Summary Report on 2019 Residue Monitoring of Irish Farmed Finfish
&
2019 Border Inspection Post Fishery Product Testing undertaken at the Marine Institute

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Foras na Mara
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Marine Institute

Rinville, Oranmore, County Galway

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The MI scope of accreditation for analysis in this report is detailed in Appendix 2

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PART A

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Part A

Summary Report on 2019 Residue Monitoring of Farmed Finfish

***Carried out under Council Directive 96/23/EC of 29 April 1996 on measures to monitor certain substances and residues thereof in live animals and animal products*.**

1. 2019 OVERALL SUMMARY

In 2019, in excess of 912 tests and a total of 2,601 measurements were carried out on 176 samples of farmed finfish for a range of residues. Implementation of the Aquaculture 2019 Plan involves taking samples at both farm and processing plant:

- 118 target samples taken at harvest: 105 farmed salmon and 13 freshwater trout.
- 58 target samples were taken at other stages of production: 50 salmon smolts and 8 freshwater trout.

All 2019 samples were compliant. For target sampling of farmed fish, a summary table of the residue results from 2005 - 2019 is outlined in Table 1. Overall, the outcome for aquaculture remains one of consistently low occurrence of residues in farmed finfish, with no non-compliant target residues results for the period 2006-2014, 0.11% and 0.10% non-compliant target residues results in 2015 and 2016 respectively and no non-compliant target results for the period 2017 to 2019.

Table 1: Summary Target Results for Residue program 2005-2019

Year	No. of Target Samples[1]	Total Group A[2]	Total Group B[2]	No. of Results[3] /non-compliant	Non-Compliant Results (%)
2005	164 (105 , 59)	163/0	164/0	2251/2	**0.09**
2006	162 (104 , 58)	162/0	162/0	2207/0	**0**
2007	161 (103 , 58)	148/0	161/0	2219/0	**0**
2008	162 (103 , 59)	144/0	162/0	2073/0	**0**
2009	146 (98 , 48)	128/0	146/0	1750/0	**0**
2010	141 (92 , 49)	109/0	141/0	1569/0	**0**
2011	140 (92 , 48)	105/0	140/0	1566/0	**0**
2012	169 (112 , 57)	101/0	169/0	1596/0	**0**
2013	137 (91 , 48)	83/0	137/0	1494/0	**0**
2014	136 (91 , 45)	83/0	136/0	1882/0	**0**
2015	124 (91 , 33)	71/0	124/2	1841/2	**0.11**
2016	126 (92 , 34)	65/0	126/2	1933/2	**0.10**
2017	141 (103 , 38)	72/0	141/0	2250/0	**0**
2018	171 (123 , 48)	108/0	171/0	2611/0	**0**
2019	176 (118 , 58)	101/0	176/0	2601/0	**0**

[1]Target samples (sampled at harvest, sampled at other stages of production)
[2] No. of samples tested/No. of samples non-compliant
[3]Total no. of results as target samples taken for Group A and Group B substances are tested for multiple residue categories within each group/No. of non-compliant results

2. BACKGROUND

As with other farmed animals, farmed finfish can be subject to disease and infestation which can have animal welfare, environmental and commercial implications. Therefore, authorised veterinary medicines and treatments may be used, and sometimes must be used, to control disease and infestation as part of health control plans e.g. antibacterial and antiparasitic treatments. The National Residues Control Plan (NRCP) sets out the monitoring requirements for residues in animal products in accordance with Council Directive 96/23/EC of 29 April 1996 *on measures to monitor certain substances and residues thereof in animals and animal products.*

On behalf of the Department of Agriculture, Food and Marine (DAFM), the Marine Institute carries out monitoring of chemical residues for aquaculture. The main objectives of the NRCP for Aquaculture are to ensure farmed fish are fit for human consumption, to provide a body of data showing that Irish farmed fish is of high quality, to promote good practices in aquaculture and to comply with EU Directive 96/23/EC.

The Food Safety Authority of Ireland (FSAI) co-ordinates the activities of the various departments and agencies involved in delivering this programme. For the aquaculture sector, the Sea Fisheries Protection Authority (SFPA) with technical support from the Marine Institute is responsible for residue controls on farmed finfish to ensure compliance with the Residue Directive (96/23/EC). A summary of each department and agencies' role with respect to the NRCP is outlined in Table 2.

Table 2: Department and Agency Roles

Department of Agriculture Food and Marine (DAFM) - Implements the overall residues controls in Ireland
Food Safety Authority of Ireland (FSAI) - Coordinates the activities of the departments and agencies involved
Sea Fisheries Protection Authority (SFPA) - Ensures compliance with the Directive for finfish aquaculture
Marine Institute - Implements the surveillance monitoring programme for farmed fish and is the official laboratory for residue sampling and analysis. The MI is National Reference Laboratory (NRL) for a number of substances in aquaculture
DAFM Veterinary Inspectors - Carry out routine on-farm inspections to verify compliance with various regulations including fish health, animal remedies, feedstuffs, etc

2.1 National Residue Control Plan (NRCP)

Annually, the Marine Institute (MI) prepares the NRCP for Aquaculture, which is reviewed and finalised by SFPA, FSAI and DAFM. The NRCP once agreed is then submitted to the European Commission (EC) for approval, this sets out the monitoring plan, including species, sample numbers and target substances in line with the specific requirements of the Directive. The national legal basis for the Residue Monitoring Plan is provided for in the Animal Remedies Act, 1993 and other relevant legislation in particular, the Control of Animal Remedies and their Residues Regulations, 2009. Figure 1 illustrates the National Aquaculture Residue Control Cycle. The 2019 NRCP is available in Appendix 5

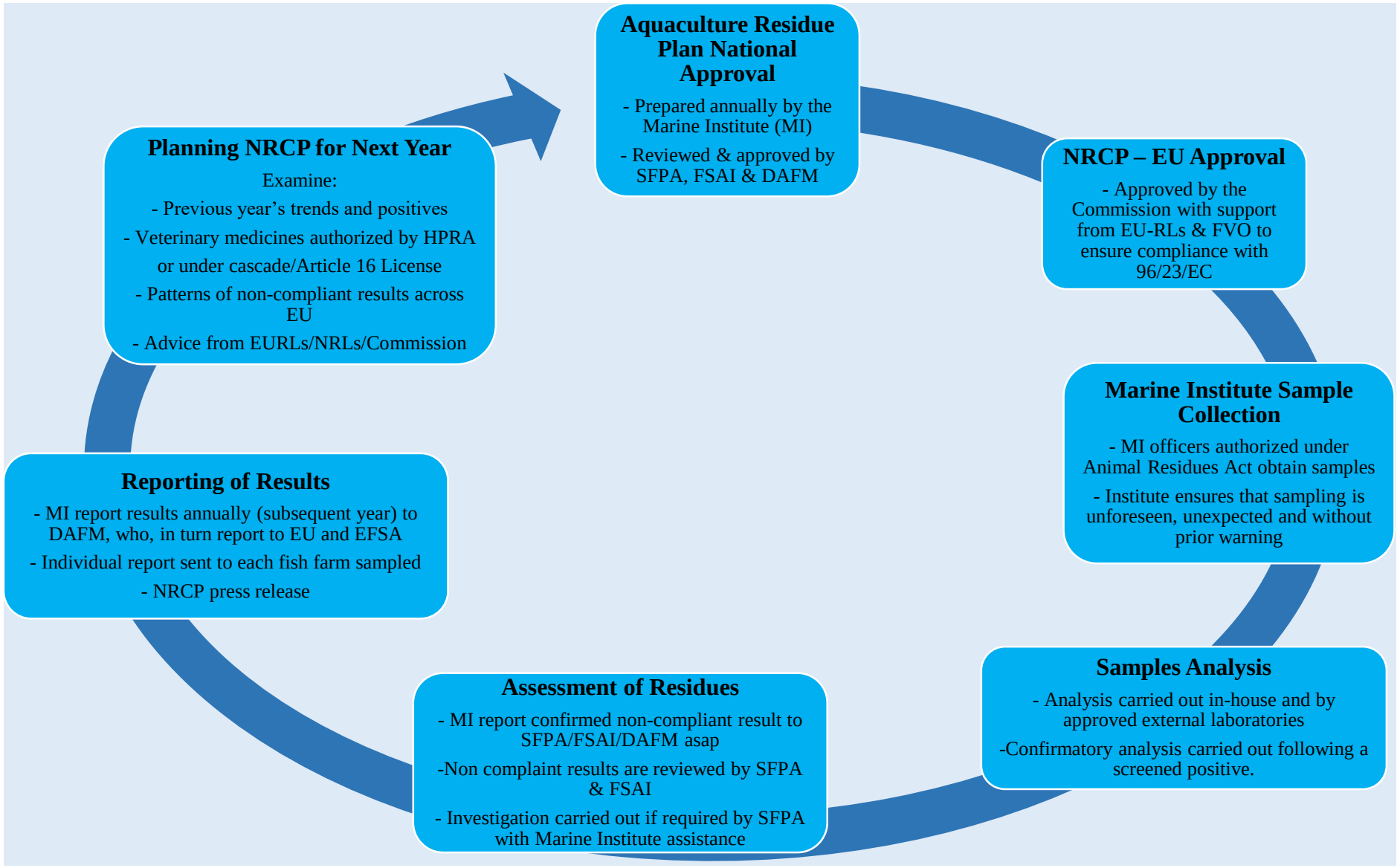

Figure 1: National Aquaculture Residue Annual Control Cycle

2.2 Scope of NRCP

The scope of this testing under the NRCP is comprehensive covering the following broad categories outlined in Table 3.

Table 3: NRCP testing categories

Category	Details
Banned	These compounds should **not** be present as no safe limit can be set for their residue e.g. steroids, chloramphenicol, nitroimidazoles
Authorised	Authorised medicines which may be used in aquaculture and should be **below statutory limit** (i.e. Maximum Residue Limit – MRL*) e.g. Sea lice treatments- emamectin, deltamethrin
Unauthorised	These compounds should **not** be present as these treatments should **not be used** in aquaculture. e.g. malachite green
Environmental contaminants	Certain contaminants occur naturally in the environment but they may also be introduced inadvertently and may accumulate in fish e.g. polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), heavy metals

*MRL = maximum concentration allowable in the edible portion of the animal which should not be exceeded at the time of harvest.

These substances are classed into 2 categories: Group A and Group B. Details are given in Table 4.

Table 4: List of substances included in the NRCP for farmed finfish

Group A–Substances having an anabolic effect	
A3	Steroids
A6	Compounds included in Annex IV of Council Regulation 2377/90/EC
Group B- Veterinary drugs and contaminants	
B1	Antimicrobials (Antibacterial)
B2a	Anthelminthics (Antiparasitic)
B2c	Pyrethroids
B2f	Other pharmacologically active substances
B3a	Organochlorine compounds
B3c	Chemical elements
B3d	Mycotoxins
B3e	Dyes

Group A:

Group A substances are banned substances and should not be present in farmed finfish. These can be categorised as the following:

- A3 steroids, 17β-oestradiol and methyltestosterone which occur naturally but also could be used for growth promotion.
- A6 compounds, nitrofurans and nitroimidazole which are antibacterial drugs, and chloramphenicol a broad spectrum antibiotic.

Group B:

Group B substances can be categorised into unauthorised substances, authorised substances and environmental contaminants. Farmed finfish can be subject to disease and infestation. which can have animal welfare, environmental and commercial implications. Therefore, similar procedures are in place for farmed finfish as for other farmed animals which may involve treatment with approved veterinary medicines such as antibiotics or anthelminthics to prevent or treat disease or infestation e.g. antibacterial agents, antifungal agents, antiparasitic treatments. Farmed finfish can also accumulate trace metals and persistent organic pollutants from their feed or the environment; therefore, levels of these contaminants are also determined.

2.3 EFSA Reporting

Annually, the Marine Institute (MI) reports the NRCP results for Aquaculture (subsequent year) to DAFM who, in turn reported to the EU and EFSA in the required EFSA format. The overall 2019 results were reported to DAFM in the EFSA format in May 2020. This reports examines the 2019 residue results for aquaculture in more detail.

3. SAMPLING

In 2019, samples were taken in accordance with Council Directive 96/23/EC by Marine Institute Authorised Sampling Officers (Authorised under the Animal Remedies Act 1993). The Institute ensures that sampling is unforeseen, unexpected and without prior warning in accordance with Article 3 of Regulation 882/2004 and Article 12 of Council Directive 96/23/EC and a strict chain of custody is maintained. Samples are taken throughout the year in an effort to spread sampling across different sites and are taken in accordance with the NRCP i.e.

- One third of the samples are taken 'on farm' at the smolt stage which is aimed at detection of illegal treatment (prohibited substances Group A and unauthorised substances Group B3 (e) - Dyes).

- Two thirds of the samples are taken at harvest stage which is aimed at controlling the compliance with the Maximum Residue Limits (MRL) and for detection of illegal treatment (prohibited substances Group A and unauthorised substances-e.g. Group B3 (e) - Dyes). These harvest samples are taken primarily at processing plants for salmon and 'on farm' for freshwater trout.

In 2019, a total of 176* target (surveillance) samples were taken from fish farms and processing plants in accordance with the NRCP for Aquaculture 2019 (Appendix 5).

- 58 target samples were taken at other stages of production (OSOP); 50 salmon smolts and 8 freshwater trout were collected from 12 farms for Group A substances and malachite green.

- 118 target samples were taken at harvest which comprised of 105 farmed salmon and 13 freshwater trout. These harvest samples were collected during 20 sampling events (samples collected from a given site at a given time) throughout the year. Salmon were collected on 17 occasions and freshwater trout on 3 occasions. In 2019 no sea reared trout samples were taken. Samples were collected from the same producers on a number of occasions due to the small number of active harvest sites in the given year.

  *Note: The 2019 plan indicated that a target of 190 samples to be taken. However, there was a shortfall of 14 samples with only 176 target samples taken. This was due to a decrease in production for aquaculture industry in 2018 and 2019 which was not reflective in 2019 plan as the plan was prepared at the start of the year using the 2017 aquaculture production data which was the most recent data available at the time. This subsequently lead to difficulties obtaining samples at the Processing plants due to sample unavailability towards end of the year.

Generally, 5 fish were taken from each producer and each individual fish was treated as a sample. However, where an individual fish was not large enough to provide sufficient test material, a number of fish were pooled to provide a sample. Samples were further subsampled as multiple tests were typically performed on individual samples.

4. RESULTS OF ANALYSIS

4.1 Interpretation of Results

Samples are tested for a broad range of substances using a variety of modern analytical techniques. The scope of testing under the Aquaculture Plan is comprehensive covering four broad categories: banned substances, unauthorised substances, authorised substances (approved substances i.e. veterinary substances) and environmental contaminants. Details of the methods and subcontract laboratories used are provided in Appendix 4.

Where a Maximum Residue Limit (MRL) has been set, samples are deemed non-compliant (i.e. positive) if concentrations of a given residue are confirmed to be in excess of the MRL.

Where no MRL is set, {e.g. for banned substances including steroids and compounds listed in Commission Regulation (EU) No 37/2010 (Table 4) and for unauthorized substances}, a Decision Limit (action level) is used. Samples are deemed non-compliant if concentrations of a given residue are confirmed to be in excess of the Decision limit (action level).

Follow up action is taken on confirmed positive samples. The sources of MRLs and Decision Limits (action level) are specified in Appendix 1.

Organochlorine compounds including Polychlorinated Biphenyls (PCBs) are persistent environmental contaminants that accumulate in lipid-rich animal tissue. For PCBs, typically, a group of indicator congeners are measured "EFSA PCB 6" which is the sum of the following 6 CB congeners – PCB 28, 52, 101, 138, 153, 180 and the Commission have set a Maximum Level (ML) of 75 µg kg^{-1} wet weight. For Organochlorine Pesticides (OCPs) there are no MRL/MLs; however, a number of OSPAR contracting countries have set levels that are presented in this report (Appendix 1).

Maximum levels for mercury, cadmium and lead in fisheries products are set out in Commission Regulation (EC) No 1881/2006 as amended *setting maximum levels for certain contaminants in foodstuffs*. For salmon and trout, the levels specified are 0.3 mg kg^{-1} for lead, 0.05 mg kg^{-1} for cadmium and 0.5 mg kg^{-1} for mercury. These are taken as the "action levels" for the following report.

A comprehensive quality assurance programme supports the monitoring programme and is detailed in Appendix 2 and 3.

4.2 Breakdown of 2019 Results

In 2019, in excess of 912 tests and a total of 2,601 measurements were carried out on 176 target samples of farmed finfish. **All 2019 samples were compliant.**

Table 5: Summary of 2019 residue monitoring results for target farmed fish samples (salmon and trout). All tests performed on muscle and skin.

RESIDUE	NUMBER TESTED	NON-COMPLIANT[1]	DETECTION LIMIT[2] (μg kg $^{-1}$)
Group A3 – Steroids			
Methyltestosterone	63	0	1.5
17β-oestradiol	24	0	1.5
Group A6 - Compounds included in Annex IV of Council Regulation 2377/90/EC			
Chloramphenicol	63	0	0.25
Nitrofurans	10	0	*See Appendix 5 for cc alphas*
Nitroimidazoles	10	0	*See Appendix 5 for cc alphas*
Group B1 - Antibacterial Substances			
Tetracyclines: **Oxytetracycline**	118	0	100 (screening)
Quinolones: **Oxolinic acid** **Flumequine**	118	0	75(screening) 150(screening)
Florfenicol	118	0	750(screening)
Sulphonamides: **Sulphadiazine**	118	0	50(screening)
Group B2a – Anthelmintics			
Emamectin B1a	113	0	9.0
Ivermectin	113	0	0.1
Doramectin	113	0	0.1
Group B2c – Pyrethroids			
Cypermethrin	113	0	5
Deltamethrin	113	0	2
Group B2f - Other pharmacologically active substances			
Corticosteroids	29	0	1.5
Teflubenzuron	113	0	80
Diflubenzuron	113	0	86
Group B3a- Organochlorine Compounds			
EFSA sum of 6 CBs	19	0	0.12
DDT and metabolites[5]	10	0	0.0498
α-HCH	10	0	0.02
β-HCH	10	0	0.02
γ-HCH (lindane)	10	0	0.02
δ –HCH	10	0	0.02
Hexachlorobenzene	10	0	0.04
Pentachlorobenzene	10	0	0.17
Aldrin + dieldrin[6]	10	0	0.026
Endrin	10	0	0.08

Table 5 (continued): Summary of 2019 residue monitoring results for target farmed fish samples (salmon and trout). All tests performed on muscle and skin.

RESIDUE	NUMBER TESTED	NON-COMPLIANT[1]	DETECTION LIMIT[2] (μg kg $^{-1}$)
Group B3a- Organochlorine Compounds			
Toxaphene 26	10	0	0.06
Toxaphene 50	10	0	0.29
Toxaphene 62	10	0	0.19
Heptachlor	10	0	0.014
Mirex	10	0	0.008
***cis*-heptachlorepoxide**	10	0	0.012
***trans*-heptachlorepoxide**	10	0	0.06
Octachlorostyrene	10	0	0.004
***trans*-nonachlor**	10	0	0.004
Oxychlordane	10	0	0.1
***trans*-chlordane (γ- chlordane)**	10	0	0.033
***cis*-chlordane (α-chlordane)**	10	0	0.008
Group B3c – Chemical Elements[7]			
Lead	10	0	7
Cadmium	10	0	1
Mercury	10	0	2
Group B3d – Mycotoxins			
Aflatoxins	6	0	0.006
Group B3e – Dyes			
Malachite Green	94	0	0.5
Leuco Malachite Green	94	0	0.5
Crystal Violet	94	0	0.5
Leuco Crystal Violet	94	0	0.5
Victoria Blue	94	0	0.5
Brilliant Green	94	0	0.5
Other – Non-NRCP Testing			

[1] Action limits to evaluate non-compliant results in Appendix 1
[2] Limit of Detection (LOD) for organochlorine compounds are averages as LOD is sample dependent.
[4] EFSA PCB 6: sum of the following 6 non dioxin like PCBS–PCB 28, 52, 101, 138, 153, 180. Commission Regulation No 1259/2011 (came into force 1st Jan 2012) amending Regulation No. 1881/2006 *setting maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs.*
[5] DDT and metabolites – sum of individual DDT metabolites (o,p'DDT, p,p' DDT, o,p'DDE, p,p' DDE o,p'DDD, and p,p' DDE) – sum of individual LODs also included.
[6] Aldrin + dieldrin sum - sum of individual LODs also included.
[7]For additional metals tested in 2019 refer to Table 6 for details and Appendix 2 for accreditation status; no maximum limit or guidance levels for these additional metals are set for fish.

4.2.1 Group A – Banned Substances

A total of 101 samples (other stage of production and harvest) were tested for at least one Group A compound.

Group A3: Steroids

77 individual samples were tested by the Irish Equine Centre (IEC) for Group A3 Steroids:

- **Methyltestosterone –** 63 samples were screened for methyltestosterone by Enzyme-Linked Immuno Sorbant Assay (ELISA) method.
- **17β-oestradiol –** 14 samples were screened for 17β-oestradiol by ELISA method.

No non-compliant (i.e. no positive) results were reported for Group A3 compounds. Although two samples from two farms gave a screening reading above the screening cut-off for 17β-oestradiol, these samples were found to be **compliant** when further quantitative confirmatory GCMSMS analysis by EURL (RIKILT) was carried out and no further action was required.

Group A6: Compounds included in Annex IV of Council Regulation 2377/90/EC

71 individual samples were tested for Group A6 Compounds.

- **Chloramphenicol –** 63 samples were screened for chloramphenicol by IEC laboratory using an ELISA method.
- **Nitrofurans –** 10 samples were analysed by Teagasc Food Research Centre (TFRC) for the marker metabolites of the nitrofurans; furazolidone, furaltadone, nitrofurantoin and nitrofurazone using a quantitative (LCMSMS) method.
- **Nitroimidazole** – 10 samples analysed by TFRC for nitroimidazole and its metabolites[1] by a quantitative (LCMSMS) method.

No non-compliant (i.e. no positive) results were reported for Group A6 compounds.

4.2.2 Group B – Veterinary Drugs and Contaminants

A total of 176 samples of farmed finfish were tested for Group B compounds which can be classed as authorised substances, unauthorised substances or environmental contaminants.

No non-compliant (i.e. no positive) results were reported for Group B compounds.

Group B1: Antibacterial Substances

- **Sulphonamides –** 118 samples were screened for sulphonamides by the Marine Institute (MI) using an Immunoassay method (Randox Evidence investigator).

[1] The following nitroimidazole metabolites are listed on the NRCP-dimetridazol, ronidazol, metronidazol, hydroxyl-dimetridazol, hydroxyl-metronidazol

No non-compliant (i.e. no positive) results were obtained for sulphonamides.

- **Quinolones, tetracyclines, florfenicol –** 118 samples were analysed by the MI for the following antibacterial substances quinolones, tetracyclines and florfenicol using a qualitative screening method (modified two plate test).

No non-compliant (i.e. no positive) results were obtained for quinolones, tetracyclines or florfenicol

Group B2: Other veterinary drugs

With the exception of corticosteroids, these are authorised and unauthorised substances that could be used in treating sea-lice infestation.

- **B2(a) Anthelmintics (**Ivermectin, emamectin B1a, doramectin) - 113 harvest samples were analysed for the above anthelmintics using UPLC-FLU in the MI. **No non-compliant results were obtained.**

- **B2(c) Pyrethroids (**Cypermethrin, deltamethrin) – 113 harvest samples were analysed for the above pyrethroids using a GC-MS screening method in the MI. **No non-compliant results were obtained for cypermethrin and deltamethrin,** however one sample from one farm required confirmatory testing for Deltamethin to be carried out on. Therefore, all 5 samples from this farm were sent for confirmatory GCMSMS analysis to FERA, UK and all were found to be compliant -**no further action was required**.

- **B2(f) Other pharmacologically active substances**

Teflubenzuron, diflubenzuron – 113 harvest samples were analysed by the MI for teflubenzuron, diflubenzuron using UPLC-DAD. **No non-compliant results were obtained.**

Corticosteroids (dexamethasone, flumethasone and betamethasone) – 29 samples (other stage of production and harvest) were screened by the IEC for the above corticosteroids using the ELISA method. **No non-compliant results were obtained for corticosteroids.**

Group B3a: Organochlorine Compounds

- **Polychlorinated Biphenyls**

Polychlorinated Biphenyls are a group of homologous man-made substances with a molecular structure comprising of a chlorinated biphenyl ring. PCBs are persistent environmental contaminants that accumulate in lipid and can be present at levels of concern in fish. PCBs can be divided into groups according to their toxicological properties e.g. dioxin-like PCBs, non-dioxin-like PCBs. As part of the NRCP, it is primarily the following six non dioxin-like PCBs

(NDL-PCB) which are monitored; PCB 28, 52, 101, 138, 153 and 180 and analysed by Eurofins. These NDL-PCBs are routinely used as a monitoring indicator as they are generally presumed to be the most persistent in fish tissue and comprise about half of the amount of total PCB present in feed and food. European legislation (Commission Regulation (EU) No 1259/2011 amending Regulation (EC) 1881/2006) has fixed maximum levels for dioxins, dioxin-like PCBs and non-dioxin-like PCBs in foodstuffs. In the case of NDL-PCBs the maximum level of 75 μg kg $^{-1}$ wet weight has been set for the sum of these six congeners. The mean and maximum concentrations measured for the sum of 6 indicator PCBs was 7.07 and 10.5 μg kg $^{-1}$ wet weight respectively (Table 6).

None of the 19 harvest samples analysed exceeded the standard for the sum of 6 PCBs (Table 6 provides details of number of samples tested and the concentration range).

Organochlorine pesticides

Organochlorine pesticides are synthetic substances used for pest control that are persistent and widespread in the marine environment despite the fact that their use has largely been phased out over recent decades. A number of OCPs are included in residues testing including DDT and its breakdown products. Chlorinated pesticides behave similarly to PCBs in the environment and do not have maximum concentrations in fish set by the EC. Due to their chemical properties (fat solubility) these substances bio-accumulate in fish tissue and also bio-magnify through the marine food chain. A number of OSPAR contracting countries have set standards/guidance values for certain OCPs and Appendix 1 presents the strictest of these in so far as Marine Institute is aware.

All the harvest samples (10 samples) analysed by Eurofins for chlorinated pesticides were below these levels and were reported as compliant.

Group B3c: Chemical elements

Levels of mercury, cadmium and lead were all very low and well below the relevant European maximum limits in all of the samples tested (Appendix 1) by the MI. Mercury has a maximum limit set in fish of 0.5 mg kg^{-1} wet weight. The highest mercury concentration obtained for the 10 samples analysed was 0.06 mg kg^{-1} wet weight. Cadmium, also an environmental contaminant, has a maximum limit set in fish of 0.05 mg kg^{-1} wet weight and cadmium was not detected above 0.003 mg kg^{-1} wet weight. Lead has a maximum limit set in fish of 0.3 mg kg^{-1} wet weight. The highest lead concentration obtained for the 10 samples analysed was 0.03 mg kg^{-1} wet weight. Table 6 provides a breakdown of the number of samples tested and the concentration range for the samples tested. **All 10 harvest samples were reported as compliant for mercury, lead and cadmium.**

In addition, in 2019 the following metals were analysed (arsenic, chromium, copper, nickel, silver, zinc, aluminium, cobalt, iron, manganese, selenium and vanadium) by the MI (Note: aluminium, cobalt, iron, manganese, selenium and vanadium are internally validated but are not accredited). Table 6 provides details of a number of samples analysed and the concentration range of these metals in samples. At present for these metals there is **no maximum limit or guidance levels set for fish.**

Table 6: Trace metal (mg kg^{-1}) and PCB (µg kg^{-1}) concentrations and maximum limits

Parameter	Median / *Mean*	Range	EC Max Limit	Number Tested
Mercury	0.04/ *0.04*	<0.007 – 0.06	0.5	10
Cadmium	nd (<0.001)	nd (<0.001) – 0.003	0.05	10
Lead	nd (<0.007)	nd (<0.007) - 0.03	0.3	10
EFSA PCB 6[1]	7.55/ *7.07*	0.12 – 10.5	75	19
Other metals				
Arsenic[2]	1.33 / 1.42	0.85 – 2.38	-	10
Chromium[2]	0.02 / 0.03	nd (<0.008) -0.14	-	10
Copper[2]	0.41 / 0.43	0.25 – 0.78	-	10
Nickel[2]	0.06 / 0.09	<0.03 – 0.32	-	10
Silver[2]	<0.001	nd (<0.0003)- <0.001	-	10
Zinc[2]	4.31 / 4.70	3.53 – 6.69	-	10
Aluminium[3]	nd (<0.62)	nd (<0.62) – 1.55	-	10
Cobalt[3]	0.008 / 0.007	0.003 – 0.01	-	10
Iron[3]	2.76 / 2.73	2.17 – 3.24	-	10
Manganese[3]	0.21 / 0.23	0.12 - 0.39	-	10
Selenium[3]	0.28 / 0.27	0.17 – 0.35	-	10
Vanadium[3]	0.003 / 0.004	0.003 – 0.01	-	10

For values reported as "nd", substances were not detected above the Limit of Detection (LOD is given in brackets)
[1]EFSA PCB 6: sum of the following non-dioxin like PCBS-PCB 28, 52, 101, 138, 153, 180
[2]For additional metals tested in 2019 reference Appendix 2 for accreditation status; no maximum limit or guidance levels for these additional metals are set for fish.
[3]Internally validated-not accredited; no maximum limit or guidance levels for these additional metals are set for fish.

Group B3d: Mycotoxins

A mycotoxin is a toxic by-product of mould growth in feed and can remain as a residue in meat tissue. The amount and type of mycotoxin varies with environmental conditions such as temperature and humidity.

The NRCP for Aquaculture 2019 analysed for the following mycotoxins: aflatoxin B1, aflatoxin B2, aflatoxin G1 and aflatoxin G2. Aflatoxin B1 is the most common in food and amongst the most potent genotoxic and carcinogenic aflatoxin. All aflatoxins were reported as <0.01 μg kg $^{-1}$ (wet weight) in the 6 samples tested by Wessling.

Currently there are no maximum limits set for aflatoxins in fish.

Group B3e: Dyes

The following triphenylmethane dyes are analysed in the MI as part of Group B3e substances, malachite green and its metabolite leuco malachite green, brilliant green, crystal violet, leuco crystal violet, and victoria blue. These dyes could be used illegally in aquaculture as they exhibit antimicrobial and antiparasitic properties. Malachite green is a common commercial fabric dye which had been widely used both prophylactically and in the treatment of fungal infection of both fish and eggs for over 60 years. It is also effective against several protozoal infestations, including agents causing proliferative kidney disease (PKD) and ichthyophthiriosis (white dot disease). Malachite green was regularly detected in aquaculture samples during the early years of the residues monitoring but as a result of increased industry awareness of its status as an unauthorised substance, supported by monitoring and enforcement, the use of malachite green has ceased with no non-compliant results reported since 2004. Its use had been primarily associated with freshwater farms and hatcheries; therefore, freshwater sites are particularly targeted by the NRCP. Malachite green is possibly both carcinogenic and genotoxic (i.e. damaging to DNA).

A minimum required performance level (MRPL) has been set for the sum of malachite green and its metabolite leuco malachite green[2] at 2 μg kg $^{-1}$ and the MI has set a decision limit of 0.5 μg kg $^{-1}$ for malachite green and leuco malachite green individually i.e. a sample is deemed non-compliant if detected above the decision limit of 0.5 μg kg $^{-1}$. There has been no evidence of brilliant green, crystal violet, leuco crystal violet, victoria blue being used in aquaculture in Ireland; however, these dyes have the potential to be used to treat Saprolegnia (fungus) either when present on the fish or as a prophylactic treatment to protect fish eggs from infection. No MRPL has been set for brilliant green, crystal violet, leuco crystal violet, victoria blue. However as these dyes are unauthorised a decision limit of 0.5 μg kg $^{-1}$ has been set for all dyes.

All 94 target samples (i.e. 36 harvest and 58 other stage of production) tested for malachite green and its metabolite leuco malachite green, crystal violet and its metabolite leuco crystal violet, brilliant green, victoria blue were found to be compliant i.e. negative.

[2] The MRPL of 2μg kg $^{-1}$ was reaffirmed by EFSA in 2016 https://www.efsa.europa.eu/de/efsajournal/pub/4530

PART B

Summary Report on 2019 Border Control Posts Product Testing undertaken at the Marine Institute

Carried out under Council Directive 97/78/EC of 18 December 1997 laying down the principles governing the organisation of veterinary checks on products entering the Community from third countries
&
Commission Regulation (EC) No 136/2004 of 22 January 2004 laying down procedures for veterinary checks at Community border control posts on products imported from third countries

Third Countries (non-EU) wishing to export animal products to the EU are required to satisfy the European Commission that their residue surveillance measures provide equivalent guarantees for EU consumers similar to EU residue surveillance 96/23/EC. Therefore, food imports of animal origin from a Third country may only be brought into the European Community through a Border Control Post (BCP) that has been approved for importation. In Ireland, the responsibility for carrying out checks at the BCP (Dublin Port and Shannon Airport) is with the DAFM BCP Officers.

In 2019, BCP samples were collected by DAFM Sampling Officers and samples for testing of antibacterials (B1a), anthelminthics (B2a), heavy metals (B3d) and dyes (B3e) were sent to the Marine Institute for testing in accordance with 2019 BCP plan (Appendix 6). In total 16 random samples were sent to the Institute by the DAFM Sampling Officers at Dublin Port and Shannon Airport. This was an increase of 5 samples compared to 2018.The 2019 BCP results as tested at the Marine Institute are presented in Table 7. **All 16 random samples were reported as compliant.**

In addition, Safeguard samples (Safeguard 2016/1774/EC) were received from DAFM, consisting of 11 shrimp samples for tetracyclines under Commission Decision 2010/381/EU '*on emergency measures applicable to consignments of aquaculture products imported from India and intended for human consumption*' and its amendment Commission Implementing Decision 2012/690/EU. Results are presented in Table 8. **All 11 safeguard samples were reported as compliant**.

Table 7: 2019 Border Control Posts results for seafood samples tested at Marine Institute

MI CODE	DAFM Sample code	BCP Office	Product type	Substances for Identification	Result
RESBIP2019/5004	DPP2019/0025	Dublin Port	Frozen Shrimp	Malachite Green	**Compliant**
RESBIP2019/5005	DPP2019/0258	Dublin Port	Frozen Cod	Cadmium	**Compliant**
RESBIP2019/5006	DPP2019/0258	Dublin Port	Frozen Cod	Mercury	**Compliant**
RESBIP2019/5007	DPP2018/11048	Dublin Port	Canned Tuna	Avermectins	**Compliant**
RESBIP2019/5008	DPP2019/0025	Dublin Port	Frozen Shrimp	Avermectins	**Compliant**
RESBIP2019/5013	DPP2019/0772	Dublin Port	Canned Tuna	Mercury	**Compliant**
RESBIP2019/5018	19/913/JG/AOC	Shannon Airport	Live Lobster	Lead, Mercury, Cadmium	**Compliant**
RESBIP2019/5019	DPP2019/1188	Dublin Port	Frozen Shrimp	Dyes	**Compliant**
RESBIP2019/5020	DPP2019/1188	Dublin Port	Frozen Shrimp	Antibiotics	**Compliant**
RESBIP2019/5021	DPP2019/1205	Dublin Port	Frozen Shrimp	Antibiotics	**Compliant**
RESBIP2019/5022	DPP2019/1205	Dublin Port	Frozen Shrimp	Dyes	**Compliant**
RESBIP2019//5023	DPP2019/1278	Dublin Port	Frozen Shrimp	Dyes	**Compliant**
RESBIP2019/5024	DPP2019/1278	Dublin Port	Frozen Shrimp	Antibiotics	**Compliant**
RESBIP2019/5025	DPP2019/1224	Dublin Port	Frozen Shrimp	Antibiotics	**Compliant**
RESBIP2019/5026	DPP2019/1245	Dublin Port	Frozen Shrimp	Mercury	**Compliant**
RESBIP2019/5029	19/1249/JON/AOC	Shannon Airport	Live Lobster	Lead, Mercury, Cadmium	**Compliant**

[1] Antibacterials – Agar Plate Method (tetracyclines, florfenicol and quinolones) and Evidence Investigator (sulphonamides)

Table 8: 2019 Safeguard results for fishery products tested at Marine Institute

MI CODE	DAFM Sample code	BCP Office	Product type	Substances for Identification	Result
RESBIP2019/5002	DPP2019/0249	Dublin Port	Farmed Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5009	DPP2019/0440	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5010	DPP2019/0506	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5011	DPP2019/0607	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5012	DPP2019/0750	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5014	DPP2019/0901	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5015	DPP2019/1152	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5016	DPP2019/1182	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5017	DPP2019/1223	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5027	DPP2019/1453	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**
RESBIP2019/5028	DPP2019/1507	Dublin Port	Frozen Shrimp	Tetracyclines	**Compliant**

Appendix 1: Source of Maximum Residues Limits, Decision Limits and Guideline Values used for comparison with the results for 2019

Parameter	Maximum Level or Decision Limit [6]	Source
Group A Compounds[1]: Methyltestosterone, 17β-Oestradiol, Chloramphenicol, Nitrofurans & Nitroimidazoles	These are banned substances and should not be detected.	
Ivermectin	1 μg kg^{-1}	Decision Limit[3]
Doramectin	1 μg kg^{-1}	Decision Limit[3]
Emamectin B1a	100 μg kg^{-1}	Maximum Residue Limit[2]
Cypermethrin	50 μg kg^{-1}	Maximum Residue Limit[2]
Deltamethrin	10 μg kg^{-1}	Maximum Residue Limit[2]
Teflubenzuron	500 μg kg^{-1}	Maximum Residue Limit[2]
Diflubenzuron	1000 μg kg^{-1}	Maximum Residue Limit[2]
Antibacterial Substances		
Sulphonamides	100 μg kg^{-1}	Maximum Residue Limit[2]
Oxytetracycline (Tetracyclines)	100 μg kg^{-1}	Maximum Residue Limit[2]
Oxolinic Acid (Quinolones)	100 μg kg^{-1}	Maximum Residue Limit[2]
Flumequine (Quinolones)	600 μg kg^{-1}	Maximum Residue Limit[2]
Sarafloxacin (Quinolones)	30 μg kg^{-1}	Maximum Residue Limit[2]
Florfenicol	1000 μg kg^{-1}	Maximum Residue Limit[2]
EFSA PCB 6 [7]	75 μg kg^{-1}	EC Maximum Limit[8]
HCB	50 μg kg^{-1}	Norway (G)[4]
γ HCH	100 μg kg^{-1}	Finland (S)[4]
p,p'DDT and metabolites	500 μg kg^{-1}	Finland (S)[4]
Aldrin + Dieldrin	100 μg kg^{-1}	Finland (S)[4]
Endrin	50 μg kg^{-1}	Finland(S)[4]
Malachite Green	0.5 μg kg^{-1}	Decision Limit[3]
Leuco Malachite Green	0.5 μg kg^{-1}	Decision Limit[3]
Brilliant Green	0.5 μg kg^{-1}	Decision Limit[3]
Crystal Violet	0.5 μg kg^{-1}	Decision Limit[3]
Leuco Crystal Violet	0.5 μg kg^{-1}	Decision Limit[3]
Victoria Blue	0.5 μg kg^{-1}	Decision Limit[3]
Lead	0.3 mg kg^{-1}	EC Maximum Limit[5]
Cadmium	0.05 mg kg^{-1}	EC Maximum Limit[5]
Mercury	0.5 mg kg^{-1}	EC Maximum Limit[5]

Notes

1. Commission Regulation (EU) No 37/2010 (Table 2) and Directive 2008/97/EC: *Substances banned and should not be detected*
2. Commission Regulation No 37/2010 (Table 1) *on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin*.
3. These compounds are not authorised for use in finfish, concentrations above the analytical methods decision limit are non-compliant.
4. OSPAR: *A compilation of standards and guidance values for contaminants in fish, crustaceans and molluscs for the assessment of possible hazards to human health*, Update 1993, JMP 17/3/10-E. (S) standard; (G) guidance value.
5. Commission Regulation (EC) No 1881/2006 *setting maximum levels for certain contaminant in foodstuffs and its amendments* Commission Regulation 629/2008/EC, Commission Regulation 420/2011/EC and Commission Regulation 488/2014/EC.
6. Maximum Residue Limits and Decision Limits concentration are on a wet weight basis.
7. EFSA PCB 6: sum of the following 6 CB congeners –PCB 28, 52, 101, 138, 153, 180.
8. Commission Regulation No 1259/2011 amending Regulation No. 1881/2006 *as regards maximum levels for dioxins, dioxin-like PCBs and non-dioxin like PCBs in foodstuffs*.

Appendix 2: Accreditation to ISO 17025

The table below outlines the parameters as tested at the Marine Institute for which the Marine Institute is accredited by the Irish National Accreditation Board (INAB) to ISO 17025 as detailed in Scope Registration Number 130T.

Scope Registration Number 130T	
Test	**SOP**
Ivermectin, Emamectin B1a , Doramectin[3]	CHE-8
Mercury[4]	CHE-32
Teflubenzuron , Diflubenzuron[3]	CHE-42
Dyes[3]: Malachite Green, Crystal Violet, Victoria Blue, Leuco Crystal Violet, Leuco Malachite Green and Brilliant Green	CHE-167
Metals[4]: Cadmium Lead, Silver, Nickel, Arsenic, Copper, Lead, Chromium and Zinc	CHE-178
Screening of Antibiotic Residues in Fish[3]	FHU-1
Screening of sulphadiazine[3]	FHU-119
Moisture %[4]	CHE-52
When collecting samples the laboratory complies with Council Directive 96/23/EC	CHE-6

[3] Accreditation is for finfish only
[4] Accreditation is for Marine Biota

Appendix 3: Quality Control

To check the quality of the data produced during the 2019 National Surveillance Scheme for chemical residues in farmed fish, Quality Control (QC) samples in the form of either reagent blanks, spiked samples or Certified Reference Materials (CRMs) were analysed with each batch of samples tested by the Marine Institute. The quality assurance results were considered sufficient for the purpose of the monitoring programme. For CRMs, z-scores were calculated using the methodology of QUASIMEME (Quality Assurance of Marine Environment and Monitoring in Europe); A Z-score of between –2 and +2 is generally considered satisfactory for the purpose of environmental monitoring programmes. Where available the MI participate in Proficiency schemes such as FAPAS, QUASIMEME to verify our analytical methods independently. Quality Control information for tests carried out at the Marine Institute is available on request.

Appendix 4: Methods of Analysis

Analysis carried out at the Marine Institute laboratories unless otherwise stated

1.1 Sample Collection and Preparation (MI SOP: CHE-6): *MI Testing Lab*

In accordance with the 2018 National Residues Control Plan for Aquaculture under Council Directive 96/23/EC, Staff authorised under the *Animal Remedies Act 1993*, collected samples at farms or at processing plants. All samples were transported to the laboratory under controlled conditions, while ensuring an unbroken chain of custody. Sub-samples were taken for both analytical and archive purposes and all sub-samples were stored frozen (< -18°C).

1.2* Analysis of Ivermectin, Doramectin and Emamectin B1a by Ultra-Fast Liquid Chromatography (UFLC) with Fluorescence Detection *(MI SOP: CHE-8): MI Testing Lab

Approximately 5g of sample from each fish was homogenised and extracted with methanol. The extract was cleaned up by liquid/liquid partition and solid phase extraction techniques. The resultant residue was derivatised and analysed by liquid chromatography (UFLC) with fluorescence detection.

At the end of 2019, the MI introduced a semi-quantitative screening for this analysis. Therefore, a sample is screened firstly by the semi-quantitative screening method and if a sample was screened at or above the screening detection limit (SDL) for that analyte it would trigger a full quantitative test and result confirmed where applicable. The SDL for Emamectin is at half the MRL i.e. the SDL is 50 ug kg^{-1} and the SDL is at half the Decision Limit for Ivermectin and Doramectin i.e. the SDL is 0.5 ug kg^{-1} for both Ivermectin and Dormaectin.

1.3 Analysis of Teflubenzuron and Diflubenzuron by Ultra-Fast Liquid Chromatography (UFLC) with Ultraviolet (UV) Detection *(MI SOP: CHE-42): MI Testing Lab*

This method involves the extraction of approximately 3g of tissue with acetonitrile followed by clean up using liquid/liquid partition and silica SPE. Quantification was carried out by reverse phase UFLC using an acetonitrile/water mobile phase and UV detection. Confirmation and peak purity was evaluated using a photodiode array detector.

1.4* Analysis for Cypermethrin and Deltamethrin by Gas Chromatography-Mass Spectrometry (GC-MS) – *[MI SOP: CHE-215] MI Testing Lab and confirmatory FERA

Samples were extracted using a modified QUECHERs approach followed by dispersive solid phase extraction(DSPE) and a secondary clean up using florisil solid phase extraction. The extract was reconstituted in iso-octane and analysed by GC-MSMS. At the end of 2019, the MI introduced a semi-quantitative screening for this analysis. Therefore, a sample is screened firstly by the semi-quantitative screening method and if a sample was screened at or above the screening detection limit (SDL) for that analyte it would trigger a full quantitative screening test. The SDL for Cypermethrin and Deltamethrin is at half the MRL i.e. the SDL is 25 µg kg^{-1} and 5 µg kg^{-1} respectively. Where confirmatory analysis was required the samples were tested by FERA-UK. In 2019, confirmatory testing by FERA-UK only required for select number of samples, reference footnote in analytical results if applicable.

1.5 Analysis of Dyes by Ultra-Fast Liquid Chromatography (UFLC) with MS/MS detection *(MI SOP: CHE-167): MI Testing Lab*

Samples were extracted for Dyes analysis with Acetonitrile by shaking in the presence of hydroxylamine and magnesium sulphate. The eluant is evaporated to dryness followed by reconstitution in a mixture of acetonitrile/water /ascorbic acid solution. This solution is centrifuged, filtered and analysed for brilliant green, crystal violet, leuco crystal violet, leuco malachite green, malachite green and victoria blue by Ultra-Fast Liquid Chromatography coupled to Mass Spectrometry (UFLC-MS/MS).

1.6 Screening for Antibacterial Substances (Quinolones, Tetracylines and Florfenicol) using modified Two Plate Test *(MI SOP: FHU-1): MI Testing Lab*

Antimicrobial screening was carried by the Fish Health Unit (FHU) of the Marine Institute, using a modification of the Two Plate Test (TPT). The aim of this method is to reveal residues of substances with antibacterial activity by testing the fish tissue using agar plates that have been seeded with suitably sensitive bacterial cultures. This method is qualitative in nature and was used to detect residues of Quinolones, Tetracyclines and Florfenicol. Where confirmatory analysis was required for oxytetracyclines the samples were tested by WFSR previously known as RIKILT.

1.7 Screening for sulphonamides by Evidence Investigator *(MI SOP: FHU-119): MI Testing Lab*

Screening for sulphonamides was carried by the Fish Health Unit (FHU) of the Marine Institute using Immunoassay. This method is qualitative in nature and tested on the Evidence Investigator instrument. Where confirmatory analysis was required the samples were tested by EURL-ANSES.

1.8 Screening for Group A Compounds by Elisa method: *IEC Testing Lab*

Screening for Group A compounds was carried out by the Irish Equine Centre (IEC) using the Enzyme-Linked Immuno Sorbant Assay (ELISA) method. This method is qualitative in nature and was used to detect residues of 17β-oestradiol, chloramphenicol and methyltesterone. Where confirmatory analysis was required the samples were tested by EURL-RIKILT. In 2019, confirmatory testing only required for 17β -estradiol for a select number of samples.

1.9 **Screening for Group B - Cortiscosteroids by Elisa method: *IEC Testing Lab***

Screening for corticosteroids was carried out by the Irish Equine Centre (IEC) using the Enzyme-Linked Immuno Sorbant Assay (ELISA) method. Where further quantitative LCMSMS screening was required for cortiscosteroids the sample was tested by EURL (RIKILT).

1.10 **Analysis of Nitrofurans by Ultra Performance Liquid Chromatography with Mass Spectrometry detection (UPLC-MS/MS): *TFRC Testing Lab***

Analysis of nitrofurans was carried out by Teagasc Food Research Centre (TFRC). Tissue bound residues of nitrofurans are hydrolysed with acid and derivatised with 2-nitrobenzaldehyde. The nitrophenyl derivatives are extracted with ethyl acetate and determined by Ultra Performance Liquid Chromatography coupled to Mass Spectrometry (UPLC-MS/MS) using deuterated analogues as internal standards for quantification. Metabolites of furazolidone, furaltadone, nitrofurantoin and nitrofurazone are analysed.

1.11 Analysis of Nitroimidazoles by UPLC-MS/MS: *TFRC Testing Lab*

Analysis of nitroimidazoles was carried out by Teagasc Food Research Centre (TFRC). Samples are extracted with acetonitrile, water, magnesium sulphate and sodium chloride; defatted with n-hexane and concentrated. The residue content is determined by Ultra Performance Liquid Chromatography coupled to Mass Spectrometry (UPLC-MS/MS) and analysed for dimetridazole and its metabolite, ipronidazole and its metabolite, metronidazole and its metabolite, ornidazole and ronidazole.

1.12 **Analysis for Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides (OCPs) by GC/HRMS: *Eurofins Testing Lab***

Analysis for PCBs and OCPs was carried out by a subcontracted laboratory (Eurofins). Prior to the extraction, ^{13}C-UL-labeled internal standards were added, followed by an extraction using a solid/lipid extraction and clean up by a multicolumn system. Concentration levels were determined by (Gas chromatography - high resolution mass spectrometry (GC/HRMS) using a DB-5 capillary column.

1.13 **Analysis of Trace metals by Inductively Coupled Plasma -Mass Spectrometry (ICP-MS) *(MI SOP CHE-178): MI Testing Lab***

Arsenic, cadmium, chromium, copper, lead, nickel, silver, zinc (and additional metals aluminium, cobalt, iron, manganese, selenium and vanadium).

Concentrated nitric acid (4ml) and hydrogen peroxide (4ml) was added to approximately 0.2g freeze-dried tissue, which was then digested in a laboratory microwave oven (CEM Mars Xpress). After cooling, samples were diluted to 50mls with deionised water. Trace metal concentrations were determined by ICP-MS (Agilent 7700x with High Matrix Introduction (HMI) system). Interferences were removed using a helium collision cell and appropriate correction equations.

1.14 **Analysis of Mercury by Cold Vapour Atomic Fluorescence Spectroscopy CV-AFS *(MI SOP CHE-42): MI Testing Lab***

Concentrated nitric acid (4 ml) was added to approximately 0.2 g freeze-dried tissue, which was then digested in a laboratory microwave oven (CEM Mars Xpress). After cooling, potassium permanganate was added until the purple colour of the solution stabilized. Sufficient hydroxylamine sulphate/sodium chloride solution was added to neutralise the excess potassium permanganate and potassium dichromate was added as a preservative. The solution was diluted to 100mls using deionised water. Following reduction of the samples with tin (II) chloride, total mercury concentration was determined by Cold Vapour Atomic Fluorescence Spectroscopy (CV-AFS) using a PSA Merlin Analyser.

1.15 Determination of Moisture Content *(MI SOP CHE-52): MI Testing Lab*

The moisture content was determined by drying approximately 1g of tissue overnight in an oven at 104ºC to constant weight.

1.16 Analysis of Mycotoxins: *Wessling Testing Lab*

Analysis of Aflatoxins B1, B2, G1 and G2 was carried out by Wessling. The method involved the extraction of about 25g of muscle using dichloromethane and the extract was cleaned up on an immunoaffinity column. The subsequent determination of aflatoxins B1, B2, G1 and G2 was achieved using Liquid Chromatography with Fluorescence Detection after post column derivatisation.

Appendix 5: 2019 Plan for the Monitoring and Detection of Residues in Aquaculture products

2019 Plan for the Monitoring and Detection of Residues in Aquaculture products

1. National Legislation on use of substances listed in Annex I of Directive 96/23/EC

Animal Remedies Act, 1993 (No. 23 of 1993)
Animal Remedies Regulations, 2007 (SI No. 786 of 2007)
Control of Animal Remedies and their Residues Regulations 2009(SI No. 183 of 2009)

2. Relevant Departments and their infrastructure

- **Marine Institute (MI)** Rinville, Oranmore, Co. Galway
- **Dept of Agriculture, Food & Marine (DAFM),** Agriculture House**,** Kildare Street, Dublin 2
- **Sea-Fisheries Protection Authority (SFPA),** Block B, Clogheen, Clonakilty, Co. Cork

3. Staff resources to carry out plan

- Authorised Officers will collect all samples.
- Analysis of Group A substances - performed by Irish Equine Centre, Kildare, Teagasc Food Research Centre, Dublin, ANSES-Fougères, France and RIKILT, The Netherlands.
- Analyses for Group B substances - performed within the Marine Institute with the exception of those indicated in the plan.

4. Approved laboratories

Marine Institute (MI)
Rinville,
Oranmore,
Co. Galway
H91 R673

Irish Equine Centre (IEC)
Johnstown,
Naas,
Co. Kildare
W91 RH93

Teagasc Food Research Centre (TFRC)
Ashtown,
Dublin 15
D15 KN3K

Wageningen Food Safety Research (WFSR)

Laboratory for Residue analysis,
Akkermaalsbos 2,
6708 WB Wageningen,
The Netherlands

ANSES - Fougères,
10B rue Claude Bourgelat, Javené CS 40608 35306, Fougères Cedex,
France

Fera Science Ltd
Sand Hutton,
York,
North Yorkshire
Y041 1LZ

Eurofins GfA GmbH,
D-48161 Münster
Germany

Wessling GmbH,
Kohlenstraße 51-55,
44795 Bochum,
Germany

5. Additional Information

For Group A analysis more than half the samples are ‘on farm’ samples, taken at various stages of production, the remainder are samples taken at harvest

DIRECTIVE 96/23/EC ANNUAL PLAN FOR THE EXAMINATION FOR RESIDUES IN FARMED FINFISH FOR THE YEAR 2019

Sampling levels and frequency:

Minimum number of fish from which samples must be taken.

Finfish.

Total Tonnes Produced 2017	**Minimum no. to be tested[a]**	**Minimum No. Group A**	**Minimum No. Group B**
18,989	Production (tonnes)/100 = 190	1/3 Total Tested = 63	2/3 Total Tested = 127

[a] min no. to be tested will be based on 2017 finfish production figures as 2018 figures are not available

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Matrix**	**Lab Method**	**CCbeta (Screening) Detection Capability**	**CCalpha (Confirmatory) Decision Limit**	**Level of action**	**Sample No.**	**Laboratory**
Group A								
A 3 Steroids	Methyltestosterone	Muscle & Skin	(1) ELISA (2) GCMSMS	1)1.5 µg kg^{-1}	2)0.05 µg kg^{-1}	Presence	63[(b)]	(1) IEC (2) EU-RL RIKILT
	17β-Oestradiol	Muscle & Skin	(1) ELISA (2) GCMSMS	1)1.5 µg kg^{-1}	2)0.17 µg kg^{-1}	0.5 µg kg^{-1}	16[(b)]	(1) IEC (2) EU-RL RIKILT
A 6 Compounds included in Annex IV Council Reg. 2377/90	**Chloramphenicol**	Muscle & Skin	(1) ELISA (2) LCMSMS	1)0.25 µg kg^{-1} 1)0.3 µg kg^{-1}[(c)]	2)0.05 µg kg^{-1}	Presence	63[(b)]	(1) IEC (2) EU-RL ANSES- Fougères
	Nitrofurans AOZ AMOZ AHD **SEM**	Muscle & Skin	UPLCMSMS		 0.041 µg kg^{-1} 0.061 µg kg^{-1} 0.057 µg kg^{-1} 0.064 µg kg^{-1}	Presence	10[(b)]	TFRC
	Nitroimidazoles Dimetridazole HMMNI Ipronidazole Hydroxyl-ipronidazole Metronidazole Hydroxyl- Metronidazole Ornidazole Ronidazole	Muscle & Skin	UPLCMSMS		 0.12 µg kg^{-1} 1.0 µg kg^{-1} 0.15 µg kg^{-1} 0.10 µg kg^{-1} 0.10 µg kg^{-1} 0.15 µg kg^{-1} 0.29 µg kg^{-1} 0.10 µg kg^{-1}	Presence	10[(b)]	TFRC

Column 4: (1) Screening Method, (2) Confirmatory Method

[(b)] At least 50% of Group A are "on farm" samples

[(c)]For screened positive samples for Chloramphenicol using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMSMS).

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Lab Method**	**CCbeta (Screening) Detection Capability**	**CCalpha (Confirmatory) Decision Limit**	**Level of action**	**Sample No.**	**Laboratory**
B 1 Antibacterial substances	Microbiological screening: Quinolones: -Oxolinic acid -Flumequine Tetracyclines[(c)] -oxytetracycline Florfenicol [(c)]	Muscle & Skin	Modified EC 2-plate method	 75 µg kg^{-1} 150 µg kg^{-1} 100 µg kg^{-1} 750 µg kg^{-1}	N/A	[(c)]	127	MI
	Screening: Sulphonamides -Sulphadiazine	Muscle & Skin	Immunoassay	50 µg kg^{-1}	N/A	[(c)]	127	MI
	Tetracycline Oxytetracycline Tetracycline Chlortetracycline Doxycycline	Muscle & Skin	LCMSMS		 140 µg kg^{-1} 123 µg kg^{-1} 116 µg kg^{-1} 114 µg kg^{-1}	 140 µg kg^{-1} 123 µg kg^{-1} 116 µg kg^{-1} 114 µg kg^{-1}	Confirmation and post screening identification of positive Microbiological Samples/ Bioassay	RIKILT
	Quinolones Ciprofloxacin Enrofloxacin Danofloxacin Difloxacin Flumequine Oxolinic acid Sarafloxacin Marbofloxacin		LC-Flu		 118.3 µg kg^{-1} 113.7 µg kg^{-1} 112.3 µg kg^{-1} 337.6 µg kg^{-1} 624.5 µg kg^{-1} 108.0 µg kg^{-1} 37.4 µg kg^{-1} 28.2 µg kg^{-1}	 118.3 µg kg^{-1} 113.7 µg kg^{-1} 112.3 µg kg^{-1} 337.6 µg kg^{-1} 624.5 µg kg^{-1} 108.0 µg kg^{-1} 37.4 µg kg^{-1} 28.2 µg kg^{-1}		EU-RL ANSES-Fougères

Column 4: (1) Screening Method, (2) Confirmatory Method

[(c)]For screened positive samples i.e. above CC_{beta} for tetracyclines, quinolones, sulphonamides using MI in-house methods, these samples will be sent to subcontract laboratory for confirmatory testing. NOTE: In 2019, MI may examine alternative methods which may result for a temporary period the subcontracting out to a validated and accredited laboratory the testing for tetracycline and florfenicol during this transition period.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Lab Method**	**CCbeta (Screening) Detection Capability**	**CCalpha (Confirmatory) Decision Limit**	**Level of action**	**Sample No.**	**Laboratory**
B 1 Antibacterial substances	Sulphonamides Sulphathiazole Sulphaquinoxaline Sulphamethoxypyridazine Sulphamonomethoxine Sulphamerazine Sulphadimethoxine Sulphadiazine Sulphachlorpyridazine Sulphamethizole Sulfacetamide Sulfachlozine Sulfadoxine Sulfamethoxazole	Muscle & Skin	LMSMS		108.0 µg kg^{-1} 113.3 µg kg^{-1} 120.4 µg kg^{-1} 104.9 µg kg^{-1} 103.8 µg kg^{-1} 109.3 µg kg^{-1} 114.4 µg kg^{-1} 109.0 µg kg^{-1} 119.3 µg kg^{-1} 122.9 µg kg^{-1} 111.9 µg kg^{-1} 106.4 µg kg^{-1} 117.8 µg kg^{-1}	108.0 µg kg^{-1} 113.3 µg kg^{-1} 120.4 µg kg^{-1} 104.9 µg kg^{-1} 103.8 µg kg^{-1} 109.3 µg kg^{-1} 114.4 µg kg^{-1} 109.0 µg kg^{-1} 119.3 µg kg^{-1} 122.9 µg kg^{-1} 111.9 µg kg^{-1} 106.4 µg kg^{-1} 117.8 µg kg^{-1}		EU-RL ANSES- Fougères
	Florfenicol		LCMSMS		[(d)]	1000 µg kg^{-1}		RIKILT
B2 Other veterinary drugs								
B2 (a) Anthelmintics	Ivermectin	Muscle & Skin	UFLC-Flu	-	0.4 µg kg^{-1}	0.4 µg kg^{-1}	127	MI
	Emamectin B1a			-	124 µg kg^{-1}	124 µg kg^{-1}		
	Doramectin			-	0.4 µg kg^{-1}	0.4 µg kg^{-1}		
B2 (c) Carbamates / Pyrethroids	Cypermethrin	Muscle & Skin	GC-MS	1)25 µg kg^{-1} 2)5 µg kg^{-1}	[(d)]	50 µg kg^{-1}	127	1) MI 2)FERA, UK
	Deltamethrin			1)5 µg kg^{-1} 2)5 µg kg^{-1}	[(d)]	10 µg kg^{-1}		
B2 (f) Other Pharmacologically active substances	Teflubenzuron	Muscle & Skin	UFLC-DAD	-	575 µg kg^{-1}	575 µg kg^{-1}	127	MI
	Diflubenzuron			-	1151 µg kg^{-1}	1151 µg kg^{-1}		
	Corticosteroids Betamethasone Dexamethasone Flumethasone	Muscle & Skin	(1) ELISA (2) LC-MS	1)1.5 µg kg^{-1} 1.5 µg kg^{-1} 1.5 µg kg^{-1}	[(d)]	Presence	29[(f)]	(1) IEC (2) EU-RL RIKILT

Column 4: (1) Screening Method, (2) Confirmatory Method

[(d)] Can provide confirmation under accreditation scope. CCalpha will be calculated at that point and level of action updated.

[(f)] At least 50% are "on farm" samples

1	2	3	4	5	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Lab Method**	**Detection limit**	**Level of action**	**No. samples**	**Laboratory**
B3 Other substances and environmental contaminants							
B3(a) Organochlorine compounds including PCBs	**PCBs** Sum of 6 PCBs [PCB28, 52, 101, 138, 153, 180]	Muscle & Skin	GCHRMS	[g] 0.07 µg kg^{-1} per individual congener	[h] 75 µg kg^{-1}	20	Eurofins
	Chlorinated Pesticides [j] γ-HCH DDT and metabolites[k] HCB Endrin Aldrin + Dieldrin		GCHRMS	 [g] 0.06 µg kg^{-1} [g] 0.15 µg kg^{-1} [g] 0.13 µg kg^{-1} [g] 0.075 µg kg^{-1} [g] 0.063 µg kg^{-1}	Excess of Guidance value [i] 100 µg kg^{-1} 500 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 100 µg kg^{-1}	10	
B3(c) Chemical elements	Lead		ICP-MS	7 µg kg $^{-1}$	[h]300 µg kg $^{-1}$	10	MI
	Cadmium		ICP-MS	1 µg kg $^{-1}$	[h]50 µg kg $^{-1}$	10	
	Mercury		CVAFS	2 µg kg $^{-1}$	[h]500 µg kg $^{-1}$	10	
B3(d) Mycotoxins	Aflatoxin B1	Muscle & Skin	HPLC-FLD	0.01 µg kg^{-1}	-	8	Wessling
	Aflatoxin B2			0.01 µg kg^{-1}			
	Aflatoxin G1			0.01 µg kg^{-1}			
	Aflatoxin G2			0.01 µg kg^{-1}			

[g] Detection limit is at limit of quantification for PCBs and OCPs
[h] Commission Regulation No. 1881/2006 as amended setting maximum levels for certain contaminants in foodstuffs; matrix: muscle & skin as skin eaten
[i] There are no national or European maximum limits for organochlorine pesticides in fish. The guidance values used represent the strictest national limits applied by contracting parties to the OSPAR convention and as compiled by OSPAR (1992), in so far as they are known. These values have no statutatory basis and are used in the absence of other criteria.
[j] Additional chlorinated pesticides are also included in routine testing but no action level or guidance values are available
[k]DDT and metabolites: sum of DDT-o,p', DDT-p,p', DDD-o,p', DDD-p,p', DDE-o,p', DDE-p,p'

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Lab Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**No. samples**	**Laboratory**
B3(e) Dyes	Malachite Green (MG) Leuco Malachite Green (LMG) Brilliant Green (BG) Crystal Violet (CV) Leuco Crystal Violet (LCV) Victoria Blue (VB)	Muscle & Skin	UFLCMSMS	- -	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	94[(m)] 24 x salmon/sea trout 12 x freshwater trout (harvest) 8 x freshwater trout (osop) 50 x salmon smolts	MI

[(m)] 70 of the 94 samples for dyes are “on farm”

Appendix 6: Annual Plan for Sampling Fishery Products and Other Seafood at Border Control Posts. Dublin Port 2019

Group	Test	TRACES sampling list	Samples to be taken	Laboratory
Microbiological	Microbiological testing against Microbiological Criteria stipulated in Regulation 2073/2005	1037, 1040, 1042, 1045, 1074, 1076, 1079	**3∞ samples,** each of *n* units. Targeting Fishery Products and other seafood for which Microbiological Criteria are stipulated in Regulation 2073/2005 and using the sampling plans (*n* values) outlined there in **∞Samples numbers subject to change**	Eurofins Food Testing Ireland Ltd Unit D13 North City Business Park North Road Dublin 11 Phone: 01 431 1306 Email: info@eurofins.ie
	Histamine	Histamine	**4∞ samples** of each of *n* units. Targeting fishery products derived from species associated with high amounts of histidine or fish sauce produced by fermentation of fishery products **∞Samples numbers subject to change**	*Dr. Brenda Lennon* *Executive Chemist,* *Public Analyst's Laboratory,* *Seamus Quirke Road,* *Galway.* *Tel: 091-581122* *Fax: 091-581212* *E-mail:* Brenda.Lennon@hse.ie

Group	Test	TRACES sampling list	Samples to be taken	Laboratory
A.6	Nitrofuran metabolites	Nitrofurazone, Nitrofurantoin, Furazolidone, Furaltadone	**7∞ aquaculture samples** (shellfish & finfish relative to consignment numbers) **∞Samples numbers subject to change**	Dr. Martin Danaher, Food Safety Department, Ashtown Food Research Centre, Teagasc, Ashtown, Dublin 15. Tel: 01 8059500 Fax: 01 8059550 martin.danaher@teagasc.ie
A.6	Chloramphenicol	Chloramphenicol	**2∞ aquaculture samples** (shellfish & finfish relative to consignment numbers) **∞Samples numbers subject to change**	Dr. John Gibbons, Irish Equine Centre, Johnstown, Naas, Co. Kildare Telephone: 045 866266 Fax: 045 866 273
B.1	Antibacterial substances General 2 plate test & Immuno assay	Antibacterial substances	**3∞ aquaculture samples** (shellfish & finfish relative to consignment numbers) **∞ Samples numbers subject to change**	Denise Glynn Residues Coordinator Marine Institute Rinville Oranmore Galway H91 R673 Phone: + 353 91 387332 Reception: 091-387200 Fax: 091-387201 E-mail: Denise.Glynn@Marine.ie
B.2.a	Anthelmintics (Avermectins)	Emamectin, Ivermectin Doramectin	**2∞ aquaculture samples** (shellfish & finfish relative to consignment numbers) **∞ Samples numbers subject to change**	
B.3.e	Dyes	Malachite Green (MG) Leuco Malachite Green (LMG) Brilliant Green (BG) Crystal Violet (CV) Leuco Crystal Violet (LCV) Victoria Blue (VB)	**4∞ aquaculture samples** (shellfish & finfish relative to consignment numbers) **∞ Samples numbers subject to change**	

Group	Test	TRACES sampling list	Samples to be taken	Laboratory
B.3.d	Chemical - Heavy Metals *(Specify Pb, Cd, or Hg*)	Pb Lead Hg Mercury Cd Cadmium	**2∞ fish samples** **∞ Samples numbers subject to change**	
	Chemical - Sulphur Dioxide 4-Hexylresorcinol	Sulphur Dioxide 4-hexylresorcinol	**2* ∞ prawn /shrimp samples** (1kg approx. per sample) **∞ Samples numbers subject to change**	Michael O'Riordan Cork Public Analyst's Laboratory St Finbarr's Hospital Cork Michael.ORiordan@hse.ie. Tel: 021 4923245 Fax: 021 4923367
	Fish Speciation	DNA	**2/4 ∞ samples of Finfish** **∞ Samples numbers subject to change**	David Lee Public Analyst's Laboratory St. Finbarr's Hospital Douglas Road Cork Phone: 021 4923364 E-mail: David.Lee@hse.ie

Annual Plan for Sampling Fishery Products and Other Seafood at Border Inspection Posts. Shannon Airport 2019

Group	Test	TRACES sampling list No.	Samples to be taken	Laboratory
Microbiological	Microbiological testing against Microbiological Criteria stipulated in Regulation 2073/2005	1037, 1040, 1042, 1045, 1074, 1076, 1079	**1∞ samples**, each of *n* units. Targeting Fishery Products and other seafood for which Microbiological Criteria are stipulated in Regulation 2073/2005 and using the sampling plans (*n* values) outlined there in ∞ **Samples numbers subject to change**	Complete Laboratory Solutions (CLS) Ros Muc Connemara Co. Galway Tel: 091 574355 Fax: 091 574356 E-mail: microfoodandwater@cls.ie Contacts: Anne O'Donnell aodonnell@cls.ie and Katie Ui Chatnaigh: kuichatnaigh@cls.ie
B.3.d	Chemical - Heavy Metals *(Specify Pb, Cd, or Hg*)	Pb Lead Hg Mercury Cd Cadmium	**1∞ fish or crustacean samples** For live lobster samples can BIP officers please freeze the sample before sending to MI for heavy metal analysis. ∞ **Samples numbers subject to change**	Denise Glynn Residues Coordinator Marine Institute Rinville Oranmore Galway H91 R673 Phone: + 353 91 387332 Reception: 091-387200 Fax: 091-387201 E-mail: Denise.Glynn@Marine.ie Website: www.marine.ie