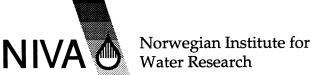
# **Report 537/93**

Client	State Pollution Control Authority
Contractor	NIVA

Joint Monitoring Programme - JMP

# Overview of Analytical Methods Employed by JMP in Norway 1981-92





# **NIVA - REPORT**

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#### Abstract:

This report is a compilation of analytical method codes and descriptions used in the Norwegian contribution to the Joint Monitoring Programme monitoring of contaminants (mainly: selected metal, organochlorines, polycyclic aromatic hydrocarbons) in sea water, sea bed sediment and marine biota collected 1981-1992 and analyzed by June 1993. The method descriptions are brief and focus on the principles involved.

- 4 keywords, Norwegian
- 1. Analytiske metoder
- 2. Mijøgifter
- 3. Sediment
- 4. Organismer

- 4 keywords, English
- 1. Analytical methods
- 2. Contaminants
- 3. Sediment
- 4. Organisms

1210

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For the Administration

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# O-80106

# Joint Monitoring Programme - JMP

# Overview of analytical methods employed by JMP in Norway 1981-92

Project coordinator;

Norman W.Green

3.11.93

### **FOREWORD**

This report presents the Norwegian method overview and brief descriptions of chemical analyses used for 1981-92 investigations for the Joint Monitoring Programme (JMP). JMP is administered by the Oslo and Paris Commissions (OSPARCOM) under the guidance of the International Council for the Exploration of the Seas (ICES). The programme is implemented by participating members comprising the Joint Monitoring Group (JMG).

Information for this report was compiled by the Norwegian Institute for Water Research (NIVA) by contract from the Norwegian State Pollution Control Authority (SFT) (NIVA contract 80106) The report is an updated version of information reported earlier (OSPAR, 1984; Green, 1988; see Section 1)

The different methods have been reviewed by representatives of the respective analytical laboratories

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# 1. Introduction

This report outlines the analytical methods employed under the Joint Monitoring Programme (JMP) in Norway 1981-1992 for the analyses of contaminants in sea water, sea bed sediment and marine biota samples (collected 1981-92). 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 largely a revision of OSPAR (1984) and Green (1988) and includes updated and extended information but retains the same codes and abbreviations. Section 2 summarizes the analytical methods employed sorted in various ways for convenient reference. Section 3 gives a brief description of each method.

# 2. Method overview

#### 2.1. Medium and method code

Overview of analytical methods for trace metals, chlorinated hydrocarbons and other substances in the marine environment.(abbreviations are defined in section 2.4)

Medium	method code	institute code	contaminant(s) etc. (JMP code)	sampling year
Sea water				
	320	SIIF	Hg	84
	322	SERI	Hg	84-86
	330	SIIF	Cd	83
	331	NIVA	Cu,Zn	85-90
	331	NIVA	Cd,Pb	84-90
	331	NIVA	Fe,Ni,Co	86
	332	NIVA	Hg	86-90
Sea bed sediment				
	350	NIVA	Hg	86,90,92
	351	NIVA	Cu,Zn	86,90,92
	352	NIVA	Cd,Pb	86,90,92
	360	NIVA	PCB <sup>1</sup>	86,92
	369	NIVA	$PAH^2$	92
	390	NIVA	NTOT,CORG	86,90,92
	760	<b>IMRN</b>	PCB <sup>1</sup>	90
	769	<b>IMRN</b>	PAH <sup>2</sup>	90

.../...

### Medium and method code overview (cont'd)

Medium	method	institute	contaminant(s) etc.	sampling
	code	code	(JMP code)	year
Marinebiota				
	120	SIIF	Hg	81-85
	121	SIIF	$Hg^3$	83-85
	130	SIIF	Ni	83
	130	SIIF	Cu,Pb	84
	130	SIIF	Cd	81-85
	130	SIIF	$Pb^3$	83-85
	131	SIIF	Zn	83
	132	SIIF	Mn,Zn	84-85
	220	VETN	Hg	82-85
	220	VETN	Se <sup>3</sup>	85
	230	VETN	Cd	82-85
	240	VETN	Se	82
	310	NIVA	Hg	86-92
	311	NIVA	Cu,Zn	86-92
	312	NIVA	Cd,Pb	86-92
	401	FIER	Hg	84,876
	402	FIER	Cd	84,87
	403	FIER	Pb	87
	404	FIER	Cu	87
	405	FIER	Zn	87
	406	FIER	$As^3$	83
	110	SIIF	PCB <sup>1</sup>	81
	111	SIIF	$PCB^1$ ,	82-91
	210	VETN	PCB,HCB,DDEPP	82-85
	211	VETN	PCB	82-85
	309	NIVA	$PAH^2$	87,92
	340	NIVA	PCB <sup>1</sup>	87,90-92
	341	NIVA	PCB <sup>1</sup>	90-92
	510	NACE	PCB <sup>1</sup>	86-89
	511	NACE	PCB	86-89
	605	SIIF	EPOCI	86-91
	610	NACE	EPOCI	86-89
	615	NIVA	EPOCI	90-92

selected individual chlorobiphenyls and pesticides (cf., Section 2.4).
 selected PAHs (cf., Section 2.4).
 not in data base

#### 2.2. Seawater and sediment

Overview of chemical analyses employed by JMP 1981-1991 in Norway for trace metals, chlorinated hydrocarbons and other substances in sea water and sea bed sediment. Detection limits (detec.) are listed. Intercalibration codes (and in some cases laboratory codes, see Table 4) 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.

medium	parameter	detection limit ppb <sup>1</sup>	institute	sample year	method code	Intercal- ibration
Sea water				•		
	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	86-90	331	<b>4</b> I
		50	NIVA	85	331	4Z
	Zn	70	NIVA	85	331	4Z
		10	NIVA	86-90	331	4I
	Cd	1	NIVA	85	331	4Z
		0.5	NIVA	86-90	331	4I
		5	NIVA	84	331	4Z
		100	SIIF	83	330	4F
	Hg	0.02	SERI	84-85	322	4H
		0.02	SERI	86-90	322	4I
		2	NIVA	86	332	<b>4</b> I
		10	SIIF	84 .	320	4F
	Pb	6	NIVA	86-90	331	4Z
Sea bed sediment						
	Cu	10	NIVA	86,87,90	351	7E
	Zn	100	NIVA	86,87,90	351	7E
	Cd	1	NIVA	86,87,90	352	7E
	Hg	10	NIVA	86,87,90	350	7E
	Pb	50	NIVA	86,87,90	352	7E
	PCB	5	NIVA	86	360	498
	PCB	0.05	NIVA	92	360	8C-
	PAH	1	NIVA	92	369	-
	NTOT	0.1*	NIVA	86,87,90	390	-
	CORG	0.2*	NIVA	86,87,90	390	_
	PCB <sup>2</sup>	0.05	<b>IMRN</b>	90	760	8B
	PAH <sup>3</sup>	1	IMRN	90	769	-

<sup>\*)</sup> mg/g

1) note definition in section 2.6.

<sup>2)</sup> all individual chlorobiphenyls and pesticides (cf., section 2.4).

<sup>3)</sup> all PAHs (cf., section 2.4).

#### 2.3. Biota

Overview of chemical analyses employed by JMP 1981-1992 in Norway for trace metals, chlorinated hydrocarbons and other substances in biota. Detection limit is the general limit for the method and not specific for a particular sample. "Count" (n) refers to number of values registered in JMP database at NIVA."N (<)" indicates number of values with less-than qualifier above detection limit. Note qualifier for detection limit in section 2.6. Code explanations for intercalibration excercies are listed in Section 2.5

This section is sorted in three ways for easy reference:

Section 2.3.1. Method, laboratory and tissue

Section 2.3.2 Tissue, method and laboratory

Section 2.3.3. Contaminant, year-laboratory and tissue

# 2.3.1. Method, laboratory and tissue

Analytical overview sorted by METHOD, LAB. and TISSUE.

Analy			ew sorted by		LAB	. and	TISS	OB.				
Method	Lab.	Tissue=	Sample Year	Contam	nants							
110	SIIF	Fish fillet Fish liver	81 81	PCB PCB								
111	ettr	Mussel	81	PCB								
111	SIIF	Mussel Mussel	82-91 83-91	PCB DDTEP,	нсв							
		Mussel Mussel	86-87,89-91 87-91	HCHG		CDES						
		Mussel	88-91	CB138,	CB180, CB153,	CB28						
		Mussel Shrimp tail	89-91 82,84,86,88,90	CB118 PCB								
		Shrimp tail	84,86,88,90	DDTEP,	HCB							
1		Shrimp tail Shrimp tail	86,90 88,90	НСНG СВ101,	CB138,	CB153,	CB180,	св28 "	CB52			
		Shrimp tail "Other	90 88	CB118			_	CB28 ,		DDTED	uce	DCD
120	SIIF	Fish fillet	81	HG	00.50,	00.35,	00100,		, ,	DUTER,	nub,	FCB
1		Fish liver Mussel	81 81-85	HG HG								
130	SIIF	Shrimp tail Fish fillet	82,84   81	HG CD								
		Fish liver	81	CD								
		Mussel Mussel	81-85 83	CD NI								
1		Mussel Mussel	83-84 83-85	CU PB								
1		Shrimp tail	82,84	CD								
131	SIIF	Shrimp tail Mussel	84   83	CU , ZN	PB							
132	SIIF	Mussel Shrimp tail	84-85 84	MN .	ZN ZN							
210	VETN	Fish fillet	83	DDEPP,	HCB							
		Fish liver Fish liver	82-85 83-85	DDEPP, HCB	PCB							
211	VETN	Fish fillet Fish fillet	82-85 83	PCB	uce							
220	VETN	Fish fillet	82-85	DDEPP,	nco							
230	VETN	Fish liver Fish liver	82   82-85	HG CD								
240	VETN	Fish fillet Fish liver	82 82	SE								
309	NIVA	Fish fillet	92-93	SE ACNE ,	ACNLE,	ANT ,	BAA_,	BAP ,	BBF ,	BEP ,	BGHIP,	BIPN .
1				BJKF ,	CHR ,	COR ,	DBA3A,	DBP ,	FLE ,	FLU ,	ICDP ,	
		Fish liver Fish liver	87 92-93	PAH							PYR	
		rish tiver	92-93	BJKF ,	ACNLE, CHR,	COR ,	DBA3A,	DBP ,	BBF , FLE ,	BEP ,	BGHIP,	
		Mussel	92	NAP1M,	NAPZM, ACNLE,	NAPDI,	NAPTM.	PA ,	PAM1 ,	PER ,	PYR	
1			1-	BJKF ,	CHR ,		DBA3A,	DBP ,	FLE ,	FLU .	BGHIP,	NAP ,
		Shrimp tail	92	ACNE ,	NAPZM, ACNLE,	NAPDI,	NAPTM, BAA_,	PA , BAP ,	PAM1, BBF.	PER , BEP .	PYR BGHIP,	BIPN .
				BJKF ,	CHR ,	COR ,	DBA3A,	DBP ,	FLE ,	FLU ,	ICDP ,	NAP ,
310	NIVA	Fish fillet	86-93	HG	MATEN,	MAPDI,	MAP IM,	ΡΑ ,	PAM1 ,	PEK ,	PYR	
		Mussel Shrimp tail	86-92 86,88,90,92	HG HG								
311	NIVA	Other	88 86-93	HG	711							
I		Mussel	86-92	CU ,	ZN							
		Shrimp tail Other	86,88,90,92 88	cu ,	711							
312	NIVA	Fish liver Mussel	86-93 86-92	CD ;	PB							
1		Mussel	92	CR ,	NI							
1		Shrimp tail Other	86,88,90,92 88	CD ,	PB PB							
340	NIVA	Fish liver Fish liver	87 90-93	PCB .		CD 170	C01E7		00000	cnaa	COFÓ	
			1	HCB ,	CB118,	HCHG ,	OCS ,	CB180, QCB,	CB209, TDEPP	CB28 ,	CBDZ ,	DDEPP,
341	NIVA	Fish liver Fish fillet	91-93 90-93	CB105, CB101	CB156 CB118	CB138	CB153	QCB , CB180,	CB200	CR28	CB52	DUEDD
		Fish fillet		HCB ,	HCHA,	HCHG ,	ocs,	CB180, QCB ,	TDEPP	,	, عددت	UUCPP,
l		Mussel	91-93 92	CBIUI,	CBIUD,	CBIIO,	CB 130,	CB155.	CB156.	CB180 -	CB209.	CB28
		Shrimp tail	92	CB52 ,	DDEPP,	HCB ,	HCHA,	HCHG CB153,	OCS ,	QCB ,	TDEPP	cpno
401	ETER			CB52 ,	DDEPP,	HCB ,	HCHA,	HCHG ,	ocs ,	QCB ,	TDEPP	LD20 ,
402	FIER FIER	Fish fillet Fish liver	84,87 84,87 87	HG CD								
403 404	FIER FIER	Fish liver Fish liver	87 87	PB CU								
405	FIER	Fish liver	87	ZN								
510	NACE	Fish liver Fish liver	86-89 89	DDEPP, CB101.	DDTPP, CB118	HCB , CB138.	HCHG , CB153	PCB CB180,	CB28	CB52		
511	NACE	Fish fillet Fish liver	86-89 86	PCB		/ /	,	,	,			
605	SIIF	Mussel	86-91	PCB EPOCL								
I		Mussel Shrimp tail	89 86,88,90	EOCL EPOCL								
610	NACE	"Other	88	EPOCL								
615	NIVA		90-91	EPOCL EPOCL								
610 615	NACE NIVA	Fish liver Fish liver	86-89 90-91	EPOCL EPOCL								-

### 2.3.2. Tissue, method and laboratory

Analytical overview sorted by TISSUE, METHOD and LAB.

Tissue	Method	Lab.=		Contaminants
Fish fillet	110 120 130 210 211 211 220 240	SIIF SIIF SIIF VETN VETN VETN VETN	81 81 81 83 82-85 83 82-85 83	PCB HG CD DDEPP, HCB PCB DDEPP, HCB HG SE
	309 310 341	NIVA NIVA NIVA	92-93 86-93 90-93	ACNE , ACNLE, ANT , BAA , BAP , BBF , BEP , BGHIP, BIPN BJKF , CHR , COR , DBA3A, DBP , FLE , FLU , ICDP , NAP NAP1M, NAP2M, NAPDI, NAPTM, PA , PAM1 , PER , PYR HG
Fish liver	341 401 511 110 120	NIVA FIER NACE SIIF SIIF	91-93 84,87 86-89 81	CB101, CB118, CB138, CB153, CB180, CB209, CB28, CB52, DDEPH HCB, HCHA, HCHG, OCS, QCB, TDEPP CB105, CB156 HG PCB PCB PCB
	130 210 210 220 230 240 309	SIIF VETN VETN VETN VETN VETN NIVA	81 82-85 83-85 82 82-85 82 87	HG CD DDEPP, PCB HG CD SE PAH
	309 311 312 340	NIVA NIVA NIVA	92-93 86-93 86-93 87	ACNE , ACNLE, ANT , BAA , BAP , BBF , BEP , BGHIP, BIPN BJKF , CRR , COR , DBA3A , DBP , FLE , FLU , ICDP , NAP NAP1M, NAP2M, NAPDI, NAPTM, PA , PAM1 , PER , PYR CU , ZN , CD , PB
	340 340 402 403	NIVA NIVA NIVA FIER FIER	90-93 91-93 84,87 87	PCB CB101, CB118, CB138, CB153, CB180, CB209, CB28, CB52, DDEPF HCB, HCHA, HCHG, OCS, QCB, TDEPP CB105, CB156 CD PB
Mussel	404 405 510 510 511 610 615 110	FIER FIER NACE NACE NACE NIVA SIIF SIIF	87 87 86-89 89 86 86-89 90-91 81	CU 2N DDEPP, DDTPP, HCB , HCHG , PCB CB101, CB118, CB138, CB153, CB180, CB28 , CB52 PCB EPOCL EPOCL PCB PCB
	111 111 111 111 120 130 130 130 131 131 132 309	SIIF SIIF SIIF SIIF SIIF SIIF SIIF SIIF	83-91 86-87,89-91 88-91 88-91 89-91 81-85 81-85 83-85 83-84 83-85 83-85	DDTEP, HCB HCHG CB101, CB180, CB52 CB138, CB153, CB28 CB118 HG CD NI CU PB ZN MN , ZN ACNE , ACNLE, ANT , BAA , BAP , BBF , BEP , BGHIP, BIPN
	310 311 312 312 312 341	NIVA NIVA NIVA NIVA NIVA	86-92 86-92 86-92 92 92	ACNE , ACNLE, ANT , BAA , BAP , BBF , BEP , BGHIP, BIPN BJKF , CHR , COR , DBA3A , DBP , FLE , FLU , ICDP , NAP NAP1M, NAP2M, NAPDI, NAPTM, PA , PAM1 , PER , PYR HG CU , ZN CD , PB CR , NI CB101, CB105, CB118, CB138, CB153, CB156, CB180, CB209, CB28
Shrimp tail	605 605 111 111 111 111	SIIF SIIF SIIF SIIF SIIF SIIF SIIF	86-91 89 82,84,86,88,90 84,86,88,90 86,90 88,90 90	CB107, CB107, CB107, CB107, CB107, CB108, CB109, CB209, CB28 CB52, DDEPP, HCB , HCHA , HCHG , OCS , QCB , TDEPP EPOCL EOCL PCB DDTEP, HCB HCHG CB101, CB138, CB153, CB180, CB28 , CB52 CB118
	120 130 130 132 309	SIIF SIIF SIIF SIIF NIVA	82,84 82,84 84 84 92	HG CD CU , PB MN , ZN ACNE , ACNLE, ANT , BAA , BAP , RBF , RFP , BCNLP, BLDN
	310 311 312 341	NIVA NIVA NIVA NIVA	86,88,90,92 86,88,90,92 86,88,90,92 92	MAPIM, NAPZM, NAPDI, NAPTM, PA , PAM1 , PER , PYR HG CU , ZN CD , PB CB101, CB105, CB118, CB138, CB153, CB156, CB180, CB209, CB28
<sup>~</sup> Other	605 111 310 311 312 605	SIIF SIIF NIVA NIVA NIVA SIIF	86,88,90 88 88 88 88 88	EDOCL CB101, CB138, CB153, CB180, CB28, CB52, DDTEP, HCB, PCBHGCU, ZNCD, PBEPOCL

# 2.3.3. Contaminant, year-laboratory and tissue

ANICHIYETCAL OVERVIEW SOFTED by CONTAMINANT, Year&Lab, Intercalibration+Basis and ordered by TISSUE.

Tissue			1	Fi	sh liver			Fish fi	llet, Shr	imptail,	Mussel,	Other
Contam.	Year& Lab.	Inter- calibr. +Basis	Analys Method Code	Detect Limit (ppb)	Total Value Count	Count Below D.Lim	N (<) Above D.Lim	Analys Method Code	Detect Limit (ppb)	Total Value Count	Count Below D.Lim	N (<) Above D.Lim
ACNE	92-NIV		309	0.10	5		2	309	0.10	43 3		15
ACNLE	93-NIV/ 92-NIV/	A W	309 309	0.10 0.10	5		2	309 309	0.10 0.10	3 43		3
ANT	93-NIV/ 92-NIV/		309 309	0.10 0.10	5			309 309	0.10 0.10	3 42		3 20
BAA	93-NIV/ 92-NIV/	A W	309 309	0.10 0.10	3 5		5	309 309	0.10 0.10	3 41		3
BAP	93-NIV/ 92-NIV/ 93-NIV/	A W	309 309	0.10 0.10	3 5		5	309 309	0.10 0.10	3		3
BBF	92-NIV/	A W	309 309	0.10 0.10	3		3	309 309	0.10 0.10	3		3
BEP	93-NIV/ 92-NIV/	A W A W	309 309	0.10 0.10	3			309 309	0.10 0.10	3		3
BGHIP	93-NIV/ 92-NIV/	A W	309 309	0.10 0.10	กทรดนทรทรทรทรทธทธกรก		. 1	309 309	0.10 0.10	43 42 41 42 42 43 43 43 43 43 43 42 31		193031139363537363123
BIPN	93-NIV/ 92-NIV/	A W	309 309	0.10 0.10	3		2	309 309	0.10 0.10	3		3
BJKF	93-NIV/ 92-NIV/	A Ü	309 309	0.10	3		3	309 309	0.10 0.10	3		3
CB101	93-NIV/ 87-SIII	À W	309	0.10 0.10	ž		3	309	0.10 0.20	3		3
	88-SIII 88-SIII	F D						111	0.10	6	1	
	89-NACE 89-SIII	E W	510	20.00	93		•	111	0.10	22		
	90-NIV/ 90-SIII	1 2G W	340	1.00	169	1		111 341	0.10 0.05	36 58 41		
	91-NIV/ 91-SIII	A 2H W	340	1.00	179		8	111 341	0.40 0.05	62	6	
	92-NIV/ 93-NIV/	A 2HW	340 340	5.00	173	3		111 341	0.20 0.10	35 137		1
CB105	91-NIV/ 92-NIV/	A 2H W	340	5.00 1.00	19 87	_	1	341 341	0.10 0.05	3 47		
CB118	93-NIV/	4 W	340 340	5.00 5.00	173 19	3		341 341	0.10 0.10	137 3		
CBIIO	89-NACE 89-SIII	F W	510	20.00				111	0.10	36 58		
	90-NIV/ 90-SIII	F 2G W	340	1.00	169			341 111	0.05 0.20	58 41	1	
	91-NIV	F 2H W	340	1.00	179			341 111	0.05 0.20	62		1
	92-NIV/ 93-NIV/	1 2H W	340 340	5.00 5.00	173 19	2		341 341	0.10 0.10	35 137 3		
CB138	88-SIII 88-SIII	F W						111	0.10 0.10	6 21		
	89-NACE 89-SIII	· W	510	20.00	93			111	0.10	36		
	90-NIVA	2 G W	340	1.00	169			341	0.05 0.30	58 41		
	91-NIVA 91-SIII	2H W 2H W	340	1.00	179			341	0.05 0.30	62		1
	92-NIVA	1 2H W	340 340	5.00 5.00	173 19			341	0.10 0.10	35 134		'
CB153	88-SIIF 88-SIIF	- D		2.00	.,			341 111 111	0.10	3 6		
	89-NACE 89-SIIF	W	510	20.00	93			1	0.10	22		
	90-NIVA	\ 2G W	340	1.00	169			111 341	0.10 0.05	36 58		
	91-NIVA 91-SIIF	1 2H W	340	1.00	179			111 341	0.30 0.05	41 62		
	92-NIVA 93-NIVA	V 2H W	340 340	5.00	173 19			111 341	0.50 0.10	35 137 3		1
CB156	91-NIVA 92-NIVA	1 2H W	340	5.00 1.00	87	-	15	341 341	0.10 0.05	47		5
CB180	93-NIVA 87-SIIF	\ W	340 340	5.00 5.00	173 19	3		341 341	0.10 0.10	137 3		
CB 100	88-SIIF	: D						111	0.20 0.10	21 6	6	
	88-SIIF 89-NACE	W	510	20.00	93	1		111	0.10	22		
	89-SIIF 90-NIVA	L 2G W	340	1.00	169			111 341	0.10 0.05	36 58		
	90-SIIF 91-NIVA	2H W	340	1.00	179			111 341	0.20 0.05	41 62	8	
	91-SIIF 92-NIVA	2H W	340	5.00	173	3		111 341	0.20 0.10	35		
CB209	93-NIVA 90-NIVA	W V	340 340	5.00 2.00	19 169	24	11	341 341	0.10 0.05	137 3 58		
	91-NIVA 92-NIVA	W V	340 340	2.00 5.00	179 173	11	88	341 341	0.05 0.10	62 137	5	7
CB28	93-NIVA 88-SIIF	. D	340	5.00	19			341 111	0.10 0.10	3 6		·
	88-SIIF	W	510	20.00	93			iii	0.10	22		
	89-SIIF 90-NIVA	2G W	340	1.00	169	2	2	111 341	0.10 0.05	36 58		1
	90-SIIF	2G W				-	-	111	0.20	41	7	l

# Contaminant, year-laboratory and tissue (cont.)

Tissue				Fis	Fish fi	llet, Shr	imptail,	Mussel,	Other			
Contam.	Year& Lab.	Inter- calibr. +Basis	Analys Method Code	Detect Limit (ppb)	Total Value Count	Count Below D.Lim	N (<) Above D.Lim	Analys Method Code	Detect Limit (ppb)	Total Value Count	Count Below D.Lim	Above
	91-NIV	A 2H W F 2H W	340	1.00	179	2	52	341 111	0.05	62	5	1
СВ52	91-SII 92-NIV 93-NIV 87-SII 88-SII 88-SII	A 2HW FW FD	340 340	5.00 5.00	173 19	3		341 341 111 111 111	0.30 0.10 0.10 0.20 0.10 0.10	35 134 3 20 6 22	1	
	89-NAC 89-SII	E ₩	510	20.00	93			111	0.10	36		
	90-NIV 90-SII	F 2G W	340	1.00	169	2	6	341 111	0.05 0.40	58 41	7	
	91-NIV 91-SII	F 2H W	340	1.00	179	1	37	341 111	0.05 0.30	62 35 134 3	7 5	1
CD	92-NIV 93-NIV 81-SII 81-SII 82-SII	A 2H W F 1E W F 1F W	340 340 130	5.00 5.00 10.00	173 19 28	3		341 341 130 130	0.10 0.10 5.00 10.00	7		
	82-VET 83-SII	N W	230	10.00	54			130 130	10.00	18 17		
	83-VET 84-FIE	N 12 W	230 402	10.00 1.00	46 23			130	10.00			
	84-SII 84-VET	N 12 W	230	10.00	66			130	10.00	27		
	85-SII 85-VET	N 12 W	230 312	10.00	45 29		3	130	10.00	35		
	86-NIV 87-FIE 87-NIV	R 1GW	402 312	30.00 1.00 30.00	29 37 59	1		312	30.00	20		
	88-NIV 89-NIV	A 1H D A 1H D	312 312 312	30.00 30.00	61 160	2 20	1 4 8	312 312	30.00 30.00	37 43		
	89-NIV 90-NIV 91-NIV 92-NIV 93-NIV	A 1H W A 1H W A 1H W	312 312 312 312 312	10.00 10.00 10.00 10.00	189 190 172	9 29 4	2	312 312 312 312 312	30.00 30.00 10.00 10.00	36 77 67 111	5	
CHR COR CR CU	92-NIV 93-NIV 92-NIV 93-NIV 92-NIV 83-SII	A W A W A W A W F 1G W	309 309 309 309 309	0.10 0.10 0.10 0.10	19 5 3 5 3			309 309 309 309 312 130	0.10 0.10 0.10 0.10 10.00	41 3 43 3 .6 12		5 3
	84-SII 86-NIV 87-FIE 87-NIV	A 1H D R 1G W A 1H D	311 404 311	150.00 50.00 150.00	29 37 59			130 311 311	10.00 150.00 150.00	12 27 20 37		
	88-NIV 89-NIV	A 1H D	311 311	150.00 150.00	61 160			311	150.00	43		
	89-NIV 90-NIV 91-NIV 92-NIV 93-NIV	A 1H W A 1H W A 1H W	311 311 311 311	150.00 50.00 10.00 10.00	189 193 172	2		311 311 311 311 311	150.00 150.00 50.00 10.00	36 77 67 111		
DBA3A	92-NIV 93-NIV	A W	309 309	0.10 0.10	5		5 3	309 309	0.10 0.10	43 3		35 3
DBP DDEPP	92-NIV 93-NIV 82-VET	A W	309 309	0.10	19 5 3 5 3 53 48			309 309	0.10 0.10	43 3		
DOEPP	83-VET 84-VET 85-VET 86-NAC 87-NAC	N 2E W N 2E W N 2E W E 2Z W	210 210 210 210 210 510 510	0.05 50.00 50.00 50.00 20.00 40.00a	53 48 66 45 31 54			211a	50.00	48		
DDTEP	88-NAC 89-NAC 90-NIV 91-NIV 92-NIV 93-NIV	E 22 W A W A W A W	510 510 510 340 340 340 340	40.00 40.00a 1.00 1.00 5.00 5.00	60 118 169 179 173 19	2		341 341 341 341	0.05 0.05 0.10 0.10	58 62 137		
DUICP	83-SII 84-SII 85-SII 86-SII 87-SII 88-SII 88-SII 89-SII 90-SII	F						111   111   111   111   111   111   111	0.50 0.50 0.50 0.50 0.50 0.50 0.50	12 24 27 21 21 6 22 36 41	1 1 1 1	1 5
DDTPP	91-SII 86-NAC 87-NAC 88-NAC 89-NAC	E W E W	510 510 510	40.00 40.00 40.00 40.00a	31 54 60			111	0.30	35	•	
EOCL EPOCL	89-SII 86-NAC	F W	610	40.00a 800.00	118 31			605	170.00	5		
	86-SII 87-NAC	F W	610	800.00	54			605	5000.00	21	21	
	87-SII 88-NAC	F W	610	800.00	59			605	40.00	20		

# Contaminant, year-laboratory and tissue (cont.)

Tissue	<del></del>				Fis	h liver			Fish fi	llet, Shri	mptail,	Mussel,	Other
Contam.			ter-	Analys	Detect	Total	Count	N (<)	Analys Method	Detect Limit	Total Value	Count Below	N (<)
•	Lab.	+B	libr. Masis	Method Code	Limit (ppb)	Value Count		Above D.Lim	Code	(ppb)	Count	D.Lim	
	88-SII 89-NAC	Œ	W	610	800.00	114	1		605	40.00	27		
	89-SII 90-NIV 90-SII	/A	W	615	40.00	117		3	605	40.00 40.00	35 41		
	91-NI\	/A	W	615	40.00	116		12	605	130.00	35		1
FLE	91-SII 92-NIV	/A	Ä	309	0.10	5		2	309 309	0.10 0.10	42 3		9
FLU	93-NI\ 92-NI\	/A	W	309 309	0.10 0.10	5 3 5 3			309	0.10	41 3		9 3 5 3
нсв	93-NI\ 83-SII 83-VEI	I F	W W 2Z W	309 210	0.10 10.00	3 48			309 111 211a	0.10 0.50 10.00	12 48		,
	84-S11 84-VE	I F	2Z W	210	10.00	66			111	0.20	24		-1
	85-SI 85-VE 86-NA	I F T N	2Z W 2Z W	210 510	10.00 10.00	45 31		4	111	0.20	30	6	2
	86-SI 87-NA	I F	2Z W 2Z W	510	40.00a	54			111	0.20	21	3	
	87-SI 88-NA	I F	22 W 22 W	510	40.00	60			111	0.20	21	4	
	88-SI 88-SI	1 F	2Z D 2Z W	310		50			111	0.20 0.20	6 22	2	
	89-NA 89-SI	CE I F	2Z W 2Z W	510 340	40.00a 1.00	118 169	2		111 341	0.05 0.05	36 58		
	90-NI 90-SI 91-NI	ΙF	22 W	340	1.00	179	4		111 341	0.05 0.05	41 62	3 5	
	91-SI 92-NI	ΙF	2Z W	340	5.00	170	3		111 341	0.10 0.10	35 137		
НСНА	93-NI 90-NI	V۸	ü	340 340	5.00 1.00	19 168			341 341	0.10 0.05	137 3 58		
	91-NI 92-NI	VA	ÿ	340 340	1.00 5.00	179 173	2	111	341 341	0.05 0.10	62 137	5	10
нсна	93-NI 86-NA	VA	Ü	340 510	5.00 30.00	19 31	_		341	0.10	3		
	86-S1 87-NA	I F		510	40.00a		1		111	3.00	21		
	87-SI 88-NA 89-NA	CE	W W	510 510	40.00 40.00a	60 118			111	5.00	21		1
	89-SI 90-NI	IF	ű	340	1.00	169	1	9	111 341	50.00 0.05	36 58		
	90-SI 91-NI	I F VA	W	340	1.00	179	3	18	111 341	0.10 0.05	41 62	5	1
	91-SI 92-NI	VA	W	340	5.00	173 19	3	3	111 341 341	0.30 0.10 0.10	35 137 3		
НG	93-NI 81-SI 82-SI	IF	1E W 1E W	340 120	5.00 10.00	15		1	120 120	10.00	35 18		
	82-VE 83-S1	TN	1E W	220	10.00	51			220 120	10.00 10.00	54 17		
	83-VE 84-F1	TN	12 W 16 W						220 401	10.00 10.00	54 17 48 39 27		
	84-S1 84-VE	IIF	16 W						120 220	10.00 10.00	66	6	1
	85-S1 85-VE	ΙIF	1G D 1Z W						120 220	10.00 10.00	30 90 49		
	86-N) 87-F)	I VA	1H D 1G W						310 401	10.00	49 38		
	87-N1 88-N1	ΙVΑ	1H D 1H D						310 310	10.00	93 104		14
	89-N	AVI	1H D 1H W						310 310	100.00a		5	;
İ	90-N 91-N	AV I	1H W						310 310	10.00	266	126	
1	92-N 93-N	IVA	2H W 2H W						310 310	100.00a 100.00a 100.00	a 283 19	109	
I CDP	92-N 93-N	IVA	Zn W	309 309	0.10 0.10	5 3		3	309 309	0.10 0.10	43 3		13 3
MN	84-S 85-S	IIF	W	337	0.10				132 132	40.00 40.00	27 35		J
NAP	92-N 93-N	IVA	W	309 309	0.10 0.10	5 3		3	309 309	0.10 0.10	27 35 43 3		5
NAP1M	92-N 93-N	IVA IVA	W	309 309	0.10 0.10	5		•	309 309	0.10 0.10	43 3		5 2
NAP2M	92-N 93-N	IVA IVA	W	309 309	0.10 0.10	3 5 3 5 3 5 3 5			309	0.10 0.10			5 2 5 1 7 3
NAPDI	92-N 93-N	IVA	W	309 309	0.10 0.10	3			309	0.10 0.10	43 3		3 11
NAPTM	92-N 93-N	IVA	W	309 309	0.10 0.10	3			309	0.10 0.10 20.00	3		3
NI	83-S 92-N	IVA	W	7/0	3.00	440	, -		130 312 4 341	10.00 0.05	- 6		1
ocs	90-N 91-N 92-N	IVA	W	340 340 340	2.00 2.00 5.00	179	1	1 2 4 8 3	341 1 341 341	0.05 0.10	62	: !	5 8
	93-N			340	5.00	19	,	-	341	0.10	3		

# Contaminant, year-laboratory and tissue (cont.)

Tissue				Fis	sh liver			Fish fi	illet, Shr	imptail,	Mussel,	Other
Contam.	Year& Lab.	Inter- calibr.	Analys Method	Detect Limit	Total Value	Count Below	N (<) Above	Analys Method	Detect Limit	Total Value	Count	N (<)
•		+Basis	Code	(ppb)	Count	D.Lim		Code	(ppb)	Count	Belo⊯ D.Lim	Above D.Lim
PA	92-NIVA 93-NIVA		309	0.10	5		1	309	0.10	42		5
PAH	87-NIVA		309 309	0.10 0.02	3 1		2	309	0.10	3		3
PAM1	92-NIVA	W	309	0.10	5		1	309	0.10	42		7
PB ·	93-NIVA		309	0.10	3		•	309	0.10	3		3
F 5	83-SIIF 84-SIIF							130	20.00	12		
	85-SIIF	1G D						130	20.00 20.00	27 35		2
	86-NIVA		312	150.00	29	4		312	150.00	20		
	87-FIER 87-NIVA		403 312	10.00 150.00	37	1	_					
	88-NIVA		312	150.00	59 61	14	. 8 7	312 312	150.00	37		
	89-NIVA	12 D	312	150.00	160	12	9	""	150.00	43		
	89-NIVA 90-NIVA		312	E0 00	407	-		312	150.00	36		
	91-NIVA		312	50.00 50.00	187 193	3 14	1	312 312	150.00	77	3	
	92-NIVA	12 W	312	50.00	172	108		312	50.00 50.00	67 111	2	
PCB	93-NIVA		312	50.00	19	11			20.00	111	٤.	
rub	81-SIIF 82-SIIF		110	10.00	27		•	110	10.00	35		
	82-VETN		210	50.00	53			111 211	5.00 50.00	17		
	83-SIIF	2E W						111	5.00	54 14		
	83-VETN 83-VETN		210	50.00				211	50.00	48		
	84-SIIF		210	20.00	48			111	E 00			
	84-VETN	ZE W	l					211	5.00 50.00	24 66		
	84-VETN 85-SIIF		210	50.00	66				50100	-		
	85-VETN							111	5.00	32		6
	85-VETN	2Z W	210	50.00	45			211	50.00	90		1
	86-NACE		511a	40.00a	31			511	20.00	31		
	86-SIIF 87-NACE	2E W 2Z W	510	40.00	E/			111	5.00	21		
	87-NIVA	22 W	340	40.00 0.10	54 2			511	20.00	54		
	87-SIIF	2E W						111	5.00	21		
	88-NACE 88-SIIF	2Z W 2E D	510	40.00	60			511	20.00	37		
	88-SIIF	2E W						111	5.00	6		
	89-NACE	2Z W	510	40.00a	118			511	5.00 20.00	22 18	4	
	89-SIIF	2E W						111	5.00	36	6	
	90-SIIF 91-SIIF	2E W						111	5.00	41		
PER	92-NIVA	W	309	0.10	5		5	111 309	5.00 0.10	35 43		71
DVD	93-NIVA	W	309	0.10	3		3	309	0.10	43 3		31 3
PYR	92-NIVA 93-NIVA	W	309 309	0.10	5			309	0.10	41		6
QCB	90-NIVA	ü	340	0.10 2.00	3 169	33	39	309 341	0.10	3		3
	91-NIVA	W	340	2.00	178	13	97	341	0.05 0.05	58 57	5	7
	92-NIVA 93-NIVA	W	340	5.00	173	3		341	0.10	122	-	'
SE	82-VETN	W	340 240	5.00 10.00	19 46			341 240	0.10	3		
TDEPP	91-NIVA	W	340	1.00	138		1	341	10.00 0.05	54 62		
	92-NIVA 93-NIVA	W	340	5.00	172	3	•	341	0.10	137		
ZN	83-SIIF	1G W	340	5.00	19			341	0.10	3		
	84-SIIF	1G W						131 132	400.00 400.00	12 27		
	85-SIIF	16 D						132	400.00	35		
	86-NIVA 87-FIER	1H D 1G W	311 405	3000.00 20.00	29 37			311	3000.00	20		
	87-NIVA	1H D	311	3000.00	37 59			311	3000.00	37		
	AVIN-88	1H D	311	3000.00	61			311	3000.00	37 43		
	89-NIVA 89-NIVA	1H D 1H W	311	3000.00	160		1					
	90-NIVA	1H W	311	3000.00	189		1	311 311	3000.00	36		
	91-NIVA	1H W	311	1000.00	193			311	3000.00 1000.00	77 67		
	92-NIVA	1H W	311	1000.00	172			311	1000.00	111		
	93-NIVA	1H W	311	1000.00	19							
Sum of C	ounts				15765	426	746	***************************************		11766	400	434
(15)				······································							700	734

a(15) > Ambiguous value in cell (Maximum value displayed).

# 2.4. Abbreviations

Abbreviation <sup>1</sup>	English	Norwegian
ELEMENTS		
Al	aluminium	aluminium
As	arsenic	arsenikk
Cd	cadmium	kadmium
Co	cobalt	kobolt
Cr	chromium	krom
Cu	copper	kobber
Fe	iron	jern
Hg	mercury	kvikksølv
Li	lithium	litium
Mn	manganese	mangan
Ni	nickel	nikkel
Pb	lead	bly
Pb210	lead-210	bly-210
Se	selenium	selen
Ti	titanium	titan
Zn	zinc	sink
PAHs		
PAH	polycyclic aromatic hydrocarbons	polysykliske aromatiske hydrokarboner
ACNE	acenaphthene	acenaften
ACNLE	acenaphthylene	acenaftylen
ANT	anthracene	antracen
BAA	benzo(a)anthracene	benzo(a)antracen
BAP	benzo(a)pyrene	benzo(a)pyren
BBF	benzo(b)fluoranthene	benzo(b)fluoranten
BBKF	benzo(b+k)fluoranthene	benzo(b+k)fluoranten
BEP	benzo(e)pyrene	benzo(e)pyren
BGHIP	benzo(ghi)perylene	benzo(ghi)perylen
BIPN	biphenyl	bifenyl
BJKF	benzo(j,k)fluoranthrene	benzo(j,k)fluorantren
CHR	chrysene	chrysen
COR	coronen	coronen
DBAHA	(see DBA3A)	(se DBA3A)
DBA3A	dibenzo(a,c/a,h)anthracene	dibenzo(a,c/a,h)anthracen
DBP	dibenzopyrener	dibenzopyren
DBT	dibenzothiophene	dibenzothiofen
DBTC1	C <sub>1</sub> -dibenzothiophenes	$C_1$ -dibenzotiofen
DBTC2	C <sub>2</sub> -dibenzothiophenes	$C_2$ -dibenzotiofen
DBTC3	C <sub>3</sub> -dibenzothiophenes	$C_3$ -dibenzotiofen
FLE	fluorene	fluoren
FLU	fluoranthene	fluoranten

Abbreviation <sup>1</sup>	English	Norwegian
PAHs (cont.)		
ICDP	indeno(1,2,3-cd)pyrene	indeno(1,2,3-cd)pyren
NAMTM	2,3,5-trimethylnaphthalene	2,3,5-trimetylnaftalen
NAP	naphthalene	naftalen
NAP1M	1-methylnaphthalene	1-metylnaftalen
NAP2M	2-methylnaphthalene	2-metylnaftalen
NAPC1	C <sub>1</sub> -naphthalenes	C <sub>1</sub> -naftalen
NAPC2	C <sub>2</sub> -naphthalenes	C <sub>2</sub> -naftalen
NAPC3	C <sub>3</sub> -naphthalenes	C₃naftalen
NAPDI	2,6-dimethylnaphthalene	2,6-dimetylnaftalen
PA	phenanthrene	fenantren
PAC1	C <sub>1</sub> -phenanthrenes	C <sub>1</sub> -fenantren
PAC2	C <sub>2</sub> -phenanthrenes	C <sub>2</sub> -fenantren
PAM1	1-methylphenanthrene	1-metylfenantren
PER	perylene	perylen
PYR	pyrene	pyren
DI-Σn	sum of "n" dicyclic "PAH"s	sum "n" disykliske "PAH"
P-Σn	sum "n" PAH	sum "n" PAH
PK-Σn	sum carcinogen PAH's	sum kreftfremkallende PAH
ΡΑΗΣΣ	DI- $\Sigma$ n + P- $\Sigma$ n etc.	DI- $\Sigma$ $n + P-\Sigma$ $n$ $mm$
SPAH	$= PAH\Sigma\Sigma$	$= PAH\Sigma \Sigma$
PCBs		
PCB	polychlorinated biphenyls	polyklorerte bifenyler
СВ	individual chlorobiphenyls (CB)	enkelte klorobifenyl
<b>CB28</b>	CB28 (IUPAC)	CB28 (IUPAC)
CB31	CB31 (IUPAC)	CB31 (IUPAC)
<b>CB44</b>	CB44 (IUPAC)	CB44 (IUPAC)
CB52	CB52 (IUPAC)	CB52 (IUPAC)
CB95	CB95 (IUPAC)	CB95 (IUPAC)
CB101	CB101 (IUPAC)	CB101 (IUPAC)
CB105	CB105 (IUPAC)	CB105 (IUPAC)
CB110	CB110 (IUPAC)	CB110 (IUPAC)
CB118	CB118 (IUPAC)	CB118 (IUPAC)
CB128	CB128 (IUPAC)	CB128 (IUPAC)
CB138	CB138 (IUPAC)	CB138 (IUPAC)
CB149	CB149 (IUPAC)	CB149 (IUPAC)
CB153	CB153 (IUPAC)	CB153 (IUPAC)
CB156	CB156 (IUPAC)	CB156 (IUPAC)

Abbreviation <sup>1</sup>	English	Norwegian
PCBs (cont.)		
CB170	CB170 (IUPAC)	CB170 (IUPAC)
CB180	CB180 (IUPAC)	CB180 (IUPAC)
CB194	CB194 (IUPAC)	CB194 (IUPAC)
CB209	CB209 (IUPAC)	CB209 (IUPAC)
<b>CB-Σ7</b>	CB: 28+52+101+118+138+153+180	CB: 28+52+101+118+138+153+180
CB-Σn	sum of CBs, $n = number$ of compounds	$sum\ CBer,\ n = antall\ forbindelser$
ALD	aldrin	aldrin
DIELD	dieldrin	dieldrin
ENDA	endrin	endrin
CCDAN	cis-chlordane	cis-chlordane
ACDAN	α-chlordane	α -chlordan
GCDAN	γ-chlordane	γ -chlordan
OCDAN	oxy-chlordane	oxy-chlordane
TNONC	trans-nonachlor	trans-nonaklor
TCDAN	trans-chlordane	trans-chlordane
ocs	octachlorostyrene	octaklorstyren
QCB	pentachlorobenzene	pentaklorbenzen
DDD	dichlorodiphenyldichloroethane	diklordifenyldikloretan
	· 1,1-dichloro-2,2-bis-	1,1-dikloro-2,2-bis-(4-klorofenyl)etan
	(4-chlorophenyl)ethane	
DDE	dichlorodiphenylethylene	diklordifenyletylen
	(principle metabolite of DDT)	(hovedmetabolitt av DDT)
	1,1-dichloro-2,2-bis-	1,1-dikloro-2,2-bis-
	(4-chlorophenyl)ethylene*	(4-klorofenyl)etylen
DDT	dichlorodiphenyltrichloroethane	diklordifenyltrikloretan
	1,1,1-trichloro-2,2-bis-	1,1,1-trikloro-2,2-bis-(4-klorofenyl)etan
	(4-chlorophenyl)ethane	
DDEOP	o,p'-DDE	o,p'-DDE
DDEPP	p,p'-DDE	p,p'-DDE
DDTOP	o,p'-DDT	o,p'-DDT
DDTPP	p,p'-DDT	p,p'- $DDT$
TDEOP	o,p'-DDD	o,p'-DDD
TDEPP	p,p'-DDD	p,p'- $DDD$
DDTEP	p,p'-DDE + p,p'-DDT	p,p'-DDE + p,p'-DDT
DD-nΣ	sum of DDT and metabolites,	sum DDT og metaboliter,
	n = number of compounds	n = antall forbindelser

Abbreviation <sup>1</sup>	English	Norwegian
НСВ	hexachlorobenzene	heksaklorbenzen
HCHA HCHB HC-nΣ	lindane $ \gamma \ HCH = gamma \ hexachlorocyclohexane $ ( $\gamma \ BHC = gamma \ benzenehexachloride, $ outdated synonym) $ \alpha \ HCH = alpha \ HCH $ $ \beta \ HCH = beta \ HCH $ sum of HCHs, $ n = count $	lindan $\gamma$ HCH = gamma heksaklorsykloheksan  ( $\gamma$ BHC = gamma benzenheksaklorid, foreldret navn) $\alpha$ HCH = alpha HCH $\beta$ HCH = beta HCH  sum av HCHs, $n$ = antall
EOCI EPOCI	extractable organically bound chlorine extractable persistant organically bound chlorine	ekstraherbart organisk bundet klor ekstraherbart persistent organisk bundet klor
NTOT CORG	total organic nitrogen organic carbon	total organisk nitrogen organisk karbon
GSAMT MOCON	grain size moisture content	kornfordeling vanninnhold

Abbreviation <sup>1</sup>	English	Norwegian
INSTITUTES		
FIER	Institute for Nutrition, Fisheries Directorate	Fiskeridirektoratets Ernæringsinstitutt
FORC	FORCE Institutes, Div. for Isotope Technique and Analysis [DK]	FORCE Institutterne, Div. for Isotopteknik og Analyse [DK]
IMRN	Institute of Marine Research (IMR)	Havforskningsinstituttet
NACE	Nordic Analytical Center	Nordisk Analyse Center
NIVA	Norwegian Institute for Water Research	Norsk institutt for vannforskning
SERI	Swedish Environmental Research Institute	Institutionen för vatten- och luftvårdsforskning
VETN	Norwegian Veterinary Institute	Veterinærinstituttet
SIIF	Fondation for Scientific and Industrial Research at the Norwegian Institute of Technology - SINTEF-SI (previously: Senter for Industrial Research SI)	Stiftelsen for industriell og teknisk forskning ved Norges tekniske høgskole- SINTEF-SI (tidligere: Senter for industriforskning

<sup>&</sup>lt;sup>1</sup>) After: ICES Environmental Data Reporting Formats. International Council for the Exploration of the Sea. January 1992.

<sup>\*)</sup> The Pesticide Index, second edition. The Royal Society of Chemistry, 1991.

# 2.5. Intercalibration excercises

Sea water:	
4H -	ICES/JMG Fifth Round Intercalibration on Trace Metals in Sea Water - Section 4, analysis for Hg - 1983 - (5/TM/SW:4).
4I -	JMG Sixth Intercalibration on Trace Metals in EstuarineWaters - 1986 - (6/TM/SW).
4Z -	Intercalibration exercise for SIIF/SERI (Cd) and NIVA/IAMK (IAMK=Chalmers Inst., Göteborg) - 1985.
Seabed sedimen	ıt:
7E	ICES, First Intercalibration Exercise on Trace metals in Marine Sediments - 1984 - (1/TM/MS).
8B	ICES/OSPARCOM, First Intercomparison Exercise on Organochlorines (individual chlorobiphenyl congeners) in Marine Sediments - Phase 1, analysis of standard solutions - 1989 - (1/OC/MS:1).
8C	ICES/OSPARCOM, First Intercomparison Exercise on Organochlorines (individual chlorobiphenyl congeners) in Marine Sediments - Phase 2, analysis of standard solutions - 1991 - (1/OC/MS:2).
Marine biota:	
1E	ICES, Fifth Intercalibration Exercise on Trace Metals in Biological Tissues - 1978 - (5/TM/BT).
1F	ICES, Sixth Intercalibration Exercise on Trace Metals (Cadmium and Lead only) in Biological Tissues - 1979 - (6/TM/BT).
1G	ICES, Seventh Intercalibration Exercise on Trace Metals in Biological Tissues - Part A - 1983 - (7/TM/BT).
1H	ICES, Seventh Intercalibration Exercise on Trace Metals in Biological Tissues - Part B - 1985 - (7/TM/BT) (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).

#### Method overview

2G	ICES/IOC Seventh Intercomparison Excercise on Organochlorines (individual chlorobiphenyl congeners) in Biological Tissue - Phase 1, analysis of standard solutions - 1989 - (7/OC7BT:1).
2Н	ICES/IOC Seventh Intercomparison Excercise on Organochlorines (individual chlorobiphenyl congeners) in Biological Tissue - Phase 2 - 1991 - (7/OC7BT:2).
2Z VETN	Interlabcalibration exercise with VETN among others, 1983, PCB and HCB in cod liver.
2Z NACE	Interlabcalibration exercise with NACE, VETNand SIIF 1986 (PCB (all labs), DDE, OCS, HCB and DCB (NACE and VETN).

# 2.6. Comment on detection limit

The detections limits given here are approximations based on 3 times the standard deviation of the 'blank' or near zero concentration of a solution. However, day-to-day variations in the analytical instrument may lead to different detection limits.

The following descriptions focus on the principles involved and hence are not intended as detailed specifications. The descriptions may vary arbitrarily in detail and may be coupled to specific time periods (cf., Section 2). Hence, they may not necessarily reflect methods currently practiced by the contributing institutes.

# 3.1. Analyses of sea water

#### 3.1.1. 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
	<i>5</i>

#### 3.1.2. Inorganic determinations

#### code description

#### 320 Mercury in sea water (SIIF)

<u>Reference</u>: 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  $SiO_2$  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  $800^{\circ}$ 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 isoptimized 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.)

<u>Reference</u>: Cossa, D, Courau, P., 1984. ICES Fifth round intercalibration for trace metals in seawater (Intercalibrations 5/TM/SW). Report of Section 4 - Round Robbin Intercalibration for total Mercury in seawater. International Council for the Exploration of the Seas, May 1984.

#### 330 Cadmium in sea water (SIIF)

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

#### 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 using a graphite furnace atomic absorption electrothermal spectrometry or GFAAS.

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.

SnCl<sub>2</sub> is added to 250ml 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 at least 500<sup>0</sup>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.

Since 1988: a maximum of 100ml sample used, diluted if Hg >50ng/l; P-E 1100 B with gold trap used, helium replaced air as carrier gas and lowest signal was 2.5ng/l.

# 3.2. Analyses of sea bed sediments

# 3.2.1. Sampling methods

~ · · · · · · · · · · · · · · · · · · ·	on the same of the		
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)		
GS	Grab sampler		
	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		
HFO	'Total' digestion with mineral acids including hydrofluoric acid (HF), in open vessels, evaporation of excess HF before analysis.		
HNO	(outdated code = HNO1 3) Extraction with 1:1 $\text{HNO}_3$ (suprapur) for inorganic analyses on "fresh" (i.e., frozen) material.		
EXN	(outdated code = EXN1 2) Extraction of (organic) contaminants by shking with non-polar solvents cyclohexane/ispropanol (1:1 v/v) on freeze dried material.		

#### 3.2.2. 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 sample is 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 7N (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**

Prior to 1988: 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 0,03µg mercury.

Since 1988: a maximum of 100ml sample used, diluted if Hg >50ng/l; P-E 1100 B with gold trap used, helium replaced air as carrier gas and lowest signal was 2.5ng/l.

#### 351 Chromium, copper, iron, manganese, nikkel and zinc in sea bed\_sediment (NIVA)

Same procedure as 350: #1-2, Drying and nitric acid Extraction.

Prior to 1992 (1990-91 JMP samples) 'total' extraction (HFO): Approximately 0.1g of the sample is accurately weighed in, 2ml of hydroflouric acid and 2+2ml of concnetrated nitric acid ('aqua regia') is added and the solution heated in a microwave oven. The solution is transferred to a 100ml volumetric clask and diluted to the marked with deionized water.

Since 1992 'total' extraction (HFO): 0.2g of freeze dried homogenated sample is digested in teflon vessels with 1ml 'aqua regia' plus 6ml hydroflouric acid neutralized with boric acid amd diluted to 100ml. (cf., Loring D.H., Rantala, R.T.T., 1992. ICES manual for the geochmeical analyses of marine sediment and suspended particulate matter).

Determinations by flame atomic absorption spectrometry using acetylene/air flame. Instrument: *Prior to 1986* a Perkin Elmer model 2380 was used and *since 1986* the P-E 560 has been used. For determinations of low concentrations (below detection limits) the flameless method (352) is used. The following are elements often analyzed by flame and

Element		μg/l
Al	aluminium	1000
Cr	chromium	50
Cu	copper	100
Fe	iron	200
Li	lithium	10
Mn	manganese	50
Ni	nickel	100
Zn	zinc	10

# Aluminium, cadmium, chromium, cobalt, copper, iron, lead, lithium, manganese, nickel, and zinc in seabed sediment (NIVA)

Same procedure as 350: #1-2, Drying and Extraction.

their respective detection limits of extract solution:

<u>"Total" extraction</u> (HFO): Approximately 0.1g of the sample is accurately weighed in, 2ml of hydroflouric acid and 2+2ml of concnetrated nitric acid (suprapur) is added and the solution heated in a microwave oven. The solution is transferred to a 100ml volumetric clask and diluted to the marked with deionized water.

Concentrations are determined by **graphite furnace atomic absorption** electrothermal spectrometry or GFAAS using a hollow cathode lamp (HCL) or an electrodeless discharge lamp (EDL) as a light source. *Prior to 1986* a Perkin-Elmer model 560 with HGA-500 graphite furnace was used and *since 1986* the P-E 2380 has been used instead of the P-E 560.

A  $20\mu l$  portion of extract, treated with HNO<sub>3</sub>, is injected into graphite tube. The sample is then heated electrothermally in a stepwise manner through drying, ashing and atomization by a programme designed for each element. The programe which controls the ramp time, holding time and temperature for each phase is often adjusted to achieve optimal results.

The elements analyzed and approximated limit of detection for the extract are:

Element		μg/l
Al	aluminium	5
Cd	cadmium	0.1
Co	cobalt	5
Cr	chromium	0.5
Cu	copper	0.5
Fe	iron	5
Li	lithium	10
Mn	manganese	0.5
Ni	nickel	5
Pb	lead	0.5
Zn	zinc	10

#### 353 Cadmium, chromium, nickel and lead in seabed sediment (NIVA)

As 352 but since 1992 the L'vov platform technique is used for these metals.

#### 650 Pb-210 dating (FORC)

<u>reference</u>: Pheiffer Madsen, P., Sørensen, J., 1979. Validation of the Lead-210 dating method. Journal of Radioanalysis and Chemistry 54:39-48.

Excerpt (Larsen, B., & Jensen, A., 1989. Marine Pollution Bulletin 20(11):556-560.): "The determination of time- dependent sediment parameters is based on the vertical distribution of the natural radioactive isotope lead-210 [= <sup>210</sup> Pb] ... The content of unsupported lead-210, that lead-210 not produced in the sediment) decreases regularly downwards in undisturbed and steadily deposited sediment owing to radioactive decay. Departures from this predictable lead-210 profile in the topmost sediment column permit an assessment of mixing and/or intermittent erosion."

Dried slices of sediment are employed.

#### 3.2.3. Organic determinations

#### code description

#### 360 PCB in sea bed sediment (NIVA)

Prior to 1990: the method is quite similar to SIIF method JMG code 110.

#### Cleaning of chemicals and equipment

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

The equipment is washed with soap and water, then rinsed in 1:5 mixture of  $HNO_3$  and  $H_2SO_4$ , respectively. Finally, the equipment is rinsed with aceton 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.

#### 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 electron-capture detector (ECD). Splitless injection at  $70^{\circ}$ C and then programmed temperature raise with  $7^{\circ}$ /min to 230°C. Column: 30mx0.259mm (inner diameter),  $0.25\mu$  DB-5 fused silica capillary column. Carrier gas:  $H_2$ , 0.8 bar.

#### Identification and quantification

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

Since 1990 the principle is the same but details have been altered as followed:

Sea bed sediment - organic

#### Cleaning of chemicals and equipment

The equipment is kept in soap and water overnight then rinsed first in water, then in destilled water and finally with aceton. The air-dried glass equipment is heated to 550°C.

All solvents are either distilled in a special room for this purpose or if the quality is satisfactory commercial solutions are used. 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 disintegrated/extracted twice with ultrasonic disintegrater with aceton and cyclohexane (1:1).

#### Clean-up of extract

2ml cyclohexane extract is shaken by whirl mixer with 6ml concentrated sulphuric acid and then centrifuged. This process is repeated.

#### Gas chromatographic condition

Gas chromatograph HP 5890 II is used with electron-capture detector (ECD). Splitless injection at 90°C and then programmed temperature raise with 3°/min to 280°C. Column: 60mx0.25mm (inner diameter),  $0.25\mu$  Rtx 05 fused silica capillary column. Carrier gas:  $H_2$ , 36 cm/sec.

#### Identification and quantification

The individual PCB-congeners are indentified by their retention times relative for the internal standard, PCB-53. The PCB's are qunatified by using internal standard and eight-level calibration in the concentration range of the CBs in the solution to be analyzed.

#### 369 PAH in sea bed sediment (NIVA)

#### Extraction

Deuterated internal standards are added to about 0.5-5g of dried sediment and the sample is extrated in Soxhlet with cyclohexane. The extract is clean first by partition with DMF:water, and then by silicagel. Finally, the sample is evaporated to a small volum before GC analysis.

#### Determination by GC

PAHs are determined on GC coupled to mass-selective detector. The compounds are detected by their respective molecular ions and retention time and quantified by using National Institute for Standards and Technology (NIST) Certified Reference Material (CRM) numbers 1491 and 1941. Coronen and Dibenzopyrener are quantified with the help of in house standards.

#### 760 PCB in sea bed sediment (IMRN)

PCB in total sediment (50g) were extracted 3 times by acetone and hexane: Acetone (3:1) using repeated ultrasonication and aggitation (Jensen et al., 1977).

Sulphur was removed with metallic mercury.

A florisil column (100-230 mesh, 30 cm x 6 mm ID) was used for the separation of the extract into 3 fractions. The first fraction eluted with 2 ml pentane was discarded; the second fraction eluted with 6.5 ml pentane contained PCB, HCB, aldrin, o,p-DDE, p,p-DDE and o,p-DDT; and the third fraction eluted with 10ml pentane:acetone (9:1) contained, alpha-HCH, beta-HCH, gamma-HCH (Lindane), o,p-DDD, p,p-DDD, o,p-DDT (20%) and p,p-DDT.

The third fraction needed further clean up on a neutral alumina column (30 cm x 6 mm ID; deactivated with 6% water). The chlorinated pesticides were eluted with 18 ml pentane. Beta-HCH was not eluted using this method.

A few samples (1990 sediment stations 15S-67S) were cleaned up before separation on the florisil column. A short silica column (10 cm x 6 mm ID) was used, followed by a alumina column (10 cm x 6 mm ID, acidic  $Al_2O_3$ ). Pentane:dichloromethane (4:1) was used for elution of the compounds.

The chlorinated compounds were quantified on GC (ECD) using two different columns: SE-54 CB, fused silica, 50 m x 0.20 mm, 0.11  $\mu$ m; SP-2330, fused silica, 60 m x 0.25 mm, 0.20  $\mu$ m.

<u>Reference</u>: Jensen, S., Renberg, L., Reutergårdh, L., 1977. Residue analysis of sediment and sewage sludge for organochlorines in the presence of elemental sulfur. Anal. Chem. 49:316-318.

#### 769 PAH in sea bed sediment (IMRN)

Ca.50 g of total sediment (< 2mm) were extracted three times with acetone and hexane:acetone (3:1) using ultrasonication and aggetation.

The clean-up of the extract was carried out on a short silica column (10 cm x 6 mm ID) using pentane:dichloromethane (9:1) as eluent. GC/MS equipped with a SE-54 fused silica cappilary column (50 m x 0.20 mm ID, 0.11  $\mu$ m film thickness) was used for the analysis of 2-6 ring aromatic hydrocarbons.

#### 3.2.4. Organic carbon determinations

#### code description

# 390 Total organic nitrogen and organic carbon (CORG) in sea bed sediment (NIVA)

5-8mg of freeze dried sample is weighed in a tin-foiled capsule and heated to over  $1800^{\circ}$ 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  $1 \mu g/mg$  and N is  $1 \mu g/mg$ .

# 3.3. Analyses of marine biota

#### 3.3.1. 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. HNO<sub>3</sub> 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 SnCl<sub>2</sub>.

#### 121 Mercury in shellfish (SIIF)

Same procedure as 120 but bombe digestion (pressurized decomposition) with HNO<sub>3</sub> at 160°C is used instead of pretreatment with HNO<sub>3</sub> 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°C to constant weight to determine the total water content.

The vessel is then placed in a cold muffel furnace and the temperature increased slowly to 450°C. The vessel is removed from the furnace and cooled down to room temperature. After wetting the ash with 1ml conc. HNO<sub>3</sub> and approximately 2ml H<sub>3</sub>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 methylisobutylketon) with flame atomic absorption spectrometry.

#### **Zinc in biota (SIIF)**

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

#### **Zinc and manganese in biota (SIIF)**

Same procedure as 131 but quantified by ICP.

#### 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).

<u>Reference</u>: 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  $\mu$ g/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 0.71 $\mu$ g 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)

<u>Reference</u>: Norheim, G. and Nymoen, U.K., 1981. Fluorimetric determination of sclenium 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°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.

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DAN is added and the solution is heated at 60°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)

Homogenizing of large samples (eg., fish fillet) by Tedal Quick Foodmaster Holberth silent cutter commercial use. Stainless steel blades are used. For smaller samples (eg., liver) a Silverson 4R Homogenizer is used.

#### **Drying procedure**

An accurately weighed sample of approximately 1g is freezedried until constant weight. If the sample has excessive fat content (e.g., fish liver, and therefore, can not be freezedried) the sample is dried at 105°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.

Since 1991 (1990 JMP samples) extracts have been made from wet (fresh) samples.

#### Extraction (oxidation)

Prior to 1991: 50-200mg freezedried sample is weighed in teflon vessels, 2ml concentrated nitric acid (suprapur) is added and capped loosely. The solution is heated for about 2hrs. at  $50^{\circ}$ C in a thermostatically controlled aluminium block until foaming ceases. The temperature is raised to  $110^{\circ}$ 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  $110^{\circ}$ 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.

Since 1991: extracts are made from 0.2-0.5g dried or 2-2.5g wet sample. When teflon vessels are used 10ml concentrated HNO<sub>3</sub> is added and digested for 30 min in autoclave and then diluted to 100ml. If there is excessive fat in sample 2 ml 30% hyrdogen pyroxide is added.

#### **Determination**

Prior to 1988: 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. Tin chloride is added as a reducing agent.

Since 1988: a maximum of 30ml sample is used, up to concentrations  $1.5\mu g/l$ , and diluted if Hg in the solution is more than  $1.5\mu g/l$ . A P-E 1100 B with gold trap used with helium as carrier gas.

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

(Same homogenizing, drying and extraction procedure as 310.)

Determinations by flame atomic absorption spectrometry using acetylene/air flame. Instrument: *Prior to 1986* a Perkin Elmer model 2380 was used and *since 1986* the P-E 560 has been used. For determinations of low concentrations (below detection limits) the flameless method (352) is used. The following are elements often analyzed by flame and their respective detection limits of extract solution:

Element		μg/l	
Cu	copper	50	
Fe	iron	200	
Zn	zinc	10	

# Cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, and zinc in biota (NIVA)

(Same homogenizing, drying and extraction procedure as 310.)

#### **Determination**

Concentrations are determined by graphite furnace atomic absorption electrothermal spectrometry or GFAAS using a hollow cathode lamp (HCL) or an electrodeless discharge lamp (EDL) as a light source. *Prior to 1986* a Perkin-Elmer model 560 with HGA-500 graphite furnace was used and since 1986 the P-E 2380 has been used instead of the P-E 560.

A  $20\mu l$  portion of extract, treated with HNO<sub>3</sub>, is injected into graphite tube. The sample is then heated electrothermally in a stepwise manner through drying, ashing and atomization by a programme designed for each element. The programe which controls the ramp time, holding time and temperature for each phase is often adjusted to achieve optimal results.

Since 1992 the L'vov platform techninques has been used for determination of cadmium and lead

The elements analyzed and approximated limit of detection for the extract are:

Element		μg/l
Al	aluminium	5
Cd	cadmium	0.1
Co	cobalt	5
Cr	chromium	0.5
Cu	copper	0.5
Fe	iron	5
Mn	manganese	0.5
Ni	nickel	5
Pb	lead	0.5
Zn	zinc	10

#### 401 Mercury in biota (FIER)

<u>Reference</u>: 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).

<u>Reference</u>: Julshamn, K., Ringdal, O., & Braekkan, O.R., 1982. Mercury concentration in liver and muscle of cod (<u>Gadus morhua</u>) 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.  $HNO_3/H_2SO_4$  (1+1) +  $V_2O_5$  (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<sub>4</sub>.

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<sub>2</sub> to avoid interference with iodine.

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

#### 402 Cadmium in biota (FIER)

<u>Reference</u>: 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).

<u>Reference</u>: 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 <u>Mytilus edulis</u>. 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  $95^{\circ}$ C for 24hr. O.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.

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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 atomic absorption electrothermal spectrometry or GFAAS, hollow cathode lamp (HCL), 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  $(NH_4)_2HPO_4$ .

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 atomic absorption electrothermal spectrometry or GFAAS, 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  $(NH_4)_2HPO_4$ .

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 atomic absorption electrothermal spectrometry or GFAAS, 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.

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#### **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 atomic absorption electrothermal spectrometry or GFAAS, 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.

#### 3.3.2. Organic determinations

#### code description

# PCB HCB DDTEP (p,p'DDE + p,p'DDT) HCHG (gHCH = gBHC) in fish and shellfish (SIIF)

#### Cleaning of chemicals and equipment

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°C.

Alternatively, the extract is evaporated to constant weight in a flask with Vigreux column in oil bath at 1 atm.,  $110^{\circ}$ C and reflux or in vacuum,  $50^{\circ}$ C, reflux and weak  $N_2$ -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  $\pm 10\%$ . Determinations are given to 0.1% fat. 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  $60^{\circ}$ C and then programmed temperature raise with  $8^{\circ}$ /min to  $230^{\circ}$ C. Column: 50mx0.3mm (inner diameter),  $0.15\mu$  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 A60 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:

SIIF code	CB code	Structure (-bifenyl)	name
3	95	2,3,6,2',5'	Pentachlorine
4	101	2,4,5,2',5'	Pentachlorine
9/	149/	2,4,5,2',3',6'/	Hexachlorine
10	118	2,4,5,3',4,	Pentachlorine
14	138	2,3,4,2',4',5'	Hexachlorine
15	128	2,3,4,2',3',4'	Hexachlorine
16	180	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 (gHCH=gBHC) 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)

# PCB HCB DDTEP (p,p'DDE + p,p'DDT) HCHG (gHCH = gBHC) in fish and shellfish (SIIF)

Same procedure as 110, except that the organochlorine standard was <u>Arochlor 1254</u> 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.

Since 1991 CB204 has been used as an internal standard.

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

<u>References</u>: Bjerk, J.E., & Sundby, R., 1970. Residues of organochlorine insecticides and polychlorinated biphenys in terrestrial and aquatic test-organisms. Norwegian Part of OECD program 1967-68. This was modified by Norheim, G., 1978. The composition and distribution of PCB in arctic fox (<u>Alopex lagopus</u>) caught near Longyearbyen on Svalbard. <u>Arta pharmacol. et toxicol.</u> 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.

<u>Quantification</u>: Phenoclor DP6 is used as standard. The peak height of 2,4,5-2',4',5' hexachlorbiphenyl is used to quantify PCB.

#### 211 PCB in fish filet samples (VETN)

<u>Reference</u>: Norheim, G., Økland, E.M., 1980. Rapid extraction of some persistent chlorinated hydrocarbons from biological material with low fat content. <u>Analyst</u> (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 x128.

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.

<u>Standard solutions</u>: 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°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 5µl is used.

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

#### 309 PAH in biota (NIVA)

#### Extraction

Deuterated internal standards are added to about 20-30g of homogenized wet sample and the sample is then saponified with KOH/methanol. After filtering through a glass filter the solution is extracted with n-pentan. The extract is clean first by partition with DMF:water, and then by silicagel. An aliquot if the homogenized sample is used for dry weight determination.

#### **Determination by GC**

PAHs are determined on GC coupled to mass-selective detector. The compounds are detected by their respective molecular ions and retention time and quantified by using National Institute for Standards and Technology (NIST) Certified Reference Material (CRM) number 1491 and number 1974 for blue mussel samples. Coronen and Dibenzopyrener are quantified with the help of in house standards.

# PCB, QCB, OCS, HCB, DDTEP (p,p'DDE + p,p'DDT), HCHB, (b HCH), HCHG (g HCH = g BHC) in fish liver (NIVA)

Prior to 1991 (1987 JMP NIVA samples): Equivalent to SIIF method 111 with the following exception:

#### Determination of fat

The extract is evaporated to constant weight in a flask with Vigreux column in oil bath at 1 atm.,  $110^{\circ}$ C and reflux or in vacuum,  $50^{\circ}$ C, reflux and weak  $N_2$ -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.

Since 1991 (1990 JMP samples):

#### Cleaning of chemicals and equipment

The equipment is kept in soap (3% RBS/Deconex) and water overnight then rinsed first in water, then in destilled water and finally with aceton. The air-dried glass equipment is heated to 550°C.

All solvents are distilled in a special room for this purpose or if the quality is satisfactory commercial solutions are used. Sodium sulphate is washed twice with distilled cyclohexane and heated to  $550^{\circ}$ C.

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#### Extraction

About 5g (dependant on species/tissue) of wet homogenized material is extracted twice by ultra sonic desintegration with a mixture of cyclohexane/aceton (1:1 v/v). Since June 1991 PCB 53 has been added as internal standard and sodium sulphate step has been omitted.

#### Determination of fat

A part of the cleaned and dried cyclohexane extract is evaporated in an oven to constant weight at 105°C.

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

#### Clean-up of extract

About 0.1g fat is dissolved in 2ml cyclohexane, shaken with 6ml concentrated sulphuric acid and then centrifuged. For further clean-up about 1ml of the sulphuric acid treated extract may be treated with a solution of KOH:

#### Gas chromatographic condition

Since 1992 the following has been used: HP 5890 series II with ECD; spitless injection at  $90^{\circ}$ C and then programmed temperature raise with  $3^{\circ}$ /min. to  $280^{\circ}$ C; column: 60mx0.25mm (inner diameter),  $0.25\mu$  Rtx 05 silica capillary column; carrier gas: H at 37 cm/s.

#### Identification and quantification

HCB, HCHG (γHCH=γBHC) and DDTEP (p,p'DDE + p,p'DDT) and the individual PCB-congeners are indentified by their retention times relative for the internal standard, PCB-53. The PCB's are qunatified by using internal standard and eight-level calibration in the concentration range of the CHC's in the solution to be analyzed.

# PCB, QCB, OCS, HCB, DDTEP (p,p'DDE + p,p'DDT), HCHB, (b HCH), HCHG (g HCH = g BHC) in shellfish and fish fillet (NIVA)

Same procedure as 340: except for detection limits:

<u>Detection limits</u>: the minium detectable amount corresponds to  $0.001\mu g/g$  wet weight for liver samples. This gives minimum quantification limits of  $0.002\mu g/g$ .

# PCB, HCB,DDEPP (p,p'DDE), DDTPP (p,p'DDT), HCHG (g HCH = g BHC) in fish liver (NACE)

<u>Pretreatment and fat determinations</u>: 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-

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sulphate, transferred to a glass column with sintered glass fritt 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°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°C and the detector at 300°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.

<u>Detection limits</u>: the minimum detectable amount corresponds to  $0.01\mu g/g$  wet weight for liver samples. This gives minimum quantification limits of  $0.04\mu g/g$  for PCB.

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#### 511 PCB in fish filet (NACE)

<u>Pretreatment</u>: Homogenised filet samples are treated with concentrated sulphuric acid for 4hr at 60°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).

<u>Detection limits</u>: the minium detectable amount corresponds to  $0.005\mu g/g$  wet weight for liver samples. This gives minimum quantification limits of  $0.02\mu g/g$ .

#### 605 EPOCl in shellfish (SIIF)

The cyclohexane extract from chlorinated hydrocarbon analysis is reduced in volume (by evaporation) and treated with concentrated  $H_2SO_4$  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 EPOCl in fish liver (NACE)

Same procedure as 605 but higher detection limit.

The detection limit is 800 ppb wet weight.

#### 615 EPOCl in fish liver (NIVA)

Same procedure as 605.

The detection limit is 40 ppb wet weight.

#### 3.3.3. Fat determinations

#### code description

- A Weight of extracted solids during chlorinated hydrocarbon determinations: procedure 110 (SIIF).
- B Weight of extracted solids during chlorinated hydrocarbon determinations: procedure 510 (NACE and VETN).
- C Weight of extracted solids from freeze dried material using ethyl acetate (FIER).
- D Weight of extracted solids during chlorinated hydrocarbon determinations: procedure 340 (NIVA).

