



Agri-Food & Biosciences Institute

VETERINARY SCIENCES DIVISION

Chemical Surveillance Branch

Annual Report UK National Reference Laboratory For Marine Biotoxins

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Glossary

AFBI: Agri-Food and Biosciences Institute

ASP: Amnesic Shellfish Poison (Domoic Acid)

Cefas: Centre for Environment, Fisheries and Aquaculture Science

LTs: Lipophilic toxins (including Diarrhetic Shellfish Poison (DSP) group)

EURL-MB: European Reference Laboratory for Marine Biotoxins

FSA: Food Standards Agency

SOP: Standard Operating Procedure

HPLC-FLD: High Performance Liquid Chromatography with fluorescence detection

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

OCL: Official Control Laboratory

PSP: Paralytic Shellfish Poison (Saxitoxin group)

TTX: Tetrodotoxin

SAMS: The Scottish Association for Marine Sciences

SOP: Standard Operating Procedure

PTs: Proficiency Tests

UK-NRL: United Kingdom National Reference Laboratory

eWG: Electronic working group

LOQ: Limit of quantification

Introduction

This report provides an outline of the work of the UK-NRL over the financial year 2016-2017. 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. A summary of the 2016 UK-NRL work programme is provided in Appendix 1.

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.

Summary of Meetings attended 2016-2017

The table below provides a summary of meetings attended as part of the NRL activities that took place during 2016-2017.

Meetings Attended 2016-2017

Date	Venue	Subject
12 th April 2016	Teleconference	EURL Phytoplankton Working Group (Madrid)
9 th May 2016	Teleconference	UK TTX meeting (FSA co-ordinated)
26 th May 2016	Teleconference	UK TTX meeting (FSA co-ordinated)
14 th June 2016	Madrid	LC-MS/MS working group
27 th June 2016	Teleconference	28 th UK NRL Network Meeting
4-8 th September 2016	Baiona	Emerging toxins workshop
25-26 th October 2016	Porto	EURL/NRL Annual workshop
10 th November 2016	London	29 th UK NRL Network Meeting

Collaboration with the EURL-MB

In 2016-2017, AFBI as UKNRL continued to participate in the following EURL co-ordinated working groups: (1) LC-MS/MS working group (2) Toxic phytoplankton working group.

The EURL convened an LC-MS/MS Working Group (WG) meeting in June 2016 to discuss and agree revisions to the EU-RL reference method for lipophilic toxin analysis by LC-MS/MS. AFBI as UK-NRL submitted recommendations prior to the meeting and during a formal presentation. The update will be reflected in version 6 of the SOP and published on the EURL website when finalised.

The phytoplankton working group was established with the aim of harmonising sampling methodology, the list of toxic species to be identified and thresholds used. Much of the work of this WG is conducted electronically with meetings as required to agree final draft documents. The UK-NRL co-ordinated all UK input/responses received from official control laboratory experts and submitted these to Dr. Pablo Serrat, chair of the Working Group, with one meeting attended by teleconference on 12th April 2016. The EU-RL has highlighted the necessity of the experts participating in the WG for Phytoplankton to communicate and report any input to the relevant NRL, as this activity remains under NRLs co-ordination.

Co-ordination of the Activities of the Monitoring Laboratories

The NRL organised two UK Network meetings, comprising representation from the FSA and monitoring laboratories. The 28th meeting of the UKNRL-Network group was conducted by teleconference on the 27th June 2016. FSA hosted the 29th meeting of the UKNRL-Network group in London on the 10th Nov 2016.

In 2016, the UK-NRL lobbied for the inclusion of Cefas in EURL proficiency testing (PT) exercises, however the EURL only offered participation to NRLs (this had also been the case in 2015). This was discussed at both UK network meetings in 2016. Tissue extracts and remaining samples from the EURL PT scheme were circulated to Cefas after the closure of the EURL test submission deadline. The final report for this PT was then forwarded to Cefas to allow them to retrospectively evaluate their performance in the EURL PT scheme. The UK-NRL collated a summary of results for PT exercises undertaken by the UK official control laboratories and these were circulated and discussed at UK network meetings in 2016.

The UK-NRL participated in a collaborative pre-trial being organised by Cefas for the analysis of PSP group toxins and TTX/epi-TTX by LC-MS/MS. The purpose of the pre-trial was to determine ease of transfer of the method between laboratories, to highlight any issues or difficulties prior to any subsequent inter-laboratory validation study, and to assess method performance between laboratories. Cefas provided documents which aided in the transfer of the method from Cefas to the NRL laboratory. Pre-Trial samples were analysed at the NRL in May 2016 and results submitted to Cefas. Cefas issued a pre-trial summary report in June 2016 showing good general agreement of UK-NRL results with those obtained at Cefas.

AFBI as UK-NRL co-ordinated a review of the BSI standard with the current NRL SOP for monitoring of toxic phytoplankton species. This was conducted with input from the Official control laboratories at AFBI, SAMS and Cefas. The UK-NRL also co-ordinated responses received from official control laboratory experts in the UK to documents circulated through the EURL phytoplankton WG, with an agreement on sampling methodologies being completed in 2016.

Proficiency Tests (PTs)

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 LTs, PSP and ASP.

- For Lipophilic Toxins (LTs), the EURL-MB has organised PTs since 2000. In total 21 laboratories participated, 19 of which were EU-NRLs and 2 OC laboratories from third countries.
- PT exercises for PSP have been organised since 2004. The exercise covered both biological methods and HPLC-FLD. The number of participants in 2016 was 23, of which 19 were EU-NRLs, with 4 official control (OC) laboratories from third countries also submitting results.
- 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 2016, there were 20 participants, of which 17 were EU-NRLs and 3 OC laboratories from third countries.

Reports on the EURL-MB proficiency tests are circulated in October and discussed at the annual EURL-NRL meeting. The results obtained by the UK-NRL are summarised in Appendix 1 and are available on the UK-NRL website (full reports generated by the EURL are confidential). The EURL-MB was not able to extend the PTs to include additional EU laboratories other than NRLs from 2015. The EURL has indicated that this was at the request of the European Commission due to budgetary pressures. Results obtained by the UK-NRL have been circulated to the UK NRL Network and were discussed in full at the Network meeting held in November 2016.

Both UK laboratories participate in the Quasimeme Proficiency test programmes for ASP/PSP/LTs and in the International Phytoplankton Intercomparison (Bequalm) taxonomic quiz. A full summary report is circulated to the NRL network prior to each Network Meeting. The results obtained by the UK-NRL are summarised in Appendix 1.

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

$2 < |Z| < 3$ Questionable performance

$|Z| > 3$ Unsatisfactory performance

Proficiency test summary

For the EURL and Quasimeme proficiency tests in 2016 for lipophilic toxins, the National Reference Laboratory reported results for individual toxins with 88% of Z scores less than 2 (satisfactory), 11.1% of the Z scores less than 3 (questionable) and 0.9% of the Z scores as >3 (1 result out of 116 narrowly reported as unsatisfactory with a z-score = -3.1; for DTX2 toxin total result). All EURL PT results were acceptable with all but one of the Z scores >2 being reported in Quasimeme round 2 results. Individual toxin results >2 were investigated and reviewed as part of the laboratories internal quality procedures, and all relate to the hydrolysed (total) OA group analysis. Overall, none of the total toxicity values were unsatisfactory, with no impact on reported results.

For PSP, 85% of individual toxin Z scores were less than 2, 7.5% between 2 to 3, and 7.5% above 3. Individual toxin results >2 were investigated and reviewed as part of the laboratories internal quality procedures. Overall, all total toxicity results were within acceptable ranges, with no impact on reported results.

For ASP (Domoic Acid) all results returned satisfactory z-scores.

All results for the International Phytoplankton Intercomparison (Bequalm) taxonomic quiz returned satisfactory z-scores.

Dissemination of Information from the EURL-MB & Provision of Scientific and Technical Assistance to the Competent Authority

Minutes and reports from EURL Working Groups and workshops attended were discussed at the UK network meetings and further information circulated on request.

The UK-NRL responded to FSA requests to discuss Dutch national measures that were introduced for Tetrodotoxin (TTX) control in bivalve molluscs in the spring of 2016, a subsequent draft UK risk assessment (produced by FSA) and UK lines at EU level. The UK-NRL attended two meetings by teleconference in respect of this, one held prior to FSA attendance at the Bivalve Molluscs Working Group meeting on 10th May 2016, and a second on the 26th May 2016. The UK-NRL agreed to co-ordinate sub-sampling and extraction of Northern Ireland (NI) samples for TTX from 31/5/16 to 19/9/16. The extracts were subsequently sent to Cefas for TTX analysis to allow inclusion of NI survey results with UK data that had been generated to date for England, Scotland and Wales.

The EURL convened an LC-MS/MS Working Group (WG) meeting in June 2016 to discuss and agree revisions to the EU-RL reference method for lipophilic toxin analysis by LC-MS/MS. AFBI as UK-NRL submitted recommendations prior to the meeting and during a formal presentation. A summary of these discussions was presented at the UK-NRL network meeting on the 27th June 2016. The NRL informed the group that the update is not expected to have any significant impact on UK monitoring laboratories. The update will be reflected in version 6 of the SOP and published on the EURL website when finalised.

The UK-NRL attended an Emerging toxins workshop held in Baiona, Spain from the 4-8th September 2016. A summary was presented by the NRL at the UK-NRL network meeting on the 10th November 2016 and is included with the minutes of that meeting.

The UK-NRL attended the annual EURL/NRL workshop on 25-26th October 2016 in Porto. The performance of NRLs in the 2016 EURL PT schemes, a review of the 2016 work programme of the EURL and activities for 2017 were presented by the EURL. The UK-NRL gave a presentation on its NRL activities for 2016 and undertook responsibility for producing minutes of that meeting. A number of issues were discussed by NRLs, including: Annex C of the harmonised procedure for lipophilic toxins for treatment of processed shellfish; harmonisation of LOQ (calculation and expression) for reporting of results to the EURL PT schemes; TTX occurrence in Europe and the pending EFSA opinion.

Links

UK-NRL Web page:

The NRL website and associated links can be accessed through the following link:

<https://www.afbini.gov.uk/articles/united-kingdom-national-reference-laboratory-marine-biotoxins>

Updated link to EURL website:

<http://www.aecosan.msssi.gob.es/en/CRLMB/web/home.html>

Appendix 1

Domoic Acid 2016 Proficiency Test Summaries

AFBI EURL 2016

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
EURLMB/16/A/01	Scallop homogenate	12.2	12.7	mg/kg	0.4
EURLMB/16/A/02	Scallop homogenate	9.6	9.2	mg/kg	-0.3

AFBI Quasimeme Round 2016.1

Sample No	Sample ID	Sample description	Determinand	Assigned value	Reported value	Units	z-score
Sample 1	QST202 SS	Standard solution	Total Domoic + Epi DA	1.30	1.24	mg/Kg	-0.28
Sample 2	QST203 BT	Oyster Homogenate	Total Domoic + Epi DA	18.57	19.18	mg/Kg	0.26
Sample 3	QST204 BT	Scallop Homogenate	Total Domoic + Epi DA	30.80	36.85	mg/Kg	1.55

AFBI Quasimeme Round 2016.2

Sample No	Sample ID	Sample description	Determinand	Assigned value	Reported value	Units	z-score
Sample 1	QST214 SS	Standard solution	Total Domoic + Epi DA	0.481	0.473	mg/Kg	-0.07
Sample 2	QST215 SS	Oyster Homogenate	Total Domoic + Epi DA	28.1	30.9	mg/Kg	0.80
Sample 3	QST216 SS	Scallop Homogenate	Total Domoic + Epi DA	19.3	21.2	mg/Kg	0.80

PSP 2016 Proficiency Test Summaries

AFBINI EURL 2016

Sample ID	Method	Matrix	Determinand	Assigned Value	Reported value	Units	Z-Score
EURLMB/16/P/01	MBA	Mussels	Total STX	545.7	452	ugSTX2HCL equiv/Kg	-1.00
EURLMB/16/P/01	HPLC	Mussels	GTX2,3	317.6	205.5	umol/kg	-1.70
EURLMB/16/P/01	HPLC	Mussels	STX	547.5	405	umol/kg	-1.50
EURLMB/16/P/01	HPLC	Mussels	Total STX	848.4	610.5	ugSTX2HCL equiv/Kg	-1.70
EURLMB/16/P/02	MBA	Mussels	NEGATIVE			ugSTX2HCL equiv/Kg	
EURLMB/16/P/02	HPLC	Mussels	NEGATIVE			ugSTX2HCL equiv/Kg	
EURLMB/16/P/03	HPLC	Mussels	dcGTX2&3	140.3	260.8	umol/kg	2.40
EURLMB/16/P/03	HPLC	Mussels	C1&2	326.6	254.3	umol/kg	-1.10
EURLMB/16/P/03	HPLC	Mussels	dcSTX	188.4	119.1	umol/kg	-1.80
EURLMB/16/P/03	HPLC	Mussels	GTX5	208.6	144.7	umol/kg	-1.40
EURLMB/16/P/03	HPLC	Mussels	C3,4	96.1	32.5	umol/kg	-2.10
EURLMB/16/P/03	HPLC	Mussels	GTX6	140	130.4	umol/kg	-0.30
EURLMB/16/P/03	HPLC	Mussels	Total STX	1084	941.9	ugSTX2HCL equiv/Kg	-0.70

AFBI Quasimeme Round 2016.1

Sample No	Sample ID	Method	Determinand	Sample description	Assigned value	Reported value	Units	z-score
Sample 1	QST210BT	HPLC	C-1,2	Mussel Homogenate	0.348	0.324	µmol/kg	-0.26
Sample 1	QST210BT	HPLC	dc-STX	Mussel Homogenate	0.968	0.760	µmol/kg	-1.22
Sample 1	QST210BT	HPLC	GTX-1,4	Mussel Homogenate	0.928	0.378	µmol/kg	-3.31
Sample 1	QST210BT	HPLC	GTX-2,3	Mussel Homogenate	1.345	0.916	µmol/kg	-1.97
Sample 1	QST210BT	HPLC	NEO	Mussel Homogenate	0.111	0.308	µmol/kg	3.09
Sample 1	QST210BT	HPLC	STX	Mussel Homogenate	0.332	0.296	µmol/kg	-0.39
Sample 1	QST210BT	HPLC	Total toxicity	Mussel Homogenate	1091	864	µgSTXdiHCleq./kg	-1.65
Sample 2	QST211BT	HPLC	Total toxicity	Oyster Homogenate	NEG	NEG	µgSTXdiHCleq./kg	NA
Sample 3	QST212BT	HPLC	dc-STX	Oyster Homogenate	0.091	0.072	µmol/kg	-0.30
Sample 3	QST212BT	HPLC	GTX-1,4	Oyster Homogenate	0.749	0.568	µmol/kg	-1.26
Sample 3	QST212BT	HPLC	GTX-2,3	Oyster Homogenate	2.291	1.924	µmol/kg	-1.09
Sample 3	QST212BT	HPLC	NEO	Oyster Homogenate	0.221	0.196	µmol/kg	-0.33
Sample 3	QST212BT	HPLC	STX	Oyster Homogenate	1.763	1.660	µmol/kg	-0.38
Sample 3	QST212BT	HPLC	Total toxicity	Oyster Homogenate	1371	1359	µgSTXdiHCleq./kg	-0.07
Sample 4	QST213BT	HPLC	GTX-2,3	Mussel Homogenate	3.131	2.324	µmol/kg	-1.83
Sample 4	QST213BT	HPLC	STX	Mussel Homogenate	1.768	1.552	µmol/kg	-0.80
Sample 4	QST213BT	HPLC	Total toxicity	Mussel Homogenate	1285	1096	µgSTXdiHCleq./kg	-1.17

Lipophilic 2016 Proficiency Test Summaries

AFBINI EURL 2016

Sample ID	Method	Matrix	Determinand	Assigned Value	Reported value	Units	Z-Score
EURLMB/16/L/01	LC-MS/MS	Mussel		NEGATIVE	NEGATIVE		
EURLMB/16/L/02	LC-MS/MS	Mussel	Total OA group	440.0	320.8	OA equivalents /kg	-1.4
EURLMB/16/L/02	LC-MS/MS	Mussel	Free OA	187.2	159.3	Free OA µg/kg	-0.7
EURLMB/16/L/02	LC-MS/MS	Mussel	Free DTX2	116.4	99.8	Free DTX-2 µg/kg	-0.6
EURLMB/16/L/02	LC-MS/MS	Mussel	Total OA	349.8	256.6	Total OA µg/kg	-1.3
EURLMB/16/L/02	LC-MS/MS	Mussel	Total DTX-2	159.9	107.0	Total DTX-2 µg/kg	-1.4
EURLMB/16/L/02	LC-MS/MS	Mussel	Total YTX group	2.19	1.94	YTX equivalents /kg	-0.7
EURLMB/16/L/02	LC-MS/MS	Mussel	YTX	0.14	0.15	YTX mg/kg	0.2
EURLMB/16/L/02	LC-MS/MS	Mussel	Homo-YTX	1.33	1.06	Homo-YTX mg/kg	-1.3
EURLMB/16/L/02	LC-MS/MS	Mussel	45-OH-YTX	0.18	0.17	45-OH-YTX mg/kg	-0.3
EURLMB/16/L/02	LC-MS/MS	Mussel	45-OH-Homo-YTX	1.18	1.11	45-OH-Homo-YTX mg/kg	-0.3
EURLMB/16/L/03	LC-MS/MS	Mussel	Total OA group	178.4	134.7	Total OA µg/kg	-1.1
EURLMB/16/L/03	LC-MS/MS	Mussel	Free OA	66.5	56.9	Free OA µg/kg	-0.6
EURLMB/16/L/03	LC-MS/MS	Mussel	Free DTX-2	52.1	47.0	Free DTX-2 µg/kg	-0.4
EURLMB/16/L/03	LC-MS/MS	Mussel	Total OA	138.7	101.1	Total OA µg/kg	-1.1
EURLMB/16/L/03	LC-MS/MS	Mussel	Total DTX-2	73.5	55.9	Total DTX-2 µg/kg	-1.0
EURLMB/16/L/03	LC-MS/MS	Mussel	Total AZA group	463.2	464.3	AZA1 equivalents /kg	0.0
EURLMB/16/L/03	LC-MS/MS	Mussel	AZA 1	294.4	300.9	AZA1 µg/kg	0.1
EURLMB/16/L/03	LC-MS/MS	Mussel	AZA 2	79.4	76.6	AZA2 µg/kg	-0.2
EURLMB/16/L/03	LC-MS/MS	Mussel	AZA 3	21.2	18.2	AZA3 µg/kg	-0.6

AFBINI Quasimeme Round 2016.1

<i>Sample No</i>	<i>Sample ID</i>	<i>Sample Description</i>	<i>Determinand</i>	<i>Assigned Value</i>	<i>Reported Value</i>	<i>Units</i>	<i>Z Score</i>
Sample 1	QST205SS	AZA Standard	AZA-1	10.29	11.1	µg/kg	0.61
Sample 1	QST205SS	AZA Standard	AZA-total	10.1	11.1	µg AZA eq./kg	0.76
Sample 2	QST206SS	Lipophilic Standard	AZA-1	17.79	19.1	µg/kg	0.57
Sample 2	QST206SS	Lipophilic Standard	AZA-2	11.88	12.8	µg/kg	0.60
Sample 2	QST206SS	Lipophilic Standard	AZA-3	8.88	10	µg/kg	0.96
Sample 2	QST206SS	Lipophilic Standard	AZA-total	50.69	56	µg AZA eq./kg	0.83
Sample 2	QST206SS	Lipophilic Standard	Free-DTX1	69.46	58.5	µg/kg	-1.26
Sample 2	QST206SS	Lipophilic Standard	Total-free-OA+DTX1+DTX2	70.19	58.5	µg OA eq./kg	-1.32
Sample 2	QST206SS	Lipophilic Standard	Total-YTX		0.026	mg YTX eq./kg	
Sample 2	QST206SS	Lipophilic Standard	YTX		0.026	mg/kg	
Sample 3	QST207BT	DSP/AZP extract	45-OH-YTX		0.0033	mg/kg	
Sample 3	QST207BT	DSP/AZP extract	AZA-1	67.23	62.8	µg/kg	-0.52
Sample 3	QST207BT	DSP/AZP extract	AZA-2	20.67	19.4	µg/kg	-0.48
Sample 3	QST207BT	DSP/AZP extract	AZA-3	15.14	14.9	µg/kg	-0.12
Sample 3	QST207BT	DSP/AZP extract	AZA-total	126.0	118.5	µg AZA eq./kg	-0.48
Sample 3	QST207BT	DSP/AZP extract	Free-DTX1	9.6	9	µg/kg	-0.48
Sample 3	QST207BT	DSP/AZP extract	Free-DTX2	35.17	27.6	µg/kg	-1.70
Sample 3	QST207BT	DSP/AZP extract	free-Okadaic-Acid	10.92	9.2	µg/kg	-1.22
Sample 3	QST207BT	DSP/AZP extract	Total OA and PTX	89.9	80.7	µg OA eq./kg	-0.81
Sample 3	QST207BT	DSP/AZP extract	Total-DTX1	15.33	13	µg/kg	-1.19
Sample 3	QST207BT	DSP/AZP extract	Total-DTX2	59.56	47.4	µg/kg	-1.62
Sample 3	QST207BT	DSP/AZP extract	Total-free-OA+DTX1+DTX2	41.12	34.8	µg OA eq./kg	-1.22
Sample 3	QST207BT	DSP/AZP extract	Total-hy-OA+DTX1+DTX2	93.63	80.7	µg OA eq./kg	-1.10
Sample 3	QST207BT	DSP/AZP extract	Total-Okadaic-Acid	41.02	39.3	µg/kg	-0.33
Sample 3	QST207BT	DSP/AZP extract	Total-YTX		0.0091	mg YTX eq./kg	
Sample 3	QST207BT	DSP/AZP extract	YTX		0.0059	mg/kg	

<i>Sample No</i>	<i>Sample ID</i>	<i>Sample Description</i>	<i>Determinand</i>	<i>Assigned Value</i>	<i>Reported Value</i>	<i>Units</i>	<i>Z Score</i>
Sample 4	QST208BT	Mussel Homogenate	AZA-1	134.53	112.5	µg/kg	-1.31
Sample 4	QST208BT	Mussel Homogenate	AZA-2	44.98	36.3	µg/kg	-1.53
Sample 4	QST208BT	Mussel Homogenate	AZA-3	34.93	28.1	µg/kg	-1.55
Sample 4	QST208BT	Mussel Homogenate	AZA-total	258.9	217	µg AZA eq./kg	-1.29
Sample 4	QST208BT	Mussel Homogenate	Free-DTX2	519.3	446.5	µg/kg	-1.12
Sample 5	QST208BT	Mussel Homogenate	free-Okadaic-Acid	100.8	70.6	µg/kg	-2.38
Sample 4	QST208BT	Mussel Homogenate	Total OA and PTX	684.4	611.7	µg OA eq./kg	-0.85
Sample 4	QST208BT	Mussel Homogenate	Total-DTX2	820.9	630.2	µg/kg	-1.86
Sample 4	QST208BT	Mussel Homogenate	Total-free-OA+DTX1+DTX2	415.8	338.5	µg OA eq./kg	-1.49
Sample 4	QST208BT	Mussel Homogenate	Total-hy-OA+DTX1+DTX2	734.8	611.7	µg OA eq./kg	-1.34
Sample 4	QST208BT	Mussel Homogenate	Total-Okadaic-Acid	238.1	233.5	µg/kg	-0.15
Sample 4	QST208BT	Mussel Homogenate	Total-YTX	1.21	1.26	mg YTX eq./kg	0.25
Sample 4	QST208BT	Mussel Homogenate	YTX	1.2	1.26	mg/kg	0.28
Sample 5	QST209BT	Mussel Homogenate	AZA-1	1318	1297	µg/kg	-0.13
Sample 5	QST209BT	Mussel Homogenate	AZA-2	332.4	330.7	µg/kg	-0.04
Sample 5	QST209BT	Mussel Homogenate	AZA-3	283.3	265.1	µg/kg	-0.51
Sample 5	QST209BT	Mussel Homogenate	AZA-total	2340.4	2263.5	µg AZA eq./kg	-0.26
Sample 5	QST209BT	Mussel Homogenate	Free-DTX2	256	217	µg/kg	-1.21
Sample 5	QST209BT	Mussel Homogenate	free-Okadaic-Acid	238.8	204.1	µg/kg	-1.16
Sample 5	QST209BT	Mussel Homogenate	Total OA and PTX	601.2	587.1	µg OA eq./kg	-0.19
Sample 5	QST209BT	Mussel Homogenate	Total-DTX2	355.6	316.3	µg/kg	-0.88
Sample 5	QST209BT	Mussel Homogenate	Total-free-OA+DTX1+DTX2	404.2	336	µg OA eq./kg	-1.35
Sample 5	QST209BT	Mussel Homogenate	Total-hy-OA+DTX1+DTX2	630.6	587.1	µg OA eq./kg	-0.55
Sample 5	QST209BT	Mussel Homogenate	Total-Okadaic-Acid	425.7	394.1	µg/kg	-0.59

AFBINI Quasimeme Round 2016.2

<i>Sample No</i>	<i>Sample ID</i>	<i>Sample Description</i>	<i>Determinand</i>	<i>Assigned Value</i>	<i>Reported Value</i>	<i>Units</i>	<i>Z Score</i>
Sample 1	QST217SS	Multitoxin standard	AZA-1	12	11.39	µg/kg	-0.35
Sample 1	QST217SS	Multitoxin standard	AZA-total	11.7	11.39	µg AZA eq./kg	-0.20
Sample 1	QST217SS	Multitoxin standard	Free-OA	79.3	69.21	µg/kg	-0.96
Sample 1	QST217SS	Multitoxin standard	Total-free-OA+DTX1+DTX2	77.2	69.21	µg OA eq./kg	-0.78
Sample 1	QST217SS	Multitoxin standard	PTX-2	26.3	27.15	µg/kg	0.23
Sample 1	QST217SS	Multitoxin standard	YTX	0.051	0.045	mg/kg	-0.37
Sample 2	QST218SS	Lipophilic standard	Free-DTX2	33.6	32.84	µg/kg	-0.18
Sample 2	QST218SS	Lipophilic standard	Free-OA	58	49.63	µg/kg	-1.09
Sample 2	QST218SS	Lipophilic standard	Total-free-OA+DTX1+DTX2	76.4	69.33	µg OA eq./kg	-0.70
Sample 2	QST218SS	Lipophilic standard	YTX	0.034	0.025	mg/kg	-0.63
Sample 3	QST219BT	DSP/AZP extract	AZA-1	15	15.5	µg/kg	0.25
Sample 3	QST219BT	DSP/AZP extract	AZA-2	3.9	4	µg/kg	0.18
Sample 3	QST219BT	DSP/AZP extract	AZA-3	5.58	5.8	µg/kg	0.28
Sample 3	QST219BT	DSP/AZP extract	AZA-total	30.1	30.7	µg AZA eq./kg	0.16
Sample 3	QST219BT	DSP/AZP extract	Free-DTX2	25.4	26.5	µg/kg	0.31
Sample 3	QST219BT	DSP/AZP extract	Free-OA	13.7	12.6	µg/kg	-0.60
Sample 3	QST219BT	Mussel Homogenate	Total-free-OA+DTX1+DTX2	29.9	28.5	µg OA eq./kg	-0.33
Sample 3	QST219BT	Mussel Homogenate	Total-DTX2	54.2	32.2	µg/kg	-2.90
Sample 3	QST219BT	Mussel Homogenate	Total-OA	42.4	29.1	µg/kg	-2.26
Sample 3	QST219BT	Mussel Homogenate	Total-hy-OA+DTX1+DTX2	75.5	48.4	µg OA eq./kg	-2.55
Sample 3	QST219BT	Mussel Homogenate	Total-hy-OA+DTX1+DTX2+PTX	75	48.4	µg OA eq./kg	-2.57
Sample 4	QST220BT	Mussel Homogenate	AZA-1	77.4	76.9	µg/kg	-0.05
Sample 4	QST220BT	Mussel Homogenate	AZA-2	25.6	29.4	µg/kg	1.16
Sample 4	QST220BT	Mussel Homogenate	AZA-3	19.1	19.8	µg/kg	0.29
Sample 4	QST220BT	Mussel Homogenate	AZA-total	153	157.7	µg AZA eq./kg	0.24

Sample No	Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
Sample 4	QST220BT	Mussel Homogenate	Free-DTX1	12.2	11	µg/kg	-0.69
Sample 4	QST220BT	Mussel Homogenate	Free-DTX2	44.2	42.8	µg/kg	-0.24
Sample 4	QST220BT	Mussel Homogenate	Free-OA	12.8	11.9	µg/kg	-0.49
Sample 4	QST220BT	Mussel Homogenate	Total-free-OA+DTX1+DTX2	54.9	48.6	µg OA eq./kg	-0.85
Sample 4	QST220BT	Mussel Homogenate	Total-DTX1	16.7	10.3	µg/kg	-2.75
Sample 4	QST220BT	Mussel Homogenate	Total-DTX2	73.9	42.8	µg/kg	-3.10
Sample 4	QST220BT	Mussel Homogenate	Total-OA	48.9	33.4	µg/kg	-2.31
Sample 4	QST220BT	Mussel Homogenate	Total-hy-OA+DTX1+DTX2	112	69.4	µg OA eq./kg	-2.91
Sample 4	QST220BT	Mussel Homogenate	Total-hy-OA+DTX1+DTX2+PTX	109	69.4	µg OA eq./kg	-2.71
Sample 4	QST220BT	Mussel Homogenate	YTX		0.0061	mg/kg	
Sample 4	QST220BT	Mussel Homogenate	45-OH-YTX		0.0033	mg/kg	
Sample 4	QST220BT	Mussel Homogenate	Total YTX		0.0094	mg YTX eq./kg	
Sample 5	QST221BT	Lipophilic extract	AZA-1	715	659.8	µg/kg	-0.59
Sample 5	QST221BT	Lipophilic extract	AZA-2	200	215.2	µg/kg	0.59
Sample 5	QST221BT	Lipophilic extract	AZA-3	117	119.1	µg/kg	0.10
Sample 5	QST221BT	Lipophilic extract	AZA-total	1246	1213.9	µg AZA eq./kg	-0.21
Sample 5	QST221BT	Lipophilic extract	Free-DTX1	83	62	µg/kg	-1.91
Sample 5	QST221BT	Lipophilic extract	Free-DTX2	566	527.1	µg/kg	-0.53
Sample 5	QST221BT	Lipophilic extract	Free-OA	148	117.8	µg/kg	-1.58
Sample 5	QST221BT	Lipophilic extract	Total-free-OA+DTX1+DTX2	575	496	µg OA eq./kg	-1.06
Sample 5	QST221BT	Lipophilic extract	Total-DTX1	114	76.7	µg/kg	-2.48
Sample 5	QST221BT	Lipophilic extract	Total-DTX2	746	490.3	µg/kg	-2.57
Sample 5	QST221BT	Lipophilic extract	Total-OA	320	260.9	µgOA eq/kg	-1.41
Sample 5	QST221BT	Lipophilic extract	Total-hy-OA+DTX1+DTX2	885	631.7	µg OA eq./kg	-2.20
Sample 5	QST221BT	Lipophilic extract	Total-hy-OA+DTX1+DTX2+PTX	872	631.7	µg OA eq./kg	-2.11
Sample 5	QST221BT	Lipophilic extract	YTX	0.215	0.216	mg/kg	0.02
Sample 5	QST221BT	Lipophilic extract	Homo YTX	1.04	1.008	mg/kg	-0.23

Sample No	Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
Sample 5	QST221BT	Lipophilic extract	45-OH-Homo YTX	0.515	0.576	mg/kg	0.74
Sample 5	QST221BT	Lipophilic extract	45-OH-YTX	0.156	0.149	mg/kg	-0.18
Sample 5	QST221BT	Lipophilic extract	Total YTX	1.68	1.661	mg YTX eq./kg	-0.08

AFBINI International Phytoplankton Intercomparison (previously Bequalm) 2016 Phytoplankton Proficiency Testing Scheme

Date	Analyst Code	Phytoplankton In sample	Species ID	z-score
2016	29	<i>Pseudo-nitzschia australis</i>	correct	-0.05
2016	29	<i>Guinardia delicatula</i>	correct	1.22
2016	29	<i>Dinophysis acuta</i>	correct	0.18
2016	29	<i>Thalassiosira gravida</i>	correct	0.91
2016	29	<i>Chaetoceros didymus</i>	correct	1
2016	29	<i>Coscinodiscus walessii</i>	correct	-0.02
2016	29	<i>Prorocentrum triestinum</i>	correct	-0.53
2016	29	<i>Alexandrium ostenfeldii</i>	correct	1.12

Result of International Phytoplankton Intercomparison (Bequalm) taxonomic quiz score: Analyst 29 achieved a proficient classification; test score >90%.

Appendix 2



Agri-Food & Biosciences Institute

VETERINARY SCIENCES DIVISION

Chemical Surveillance Branch

Work Programme UK National Reference Laboratory For Marine Biotoxins

2016

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Glossary

AFBI: Agri-Food and Biosciences Institute

NRL: National Reference Laboratory

Cefas: Centre for Environment, Fisheries and Aquaculture Science

LTs: Lipophilic toxins (including Diarrhetic Shellfish Poison (DSP) group)

EURL-MB: European Reference Laboratory for Marine Biotoxins

FSA: Food Standards Agency

TEF: Toxic Equivalence Factor

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

MU: Measurement Uncertainty

PSP: Paralytic Shellfish Poison (Saxitoxin group)

UK-NRL: United Kingdom National Reference Laboratory

National Reference Laboratory Annual Report

The annual report for 2016-2017 will be drafted and submitted to the Competent Authority (FSA) for comment in May 2017.

NRL Standard Operating Procedures

The NRL Standard Operating procedures will be reviewed and updated, if required.

Proficiency tests 2016

Official control testing is carried out at two laboratories (AFBI and Cefas) making UK proficiency tests / ring trials of limited value. Both UK laboratories participate in marine biotoxin proficiency schemes organised by Quasimeme and share the data with the UK-NRL and the Competent Authority (FSA). Similarly, the official control laboratories undertaking phytoplankton analysis participate in the Bequalm scheme and share the data with the NRL and the Competent Authority.

The UK-NRL has negotiated participation of Cefas in the EURL-MB proficiency tests in the past and whilst the EURL has indicated that the 2016 programme will be limited because of resource issues and the number of non-NRLs taking part will be restricted, the UKNRL has requested that both UK laboratories be included in the 2016 programme.

Meetings

EURL – NRLs Workshop 2016: Date and location to be confirmed (October 2016) by EURL.

EURL working group on LC-MS/MS: The working group will be reconvened in June 2016 to discuss revision of the LTs method by LC-MS/MS. Further meetings may be convened during 2016.

EURL working Group on PSP: The working group may be reconvened in 2016 to discuss performance in the 2016 EURL proficiency test, consider new data on TEFs for PSP analogues, and consider the implementation and application of new technology and modifications to methods.

EURL working Group on Phytoplankton: The NRL will continue to participate and be represented at meetings of the working Group in 2016-17. Most of the work is conducted electronically and the NRL will continue to co-ordinate all UK responses and input from UK experts.

EURL working Group on MU: The NRL will continue to participate in this working group as required. Work to date has been conducted electronically and the NRL will continue to co-ordinate all UK responses and input from UK experts.

CEN/TC275/WG 14 on Marine Biotoxins: AFBI will continue to support the Competent Authority in its standardisation activities through active participation in the BSI / CEN processes.

UK-NRL Network Group: Two meetings to be held in 2016. It is proposed that two meeting will be held in 2016. The first will be in June 2016 and may be conducted by teleconference, depending on the agreed agenda. The second will be held in October/November 2016, with arrangements and location to be confirmed at the meeting in June 2016.