# Summary Report on 2015 Residue Monitoring of Irish Farmed Finfish & 2015 Border Inspection Post Fishery and Fishery Product Sample Testing

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Marine Institute
Foras na Mara

# Summary Report on 2015 Residue Monitoring of Irish Farmed Finfish & 2015 Border Inspection Post Fishery and Fishery Product Sample Testing

Residues samples collected by Marine Institute in accordance with the National Residue Control Plan for Aquaculture 2015

Border Inspection samples taken by DAFM in accordance with Border Inspection Post Plan 2015

**Issue date: March 2017**

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ISO 17025 INAB ACCREDITED TESTING DETAILED IN SCOPE REG NO. 130T

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

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## Part A



## PART B



## APPENDICES

# Part A

# Summary Report on 2015 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*.**

- **2015 OVERALL SUMMARY**

In 2015, in excess of 676 tests and a total of 1,845 measurements were carried out on 128 samples (i.e. 124 target samples & 4 suspect samples) of farmed finfish for a range of residues. Implementation of the Aquaculture 2015 Plan involves taking samples at both farm and processing plant:

- 91 target samples taken at harvest: 83 farmed salmon and 8 freshwater trout.
- 33 target samples were taken at other stages of production: 25 salmon smolts and 8 freshwater trout.
- 4 suspect harvest freshwater trout samples were taken as part of SFPA on-farm investigations.

All 2015 samples were compliant with the exception of two harvest freshwater trout samples from one farm which was found to have sulphadiazine (Group B1- antibacterial substance) present in excess of the Maximum Residue Limit. As part of the on-farm investigation carried out by the SFPA the Marine Institute additionally collected 4 suspect samples.

For target sampling of farmed fish, a summary table of the residue results from 2005 - 2015 are outlined in Table 1. Overall, the outcome for aquaculture remains one of consistently low occurrence of residues in farmed finfish, with 0% non-compliant target residues results for the period 2006-2014, with a slight increase to 0.11% in 2015.

**Table 1:** Summary Target Results for Residue program since 2005-2015.

| | 2005 | 2006 | 2007 | 2008 | 2009 | 2010 | 2011 | 2012 | 2013 | 2014 | 2015 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **No. Target samples[1]** | 164 (105, 59) | 162 (104, 58) | 161 (103, 58) | 162 (103, 59) | 146 (98, 48) | 141 (92, 49) | 140 (92, 48) | 169 (112, 57) | 137 (91, 48) | 136 (91, 45) | 124 (91, 33) |
| **Total Group A[2]** | 163/0 | 162/0 | 148/0 | 144/0 | 128/0 | 109/0 | 105/0 | 101/0 | 83/0 | 83/0 | 71/0 |
| **Total Group B[2]** | 164/0 | 162/0 | 161/0 | 162/0 | 146/0 | 141/0 | 140/0 | 169/0 | 137/0 | 136/0 | 124/2 |
| **Total No. of Results[3]** | 2251/2 | 2207/0 | 2219/0 | 2073/0 | 1750/0 | 1569/0 | 1566/0 | 1596/0 | 1494/0 | 1882/0 | 1841/2 |
| **% non -compliant results** | **0.09** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | **0** | **0.11** |

[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*

- **BACKGROUND**

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). Outlined in Table 2 is a summary of each department and agency's role with respect to the NRCP.

**Table 2: Residues Directive and Aquaculture:** Department and Agencies' 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)** |
| **Responsible for ensuring 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 2 illustrates the National Aquaculture Residue Control Cycle. The 2015 NRCP is available in Appendix 5.

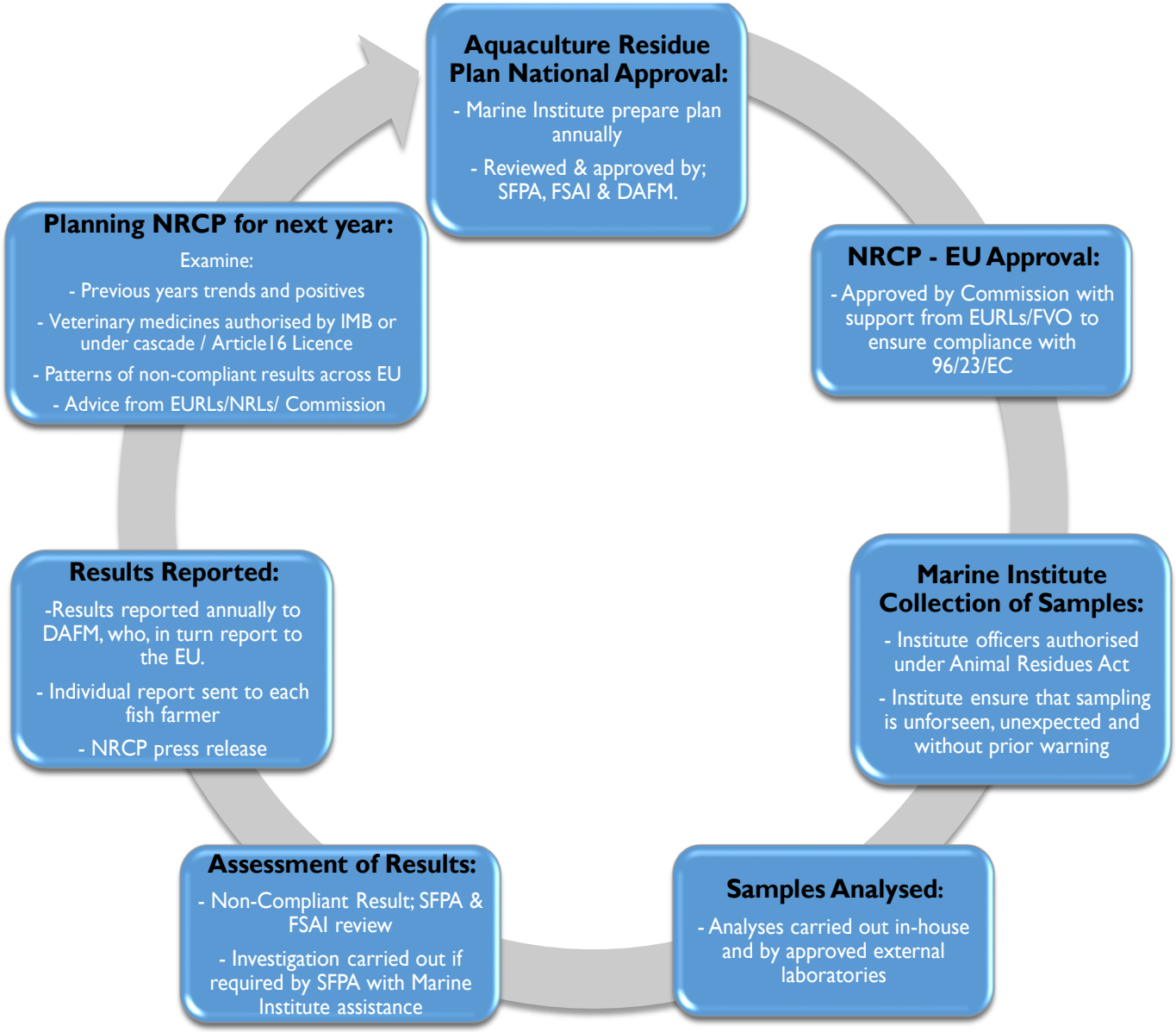


**Figure 2:** National Aquaculture Residue Control Cycle

## 2.2 Scope of NRCP:

As with other farmed animals, farmed finfish can be subject to disease and infestation which can have animal welfare, environmental and commercial implications. 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 can be introduced inadvertently which may accumulate in fish e.g. polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), heavy metals |
| ***MRL is the 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 anabolic effect | | Group B- Veterinary drugs and contaminants | |
|---|---|---|---|
| **A3** | Steroids | **B1** | Antimicrobials (Antibacterial) |
| **A6** | Compounds included in Annex IV of Council Regulation 2377/90/EC | **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 including 17 β-oestradiol, methyltestosterone which occur naturally but also could be used for growth promotion.
- A6 compounds including nitrofurans and nitroimidazole which are antibacterial drugs, and chloramphenicol a broad spectrum antibiotic.

### Group B (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.

- **SAMPLING**

In 2015, 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 sea-reared trout and 'on farm' for freshwater trout.

In 2015, a total of 124 target (surveillance) samples were collected from fish farms and processing plants in accordance with the NRCP for Aquaculture 2015 (Appendix 5) as follows:

- 33 target samples were taken at other stages of production (OSOP); 25 salmon smolts and 8 freshwater trout from seven farms for Group A and malachite green analysis.

- 91 target samples taken at harvest which comprised of 83 farmed salmon and 8 freshwater trout. These harvest samples were collected during 19 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 two occasions. In 2015 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.

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 are further subsampled as multiple tests are typically performed on individual samples.

**Suspect sampling** took place in 2015 following the confirmation of sulphadiazine in excess of the MRL (Group B1- antibacterial substance) in two harvest freshwater trout samples from one farm. As a result of this finding, an on-farm investigation was carried out by the SFPA which included further sampling by the Marine Institute where 4 suspect samples were taken in October 2015.

- **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.

Samples are deemed **non-compliant (i.e. positive)** if concentrations of a given residue are confirmed to be in **excess of the Maximum Residue Limit (MRL)** where an MRL has been set. Follow up action is taken on samples with detected concentrations in excess of the MRL. The source of MRLs and Decision Limit (action level) are specified in Appendix 1.

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. The sources of Decision Limits are specified in Appendix 1. **A non-compliant result is one above a Decision limit (action level).**

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 "ICES 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 of 75 µg $kg^{-1}$ wet weight. For Organochlorine Pesticides (OCPs) there are no MRLs, 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, reference Appendix 2 and 3.

## 4.2 Breakdown of 2015 Results

In 2015, in excess of 676 tests and a total of 1,845 measurements were carried out on 128 samples (i.e. 124 target samples & 4 suspect samples) of farmed finfish. All 2015 samples were compliant with the **exception of two harvest freshwater trout samples from one farm which was found to have sulphadiazine** (Group B1- antibacterial substance) **present.**

**Table 5:** Summary of 2015 Residue Monitoring Results for Target farmed fish samples (salmon and trout). All tests performed on muscle tissue.

| RESIDUE | GROUP | NUMBER EXAMINED | Non-Compliant[1] | DETECTION LIMIT[2] (µg kg $^{-1}$) |
|---|---|---|---|---|
| **Methyltestosterone** | A3 | 38 | 0 | 1.5 |
| **17β-oestradiol** | A3 | 10 | 0 | 1.5 |
| **Chloramphenicol** | A6 | 38 | 0 | 0.25 |
| **Nitrofurans** | A6 | 9 | 0 | 0.06 |
| **Nitroimidazole** | A6 | 9 | 0 | 3.0 |
| **Tetracyclines: oxytetracycline** | B1 | 91 | 0 | 50 |
| **Quinolones: Oxolinic acid Flumequine** | B1 | 91 | 0 | 75 150 |
| **Florfenicol** | B1 | 91 | 0 | 250 |
| **Sulphonamides: Sulphadiazine** | B1 | 91 | 2 | 50 |
| **Emamectin B1a** | B2a | 91 | 0 | 9.0 |
| **Ivermectin** | B2a | 91 | 0 | 0.1 |
| **Doramectin** | B2a | 91 | 0 | 0.1 |
| **Cypermethrin[4]** | B2c | 5 | 0 | 5 |
| **Cyoermethrin[5]** | B2c | 86 | 0 | 12 |
| **Deltamethrin[4]** | B2c | 5 | 0 | 4 |
| **Deltamethrin[5]** | B2c | 86 | 0 | 3 |
| **Corticosteroids** | B2f | 27 | 0 | 1.5 |
| **Teflubenzuron** | B2f | 91 | 0 | 80 |
| **Diflubenzuron** | B2f | 91 | 0 | 86 |
| ICES PCB 6 (incl. LOQ)[3] | B3a | 19 | 0 | 0.128 |
| DDT (o p') | B3a | 10 | 0 | 0.02 |
| DDE (o p') | B3a | 10 | 0 | 0.02 |
| DDD (o p') | B3a | 10 | 0 | 0.02 |
| DDD (p p') | B3a | 10 | 0 | 0.02 |
| DDE (p p') | B3a | 10 | 0 | 0.02 |
| DDT (p p') | B3a | 10 | 0 | 0.02 |
| hexachlorobenzene | B3a | 10 | 0 | 0.08 |
| α-HCH | B3a | 10 | 0 | 0.04 |
| β-HCH | B3a | 10 | 0 | 0.04 |
| γ-HCH (lindane) | B3a | 10 | 0 | 0.04 |
| δ -HCH | B3a | 10 | 0 | 0.04 |
| Pentachlorobenzene | B3a | 10 | 0 | 0.08 |
| aldrin | B3a | 10 | 0 | 0.02 |
| dieldrin | B3a | 10 | 0 | 0.03 |
| endrin | B3a | 10 | 0 | 0.05 |

| RESIDUE | GROUP | NUMBER EXAMINED | Non-Compliant[1] | DETECTION LIMIT[2] (µg kg $^{-1}$) |
|---|---|---|---|---|
| Toxaphene 26 | B3a | 10 | 0 | 0.08 |
| Toxaphene 50 | B3a | 10 | 0 | 0.08 |
| Toxaphene 62 | B3a | 10 | 0 | 0.16 |
| heptachlor | B3a | 10 | 0 | 0.02 |
| mirex | B3a | 10 | 0 | 0.02 |
| oxychlordane | B3a | 10 | 0 | 0.08 |
| cis-heptachlorepoxide | B3a | 10 | 0 | 0.02 |
| trans-heptachlorepoxide | B3a | 10 | 0 | 0.05 |
| octachlorostyrene | B3a | 10 | 0 | 0.01 |
| trans-nonachlor | B3a | 10 | 0 | 0.01 |
| trans-chlordane (γ- chlordane) | B3a | 10 | 0 | 0.02 |
| cis-chlordane (α-chlordane) | B3a | 10 | 0 | 0.02 |
| **Lead** | B3c | 10 | 0 | 7 |
| **Cadmium** | B3c | 10 | 0 | 1 |
| **Mercury** | B3c | 10 | 0 | 2 |
| **Aflatoxins** | B3d | 6 | 0 | 0.01 |
| **Malachite Green** | B3e | 58 | 0 | 0.5 |
| Leuco Malachite Green | B3e | 58 | 0 | 0.5 |
| Crystal Violet | B3e | 58 | 0 | 0.5 |
| Leuco Crystal Violet | B3e | 58 | 0 | 0.5 |
| Victoria Blue | B3e | 58 | 0 | 0.5 |
| Brilliant Green | B3e | 58 | 0 | 0.5 |

[1] Action limit reference Appendix 1.
[2] Limit of Detection (LOD) for organochlorine compounds are averages as LOD is sample dependant.
[3] ICES 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.*
[4] Analysed by GC-ECD method
[5]Analysed by GC-MS method

### 4.2.1 Group A

A total of 71 (other stage of production and harvest) samples were tested for Group A substances and **no non-compliant results were obtained for these banned compounds;**

- **Group A3:**

48 tests on aquaculture samples were carried out for Group A3 Steroids

- **Methyltestosterone:** 38 samples analysed for methyltestosterone by screening Enzyme-Linked ImmunoSorbant Assay (ELISA) method.
- **17β-oestradiol:** 10 samples analysed for 17β-oestradiol by screening ELISA method.

The A3 tests were carried out on 43 individual samples in total, as some samples were tested for more than one of the above target substances.

All samples were compliant (i.e. no positive) for Group A3. However, two of four samples of fresh water trout (*O. mykiss*) which were sent to the RIKILT for GC-MSMS confirmatory analysis were found to have 17β-oestradiol present at trace levels. However, as 17β-oestradiol is a naturally occurring hormone and there is no information available as to the natural levels and variability in fish it was concluded that the presence of 17β-oestradiol at these trace levels was

most likely of a probable natural occurrence; therefore, these samples are reported as **compliant.**

**Therefore no non-compliant (i.e. no positive) results are reported for Group A3 samples.**

- **Group A6:**

In addition, 56 tests on aquaculture other stage of production and harvest samples were carried out for Group A6 substances as follows:

- **Chloramphenicol:** 38 samples analysed for chloramphenicol by screening ELISA method.
- **Nitrofurans:** 9 samples analysed for the marker metabolites of the nitrofurans; furazolidone, furaltadone, nitrofurantoin and nitrofurazone using a quantitative test (LCMSMS)
- **Nitroimidazole:** 9 samples analysed for nitroimidazole and its metabolites[1] by a quantitative test (LCMSMS)

The A6 tests were carried out on 45 individual samples in total, as some samples were tested for more than one of the above target substance groups.

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

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

### 4.2.2 Group B

A total of 128 samples (i.e. 124 target samples & 4 suspect samples) of farmed finfish were tested for Group B substances which can be classed as authorised substances, unauthorised substances or environmental contaminants. **All results were compliant with the exception of two harvest freshwater trout samples from one farm which was found to have sulphadiazine (Group B1- antibacterial substance) present in excess of the MRL**.

- **Group B1:**

Antibiotic Residues (Group B1) were screened using a qualitative screening method for the detection of quinolones, tetracyclines and florfenicol. **No non-compliant (i.e. no positive) results were obtained out of the 91 harvest samples tested for the above antibiotic residues.**

A total of 91 harvest samples were screened for sulphonamides by Immuno-assay (Evidence Investigator) and all samples were compliant with the exception of four harvest freshwater trout samples from one farm which screened as suspect positives and required confirmatory analysis. This LCMSMS confirmatory analysis was carried out by LGC, UK **and two samples were found to be non-compliant for sulphadiazine. i.e. sulphadiazine was confirmed to present in excess of the EU MRL** An investigation was carried out by the SFPA which included the Marine Institute taking 4 suspect samples. These 4 samples were tested by LGC, UK and found to be compliant.

- **Group B2:**

The following Group B2 groupings were analysed for the NRCP for Aquaculture; B2a Anthelmintics, B2c Pyrethroids, B2f other – Corticosteroids, B2f other veterinary drugs – such as insect growth regulators.

Twenty-seven samples (i.e. other stage of production and harvest) were screened for the following corticosteroids; dexamethasone, flumethasone and betamethasone and **no non-compliant results were obtained.**

The other veterinary drugs which were analysed as part of Group B2 are all generally authorised or unauthorised sea lice treatments these include; cypermethrin, deltamethrin (both group B2c), ivermectin, doramectin, emamectin B1a (both group B2a), teflubenzuron and diflubenzuron (both group B2f). 91 harvest samples were tested for each of these substances. **No non-compliant (i.e. no positive) results were obtained for Group B2 compounds.**

- **Group B3a:** Environmental Contaminants

**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. 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. Recent European legislation (Commission Regulation (EU) No 1259/2011 which came into force 1st January 2012 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. **None of the 19 harvest samples analysed exceeded the standard for the sum of ICES 6 PCBs (reference Table 6 for details of the number of samples tested and the concentration range).**

**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 with high levels especially found in marine mammals. A number of OSPAR contracting countries have set levels and Appendix 1 shows the available standards/guidance values in so far as Marine Institute is aware of these. **All the harvest samples analysed 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). 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 0.07 mg $kg^{-1}$ (wet weight). **All 10 harvest samples were reported as compliant for mercury.**

Cadmium, also an environmental contaminant, has a maximum limit set in fish of 0.05 mg $kg^{-1}$ (wet weight). Cadmium was not detected in any of the samples above 0.006 mg $kg^{-1}$ (wet weight). **All 10 harvest samples were reported as compliant for cadmium.**

Lead has a maximum limit set in fish of 0.3 mg $kg^{-1}$ (wet weight). Lead was not detected in any of the samples above 0.02 mg $kg^{-1}$ (wet weight). **All 10 harvest samples were reported as compliant for lead. Table 6 provides a breakdown of the number of samples tested and the concentration range for the samples tested.**

**Table 6**: Levels of mercury, cadmium, lead and PCBs

| | Maximum concentration | Median (Upper Bound mean) | Range | EC Maximum Limit | n |
|---|---|---|---|---|---|
| | mg $kg^{-1}$ ww | | | | |
| **Mercury** | 0.07 | 0.03 (0.03) | 0.02-0.07 | 0.5 | 10 |
| **Cadmium** | 0.006 | 0.005 (0.005) | 0.003-0.006 | 0.05 | 10 |
| **Lead** | 0.02 | 0.007 (0.012) | ND-0.02 | 0.3 | 10 |
| | µg $kg^{-1}$ ww | | | | |
| **ICES PCB 6[1]** | 18.8 | 7.74 (8.72) | 2.04-18.8 | 75 | 19 |

n: number sampled

[1]ICES PCB 6: sum of the following non-dioxin like PCBS-PCB 28, 52, 101, 138, 153, 180

- **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 2015 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 aflatoxins. The highest aflatoxin concentration obtained for the 6 harvest samples analysed was 0.07 µg $kg^{-1}$ (wet weight) for aflatoxin G2. **Currently there are no maximum limits set for aflatoxins in fish.**

- **Group B3e:** Dyes

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 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 $^{-1}$.

In 2014, in addition to routine testing for malachite green and its metabolite leuco malachite green testing was extended to include the following additional triphenylmethane dyes: brilliant green, crystal violet, leuco crystal violet, victoria blue. 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 58 target samples (i.e. 25 harvest and 33 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 2015 Border Inspection Posts Fishery and Fishery Product Sample Testing

***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 inspection 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 Inspection Post (BIP) that has been approved for importation. In Ireland, the responsibility for carrying out checks at the BIP (Dublin Port and Shannon Airport) is with the DAFM BIP Officers.

In 2015, BIP samples were collected by DAFM Sampling Officers and sent to the Marine Institute for testing in accordance with 2015 BIP plan (Appendix 6). In total 10 samples were sent to the Institute by the DAFM Sampling Officers at Dublin Port and Shannon Airport. The 2015 BIP results are presented in Table 6; **all samples were compliant.**

In addition, **2 Safeguard samples from India** were received from DAFM and required testing 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. **These samples were reported as compliant** (Table 7).

**Table 7:** 2015 Border Inspection Posts results for seafood samples tested at Marine Institute.

| MI CODE | DAFM Sample code | Product type | Substances for Identification | Result |
|---|---|---|---|---|
| **RESBIP 2015-5001** | ARA983059 | Lobster | Lead, cadmium, mercury | **Compliant** |
| **RESBIP 2015-5002** | ARA718318 | Canned Tuna | Cadmium | **Compliant** |
| **RESBIP 2015-5004** | ARA718332 | Frozen Shrimp | Malachite Green and other Dyes | **Compliant** |
| **RESBIP 2015-5005** | ARA718333 | Frozen Shrimp | Avermectins | **Compliant** |
| **RESBIP 2015-5007** | ARA718362 | Salmon | Malachite Green | **Compliant** |
| **RESBIP 2015-5008** | ARA718386 | Prawn | Mercury | **Compliant** |
| **RESBIP 2015-5010** | ARA718390 | Cooked Prawn | Antibacterials | **Compliant** |
| **RESBIP 2015-5011** | ARA718391 | Cooked Prawn | Avermectins | **Compliant** |
| **RESBIP 2015-5012** | ARA718396 | Raw Prawn | Malachite Green | **Compliant** |
| **RESBIP 2015-5013** | ARA718395 | Raw Prawn | Antibacterials | **Compliant** |

**Table 8:** 2015 Safeguard results.

| MI CODE | DAFM Sample code | Product type | Substances for Identification | Result |
|---|---|---|---|---|
| **RESBIP 2015-5003** | ARA718329 | Raw Prawn | Tetracyclines | **Compliant** |
| **RESBIP 2015-5006** | ARA718373 | Shrimp | Tetracyclines | **Compliant** |

## Appendix 1: Source of Maximum Residues Limits, Decision Limits and Guideline Values used for comparison with the results for 2015

| 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** | 0.4 µg $kg^{-1}$ | Decision Limit[3] |
| **Doramectin** | 0.4 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] |
| **ICES 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 mg $kg^{-1}$ | 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 and 565/2008/EC.
6. Maximum Residue Limits and Decision Limits concentration are on a wet weight basis.
7. ICES 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 for accreditation tests carried out by the Marine Institute

The following lists the reported analytes, which the Marine Institute are accredited for by the Irish National Accreditation Board (INAB) to ISO 17025 as detailed in Scope Registration Number 130T. For certain substance testing is carried out by other specialist laboratories in accordance with the annual place for Aquaculture (Appendix 5)

| Scope Registration Number 130T | | |
|---|---|---|
| **Analyte** | **SOP** | **Method** |
| • **Ivermectin, Emamectin B1a, Doramectin**[1] | (CHE-8) | UFLC-Flu |
| • **Mercury**[2] | (CHE-32) | CV-AFS |
| • **Teflubenzuron , Diflubenzuron**[1] | (CHE-42) | UFLC-DAD |
| • **Cadmium**[2, 3] | (CHE-178) | ICP-MS |
| • **Lead**[2] | (CHE-178) | ICP-MS |
| • **Screening of Antibiotic Residues in Fish**[1] | (FHU-1) | Modified EC 2-plate test |
| • **Screening of sulphadiazine**[1] | (FHU-119) | Immunoassay |
| • **Dyes**[1]**: Malachite Green, Crystal Violet, Victoria Blue, Leuco Crystal Violet, Leuco Malachite Green, Brilliant Green** | (CHE-167) | UFLC-MS/MS |
| • **Moisture**[2] **%** | (CHE-52) | See Appendix 3 |
| **When Collecting Samples the Laboratory Complies with Council Directive 96/23/EC** | (CHE-6) | |
| Notes:<br>1. Accreditation for finfish only<br>2. Accreditation for Marine Biota<br>3. BIP samples were tested prior to INAB accreditation for 07/10/15 | | |

## Appendix 3: Quality Control for tests carried out at the Marine Institute

To check the quality of the data produced during the 2015 National Surveillance Scheme for chemical residues in farmed fish, Quality Control (QC) samples in the form of either reagent blanks, replicates, spiked samples or Certified Reference Materials (CRMs) were analysed with each batch of samples tested by the Marine Institute. The quality assurance results obtained, as shown below ± 1 Standard Deviations (SD), 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.

| Analyte | QC Type | Target Value | Result ± SD | Mean Z – Score |
|---|---|---|---|---|
| • **Benzoylurea (µg kg$^{-1}$)** | | | | |
| Teflubenzuron | Spike (n=14) | 500 | 77.5 ± 5.9 % Recovery | -- |
| Diflubenzuron | Spike (n=14) | 1000 | 89.4 ± 9.0 % Recovery | -- |
| • **Pyrethroids (µg kg$^{-1}$) GC/ECD** | | | | |
| Cypermethrin | Spike (n=2) | 50 | 67.4 % Recovery | -- |
| Deltamethrin | Spike (n=2) | 10 | 89.7 % Recovery | -- |
| • **Pyrethroids (µg kg$^{-1}$) GC/MS** | | | | |
| Cypermethrin | Spike (n=21) | 50 | 99.8 ± 10.3% Recovery | -- |
| Deltamethrin | Spike (n=21) | 10 | 88.6 ± 21.6% Recovery | -- |
| • **Anthelmintics (µg kg$^{-1}$)** | | | | |
| Ivermectin | Spike (n=16) | 2 | 89.6 ± 7.8 % Recovery | -- |
| Emamectin B1a | Spike (n=16) | 100 | 82.0 ± 11.1 % Recovery | -- |
| Doramectin | Spike (n=16) | 2 | 100.1 ± 6.7 % Recovery | -- |
| • **Dyes (µg kg$^{-1}$)** | | | | |
| Brilliant Green | Spike (n=22) | 2 | 95.0 ± 13.9 % Recovery | -- |
| Crystal Violet | Spike (n=22) | 2 | 100.2 ± 5.3 % Recovery | -- |
| Leuco Crystal Violet | Spike (n=22) | 2 | 110.4 ± 14.0 % Recovery | -- |
| Leuco Malachite Green | Spike (n=22) | 2 | 105.9±11.3 % Recovery | -- |
| Malachite Green | Spike (n=22) | 2 | 100.6 ± 8.6 % Recovery | -- |
| Victoria Blue | Spike (n=20) | 2 | 101.6±15.7 % Recovery | -- |
| • **Chemical Elements (mg kg$^{-1}$ dry weight)** | | | | |
| Lead | SRM 2976 (n=1) | 1.19 | 112 % Recovery | 0.91 |
| Cadmium | SRM 2976 (n=1) | 0.82 | 105 % Recovery | 0.39 |
| Mercury | DORM2 (n=1) | 4.64 | 95.7% Recovery | -0.34 |
| Dry weight (%) | QTMO58BT (n=1) | 23.56 | 98.7 % Recovery | -0.10 |

**Note**: n is the sample number

## Appendix 4: Methods of Analysis

### 1.1 Sample Collection and Preparation

In accordance with the 2015 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 (<-20°C).

### 1.2 Dyes Analysis

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 Fast Liquid Chromatography coupled to Mass Spectrometry (UFLC-MS/MS).

### 1.3 Nitrofuran Analysis

The 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.

### 1.4 Screening for Group A Compounds & Group B Cortiscosteroids

Group A compounds were screened using the Elisa method, this testing was subcontracted out to Irish Equine Centre (IEC). This method is qualitative in nature and was used to detect residues of 17β-oestradiol, chloramphenicol and methyltesterone. Corticosteroids were also screened using this method. Where confirmatory analysis was required for 17 β-oestradiol the samples were tested by RIKILT Laboratories, Netherlands.

### 1.5 Nitroimidazoles

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.

### 1.6 Analysis for Cypermethrin and Deltramethrin

Approximately 2g of sample was extracted using a hexane/acetone mixture, followed by liquid/liquid partition and solid phase extraction techniques. The extract was then evaporated to dryness and reconstituted in 2,2,4-trimethylpentane. The analysis was carried out using Agilent 7890A Gas Chromatography coupled with electron capture detector (GC-ECD) with a Chrompack 15m CpSil 8 column.

During 2015 the analysis was transferred over to a new method, for details of this see below:
Approximately 2g of sample was extracted using a hexane/acetone mixture, followed by liquid/liquid partition and clean up using alumina and silica. The extract was then evaporated to dryness and reconstituted in 2,2,4-trimethylpentane. The analysis was carried out by Gas Chromatography coupled mass spectrometry (GCMS).

### 1.7 Analysis of Ivermectin, Doramectin and Emamectin B1a

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.

### 1.8 Analysis of Teflubenzuron and Diflubenzuron

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.

### 1.9 Antibacterial Substances

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. Analysis of Sulphonamide was carried out by Immunoassay, this method is qualitative in nature and tested on the Evidence Investigator instrument. Where confirmatory analysis was required for sulphonamides the samples were tested by LGC, UK.

### 1.10 Analysis for Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides (OCPs)

Analysis for Polycholorinated Biphenyls (PCBs) and Organochlorine Pesticides (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 (high resolution gas chromatography and high resolution mass spectrometry (HRGC/HRMS) using a DB-5 capillary column.

### 1.11 Cadmium and Lead Analysis

Concentrated nitric acid (4 ml) and hydrogen peroxide (4 ml) were added to approximately 0.2 g 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. Concentrations were determined by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS, Agilent 7700x with High Matrix Introduction (HMI) system).

### 1.12 Mercury Analysis

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 100 mls 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.

### 1.13 Moisture Content

The moisture content was determined by drying approximately 1g of tissue overnight in an oven at 104$^{o}$C to constant weight.

### 1.14 Analysis for Mycotoxins

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: 2015 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**

Dept of Agriculture, Food and Marine
Agriculture House
Kildare Street
Dublin 2

Sea-Fisheries Protection Authority
Block B
Clogheen
Clonakilty
Co. Cork

Marine Institute
Rinville
Oranmore
Co. Galway

**3. Staff resources to carry out plan**

Authorised Officers will collect all samples.

Group A substances will be performed by the Irish Equine Centre- Kildare, Laboratory of the Government Chemist-UK, Ashtown Food Research Centre-Dublin & EU-RL –RIKILT

Analyses for Group B substances will be performed within the Marine Institute with the exception of those indicated in the plan.

**4. Approved laboratories**

**Marine Institute (MI)**
Rinville
Oranmore
Co. Galway

**Irish Equine Centre (IEC)**
Johnstown
Naas
Co. Kildare

**Teagasc Food Research Centre (TFRC)**
Teagasc
Ashtown
Dublin 15

**Eurofins GfA GmbH,**
D-48161 Münster
Germany

**RIKILT EU-RL**
Laboratory for Residue analysis,
NL-3720 BA BILTHOVEN
The Netherlands

**Laboratory of the Government Chemist** (LGC)
Queens Road
Teddington
Middlesex
TW11 OLY, UK

**Wessling GmbH,**
Bochum
Germany

**ANSES EU-RL**
Fougeres
France

**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 2015

**Sampling levels and frequency**:

Minimum number of fish from which samples must be taken.

**Finfish.**

| Total Tonnes Produced 2013 | Total min. no. to be tested** | Min. no. Group A | Min. no. Group B |
|---|---|---|---|
| 10,033 | Production (tonnes)/100 =100 | 1/3 Total Tested = 33 | 2/3 Total Tested = 67 |
| | | | |

** min no. to be tested will be based on 2013 finfish production numbers as production numbers not available for 2014

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|
| **Group of Substances** | **Compounds** | **Matrix** | **Laboratory Method** | **CCbeta (screening) Detection capability** | **CCalpha (confirmatory) decision limit** | **Level of action** | **Number of samples** | **Laboratory** |
| Group A | | | | | | | | |
| A 3 Steroids[ж] | Methyltestosterone | Muscle & Skin | (1) ELISA<br>(2) GCMS | 1)1.5 µg $kg^{-1}$ | 2)0.5 µg $kg^{-1}$ | Presence | 38* | (1) IEC<br>(2) EU-RL |
| | 17β-Oestradiol | Muscle & Skin | (1) ELISA<br>(2) GCMS | 1)1.5 µg $kg^{-1}$ | 2)0.5 µg $kg^{-1}$ | 0.5 µg $kg^{-1}$ | 7* | (1) IEC<br>(2) EU-RL |
| A 6 Compounds included in Annex IV Council Reg. 2377/90 | Chloramphenicol | Muscle & Skin | (1) ELISA[∞]<br>(2) GCMS | 1)0.25 µg $kg^{-1}$<br>1)0.09 µg $kg^{-1}$[∞] | 2)0.19 µg $kg^{-1}$ | Presence | 38* | (1) IEC[∞]<br>(2) EU-RL |
| | Nitrofurans<br>AOZ<br>AMOZ<br>AHD<br>SEM | Muscle & Skin | UPLCMSMS | - | <br>0.041 µg $kg^{-1}$<br>0.061 µg $kg^{-1}$<br>0.057 µg $kg^{-1}$<br>0.064 µg $kg^{-1}$ | Presence | 9* | TFRC |
| | Nitroimidazoles<br>Dimetridazole<br>HMMNI<br>Metronidazole<br>Hydroxyl- Metronidazole<br>Ornidazole<br>Ronidazole<br>Ipronidazole<br>Hydroxyl-ipronidazole | Muscle & Skin | UPLCMSMS | - | <br>0.12 µg $kg^{-1}$<br>1.0 µg $kg^{-1}$<br>0.08 µg $kg^{-1}$<br>0.15 µg $kg^{-1}$<br>0.29 µg $kg^{-1}$<br>0.10 µg $kg^{-1}$<br>0.15 µg $kg^{-1}$<br>0.09 µg $kg^{-1}$ | Presence | 9* | TFRC |

* At least 50% of Group A are "on farm" samples

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

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

ж Corticosteroids: re-categorised as B2f

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|
| **Group of Substances** | **Compounds** | **Tissue** | **Laboratory Method** | **CCbeta (screening) Detection capability** | **CCalpha (confirmatory ) decision limit** | **Level of action** | **Number of samples** | **Laboratory** |
| B 1 Antibacterial substances | Microbiological screening:<br><u>Quinolones</u>:<br>-Oxolinic acid<br>-Flumequine<br><u>Tetracyclines:</u><br>-oxytetracycline<br><u>Florfenicol</u> | Muscle & Skin | Modified EC 2-plate method. | 75<br>150<br><br>50<br>250 | N/A | >MRL | 91 | MI |
| | Screening:<br><u>Sulphonamides</u><br>-Sulphadiazine | Muscle & Skin | 1)Immuno assay | 50 µg $kg^{-1}$ | N/A | >MRL | 91 | MI |
| | <u>Tetracycline</u><br><br>Chlortetracycline<br>Epi-Chlortetracycline<br>Oxytetracycline<br>Epi-Oxytetracycline<br>Tetracycline<br>Epi-Tetracycline<br>Doxycycline | Muscle & Skin | 1)LC-TOF<br>2)HPLC-DAD | <br><br>72 ug kg-1<br>75 ug kg-1<br>68 ug kg-1<br>81 ug kg-1<br>75 ug kg-1<br>78 ug kg-1<br>70 ug kg-1 | ^* | ^* | Confirmation and post screening identification of positive Microbiological Samples/Bioassay | 1)LGC∞∞<br>2) EU-RL |
| | <u>Quinolones</u><br><br>Ciprofloxacin<br>Enrofloxacin<br>Danofloxacin<br>Difloxacin<br>Flumequine<br>Oxolinic acid<br>Sarafloxacin | | 1)LC-TOF<br>2)HPLC-FLU | <br><br>74 ug kg-1<br>79 ug kg-1<br>79 ug kg-1<br>237 ug kg-1<br>396 ug kg-1<br>69 ug kg-1<br>** | ^* | ^* | | 1)LGC∞∞<br>2) EU-RL |

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

** Validation pending

∞∞ For screened positive samples for tetracyclines, quinolones, sulphonamides using MI in-house methods, these samples will be sent to subcontract laboratory LGC for further screening by LC-TOF

^* EU-RL calculates on the day of confirmatory analysis under ISO11843-2

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|
| Group of Substances | Compounds | Tissue | Laboratory Method | CCbeta (screening) Detection capability | CCalpha (confirmatory) decision limit | Level of action | Number of samples | Laboratory |
| | Sulphonamides<br>Sulphathiazole<br>Sulphaquinoxaline<br>Sulphapyridine<br>Sulphamethoxy-pyridazine<br>Sulphamonomethoxine<br>Sulphamethazine<br>Sulphamerazine<br>Sulphisoxazole<br>Sulphadimethoxine<br>Sulphadiazine<br>Sulphachlorpyridazine<br>Sulphamethizole | Muscle & Skin | 1)LC-TOF<br>2)LCMSMS | <br>82 ug $kg^{-1}$<br>78 ug $kg^{-1}$<br>74 ug $kg^{-1}$<br>78 ug $kg^{-1}$<br><br>50 ug $kg^{-1}$<br>72 ug $kg^{-1}$<br>91 ug $kg^{-1}$<br>73 ug $kg^{-1}$<br>76 ug $kg^{-1}$<br>84 ug $kg^{-1}$<br>74 ug $kg^{-1}$<br>70 ug $kg^{-1}$ | ** | ** | | 1)LGC[∞∞]<br>2) LGC |
| **B2 Other veterinary drugs** | | | | | | | | |
| B2 (a) Anthelmintics | Ivermectin | Muscle & Skin | UFLC-Flu | - | 0.4 µg $kg^{-1}$ | 0.4 µg $kg^{-1}$ | 91 | MI |
| | Emamectin B1a | | | - | 129µg $kg^{-1}$ | 129 µg $kg^{-1}$ | | |
| | Doramectin | | | - | 0.4 µg $kg^{-1}$ | 0.4 µg $kg^{-1}$ | | |
| B2 (c) Carbamates / Pyrethroids | Cypermethrin | Muscle & Skin | 1)GC-ECD<br>2)GC-MS | 1)41 µg $kg^{-1}$ | 2)65 µg $kg^{-1}$ | 65 µg $kg^{-1}$ | 91 | 1)MI<br>2)LGC |
| | Deltamethrin | | | 1)9 µg $kg^{-1}$ | 2)15 µg $kg^{-1}$ | 15 µg $kg^{-1}$ | | |
| B2 (f) Other Pharmacologically active substances | Teflubenzuron | Muscle & Skin | UFLC-DAD | - | 572 µg $kg^{-1}$ | 572 µg $kg^{-1}$ | 91 | MI |
| | Diflubenzuron | | | - | 1119 µg $kg^{-1}$ | 1119 µg $kg^{-1}$ | | |
| | Corticosteroids<br>Betamethasone<br>Dexamethasone<br>Flumethasone | Muscle & Skin | (1)ELISA<br>(2) LC-MS | 1)1.5 µg $kg^{-1}$<br>1.5 µg $kg^{-1}$<br>1.5 µg $kg^{-1}$ | 0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$ | 0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$ | 27* | (1) IEC<br>(2) EU-RL |

* At least 50% are "on farm" samples

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

**LGC under flexible scope of accreditation has an accredited procedure for the development and validation of methods in place in the event that a fish sample tested screen positive using the LC-TOF method. CC alpha will be calculated at that point

| 1 | 2 | 3 | 4 | 5 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|
| **Group of Substances** | **Compounds** | **Tissue** | **Laboratory Method** | **Detection limit** | **Level of action** | **Number of samples** | **Laboratory** |
| **B3 Other substances and environmental contaminants** | | | | | | | |
| B3 (a) Organochlorine compounds including PCBs | **PCBs**<br>CB Congener 28<br>CB Congener 52<br>CB Congener 101<br>CB Congener 118<br>CB Congener 138<br>CB Congener 153<br>CB Congener 180 | Muscle & Skin | GCHRMS | <br>0.07 µg $kg^{-1}$^^<br>0.07 µg $kg^{-1}$^^<br>0.07 µg $kg^{-1}$^^<br>0.01 µg $kg^{-1}$^^<br>0.07 µg $kg^{-1}$^^<br>0.07 µg $kg^{-1}$^^<br>0.07 µg $kg^{-1}$^^ | Excess of Guideline value | 19 | Eurofins |
| | **Chlorinated Pest.**<br>α-HCH<br>β-HCH<br>γ-HCH<br>δ-HCH<br>DDT-o,p'<br>DDT-p,p'<br>DDD-o,p'<br>DDD-p,p'<br>DDE-o,p'<br>DDE-p,p'<br>HCB<br>Aldrin<br>Dieldrin<br>Endrin<br>*cis*-Chlordane<br>*trans*-Chlordane<br>*oxy*chlordane<br>*trans*-Nonachlordane | | GCHRMS | <br>0.0625 µg $kg^{-1}$^^<br>0.0625 µg $kg^{-1}$^^<br>0.0625 µg $kg^{-1}$^^<br>0.0625 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.125 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.0375 µg $kg^{-1}$^^<br>0.075 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.125 µg $kg^{-1}$^^<br>0.025 µg $kg^{-1}$^^<br>0.125 µg $kg^{-1}$^^ | Excess of Guideline value | 10 | |
| B3 (c) Chemical elements | Lead | | GFAAS | 8 µg $kg^{-1}$ | 300 µg $kg^{-1}$ | 10 | MI |
| | Cadmium | | GFAAS | 2 µg $kg^{-1}$ | 50 µg $kg^{-1}$ | 10 | |
| | Mercury | | CVAFS | 8 µg $kg^{-1}$ | 500 µg $kg^{-1}$ | 10 | |
| B3 (d) Mycotoxins | Aflatoxin B1 | Muscle & Skin | HPLC-Flu | 0.01 µg $kg^{-1}$ | - | 6 | Wessling |
| | Aflatoxin B2 | | | 0.01 µg $kg^{-1}$ | | | |
| | Aflatoxin G1 | | | 0.01 µg $kg^{-1}$ | | | |
| | Aflatoxin G2 | | | 0.01 µg $kg^{-1}$ | | | |

^^detection limit is at limit of quantification for PCBs and OCPs

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|
| **Group of Substances** | **Compounds** | **Tissue** | **Laboratory Method** | **CCbeta (screening) Detection capability** | **CCalpha (confirmatory) decision limit** | **Level of action** | **Number of samples** | **Laboratory** |
| B3 (e) Dyes | Malachite Green (MG)<br>Leuco Malachite Green (LMG)<br>Brilliant Green (BG)<br>Crystal Violet (CV)<br>Leuco Crystal Violet (LCV)<br>Victoria Blue (VB) | Muscle & Skin | UFLCMSMS | -<br>- | 0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$ | 0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$<br>0.5 µg $kg^{-1}$ | 58#<br>(17 salmon/sea trout, 8 Freshwater trout harvest, 8 Freshwater trout osop, 25 salmon smolts) | MI |

#41 of the 58 samples for dyes are “on farm” samples.

## Appendix 6: Annual Plan for Sampling Fishery Products and Other Seafood at Border Inspection Posts

# Dublin Port 2015

| 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 (See Note 1 below). | **Eurofins Food Testing Ireland Ltd**<br>**Unit D13 North City Business Park**<br>**North Road**<br>**Dublin 11**<br>**Phone: 01 431 1306**<br>**Email: info@eurofins.ie**<br><br>**Please quote Quotation reference 12-7384P when submitting samples.**<br><br>**For results & analytical queries contact : Anna Coffey**<br>**Email : AnnaCoffey@eurofins.ie**<br><br>**Eurofins offer a free collection service from Dublin. See Annex II.**<br><br>**For queries on sample submission / collection:**<br>**Tel: 01 – 4311306**<br>**Email : samplereception@eurofins.ie**<br><br>**Please give lab as much notice as possible prior submission of sample.** |
| | 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 (See Note 2 below). | ***Dr. Brenda Lennon***<br>***Executive Chemist,***<br>***Public Analyst's Laboratory,***<br>***Seamus Quirke Road,***<br>***Galway.***<br>***Tel: 091-581122***<br>***Fax: 091-581212***<br>***E-mail:* Brenda.Lennon@hse.ie**<br><br>**Please phone lab prior to submission of samples.** |
| **A.6** | Nitrofuran metabolites | Nitrofurazone, Nitrofurantoin, Nitrofurans, Furazolidone, Furaltadone | 4 aquaculture samples | **Dr. Martin Danaher,**<br>**Food Safety Department,**<br>**Ashtown Food Research Centre,**<br>**Teagasc, Ashtown, Dublin 15.**<br>**Tel: 01 8059500**<br>**Fax: 01 8059550**<br>**martin.danaher@teagasc.ie** |

| | | | | |
|---|---|---|---|---|
| **A.6** | Chloramphenicol | Chloramphenicol | 2 aquaculture samples | **Prof. Tom Buckley,<br>Irish Equine Centre,<br>Johnstown,<br>Naas,<br>Co. Kildare<br>Telephone: 045 866266<br>Fax: 045 866 273<br>tbuckley@equine-centre.ie** |
| **B.1** | Antibacterial substances<br>General 2 plate test &Immuno assay | Antibacterial substances | 3 aquaculture samples<br>(See Note 3) | **Denise Glynn,<br>Residues Scientific Coordinator,<br>Marine Environment & Food Safety Services,<br>Marine Institute,<br>Rinville,<br>Oranmore,<br>Galway.<br>Direct: 091-387339<br>Reception: 091-387200<br>Fax: 091-387201<br>denise.glynn@marine.ie<br><br>Please ensure lab receive prior notice where possible** |
| **B.2.a** | Anthelmintics (Avermectins) | Emamectin,<br>Ivermectin<br>Doramectin | 2 aquaculture samples<br>(See Note 3) | |
| **B.3.e** | Dyes | Malachite Green (MG)<br>Leuco Malachite Green (LMG)<br>Brillant Green (BG)<br>Crystal Violet (CV)<br>Leuco Crystal Violet (LCV)<br>Victoria Blue (VB) | 4 aquaculture samples<br>(See Note 3) | |
| **B.3.d** | Chemical - Heavy Metals<br>*(Specify Pb, Cd, or Hg*) | Pb Lead<br>Hg Mercury<br>Cd Cadmium | 2 fish samples<br>(See Note 3) | |
| | Sulphur Dioxide and 4-Hexylresorcinol | | 2 prawn /shrimp samples (1kg approx. per sample)<br><br>Cork PAL can facilitate Sulphur Dioxide and 4-Hexylresorcinol analyses of prawns in the following sampling periods in 2015:<br><br>January 19th – 30th<br>February 10th – 21st<br>June 9th – 20th<br>July 20th – 31st<br>November 10th - 21st<br>These sampling periods are in conjunction with SFPA and HSE sampling so where possible if these dates could be met please, however PAL will strive to fit in the samples at other times, but the turnaround times may be longer. | **Dr. Fred Davidson<br>Cork Public Analyst's Laboratory<br>St Finbarr's Hospital<br>Cork<br>Public Analyst.<br>Fred.Davidson@hse.ie<br>Tel: 021 4923245<br>Fax: 021 4923367<br><br>Please copy sfpafoodsafety@sfpa.ie when emailing Fred to inform him of sample submission.** |

# Shannon Airport 2015

| Group | Test | TRACES residue 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 | 2 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 (See Note 1 below). | **Complete Laboratory Solutions (CLS)**<br>**Ros Muc**<br>**Connemara**<br>Co. Galway<br>**Tel: 091 574355**<br>**Fax: 091 574356**<br>**Email : microfoodandwater@cls.ie**<br>**For scheduling/testing/reporting requirements contact Olivia Ryan Email: oryan@cls.ie**<br>**For quality related queries phone: 091574355**<br><br>**Please quote Quotation reference 8143 when submitting samples.**<br><br>**CLS operate a weekly collection service from Limerick. See Annex II.**<br><br>**Please give lab as much notice as possible prior submission of sample.** |
| **B.3.d** | Chemical - Heavy Metals *(Specify Pb, Cd, or Hg*) | Pb Lead<br>Hg Mercury<br>Cd Cadmium | 2 fish or crustacean samples For live lobster samples can BIP officers please freeze the sample before sending to MI for heavy metal analysis. (See Note 3) | **Denise Glynn,**<br>**Residues Scientific Coordinator,**<br>**Marine Environment & Food Safety Services,**<br>**Marine Institute,**<br>**Rinville,**<br>**Oranmore,**<br>**Galway.**<br>**Direct: 091-387339**<br>**Reception: 091-387200**<br>**Fax: 091-387201**<br>**denise.glynn@marine.ie** |

## ANNEX I – NOTES SAMPLING & ANALYSIS

**1. Notes on Microbiological Sampling and Analysis**
The aim of sampling should be to verify conformity with microbiological criteria set for certain food types in Regulation 2073/2005 (Annex 1 Chapter 1).
(It is possible for more than one criterion to be applicable to a food, e.g. both *Listeria monocytogenes* and *Salmonella* criteria can apply to cooked crustaceans/ shellfish.)

Micro. Criteria 1.2 and 1.3
*Listeria monocytogenes* enumeration in Ready-to-eat (RTE) food (Food Safety Criteria).
Useful targets here might be RTE smoked or gravid fish (e.g. smoked salmon) or RTE dried fish (e.g. dried catfish) or cooked (and possibly chilled or frozen) fishery products (e.g. cooked tuna, cooked shrimp, cooked prawn, cooked lobster, cooked crab/crabmeat or cooked mussels).
**(Testing is not required for canned fish or other RTE fishery products heat treated in their final package. Testing is not required for RTE Live Bivalve Molluscs.)**
The legislation requires the testing of 5 sample units from the same batch (n=5).
The limit is 100 cfu/g.
The batch is satisfactory if all samples units are less than or equal to 100 cfu/g
The batch is unsatisfactory if one or more sample units is >100 cfu/g.
(A different criterion applies to RTE foods intended for infants and to RTE foods for special medial purposes. See Reg. 2073/2005 Annex 1 Chapter 1 Micro Criterion 1.1 for full details.)

Micro. Criterion 1.16
*Salmonella* detection in cooked crustaceans and molluscan shellfish (Food Safety Criterion).
Useful targets here would be cooked (and possibly chilled or frozen) shrimp, prawn, lobsters, crab/crabmeat or mussels.
The legislation requires the testing of 5 sample units from the same batch (n=5).
The batch is satisfactory if a result of absent is obtained for all 5 sample units.
The batch is unsatisfactory if a result of present is obtained for one or more sample units.

Micro. Criteria 1.17 and 1.25
*Salmonella* detection and *E. coli* enumeration (Most Probable Number, MPN) in live bivalve molluscs, echinoderms, tunicates and gastropods (Food Safety Criteria). Entry of these animals live for human consumption at Irish BIPs is rare.

For *Salmonella*,
The legislation requires the testing of 5 sample units from the same batch (n=5).
The batch is satisfactory if a result of absent is obtained for all 5 sample units.
The batch is unsatisfactory if a result of present is obtained for one or more sample units.

For *E. coli*
The legislation requires the testing of a pooled sample of a minimum of 10 animals, (i.e. n = a pooled sample of a minimum of 10 animals).
The sample is satisfactory if a result of less than or equal to 230 MPN/100g of flesh and intra-valvular liquid is obtained.
The sample is unsatisfactory if a result of >230 MPN/100g of flesh and intra-valvular liquid is obtained.

Process Hygiene Criteria
Process Hygiene criterion for *E. coli* and Coagulase positive Staphylococci in shelled and shucked products of cooked crustaceans and molluscan shellfish are set in Regulation 2073/2005 (Annex 1 Chapter 2 Criteria 2.4.1).

These are process hygiene criteria applicable at the end of the manufacturing process, so while conformity at the BIP stage is consistent with effective implementation of these criteria, non-conformity with these criteria in BIP samples does not necessarily indicate non-conformity with requirements. Therefore we propose that BIP samples focus on the Food Safety Criteria outlined above rather than the Process Hygiene Criteria.

**2. Note on Histamine Sampling**

Histamine Micro Criteria 1.26, 1.27 and 1.27a (Food Safety Criteria) are also set in Regulation 2073/2005.

Micro Criterion 1.26 sets a histamine criterion for fishery products from fish species associated with a high amount of histidine.
Micro Criterion 1.27 sets a histamine criterion for fishery products, except those in food category 1.27a, which have undergone enzyme maturation treatment in brine, manufactured from fish species associated with a high amount of histidine.
Micro Criterion 1.27a sets a histamine criterion for fish sauce produced by fermentation of fishery products

The fish species associated with a high amount of histidine (as mentioned in Micro Criteria 1.26 and 1.27) are particularly species of the families *Scombridae, Clupeidae, Engraulidae, Coryfenidae, Pomatomidae, Scombresosidae. (Scombridae* include mackerels, tunas and bonitos; *Clupeidae* include herrings, shads and sardines*; Engraulidae* include anchovies*; Coryphaenidae* are dolphinfishes; *Pomatomidae* are Bluefish*; Scomberesocidae* include sauries).
Typical products of relevance entering Irish BIPs include mackerel, tuna, herring, sardines or anchovies. It would be useful to target cooked, canned or raw variants of these species.

Micro Criterion 1.27a is applicable to any fish sauce produced by fermentation of fishery products (i.e. not just those from the fish species associated with a high amount of histidine).

For 1.26 histamine criterion applicable to fishery products from fish species associated with a high amount of histidine:
The histamine criterion has a lower limit, m=100mg/kg and an upper limit, M=200 mg/kg.
n=9 (i.e. 9 sample units (fish / packs /cans) to be tested from a batch).
The number of sample units allowed to exceed the lower limit (m), is 2 (so called c=2).
A batch is satisfactory if the **mean** of all 9 values is less than or equal to m (i.e. less than or equal to 100mg/kg) and a maximum of 2 of 9 sample units are between m & M (i.e. >100 mg/kg & less than or equal to 200 mg/kg) and no value is >M (i.e. >200 mg/kg).
A batch is unsatisfactory if the mean is > m (i.e. mean >100mg/kg) or if more than 2 of 9 samples are between m & M (i.e. >100 mg/kg & less than or equal to 200 mg/kg) or if 1 or more values are >M (i.e. >200 mg/kg).

For 1.27 histamine criterion applicable to fishery products, except those in food category 1.27a, which have undergone enzyme maturation treatment in brine, manufactured from fish species associated with a high amount of histidine:
The criterion has a lower limit, m=200mg/kg and an upper limit, M=400 mg/kg. n=9; c=2.
A batch is satisfactory if the mean of all 9 values is less than or equal to m (i.e. less than or equal to 200mg/kg) and a maximum of 2 of 9 sample units are between m & M (i.e. >200 mg/kg & less than or equal to 400 mg/kg) and no value is >M (i.e. >400 mg/kg).
A batch is unsatisfactory if the mean is > m (i.e. mean. > 200mg/kg) or if more than 2 of 9 samples are between m & M (i.e. >200 mg/kg & less than or equal to 400 mg/kg) or if 1 or more values >M (i.e. >400 mg/kg).

For 1.27a histamine criterion applicable to fish sauce produced by fermentation of fishery products
The histamine criterion has a single limit of 400mg/kg (i.e. m=M=400mg/kg).
n=1 (i.e. 1 sample unit (packs /can /jar) to be tested from a batch).
A batch is satisfactory if the result is less than or equal to 400mg/kg.

A batch is unsatisfactory if the result is greater than 400mg/kg.

**3. Notes on Chemical Sampling and Analysis**

The Marine Institute has INAB accreditation for the following matrices: Salmon and trout; Marine Biota (Shellfish and Finfish species). If other species are sampled, they will be tested, but INAB accreditation will not be claimed.

Note: Samples from frozen consignments are to be kept frozen prior to dispatch and during dispatch if possible. Samples should arrive at laboratory within 48 hours of dispatch.