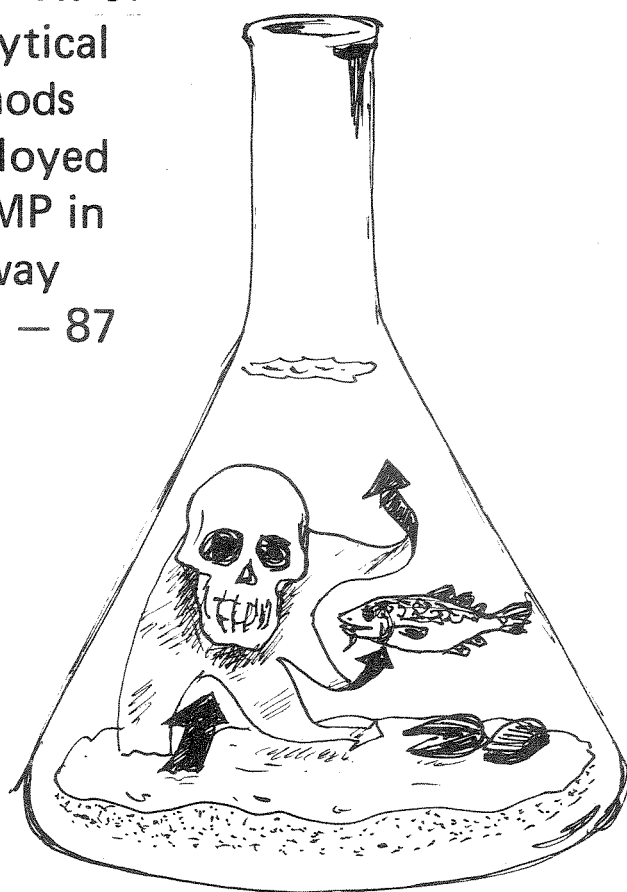


O-80106

# Joint Monitoring Programme

Overview of  
Analytical  
Methods  
Employed  
by JMP in  
Norway  
1981 – 87



# NIVA - REPORT

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<b>Abstract:</b> Brief descriptions are given of the analytical methods employed by Norway under the Joint Monitoring Programme (JMP) and in cooperation with the International Council for the Exploration of the Sea (ICES). Methods concern determination of trace metals and chlorinated hydrocarbons in sea water, sea bed sediment and marine organisms. Included are the detection limits, analytical laboratories, and references to the intercalibration exercises that applied to samples collected 1981-86.
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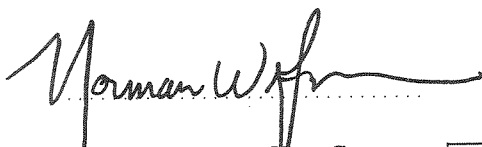
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Project leader



Norman W. Green

For the Administration



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0-80106

JOINT MONITORING PROGRAMME (JMP).

OVERVIEW OF ANALYTICAL METHODS EMPLOYED BY JMP IN NORWAY 1981-1987

Oslo, 29.01.1988

Project manager: Norman W. Green

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## FOREWORD

This report outlines the analytical methods employed under the Joint Monitoring Programme (JMP) in Norway 1981-1987 for the analyses of contaminants in sea water, sea bed sediment and marine biota samples (collected 1981-86). It is largely based on information filed at the International Council for the Exploration of the Sea (ICES) and in particular the Norwegian contribution to the report:

OSPAR, 1984. "Automatic data processing codes and descriptions of the sampling procedures and methods of analysis used in the Joint Monitoring Programme". Section 8 (Norway), 58/A/1-5/84 and 58/B/1/84. Oslo and Paris Commissions, October, 1984.

This report is basically a revision of OSPAR (1984) and includes updated and extended information but retains the same codes and abbreviations. The report is divided into three parts. The first part summarizes the analytical methods employed sorted by medium and method code and listing the analytical laboratories, contaminants (and other substances) quantified and sampling year(s) (Table 1).

The second part consists of a tabulized summary of contaminants and media analyzed, detection limits, analytical laboratories, the years samples were collected, method and intercalibration codes (Table 2).

The third part gives a more detailed description of each method. These are not meant to be complete but rather are intended to provide adequate information to distinguish analytical procedures. In some cases of older or rarely used analytical procedures only the literature reference is given. The revisions of the different methods have been reviewed by representatives of the respective analytical laboratories (listed below) during the spring and summer of 1987:

Institute for Nutrition, Fisheries Directorate: Kåre Julshamm  
Nordic Analytical Center (NAC): Paul D., Edminson and Beate Enger\*  
Norwegian Institute for Water Research: Håvard Hovind and  
Lasse Berglind  
Swedish Environmental Research Institute: Åke Iverfeldt  
Senter for Industrial Research (SI): Kari Martinsen  
Norwegian Veterinary Institute: Gunnar Norheim

\* B.E. previously employed at SI for metal analyses.

To these representatives and secretary Ingegerd Svensson I extend my gratitude in preparation of this report.

Norman W. Green  
Oslo, 29.01.1988

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**1. Table 1. Method overview, by medium and method code**

Table 1. Overview of analytical methods for trace metals, chlorinated hydrocarbons and other substances in the marine environment. (See abbreviation key at end of Table.)

method no.	institute (JMP code)	contaminant(s) etc.	sampling year
<u>Sea water</u>			
320	SIIF	Hg	84
322	SERI	Hg	84-86
330	SIIF	Cd	83
331	NIVA	Cu, Zn	85-86
331	NIVA	Cd, Pb	84-86
331	NIVA	Fe, Ni, Co	86
332	NIVA	Hg	86
<u>Sea bed sediment</u>			
350	NIVA	Hg	86
351	NIVA	Cu, Zn	86
352	NIVA	Cd, Pb	86
360	NIVA	PCB	86
390	NIVA	NTOT, CORG	86
<u>Marine biota</u>			
120	SIIF	Hg	81-85
121	SIIF	Hg	83-85
130	SIIF	Ni	83
130	SIIF	Cu	83-84
130	SIIF	Cd	81-85
130	SIIF	Pb	83-85
131	SIIF	Mn, Zn	83-85
220	VETN	Hg	82-86
220	VETN	Se	85
230	VETN	Cd	82-86
240	VETN	Se	82
310	NIVA	Hg	86
311	NIVA	Cu, Zn	86
312	NIVA	Cd, Pb	86
401	FIER	Hg	83-86
402	FIER	Cd	83-84
403	FIER	Pb	83, 86
404	FIER	Cu	83, 86
405	FIER	Zn	83, 86
406	FIER	As	83
110	SIIF	PCB, HCB, DDTEP, HCHG	-
111	SIIF	PCB, HCB, DDTEP, HCHG	83-86
210	VETN	PCB, HCB, DDEPP	82-85
211	VETN	PCB	82-85
510	NACE	PCB, HCB, DDEPP, DDTTP, HCHG	86
511	NACE	PCB	86
605	SIIF	EPOC1	86
610	NACE	EPOC1	86

/...

/...

## Abbreviations - English

- 
- DDEPP -  $\Sigma$  p,p'-DDE  
 DDTEP -  $\Sigma$  p,p'-DDE og p,p'-DDT  
 DDTPP - p,p'-DDT  
 DDE - dichlorodiphenylethylene principle metabolite of DDT  
 - p,p'DDE = 2,2-bis-(4-chlorophenyl)-1,1-dichlorethylene  
     NAC - Edminson (letter, 1987)  
     1,1-dichloro-2,2-bis-(p-chlorophenyl)ethylene  
     Norheim & Økland (1980)  
 DDT - dichlorodiphenyltrichloroethane  
 - p,p'DDT = 1,1-bis-(4-chlorophenyl)-1,2,2-trichloroethane  
     NAC - Edminson (letter, 1987)  
 - 1,1,1-trichloro-2,2-bis-(p-chlorophenyl)ethane  
     Norheim & Økland (1980)  
 HCB - hexachlorbenzene  
 HCHG - Lindan =  $\gamma$  HCH = hexachlorocyclohexene  
     =  $\gamma$  BHC = benzenehexachloride (outdated synonym)  
 PCB - polychlorinated biphenyls  
 EPOC1 - extractable persistent organically bound chlorine  
 NTOT - total organic nitrogen  
 CORG - organic carbon  
 FIER - Institute for Nutrition, Fisheries Directorate  
 NACE - Nordic Analytical Center (NAC)  
 NIVA - Norwegian Institute for Water Research  
 SERI - Swedish Environmental Research Institute  
 SIIF - Senter for Industrial Research (SI)  
 VETN - Norwegian Veterinary Institute

/...

## Abbreviations - Norwegian

/...

## Abbreviations - Norwegian

- 
- DDEPP -  $\Sigma$  p,p'-DDE  
 DDTEP -  $\Sigma$  p,p'-DDE og p,p'-DDT  
 DDTTP - p,p'-DDT  
 DDE - diklordifenyletylen hovedmetabolitt av DDT  
     - p,p'DDE = 2,2 - bis - (4-klorfenyl)- 1, 1 - dikloretylen  
     NAC - Edminson (brev, 1987)  
     1,1-dikloro-2,2-bis-(p-klorofenyl)etylen  
     Norheim og Økland (1980)  
 DDT - diklordifenyltrikloretan  
     - p,p'DDT = 1,1 - bis - (4-klorfenyl)- 1,2,2 - trikloretylen  
     NAC - Edminson (brev, 1987)  
     - 1,1,1-trikloro-2,2-bis-(p-klorofenyl)etan  
     Norheim og Økland (1980)  
 HCB - heksaklorbenzen  
 HCHG - Lindan =  $\gamma$  HCH = heksaklorcycloheksen  
     =  $\gamma$  BHC = benzenheksaklorid (foreldet navn)  
 PCB - polyklorerte bifenyler  
  
 EPOC1 - ekstraherbart persistent organisk bundet klor  
  
 NTOT - total organisk nitrogen  
 CORG - organisk karbon  
  
 FIER - Fiskeridirektoratets Ernæringsinstitutt  
 NACE - Nordisk Analyse Center (NAC)  
 NIVA - Norsk institutt for vannforskning  
 SERI - Institutionen för vatten- och luftvårdsforskning  
 SIIF - Senter for industriforskning (SI)  
     - Sentral institutt for industriell forskning (foreldet  
     navn)  
 VETN - Veterinærinstituttet



## Method overview, by medium and contaminant

**2. Table 2. Method overview, by medium and contaminant**

Table 2. Overview of chemical analyses employed by JMP 1981-1987 in Norway for trace metals, chlorinated hydrocarbons and other substances in sea water, sea bed sediment and marine biota. Detection limits (detec.) are listed. Intercalibration codes (and in some cases laboratory codes) are given to distinguish different ICES exercises. "x" indicates data is registered at ICES but that no intercalibration exercise was filed. "-" indicates that no data are registered at ICES. Count n indicates the number of samples involved in cases where detection limit varies from the normal. (See also key at end of Table).

medium	parameter	detec. ng/l	institute (JMP code)	sample year	method no.	Interca- libration	count
<u>Sea water</u>							
	Cr	200	NIVA	87	331	-	
	Mn	500	NIVA	87	331	-	
	Fe	50	NIVA	87	331	-	
	Co	5	NIVA	87	331	-	
	Ni	10	NIVA	87	331	-	
	Cu	10	NIVA	86	331	4I	
		50	NIVA	85	331	4Z	
	Zn	70	NIVA	85	331	4Z	
		10	NIVA	86	331	4I	
	Cd	1	NIVA	85	331	4Z	
		0.5	NIVA	86	331	4I	
		5	NIVA	84	331	4Z	
		100	SIIF	83	330	4F	
	Hg	0.02	SERI	84-85	322	4H	
		0.02	SERI	86	322	4I	
		2	NIVA	86	332	4I	
		10	SIIF	84	320	4F	
	Pb	6	NIVA	86	331	4Z NIVA	
-----							
medium	parameter	detec. ppb d.w.	institute (JMP code)	sample year	method no.	Interca- libration	count
<u>Sea bed sediment</u>							
	Cu	10	NIVA	86	351	7E	
	Zn	100	NIVA	86	351	7E	
	Cd	1	NIVA	86	352	7E	
	Hg	10	NIVA	86	350	7E	
	Pb	50	NIVA	86	352	7E	
	PCB	5	NIVA	86	360	-	
	NTOT	0.1*	NIVA	86	390	-	
	CORG	0.2*	NIVA	86	390	-	

\* mg/g

/...

Table 2. (cont.)  
Trace metals in marine biota

/...

medium	parameter	detec. ppb d.w.	institute (JMP code)	sample year	method no.	Interca- libration	count
<b>Marine biota</b>							
	Cr	-	-	-	-	-	
	Mn	1000	SIIF	83-85	131	x	
		100	SIIF	83-85	132	1G	
	Fe	-	-	-	-	-	
	Co	-	-	-	-	-	
	Ni	100	SIIF	83	130	x	
		200	SIIF	83-85	132	1G	
	Cu	40	SIIF	83-84	130	x	
		20	SIIF	83-84	130	1G	
		500*	VETN	86	-	-	NIVA
		50	FIER	83	404	1G	
		50	FIER	86	404	1Z	NIVA
		10	NIVA	86	311	1H	
		10	NIVA	86	311	1Z	NIVA
	Zn	2000	SIIF	83-85	131	x	
		10	SIIF	83-85	130	1G	
		100	VETN	86	-	1Z	NIVA
		20	FIER	83	405	1G	
		20	FIER	86	405	1Z	NIVA
		100	NIVA	86	311	1H	
		100	NIVA	86	311	1Z	NIVA
	As	5	FIER	83	406	1G	
	Se	10	VETN	82	240	x	
		10	VETN	85	220	-	
	Cd	5	SIIF	81	130	x	
		60	SIIF	81	130	1E	
		50	SIIF	81-82	130	1F	
		20	SIIF	83-85	130	1G	
		40	SIIF	82-84	130	x	
		50	SIIF	85	130	x	
		10*	VETN	82-85	230	1Z	VETN
		10*	VETN	86	230	1Z	NIVA
		1	FIER	83-84	402	1G	
		0.5	FIER	86	402	1Z	NIVA
		1	NIVA	86	312	1H	
		1	NIVA	86	312	1Z	NIVA

\* on wet weight basis

/...

/...

medium	parameter	detec. ppb d.w.	institute (JMP code)	sample year	method no.	Interca- libration	count
<u>Marine biota</u>							
	Hg	20	SIIF	81	120	x	
		30	SIIF	82-85	120	x	
		30	SIIF	81-82	120	1E	
		25	SIIF	83-85	121	1G	
		10*	VETN	82-85	220	1Z	VETN
		10*	VETN	86	220	1Z	NIVA
		5	FIER	83-85	401	1G	
		5	FIER	86	401	1Z	NIVA
		10	NIVA	86	310	1H	
		10	NIVA	86	310	1Z	NIVA
	Pb	100	SIIF	83-84	130	x	
		60	SIIF	84	130	x	n = 2 <sup>A</sup>
		50	SIIF	83-85	130	1G	
		200	SIIF	85	130	x	
		10	FIER	83	403	1G	
		10	FIER	86	403	1Z	NIVA
		50	NIVA	86	312	1Z	NIVA
	DDEPP	50*	VETN	82-85	210	2E	
		50*	VETN	86	210	2Z	NACE
		20*	NACE	86	510	2Z	NACE
	DDTPP	40*	NACE	86	510	x	
	DDTEP	0.5	SIIF	83-86	111	x	
		1.5	SIIF	84	111	x	n = 1 <sup>B</sup>
		0.2 <sup>C</sup>	SIIF	85	111	x	n = 1
		0.9 <sup>C</sup>	SIIF	85	111	x	n = 1
		1.0 <sup>C</sup>	SIIF	85	111	x	n = 2
		3.0 <sup>C</sup>	SIIF	85	111	x	n = 2

-----

\* on wet weight basis  
<sup>A</sup> 2 prawn samples, 1984  
<sup>B</sup> 1 mussel sample, 1984  
<sup>C</sup> variable detection limit due to sample quantity  
 /...

Table 2. (cont.)  
Chlorinated hydrocarbons in marine biota

/...

medium	parameter	detec. ppb	institute d.w.(JMP code)	sample year	method no.	Interca- libration	count
<b>Marine biota</b>							
HCB	0.5 <sup>A</sup>	SIIF	83-84	111	x		
	0.2	SIIF	85-86	111	x		
	0.1	SIIF	86	111	x		n = 2
	0.02 <sup>B</sup>	SIIF	84	111	x		n = 1
	0.04 <sup>B</sup>	SIIF	84	111	x		n = 1
	0.05 <sup>B</sup>	SIIF	84	111	x		n = 1
	0.06 <sup>B</sup>	SIIF	84	111	x		n = 1
	0.07 <sup>B</sup>	SIIF	85	111	x		n = 1
	0.08 <sup>B</sup>	SIIF	84	111	x		n = 1
	0.08 <sup>B</sup>	SIIF	85	111	x		n = 1
	0.1 <sup>B</sup>	SIIF	85	111	x		n = 4
	0.11 <sup>B</sup>	SIIF	84	111	x		n = 1
	0.9 <sup>B</sup>	SIIF	85	111	x		n = 1
	2.0 <sup>B</sup>	SIIF	85	111	x		n = 1
	10 <sup>*</sup>	VETN	83-85	210	2Z	VETN	
	0.2	SIIF	86	111	2Z	NACE	
	5 <sup>*</sup>	NACE	86	510	2Z	NACE	
10 <sup>*</sup>	VETN	86	210	2Z	NACE		
HCHG	0.4 <sup>B</sup>	SIIF	85	111	x		n = 2
	0.9 <sup>B</sup>	SIIF	85	111	x		n = 2
	1.0 <sup>B</sup>	SIIF	85	111	x		n = 3
	10 <sup>B</sup>	SIIF	85	111	x		n = 1
	25 <sup>*</sup>	NACE	86	510	2Z		
	3.0	SIIF	86	111	x		
PCB	5	SIIF	81-82	111	2D		
	5	SIIF	83-86	111	2E		
	6 <sup>B</sup>	SIIF	85	111	x		n = 1
	11 <sup>B</sup>	SIIF	85	111	x		n = 1
	13 <sup>B</sup>	SIIF	85	111	x		n = 1
	17 <sup>B</sup>	SIIF	85	111	x		n = 1
	23 <sup>B</sup>	SIIF	85	111	x		n = 1
	26 <sup>B</sup>	SIIF	85	111	x		n = 1
	liver 50 <sup>*</sup>	VETN	82-85	210	2Z	VETN	
	filet 50 <sup>*</sup>	VETN	82-85	211	2E		
liver 40 <sup>*</sup>	NACE	86	510	2Z			
filet 20 <sup>*</sup>	NACE	86	511	2Z			
5	SIIF	86	111	2Z	NACE		
EPOC1	5	SIIF	86	605	x		
EPOC1 liver	800	NACE	86	610	x		

-----  
<sup>\*</sup> on wet weight basis  
<sup>A</sup> 2 prawn and 1 mussel sample, 1984  
<sup>B</sup> variable detection limit due to sample quantity  
/...

/...

## Intercalibration exercises

Sea water:

- 4H - ICES/JMG Fifth Intercalibration on Trace Metals in Estuarine Waters (JMG 5/TM/SW) - 1983, Preliminary report 1986.
- 4I - ICES/JMG Sixth Intercalibration on Trace Metals in Estuarine Waters (JMG 6/TM/SW) - 1986, Preliminary report 1987.
- 4Z - Intercalibration exercise for SIIF/SERI (Cd) and NIVA/IAMK (IAMK=Chalmers Inst., Göteborg) - 1985.

Sea bed sediment:

- 7E - ICES, First Intercalibration Exercise on Trace metals in Marine Sediments (1/TM/MS) - 1984.

Marine biota:

- 1E - ICES, Fifth Intercalibration Exercise on Trace Metals in Biological Tissues (5/TM/BT) - 1978.
- 1F - ICES, Sixth Intercalibration Exercise on Trace Metals (Cadmium and Lead only) in Biological Tissues (6/TM/BT) - 1979.
- 1G - ICES, Seventh Intercalibration Exercise on Trace Metals in Biological Tissues - Part A (7/TM/BT) - 1983.
- 1H - ICES, Seventh Intercalibration Exercise on Trace Metals in Biological Tissues - Part B (7/TM/BT) - 1985 (preliminary report 1987).
- 1Z VETN - Interlabcalibration exercise with VETN and SIIF 1983, mercury and cadmium in cod filet and liver.
- 1Z NIVA - Interlabcalibration exercise with VETN, NACE and NIVA 1986 (Hg, Cd, Cu, Pb and Zn in 6 samples).
- 2D - ICES Fourth Intercalibration Exercise on Organochlorines (mainly PCBs) in Biological Tissues (Sample No.5) - 1979 (4/OC/BT).
- 2E - ICES Fifth Intercalibration Exercise on Organochlorines (PCBs only) in Biological Tissues - 1982 (5/OC/BT).
- 2Z VETN - Interlabcalibration exercise with VETN among others, 1983, PCB and HCB in cod liver.
- 2Z NACE - Interlabcalibration exercise with NACE, VETN and SIIF 1986 (PCB (all labs), DDE, OCS, HCB and DCB (NACE and VETN)).

### 3. Analyses of sea water

#### 3.1 Sampling method codes

code      description

##### Samplers for sea water

- 1      Hydrobios hydrographical water sampler
- 2      Ruttner industrial water sampler
- 3      Ultracleaned polyethylene flask
- 4      Ultracleaned glass Erlenmeyer flask

##### Sampler deployment for sea water

- 1      2 nylon lines
- 2      Nylon line, brass messenger
- 3      By plastic-gloved hands

##### Methods of pretreatment of sea water samples

- 0      None
- 1      Membrane filtre

##### Methods of preservation of sea water samples

- 0      None
- 1      Nitric acid addition
- 2      Freezing
- 3      Nitric acid addition and freezing

#### 3.2 Inorganic determinations

code      description

320      Mercury in sea water (SIIF)

Reference: S.H. Omang, 1971. Determination of mercury in natural waters and effluents by flameless atomic absorption spectrophotometry. Anal. Chim. Acta (1971) 53: 415-420.

322      Mercury in sea water (SERI)

Reference: Iverfeldt, Å, 1984. Structural, thermodynamic and kinetic studies of mercury compounds; applications within the environmental mercury cycle. PhD thesis. Department of Inorganic Chemistry, Chalmers University of Technology and University of Göteborg. 48pp. + 7 appendices.

Abstract (Iverfeldt, 1984)

The 0.5-liter glass Erlenmeyer sampling bottles are specially cleaned: first filled with 6M HCl for 7 days, then 7M HNO<sub>3</sub> for 7 days and finally with deionized water with 1ml conc. HNO<sub>3</sub>/liter sample for 7 days. The bottles are rinsed extensively between each stage. After sampling, ≈1ml conc. HNO<sub>3</sub>/liter sample is added to achieve a pH of 1-2.

Mercury is preconcentrated on a gold trap after being reduced and volatilized by NaBH<sub>4</sub> from an all quartz glass reduction vessel. Mercury free nitrogen gas is used for purging.

The gold trap is constructed as gold grains in layers separated by SiO<sub>2</sub> glass pieces. All gold traps used are individually calibrated and give the same response.

The gold traps are analysed by a double amalgamation step; i.e. the mercury is transferred by heating, to about 800°C, to a second gold trap. This gold trap is analyzed using a helium direct current-plasma emission spectrometer (DCPAES). A Keithley 427 Current Amplifier and a Shimadzu Chromatopac C-R2AX Integrator are used. The system is optimized for the mercury line at 253.65 nm using a Hg(0) diffusion tube.

The stable sensitivity of the DCPAES instrument is ensured by a check before and after every sample determination.

Standard solutions are prepared from commercial stock solutions of CH<sub>3</sub>HgCl (1000 ppm, Alfa Products) and mercuric nitrate (1ml = 1mg Hg, BDH Spectrosol).

This combination resulted in the extremely low detection limit of 0.02ng/litre with 5% reproducibility (Cossa & Courau, 1984.)

Reference: Cossa, D, Courau, P., 1984. ICES Fifth round

## Sea water

intercalibration for trace metals in seawater (Intercalibrations 5/TM/SW). Report of Section 4 - Round Robin Intercalibration for total Mercury in seawater. International Council for the Exploration of the Seas, May 1984.

330 Cadmium in sea water (SIIF)

Reference: P.E. Paus, 1973. Determination of Heavy Metals in Seawater by AAS. J. Anal. Chem. 118-122 (1973).

331 Cadmium, copper, zinc, lead, iron, nickel, cobalt in sea water (NIVA)

The 1-liter polyethylene sampling bottles are specially cleaned: first filled with 6M HCl for 7 days, then 7M HNO<sub>3</sub> for 7 days and finally with deionized water with 1ml conc. HNO<sub>3</sub>/liter sample for 7 days. The bottles are rinsed extensively between each stage. After sampling, ≈1ml conc. HNO<sub>3</sub>/liter sample is added to achieve a pH of 1-2.

The analysis uses chelation with APDC (ammonium-pyrrolidine dithio-carbamate) and DDTC (diethylammonium-N,N-diethyl-dithio-carbamate) extraction with freon, reversed extraction back into water and reading on a graphite furnace AAS or AAES (atomic absorption electrothermal spectrometry).

50-250ml of the acidified sample is transferred to a separatory funnel, buffer is added to pH=4.75 and 20ml of freon. The mixture is vigorously shaken for 120 seconds. After separation of the phases, the organic one is transferred to a 50ml plastic bottle. The extraction is repeated with further 10ml of freon, and the organic phase added to the first 20ml after separation. 0.1ml concentrated nitric acid is added to the organic phase. The bottle is vigorously shaken and let to stand at least 5 minutes. 4.9ml deionized water is added and the solution is shaken; this process is repeated after 30 minutes. More acid and water are used for back-extraction if a greater volume of the solution is necessary for the determination.

Apparatus: Perkin Elmer (P-E) 2380 AAS, HGA 500 (P-E graphite furnace), AS 40 (P-E autosampler), HCL (hollow cathode lamp).



332 Mercury in sea water (NIVA)

Sample-bottle preparation and sampling is the same procedure as 331.

$\text{SnCl}_2$  is added to 250 ml of acidified sample. The liberated mercury is driven off with air as carrier gas through a gold trap onto which the mercury is amalgamated. CVAAS (cold-vapour atomic absorption spectrometry) is used to quantify the sample. When all the mercury is trapped the gold is heated to  $500^\circ\text{C}$  and the mercury is driven off by the carrier gas into a quartz cell where the atomic absorption signal is measured at 253.7nm. Apparatus: Perkin Elmer (P-E) 300SG AAS converted with gold trap.

#### 4. Analyses of sea bed sediments

##### 4.1 Sampling methods

code      description

Sampling of sediment

GC      Reference: Niemistö (1974) gravity corer, inner diameter 50mm.  
(Niemistö, H., 1974. A gravity corer for studies of soft  
sediment. Havforskningsinst. Skr. Helsinki, 238:33-38)

Methods of storage/preservation of sea bed sediment samples

01      Frozen (prior to inorganic analyses) and freeze dried (prior  
to organic analyses)

Methods of grain size analysis of sea bed sediment

01      Dry sieving

Methods of structural analysis of sea bed sediment

01      Visual observation through clear plastic cores

Methods of sea bed sediment extraction

HN01 3      Concentrated HNO<sub>3</sub> (suprapur) for inorganic analyses on "fresh"  
(i.e., frozen) material.

EXN1 2      Shaking with non-polar solvents cyclohexane/isopropanol (1:1  
v/v) on freeze dried material.

## 4.2 Inorganic determinations

code      description

350      Mercury in sea bed sediment (NIVA)

### Drying procedure

An accurately weighed sample of approximately 1g is dried at 105°C for one hour. The samples are cooled in a desiccator for one hour before weighing. Normally, determinations are on wet samples and the water content is determined of a subsample.

### Extraction (oxidation)

Approximately 1g of the sample is accurately weighed in pyrex flasks, 20ml concentrated nitric acid (suprapur) is added and the solution heated 120°C for 30min in an autoclave. The solution is transferred to a 100ml volumetric flask and diluted to the mark with deionized water.

### Determination

Mercury is determined by CVAAS (cold-vapour atomic absorption spectrometry), using the instrument Coleman Model MAS-50. 50ml of the sample solution is transferred to the aeration flask. The lowest signal detectable is corresponding to 0,01µg mercury.

351      Copper and zinc in sea bed sediment (NIVA)

(Same procedure as 350: par.#1-3)

Copper and zinc is determined by flame atomic absorption spectrometry using acetylene/air flame. Instrument: Perkin Elmer model 2380. The wavelengths used are:

copper	324.7 nm
zinc	213.9 nm

352 Cadmium and lead in sea bed sediment (NIVA)

(Same procedure as 350: par.#1-3)

Cadmium and lead is determined by flameless atomic absorption spectrometry. Instrument: Perkin Elmer model 560 and graphite furnace HGA-500. The analysis program:

	<u>Cadmium</u>	<u>Lead</u>
Wavelength, nm	228.8	283.3
Drying temperature, °C	110	110
Ramp time, sec.	1	1
Drying time, sec.	30	30
Ashing temperature, °C	300	300
Ramp time, sec.	1	1
Ashing time, sec.	40	40
Atomization temperature, °C	2000	2100
Ramp time, sec.	0	0
Internal gas flow, ml/min	50	50
Atomization time, sec.	7	7

#### 4.3 Organic determinations

code      description

360      PCB in sea bed sediment (NIVA)

The method is quite similar to SIIF method JMG code 110.

##### Cleaning of chemicals and equipment

The equipment is washed with soap and water, then rinsed in 1:5 mixture of HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>, respectively. Finally, the equipment is rinsed with acetone and cyclohexane.

All solvents are distilled in a special room for this purpose. Distilled water is shaken twice with distilled cyclohexane before use. Sodium sulphate is washed twice with distilled cyclohexane and heated to 550°C.

### Extraction

10g freeze dried, homogenized material is extracted twice with a mixture of non-polar solvents cyclohexane/isopropanol (1:1 v/v). The two hour extraction is performed in a continuously shaken Erlenmeyer flask with 200ml solvent mixture. The extraction is repeated with half solvent volume. The two extracts are combined and the cyclohexane phase separated by addition of 150+ml distilled water (excess water relative to isopropanol). The isopropanol/water phase is decanted and the cyclohexane phase washed once with distilled water (several times if also determination of total persistent organic chlorine). The extract is dried over sodium sulphate and then weighed.

### Determination of fat

A part of the cleaned and dried cyclohexane extract is evaporated in an oven to constant weight at 100<sup>0</sup>C.

The results are corrected for loss of cyclohexane. Cyclohexane output is calculated as sum cyclohexane extracts from first and second extraction (grams) minus fat in the same extract.

### Clean-up of extract

2ml cyclohexane extract is shaken vigorously with 2ml concentrated sulphuric acid and then centrifuged. This process is repeated.

### Gas chromatographic condition

Carlo Erba 2350 with ECD. Splitless injection at 70<sup>0</sup>C and then programmed temperature raise with 7<sup>0</sup>/min to 230<sup>0</sup>C. Column: 30mx0.259mm (inner diameter), 0.25 $\mu$  DB-5 fused silica capillary column. Carrier gas: H<sub>2</sub>, 0.8 bar.

### Identification and quantification

The sample is quantified using 4-5 dominant peaks in the Clophen A60 standard.

#### 4.4 Organic carbon determinations

<u>code</u>	<u>description</u>
390	<u>Total organic nitrogen and organic carbon (CORG) in sea bed sediment (NIVA)</u>  5-8mg of freeze dried sample is weighed in a tin-foiled capsule and heated to over 1000 <sup>0</sup> C in an oven. The carbon in the gas is analyzed in a C-N 1106 Carlo-Erba element analyzer. Detection limit for C is 0.2 µg/mg and N is 0.1 µg/mg.

## 5. Analyses of marine biota

### 5.1 Inorganic determinations

code      description

120      Mercury in biota (SIIF)

Representative samples are homogenized in a whirlmixer.

1.0g sample is weighed into a special digestion apparatus with reflux (Bethge apparatus).

10ml conc.  $\text{HNO}_3$  and 1ml 47% HBr is added and the solution boiled for approximately 30min. under reflux.

The solution is cooled down to room temperature and diluted to volume into a 50 ml volumetric flask with distilled water.

Mercury is determined with CVAAS (cold-vapour atomic absorption spectrometry). Mercury is reduced with  $\text{SnCl}_2$ .

121      Mercury in shellfish (SIIF)

Same procedure as 120 but bombe digestion (pressurized decomposition) with  $\text{HNO}_3$  at  $160^\circ\text{C}$  is used instead of pretreatment with  $\text{HNO}_3$  and HBr.

130      Cadmium, lead, copper and nickel in biota (SIIF)

Representative samples are homogenized in a whirlmixer. 1g freeze dried sample is weighed into a vitrosil vessel and dried at  $110^\circ\text{C}$  to constant weight to determine the total water content.

The vessel is then placed in a cold muffle furnace and the temperature increased slowly to  $450^\circ\text{C}$ . The vessel is removed from the furnace and cooled down to room temperature. After wetting the ash with 1ml conc.  $\text{HNO}_3$  and approximately 2ml  $\text{H}_2\text{O}$ , gentle heating on a hot plate is performed.

The final solution is diluted to volume into a 50 ml volumetric flask with distilled water.

Cadmium is determined after extraction with APDC and MIBK (ammonium-pyrrolidine-dithio-carbamate and methylisobutylketone) with flame atomic absorption spectrometry.

131 Zinc and manganese in biota (SIIF)

Same procedure as 130 (cadmium) but without extraction with APDC/MIBK.

220 Mercury and selenium in fish (VETN)

Samples are digested in a mixture of nitric and perchloric acid and the mercury content is determined by CVAAS (cold-vapour atomic absorption spectrometry).

Reference: Haugen, A., Høle, R., and Norheim, G., 1985. Automated hydride generator determination of selenium and mercury in biochemical material. Proc. from 10th. Nordic Atomic Spectroscopy and Trace Element Conference, August 6-9. 1985, Turku, Finland.

Abstract (Haugen et al., 1985)

Tissue samples are digested in a mixture of nitric and perchloric acid in a temperature programmable aluminium block. Maximum temperatures for the mercury and selenium determinations are 180 and 225°C, respectively. After reduction of hexavalent selenium with hydrochloric acid and dilution, the samples are transferred to a programmable sample changer. Both elements are determined with hydride generator producing a continuous, integratable signal. The precision at an absorbance reading of 0.4 is better than 1% and the quantification limit is better than 0.02 µg/g, when using a 1.0g sample. Good agreement was obtained with other methods. Seven determinations of selenium in NBS bovine liver (1577a) gave an average of 0.71µg Se/g, which is equivalent to the certified value.



230 Cadmium in fish (VETN)

Samples are digested by boiling with concentrated nitric acid (Suprapur) during several hours. The metal content is recorded by graphite furnace atomic absorption spectrometry. Quantification is based on standard addition to the digested samples.

240 Selenium in biota (VETN)

Reference: Norheim, G. and Nymoen, U.K., 1981. Fluorimetric determination of selenium in biological material using automatic digestion. Lecture presented at 8th. Nordic trace element and microchemistry conference, Sandefjord, Norway, 10-13, June 1981.

Abstract (Norheim & Nymoen, 1981)

The fluorimetric method is used, employing 2,3-diaminonaphtalene (DAN) as a complexing agent. The method uses 5g of material in an automatic wet digestion procedure with 17ml of 3+7 mixture of perchloric and nitric acid. The solution is heated slowly (225<sup>0</sup>C) in a thermostatically controlled aluminium block to distil off the nitric acid without charring. After digestion the hexavalent selenium is reduced with hydrochloric acid. EDTA (ethylenediaminetetraacetic acid) is added and aminoacetic acid is used as buffer. The pH is adjusted to 2.4 using a 35cm long electrode. DAN is added and the solution is heated at 60<sup>0</sup>C for 1hr. Finally, the solution is extracted with cyclohexane and the selenium content is measured fluorometrically on a Perkin-Elmer Model 1000 filter instrument. The detection limit is 10ppb wet weight.

310 Mercury in biota (NIVA)Drying procedure

An accurately weighed sample of approximately 1g is dried at 105<sup>0</sup>C for one hour. The samples are cooled in a desiccator for one hour before weighing. Normally mercury is determined on wet samples and the water content is determined of a subsample.

### Extraction (oxidation)

50-200mg freeze-dried sample is weighed in teflon vessels, 2ml concentrated nitric acid (suprapur) is added and capped loosely. The solution is heated for about 2hrs. at 50°C in a thermostatically controlled aluminium block until foaming ceases. The temperature is raised to 110°C and kept there for 6-8hrs. The solution is then cooled.

For samples with high fat content (e.g., liver) 2ml of 30% H<sub>2</sub>O<sub>2</sub> is added and the solution is heated again to 110°C for 3-4hrs.

After cooling the solution is diluted to 25ml. For mercury samples approximately 200mg material is used and the solution is diluted to 100ml.

### Determination

Mercury is determined by CVAAS (cold-vapour atomic absorption spectrometry), using the instrument Coleman Model MAS-50. 50ml of the sample solution is transferred to the aeration flask. The lowest signal detectable is corresponding to 0.01µg mercury.

### 311 Copper and zinc in biota (NIVA)

(Same procedure as 310: par.#1-5)

Copper and zinc are determined by flame atomic absorption spectrometry using acetylene/air flame. Instrument: Perkin Elmer model 2380. The wavelengths used are:

copper	324.7 nm
zinc	213.9 nm

312 Cadmium and lead in biota (NIVA)

(Same procedure as 310: par.#1-5)

Cadmium and lead are determined by flameless atomic absorption spectrometry. Instrument: Perkin Elmer model 560 and graphite furnace HGA-500. The analysis program:

	<u>Cadmium</u>	<u>Lead</u>
Wavelength, nm	228.8	283.3
Drying temperature, °C	110	110
Ramp time, sec.	1	1
Drying time, sec.	30	30
Ashing temperature, °C	300	300
Ramp time, sec.	1	1
Ashing time, sec.	40	40
Atomization temperature, °C	2000	2100
Ramp time, sec.	0	0
Internal gas flow, ml/min	50	50
Atomization time, sec.	7	7

401 Mercury in biota (FIER)

Reference: Eliann E. & Julshamn, K., 1978. A method for the determination of selenium and mercury in fish products using the same digestion procedure. Atomic Absorption Newsletter, 17(6):135-138 (November - December 1978).

Reference: Julshamn, K., Ringdal, O., & Braekkan, O.R., 1982. Mercury concentration in liver and muscle of cod (Gadus morhua) as an evidence of migration between waters with different levels of mercury. Bull. Environm. Contam. Toxicol. 29:544-549 (1982).

Abstract (Eliann & Julshamn, 1978; Julshamn et al., 1982)

Representative samples are homogenized in a whirlmixer. About 1g of sample tissue is dried at 95°C for 24 hrs. 0.5g sample is weighed into a special digestion apparatus with reflux (Bethge apparatus).

10 ml conc.  $\text{HNO}_3/\text{H}_2\text{SO}_4$  (1+1) +  $\text{V}_2\text{O}_5$  (0.1% w/v) are added. The solution is boiled for approximately 30 min under reflux.

The solution is cooled down to room temperature and diluted to volume into a 50ml volumetric flask with distilled water and  $MnO_4$ .

Mercury is determined by CVAAS (cold-vapour atomic absorption spectrometry), EDL (electrodeless discharge lamp), wavelength = 253.6nm, Perkin Elmer (P-E) 370 AAS, P-E mercury analysis system (303-0830 and 303-0832). Mercury is reduced with  $SnCl_2$  to avoid interference with iodine.

Quantification is based on standard curves. Detection limit: dry weight sample. 5 ng/g.

402 Cadmium in biota (FIER)

Reference: Julshamn, K. & Brækkan, O.R., 1975. Determination of trace elements in fish tissues by the standard addition method. Atomic Absorption Newsletter, 11(3):49-52. (May-June 1975). (Concerns: Mn, Fe, Cu, Zn, Cd and Pb).

Reference: Julshamn, K., 1977. Inhibition of response by perchloric acid in flameless atomic absorption. Atomic Absorption Newsletter, 16(6):149-150 (November - December 1977). (Concerns: Mg, Al, Cr, Mn, Fe, Co, Ni, Cu, Zn, Cd and Pb).

Reference: Julshamn, K. & Andersen, K.-J., 1983. Subcellular distribution of major and minor elements in unexposed molluscs in Western Norway - II. The distribution and binding of cadmium, zinc, copper, magnesium, manganese and iron in the kidney and the digestive system of the common mussel Mytilus edulis. Comp. Biochem. Physiol. 75A(1):13-16 (1983). Pergamon Press.

Abstract (Julshamn & Brækan, 1975; Julshamn, 1977; Julshamn & Andersen, 1983)

Representative samples are homogenized in a whirlmixer. 1g of sample tissue is dried at 95<sup>0</sup>C for 24hr. 0.1g sample is weighed into a Sovirel test-tube (20ml). 2ml conc.  $HNO_3/HClO_4$  (9:1) is added and is boiled under pressure.

The solution is cooled down to room temperature and diluted to volume into a 10ml volumetric flask with distilled water.

Concentrations are determined by graphite furnace AAS or AAES (atomic absorption electrothermal spectrometry), HCL (hollow-cathode lamp, Perkin Elmer (P-E) 5000 AAS, HGA 500 (P-E graphite furnace), AS 50 (P-E autosampler) and wavelength = 228.8nm. Matrix modification reagent is  $(\text{NH}_4)_2\text{HPO}_4$ .

Quantification is based on standard addition to the digested samples: Amount added in the analyses: Cd 1ng/ml. Detection limit for dry tissue sample: 0.5ng/g.

403 Lead in biota (FIER)

(Same procedure as 402: par.#1 and #3-5).

Concentrations are determined by graphite furnace AAS or AAES (atomic absorption electrothermal spectrometry), EDL (electrodeless discharge lamp) Perkin Elmer (P-E) 5000 AAS, HGA 500 (P-E graphite furnace), AS 50 (P-E autosampler) and wavelength = 283.3nm. Matrix modification reagent is  $(\text{NH}_4)_2\text{HPO}_4$ .

Quantification is based on standard addition to the digested samples: amount added in the analysis Pb 10ng/ml. Detection limit for wet tissue sample: 10ng/g.

404 Copper in biota (FIER)

with high Cu content (> 1.5 ppm d.w.)

(Same procedure as 402: par.#1-5).

Concentrations are determined by flame AAS (atomic absorption spectrometry), HCL (hollow-cathode lamp, Perkin Elmer (P-E) 370 AAS and wavelength = 324.7nm without background correction.

with low Cu content (< 1.5 ppm d.w.)

(Same procedure as 402: par.#1-5).

Concentrations are determined by graphite furnace AAS or AAES (atomic absorption electrothermal spectrometry), HCL (hollow-cathode lamp, Perkin Elmer (P-E) 5000 AAS, HGA 500 (P-E graphite furnace), AS 50 (P-E autosampler) and wavelength = 324.7nm. No matrix modification reagent is used.

Quantification is based on standard addition to the digested samples: 10 ng/ml.

405 Zn in biota (FIER)

(Same procedure as 402: par.#1-5).

Concentrations are determined by flame AAS (atomic absorption spectrometry), HCL (hollow-cathode lamp, Perkin Elmer (P-E) 370 AAS and wavelength = 213.9 nm without background correction.

Quantification is based on standard curves.

406 Arsenic in biota (FIER)

(Same procedure as 402: par.#4-5).

Concentrations are determined by graphite furnace AAS or AAES (atomic absorption electrothermal spectrometry), HCL (hollow-cathode lamp) Perkin Elmer (P-E) 5000 AAS, HGA 500 (P-E graphite furnace) with a conventional tube without platform, AS 50 (P-E autosampler) and wavelength = 193.7 nm. Matrix modification reagent is a Ni-solution.

## 5.2 Organic determinations

<u>code</u>	<u>description</u>
110	<u>PCB HCB DDTEP (p,p'DDE + p,p'DDT) HCHG (<math>\gamma</math>HCH = <math>\gamma</math>BHC) in fish and shellfish (SIIF)</u>

### Cleaning of chemicals and equipment

The equipment is washed with soap and water, rinsed in water first and then with acetone. Finally, the glass equipment is heated to 550°C.

All solvents are distilled in a special room for this purpose. Distilled water is shaken twice with distilled cyclohexane before use. Sodium sulphate is washed twice with distilled cyclohexane and heated in the same way as the glass equipment.

#### Extraction

10g wet, homogenized material is extracted twice with a mixture of cyclohexane/isopropanol (1:1 v/v). The two hour extraction is performed in a continuously shaken Erlenmeyer flask with 200 ml solvent mixture. The extraction is repeated with half solvent volume. The two extracts are combined and the cyclohexane phase separated by addition of 150+ ml distilled water (excess water relative to isopropanol). The isopropanol/water phase is decanted and the cyclohexane phase washed once with distilled water (several times if also determination of total persistent organic chlorine). The extract is dried over sodium sulphate and then weighed.

#### Determination of fat

A part of the cleaned and dried cyclohexane extract is evaporated in an oven to constant weight at 100<sup>0</sup>C.

Alternatively, the extract is evaporated to constant weight in a flask with Vigreux column in oil bath at 1 atm., 110<sup>0</sup>C and reflux or in vacuum, 50<sup>0</sup>C, reflux and weak N<sub>2</sub>-stream.

The results are corrected for loss of cyclohexane. Cyclohexane output is calculated as sum cyclohexane extracts from first and second extraction (grams) minus fat in the same extract.

The precision of the fat determinations is roughly ±10%. Determinations are given at 0.1%. The two methods give comparable results.

#### Clean-up of extract

0.2g fat is dissolved in 2ml cyclohexane, shaken vigorously with 2ml concentrated sulphuric acid and then centrifuged. For further clean-up about 1ml of the sulphuric acid treated extract is treated with a solution of KOH.

Gas chromatographic condition

Hewlett-Packard 5730 A with ECD. Splitless injection at 60<sup>0</sup>C and then programmed temperature raise with 8<sup>0</sup>/min to 230<sup>0</sup>C. Column: 50mx0.3mm (inner diameter), 0.15µ SE-54 glass capillary column. Carrier gas: He, 20 psi.

Identification and quantification

By comparing the whole pattern with various commercial standard mixtures, it was found that Clophen 60 was in best accordance with the sample types. Some of the isomers in the PCB pattern were selected by comparison with standards of specific isomers and these were used for quantification:

<u>Internal code</u>	<u>Structure (-bifeny1)</u>	
3	2,3,6,2',5'	Pentachlorine
4	2,4,5,2',5'	Pentachlorine
9/10	2,4,5,2',3',6' / 2,4,5,3',4,	Hexachlorine/Penta-
14	2,3,4,2',4',5'	Hexachlorine/Penta-
15	2,3,4,2',3',4'	Hexachlorine/Penta-
16	2,3,4,5,2',4',5'	Heptachlorine

By the GC conditions used it was not possible to separate isomers 9 and 10.

HCB, HCHG ( $\gamma$ HCH= $\gamma$ BHC) and DDTEP (p,p'DDE + p,p'DDT) is determined by multi-level calibration curve. HCHG is identified and quantified by the breakdown product of HCHG (three trichlorobenzene peaks) after treatment with sodium hydroxide (NaOH)

111 PCB HCB DDTEP (p,p'DDE + p,p'DDT) HCHG ( $\gamma$ HCH =  $\gamma$ BHC) in fish and shellfish (SIIF)

Same procedure as 110, except that for organochlorine standard was Arochlor 1254 instead of Clophen A60 which is used earlier. The detection limit given for 1982 JMP data for this method is erroneously low. Corrected limits are given in the 1983 JMP data submitted

The detection limit is dependant on sample quantity. For example the detection limit for HCHG is approximately: 0.1ppb



with 10g dry weight material, 0.03ppb with 25g and 0.01 with 80g.

210 PCB, HCB, DDEPP in fish liver samples (VETN)

Reference: Norheim, G., 1978. The composition and distribution of PCB in arctic fox (Alopex lagopus) caught near Longyearbyen on Svalbard. Acta pharmacol. et toxicol. 42:7-13.

Abstract (Norheim, 1978)

Extraction: 0.5g of sample is homogenized in a mortar with 2.5g of anhydrous sodium sulphate and 2.5g purified sand and allowed to stand overnight in the dark. After being mixed with 2g magnesium sulphate, the dry powder is transferred to a short chromatographic column (20mmx10cm) equipped with ground glass stoppers and elutriated with 2x10ml diethyl ether. The column is carefully rotated to release air bubbles and the ether is allowed to stand for 2hrs in the column before elutriation. The ether is evaporated in centrifuge tubes and the residue dissolved in 1.0ml n-heptane. The extract is finally treated with 2.0ml concentrated sulphuric acid for about 1 hour. 5µl n-heptane is injected into the gas chromatograph.

Quantification: Phenoclor DP6 is used as standard. The peak height of 2,4,5-2',4',5' hexachlorobiphenyl is used to quantify PCB.

211 PCB in fish filet samples (VETN)

Reference: Norheim, G., Økland, E.M., 1980. Rapid extraction of some persistent chlorinated hydrocarbons from biological material with low fat content. Analyst (Okt.1980)105:990-992.

Abstract (Norheim & Økland, 1980)

Apparatus: A Carlo Erba 2100 gas chromatograph equipped with a nickel-63 electron-capture detector and a 2m x 3mm (inner diameter) glass column is used. The column material is 1.5% SP-2250 - 15.9% SP-2401 on 100-120-mesh Supelcon AW DMCS. The column, injector and detector temperatures are 200, 250 and 275<sup>0</sup>C, respectively. Argon

-methane (95+5) is used as the carrier gas, the flow-rate being  $55\text{ml}\cdot\text{min}^{-1}$ . The electrometer attenuation is  $\times 128$ .

Reagents: Sulphuric acid, 95-97%. Pro analysi grade (Merck). Heptane. Pro analysi grade (Merck). Hexachlorobenzene. Pract. grade (Fluka). Octachlorostyrene. Obtained as a gift from Norsk Hydro.

Standard solutions: Amounts of 100mg each of hexachlorobenzene and octachlorostyrene are dissolved in 100ml of heptane and the mixture is diluted 1 + 50 000 with heptane.

Procedure: A 0.5g amount of sample is accurately weighed into a 10ml Soveril glass tube fitted with a screw-cap, and 6ml of concentrated sulphuric acid are measured into the tube. The tube is placed in a thermostatically controlled oven at  $60^{\circ}\text{C}$  for 4hr, during which time it is shaken lightly a few times to ensure complete solubilisation of the sample. After cooling, 1.0ml of heptane is pipetted into the tube, the screw-cap put on and the tube shaken for about 3min. Finally, the tube is centrifuged with the screw-cap on, after which the sample is ready for gas chromatography. An injection volume of  $5\mu\text{l}$  is used.

Quantification: The same standard and isomer as in 210 are used to quantify the sample.

510

PCB, HCB, DDEPP (p,p'DDE), DDTTP (p,p'DDT), HCHG ( $\gamma$  HCH =  $\gamma$  BHC) in fish liver (NACE)

Pretreatment and fat determinations: Samples are homogenized in a Waring blender. Homogenised liver samples are ground in a mortar with sea sand and anhydrous sodium sulphate and allowed to stand overnight. The samples are mixed with magnesium-sulphate, transferred to a glass column with sintered glass frit and extracted with diethyl ether. The ether is collected in pre-weighed tubes, evaporated and the amount of fat determined by weighing.

The fat extract is dissolved in hexane for pesticide

analyses and treated with concentrated sulphuric acid with gentle agitation. After centrifugation the hexane phase is used for gas chromatography (GC) analysis for pesticides and PCBs. An aliquot of the hexane phase is also treated with sodium alcoholate to convert p,p'DDT to p,p'DDE for the determination of DDT by the increase in DDE.

Moisture content: samples are dried in an oven overnight (16hr) at 105<sup>0</sup>C, equilibrated in a desiccator for 1hr and re-weighed. Filet samples are also dried for 72hr at 45<sup>0</sup>C for later determination of mercury.

GC analyses: a Perkin Elmer 8500 GC equipped with an auto-sampler and an electron capture detector (Ni-63) and connected to a 7500 computer with Chrom 3 software is used. The column is a glass 2mx1/4", 2mm (inner diameter) packed with 1.5% SP-2250/1.95% SP-2400 on Suplecoport 100/120. The carrier gas is argon with 5% methane at a flow rate of 40ml/min. The oven temperature is 210<sup>0</sup>C, with the injector at 250<sup>0</sup>C and the detector at 300<sup>0</sup>C. The amount of sample injected is 2µl and the analysis takes 40min.

Reference standards: commercially available Aroclor 1242, Aroclor 1254, Aroclor 1260 and Supelco's CP pesticide mix are used in addition to a special mixture containing Phenoclor DPG (60% chlorination), hexachlorobenzene (HCB), octachlorostyrene (OCS), p,p'-DDE and decachlorobiphenyl (DCB).

Quantification: response factors are calculated from the integrated areas for each component and the amount injected. The corresponding peaks for the samples are integrated and the concentrations calculated from the area and the response factor. A simplified method for the calculation of the concentrations of PCBs is used. This is based on using the area for the peak in the Phenoclor standard corresponding to 2,4,5,2',4',5'-hexachlorobiphenyl and the total amount of PCB components injected. The concentrations for samples are calculated from the area of the peak corresponding to that used in

the standard. This requires that the pattern of PCB components in the sample corresponds to that of the Phenoclor standard.

Detection limits: the minimum detectable amount corresponds to 0.01µg/g wet weight for liver samples. This gives minimum quantification limits of 0.04µg/g for PCB.

511 PCB in fish filet (NACE)

Pretreatment: Homogenised filet samples are treated with concentrated sulphuric acid for 4hr at 60<sup>0</sup>C and PCBs extracted with hexane. After centrifugation the hexane phase is used for gas chromatography (GC) analysis.

(Same procedure as 510: par.#3-6).

Detection limits: the minimum detectable amount corresponds to 0.005µg/g wet weight for liver samples. This gives minimum quantification limits of 0.02µg/g.

605 EPOC1 in shellfish (SIIF)

The cyclohexane extract from chlorinated hydrocarbon analysis is reduced in volume (by evaporation) and treated with concentrated H<sub>2</sub>SO<sub>4</sub> until the extract is clear. An aliquot is sent to the Institue for Energy Technology (Kjeller, Norway) to be exposed to neutron bombardment in a JEEP II atomic reactor. The radioactivity of the persistant chlorine isotope is measured and quantified against a complete procedural blank.

The detection limit is 5 ppb wet weight.

610 EPOC1 in fish liver (NACE)

Same procedure as 605 but higher detection limit.

The detection limit is 800 ppb wet weight.

### 5.3 Fat determinations

<u>code</u>	<u>description</u>
A	Weight of extracted solids during chlorinated hydrocarbon determinations: procedure 110 (SIIF)
B	Weight of extracted solids during chlorinated hydrocarbon determinations: procedure 510 (NACE)