE	LDPE	LDPE
Mängd analyserat prov färskvikt:	-	-	-	-
Enhet:	pg/prov	pg/prov	pg/prov	pg/prov
PCDD och PCDF:				
Σ WHO 2005 TEQ _{diox}				
Övre koncentration	131.9	51.7	11.2	32.7
Medel koncentration	131.8	51.6	11.2	32.5
Nedre koncentration	131.7	51.5	11.2	32.4
HCB:	5751	5931	5246	2071
Återfunnen ¹³C IS(%)	98	61	77	66
OCS:	301	387	53	76
Återfunnen ¹³C IS(%)	98	93	93	113

Vår provmärkning:	3469:5	3469:6
Er provmärkning:	B7=FO4b	B9=FO3b
Provtyp:	LDPE	LDPE
Mängd analyserat prov färskvikt:	-	-
Enhet:	pg/prov	pg/prov
PCDD och PCDF:		
Σ WHO 2005 TEQ _{diox}		
Övre koncentration	8.22	3.11
Medel koncentration	7.48	2.09
Nedre koncentration	6.74	1.06
HCB:	6153	6609
Återfunnen ¹³C IS(%)	102	88
OCS:	49	22
Återfunnen ¹³C IS(%)	98	92

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:1
Er provmärkning : A5=FO2b
Provtyp : LDPE
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.160)	0.12%	92%
12378 PeCDD	14.1	11%	83%
123478 HxCDD	12.8	0.97%	89%
123678 HxCDD	19.9	1.51%	93%
123789 HxCDD	12.1	0.92%	87%
1234678 HpCDD	44.3	0.34%	88%
OCDD	36.9	0.01%	95%
2378 TeCDF	98.8	7.5%	99%
12378 PeCDF	243	5.5%	57%
23478 PeCDF ^a	135	31%	128%
123478 HxCDF	259	20%	95%
123678 HxCDF	126	9.6%	97%
234678 HxCDF	64.9	4.9%	110%
123789 HxCDF ^b	60.2	4.6%	102%
1234678 HpCDF	291	2.2%	100%
1234789 HpCDF	88.1	0.67%	97%
OCDF	428	0.10%	110%
Sum	övre konc.	131.9	
WHO-	medelkonc.	131.8	
TEQ₂₀₀₅	nedre konc.	131.7	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	20110401	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	20110622	Upparbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829	Analys:	Anna Kitti-Sjöström

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:2
Er provmärkning : A4=FE6a
Provtyp : SPMD
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.160)	0.31%	98%
12378 PeCDD	5.41	10%	84%
123478 HxCDD	6.48	1.3%	92%
123678 HxCDD	10.4	2.0%	93%
123789 HxCDD	6.34	1.2%	89%
1234678 HpCDD	25.7	0.50%	92%
OCDD	25.2	0.01%	94%
2378 TeCDF	27.9	5.4%	104%
12378 PeCDF	51.1	3.0%	93%
23478 PeCDF ^a	40.0	23%	125%
123478 HxCDF	118	23%	99%
123678 HxCDF	67.3	13%	100%
234678 HxCDF	33.3	6.4%	112%
123789 HxCDF ^b	28.1	5.4%	103%
1234678 HpCDF	189	3.7%	98%
1234789 HpCDF	55.0	1.1%	99%
OCDF	285	0.17%	105%
Sum	övre konc.	51.7	
WHO-	medelkonc.	51.6	
TEQ₂₀₀₅	nedre konc.	51.5	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	20110401	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppabetning:	20110622	Upparbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829	Analys:	Anna Kitti-Sjöström

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:3
Er provmärkning : B3=FE6a
Provtyp : LDPE
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.0006)	0.005%	93%
12378 PeCDD	ND(0.0011)	0.010%	89%
123478 HxCDD	1.41	1.3%	92%
123678 HxCDD	1.91	1.7%	92%
123789 HxCDD	2.80	2.5%	93%
1234678 HpCDD	10.3	0.92%	90%
OCDD	48.3	0.13%	93%
2378 TeCDF	17.1	15%	93%
12378 PeCDF	12.7	3.4%	91%
23478 PeCDF ^a	10.6	28%	102%
123478 HxCDF	24.3	22%	94%
123678 HxCDF	11.2	10.0%	96%
234678 HxCDF	5.84	5.2%	94%
123789 HxCDF ^b	6.03	5.4%	92%
1234678 HpCDF	32.8	2.9%	94%
1234789 HpCDF	9.6	0.86%	92%
OCDF	66.9	0.18%	99%
Sum	övre konc.	11.2	
WHO-	medelkonc.	11.2	
TEQ₂₀₀₅	nedre konc.	11.2	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	20110401	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppärbetning:	20110622	Uppärbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829		Anna Kitti-Sjöström

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:4
Er provmärkning : B4=FO1b
Provtyp : LDPE
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.160)	0.49%	89%
12378 PeCDD	3.59	11%	77%
123478 HxCDD	ND(1.34)	0.41%	89%
123678 HxCDD	5.46	1.7%	85%
123789 HxCDD	3.53	1.1%	87%
1234678 HpCDD	17.8	0.55%	92%
OCDD	17.5	0.02%	103%
2378 TeCDF	14.8	4.5%	98%
12378 PeCDF	30.7	2.8%	89%
23478 PeCDF ^a	25.2	23%	128%
123478 HxCDF	80.4	25%	96%
123678 HxCDF	35.8	11%	96%
234678 HxCDF	22.3	6.8%	107%
123789 HxCDF ^b	22.2	6.8%	100%
1234678 HpCDF	134	4.1%	96%
1234789 HpCDF	27.7	0.85%	107%
OCDF	194	0.18%	118%
Sum	övre konc.	32.7	
WHO-	medelkonc.	32.5	
TEQ₂₀₀₅	nedre konc.	32.4	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	20110401	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	20110622	Upparbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829	Analys:	Anna Kitti-Sjöström

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:5
Er provmärkning : B7=FO4b
Provtyp : LDPE
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.160)	1.9%	93%
12378 PeCDD	ND(1.06)	13%	88%
123478 HxCDD	ND(1.34)	1.6%	94%
123678 HxCDD	<i>2.15</i>	2.6%	91%
123789 HxCDD	ND(1.35)	1.6%	90%
1234678 HpCDD	6.17	0.75%	93%
OCDD	<i>6.16</i>	0.02%	89%
2378 TeCDF	9.55	12%	95%
12378 PeCDF	9.96	3.6%	92%
23478 PeCDF ^a	4.79	17%	113%
123478 HxCDF	16.0	20%	96%
123678 HxCDF	8.16	9.9%	96%
234678 HxCDF	5.55	6.8%	108%
123789 HxCDF ^b	<i>5.06</i>	6.1%	100%
1234678 HpCDF	21.6	2.6%	101%
1234789 HpCDF	5.88	0.72%	94%
OCDF	32.6	0.12%	88%
Sum	övre konc.	8.22	
WHO-	medelkonc.	7.48	
TEQ₂₀₀₅	nedre konc.	6.74	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	20110401	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	20110622	Upparbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829	Analys:	Anna Kitti-Sjöström

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:6
Er provmärkning : B9=FO3b
Provtyp : LDPE
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.160)	5.1%	91%
12378 PeCDD	ND(1.06)	34%	83%
123478 HxCDD	ND(1.34)	4.3%	89%
123678 HxCDD	ND(1.26)	4.0%	93%
123789 HxCDD	ND(1.35)	4.3%	88%
1234678 HpCDD	3.92	1.3%	88%
OCDD	7.74	0.07%	87%
2378 TeCDF	<i>0.885</i>	2.8%	100%
12378 PeCDF	ND(0.890)	0.9%	89%
23478 PeCDF ^a	ND(0.950)	9%	111%
123478 HxCDF	4.64	15%	96%
123678 HxCDF	<i>1.24</i>	4.0%	99%
234678 HxCDF	ND(1.25)	4.0%	98%
123789 HxCDF ^b	<i>2.43</i>	7.8%	96%
1234678 HpCDF	7.44	2.4%	95%
1234789 HpCDF	<i>2.33</i>	0.75%	92%
OCDF	ND(2.57)	0.02%	97%
Sum	övre konc.	3.11	3.11
WHO-	medelkonc.	2.09	2.09
TEQ₂₀₀₅	nedre konc.	1.06	1.06

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	20110401	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	20110622	Upparbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829	Analys:	Anna Kitti-Sjöström

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3469:B
Er provmärkning : -
Provtyp : Laboratorieblank
Mängd analyserat prov (g) : -
Fettvikt (g) : -
Provsort : pg/prov
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/prov)		Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.160)	6.3%	95%
12378 PeCDD	ND(1.06)	42%	92%
123478 HxCDD	ND(1.34)	5.3%	93%
123678 HxCDD	ND(1.26)	5.0%	96%
123789 HxCDD	ND(1.35)	5.3%	95%
1234678 HpCDD	1.82	0.72%	92%
OCDD	ND(2.19)	0.03%	90%
2378 TeCDF	ND(0.360)	1.4%	94%
12378 PeCDF	ND(0.890)	1.1%	93%
23478 PeCDF ^a	ND(0.950)	11%	96%
123478 HxCDF	ND(1.23)	4.9%	92%
123678 HxCDF	ND(1.19)	4.7%	96%
234678 HxCDF	ND(1.25)	4.9%	92%
123789 HxCDF ^b	ND(1.58)	6.2%	89%
1234678 HpCDF	ND(0.930)	0.37%	96%
1234789 HpCDF	ND(1.50)	0.59%	92%
OCDF	ND(2.57)	0.03%	86%
Sum	övre konc.	2.53	HCB: 112
WHO-TEQ₂₀₀₅	medelkonc.	1.27	OCS: <10
	nedre konc.	0.02	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:		Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :		Person ansvarig för	
Startdatum för upparbetning:	20110622	Upparbetning:	Anna Kitti-Sjöström
Startdatum för analys:	20110829	Analys:	Anna Kitti-Sjöström

Appendix C

UMEÅ UNIVERSITET
Kemiska institutionen,
Miljökemiska Laboratoriet
Sture Bergek
Maria Hjelt
Per Liljelind
Tel: 090-786 9321



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NO-0349 Oslo, Norway

Resultat från analys av polyklorerade dibenso-*p*-dioxiner, polyklorerade dibensofuraner (PCDD/F), hexaklorbensen (HCB) och oktaklorstyren (OCS) i biologiska prov (Hinia och Nereis).

Laboratorier ackrediteras av Styrelsen för ackreditering och teknisk kontroll (SWEDAC) enligt svensk lag. Den ackrediterade verksamheten vid laboratorierna uppfyller kraven i SS-EN ISO/IEC 17 025 (2005).

Förkortningar i analysrapporten

TrC	Triklor (3 klor)	-DD	Dibenso- <i>p</i> -dioxin(er)
TeC-	Tetraklor (4 klor)	-DF	Dibensofuran(er)
PeC-	Pentaklor (5 klor)	-B	Bifenyl(er)
HxC-	Hexaklor (6 klor)		
HpC-	Heptaklor (7 klor)		
OC-	Oktaklor (8 klor)		
NC-	Nonaklor (9 klor)		
DeC-	Dekaklor (10 klor)		
ng	Nanogram (10 ⁻⁹ gram)		
pg	Picogram (10 ⁻¹² gram)		
IS	Internstandard		
fw	Färskvikt (fresh weight)		
lw	Fettvikt (lipid weight)		
ND	Icke-detekterad (Not Detected)		
LOD	Detektionsgräns (Limit of Detection)		
LOQ	Kvantifieringsgräns (Limit of Quantification)		
TEF	Toxisk ekvivalentfaktor (Toxic Equivalency Factor)		
TEQ	Total koncentration i TCDD-ekvivalenter (TCDD toxic equivalent concentration)		

Metodbeskrivning

Använda uppberedningsmetoder är väl validerade genom ett flertal internationella interkalibreringar och GC-MS analyserna utförs enligt Svensk standard SS-EN 1948:1-3. De genomförda analyserna uppfyller även kvalitetskraven för analys av dioxiner och dioxinlika PCB, EU-direktiv 2002/69/EC, som publicerades i EUs officiella tidskrift (Official journal of the European Communities) den 30 juli 2002. En sammanfattning av analysmetoderna följer nedan.

Extraktion och fettviktsbestämning

Före extraktionen tillsattes internstandard bestående av ^{13}C -anrikade isotoper av merparten av de ämnen (kongener) som skall bestämmas. Proven extraherades med organiska lösningsmedel. Lösningssmedlet avlägsnades genom indunstning och mängden fett bestämdes genom vägning.

Upprening

Uppreningen av polyklorerade dibenso-p-dioxiner (PCDD), polyklorerade dibensofuraner (PCDF), hexaklorbensen (HCB) och oktaklorstyren (OCS) utfördes med två vätskekromatografikolonner: en flerskiktsskolonn bestående av kiselgel, svavelsyra- och kaliumhydroxidimpregnerad kiselgel samt en kolonn med aktivt kol. På den sistnämnda separeras provet i två fraktioner innehållande 1) OCS, 2) PCDD/F och HCB. Innan den slutliga analysen tillsattes ytterligare ^{13}C -kongener, sk. återfinningsstandarder.

Analys

Isomerspecifik analys har skett med gaskromatografi (GC) kopplat till masspektrometri (MS). Separationen av ämnena sker på GC:n och detektionen med masspektrometern. Vid MS-analysen detekterades ämnen med olika masstal selektivt vilket möjliggjorde utnyttjandet av syntetiska ^{13}C -isotopanrikade ämnen (^{13}C -kongener) vilka användes som interna standarder med så kallad isotoputspädningsmetodik. Härvidlag jämfördes responskvoten mellan naturliga kongener och ^{13}C -kongener i provet med motsvarande kvot i en kvantifieringsstandard innehållande kända mängder av naturliga och tillsatta ^{13}C -kongener. Detta förfarande medförde att resultaten automatiskt blev kompenserade för uppberedningsförluster. En MS (Waters Autospec Ultima) med hög massupplösning (~9000) har använts. Den använde elektronstötjonisering (EI) där sedan utvalda joner registrerades (SIR).

Koncentrationsbestämningen har utförts enligt ovan nämnda norm, SS-EN 1948:3 och återfinningsgraden av de internstandarder (IS) som tillsatts proven beräknas och uttrycks i procent av ursprunglig mängd.

När en kongen ej kan detekteras räknas detektionsgränsen ut (LOD – limit of detection). Den motsvarar en signal från analysinstrumentet som är tre gånger högre än brusnivån och anges som ett mindre än-värde. LOD beror av ett antal faktorer och varierar därför något från prov till prov, mellan olika kongener och från ett analystillfälle till ett annat. Akkrediterade resultat kan endast fås ner till kvantifieringsgränsen (LOQ – limit of

quantification) som defineras av signaler som överstiger tio gånger brusnivån. Det framgår av analysrapporten för vilka kongener detta kriterium inte är uppfyllt. I området mellan tre och tio gånger brusnivån är mätosäkerheten förhöjd men ger ändå ett värdefullt bidrag till resultaten och TEQ-beräkningen.

Laboratorieblankens koncentration har redovisats separat, ingen subtraktion har gjorts från de verkliga provens koncentrationer. Normalisering har gjorts till samma enhet som för proven med medelvärdet av de provmängder som använts till dessa.

Beräkning av TCDD-ekvivalenter (TEQ)

Utifrån de enskilda kongenernas koncentration har s.k. TCDD-ekvivalenter (TEQ) beräknats. TCDD-ekvivalenterna relaterar de toxiska kongenerna till den mest toxiska, 2,3,7,8-TeCDD.

$$\text{TEQ} = \text{koncentration} \times \text{TEF}$$

Det finns ett antal olika TEF-skalar som använts genom åren. Idag är WHO-TEF-skalan den vedertagna men resultaten kan omräknas enligt den skala som önskas (tabell 1, sid. 5).

När en kongen ej kan detekteras räknas detektionsgränsen ut. Den motsvarar en signal från analysinstrumentet som är tre gånger högre än brusnivån. Detektionsgränsen beror av ett antal faktorer och varierar därför något från prov till prov, mellan olika kongener och från ett analystillfälle till ett annat.

TEQ beräknas på tre nivåer. En nedre koncentrationsgräns där koncentrationerna av icke detekterade ämnen satts till noll, en övre koncentrationsgräns där koncentrationerna av icke detekterade ämnen ersatts med detektionsgränsen samt en medelkoncentration (medelvärdet av de båda).

En sammanfattning av analysrapporten finns på sidan 6-8 med WHO-TEQ-värden för samtliga prov och ev kommentarer.

Umeå som ovan,

Tabell 1. Skalor för beräkning av toxiska ekvivalenter (TEQ):

Kongen	TEF			
	WHO ₂₀₀₅	WHO ₁₉₉₈	Internationella	Nordic
2378-TeCDD	1	1	1	1
12378-PeCDD	1	1	0,5	0,5
123478-HxCDD	0,1	0,1	0,1	0,1
123678-HxCDD	0,1	0,1	0,1	0,1
123789-HxCDD	0,1	0,1	0,1	0,1
1234678-HpCDD	0,01	0,01	0,01	0,01
OCDD	0,0003	0,0001	0,001	0,001
2378-TeCDF	0,1	0,1	0,1	0,1
12378-PeCDF	0,03	0,05	0,05	0,01
23478-PeCDF	0,3	0,5	0,5	0,5
123478-HxCDF	0,1	0,1	0,1	0,1
123678-HxCDF	0,1	0,1	0,1	0,1
123789-HxCDF	0,1	0,1	0,1	0,1
234678-HxCDF	0,1	0,1	0,1	0,1
1234678-HpCDF	0,01	0,01	0,01	0,01
1234789-HpCDF	0,01	0,01	0,01	0,01
OCDF	0,0003	0,0001	0,001	0,001

Resultatsammanställning

Vår provmärkning:	3492:1	3492:2	3492:3	3492:4
Er provmärkning:	2182:1	2182:2	2182:3	2182:4
Provtyp:	Hinia	Hinia	Hinia	Hinia
Mängd analyserat prov färskvikt:	22.25	20.68	21.38	20.20
Enhet:	pg/g	pg/g	pg/g	pg/g

PCDD och PCDF:

Σ WHO 2005 TEQ_{diox}

Övre koncentration	2.0	2.9	0.63	3.7
Medel koncentration	2.0	2.9	0.53	3.7
Nedre koncentration	2.0	2.9	0.43	3.7

HCB:	67	110	43	140
Återfunnen ¹³C IS(%)	54%	65%	55%	58%

OCS:	3.9	7.5	1.5	8.7
Återfunnen ¹³C IS(%)	76%	77%	77%	77%

Vår provmärkning:	3492:5	3492:6	3492:7	3492:8
Er provmärkning:	2182:5	2182:6	2182:7	2182:8
Provtyp:	Hinia	Hinia	Hinia	Nereis
Mängd analyserat prov färskvikt:	20.41	21.27	8.87	1.75
Enhet:	pg/g	pg/g	pg/g	pg/g
PCDD och PCDF:				
Σ WHO 2005 TEQ _{diox}				
Övre koncentration	1.1	6.1	0.60	11
Medel koncentration	1.0	6.1	0.32	9.4
Nedre koncentration	0.96	6.1	0.033	8.2
HCB:	65	190	65	210
Återfunnen ¹³C IS(%)	54%	53%	63%	52%
OCS:	9.6	39	0.80	67
Återfunnen ¹³C IS(%)	75%	75%	74%	92%

Vår provmärkning:	3492:9	3492:10	3492:11	3492:12	3492:13
Er provmärkning:	2182:9	2182:10	2182:11	2182:12	2182:13
Provtyp:	Nereis	Nereis	Nereis	Nereis	Nereis
Mängd analyserat prov färskvikt:	4.99	7.07	6.00	2.41	1.95
Enhet:	pg/g	pg/g	pg/g	pg/g	pg/g
PCDD och PCDF:					
Σ WHO 2005 TEQ _{diox}					
Övre koncentration	4.8	1.2	5.1	26	7.6
Medel koncentration	4.6	0.86	5.0	26	6.5
Nedre koncentration	4.4	0.55	4.8	25	5.5
HCB:	230	41	250	370	520
Återfunnen ¹³C IS(%)	57%	54%	60%	60%	64%
OCS:	69	13	61	1100	410
Återfunnen ¹³C IS(%)	89%	72%	78%	74%	73%

Kommentar: Mätvärden för HCB och OCS i laboratorieblanken finns på sista sidan i rapporten tillsammans med dioxinvärdena.

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:1
Er provmärkning : 2182:1
Provtyp : Hinia
Mängd analyserat prov (g) : 22.25
Fettvikt (g) : 0.459
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	<i>0.044</i>	2.2%	93%
12378 PeCDD	<i>0.23</i>	11%	96%
123478 HxCDD	<i>0.17</i>	0.85%	114%
123678 HxCDD	<i>0.27</i>	1.3%	114%
123789 HxCDD	<i>0.21</i>	1.0%	103%
1234678 HpCDD	<i>1.7</i>	0.84%	95%
OCDD	<i>3.2</i>	0.048%	101%
2378 TeCDF	<i>1.3</i>	6.7%	97%
12378 PeCDF	<i>1.5</i>	2.2%	105%
23478 PeCDF ^a	<i>1.1</i>	16%	117%
123478 HxCDF	<i>4.8</i>	24%	98%
123678 HxCDF	<i>2.4</i>	12%	98%
234678 HxCDF	<i>1.4</i>	6.8%	92%
123789 HxCDF ^b	<i>1.0</i>	5.2%	97%
1234678 HpCDF	<i>14</i>	6.8%	99%
1234789 HpCDF	<i>4.2</i>	2.1%	97%
OCDF	<i>37</i>	0.56%	99%
Sum	övre konc.	2.0	
WHO- TEQ₂₀₀₅	medelkonc.	2.0	
	nedre konc.	2.0	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-05	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:2
Er provmärkning : 2182:2
Provtyp : Hinia
Mängd analyserat prov (g) : 20.68
Fettvikt (g) : 0.410
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	0.083	2.8%	94%
12378 PeCDD	<i>0.34</i>	11%	93%
123478 HxCDD	0.25	0.84%	113%
123678 HxCDD	0.38	1.3%	110%
123789 HxCDD	ND(0.33)	1.1%	97%
1234678 HpCDD	1.8	0.60%	96%
OCDD	3.3	0.034%	96%
2378 TeCDF	2.9	9.8%	98%
12378 PeCDF	2.6	2.6%	103%
23478 PeCDF ^a	1.9	19%	117%
123478 HxCDF	6.4	22%	98%
123678 HxCDF	3.1	11%	98%
234678 HxCDF	1.8	6.1%	92%
123789 HxCDF ^b	1.4	4.7%	95%
1234678 HpCDF	14	4.8%	99%
1234789 HpCDF	4.8	1.6%	97%
OCDF	38	0.38%	100%
Sum	övre konc.	2.9	
WHO- TEQ₂₀₀₅	medelkonc.	2.9	
	nedre konc.	2.9	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-05	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:3
Er provmärkning : 2182:3
Provtyp : Hinia
Mängd analyserat prov (g) : 21.38
Fettvikt (g) : 0.383
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.028)	4.4%	95%
12378 PeCDD	ND(0.14)	22%	88%
123478 HxCDD	ND(0.085)	1.3%	121%
123678 HxCDD	ND(0.085)	1.3%	122%
123789 HxCDD	ND(0.18)	2.9%	119%
1234678 HpCDD	0.61	0.96%	96%
OCDD	1.4	0.066%	97%
2378 TeCDF	0.64	10%	97%
12378 PeCDF	0.35	1.6%	97%
23478 PeCDF ^a	<i>0.27</i>	13%	121%
123478 HxCDF	1.0	16%	99%
123678 HxCDF	0.52	8.2%	101%
234678 HxCDF	<i>0.31</i>	4.9%	95%
123789 HxCDF ^b	<i>0.27</i>	4.2%	96%
1234678 HpCDF	3.7	5.9%	102%
1234789 HpCDF	1.1	1.8%	96%
OCDF	11	0.52%	98%
Sum	övre konc.	0.63	
WHO- TEQ₂₀₀₅	medelkonc.	0.53	
	nedre konc.	0.43	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-05	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:4
Er provmärkning : 2182:4
Provtyp : Hinia
Mängd analyserat prov (g) : 20.20
Fettvikt (g) : 0.383
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	0.092	2.5%	96%
12378 PeCDD	<i>0.39</i>	11%	90%
123478 HxCDD	0.31	0.84%	120%
123678 HxCDD	0.43	1.2%	122%
123789 HxCDD	<i>0.29</i>	0.78%	120%
1234678 HpCDD	2.1	0.55%	96%
OCDD	3.6	0.029%	97%
2378 TeCDF	4.0	11%	100%
12378 PeCDF	3.9	3.2%	101%
23478 PeCDF ^a	2.8	23%	120%
123478 HxCDF	7.8	21%	99%
123678 HxCDF	3.6	9.7%	102%
234678 HxCDF	2.1	5.7%	95%
123789 HxCDF ^b	1.6	4.3%	97%
1234678 HpCDF	16	4.4%	100%
1234789 HpCDF	5.3	1.4%	97%
OCDF	41	0.33%	100%
Sum	övre konc.	3.7	
WHO- TEQ₂₀₀₅	medelkonc.	3.7	
	nedre konc.	3.7	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-05	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:5
Er provmärkning : 2182:5
Provtyp : Hinia
Mängd analyserat prov (g) : 20.41
Fettvikt (g) : 0.394
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	<i>0.036</i>	3.2%	95%
12378 PeCDD	ND(0.12)	11%	85%
123478 HxCDD	ND(0.12)	1.0%	117%
123678 HxCDD	<i>0.13</i>	1.2%	114%
123789 HxCDD	ND(0.25)	2.3%	111%
1234678 HpCDD	1.1	0.96%	96%
OCDD	2.6	0.069%	98%
2378 TeCDF	0.87	7.9%	97%
12378 PeCDF	0.74	2.0%	91%
23478 PeCDF ^a	0.54	15%	115%
123478 HxCDF	2.4	22%	96%
123678 HxCDF	1.3	12%	99%
234678 HxCDF	0.70	6.3%	93%
123789 HxCDF ^b	0.61	5.5%	98%
1234678 HpCDF	8.4	7.5%	94%
1234789 HpCDF	2.7	2.5%	103%
OCDF	29	0.79%	121%
Sum	övre konc.	1.1	
WHO- TEQ₂₀₀₅	medelkonc.	1.0	
	nedre konc.	0.96	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-05	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:6
Er provmärkning : 2182:6
Provtyp : Hinia
Mängd analyserat prov (g) : 21.27
Fettvikt (g) : 0.359
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	0.13	2.1%	97%
12378 PeCDD	0.72	12%	86%
123478 HxCDD	0.45	0.75%	123%
123678 HxCDD	0.68	1.1%	122%
123789 HxCDD	<i>0.45</i>	0.74%	119%
1234678 HpCDD	3.3	0.55%	96%
OCDD	5.8	0.029%	97%
2378 TeCDF	4.9	8.0%	98%
12378 PeCDF	6.5	3.2%	94%
23478 PeCDF ^a	4.5	22%	120%
123478 HxCDF	13	21%	99%
123678 HxCDF	6.7	11%	104%
234678 HxCDF	3.7	6.0%	95%
123789 HxCDF ^b	2.8	4.6%	96%
1234678 HpCDF	27	4.5%	100%
1234789 HpCDF	9.5	1.6%	99%
OCDF	74	0.37%	105%
Sum	övre konc.	6.1	
WHO- TEQ₂₀₀₅	medelkonc.	6.1	
	nedre konc.	6.1	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:7
Er provmärkning : 2182:7
Provtyp : Hinia
Mängd analyserat prov (g) : 8.87
Fettvikt (g) : 0.087
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.064)	11%	94%
12378 PeCDD	ND(0.26)	44%	85%
123478 HxCDD	ND(0.20)	3.3%	110%
123678 HxCDD	ND(0.20)	3.3%	113%
123789 HxCDD	ND(0.43)	7.3%	110%
1234678 HpCDD	<i>0.28</i>	0.47%	96%
OCDD	0.99	0.050%	95%
2378 TeCDF	<i>0.24</i>	4.0%	96%
12378 PeCDF	ND(0.20)	0.99%	98%
23478 PeCDF ^a	ND(0.21)	10%	112%
123478 HxCDF	ND(0.21)	3.5%	93%
123678 HxCDF	ND(0.18)	3.1%	100%
234678 HxCDF	ND(0.22)	3.6%	96%
123789 HxCDF ^b	ND(0.23)	3.9%	96%
1234678 HpCDF	<i>0.48</i>	0.81%	101%
1234789 HpCDF	ND(0.41)	0.69%	94%
OCDF	1.3	0.067%	96%
Sum	övre konc.	0.60	
WHO- TEQ₂₀₀₅	medelkonc.	0.32	
	nedre konc.	0.033	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:8
Er provmärkning : 2182:8
Provtyp : Nereis
Mängd analyserat prov (g) : 1.75
Fettvikt (g) : 0.017
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.29)	2.8%	96%
12378 PeCDD	ND(1.8)	17%	83%
123478 HxCDD	1.0	0.95%	113%
123678 HxCDD	1.4	1.3%	117%
123789 HxCDD	ND(1.7)	1.6%	113%
1234678 HpCDD	4.8	0.45%	96%
OCDD	9.6	0.027%	114%
2378 TeCDF	4.9	4.6%	98%
12378 PeCDF	10	2.9%	92%
23478 PeCDF ^a	6.8	20%	111%
123478 HxCDF	24	23%	94%
123678 HxCDF	8.6	8.2%	103%
234678 HxCDF	6.0	5.7%	98%
123789 HxCDF ^b	6.5	6.1%	96%
1234678 HpCDF	43	4.0%	100%
1234789 HpCDF	14	1.3%	94%
OCDF	109	0.31%	94%
Sum	övre konc.	11	
WHO- TEQ₂₀₀₅	medelkonc.	9.4	
	nedre konc.	8.2	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:9
Er provmärkning : 2182:9
Provtyp : Nereis
Mängd analyserat prov (g) : 4.99
Fettvikt (g) : 0.060
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.21)	4.4%	93%
12378 PeCDD	<i>0.61</i>	13%	81%
123478 HxCDD	ND(0.38)	0.80%	109%
123678 HxCDD	<i>0.45</i>	0.94%	108%
123789 HxCDD	ND(0.86)	1.8%	107%
1234678 HpCDD	1.7	0.35%	93%
OCDD	3.4	0.021%	121%
2378 TeCDF	5.5	12%	93%
12378 PeCDF	6.5	4.1%	90%
23478 PeCDF ^a	3.9	24%	105%
123478 HxCDF	9.3	20%	92%
123678 HxCDF	3.1	6.6%	99%
234678 HxCDF	1.8	3.8%	100%
123789 HxCDF ^b	2.4	5.1%	94%
1234678 HpCDF	14	2.9%	98%
1234789 HpCDF	4.3	0.89%	96%
OCDF	35	0.22%	98%
Sum	övre konc.	4.8	
WHO- TEQ₂₀₀₅	medelkonc.	4.6	
	nedre konc.	4.4	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:10
Er provmärkning : 2182:10
Provtyp : Nereis
Mängd analyserat prov (g) : 7.07
Fettvikt (g) : 0.055
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.072)	6.2%	92%
12378 PeCDD	ND(0.27)	23%	84%
123478 HxCDD	ND(0.30)	2.6%	116%
123678 HxCDD	ND(0.30)	2.5%	114%
123789 HxCDD	ND(0.67)	5.8%	112%
1234678 HpCDD	0.70	0.60%	91%
OCDD	2.3	0.058%	90%
2378 TeCDF	<i>0.50</i>	4.3%	94%
12378 PeCDF	0.72	1.9%	94%
23478 PeCDF ^a	ND(0.48)	12%	111%
123478 HxCDF	2.0	18%	96%
123678 HxCDF	0.83	7.1%	97%
234678 HxCDF	<i>0.54</i>	4.6%	92%
123789 HxCDF ^b	<i>0.63</i>	5.4%	93%
1234678 HpCDF	4.6	4.0%	94%
1234789 HpCDF	1.5	1.3%	90%
OCDF	14	0.36%	97%
Sum	övre konc.	1.2	
WHO- TEQ₂₀₀₅	medelkonc.	0.86	
	nedre konc.	0.55	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:11
Er provmärkning : 2182:11
Provtyp : Nereis
Mängd analyserat prov (g) : 6.00
Fettvikt (g) : 0.057
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.12)	2.4%	95%
12378 PeCDD	<i>0.68</i>	13%	83%
123478 HxCDD	ND(0.40)	0.78%	118%
123678 HxCDD	<i>0.41</i>	0.80%	117%
123789 HxCDD	ND(0.88)	1.7%	117%
1234678 HpCDD	1.6	0.31%	94%
OCDD	4.1	0.024%	91%
2378 TeCDF	5.3	10%	96%
12378 PeCDF	7.3	4.3%	93%
23478 PeCDF ^a	4.4	26%	120%
123478 HxCDF	10	20%	98%
123678 HxCDF	3.8	7.5%	99%
234678 HxCDF	1.9	3.8%	95%
123789 HxCDF ^b	2.5	4.9%	97%
1234678 HpCDF	13	2.5%	97%
1234789 HpCDF	4.4	0.87%	94%
OCDF	35	0.21%	97%
Sum	övre konc.	5.1	
WHO- TEQ₂₀₀₅	medelkonc.	5.0	
	nedre konc.	4.8	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:12
Er provmärkning : 2182:12
Provtyp : Nereis
Mängd analyserat prov (g) : 2.41
Fettvikt (g) : 0.031
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.57)	2.2%	92%
12378 PeCDD	4.2	16%	79%
123478 HxCDD	1.8	0.70%	105%
123678 HxCDD	2.5	0.97%	108%
123789 HxCDD	ND(1.8)	0.69%	107%
1234678 HpCDD	4.4	0.17%	90%
OCDD	8.1	0.009%	87%
2378 TeCDF	19	7.2%	93%
12378 PeCDF	33	3.8%	91%
23478 PeCDF ^a	27	31%	106%
123478 HxCDF	48	18%	89%
123678 HxCDF	22	8.4%	96%
234678 HxCDF	11	4.2%	95%
123789 HxCDF ^b	12	4.4%	93%
1234678 HpCDF	41	1.6%	97%
1234789 HpCDF	12	0.47%	91%
OCDF	66	0.076%	91%
Sum	övre konc.	26	
WHO- TEQ₂₀₀₅	medelkonc.	26	
	nedre konc.	25	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:13
Er provmärkning : 2182:13
Provtyp : Nereis
Mängd analyserat prov (g) : 1.95
Fettvikt (g) : 0.017
Provsort : pg/g
Mätosäkerhet : ± **26** % (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.50)	6.6%	94%
12378 PeCDD	ND(1.1)	15%	80%
123478 HxCDD	ND(1.0)	1.4%	101%
123678 HxCDD	ND(1.1)	1.4%	99%
123789 HxCDD	ND(2.4)	3.1%	99%
1234678 HpCDD	2.9	0.39%	94%
OCDD	7.4	0.029%	89%
2378 TeCDF	7.9	10%	96%
12378 PeCDF	9.7	3.8%	90%
23478 PeCDF ^a	5.5	22%	101%
123478 HxCDF	13	17%	92%
123678 HxCDF	5.0	6.6%	99%
234678 HxCDF	2.8	3.6%	108%
123789 HxCDF ^b	3.9	5.1%	95%
1234678 HpCDF	22	2.9%	100%
1234789 HpCDF	7.9	1.0%	91%
OCDF	60	0.24%	91%
Sum	övre konc.	7.6	
WHO- TEQ₂₀₀₅	medelkonc.	6.5	
	nedre konc.	5.5	

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:	2011-10-05	Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för upparbetning:	2011-10-06	Upparbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

Analysrapport klorerade dioxiner och furaner

Vår provmärkning : MPR 3492:B1
Er provmärkning : -
Provtyp : Laboratorieblank
Mängd analyserat prov (g) : "12"
Fettvikt (g) : -
Provsort : pg/g
Mätosäkerhet : ± **26 %** (95% konfidensintervall)

Kongen	Konc. (pg/g färskvikt)	WHO- TEQ(%)	Återfunnen ¹³ C IS (%)
2378 TeCDD	ND(0.036)	12%	93%
12378 PeCDD	ND(0.14)	44%	100%
123478 HxCDD	ND(0.15)	4.9%	120%
123678 HxCDD	ND(0.14)	4.6%	121%
123789 HxCDD	ND(0.33)	11%	114%
1234678 HpCDD	ND(0.24)	0.79%	94%
OCDD	<i>0.20</i>	0.020%	105%
2378 TeCDF	ND(0.083)	2.7%	94%
12378 PeCDF	ND(0.067)	0.66%	106%
23478 PeCDF ^a	ND(0.033)	3.2%	104%
123478 HxCDF	ND(0.12)	3.8%	98%
123678 HxCDF	ND(0.11)	3.6%	100%
234678 HxCDF	ND(0.12)	3.9%	95%
123789 HxCDF ^b	ND(0.13)	4.1%	96%
1234678 HpCDF	ND(0.13)	0.41%	98%
1234789 HpCDF	ND(0.13)	0.43%	94%
OCDF	<i>0.11</i>	0.011%	91%
Sum	övre konc.	0.30	HCB: 2.7
WHO- TEQ₂₀₀₅	medelkonc.	0.15	OCS: 0.12
	nedre konc.	0.000	65%
			95%

Koncentrationer angivna med *kursiv stil* ligger mellan LOD och LOQ med större mätosäkerhet (38%).

^a Sameluerar med 12489-PeCDF

^b Sameluerar med 123489-HxCDF

Provet ankom:		Typ av GC-kolonn:	DB-5ms
Lagringsbetingelser :	Frys	Person ansvarig för	
Startdatum för uppärbetning:	2011-10-06	Uppärbetning:	Sture Bergek
Startdatum för analys:	2011-10-21	Analys:	Per Liljelind

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