MONITORING PROGRAM FOR PHARMACEUTICALS, ILLEGAL SUBSTANCES AND CONTAMINANTS IN FARMED FISH

Annual report for 2020

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Summary (English):

This report summarises the monitoring data collected in 2020 on the status of illegal substances, pharmaceuticals and contaminants in Norwegian farmed fish. In 2020, a total of 13 845 fish were sampled. Samples examined for illegal compounds were collected at all stages of farming and are representative of farmed fish under production. The samples were analysed for substances with anabolic effects or unauthorized substances. No residues of illegal compounds were detected. Samples tested for approved veterinary drugs and contaminants were collected at processing plants and are representative of Norwegian farmed fish ready for human consumption. Residues of the anti-sea-lice agent emamectin were found in one sample, with a concentration below the Maximum Residue Limit (MRL). Other veterinary drugs, like antibiotics or drugs used against internal parasites were not found. No environmental contaminants were found above the EU maximum level.

Summary (Norwegian):

Denne rapporten oppsummerer overvåkingsresultatene fra 2020 for ulovlige stoffer, legemidler og miljøgifter i norsk oppdrettsfisk. I 2020, ble det tatt ut prøver av totalt 13 845 fisk. Prøver som ble analysert for ulovlige forbindelser, som stoffer med anabole effekter eller uautoriserte legemidler, ble tatt ut under alle livsstadier, og er representative for oppdrettsfisk under produksjon. Ingen rester av ulovlige forbindelser ble detektert. Prøver som ble testet for godkjente veterinære legemidler og miljøgifter ble samlet inn på slakterier, og er representative for norsk oppdrettsfisk som er klar for markedet. Rester av lusemiddelet emamectin ble funnet i én prøve, med et nivå under grenseverdien (MRL). Andre veterinære legemidler, som antibiotika eller legemidler brukt mot interne parasitter ble ikke funnet. Ingen miljøgifter ble funnet over EUs maksimumsgrenser.

Content

1 - Introduction

1.1 - Background

According to EU legislation (EU 2017/625, replacing Directive 96/23/EC), all food producing animals should be monitored for certain substances and residues thereof. The following residues or substance groups are monitored in Norwegian farmed fish:

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

B2d: Sedatives

B3a: Organochlorine compounds

B3b: Organophosphorus compounds

B3c: Chemical elements

B3d: Mycotoxins

B3e: Dyes

B3f: Others

1.2 - Group A, Substances with anabolic effects and unauthorized substances

Fish tested for illegal compounds were collected at the farm by official inspectors from the Norwegian Food Safety Authority, without prior notification to the farmers. Samples were taken at all stages of farming in order to represent farmed fish during production. Substances monitored in Group A include growth promoters like steroids and stilbenes, and unauthorized drugs. Unauthorized drugs considered most relevant for aquaculture are chloramphenicol, nitrofurans, metronidazole and dyes. Since the use of the dyes malachite green, crystal violet and brilliant green is not allowed for food producing species (EU 37/2010), they are considered Group A substances and hence monitored in samples throughout the production chain. However, according to Regulation (EU) 2017/625, these dyes belong to the group B3e. Thus, in order to fulfill criteria for group B sampling, some of the samples assigned to analysis of dyes were also collected at the slaughterhouse.

To ensure harmonized levels for the control of unauthorized substances, the analytical methods should meet a minimum required performance limits (MRPLs) set by the European Union (EU 2003/1881, EU 2004/25, CRL 2007), and European reference laboratories (EU-RLs), (EU 2003/1881, EU 2004/25, CRL 2007). Table 5.3 gives an overview of MRPLs of relevant compounds.

1.3 - Group B, Veterinary drugs

In order to protect public health, current EU legislation (EU 37/2010) provisions the assignment of Maximum Residue Limits (MRLs) for all legally applied pharmacologically active substances in products intended for human consumption. An MRL denotes the highest permitted residual concentration of a legally applied veterinary drug and is evaluated for each substance and each food product individually. Consumption of food with drug residues below the MRL should not pose a health risk to the consumer. For fish, the MRLs are set for muscle and skin in natural proportions. Samples examined for veterinary drugs were collected from fish at processing plants and the samples are representative of fish ready to be placed on the market for human consumption.

1.4 - Group B, Contaminants

Samples examined for contaminants were collected from fish at processing plants and are representative of fish ready for human consumption. The EU (EU 1881/2006) has set a Maximum limit (ML) for some of the contaminants in fish, while for others, like the pesticides, PAH, PFC and BFR, maximum limits have not yet been established.

2 - Material and methods

2.1 - Sampling

Samples were taken on fish farms or slaughterhouses, by official inspectors from the NFSA, in all fish-producing regions in Norway. The sampling plan was randomised according to season and region. In 2020, the monitoring program included Atlantic salmon (*Salmo salar*), rainbow trout (*Oncorhynchus mykiss*), turbot (*Scophthalmus maximus*), Atlantic halibut (*Hippoglossus hippoglossus*), Arctic char (*Salvelinus alpinus*), Atlantic cod (*Gadus morhua*) and spotted wolffish (*Anarhichas minor*) .

Samples were transported to IMR in a frozen state. For most analyses, the Norwegian quality cut (NQC) was used (Johnsen, Hagen et al. 2011). However, both NQC and individual liver samples were collected for analysis of antibiotics. Samples to be used for analyses of substances with anabolic effects or unauthorized substances also included small fish from early life stages, and in these cases, the whole fish except head, tail and gut were homogenised. The samples were analysed as pooled samples comprising five fish from the same cage/farm.

2.2 - Pre-treatment

Upon arrival at IMR the sample identification was anonymised for the analysts. A back-up sample was stored for all samples. Pooled samples of muscle from five fish from the same cage/farm were homogenised before analyses. Samples of liver were excised from the fish to be screened for residues of antimicrobial agents by the microbiological inhibition zone assay. Liver samples were examined individually, if residues were detected, the back-up sample of muscle was analysed by chemical methods. The maximum residue limits for veterinary drugs are set for muscle and skin in natural proportions (EU 37/2010). Therefore, according to the analytical protocol, any detection of drug residues in the muscle or liver was followed by a re-analysis of the back-up sample, consisting of muscle and skin in natural proportions, in duplicate.

2.3 - Analytical methods

The laboratory routines and most of the analytical methods are accredited in accordance with the standard ISO 17025. A summary of the analytical methods and their limit of detection (LOD) or limit of quantification (LOQ) is shown in Table 5.4. 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 sample blank and a quality control sample (QC) with a known composition and concentration of target analyte are included in each series. The methods are regularly verified by participation in inter-laboratory proficiency tests, or by analysing certified reference material (CRM), where such exist.

2.3.1 - Group A substances

A1, Stilbenes

Stilbenes were extracted by water and acetonitrile. Liquid-liquid extraction was used for sample clean-up. The stilbenes were and analysed by LC-MS/MS.

A3, Steroids

Steroids were extracted by water and acetonitrile. Liquid-liquid extraction followed by solid phase extraction was used for sample clean-up, before the samples were analysed by LC-MS/MS.

A6, Illegal veterinary drugs

Chloramphenicol

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

Nitrofurans

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.

Metronidazole

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

Malachite green (MG), crystal violet (CV), brilliant green (BG)

The analytes were extracted with acetonitrile. Sample clean-ups were performed by solid phase extraction. MG, CV, BG and the metabolites leuco malachite green (LMG) and leuco crystal violet (LCV), were determined by LC-MS/MS.

2.3.2 - Group B substances

B1, Antibacterial agents (antibiotics)

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

Microbiological assay

For the three-plate microbiological inhibition method, a specific bacterial strain was added to a plate containing growth agar. 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 was verified by chemical analysis of muscle and skin.

Oxolinic acid, flumequine, florfenicol, enrofloxacin, ciprofloxacin and trimethoprim

The analytes were extracted with acetonitrile and water. The analysis was performed by LC-MS/MS.

Tetracyclin

Oxytetracycline, doxycycline, chlortetracycline, and tetracycline were extracted with acetonitrile. Liquid-liquid extraction was used to purify the extract. The analytes were analysed by LC-MS/MS.

B2a, Anthelmintics

Flubenzurons

Diflubenzuron, teflubenzuron, lufenuron, hexaflumuron and fluazuron were extracted with acetone. Solid phase extraction was used for sample clean-up. The samples were analysed by LC-MS/MS (Samuelsen, Lunestad et al. 2014).

Emamectin

Emamectin was extracted with acetonitrile, and analysed by LC-MS/MS.

Ivermectin

Ivermectin was extracted with organic solvent, and the extract were purified by solid phase extraction. The samples were analysed by LC-MS/MS.

Cypermethrin and deltamethrin

Cypermethrin and deltamethrin were extracted by soxhlet extraction. The extracts were purified by gel permeation chromatography. The samples were analysed by GC-MS/MS.

Fenbendazole and praziquantel

Fenbendazole and praziquantel were extracted using acetone. The samples were analysed by LC-MS/MS.

B2d, Sedatives

Isoeugenol and eugenol

Isoeugenol and eugenol were analysed by GC coupled to a flame ionization detector (FID).

B3a, Organochlorine compounds

Dioxins, dl-PCBs, PCB-6 and PBDEs.

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 measures 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). The PCBs included in PCB-6, PCBs no. 28, 52, 101, 138, 153 and 180, were analysed by GC-MS/MS. The PBDEs were analysed by GC/MS in a relevant solvent fraction from the EPA clean-up procedure (Pirard, De Pauw et al. 2003). Tri-hepta PBDEs (no. 28, 35, 47, 49, 66, 71, 75, 77, 85, 99, 100, 119, 138, 153, 154, 183) were analysed by GC-MS/MS. Okta-deca PBDEs (no. 196, 197, 206, 207, 209) were analysed by GC-MS in negative chemical ionization mode (NCI).

Chlorinated pesticides

Pesticides were extracted by organic solvent, and the extract were cleaned up by column chromatography, before the pesticides were analysed by HRGC-HRMS.

B3b, Organophosphorus compounds

Azamethiphos and dichlorvos

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

B3c, Elements

Lead, mercury, cadmium, arsenic, cobalt, chromium, copper, iron, manganese, molybdenum, nickel, selenium, silver, vanadium, and zinc

The sample was decomposed by acid treatment, assisted by heat and high pressure. The metals were analysed 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 exchange 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

Enniatin and beauvericin

Beauvericin, enniatin A, enniatin A1, enniatin B and enniatin B1 were extracted with acetonitrile and water. Solid phase extraction was used for sample clean up. The mycotoxins were analysed by LC-MS/MS.

B3f, Others

HBCD

HBCD was extracted by a soxhlet apparatus, using a mixture of acetone and hexane. Sulfuric acid was used for purification. The extract was further cleaned up by an alumina column. The HBCD isomers were analysed by LC-MS/MS.

TBBPA

TBBPA was extracted by a soxhlet apparatus using a mixture of acetone and hexane. Sulfuric acid was used for purification. O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) was used for derivatization. The extract was purified using column chromatography. TBBPA was analyzed by GC-MS using Electron Ionization (EI).

PFC

PFCs were extracted by methanol, the extract was purified by solid phase extraction. PFCs were analysed by LC-MS/MS.

PAH

PAHs were extracted by dichloromethane and cyclohexane using an Accelerated Solvent Extractor (ASE). The extract was purified by solid phase extraction and analysed by GC-MS/MS.

Ethoxyquin

EQ and EQDM were extracted by hexane, after saponification in a mixture of ethanol, NaCl and NaOH. EQ and EQDM were quantified by reversed-phase high-performance liquid chromatography with fluorescence detection, using an external standard curve (Bohne, Hove et al. 2007, Ørnsrud, Arukwe et al. 2011).

Table 2.1. Number of fish analysed for each substance.

Compounds	Fish	Atlantic salmon	Rainbow trout	Atlantic halibut	Arctic char	Turbot	Atlantic cod	Spotted wolffish
A1 Stilbenes								
Zeranol 17alpha-Estradiol 17alpha-Ethinyl-estradiol 17beta-Estradiol beta-Zearalanol Dienestrol Diethylstilbestrol Estriol Estrone Hexestrol	825	785	35		5			
16-Hydroxystanozolol 17alpha-Boldenone 17alpha-Trenbolone alpha-Nandrolone Boldenone Chlor-Testosterone Epitestosterone Methyl-Boldenone Methyltestosterone Nortestosterone Stanozolol Testosterone Testosterone propionate Trenbolone Trenbolone-acetate	830	785	45					
A6 Illegal substances								
Chloramphenicol	830	785	40		5			
Metronidazole	835	790	35	5	5			
Nitrofuran metabolites (AOZ, AMOZ, AHD, SEM)	825	780	35	5	5			

Compounds	Fish	Atlantic salmon	Rainbow trout	Atlantic halibut	Arctic char	Turbot	Atlantic cod	Spotted wolffish
Malachite green* Crystal violet Brilliant green	825	785	30		5		5	
B1 Antibiotics								
Oxytetracycline Doxycycline Chlortetracycline Tetracycline	105	95	10					
Florfenicol Flumequine Oxolinic acid Enrofloxacin Ciprofloxacin Trimethoprim	460	420	35			5		
Quinolones (liver) Tetracyclines (liver) Amphenicols (liver) Sulphonamides (liver)	1470	1325	125		15			5
B2 Other veterinary drugs								
Emamectin	590	550	35	5				
Cypermethrin Deltamethrin	605	550	50		5			
Diflubenzuron Teflubenzuron Hexaflumeron Lufenuron Fluazuron	545	515	30					
Ivermectin Abamectin Doramectin Eprinomectin Moxidectin	80	70	10					

Compounds	Fish	Atlantic salmon	Rainbow trout	Atlantic halibut	Arctic char	Turbot	Atlantic cod	Spotted wolffish
Praziquantel Fenbendazole	495	465	30					
Isoeugenol Eugenol	195	165	30					
B3a Organochlorine compounds								
Pesticides	505	440	60		5			
Dioxin and dl-PCBs PCB-6	425	375	45		5			
B3b Organophosphorous compounds								
Azamethiphos Dichlorvos	250	230	15	5				
B3c Chemical elements								
Lead Cadmium Mercury Arsenic Cobalt Chromium Copper Iron Manganese Molybdenum Nickel Selenium Silver Vanadium Zinc	300	260	35			5		
Inorganic arsenic Methylmercury	105	95	10					
Tributyltin	250	220	30					
B3d Mycotoxins								

Compounds	Fish	Atlantic salmon	Rainbow trout	Atlantic halibut	Arctic char	Turbot	Atlantic cod	Spotted wolffish
Beauvericin Enniatin	500	455	40			5		
B3e Dyes								
Malachite green* Crystal violet Brilliant green	465	420	45					
B3f Others								
PBDE	425	375	45		5			
HBCD and TBBPA	355	345	10					
PAH	360	335	20		5			
PFC	360	310	45		5			
Ethoxyquin	400	380	15	5				

Some of the samples collected have been analysed by more than one method. Therefore, the total of fish in this table will be higher than the number of fish collected.

* Malachite green, crystal violet and brilliant green belongs to the group B3e. However, these dyes are not allowed to be used for food producing animals, therefore samples analysed for dyes have been collected as both group A samples (illegal drugs) and group B samples (dyes) (EU 2017/625).

3 - Results

3.1 - Substances with anabolic effects and unauthorized substances

3.1.1 - Stilbenes

In 2020, a total of 165 pooled samples from Atlantic salmon, rainbow trout and Arctic char were examined for presence of stilbenes. None of the included stilbenes were detected in the samples analysed.

3.1.2 - Steroids

The presence of steroids was examined in 166 pooled fillet samples of Atlantic salmon and rainbow trout. None of the substances were detected in the samples analysed.

3.1.3 - Unauthorized veterinary drugs

A total of 663 pooled samples from Atlantic salmon, rainbow trout, Atlantic halibut, Arctic char and Atlantic cod were analyzed for unauthorized veterinary drugs, including chloramphenicol, nitrofurans, metronidazole and dyes (malachite green, crystal violet, brilliant green). No residues of the included substances were detected in any of the samples.

3.2 - Veterinary drugs

Samples analysed for veterinary drugs were collected from fish at processing plants, representing fish ready for human consumption. The maximum residue limit (MRL) for veterinary drugs is defined for muscle and skin in natural proportions (EU 37/2010). Therefore, according to the analytical protocol, any detection of drug residues in a sample of muscle or liver would be followed by a re-analysis of the backup sample, consisting of muscle and skin in natural proportions, in duplicate.

3.2.1 - Group B1, Antibacterial agents

The antibacterial agents were determined by a combination of the three-plate bioassay and chemical methods. The broad groups a) quinolones, b) amphenicols and tetracyclines and c) sulphonamides were measured in liver samples from 1470 fish. Oxytetracyclin (21 pooled samples) and florfenicol, flumequin, oxolinic acid, enrofloxacin, ciprofloxacin and trimethoprim (92 pooled samples) were also analysed using chemical methods. No residues were detected in any of the analysed samples. The LOQs of the respective compounds are listed in Table 5.4.

3.2.2 - Group B2a, Anthelmintics

The levels of the anthelmintics; teflubenzuron, diflubenzuron, hexaflumenuron, lufenuron, fluazuron, cypermethrin, deltamethrin, emamectin, ivermectin, abamectin, doramectin, eprinomectin, moxidectin, praziquantel and fenbendazole were determined in 463 pooled muscle samples representing 2315 fish. Emamectin was detected in one out of 118 pooled samples of Atlantic salmon, at a concentration of 7.1 µg/kg ww. This concentration is below the MRL of 100 µg/kg (EU 37/2010). No residues were detected for the other anthelmintics. LOQs for the substances are given in Table 5.4.

3.2.3 - Group B2b, Organophosphorous compounds

The levels of the B2b substances azamethiphos and dichlorvos were determined in 46, three and one pooled fillet samples of Atlantic salmon, rainbow trout and Atlantic halibut, respectively. No residues of these agents were detected in any of the examined samples.

3.2.4 - Group B2d, Sedatives

No residues of isoeugenol were detected in any of the 39 samples analysed.

3.3 - Contaminants

Samples analysed for contaminants were collected from fish at processing plants and are representative of fish ready for human consumption.

3.3.1 - Group B3a, Organochlorine compounds

The levels of organochlorine compounds were determined in 186 pooled samples. The results are summarised in Table 3.1 to 3.3.

3.3.1.1 - Organochlorine pesticides

For several of the pesticides, the MRL residue definitions include not only the parent compound, but also its metabolites or other transformation products (EU GD SANTE 2017). To calculate the sum of the components, conversion factors (Table 5.5) are used to adjust for different molecular weights (EU GD SANTE 2017). The results for this group of pesticides are presented in Table 3.1. The sums in Table 3.1 were calculated according to the upper bound (UB) formula. When using UB calculations, the numerical value of LOQ is substituted for analytes with levels below LOQ. UB represents a “worst case scenario”. As an example, all measurements of endosulfan were below LOQ, however, a sum was generated based on the LOQ-values. There are currently no MRLs established in fish fillet for any of the listed pesticides.

Table 3.1. Sums of pesticides (µg/kg w.w.) in fillets of farmed fish.

Pesticide		Atlantic salmon	Rainbow trout	Arctic char
Sum	*n*	88	12	1
DDT	Median (UB)	4.7	4.3	-
	Max (UB)	21	9.0	5.5
Endosulfan	Median (UB)	1.3	1.3	-
	Max (UB)	3.4	3.1	1.3
Aldrin and dieldrin	Median (UB)	1.0	1.0	-
	Max (UB)	3.3	1.8	0.86
Chlordane	Median (UB)	0.90	0.90	-
	Max (UB)	2.0	1.8	0.79
Heptachlor	Median (UB)	0.53	0.53	-
	Max (UB)	1.3	1.3	0.53
Toxaphene	Median (UB)	2.3	2.0	-
	Max (UB)	7.6	4.8	2.0

The levels of pesticides calculated from a sum of several components were comparable to the previous years. The highest values measured in Atlantic salmon fillet were 21 µg/kg w.w. of DDT, and 7.6 µg/kg w.w. Toxaphene. DDT and Toxaphene were also the highest measured concentrations in rainbow trout, with 9.0 and 4.8 µg/kg w.w, respectively.

The results for the other pesticides are summarised in Table 3.2. Hexachlorbenzene and trans-nonachlor were present in concentrations above LOQ in most of the samples. In 2020, the highest levels measured were 3.0 µg/kg w.w. of hexachlorobenzene and 2.1 µg/kg w.w. of trans-nonachlor in Atlantic salmon samples.

Table 3.2. Pesticides (µg/kg w.w.) in fillets of farmed fish.

Pesticide		Atlantic salmon		Rainbow trout	Arctic char	LOQ
	n	88	12	1		
α-Hexachlorocyclo-hexane	#Values	1	0	0		
	Median	LOQ	-	-		
	Max	0.27	LOQ	LOQ		0.13-0.60
β-Hexachlorocyclo-hexane	#Values	3	0	0		
	Median	LOQ	-	-		
	Max	0.66	LOQ	LOQ		0.13-0.60
γ-Hexachlorocyclo-hexane	#Values	1	0	0		
	Median	LOQ	-	-		
	Max	0.25	LOQ	LOQ		0.13-0.60
δ -Hexachlorocyclo-hexane	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ		0.13-0.60
Hexachlorobenzene	#Values	83	10	1		
	Median	0.80	0.75	-		
	Max	3.0	1.4	0.80		0.06-1.0
Pentachlorobenzene	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ		0.30-1.2
Trans-nonachlor	#Values	86	10	1		
	Median	0.50	0.56	-		
	Max	2.1	1.0	0.50		0.13-0.60
Endrin	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ		0.15-0.71
Mirex	#Values	2	0	0		
	Median	LOQ	-	-		
	Max	0.14	LOQ	LOQ		0.05-0.24
Octachlorstyrene	#Values	5	1	0		
	Median	LOQ	LOQ	-		
	Max	0.25	0.14	LOQ		0.03-0.12

3.3.1.2 - Dioxin, dl-PCBs and PCB-6

The levels of dioxin (PCDD+PCDF), dl-PCBs and PCB-6 in farmed fish are shown in Table 3.3. The data is mainly represented by Atlantic salmon, but also samples from rainbow trout and Arctic char were examined. The sums of dioxins, dioxins+dl-PCBs and PCB-6 are calculated as upper bound (EU 1259/2011). Accordingly, the numerical LOQ values were used for congeners with levels below LOQ.

The levels of dioxins and dl-PCBs are reported as ng toxic equivalents 2005 (TEQ05)/kg and represent the sum of 17 different PCDD/F and 12 dl-PCBs where each congener was multiplied by a Toxic Equivalency Factor (TEF). TEF values are determined by WHO, and the toxicity of each congener is expressed relative to the most toxic form of dioxin, which has a TEF value of 1 (EU 1259/2011, Van den Berg, Birnbaum et al. 2006).

For salmon, the median of the sum of dioxins was 0.24 ng TEQ/kg w.w. The maximum value found in salmon (0.61 ng TEQ/kg w.w.) was below the EU maximum limit of 3.5 ng TEQ/kg w.w.

Corresponding to the concentrations found in 2019, in 2020, the median of the sum of all 29 PCDD/F and dl-PCBs was 0.46 ng TEQ/kg w.w for salmon and 0.41 ng TEQ/kg w.w for rainbow trout. The highest result for sum dioxin and dl-like PCBs in salmon was 1.81 ng TEQ/kg w.w. All measured values were below the EU maximum limit of 6.5 ng TEQ/kg w.w.

The median of PCB-6 for salmon was 3.4 µ g/kg w.w and 3.7 in rainbow trout, with maximum concentrations of 12.3 and 5.8 µ g/kg w.w, respectively. The concentration of PCB-6 in Arctic char was 7.7 µ g/kg w.w. For PCB-6, a maximum limit is set at 75 µ g/kg w.w. in the EU.

Table 3.3. Dioxins, dl-PCBs and PCB-6 in fillets of farmed fish.

		Atlantic salmon	Rainbow trout	Arctic char	Maximum limit
	Samples	75	9	1	
Sum dioxins (ng TEQ/kg w.w.)	Median	0.24	0.21	-	
	Max	0.61	0.33	0.30	3.5
Sum dioxin + dl-PCBs (ng TEQ/kg w.w.)	Median	0.46	0.41	-	
	Max	1.81	0.64	0.82	6.5
PCB-6 (µg/kg w.w.)	Median	3.4	3.7	-	
	Max	12	5.8	7.7	75

3.3.2 - Group B3c, Chemical elements

In 2020, monitoring of the levels of chemical elements, such as arsenic (and inorganic arsenic), total mercury in addition to methylmercury, cadmium, lead included 52 samples of Atlantic salmon, 7 samples of rainbow trout, and one sample of turbot. Mono-, di- and tributyltin were analysed in 44 samples of Atlantic salmon and 6 samples of rainbow trout.

The concentrations of total mercury were found below the EU maximum limit, which is set at 0.50 mg/kg w.w. for these species. The highest concentrations of total mercury were 0.05 mg/kg w.w. in salmon and turbot, and 0.03 mg/kg w.w. in rainbow trout (Table 3.4), mainly present as methylmercury (Table 5.1).

Cadmium was at concentrations above the LOQ was found in two out of a total of 60 samples. With a level of 0.004 mg/kg w.w. in the fillet salmon, and 0.003 mg/kg w.w. in rainbow trout, the measured concentration was well below EUs maximum limit of 0.05 mg/kg w.w. (EU 1881/2006).

Arsenic is determined as “total arsenic”, comprising the sum of all arsenic species. In addition, inorganic arsenic was determined in 21 of the samples. The median level of total arsenic in Atlantic salmon was 0.63 mg/kg w.w., and, same as in the previous year, the highest concentration measured was 2.1 mg/kg w.w. (Table 3.4). The concentrations of inorganic arsenic were below the LOQ in all samples measured (Table 5.1), 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.

Of the 60 samples analyzed, a lead concentration above LOQ was found in one sample of salmon. With a fillet concentration of 0.02 mg/kg w.w., the lead concentration was well below the EU maximum level, which is currently set at 0.30 mg/kg w.w. in muscle meat of fish (EU 1881/2006).

Eleven additional chemical elements were included into the surveillance from 2019. There is currently no EU-limit established for any of the newly included elements.

Copper, iron, manganese, selenium and zinc were found at levels above LOQ in all samples analysed (Table 3.4), with median values similar to the year before. The maximum concentrations were 0.6 mg copper/kg, 4 mg iron/kg, 0.43 mg manganese/kg, 0.33 mg selenium/kg and 7.9 mg zinc/kg, respectively, and were all found in salmon. Cobalt, silver, molybdenum or nickel were not detected in any of the analysed samples. Chromium and vanadium were detected in 15 and 44 out of 60 samples, respectively. The highest concentrations were 0.14 mg chromium/kg and 0.013 mg vanadium/kg (both salmon) in 2020.

Mono-, di- and tributyltin were monitored in a total of 50 pooled fillet samples of both salmon and rainbow trout. There is currently no EU upper limit for tin in fish fillet. Monobutyltin was found at levels above LOQ in 3 samples, with the maximum concentrations of 0.6 µg/kg w.w. and 0.5 µg/kg w.w. in salmon and rainbow trout, respectively. One sample of salmon contained dibutyltin at a concentration above LOQ (0.2 µg/kg w.w.). A total of 14 samples contained tributyltin above the LOQ, with the highest measured level of 1.7 µg/kg w.w. found in rainbow trout (median 0.15 µg/kg w.w.).

Table 3.4. Chemical elements in fillets of farmed fish.

Element		Atlantic salmon	Rainbow trout	Turbot	LOQ	EU- Limit
	n	52	7	1		
Mercury (mg/kg w.w.)	#Values	52	7	1		
	Median	0.016	0.025	-		
	Max	0.054	0.032	0.047	0.002	0.50
Arsenic (mg/kg w.w.)	#Values	52	7	1		
	Median	0.63	0.87	-		
	Max	1.8	1.8	2.1	0.003	n.a.
Cadmium (mg/kg w.w.)	#Values	1	0	1		
	Median	LOQ	-	-		
	Max	0.004	LOQ	0.003	0.001-0.002	0.05
Lead (mg/kg w.w.)	#Values	1	0	0		
	Median	LOQ	-	-		
	Max	0.018	LOQ	LOQ	0.006-0.01	0.30
Cobalt (mg/kg w.w.)	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ	0.006-0.009	n.a.
	#Values	12	3	0		

Element		Atlantic salmon	Rainbow trout	Turbot	LOQ	EU- Limit
Chromium (mg/kg w.w.)						
	Median	LOQ	LOQ	-		
	Max	0.14	0.022	LOQ	0.006-0.01	n.a.
Copper (mg/kg w.w.)	#Values	52	7	1		
	Median	0.37	0.37	-		
	Max	0.58	0.48	0.23	0.1	n.a.
Iron (mg/kg w.w.)	#Values	52	7	1		
	Median	2.7	2.9	-		
	Max	4.0	3.3	0.75	0.1	n.a.
Manganese (mg/kg w.w.)	#Values	52	7	1		
	Median	0.076	0.073	-		
	Max	0.43	0.082	0.24	0.03	n.a.
Molybdenum (mg/kg w.w.)	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ	0.01-0.4	n.a.
Nickel (mg/kg w.w.)	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ	0.07-0.1	n.a.
Selenium (mg/kg w.w.)	#Values	52	7	1		
	Median	0.16	0.18	-		
	Max	0.32	0.33	0.20	0.01	n.a.
Silver (mg/kg w.w.)	#Values	0	0	0		
	Median	-	-	-		
	Max	LOQ	LOQ	LOQ	0.002-0.004	n.a.
Vanadium (mg/kg w.w.)	#Values	41	3	0		
	Median	0.004	LOQ	-		
	Max	0.013	0.003	LOQ	0.001-0.002	n.a.
Zinc (mg/kg w.w.)	#Values	52	7	1		
	Median	3.9	3.6	-		
	Max	6.2	4.0	7.9	0.5	n.a.
	n	44	6			
Monobutyltin (µg Sn/kg w.w.)	#Values	2	1			
	Median	LOQ	LOQ			
	Max	0.6	0.5		0.4-1	n.a.
Dibutyltin (µg Sn/kg w.w.)	#Values	1	0			
	Median	LOQ	-			
	Max	0.2	LOQ		0.2-0.5	n.a.

Element		Atlantic salmon	Rainbow trout	Turbot	LOQ	EU- Limit
Tributyltin (µg Sn/kg w.w.)	#Values	9	5			
	Median	LOQ	0.15			
	Max	0.2	1.7		0.06-0.09	n.a.

3.3.3 - Group B3d, Mycotoxins

The mycotoxins enniatin A, enniatin A1, enniatin B, enniatin B1 and beauvericin were measured in 91 pooled samples of Atlantic salmon, 8 pooled samples of rainbow trout and one pooled sample of turbot. No residues of these mycotoxins were detected in any of the samples.

3.3.4 - Group B3e, Dyes

In addition to 164 samples analysed for residues of dyes as group A (illegal drugs) samples, dyes were measured in 93 pooled group B samples (contaminants; Group B3e, dyes). No residues of malachite green, crystal violet and brilliant green were detected in any of the group B samples.

3.3.5 - Group B3f, Others

The group B3f, others is a group not required for finfish products by Regulation (EU) 2017/625, but are deemed relevant for analyses in Norwegian aquaculture by the NFSA (Mattilsynet) and IMR, because these undesirable compounds are present in the environment and may affect food safety. This group currently consist of brominated flame retardants (BFR), perfluorinated compounds (PFC), polyaromatic hydrocarbons (PAHs), and since 2018 also the technological feed additive ethoxyquin (EQ) and its main transformation product ethoxyquin dimer (EQDM).

3.3.5.1 - Brominated flame retardants

In addition to the PBDEs included in PBDE-7 (PBDE 28, 47, 99, 100, 153, 154, 183), 10 other tri-hepta PBDEs (PBDE 35, 49, 66, 71, 75, 77, 85, 118, 119, 138) and five octa-deca PBDEs (PBDE 196, 197, 206, 207 and 209) were measured. Median values of PBDE-7 were 0.31 µ g/kg w.w. and 0.33 µ g/kg w.w for salmon and rainbow trout, respectively (Table 3.5). The results for all individually measured PBDE congeners are summarized in Table 5.2. Of 69 pooled Atlantic salmon samples and two pooled rainbow trout samples, TBBPA concentrations were detected above LOQ in four samples of salmon. The highest measured TBBPA value was 0.2 µ g/kg w.w.. HBCD was analysed in 69 salmon fillet samples and two rainbow trout fillet samples. The median HBCD concentration for salmon was 0.07 µ g/kg w.w., with a maximum concentration of 0.97 µ g/kg w.w.. There is currently no EU maximum limit for BFRs in food.

Table 3.5 . BFR (µg/kg w.w.) in fillets of farmed fish.

		Atlantic salmon	Rainbow trout	Arctic char	LOQ
	Samples	75	9	1	
UB-Sum PBDE 7	Median	0.31	0.33	-	
	Max	1.4	0.54	0.57	
	Samples	69	2	0	
TBBPA	#Values	4	0		

		Atlantic salmon	Rainbow trout	Arctic char	LOQ
	Median	LOQ	-		
	Max	0.20	LOQ		0.03-0.14
	Samples	69	2	0	
UB-Sum HBCD(α,β,γ)	Median	0.07	-		
	Max	0.97	0.05		

3.3.5.2 - Perfluorinated compounds

In 2020, a total of 72 samples were analysed for the PFCs. All results were below the LOQ (Table 5.3). EU has currently no maximum level for PFC in fish.

3.3.5.3 - Polycyclic aromatic hydrocarbons

PAHs were analysed in 67 samples of salmon, four samples of rainbow trout and one sample of Arctic char. The results for PAH are summarised in Table 3.6. Compared to the previous years, in 2020 elevated levels of PAHs were noted in four out of 72 analyzed fillet samples. However, there is currently no maximum limit for PAH in fresh fish (EU 835/2011).

Table 3.6. PAH (µg/kg w.w.) in fillets of farmed fish.

PAH		Atlantic salmon	Rainbow trout	Arctic char	LOQ
	Samples	67	4	1	
5-methylchrysene	#Values	1	0	0	0.09 - 0.13
	Max	0.2	LOQ	LOQ	
Benz(a)anthracene	#Values	7	1	0	0.09 - 0.13
	Max	2.9	1.2	LOQ	
Benzo(a)pyrene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	
Benzo(b)fluoranthene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	
Benzo(c)fluorine	#Values	4	1	0	0.09 - 0.13
	Max	0.8	0.6	LOQ	
Benzo(ghi)perylene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	
Benzo(j)fluoranthene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	
Benzo(k)fluoranthene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	
Chrysene	#Values	10	1	0	0.09 - 0.13
	Max	5.0	1.9	LOQ	
Cyclopenta(cd)pyrene	#Values	4	2	0	0.09 - 0.13
	Max	0.3	0.3	LOQ	
Dibenz(ah)anthracene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	
Dibenzo(a,e)pyrene	#Values	0	0	0	0.44 – 0.66
	Max	LOQ	LOQ	LOQ	
Dibenzo(a,h)pyrene	#Values	0	0	0	0.44 – 0.66
	Max	LOQ	LOQ	LOQ	
Dibenzo(a,i)pyrene	#Values	0	0	0	0.44 – 0.66
	Max	LOQ	LOQ	LOQ	
Dibenzo(a,l)pyrene	#Values	0	0	0	0.44 – 0.66

PAH		Atlantic salmon	Rainbow trout	Arctic char	LOQ
Dibenzo(a,i)pyrene	Max	LOQ	LOQ	LOQ	
Indeno(1,2,3,-cd)pyrene	#Values	0	0	0	0.09 - 0.13
	Max	LOQ	LOQ	LOQ	

3.3.5.4 - Ethoxyquin

EQ and EQDM levels were measured in a total of 80 samples, mostly taken from Atlantic salmon, but also samples of rainbow trout and a sample of Atlantic halibut were included (Table 3.7). The fillet concentrations of the sum EQ and EQDM were calculated as upper bound (UB), using the numerical LOQ values (0.001 and 0.005 mg/kg ww, respectively) for measurements below LOQ. The number of samples with measurements above LOQ is indicated in Table 3.7 as the number of values.

In 2020, EQDM was found at concentrations above LOQ in 26 out of the 80 samples analyzed (23%), while only one sample (salmon) contained a concentration of EQ above LOQ. The median level of the sum of EQ & EQDM was 0.006 mg/kg ww in salmon. Rainbow trout contained sum EQ & EQDM at a median concentration of 0.01 mg/kg ww. One sample of Atlantic halibut was analysed, which contained 0.03 mg/ kg ww (UB) EQ&EQDM. The maximum values of EQ and EQDM were 0.001 and 0.09 mg/kg ww, respectively, and were found in salmon. There are no MRLs established for EQ or EQDM in fish fillet. The use of EQ as feed additive in animal feed has been phased out since July 2020. Samples taken after the phase out did not contain EQ above LOQ, and the maximum level of EQDM was 0.02 mg/kg ww (salmon).

Table 3.7. Ethoxyquin and ethoxyquin dimer in fillets of farmed fish.

		Atlantic salmon	Rainbow trout	Atlantic halibut	LOQ
	n	76	3	1	
EQ (mg/kg ww)	#Values	1	0	0	
	Median	LOQ	-	-	
	Max	0.001	LOQ	LOQ	0.001
EQDM (mg/kg ww)	#Values	26	2	1	
	Median	LOQ	-	-	
	Max	0.09	0.02	0.03	0.005
Sum EQ&EQDM (mg/kg ww) UB	#Values	76	3	1	
	Median	0.006	0.01	-	
	Max	0.09	0.02	0.03	

4 - Conclusions

No residues of unauthorized substances were detected in any of the samples analysed.

Residues of the anti-sea-lice agent emamectin was detected in one sample of salmon. However, the concentration was well below the MRL for emamectin established for fish.

Consistent with the data gathered in the recent years, no residues of antibiotics, endoparasitic agents were detected in any of the samples.

The contaminant levels in 2020 were comparable to the year before. None of the samples exceeded the EUs maximum levels, where such levels have been established (sum dioxins, sum dioxins and dl-PCBs, PCB-6, mercury, lead and cadmium).

5 - Tables

Table 5.1. Inorganic arsenic and methylmercury in fillets of farmed fish.

		Atlantic salmon	Rainbow trout	LOQ
	n	19	2	
Inorganic arsenic (µg/kg w.w.)	#Values	0	0	
	Median	-	-	
	Max	LOQ	LOQ	2-3
Methyl-mercury (mg Hg/kg w.w.)	#Values	19	2	
	Median	0.020	-	
	Max	0.052	0.037	0.001

Table 5.2. PBDEs (µg/kg w.w.) in fillets of farmed fish.

		Atlantic salmon	Rainbow trout	Arctic char	LOQ
PBDE congener	**Samples**	**75**	**9**	**1**	
PBDE 28	#Values	75	9	1	
	Median	0.011	0.012	-	
	Max	0.039	0.022	0.019	0.002-0.003
PBDE 35	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.003-0.005
PBDE 47	#Values	75	9	1	
	Median	0.18	0.20	-	
	Max	0.74	0.31	0.34	0.014-0.021
PBDE 49	#Values	75	9	1	
	Median	0.049	0.056	-	
	Max	0.210	0.091	0.11	0.003-0.005
PBDE 66	#Values	73	7	1	
	Median	0.006	0.006	-	
	Max	0.030	0.013	0.014	0.002-0.01
PBDE 71	#Values	4	2	0	
	Median	LOQ	LOQ	-	
	Max	0.005	0.004	LOQ	0.002-0.003
PBDE 75	#Values	75	9	1	
	Median	0.010	0.009	-	
	Max	0.044	0.019	0.026	0.002-0.003

		Atlantic salmon	Rainbow trout	Arctic char	LOQ
PBDE 77	#Values	2	0	0	
	Median	LOQ	-	-	
	Max	0.017	LOQ	LOQ	0.007-0.01
PBDE 85	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.003-0.005
PBDE 99	#Values	75	9	1	
	Median	0.026	0.022	-	
	Max	0.140	0.080	0.055	0.007-0.01
PBDE 100	#Values	64	7	1	
	Median (UB)	0.044	0.049	-	
	Max	0.220	0.086	0.089	0.007-0.01
PBDE 118	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.004-0.01
PBDE 119	#Values	7	1	1	
	Median	LOQ	LOQ	-	
	Max	0.012	0.008	0.005	0.002-0.01
PBDE 138	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.003-0.02
PBDE 153	#Values	21	3	1	
	Median	LOQ	LOQ	-	
	Max	0.043	0.017	0.015	0.007-0.01
PBDE 154	#Values	75	9	1	
	Median	0.028	0.027	-	
	Max	0.160	0.059	0.052	0.007-0.01
PBDE 183	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.007-0.01
PBDE 196	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.017-0.026
PBDE 197	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.01-0.016

		Atlantic salmon	Rainbow trout	Arctic char	LOQ
PBDE 206	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.01-0.016
PBDE 207	#Values	0	0	0	
	Median	-	-	-	
	Max	LOQ	LOQ	LOQ	0.01-0.016
PBDE 209	#Values	11	0	0	
	Median	LOQ	-	-	
	Max	0.088	LOQ	LOQ	0.014-0.021

Table 5.3. PFCs (µg/kg w.w.) in fillets of farmed fish

Compound	Atlantic Salmon	Rainbow trout	Arctic char	Max value	LOQ
PFBA	62	9	1	<LOQ	1.0
PFBS					1.0
PFDA					0.2
PFDoDA					0.2
PFDS					0.2
PFHpA					0.2
PFHxA					0.5
PFHxS					1.0
PFNA					0.2
PFOA					0.6
PFOS					0.2
PFOSA					0.5
PFTeDA					0.2
PFTrDA					0.2
PFUdA					0.2
N-EtFOSA					1.5
N-EtFOSE					1
N-MeFOSA					1
N-MeFOSE					0.5

Table 5.4. Summary of analytical methods.

Group of substances	Analyte	Method	LOD (µg/kg w.w.)	LOQ (µg/kg w.w.)	Level of action (µg/kg w.w.)	Laboratory
A1, Stilbenes	Diethylstilbestrol	LC-MS/MS	1		Presence	Eurofins
	Dienestrol		1			
	Hexestrol		1			
	β-Estradiol		1			
	α-Estradiol		1			
	Estriol		1			
	Estrone		1			
	Ethinyl estradiol		1			
A3, Steroids	α-nandrolon	LC-MS/MS	1		Presence	Eurofins
	β-nandrolon		1			
	α-trenbolon		1			
	β-trenbolon		1			
	Trenbolone-acetate		2			
	16-Hydroxy stanozolol		1			
	α -Boldenone		1			
	Boldenone		1			
	Chlor-Testosterone (Clostebol)		1			
	Epitestosterone		1			
	Methyl-Boldenone (Dianabol)		1			
	Methyltestosterone		1			
	Nortestosterone/ Nandrolone		1			
	Stanozolol		1			
	Testosterone		1			
	Testosterone-propionate		2			
A6, Annex IV substances	Chloramphenicol	LC-MS/MS	0.25		Presence (MRPL = 0.3)	IMR
	Metronidazole	LC-MS/MS	0.3		Presence (MRPL = 3.0)	
	Hydroxy-metronidazole		2.0			
	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)	

Group of substances	Analyte	Method	LOD (µg/kg w.w.)	LOQ (µg/kg w.w.)	Level of action (µg/kg w.w.)	Laboratory
	Nitrofuran SEM		0.5		Presence (MRPL= 1.0)	
B1, Antibacterial Substances Micro-biological method[1]	Quinolones	3-plate Screening Method [2]	200		100-600	IMR
	Tetracyclines		200		100	
	Amphenicols		200		1000	
	Sulfonamides		400		100	
B1, Antibacterial substances Chemical method	Oxolinic acid	LC-MS/MS		40	100	IMR
	Flumequine			40	600	
	Enrofloxacin			10	100	
	Ciprofloxacin			10	100	
	Trimethoprim			2	50	
	Oxytetracycline	LC-MS/MS		30	100	Eurofins
	Florfenicol	LC-MS/MS		0.5	1000	IMR
B2a, Anthelmintics	Praziquantel	LC-MS/MS		1	n.a.	IMR/ Eurofins
	Fenbendazole	LC-MS/MS		1	n.a.	
	Emamectin	LC-MS/MS		2-10	100	
	Diflubenzuron	LC-MS/MS		1-10	10	
	Teflubenzuron			1-50	500	
	Hexaflumuron			1-50	500	
	Lufenuron			1-50	1350	
	Ivermectin	LC-MS/MS		2	n.a.	Eurofins
	Cypermethrin	GC-MS		5	50	
	Deltamethrin			10	10	
	Isoeugenol	GC-FID		50	6000	
B3a, Organo-chlorine compounds	Dioxins and dlPCB	HRGC-HRMS		0.0001- 0.1 ng TEQ/kg	6.5 ng TEQ/kg	IMR
	PCB-6	GC-MS GC-MS/MS		0.004 – 0.5	75	
	Pesticides	HRGC-HRMS		0.003-0.8	n.a.	Eurofins
B3b, Organo-phosphorus compounds	Azametiphos	LC-MS/MS		10	n.a.	Eurofins
	Dichlorvos					
B3c, Chemical elements	Lead	ICP-MS		0.005- 0.01 mg/kg	0.3 mg/kg	IMR
	Cadmium			0.001- 0.002 mg/kg	0.05 mg/kg.	
	Arsenic			0.003 mg/kg	n.a.	
	Mercury			0.002 mg/kg	0.5 mg/kg	

Group of substances	Analyte	Method	LOD (µg/kg w.w.)	LOQ (µg/kg w.w.)	Level of action (µg/kg w.w.)	Laboratory
	Inorganic arsenic	LC-ICP-MS		4-6	n.a.	
	Methylmercury	GC-ICP-MS		1	n.a.	
	Tributyltin	GC-ICP-MS		0.3-0.5	n.a.	
B3d, Mycotoxins	Beauvericin, Enniatin A, A1, B and B1	LC-MS/MS		10	n.a.	Eurofins
B3e, Dyes	Malachite green	LC-MS/MS	0.15		Presence (MRPL=2)	IMR
	Leuco malachite green		0.15			
	Crystal violet		0.30		Presence	
	Leuco crystal violet		0.15		Presence	
	Brilliant green		0.15		Presence	
B3f, Others	PBDE	GC-MS		0.002-0.01	n.a.	IMR
	HBCD	LC-MS/MS		0.006-0.01	n.a.	Eurofins
	TBBPA	GC-MS		0.03-0.2	n.a.	Eurofins
	PAH	GC-MS/MS		0.5-1.0	n.a.	IMR
	PFC	LC-MS/MS		0.5-13	n.a.	IMR
	Ethoxyquin	HPLC-FLD		0.001	n.a.	IMR
	Ethoxyquin dimer			0.005	n.a.	

[1] All methods used muscle as sample matrix except for microbiological methods for antibacterial substances (B1), where liver was used. [2] Only screening method, positive results must be confirmed by a chemical method.

Table 5.5. Calculation of sums for certain pesticides.

Sum	Substances included in the sum	Conversion factor
DDT (sum of p,p-DDT, o,p-DDT, p,p-DDD, o,p-DDD, p,p-DDE,and o,p-DDE expressed as DDT)	op-DDT	1
	pp-DDT	1
	op-DDD	1.108
	pp-DDD	1.108
	op-DDE	1.115
	pp-DDE	1.115
Endosulfan (sum of alpha- and beta-isomers and endosulfan-sulphate expressed as endosulfan)	alpha-endosulfan	1
	beta-endosulfan	1
	endosulfan sulphate	0.962
Aldrin and dieldrin (Aldrin and dieldrin combined expressed as dieldrin)	dieldrin	1
	aldrin	1.044

Sum	Substances included in the sum	Conversion factor
Chlordane (Sum of cis- and trans-isomers and oxychlordane expressed as chlordane)	trans-chlordane	1
	cis-chlordane	1
	oxychlordane	0.967
Heptachlor (sum of heptachlor and heptachlor epoxide expressed as heptachlor)	heptachlor	1
	trans-heptachlor epoxide	0.959
	cis-heptachlor epoxide	0.959
Toxaphene (sum of toxaphene 26, toxaphene 50 and toxaphene 62)	Toxaphene 26	1
	Toxaphene 50	1
	Toxaphene 62	1

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