



# Agri-Food & Biosciences Institute

## VETERINARY SCIENCES DIVISION

### Chemical and Immunodiagnostic Sciences Branch

#### Annual Report UK National Reference Laboratory For Marine Biotoxins

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#### Contacts:

##### **Cowan Higgins**

Chemical and Immunodiagnostic Sciences  
Branch

Tel 02890 525785

Email [cowan.higgins@afbini.gov.uk](mailto:cowan.higgins@afbini.gov.uk)

##### **Alastair Douglas**

Head of Chemical and Immunodiagnostic Sciences  
Branch

Tel 02890 525 813

Email [alastair.douglas@afbini.gov.uk](mailto:alastair.douglas@afbini.gov.uk)

## **Glossary**

**AFBI:** Agri-Food and Biosciences Institute

**ASP:** Amnesic Shellfish Poison (Domoic Acid)

**Cefas:** Centre for Environment, Fisheries and Aquaculture Science

**DSP:** Diarrhetic Shellfish Poison (Lipophilic Toxin group)

**EURL-MB:** European Reference Laboratory for Marine Biotoxins

**FSA:** Food Standards Agency

**CEN:** Comité Européen de Normalisation (European Committee for Standardization)

**CCMAS:** Codex Committee on Methods of Analysis and Sampling

**DG-SANCO:** Directorate-General for Health and Consumers

**HO:** Home Office

**HPLC-FLD:** High Performance Liquid Chromatography with fluorescence detection

**LC-MS/MS:** Liquid Chromatography coupled with tandem Mass Spectrometry

**MBA:** Mouse bioassay

**OCL:** Official Control Laboratory

**PSP:** Paralytic Shellfish Poison (Saxitoxin group)

**SAMS:** The Scottish Association for Marine Sciences

**SOP:** Standard Operating Procedure

**UK-NRL:** United Kingdom National Reference Laboratory

## **Introduction**

This report provides an outline of the work of the UK-NRL over the financial year 2014-2015. It is not a comprehensive review but highlights some of the areas to which it has contributed throughout the year. The UK-NRL acknowledges the support of the FSA and the help of AFBI and Cefas in fulfilling its duties.

For the purposes of Regulation (EC) 882/2004 regarding Official Feed and Food Controls, the FSA is designated as the Competent Authority and as such the FSA is responsible for establishing the location and boundaries of classified production and relaying areas for live bivalve molluscs. It has responsibility for the organisation of official controls including the organisation of statutory monitoring for the presence of marine biotoxins in shellfish and toxin-producing phytoplankton in the classified production and relaying areas. The appointment of the UK-NRL for marine biotoxins is also the responsibility of the FSA. The role of the NRL is to carry out the requirements and duties set out in Article 33 of Regulation (EC) 882/2004, namely:

1. Collaborate with the European EURL-MB in their area of competence.
2. Co-ordinate, for their area of competence, the activities of official laboratories responsible for the analysis of samples.
3. Where appropriate, organise comparative tests between the official national laboratories and ensure an appropriate follow-up of such comparative testing.
4. Ensure the dissemination to the competent authority and official national laboratories of information that the EURL-MB supplies.
5. Provide scientific and technical assistance to the competent authority for the implementation of co-ordinated control plans adopted in accordance with Article 33.
6. Be responsible for carrying out other specific duties provided for in accordance with the procedure referred to in Article 33 without prejudice to existing additional national duties.

### **Co-ordination of the Activities of the Monitoring Laboratories**

Following changes to the Coleman transportation boxes used in the monitoring programme, the UK NRL issued an updated Standard Operating Procedure for the Transport and Storage of samples prior to testing. The UKNRL acknowledged that further amendment may be required to the sections relating to the storage of samples prior to testing if the requirement for bioassays is removed from the monitoring programme.

The introduction of a new proficiency test database (WEPAL) by Quasimeme caused some issues in the extraction of data from the new format of report generated by Quasimeme. The new format was implemented in April 2014 however the revised format made it virtually impossible to transfer data to a “trend monitoring” spreadsheet in any way other than manually re-typing the data. Quasimeme received numerous e-mails, including a response on behalf of the UK participants by the NRL, stating the preference for a Z-score Certificate to be made available to participants. It was pointed out to Quasimeme that the certificates were useful in reviewing External Quality Control performance during accreditation audits. The issue was discussed at the annual Scientific Assessment Board meeting but to date there has been no revision of the format.

The FSA hosted the 24<sup>th</sup> meeting of the UKNRL-Network group, held in London on the 13<sup>th</sup> May 2014. The 25<sup>th</sup> meeting of the Network group was held at AFBI, Belfast on the 14<sup>th</sup> November 2014.

## EURL MB Proficiency Tests

The EURL-MB evaluates the performance of the EU NRLs and checks the equivalency of the methods used by the laboratories for the official control of marine biotoxins in bivalve molluscs through annual proficiency exercises for PSP, DSP and ASP. Proficiency exercises for PSP have been organised since 2004. The number of participants in 2014 was 35 and the exercise covered both biological methods and HPLC-FLD. For ASP, proficiency exercises have been organised since 2007 to evaluate method and laboratory performance, with participants requested to use the method usually employed for official control. In 2014, there were 31 participants. For lipophilic toxins, the EURL-MB has organised proficiency exercises since 2000. A total of 33 laboratories participated in the 2014 study.

Reports on the EURL-MB proficiency tests and have been circulated throughout the year and the results are summarised below. The full reports are available for download from either the EURL-MB or the UK-NRL website. The EURL-MB was able to extend the proficiency tests to include both the UK-NRL and Cefas laboratories in 2014 and results from both laboratories have been circulated to the UK NRL Network and discussed in full at the Network meeting held in November 2014. Both laboratories participate in the Quasimeme Proficiency test programme and the full report is circulated to the NRL network prior to each Network Meeting.

A z-score is calculated for each participant's data for each matrix / determinand combination which is given an assigned value. The z-score is calculated as follows:

$$z - \text{score} = \frac{\text{Mean from Laboratory} - \text{Assigned Value}}{\text{Total Error}}$$

Total Error

$|Z| < 2$  Satisfactory performance (95.4% of z-scores)

$2 < |Z| < 3$  Questionable performance (4.3% of z-scores)

$|Z| > 3$  Unsatisfactory performance (0.3% of z-scores)

## Domoic Acid 2014 Proficiency Test Summaries

### AFBI EURL 2014

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
EURLMB/14/A/01	Mussel homogenate	20.4	20.0	mg/kg	-0.19
EURLMB/14/A/02	Scallop homogenate	10.8	11.2	mg/kg	0.30

### AFBI Quasimeme Round 2014.1

Sample ID	Sample description	Round	Assigned value	Reported value	Units	Z-score
Sample 1	Standard solution	2014.1	2.002	2.10	mg/Kg	0.33
Sample 2	Mussel Homogenate	2014.1	29.47	28.88	mg/Kg	-0.16
Sample 3	Oyster Homogenate	2014.1	18.08	18.89	mg/Kg	0.35

### AFBI Quasimeme Round 2014.2

Sample ID	Sample description	Round	Assigned value	Reported value	Units	Z-score
Sample 1	Standard solution	2014.2	1.01	1.03	mg/Kg	0.10
Sample 2	Mussel Homogenate	2014.2	42.04	44.80	mg/Kg	0.51
Sample 3	Scallop Homogenate	2014.2	28.71	33.40	mg/Kg	1.28

## PSP 2014 Proficiency Test Summaries

### AFBI EURL 2014

Sample ID	Method	Matrix	Determinand	Assigned Value	Reported Value	Units	Z-Score
EURLMB/14/P/01	HPLC	Mussel	C1C2	19	14	µmol/kg	-1.15
EURLMB/14/P/01	HPLC	Mussel	dcSTX	172	140	µmol/kg	-0.86
EURLMB/14/P/01	HPLC	Mussel	GTX2,3	259	177	µmol/kg	-1.48
EURLMB/14/P/01	HPLC	Mussel	GTX5 (B1)	16	13	µmol/kg	-0.79
EURLMB/14/P/01	HPLC	Mussel	STX	87	76	µmol/kg	-0.55
<b>EURLMB/14/P/01</b>	<b>HPLC</b>	<b>Mussel</b>	<b>Total STX</b>	<b>562</b>	<b>420</b>	<b>ugSTX2HCL equiv/Kg</b>	<b>-1.36</b>
EURLMB/14/P/02	HPLC	Mussel	GTX2,3	383	265	µmol/kg	-1.67
EURLMB/14/P/02	HPLC	Mussel	STX	798	710	µmol/kg	-0.63
<b>EURLMB/14/P/02</b>	<b>HPLC</b>	<b>Mussel</b>	<b>Total STX</b>	<b>1203</b>	<b>976</b>	<b>ugSTX2HCL equiv/Kg</b>	<b>-1.10</b>
EURLMB/14/P/02	MBA	Mussel		841	849	ugSTX2HCL equiv/Kg	0.06
EURLMB/14/P/03	HPLC	Razor clam	dcGTX2,3	72	56	µmol/kg	-0.88
EURLMB/14/P/03	HPLC	Razor clam	C1C2	80	70	µmol/kg	-0.58
EURLMB/14/P/03	HPLC	Razor clam	dcSTX	1333	1204	µmol/kg	-0.56
EURLMB/14/P/03	HPLC	Razor clam	GTX5 (B1)	270	243	µmol/kg	-0.50
EURLMB/14/P/03	HPLC	Razor clam	GTX6 (B2)	97	104	µmol/kg	0.33
<b>EURLMB/14/P/03</b>	<b>HPLC</b>	<b>Razor clam</b>	<b>Total STX</b>	<b>1972</b>	<b>1678</b>	<b>ugSTX2HCL equiv/Kg</b>	<b>-0.94</b>
EURLMB/14/P/03	MBA	Razor clam		1632	1527	ugSTX2HCL equiv/Kg	-0.40

## AFBI Quasimeme Round 2014.1

Sample ID	Round	Method	Determinand	Sample description	Assigned Value	Reported Value	Units	z-score
Sample 1	2014.1	HPLC	STX	Mussel Homogenate	0.097	<0.056	µmol/kg	<
Sample 1	2014.1	HPLC	dc-STX	Mussel Homogenate	1.961	1.68	µmol/kg	-0.95
Sample 1	2014.1	HPLC	GTX5, (B1)	Mussel Homogenate	0.199	<0.16	µmol/kg	<
Sample 1	2014.1	<b>HPLC</b>	<b>Total</b>	<b>Mussel Homogenate</b>	<b>750.4</b>	<b>625</b>	<b>µgSTXdiHCleq./kg</b>	<b>-1.32</b>
Sample 2	2014.1	HPLC	dcGTX-2,3	Oyster Homogenate	10.24	7.224	µmol/kg	-2.27
Sample 2	2014.1	HPLC	dc-Neo	Oyster Homogenate	1.463	1.252	µmol/kg	-0.91
Sample 2	2014.1	HPLC	dc-STX	Oyster Homogenate	3.462	3.18	µmol/kg	-0.58
Sample 2	2014.1	<b>HPLC</b>	<b>Total</b>	Oyster Homogenate	3045	2445	<b>µgSTXdiHCleq./kg</b>	<b>-1.57</b>
Sample 3	2014.1	HPLC	STX	Mussel Homogenate	2.608	2.452	µmol/kg	-0.41
Sample 3	2014.1	HPLC	GTX-2,3	Mussel Homogenate	1.207	1.048	µmol/kg	-0.79
Sample 3	2014.1	<b>HPLC</b>	<b>Total</b>	<b>Mussel Homogenate</b>	<b>1289</b>	<b>1147</b>	<b>µgSTXdiHCleq./kg</b>	<b>-0.87</b>
Sample 4	2014.1	HPLC	STX	Oyster Homogenate	1.936	1.712	µmol/kg	-0.77
Sample 4	2014.1	HPLC	GTX-2,3	Oyster Homogenate	0.0923	0	µmol/kg	0.00
Sample 4	2014.1	HPLC	GTX-2,3	Oyster Homogenate	2.412	2.164	µmol/kg	-0.70
Sample 4	2014.1	HPLC	GTX-1,4	Oyster Homogenate	0.7408	0.756	µmol/kg	0.11
Sample 4	2014.1	<b>HPLC</b>	<b>Total</b>	<b>Oyster Homogenate</b>	<b>1569</b>	<b>1402</b>	<b>µgSTXdiHCleq./kg</b>	<b>-0.85</b>

## Lipophilic Toxins 2014 Proficiency Test Summaries

### AFBI EURL 2014

Sample ID	Method	Matrix	Determinand	Assigned Value	Reported Value	Units	Z-Score
EURLMB/14/L/02	MBA	Mussel	N/A	POSITIVE	POSITIVE	N/A	N/A
EURLMB/14/L/03	MBA	Mussel	N/A	POSITIVE	POSITIVE	N/A	N/A
EURLMB/14/L/01	LCMS:MS	Mussel	YTX	0.45	0.46	mg/kg	0.1
EURLMB/14/L/01	LCMS:MS	Mussel	Homo YTX	0.47	0.631	mg/kg	1.7
<b>EURLMB/13/L/01</b>	<b>LCMS:MS</b>	<b>Mussel</b>	<b>Total YTX group</b>	<b>0.89</b>	<b>1.09</b>	<b>mg YTX eq./kg</b>	<b>1.3</b>
<b>EURLMB/14/L/02</b>	<b>LCMS:MS</b>	<b>Mussel</b>	<b>All toxins considered</b>	<b>&lt;LOQ</b>	<b>&lt;LOQ</b>	<b>µg/kg</b>	
EURLMB/14/L/03	LCMS:MS	Mussel	Total Free OA group	91.7	79.9	µgOA eq./kg	-0.6
EURLMB/14/L/03	LCMS:MS	Mussel	Total OA+PTX group	258.4	252.3	µgOA eq./kg	-0.1
EURLMB/14/L/03	LCMS:MS	Mussel	AZA1	191.5	197.2	µg/kg	0.1
EURLMB/14/L/03	LCMS:MS	Mussel	AZA2	58.9	59.3	µg/kg	0.0
EURLMB/14/L/03	LCMS:MS	Mussel	AZA3	74.6	84.3	µg/kg	0.6
<b>EURLMB/14/L/03</b>	<b>LCMS:MS</b>	<b>Mussel</b>	<b>Total AZA group</b>	<b>401.6</b>	<b>422</b>	<b>µg AZA eq./kg</b>	<b>0.3</b>

### AFBI Quasimeme Round 2014.1

Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
Sample 1	AZA Standard Solution	AZA-1	10.92	9.2	µg/kg	-1.21
<b>Sample 1</b>	<b>AZA Standard Solution</b>	<b>AZA-total</b>	<b>10.77</b>	<b>9.2</b>	<b>µgAZA eq/kg</b>	<b>-1.13</b>
Sample 2	DSP/AZP Extract Solution	AZA-1	16.05	15.5	µg/kg	-0.27
Sample 2	DSP/AZP Extract Solution	AZA-2	3.618	3.1	µg/kg	-1.03
Sample 2	DSP/AZP Extract Solution	AZA-3	5.049	5.8	µg/kg	1.1
<b>Sample 2</b>	<b>DSP/AZP Extract Solution</b>	<b>AZA-total</b>	<b>28.51</b>	<b>29.2</b>	<b>µgAZA eq/kg</b>	<b>0.19</b>
Sample 2	DSP/AZP Extract Solution	Free-DTX2	27.11	25	µg/kg	-0.61
Sample 2	DSP/AZP Extract Solution	Free-OA	13.13	12.4	µg/kg	-0.43
Sample 2	DSP/AZP Extract Solution	Total-free-OA+DTX1+DTX 2	31.25	27.4	µgOA eq/kg	-0.97
Sample 2	DSP/AZP Extract Solution	Total-DTX2	51.35	52.1	µg/kg	0.12
Sample 2	DSP/AZP Extract Solution	Total-OA	42.16	45.2	µg/kg	0.57
<b>Sample 2</b>	<b>DSP/AZP Extract Solution</b>	<b>Total-hy-OA+DTX1+DTX 2</b>	<b>73.56</b>	<b>76.5</b>	<b>µgOA eq/kg</b>	<b>0.32</b>
Sample 2	DSP/AZP Extract Solution	Total OA + PTX	70.98	76.5	µgOA eq/kg	0.62
Sample 3	Mussel Homogenate	AZA-1	1105	1206.5	µg/kg	0.74
Sample 3	Mussel Homogenate	AZA-2	285.2	317.7	µg/kg	0.91
Sample 3	Mussel Homogenate	AZA-3	196.1	221.1	µg/kg	1.02
<b>Sample 3</b>	<b>Mussel Homogenate</b>	<b>AZA-total</b>	<b>1907</b>	<b>2089.9</b>	<b>µgAZA eq/kg</b>	<b>0.77</b>
Sample 3	Mussel Homogenate	Free-DTX1	250.9	238.1	µg/kg	-0.41
Sample 3	Mussel Homogenate	Free-DTX2	90.22	76.5	µg/kg	-1.21
Sample 3	Mussel Homogenate	Free-OA	160	171.9	µg/kg	0.59
Sample 3	Mussel Homogenate	Total-free-OA+DTX1+DTX 2	489.3	455.9	µgOA eq/kg	-0.55
Sample 3	Mussel Homogenate	Total-DTX1	426.9	457.7	µg/kg	0.58
Sample 3	Mussel Homogenate	Total-DTX2	213.3	216	µg/kg	0.10
Sample 3	Mussel Homogenate	Total-OA	526.5	574.3	µg/kg	0.73
<b>Sample 3</b>	<b>Mussel Homogenate</b>	<b>Total-hy-OA+DTX1+DTX 2</b>	<b>1082</b>	<b>1161.6</b>	<b>µgOA eq/kg</b>	<b>0.59</b>
Sample 3	Mussel Homogenate	Total OA + PTX	1051	1161.6	µgOA eq/kg	0.84
Sample 3	Mussel Homogenate	YTX	0.1625	0.1511	mg/kg	-0.16
Sample 3	Mussel Homogenate	45-OH-homo-YTX	NA	0.0804	mg/kg	0
<b>Sample 3</b>	<b>Mussel Homogenate</b>	<b>Total-YTX</b>	<b>0.1822</b>	<b>0.2315</b>	<b>mg YTXeq./kg</b>	<b>0.68</b>
Sample 4	Mussel Homogenate	AZA-1	116.7	106.2	µg/kg	-0.72
Sample 4	Mussel Homogenate	AZA-2	35.95	35.2	µg/kg	-0.17
Sample 4	Mussel Homogenate	AZA-3	22.56	26.9	µg/kg	1.51
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>AZA-total</b>	<b>215.9</b>	<b>207.1</b>	<b>µgAZA</b>	<b>-0.33</b>

Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
					eq/kg	
Sample 4	Mussel Homogenate	Free-DTX1	75.14	67.1	µg/kg	-0.85
Sample 4	Mussel Homogenate	Free-DTX2	477.1	415	µg/kg	-1.04
Sample 4	Mussel Homogenate	Free-OA	91.99	81.3	µg/kg	-0.93
Sample 4	Mussel Homogenate	Total-free-OA+DTX1+DTX2	476.5	397.4	µgOA eq/kg	-1.33
Sample 4	Mussel Homogenate	Total-DTX1	109.2	100	µg/kg	-0.67
Sample 4	Mussel Homogenate	Total-DTX2	729.4	611	µg/kg	-1.3
Sample 4	Mussel Homogenate	Total-OA	214.9	199.1	µg/kg	-0.59
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>Total-hy-OA+DTX1+DTX2</b>	<b>790.6</b>	<b>665.7</b>	<b>µgOA eq/kg</b>	<b>-1.26</b>
Sample 4	Mussel Homogenate	Total OA + PTX	742.8	665.7	µgOA eq/kg	-0.83
Sample 4	Mussel Homogenate	YTX	1.025	0.9553	mg/kg	-0.39
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>Total-YTX</b>	<b>0.9967</b>	<b>0.9553</b>	<b>mg YTXeq./kg</b>	<b>-0.24</b>

### AFBI Quasimeme Round 2014.2

Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
Sample 1	AZA Standard Solution	AZA-1	18.01	17.3	µg/kg	-0.31
<b>Sample 1</b>	<b>AZA Standard Solution</b>	<b>AZA-total</b>	<b>18.09</b>	<b>17.3</b>	<b>µgAZA eq/kg</b>	<b>-0.34</b>
Sample 2	Lipo Standard Solution	Free-DTX2	33.96	35.24	µg/kg	0.30
Sample 2	Lipo Standard Solution	Free-OA	57.97	56.70	µg/kg	-0.17
<b>Sample 2</b>	<b>Lipo Standard Solution</b>	<b>Total-free-OA+DTX1+DTX2</b>	<b>79.35</b>	<b>77.85</b>	<b>µgOA eq/kg</b>	<b>-0.15</b>
Sample 3	DSP/AZP Extract Solution	AZA-1	69.11	70.00	µg/kg	0.10
Sample 3	DSP/AZP Extract Solution	AZA-2	19.50	21.10	µg/kg	0.64
Sample 3	DSP/AZP Extract Solution	AZA-3	15.40	15.20	µg/kg	-0.13
<b>Sample 3</b>	<b>DSP/AZP Extract Solution</b>	<b>AZA-total</b>	<b>123.60</b>	<b>129.30</b>	<b>µgAZA eq/kg</b>	<b>0.37</b>
Sample 3	DSP/AZP Extract Solution	Free-DTX1	10.06	10.40	µg/kg	0.26
Sample 3	DSP/AZP Extract Solution	Free-DTX2	38.76	39.90	µg/kg	0.23
Sample 3	DSP/AZP Extract Solution	Free-OA	12.52	10.40	µg/kg	-1.31
<b>Sample 3</b>	<b>DSP/AZP Extract Solution</b>	<b>Total OA + PTX</b>	<b>94.38</b>	<b>92.50</b>	<b>µgOA eq/kg</b>	<b>-0.31</b>
Sample 3	DSP/AZP Extract Solution	Total-DTX1	13.93	14.00	µg/kg	0.04
Sample 3	DSP/AZP Extract Solution	Total-DTX2	63.36	67.90	µg/kg	0.57
<b>Sample 3</b>	<b>DSP/AZP Extract Solution</b>	<b>Total-free-OA+DTX1+DTX2</b>	<b>46.66</b>	<b>44.70</b>	<b>µgOA eq/kg</b>	<b>-0.33</b>
<b>Sample 3</b>	<b>DSP/AZP Extract Solution</b>	<b>Total-hy-OA+DTX1+DTX2</b>	<b>96.23</b>	<b>92.50</b>	<b>µgOA eq/kg</b>	<b>-0.16</b>
Sample 3	DSP/AZP Extract Solution	Total-OA	41.33	37.80	µg/kg	-0.68
Sample 4	Mussel Homogenate	45-OH-YTX	N/A	0.078	mg/kg	N/A
Sample 4	Mussel Homogenate	AZA-1	1100.00	1104.20	µg/kg	0.03



<b>Sample ID</b>	<b>Sample Description</b>	<b>Determinand</b>	<b>Assigned Value</b>	<b>Reported Value</b>	<b>Units</b>	<b>Z Score</b>
Sample 4	Mussel Homogenate	AZA-2	279.40	292.50	µg/kg	0.38
Sample 4	Mussel Homogenate	AZA-3	189.20	195.40	µg/kg	0.26
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>AZA-total</b>	<b>1887.00</b>	<b>1904.20</b>	<b>µgAZA eq/kg</b>	<b>0.07</b>
Sample 4	Mussel Homogenate	Free-DTX1	237.10	253.00	µg/kg	0.54
Sample 4	<b>Mussel Homogenate</b>	Free-DTX2	<b>87.28</b>	<b>84.10</b>	µg/kg	<b>-0.29</b>
Sample 4	Mussel Homogenate	Free-OA	169.90	156.30	µg/kg	-0.64
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>Total OA + PTX</b>	<b>1104.00</b>	<b>946.10</b>	<b>µgOA eq/kg</b>	<b>-1.19</b>
Sample 4	Mussel Homogenate	Total-DTX1	434.10	415.30	µg/kg	-0.35
Sample 4	Mussel Homogenate	Total-DTX2	224.20	176.40	µg/kg	-1.70
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>Total-free-OA+DTX1+DTX2</b>	<b>465.50</b>	<b>459.70</b>	<b>µgOA eq/kg</b>	<b>-0.10</b>
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>Total-hy-OA+DTX1+DTX2</b>	<b>1111.00</b>	<b>946.10</b>	<b>µgOA eq/kg</b>	<b>-1.14</b>
Sample 4	Mussel Homogenate	Total-OA	535.10	425.00	µg/kg	-1.64
<b>Sample 4</b>	<b>Mussel Homogenate</b>	<b>Total-YTX</b>	<b>0.1834</b>	<b>0.2406</b>	<b>mgYTXeq /kg</b>	<b>0.78</b>
Sample 4	Mussel Homogenate	YTX	0.1292	0.1626	mg/kg	-0.55
Sample 5	Mussel Homogenate	AZA-1	130.9	137.6	µg/kg	0.41
Sample 5	Mussel Homogenate	AZA-2	44.94	46.6	µg/kg	0.29
Sample 5	Mussel Homogenate	AZA-3	32.87	32.90	µg/kg	0.01
<b>Sample 5</b>	<b>Mussel Homogenate</b>	<b>AZA-total</b>	<b>252.20</b>	<b>267.70</b>	<b>µgAZA eq/kg</b>	<b>0.49</b>
Sample 5	Mussel Homogenate	Free-DTX2	539.50	496.20	µg/kg	-0.64
Sample 5	Mussel Homogenate	Free-OA	94.78	86.10	µg/kg	-0.73
<b>Sample 5</b>	<b>Mussel Homogenate</b>	<b>Total OA + PTX</b>	<b>758.00</b>	<b>684.20</b>	<b>µgOA eq/kg</b>	<b>-0.89</b>
Sample 5	Mussel Homogenate	Total-DTX2	845.00	774.30	µg/kg	-0.67
<b>Sample 5</b>	<b>Mussel Homogenate</b>	<b>Total-free-OA+DTX1+DTX2</b>	<b>454.40</b>	<b>383.80</b>	<b>µgOA eq/kg</b>	<b>-1.24</b>
<b>Sample 5</b>	<b>Mussel Homogenate</b>	<b>Total-hy-OA+DTX1+DTX2</b>	<b>769.80</b>	<b>684.20</b>	<b>µgOA eq/kg</b>	<b>-0.78</b>
Sample 5	Mussel Homogenate	Total-OA	236.80	219.70	µg/kg	-0.58
<b>Sample 5</b>	<b>Mussel Homogenate</b>	<b>Total-YTX</b>	<b>1.1920</b>	<b>1.0920</b>	<b>mgYTXeq /kg</b>	<b>-0.50</b>
Sample 5	Mussel Homogenate	YTX	1.2030	1.0920	mg/kg	-0.55

### Summary

For the EURL and Quasimeme proficiency tests for lipophilic toxins, the National Reference Laboratory reported results with 100% of the Z scores less than 2 (satisfactory). For PSP, 97% of Z scores were less than 2 and 3% were between 2 and 3 (questionable). The questionable result related to an individual toxin analogue whilst all figures for Total Toxicity were less than 2. For ASP (Domoic Acid) all results had satisfactory z-scores.

## **Dissemination of Information from the EURL-MB**

Minutes and reports from the EURL Working Groups and Network workshop were circulated as they became available, to the UK Network Group. In response to a report of a study submitted to DGSANCO by the Spanish Competent Authority (AECOSAN), the EURL Working Group on LC-MS/MS for lipophilic toxins focused on the application of the method to cooked and processed shellfish. The EURL standard operating procedure for lipophilic toxins by LC-MS/MS was modified to cover the application of the method to cooked and processed shellfish. The UKNRL provided the EURL with information on the application of measurement uncertainty, in response to discussions at the EURL Workshop where it became clear that the application of uncertainty varied significantly across member states.

The use of the mouse bioassay for the determination of lipophilic toxins ceased from the 31<sup>st</sup> December 2014, and in a response to a request from the EURL on behalf of DGSANCO, the NRL confirmed that the mouse bioassay would no longer be used for the detection of lipophilic toxins.

The annual EURL Network workshop was held in Lisbon on the 23<sup>rd</sup> – 24<sup>th</sup> October 2014 and saw the reintroduction of presentations by the participants on the work of their NRL. This was an attempt by the EURL to encourage more wide-spread participation by the NRLs. The UK NRL gave an overview of the role of the NRL within the UK and outlined some of the work it had undertaken during the year.

### **Provide Scientific and Technical Assistance to the Competent Authority**

The NRL undertook a peer review on behalf of the FSA on Project FS616037, a critical review of the current evidence for the use of indicator shellfish species for the purposes of biotoxin and chemical contaminant monitoring.

In a response to a request from FSA clarification on the application of measurement uncertainty was provided by the NRL. The information explained the current application of measurement uncertainty to results obtained for lipophilic toxins by LC-MS/MS.

The NRL continued to provide technical information to the FSA on the application of the mouse bioassay for the determination of PSP. The information provided background information for

the Competent Authority during discussions by the CCMAS Working Party on the status of bioassays.

Discussions continued within the CEN Working Group on Marine Toxins on the revision of the protocols for Domoic Acid and PSP. New draft methods were prepared and discussed at the meeting of the Working Group in May 2014.

From the 31<sup>st</sup> December 2014, the mouse bioassay can no longer be used for the detection of lipophilic toxins but may be used for the periodic detection of emerging toxins. In response to this change in the use of the MBA, the NRL sought information from the EURL on the extent to which emerging toxins are being monitored for and in particular the use of the MBA for such monitoring. Initial information indicates that France and some Scandinavian countries may be using the MBA for some periodic monitoring.

The NRL organised two Network meetings, comprising representation from the FSA and monitoring laboratories. An invitation to attend was extended to the NRL-Ireland (Marine Institute) who attended the meeting in Belfast and updated the Group on monitoring for Azaspiracid-producing phytoplankton.

#### Meetings Attended

Date	Venue	Subject
13 <sup>th</sup> May 2014	London	UK NRL Network Meeting
27 <sup>th</sup> May 2014	Berlin	CEN WG on Marine Biotoxins
10 <sup>th</sup> June 2014	Vigo	EURL WG on LC-MS/MS
22 <sup>nd</sup> October 2014	Lisbon	EURL WG on LC-MS/MS
23-24 <sup>th</sup> October 2014	Lisbon	EURL Workshop meeting
7 <sup>th</sup> November 2014	Dublin	SafeFood Biotoxin meeting
14 <sup>th</sup> November 2014	Belfast	UK NRL Network Meeting
27 <sup>th</sup> January 2015	Vigo	EURL WG on LC-MS/MS
11 <sup>th</sup> February 2015	Brussels	CCMAS Working Party meeting on Marine Biotoxins

#### Links

UK-NRL Web page:

<http://www.afbini.gov.uk/index/services/services-diagnostic-and-analytical/marine-biotoxins-nrl.htm>

The following document was out of date and was removed from the UK NRL Marine Biotoxin web-site during 2014-2015

Documents added to the web-site over the same period;

EURL-MB Web page:

<http://aesan.msssi.gob.es/en/CRLMB/web/home.shtml>

EURL-MB Work Programme 2014

[http://aesan.msssi.gob.es/CRLMB/docs/docs/program\\_de\\_trabajo\\_anual/EURLMB-Work\\_Program-2014.pdf](http://aesan.msssi.gob.es/CRLMB/docs/docs/program_de_trabajo_anual/EURLMB-Work_Program-2014.pdf)