NIFES
NATIONAL INSTITUTE OF NUTRITION AND SEAFOOD RESEARCH

Report
2014

Monitoring program for pharmaceuticals, illegal substances, and contaminants in farmed fish

ANNUAL REPORT FOR 2013

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05.12.2014

Commissioned by

the Norwegian Food Safety Authority

ACKNOWLEDGEMENTS

Most of the analyses for the monitoring programme were conducted at NIFES. Annette Bjordal, Marita Kristoffersen and Anette Kausland were in charge of the analytical work, while Anne Margrethe Aase was responsible for the work related to sample storage, preparation and distribution within the institute. Manfred Torsvik, Vidar Fauskanger, Nawaraj Gautam, Kari Pettersen and Kjersti Pisani carried out the sample pre-treatment. Tore Tjensvoll and Felicia Dawn Couillard were responsible for chemical analysis of residues of therapeutics. Karstein Heggstad, Jannicke Bakkejord, Dagmar Nordgård, Lene H. Johannessen, Britt Elin Øye, Teclu Habtemariam Weldegebriel, Kari B. Sæle, Kjersti Kolås and Per Ola Rasmussen were responsible for analyses of organic contaminants. Siri Bargård, Tonja Lill Eidsvik, Berit Solli, Vivian Mui, Edel Erdal and Laila Sedal carried out the analysis of the chemical elements. Tone Galluzzi and Leikny Fjeldstad conducted the analyses of the antibacterial agents by the bioassay method.

In 2013, NIFES used sub-contractors for analyses of some parameters: Oslo University Hospital (OUH) for stilbenes and steroids, the Norwegian Veterinary Institute (NVI) for mycotoxins and Eurofins for the analyses of selected therapeutic compounds (see Table 8.9 for details).

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1. SUMMARY

This report summarises the results of the analyses performed on Norwegian farmed fish according to directive 96/23/EC. In 2013, 12 310 farmed fish were collected. From 10 595 fish, 2 119 muscle samples (pooled samples of five fish) were analysed. In addition, 1 715 liver samples (individually samples) were analysed.

As defined in the 96/23 directive, group A substances include substances with anabolic effects and unauthorised substances. Approximately 30% of the samples collected were analysed for group A substances. Group A samples were collected by official inspectors at the farm without prior notification to the farmers. Samples were collected at all stages of farming and are representative of farmed fish under production. Group B substances include veterinary drugs and contaminants. These samples were collected at processing plants, and are representative of Norwegian farmed fish ready for the market.

In 2013, 3 820 fish were analysed for banned substances (Group A); no residues were found in any of the samples.

For the veterinary drugs belonging to group B, emamectin was detected in two of the 126 pooled samples of farmed fish analysed. The highest concentration measured was 32 µg/kg, which is well below the current Maximum Residue Limit (MRL) for emamectin of 100 µg/kg. No other veterinary drug residues were detected in 2013.

For organic contaminants, no samples exceeded the EUs maximum limits, where such limits have been established. For farmed salmon, the concentrations of sum dioxins (sum of polychlorinated dibenzo-para-dioxins (PCDDs) and polychlorinated dibenzofurans PCDFs), dioxin like PCBs (dl-PCBs), indicator PCB (PCB-6) and some pesticides have decreased over the last years.

The concentrations of mercury, cadmium and lead in farmed fish were below the EU maximum limits for these elements in fish. Cadmium and lead were below the limit of quantification (LOQ) in most of the samples. The levels of both mercury and arsenic have decreased over the last years.

Of the brominated flame retardants (BFRs), polybrominated diphenyl ethers (PBDEs), Tetrabromobisphenol A (TBBPA) and Hexabromocyclododecane (HBCD) were analysed in 2013. There are currently no maximum limits for BFR.

2. INTRODUCTION

The aim of this program is to monitor the presence of illegal substances, pharmaceuticals and contaminants in Norwegian farmed fish. The residues or substance groups that are relevant to monitor for aquaculture animals are specified in Directive 96/23/EC:

Group A Substances with anabolic effects and unauthorized substances:

A1: Stilbenes, derivatives and their salts and esters

A3: Steroids

A6: Prohibited substances

Group B Veterinary drugs and contaminants:

B1: Antibacterial agents

B2a: Anthelmintics

B3a: Organochlorine compounds

B3b: Organophosphorus compounds

B3c: Chemical elements

B3d: Mycotoxins

B3e: Dyes

In addition, BFR, PFC and PAH, which belongs to group B3f, others, are included.

1.1 Group A, banned substances

Group A includes growth promoters: steroids and stilbenes and substances listed in Commission Regulation (EU) No 37/2010 under prohibited substances for which MRLs cannot be established. Prohibited compounds considered relevant for aquaculture is chloramphenicol, nitrofurans, and metronidazole. To ensure harmonized levels for the control of banned substances, analytical methods used for banned compounds should meet minimum required performance limits (MRPLs) set by the community reference laboratories (CRLs), national reference Laboratories (NRLs) and member states

of the European Union (2003/181/EC; 2004/25/EC; CRL 2007). Table 8.9 gives an overview of MRPLs of relevant compounds.

Group A substances are analysed in samples that are collected by official inspectors at the farm without prior notification to the farmers. Fish are sampled at all stages of farming and are representative of farmed fish during production.

1.2 Group B, veterinary drugs

In order to protect public health, Maximum Residue Limits (MRLs) have been established. According to current EU legislation (EU 37/2010) each substance is assigned a MRL, which is the highest permitted residual concentration of legally applied pharmacologically active substances in products intended for human consumption. Consumption of food with drug residues below the MRL should, by a wide safety margin, not pose a health risk to the consumer. The MRLs for fish are set for muscle and skin in natural proportions.

Samples examined for veterinary drugs are collected from fish at processing plants and the samples are representative of fish ready to be placed on the market for human consumption.

1.3 Group B, contaminants

Contrary to veterinary drugs, which are given to the fish intentionally, contaminants are unwanted substances that the fish receive primarily from the feed. The main contributor of organic contaminants like dioxin, dl-PCB and PCB-6 is the fish oil used in the feed, while the main contributor of mercury is the fishmeal. Maximum limits for some of the contaminants are set for fish, while for others, like the pesticides and BFR, maximum limits have yet to be established.

As for the veterinary drugs, these samples are collected from fish at processing plants, and are representative of fish ready for human consumption.

3. MATERIAL AND METHODS

3.1 Sampling

Samples are taken on fish farms in all fish-producing regions in Norway. Fish species included in 2013 were Atlantic salmon (*Salmo salar*), rainbow trout (*Oncorhynchus mykiss*), Turbot (*Scophthalmus maximus*), Atlantic halibut (*Hippoglossus hippoglossus*), Atlantic cod (*Gadhus morhua*, Arctic char (*Salvelinus alpinus*) and Wolffish (*Anarhichas lupus*).

The sampling plan was randomised with regards to season and region, and the sample identification was blinded for the analysts. Samples consisted of muscle and in some cases liver tissue, and were transported to NIFES in a frozen state.

3.2 Pre-treatment

On arrival at NIFES, the Norwegian quality cut (NQC) was obtained from the fish (Johnsen 2011). Pooled samples of five fish from the same cage/farm were homogenised before analyses. Samples collected for analyses of group A compounds may include small fish from early life stages. In these cases, head, tail and gut were removed before the rest of the fish were analysed. If the fish was very small, more than 5 fish were required in the pooling. Consequently, the number of fish analysed may be slightly higher than reported. In opposite to muscle samples, the liver tissues were analysed individually. A back-up sample is stored for all samples. For samples to be analysed for veterinary drugs with a MRL, skin is included in the back-up sample. If a veterinary drug is detected in an initially screening, the back-up sample will be analysed.

3.3 Analytical methods

The laboratory routines and most of the analytical methods are accredited in accordance with the standard ISO 17025 (Table 8.9). A summary of the analytical methods and their Limit of detection (LOD) and Limit of quantification (LOQ) are shown in Annex I. The LOD is the lowest level at which the method is able to detect the substance, while the LOQ is the lowest level for a reliable quantitative measurement. For all methods, a quality control sample (QCS) with a known composition and concentration of target analyte, is included in each series. The QCS results are checked to be within pre-defined limits before the results are approved. The methods are regularly verified by participation

in inter laboratory proficiency tests, or by analysing certified reference material (CRM), where such exist.

3.3.1 Group A substances

The group-A samples were analysed for hormone-like substances in the group of stilbenes (A1), steroids (A3), and for illegal drugs (A6).

Group A1 and A3

The stilbenes (A1) diethylstilbestrol, dienesterol, hexosterol and steroid compounds (A3) compounds α-nandrolon, β-nandrolon, α-trenbolon and β-trenbolon, were analysed by GC/ MS. If positive findings should occur they would be verified by confirmatory methods, including an additional clean-up step by HPLC before a new derivatization step followed by a final analytical determination by GC/MS.

Group A6

Chloramphenicol was extracted with ethyl acetate. Liquid-liquid extraction was used to purify the extract. The samples were analysed by LC-MS.

The nitrofuran metabolites were extracted with aqueous hydrochloric acid and derivatized with nitrobenzaldehyde. Solid phase extraction was used for sample clean up. The analytes were determined by LC-MS/MS in the positive ionisation mode.

Metronidazole and its metabolite hydroxymetronidazole were extracted by ethyl acetate. Solid phase extraction was used for sample clean up. The analytes were determined by LC-MS/MS

3.3.2 Group B substances

B1, Antibacterial agents (antibiotics)

The presence of antibacterial agents was determined by chemical analysis or a three plate microbiological assay, or by a combination of both.

For the three-plate microbiological inhibition method, a plate containing growth agar and a specific bacterial strain was added. Small pieces of liver were placed on the plates before incubation. If the samples contained residues of antibacterial agents, the bacterial growth would be inhibited in a zone around each piece of liver tissue. Thus, a transparent zone with no bacterial growth surrounding the liver sample would indicate a positive sample. Any positive detection has to be verified by chemical analysis of the corresponding fillet sample sampled from the same fish.

Oxolinic acid and flumequine:

The analytes were extracted with acetonitrile, and analysis was performed by LC-MS/MS in the positive mode.

Oxytetracycline

The analyte was extracted with an EDTA-succinate aquatic buffer. Solid phase extraction was used for sample clean-up. The analyte was determined by LC-MS/MS.

Florfenicol

The analyte was extracted with ethyl acetate. Liquid-liquid extraction was used to purify the extract. The samples were analysed by LC-MS.

B2a, Anthelmintics

Diflubenzuron and teflubenzuron

The analytes were extracted with acetone. Solid phase extraction was used for sample clean up. The samples were analysed and quantified by LC-MS (Samuelsen, Lunestad et al. 2014).

Emamectin and ivermectin

Emamectin and ivermectin were extracted with acetonitrile, and the extract were purified by solid phase extraction. The samples was analysed and quantified by LC-MS (Hamre, Lunestad et al. 2011).

Cypermethrin and deltamethrin

Cypermethrin and deltamethrin were extracted from the samples with acetone. The samples were analysed and quantified by gas chromatography-electron capture detector (GC-ECD).

Fenbendazole

Fenbendazole was extracted using methanol and water. Sample clean up was performed by liquid-liquid extraction. The samples were analysed and quantified by LC-MS/MS.

Praziquantel

Praziquantel was extracted from the sample by acetone. Diethyl ether and hexane were used for sample clean up. Praziquantel was determined by LC-UV.

B3a, Organochlorine compounds

PCDD/PCDF and dl-PCBs.

This is an adaptation to modern clean-up equipment of the US-EPAs (Environmental Protection Agency) methods No. 1613 and 1668. Separation and quantification were performed by high

resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). The method determines all of the 29 compounds on the WHO list: 17 PCDD / PCDF congeners, four non-ortho substituted PCBs: PCB -77, 81, 126 and 169 and eight mono-ortho substituted PCBs: PCB-105, 114, 118, 123, 156, 157, 167 and 189 (Berntssen, Julshamn et al. 2010).

PCB-6

PCB-6 were extracted by hexane. The extract was purified before detection and quantification by GC-MS/MS (Berntssen, Julshamn et al. 2010) or GC-MS (Berntssen, Maage et al. 2011). The method quantified the PCBs no. 28, 52, 101, 138, 153 and 180. The LOQ values for the compounds are listed in Table 8.4.

Polychlorinated pesticides

Pesticides were extracted using hexane at 75^{o}C under 1500 psi pressure. The sample extract was then divided in two. The extract was either acid treated and analysed on GC/MS in EI, or cleaned up through three columns, ChemElut, QuEChERS and C18, and subsequently detected on GC/MS in NCI (Berntssen, Julshamn et al. 2010).

B3b, Organophosphorus compounds

Azamethiphos and dichlorvos

The sample material was extracted with acetone. The extract was cleaned up by gel permeation chromatography and analysed by GC-FPD.

B3c, Chemical elements

Lead, mercury, cadmium and tin

The sample was decomposed in acid, assisted by heat and high pressure. The metals were detected and quantified by inductively coupled plasma mass spectrometer (ICP-MS) (Julshamn, Maage et al. 2007).

Inorganic Arsenic

Inorganic arsenic was extracted by hydrochloric acid in hydrogen peroxide at 90 °C. Inorganic arsenic includes As (III) and As (V). As (III) was oxidised to As (V) during the extraction. Inorganic arsenic was separated from other arsenic compounds by anionic axchange HPLC, and detected by ICP-MS.

Methylmercury

Methylmercury was extracted by Tetramethylammonium Hydroxide. The pH was adjusted before derivatization and extraction by hexane. The samples were analysed by GC-ICP-MS.

Tributyltin

Tributyltin was extracted by acetic acid/methanol. The pH was adjusted before derivatization and extraction by hexane. The samples were analysed by GC-ICP-MS.

B3d, Mycotoxins

Ochratoxin A.

The sample material was weighed in together with Celite, before chloroform and phosphoric acid was added. The sample was further subjected to clean-up by an immunoaffinity column and quantification by HPLC with fluorescence detection.

B3e, Dyes

Malachite green (MG), crystal violet (CV), brilliant green (BG) and their metabolites.

The analytes were extracted with acetonitrile and dichloromethane, and analysed by LC-MS/MS.

B3f, Others

PBDE

PBDEs were measured with the same principle as the PCDD/PCDF and dl-PCBs (B3a, Organochlorine compounds) (Berntssen, Julshamn et al. 2010).

HBCD and TBBPA

The analytes were extracted from the sample by toluene. A liquid/liquid extraction were used for sample clean up before he analytes were detected and quantified by GC-MS.

PFC

Mass-labelled internal standards were added to the sample prior to extraction and sample clean up. PFCs (18 different forms including PFOS and PFOA) were analysed by LC/MS/MS.

PAH

Cyclohexane was added to extract the PAHs from the sample. PAHs were analysed by GC/MS.

Table 3.1. Number of fish of each species and the number of parameters analysed

	Compounds	Fish	Atlantic salmon	Rainbow trout	Turbot	Atlantic halibut	Atlantic cod	Arctic char	Wolf-fish
A1 Stilbenes	Diethylstilboestrol Dienoestrol Hexoestrol	300	275	15			10		
A3 Steroids	α- and β-Nandrolon α- and β-Trenbolon	320	285	15			10	10	
A6 Illegal drugs	Chloramphenicol	1110	1015	45	5	10	20	10	5
	Metronidazole Metronidazole-OH	720	660	45			15		
	Nitrofuran metabolites (AOZ, AMOZ, AHD, SEM)	875	800	40		5	20	10	
	Malachite green * Leucomalachite green Crystal violet Leucocrystal violet Brilliant green	495	450	40			5		
B1 Chemical method in muscle	Florfenicol	100	95	5					
	Oxytetracycline	100	95	5					
	Flumequine	100	95	5					
	Oxolinic acid	95	85	10					
B1 Microbiological assay in liver	Quinolones	1715	1555	125	5	5	10	10	5
	Tetracyclines and Amphenicols								
	Sulphonamides								
B2 Other veterinary drugs	Teflubenzuron	255	245	10					
	Diflubenzuron	250	240	10					
	Cypermethrin	110	105	5					
	Praziquantel	495	460	30			5		

	Fenbendazole	50	50						
	Emamectin	630	600	30					
	Ivermectin	50	45	5					
	Deltamethrin	90	85	5					
B3a Organochlorine compounds	DDT	545	500	35			10		
	Pesticides other than DDT	530	485	35			10		
	Dioxins and dl-PCBs	555	510	30	5	10			
	PCB-6	1110	1035	60	5	10			
B3b, Organophosphorus compounds	Azamethiphos	225	215	10					
	Dichlorvos	50	50						
B3c Chemical elements	Lead Cadmium Mercury Arsenic	770	660	100			10		
	Inorganic Arsenic Methylmercury Tributyltin	90	80	10					
B3d	Mycotoxins	240	235	5					
B3e, Dyes	Malachite green * Leucomalachite green Crystal violet Leucocrystal violet Brilliant green	805	740	60				5	
B3f, Others	PBDE	555	510	30	5	10			
	TBBPA and HBCD	250	230	10			5		5
	PAH	240	240						
	PFC	245	235				5	5	

*According to directive 96/23, malachite green belongs to the group B3e. However, malachite green is not allowed to be used for food producing animals, therefore samples analysed for dyes have been collected as both group A samples and group B samples.

4. RESULTS

4.1 Group A

Totally 764 pooled fillet samples from 3 820 fish, were examined with respect to residues of substances in group A. For banned substances, any presence of a compound, regardless of concentration, will lead to a non-compliant result.

4.1.1 Group A1

The levels of the group A1 substances diethylstilbestrol, dienestrol and hexoesterol were examined in 60 pooled samples from a total of 300 fish from three species. The detection limits (LODs) are listed in Annex I, and the number of fish from each species is listed in Table 3.1. None of the substances was detected in any of the samples analysed.

4.1.2 Group A3

The levels of group A3 substances (α- and β nandrolon and α- and β trenbolon) were analysed in 64 pooled samples from 320 fish from four different species. LODs are listed in Annex I, the number of fish from each species is listed in Table 3.1. None of the substances was detected.

4.1.3 Group A6

A total of 640 pooled samples from 3 200 fish were analysed in this group. The samples were analysed for chloramphenicol, metronidazole, nitrofurans or dyes. LODs are listed in Annex I, and the number of fish analysed of each species is listed in Table 3.1. No residues was detected in this group.

4.2 Group B

A total of 1 698 pooled fish samples of fillets from a total of 8 490 fish, and additionally 1 715 individual fish liver samples for the inhibition test, were analysed. Samples were collected at processing plants of fish that were of market-size. The samples were analysed for veterinary drugs or contaminants.

4.2.1 Group B1, antibacterial agents

The antibacterial agents in class B1 was determined by a combination of chemical methods and the three plate bioassay. The broad groups a) quinolones, b) amphenicols and tetracyclines and c) sulphonamides, were measured in livers from 1 715 fish, giving a total of 5 145 bioassay determinations. The B1 antibacterial agents: florfenicol, oxytetracyclin, flumequin and oxolinic acid,

were also analysed by chemical methods in 79 pooled fillet samples, representing 395 fish. The LODs/LOQs for each compound are listed in Table 8.9.

4.2.2 Group B2a anthelmintics

The levels of the anthelmintics; teflubenzuron, diflubenzuron, cypermethrin, praziquantel, fenbendazole, emamectin, ivermectin and deltamethrin were determined in 386 pooled fillet samples representing 1 930 fish from three species. Emamectin was detected in two out of 126 pooled samples. According to the analytical protocol, any detection of drug residues would be followed by a re-analysis of the back up sample, consisting of muscle and skin in natural proportions, in duplicate. Analyses of muscle and skin gave concentrations ranging from between LOD and LOQ to 32 µg/kg for emamectin. This concentration was well below the MRL of 100 µg/kg (EU 37/2010). Residues of other agents in this group, or their metabolites were not detected in any of the samples. LODs/LOQs for the substances are specified in Table 8.9.

4.2.3 Group B3b. Organophosphorous compounds

The levels of the B3b substances azamethiphos and dichlorvos were determined in 45 and 10 pooled fillet samples respectively, representing 225 and 50 fish from Atlantic salmon and rainbow trout. Residues of these agents were not detected in any of the examined samples.

4.2.4 Group B3a, Organochlorine compounds

In 2013, there were 328 pooled samples of 1 640 fish analysed for these compounds. The results are summarised in Table 4.1 to 4.3.

4.2.5 Organochlorine pesticides

The sum of DDT is calculated as both lower bound (LB) and upper bound (UB). For LB calculation, analytes with levels below LOQ are calculated as zero. When using UB calculations, the numerical value of LOQ is used for analytes with levels below LOQ. UB sum represents a “worst case scenario”.

The UB-mean of sum DDT was 5 µg/kg w.w., and the highest concentration was 11 µg/kg w.w. Data suggest that there is a significant variation in levels among fish species. The levels reflect the variation in their fat content, which is consistent with the lipophilic nature of DDT.

Table 4.1. DDT (µg/kg w.w.) in fillets of farmed fish

		Atlantic Salmon	Rainbow trout	Atlantic Cod	All Groups
LB-SUM DDT	N	100	7	2	109
	#values	100	7	2	109
	LB-Mean	3.8	4	0.4	3.7
	Min	0.8	3.8	0.2	0.2
	Max	10	5	0.7	10
UB-SUM DDT	N	100	7	2	109
	#values	100	7	2	109
	UB-Mean	5	6	1.1	5
	Min	2.8	5	0.9	0.9
	Max	11	6	1.3	11

LB-mean: Zero substituted for all values <LOQ in the calculation.
UB-mean: Numerical value of LOQ substituted for all values <LOQ in the calculation.

The results for the other 25 pesticides analysed are summarised in Table 4.2. The highest concentrations measured in 2013 were for dieldrin (3.1 µg/kg w.w.) and toxaphene (TOX)-62 (3.8 µg/kg w.w.).

Table 4.2. Pesticides (µg/kg w.w.) in fillets of farmed fish.

Pesticide		Atlantic salmon	Rainbow Trout	Atlantic Cod	All Groups	LOQ
α- Hexachlorocyclo-hexane	N	94	7	2	103	
	#Values	83	7	0	90	
	UB-mean	0.1	0.2	-	0.1	
	Min	LOQ	0.1	-	LOQ	0.03
	Max	0.3	0.2	LOQ	0.3	0.08
γ-Hexachlorocyclo-hexane	N	97	7	2	106	
	#Values	63	4	0	67	
	UB-mean	0.1	0.1	-	0.1	
	Min	LOQ	LOQ	-	LOQ	0.03
	Max	0.2	0.2	LOQ	0.2	0.15
Hexachlorobenzene	N	97	7	2	106	
	#Values	96	7	2	105	
	UB-mean	1.0	1.1	0.2	1.0	
	Min	LOQ	0.8	0.07	LOQ	0.05
	Max	2.5	1.6	0.3	2.5	1.0
Pentachlorobenzene	N	97	7	2	106	
	#Values	30	3	0	33	
	UB-mean	-	-	-	-	

	Min	LOQ	LOQ	-	LOQ	0.05
	Max	0.3	0.3	LOQ	0.3	0.2
Heptachlor	N	97	7	2	106	
	#Values	14	1	1	16	
	UB-mean	-	-	0.02	-	
	Min	LOQ	LOQ	LOQ	LOQ	0.02
	Max	0.09	0.06	0.02	0.09	0.1
Heptachlor A	N	97	7	2	106	
	#Values	4	0	0	4	
	UB-mean	-	-	-	-	
	Min	LOQ	-	-	LOQ	0.02
	Max	0.06	LOQ	LOQ	0.06	0.2
Aldrin	N	97	7	2	106	
	#Values	2	1	0	3	
	UB-mean	-	-	-	-	
	Min	LOQ	LOQ	-	LOQ	0.03
	Max	0.08	0.06	LOQ	0.08	0.4
Isodrin	N	93	6	2	101	
	#Values	45	2	0	47	
	UB-mean	-	-	-	-	
	Min	LOQ	LOQ	-	LOQ	0.03
	Max	2.2	0.2	LOQ	2.2	1.0
Dieldrin	N	97	7	2	106	
	#Values	97	7	2	106	
	UB-mean	1.2	1.3	0.07	1.2	
	Min	0.5	1.0	0.06	0.06	
	Max	3.1	1.6	0.08	3.1	0.03
α-endosulfan	N	97	7	2	106	
	#Values	0	1	0	1	
	UB-mean	-	-	-	-	
	Min	-	LOQ	-	LOQ	0.02
	Max	LOQ	0.05	LOQ	0.05	0.2
β-endosulfan	N	96	7	2	105	
	#Values	0	0	0	0	
	UB-mean	-	-	-	-	
	Min	-	-	-	-	0.02
	Max	LOQ	LOQ	LOQ	LOQ	0.2
Endosulfan sulphate	N	97	7	2	106	
	#Values	39	3	0	42	
	UB-mean	-	-	-	-	
	Min	LOQ	LOQ	-	LOQ	0.02
	Max	0.6	0.1	LOQ	0.6	0.3
***cis*-chlordane**	N	97	7	2	106	
	#Values	93	7	2	102	
	UB-mean	0.6	0.5	0.03	0.6	
	Min	LOQ	0.2	0.03	LOQ	0.08
	Max	1.9	0.7	0.03	1.9	0.1

***oxy*-chlordane**	N	97	7	2	106	
	#Values	93	7	0	100	
	UB-mean	0.1	0.1	-	0.1	
	Min	LOQ	0.1	-	LOQ	0.02
	Max	0.5	0.2	LOQ	0.5	0.05
***trans*-chlordane**	N	94	7	2	103	
	#Values	74	5	1	80	
	UB-mean	0.1	0.1	0.01	0.1	
	Min	LOQ	LOQ	LOQ	LOQ	0.01
	Max	0.2	0.09	0.01	0.2	0.2
***cis*-nonachlor**	N	97	7	2	106	
	#Values	97	7	2	106	
	UB-mean	0.3	0.3	0.02	0.3	
	Min	0.1	0.2	0.01	0.01	
	Max	1.0	0.4	0.02	1.0	0.01
***trans*-nonachlor**	N	97	7	2	106	
	#Values	97	7	1	105	
	UB-mean	0.6	0.6	0.03	0.6	
	Min	0.2	0.4	LOQ	LOQ	0.01
	Max	2.0	0.8	0.03	2.0	0.02
TOX-26	N	97	7	2	106	
	#Values	82	6	0	88	
	UB-mean	0.5	0.4	-	0.5	
	Min	LOQ	LOQ	-	LOQ	0.04
	Max	1.8	0.6	LOQ	1.8	0.5
TOX-32	N	97	7	2	106	
	#Values	0	0	0	0	
	UB-mean	-	-	-	-	
	Min	-	-	-	-	0.1
	Max	LOQ	LOQ	LOQ	LOQ	0.4
TOX-40+41	N	97	7	2	106	
	#Values	82	6	1	89	
	UB-mean	0.3	0.2	0.02	0.2	
	Min	LOQ	LOQ	LOQ	LOQ	0.02
	Max	0.8	0.3	0.02	0.8	0.3
TOX-42a	N	97	7	2	106	
	#Values	80	6	0	86	
	UB-mean	0.2	0.1	-	0.2	
	Min	LOQ	LOQ	-	LOQ	0.02
	Max	0.5	0.3	LOQ	0.5	0.3
TOX-50	N	97	7	2	106	
	#Values	82	6	2	90	
	UB-mean	0.7	0.6	0.04	0.7	
	Min	LOQ	LOQ	0.03	LOQ	0.01
	Max	2.6	1.1	0.04	2.6	0.8
TOX-62	N	80	6	2	88	
	#Values	80	6	2	88	

	UB-mean	1.4	0.7	0.03	1.3	
	Min	0.3	0.6	0.03	0.03	0.03
	Max	3.8	1	0.03	3.8	0.08
Mirex	N	93	6	2	101	
	#Values	2	0	0	2	
	UB-mean	-	-	-	-	
	Min	LOQ	-	-	LOQ	0.03
	Max	0.08	LOQ	LOQ	0.08	0.08

UB-mean: LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ
Max value is defined as the highest measured concentration, irrespective of the varying LOQ values.

4.2.6 Dioxin, dl-PCBs and PCB-6

The sum of dioxins, dioxins + dl-PCBs and PCB-6 are calculated as upper bound (EU 1259/2011), meaning that for congeners with levels below LOQ, the numerical LOQ value should be used.

The level of dioxin is reported as ng toxic equivalents (TEQ)/kg, and represents the sum of 17 different PCDD/F where each congener has been multiplied by a Toxic equivalency factor (TEF). TEF values are determined by WHO, and the toxicity of each congeners has been expressed relative to the most toxic form of dioxin, 2,3,7,8-TCDD which has a TEF value of 1 (EU 1259/2011). Similar, the level of dioxins + dl-PCBs is the sum of 17 PCDD/F and 12 dl-PCBs, each multiplied by their corresponding TEF value.

Sum dioxins ranged from 0.1 ng TEQ/kg to 0.6 ng TEQ/kg w.w., and the UB-mean sum was 0.2 ng TEQ/kg w.w. The maximum value of 0.6 ng TEQ/kg w.w. is below the EU maximum limit of 3.5 ng TEQ/kg w.w.

The sum of all 29 PCDD/F and dl-PCBs ranged from 0.2 to 1.5 ng TEQ/kg w.w. The UB-mean concentration was 0.5 ng TEQ/kg w.w. All values were below the EU maximum limit of 6.5 ng TEQ/kg w.w.

The concentrations of each of the 17 PCDD/F congeners are listed in Table 8.2 and the concentration of each of the 12 dl-PCB congeners are listed in Table 8.3.

The concentrations of PCB-6 in farmed fish are shown in Table 4.3. In 2013, the data is mainly represented by Atlantic salmon (207 samples), but also samples from rainbow trout, Atlantic halibut and turbot have been examined. The UB-mean of PCB-6 for all species was 4.0 µg/kg w.w. The congeners PCB-138 and PCB-153 have been the main contributors to the sum PCB-6 (Table 8.4). The EUs maximum limit for indicator PCBs in fish is 75 µg/kg w.w. The highest concentration of indicator PCBs measured in 2013 was 15 µg/kg w.w., which is well below the maximum limit.

Table 4.3 Dioxins, dlPCBs and PCB-6 in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Atlantic halibut	Turbot	All Groups	Maximum limit
Sum dioxins (ng TEQ/kg w.w.)	Samples	102	6	2	1	111	
	Median	0.2	0.3	0.2	-	0.2	
	UB-Mean	0.2	0.3	0.2	-	0.2	
	Min	0.1	0.2	0.1	-	0.1	
	Max	0. 6	0.4	0.3	0.2	0.6	3.5
Sum dioxin + dl-PCBs (ng TEQ/kg w.w.)	Samples	102	6	2	1	111	
	Median	0.5	0.6	0.6	0.4	0.5	
	UB-Mean	0.5	0.6	0.6	0.4	0.5	
	Min	0.2	0.3	0.2	-	0.2	
	Max	1.5	0.9	0.9	0.4	1.5	6.5
PCB-6 (µg/kg w.w.)	Samples	207	12	2	1	222	
	Median	3.6	3.7	6	2.8	3.6	
	UB-Mean	4.0	4.0	6	2.8	4.0	
	Min	0.4	2.4	2.2	-	0.4	
	Max	15	7	10	2.8	15	75

UB-mean: LOQ substituted for all values <LOQ in the calculation.

4.2.7 Group B3c, Chemical elements

The concentrations of chemical elements were determined in 154 pooled fish samples from the fillets of 770 fish (Table 4.4).

Arsenic

Arsenic is determined as "total arsenic", comprising the sum of all arsenic molecular species, as well as inorganic arsenic. Total arsenic was detected above the LOQ in all samples, and the level ranged from 0.18 to 2.0 mg/kg w.w. (Table 4.4). None of the samples had concentrations of inorganic arsenic above the LOQ (4 µg/kg w.w.) (Table 8.5), indicating that arsenic in fish is present mainly as organo-arsenic compounds of low toxicity (Shiomi 1994) There is currently no EU upper limit for neither total arsenic nor inorganic arsenic in fish fillets.

Cadmium

The concentrations of cadmium in most samples analysed since 2002 have been lower than the LOQ. In 2013, 18% of the samples were above LOQ. The highest concentration measured were 0.01 mg/kg w.w. which is well below EUs maximum limit of 0.05 mg/kg w.w. (EU 1881/2006).

Mercury

In 2013 the concentration of total mercury in farmed fish ranged from 0.01 to 0.06 mg/kg w.w. (Table 4.4). The median concentration of mercury was 0.05 mg/kg w.w. in cod, which was substantially higher than in salmon and rainbow trout, which both had a median of 0.01 mg/kg w.w. The EU maximum limit is 0.50 mg/kg w.w. for mercury in the species analysed in this report (EU 1881/2006). Thus, all samples are well below the maximum limit. In addition to mercury, methylmercury was measured in 18 samples. The result showed that the levels of methylmercury (Table 8.5) were similar to the level of mercury, showing that mercury in salmon and rainbow trout is mainly present as methylmercury.

Lead

Only five of 154 samples of farmed fish fillets analysed had detectable concentrations of lead (Table 4.4). The highest concentration was 0.015 mg/kg w.w. The EU maximum level for lead in muscle meat of fish is 0.30 mg/kg w.w. (EU 1881/2006). Thus, all samples were well below the limit.

Tributyltin

Tributyltin was detected in two of the samples analysed. The highest level found was 0.35 µg/kg w.w. (Table 4.4).

Table 4.4. Chemical elements in fillets of farmed fish

Element		Salmon	Rainbow trout	Cod	All Groups	LOQ	EU-Limit
Arsenic (mg/kg w.w.)	N	132	20	2	154		
	#Values	132	20	2	154		
	Median	0.50	0.49	0.66	0.49		
	UB-Mean	0.55	0.64	0.66	0.56		
	Min	0.18	0.37	0.53	0.18		
	Max	1.6	2.0	0.78	2.0	0.003	
Cadmium (mg/kg w.w.)	N	132	20	2	154		
	#Values	27	1	0	28		
	Median	-	-	-	-		
	UB-Mean	-	-	-	-		
	Min	LOQ	LOQ	-	LOQ	0.001	
	Max	0.01	0.01	LOQ	0.01	0.002	0.050
Mercury (mg/kg w.w.)	N	132	20	2	154		
	#Values	132	20	2	154		
	Median	0.013	0.014	0.047	0.013		
	UB-Mean	0.014	0.018	0.047	0.015		
	Min	0.007	0.011	0.039	0.007		
	Max	0.041	0.053	0.055	0.055	0.002	0.50
Lead (mg/kg w.w.)	N	132	20	2	154		
	#Values	6	0	0	6		
	Median	-	-	-	-		
	UB-Mean	-	-	-	-		
	Min	LOQ			LOQ	0.006	
	Max	0.015	LOQ	LOQ	0.015	0.01	0.30
Tri butyltin (µg/kg w.w.)	N	16	2	0	18		
	#Values	2	0		2		
	Median	-	-	-	-		
	UB-Mean	-	-		-		
	Min	LOQ	-		LOQ		
	Max	0.35	LOQ		0.35	0.30	

UB-mean: LOQ substituted for all values <LOQ in the calculation.
No mean/median is given if more than 50% of the results are below LOQ.

4.2.8 Group B3d, Mycotoxins

In 2013, 48 pooled samples were analysed for Ochratoxin-A. All samples, except one sample of rainbow trout, were from salmon. Ochratoxin-A was not detected in any of the samples.

4.2.9 Group B3e, Dyes

A total of 161 pooled samples from 805 fish, sampled at processing plants, were examined with respect to malachite green and its metabolite leuco malachite green, crystal violet and its metabolite leuco crystal violet, and brilliant green. No residues of these agents were detected.

4.2.10 Group B3f, others

Both PBDE, TBBPA and HBCD are compounds used as flame retardants. The summarised PBDE-7 (28, 47, 99, 100, 153, 154, 183) values are shown in Table 4.5. The levels in salmon ranged from 0.05 to 1.2 µg/kg w.w. with a mean value of 0.4 µg/kg w.w. The level for each PBDE congeners is reported in Table 8.6. Most of the samples had TBBPA level below the LOQ. The highest concentration of HBCD were 1.8 µg/kg w.w. There is currently no EU maximum limit for BFRs in food.

Table 4.5 BFR (µg/kg w.w.) in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Turbot	Atlantic halibut	All Groups	LOQ
PBDE 7	Samples	102	6	1	2	111	
	#Values	102	6	1	2	111	
	UB-Mean	0.4	0.4	-	0.6	0.4	
	Min	0.05	0.3	-	0.2	0.05	
	Max	1.2	0.8	0.2	1.1	1.2	
				Atlantic cod	**Wolffish**		
TBBPA	Samples	46	2	1	1	50	
	#Values	5	0	0	0	5	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.04
	Max	0.15	LOQ	LOQ	LOQ	0.15	0.20
HBCD	Samples	46	2	1	1	50	
	#Values	45	2	1	1	49	
	UB-Mean	0.4	0.1	0.01	0.5	0.4	
	Min	LOQ	0.1	-	-	LOQ	
	Max	1. 8	0.15	0.01	0.5	1.8	0.01

UB-mean: LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ.
Max value is defined as the highest measured concentration, irrespective of the varying LOQ values.

A total of 49 samples were analysed for the PFCs, of which one sample was Atlantic cod, one Arctic char, and the rest were Atlantic salmon. The results are given in Table 8.7. All measurements were below the LOQ.

Table 8.8 summarises the results for the PAH compounds analysed in farmed fish in 2013. PAH was analysed in 48 salmon samples. Benzo(a)pyrene was detected in four samples, and Chrysene/Triphenylene was detected in one sample. PAH does not accumulate in muscle meat due to rapid metabolism. Therefore, maintaining the previous maximum limit (EU 1881/2006) was no longer appropriate (EU 835/2011).

5. DISCUSSION

5.1 Veterinary drugs

Most samples reviewed in this report are from fillet of farmed fish. However, as the liver has central function in the distribution and elimination of drugs, liver samples were analysed for certain antibiotics. Even though the bioassay used for the antibacterial agents is less sensitive than the chemical analytical methods, the higher concentrations of antibacterial agents in liver compared to fillet enhance the ability to detect any antibiotics. Moreover, the ability of the bio-assay to detect a wider range of antibiotics than the more specific chemical methods renders the method useful for screening purposes. Any positive detection by the inhibition assay has to be verified by chemical analysis of the corresponding fillet sample sampled from the same fish.

A total of 1024 samples, consisting of 5120 farmed fish were analysed for veterinary drugs in 2013. Veterinary drugs were detected in two of the samples, both samples containing residues of emamectin. No residues of other veterinary drugs were detected. The amount of anti sealice agents used has increased significantly over the last years (Norwegian Institute of Public Health 2014). This may partly be explained by a shift in the use from drugs in the feed towards drugs used as bath treatment, the amount of veterinary drugs needed per bath treatment by far exceeds the amount used in feed during treatment. The reason for this shift is resistance towards several of the drugs used (Midtlyng, Grave et al. 2011).

The use of antibiotics in farmed fish has been relatively stable during the last decade and no residues of antibiotics has been detected in this period. Similarly, no residues of endoparasitic agent has been detected the last decade in Norwegian farmed fish.

5.2 Contaminants

The monitoring of undesirables in Norwegian farmed fish has been executed at NIFES since the late 90s. The general trend for most contaminants analysed in this program, is that the levels in farmed salmon are significantly declining, reflecting the shift from fish based to more vegetable based raw materials in the feed. The levels of sum dioxins + dl-PCBs have decreased from 1.4 ng TEQ/kg w.w. to 0.5 ng from 2002 to 2013. Since 2005, when the metals were included in this monitoring program, the level of mercury has declined from 0.037 mg/kg w.w. to 0.014 mg/kg w.w., and the level of arsenic has declined from 2.0 mg/kg w.w. to 0.55 mg/kg w.w.

Similarly, the level of DDT has decreased in farmed salmon. Sum DDT has declined from 11.8 µg/kg in 2002 to 5 µg/kg in 2013. Since DDT is banned for use, one can normally find it in the aquatic environment rather than in the terrestrial, due to runoff and consequent accumulation in the marine biota. The shift in fish-feed towards less fish and more vegetables could therefore explain the decline of DDT in fillets.

Apart from the "classic aquatic" contaminants, also the PBDE have declined during the last years. The first analyses of PBDE in this program were performed in 2007 and the average measured concentration in salmon fillet was 1.5 µg/kg w.w. compared to 0.4 µg/kg w.w. in 2013.

6. CONCLUSION

None of the substances with anabolic effect (group A1 and A3) was detected in any of the samples analysed in 2013. Nor were any residues found for the illegal compounds in group A6.

None of the veterinary drugs exceeded the MRL established for fish, in the monitoring program in 2013. Emamectin was detected in two samples; the levels measured were well below the MRL.

Similarly to veterinary drugs, all the environmental contaminants (organochlorine compounds and chemical elements) analysed in farmed fish in 2013 were found at levels below the EU maximum limit, for those compounds for which such limits have been established (sum dioxins, dl-PCBs, PCB-6, mercury, lead and cadmium).

The general trend for most contaminants analysed in this program shows that the level in farmed salmon is significantly declining, which reflects the shift from fish based raw materials in the feed to more vegetable based.

7. RECOMMENDATIONS

The results shows that there is now detection of illegal compounds, and that no veterinary drugs or contaminants are found above their MRL/maximum limit. Based on the results in this report, farmed fish is safe food.

8. TABLES

Table 8.1 DDT, DDD and DDE (µg/kg w.w.) in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Atlantic cod	All groups	LOQ
	N	100	7	2	109	
op-DDT	# values	3	0	0	3	
	UB-Mean	-	-	-	-	
	Min	LOQ	-	-	LOQ	0.15
	Max	0.2	LOQ	LOQ	0.2	0.4
pp-DDT	# values	50	5	0	55	
	UB-Mean	0.5	0.5	-	0.5	
	Min	LOQ	LOQ	-	LOQ	0.15
	Max	0.9	0.6	LOQ	0.9	0.6
op-DDD	# values	1	1	0	2	
	UB-mean	-	-	-	-	
	Min	LOQ	LOQ	-	LOQ	0.15
	Max	0.4	0.5	LOQ	0.5	0.5
pp-DDD	# values	98	7	1	106	
	UB-mean	0.9	1.1	0.17	0.9	
	Min	LOQ	0.9	LOQ	LOQ	0.15
	Max	1.9	1.3	0.19	1.9	0.5
op-DDE	# values	0	0	0	0	
	UB-mean	-	-	-	-	
	Min	-	-	-	-	0.10
	Max	LOQ	LOQ	LOQ	LOQ	0.5
pp-DDE	# values	100	7	2	109	
	UB-mean	2.6	2.9	0.3	2.6	
	Min	0.8	2.5	0.2	0.2	0.15
	Max	7	3.3	0.5	7	0.4

UB-mean:LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ
Max value is defined as the highest measured concentration, irrespective of the varying LOQ values.

Table 8.2 Dioxins (PCDD/F) (ng TEQ/kg w.w.) in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Atlantic halibut	Turbot	All Groups	LOQ
	N	102	6	2	1	111	
2378-TCDD	Values	4	0	0	0	4	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.02
	Max	0.07	LOQ	LOQ	LOQ	0.07	0.1
12378-PeCDD	Values	5	0	1	0	6	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	LOQ	-	LOQ	0.03
	Max	0.1	LOQ	0.09	LOQ	0.1	0.4
123478-HxCDD	Values	0	0	0	0	0	
	UB-Mean	-	-	-	-	-	
	Min	-	-	--	-	-	0.002
	Max	LOQ	LOQ	LOQ	LOQ	LOQ	0.01
123678-HxCDD	Values	3	0	1	0	4	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	LOQ	--	LOQ	0.002
	Max	0.006	LOQ	0.005	LOQ	0.006	0.02
123789-HxCDD	Values	0	0	0	0	0	
	UB-Mean	-	-	-	-	-	
	Min	-	-	-	-	-	0.002
	Max	LOQ	LOQ	LOQ	LOQ	LOQ	0.01
1234678-HpCDD	Values	2	0	0	0	2	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.0001
	Max	0.0006	LOQ	LOQ	LOQ	0.0006	0.001
OCDD	Values	6	0	0	0	6	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.000006
	Max	0.00006	LOQ	LOQ	LOQ	0.00006	0.00006
2378-TCDF	Values	101	6	2	1	110	
	UB-Mean	0.04	0.04	0.04	-	0.04	
	Min	0.005	0.02	0.02	-	LOQ	
	Max	0.2	0.06	0.06	0.01	0.2	0.005
12378-PeCDF	Values	30	0	1	0	31	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	LOQ	-	LOQ	0.0006
	Max	0.006	LOQ	0.002	LOQ	0.006	0.006

23478-PeCDF	Values	94	5	2	1	102	
	UB-Mean	0.04	0.03	0.06	-	0.04	
	Min	0.01	LOQ	0.03	-	LOQ	0.01
	Max	0.2	0.03	0.09		0.2	0.06
123478-HxCDF	Values	1	0	0	0	1	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.001
	Max	0.004	LOQ	LOQ	LOQ	0.004	0.008
123678-HxCDF	Values	2	0	1	0	3	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	LOQ	-	LOQ	0.001
	Max	0.005	LOQ	0.003	LOQ	0.005	0.007
123789-HxCDF	Values	3	0	0	0	3	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-		-	LOQ	0.001
	Max	0.007	LOQ	LOQ	LOQ	0.007	0.01
234678-HxCDF	Values	12	0	0	0	12	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.001
	Max	0.006	LOQ	LOQ	LOQ	0.006	0.009
1234678-HpCDF	Values	1	0	0	0	1	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.0001
	Max	0.0004	LOQ	LOQ	LOQ	0.0004	0.001
1234789-HpCDF	Values	3	0	0	0	3	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.0001
	Max	0.0008	LOQ	LOQ	LOQ	0.0008	0.001
OCDF	Values	5	0	0	0	5	
	UB-Mean		-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.000003
	Max	0.00006	LOQ	LOQ	LOQ	0.00006	0.00003

UB-mean:LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ.
Max value is defined as the highest measured concentration, irrespective of the varying LOQ values.

Table 8.3 dl-PCB (ng TEQ/kg w.w.) in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Atlantic halibut	Turbot	All Groups	LOQ
	N	102	6	2	1	111	
PCB-77	Values	102	6	2	1	111	
	UB-Mean	0.0009	0.001	0.001	-	0.001	
	Min	0.0001	0.0006	0.0003	-	0.0001	
	Max	0.002	0.001	0.001	0.0007	0.002	0.00004
PCB-81	Values	61	0	2	1	64	
	UB-Mean	0.0001	-	0.0001	-	0.0001	
	Min	LOQ	-	0.0001	-	LOQ	0.00009
	Max	0.0003	LOQ	0.0002	0.00006	0.0003	0.0003
PCB-126	Values	102	6	2	1	111	
	UB-Mean	0.2	0.2	0.3	-	0.2	
	Min	0.02	0.1	0.1	-	0.02	
	Max	0.8	0.4	0.5	0.2	0.8	0.04
PCB-169	Values	101	6	2	1	110	
	UB-Mean	0.02	0.01	0.02	-	0.02	
	Min	LOQ	0.006	0.01	-	LOQ	
	Max	0.06	0.02	0.03	0.01	0.06	0.003
PCB-105	Values	101	6	2	1	110	
	UB-Mean	0.005	0.005	0.006	-	0.005	
	Min	LOQ	0.003	0.002	-	LOQ	
	Max	0.02	0.008	0.01	0.003	0.02	0.0003
PCB-114	Values	10	0	1	0	11	
	UB-Mean		-	-	-	-	
	Min	LOQ	-	LOQ	-	LOQ	0.0002
	Max	0.0009	LOQ	0.0006	LOQ	0.0009	0.0006
PCB-118	Values	102	6	2	1	111	
	UB-Mean	0.02	0.02	0.02	-	0.02	
	Min	0.001	0.01	0.008	-	0.001	
	Max	0.05	0.02	0.03	0.009	0.05	0.0003
PCB-123	Values	74	6	1	1	82	
	UB-Mean	0.0005	0.0005	-	-	0.0005	
	Min	LOQ	0.0004	LOQ	-	LOQ	0.0002
	Max	0.001	0.0007	0.001	0.0002	0.001	0.0006
PCB-156	Values	101	6	2	1	110	
	UB-Mean	0.001	0.001	0.002	-	0.001	
	Min	LOQ	0.001	0.001	-	LOQ	
	Max	0.004	0.002	0.003	0.001	0.004	0.0003

PCB-157	Values	69	3	1	1	74	
	UB-Mean	0.0005	0.0004	-	-	0.0004	
	Min	LOQ	LOQ	LOQ	-	LOQ	0.0002
	Max	0.001	0.0007	0.0009	0.0003	0.001	0.0006
PCB-167	Values	101	6	2	1	110	
	UB-Mean	0.0009	0.0009	0.001	-	0.001	
	Min	LOQ	0.0005	0.0004	-	LOQ	
	Max	0.002	0.001	0.002	0.0007	0.002	0.0003
PCB-189	Values	1	0	1	0	2	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	LOQ	-	LOQ	0.0002
	Max	0.0004	LOQ	0.0004	LOQ	0.0004	0.0006

UB-mean: LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ
Max value is defined as the highest measured concentration, irrespective of the varying LOQ values.

Table 8.4 PCB-6 (µg/kg w.w.) in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Turbot	Atlantic Halibut	All groups	LOQ
	N	207	12	1	2	222	
PCB-28	# Values	133	7	1	2	143	
	UB-Mean	0.2	0.2	-	0.3	0.2	
	Min	LOQ	LOQ	-	0.1	LOQ	0.01
	Max	0.9	0.5	0.2	0.4	0.9	0.3
PCB-52	# Values	194	11	1	2	209	
	UB-Mean	0.4	0.4	-	0.5	0.4	
	Min	LOQ	LOQ	-	0.2	LOQ	0.01
	Max	1.7	0.7	0.2	0.7	1.7	1.0
PCB-101	# Values	207	12	1	2	222	
	UB-Mean	0.7	0.7	-	1.2	0.7	
	Min	0.08	0.4	-	0.4	0.08	0.01
	Max	2.7	1.0	0.4	1.7	2.7	0.03
PCB-138	# Values	207	12	1	2	222	
	UB-Mean	1.0	0.9	-	1.3	1.0	
	Min	0.07	0.5	-	0.5	0.07	0.01
	Max	4.2	1.3	0.7	2.2	4.2	0.03
PCB-153	# Values	207	12	1	2	222	
	UB-Mean	1.3	1.4	-	2.4	1.3	
	Min	0.1	0.7	-	0.8	0.1	0.01
	Max	4.3	3.0	1.0	3.6	4.3	0.03
PCB-180	# Values	207	12	1	2	222	
	UB-Mean	0.4	0.3	-	0.6	0.4	
	Min	0.03	0.2	-	0.2	0.03	0.01
	Max	0.9	0.7	0.3	1.1	1.1	0.03

UB-mean: LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ.

Table 8.5. Inorganic arsenic and methylmercury in fillets of farmed fish

		Salmon	Rainbow trout	All Groups	LOQ
	N	16	2	18	
Inorganic arsenic (µg/kg w.w.)	#Values	0	0	0	
	UB-Mean	-	-	-	
	Min	-	-	-	4
	Max	LOQ	LOQ	LOQ	5
Methyl-mercury (mg/kg w.w.)	#Values	16	2	18	
	UB-Mean	0.016	0.017	0.016	
	Min	0.009	0.016	0.009	
	Max	0.025	0.017	0.025	0.001

UB-mean: LOQ substituted for all values <LOQ in the calculation.
No mean is given if more than 50% of the results are below LOQ.

Table 8.6 PBDE (µg/kg w.w.) in fillets of farmed fish.

		Atlantic Salmon	Rainbow trout	Turbot	Atlantic halibut	All Groups	LOQ
	N	102	6	1	2	111	
PBDE 28	#Values	90	6	1	2	99	
	UB-Mean	0.01	0.01	0.01	0.02	0.01	
	Min	LOQ	0.01	-	0.01	0.00	0.002
	Max	0.04	0.02	0.01	0.03	0.04	0.004
PBDE 47	#Values	102	6	1	2	111	
	UB-Mean	0.27	0.27	0.10	0.40	0.27	
	Min	0.02	0.20	-	0.10	0.02	0.002
	Max	0.70	0.50	0.10	0.70	0.70	0.01
PBDE 66	#Values	89	6	1	2	98	
	UB-Mean	0.01	0.01	0.01	0.01	0.01	
	Min	LOQ	0.004	-	0.004	0.00	0.002
	Max	0.04	0.02	0.01	0.02	0.04	0.006
PBDE 99	#Values	101	6	1	2	110	
	UB-Mean	0.05	0.05	0.02	0.11	0.05	
	Min	LOQ	0.04	-	0.02	0.01	0.002
	Max	0.10	0.09	0.02	0.20	0.20	0.008
PBDE 100	#Values	101	6	1	2	110	
	UB-Mean	0.06	0.06	0.04	0.07	0.06	
	Min	LOQ	0.03	-	0.03	LOQ	0.002
	Max	0.20	0.10	0.04	0.10	0.20	0.004
PBDE 119	#Values	2	0	0	0	2	
	UB-Mean	-	-	-	-	-	
	Min	LOQ	-	-	-	LOQ	0.002
	Max	0.01	LOQ	LOQ	LOQ	0.01	0.006
PBDE 138	#Values	0	0	1	0	1	
	UB-Mean	-	-	0.07	-	-	
	Min	-	-	-	-	-	0.002
	Max	LOQ	LOQ	0.07	LOQ	0.07	0.01
PBDE 153	#Values	98	6	1	2	107	
	UB-Mean	0.01	0.01	0.01	0.01	0.01	
	Min	LOQ	0.01	-	0.01	LOQ	0.002
	Max	0.02	0.01	0.01	0.02	0.02	0.006
PBDE 154	#Values	101	6	1	2	110	
	UB-Mean	0.03	0.03	0.01	0.03	0.03	
	Min	LOQ	0.02	-	0.01	LOQ	0.002
	Max	0.09	0.04	0.01	0.05	0.09	0.004
PBDE 183	#Values	0	0	0	0	0	
	UB-Mean	-	-	-	-	-	
	Min	-	-	-	-	-	0.002
	Max	LOQ	LOQ	LOQ	LOQ	LOQ	0.01

UB-mean: LOQ substituted for all values <LOQ in the calculation
No mean is given if more than 50% of the results are below LOQ.

Table 8.7. PFCs (µg/kg w.w.) in fillets of farmed fish

Compound	N	#Values	Max value	LOQ
PFBA	49	0	<LOQ	1.0-1.5
PFBS				0.8-1.5
PFDA				0.3-0.5
PFDoDA				0.3-0.8
PFDS				0.3-1.0
PFHpA				0.3-0.7
PFHxA				0.3-0.9
PFHxDA				13-24
PFHxS				0.3-0.8
PFNA				0.3-0.9
PFOA				0.3-1.3
PFODA				7-24
PFOS				0.3-0.8
PFOSA				0.3-1.2
PFPeA				0.3-6.0
PFTeDA				0.3-1.1
PFTrDA				0.3-1.2
PFUdA				0.3-1.0

Table 8.8. PAH (µg/kg w.w.) in fillets of farmed salmon

PAH congener	N	#values	Max	LOQ
5-Methylchrysene	48	0	<LOQ	0.1-1.0
Benzo(a)antracene		0	<LOQ	0.1-0.5
Benzo(a)pyrene		4	0.6	0.1-0.5
Benzo(b)fluoranthene		0	<LOQ	0.1-0.5
Benzo(ghi)perylene		0	<LOQ	0.1-0.5
Benzo(j)fluoranthene		0	<LOQ	0.1-0.5
Benzo(k)fluoranthene		0	<LOQ	0.1-0.5
Benzo(c)Fluorene		0	<LOQ	0.1-1.0
Chrysene/Triphenylene		1	0.11	0.1-0.5
Cyclopenta(c,d)pyrene		0	<LOQ	0.1-1.0
Dibenzo(a,e)pyrene		0	<LOQ	0.5-1.5
Dibenzo(a,h)anthracene		0	<LOQ	0.1-0.5
Dibenzo(a,h)pyrene		0	<LOQ	0.5-1.5
Dibenzo(a,i)pyrene		0	<LOQ	0.5-1.5
Dibenzo(a,l)pyrene		0	<LOQ	0.5-1.5
Indeno(1,2,3-cd)pyrene		0	<LOQ	0.1-0.5

Table 8.9. Summary of analytical methods

Group of substances	Compounds[1]	Method	LOD (µg/kg w.w.)	LOQ (µg/kg w.w.)	Level of action (µg/kg w.w.)	Labora-tory

A1 Stilbenes	Diethylstilbestrol	GC-MS	0.4		Presence	OUH
	Dienestrol		0.7		Presence	
	Hexoestrol		0.6		Presence	
A3 Steroids	α-nandrolon	GC-MS	0.6		Presence	OUH
	β-nandrolon		0.6		Presence	
	α-trenbolon		0.6		Presence	
	β-trenbolon		0.6		Presence	
A6 Annex IV substances	Chloramphenicol	LC-MS	0.25		Presence (MRPL = 0.3)	NIFES
	Metronidazole[3]	LC-MS/MS	0.3		Presence	
	Hydroxy-metronidazole[3]		2.0		Presence	
	Nitrofuran AOZ	LC-MS/MS	0.5		Presence (MRPL =1.0)	
	Nitrofuran AHD		0.6		Presence (MRPL =1.0)	
	Nitrofuran AMOZ		0.4		Presence (MRPL =1.0)	
	Nitrofuran SEM		0.5		Presence (MRPL= 1.0)	
B1 Antibacterial Substances Micro-biological Method	Quinolones	3-plate Screening Method[2]	200		100-600	NIFES
	Tetracyclines		200		100	
	Amphenicols		200		1000	
	Sulfonamides		400		100	
B1 Antibacterial substances Chemical method	Oxolinic acid	LC-MS/MS		50	100	Eurofins
	Flumequine			50	600	
	Oxytetracycline	LC-MS/MS		50	100	
	Florfenicol	LC-MS/M	0.2	0.5	1000	NIFES
B2a Anthelmintics	Praziquantel	LC-UV	50	100	n.a.	NIFES
	Fenbendazole[3]	LC-MS/MS	0.3	1.0	n.a.	
	Emamectin	LC-MS	3	5.0	100	
	Ivermectin		25	50	n.a.	
	Diflubenzuron	LC-MS	10	20	1000	
	Teflubenzuron		5	15	500	
	Cypermethrin	GC-EC		10	50	Eurofins
	Deltamethrin			10	10	
B3a Organo-chlorine compounds	Dioxins and dlPCB	GC-HRMS		0.006-10 ng/kg	6.5 ng TEQ/kg	NIFES
	PCB-6	GC-MS		0.01 – 1.0	75	
	Pesticides	GC-MS		0.02-1.0	n.a.	
B3b Organo-phosphorus compounds	Azametiphos	GC-FPD		20	n.a.	Eurofins
	Dichlorvos			10	n.a.	
B3c Chemical elements	Lead	ICP-MS		0.01 mg/kg	0.3 mg/kg	NIFES
	Cadmium			0.02 mg/kg	0.05 mg/kg.	
	Arsenic			0.003 mg/kg	n.a.	

	Mercury			0.002 mg/kg	0.5 mg/kg	
	Inorganic arsenic	LC-ICP-MS		4-5		NIFES
	Methylmercury	GC-ICP-MS		1.0		
	Tributyltin[3]	GC-ICP-MS		0.3		
B3d Mycotoxins	Ochratoxin A	HPLC-FLU	0.06		n.a.	NVI
B3e, dyes	Malachite green	LC-MS/MS	0.15		Presence (MRPL=2)	NIFES
	Leuco-malachite green		0.15			
	Crystal violet		0.30		Presence	
	Leuco-crystal violet		0.15		Presence	
	Brilliant green[3]		0.15		Presence	
B3f, others	PBDE	GC-MS		0.002-0.01	n.a.	NIFES
	HBCD	GC-MS		0.01	n.a.	Eurofins
	TBBPA	GC-MS		0.04-0.2	n.a.	
	PAH	GC-MS		0.1-1.5	n.a.	NIFES
	PFC	LC-MS/MS		0.3-24	n.a.	

[1] All methods used muscle as sample matrix except for microbiological methods for antibacterial substances (B1), were liver was used

[2] Only screening method, positive results have to be confirmed by a chemical method.

[3] Not accreditated

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