Agri-Food & Biosciences Institute

VETERINARY SCIENCES DIVISION

Chemical Surveillance Branch

Annual Report
UK National Reference Laboratory
For Marine Biotoxins

1st April 2015 – 31st March 2016

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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

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)

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

Introduction

This report provides an outline of the work of the UK-NRL over the financial year 2015-2016. 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 2015 UK-NRL work programme is provided in Appendix 1.

Internal restructuring of the NRL took place in September 2015 due to the retirement of Cowan Higgins (biotoxins) and Dr. Richard Gowan (phtyoplankton). Dermot Faulkner (biotoxins) and Dr. Matthew Service (phytoplankton) have now assumed their respective NRL responsibilities. The NRL for marine biotoxins sits within the newly created Chemical and Immunodiagnostic Sciences Branch (CISB), headed up by Dr. Alastair Douglas.

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.

Collaboration with the EURL-MB

In 2015-2016, requests were made by the EURL to all NRLs to confirm their willingness to participate in two additional working groups (in addition to the existing LC-MS/MS working group). The UK-NRL confirmed that they would contribute to both of these. The working groups were aimed at the harmonisation of (1) method uncertainty and (2) EU monitoring programmes for toxic phytoplankton within the EU.

The UK-NRL provided a documented summary to the EURL on the UK approach for the calculation and application of method uncertainty to biotoxin monitoring results within the UK. A phytoplankton working group was established with the aim of harmonising sampling methodology, the list of toxic species to be identified and thresholds used. The UK-NRL collated information from the English, Scottish and Northern Ireland water sampling programmes on the methodologies used and target species monitored for. This information was then sent to Dr. Pablo Serrat, chair of the Working Group. 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 table below provides a summary of meetings attended as part of the NRL activities that took place during 2015-2016.

Meetings Attended 2015-2016

Date	**Venue**	**Subject**
18th June 2015	Baiona	Symposium and advisory group meeting
25th June 2015	Aberdeen	UK NRL Network Meeting
22-23rd October 2015	Cesenatico	EURL Workshop meeting (hosted by Italian NRL)
19th November 2015	Berlin	CEN WG on Marine Biotoxins
24th November 2015	London	UK NRL Network Meeting

FSS hosted the 25th meeting of the Network group in Aberdeen on the 25th June 2015. FSA hosted the 26th meeting of the UKNRL-Network group, held in London on the 19th Nov 2015.

In 2015, the EURL only offered participation in proficiency testing exercises to NRLs. This was discussed at the UK network meeting in June 2015 and tissue extracts and remaining samples were circulated to Cefas after the closure of the 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.

At the request of the NRL, positive materials were circulated by Cefas to AFBI to permit production of laboratory reference materials for method internal quality control monitoring.

In support of contingency planning within the UK laboratory network it was recommended that the AFBI official control laboratory extend ISO17025 accreditation of the PSP quantification method to King and Queen Scallop through additional validation (matrices currently under screening scope). Validation has since been completed for King Scallop and ready for submission to UKAS for assessment.

The UK-NRL agreed to participate in a collaborative pre-trial being organised by Cefas for the analysis of PSP group toxins and TTX/epi-TTX by LC-MS/MS. Cefas kindly provided documents which would aid in the transfer of the method from Cefas to the NRL laboratory. Due to different vendor equipment specification between laboratories, significant efforts were made in the setup and the optimisation of LC-MS/MS parameters and an initial evaluation of the method then carried out at the NRL.

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 PSP, DSP and ASP. PT exercises for PSP have been organised since 2004. The number of participants in 2015 was 24 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 2015, there were 22 participants. For lipophilic toxins, the EURL-MB has organised PTs since 2000. A total of 19 laboratories participated in the 2015 study. This reflects a reduction on previous years due to the exclusion of several Spanish official monitoring laboratories from these PT schemes.

Reports on the EURL-MB proficiency tests were circulated throughout the year and the results obtained by the UK-NRL are summarised in Appendix 1. The full reports are available for download from either the EURL-MB or the UK-NRL website. 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 2015.

Both laboratories participate in the Quasimeme Proficiency test programme and the full 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:

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

$|Z| < 2$ 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)

Proficiency test summary

For the EURL and Quasimeme proficiency tests for lipophilic toxins, the National Reference Laboratory reported results with 95.4% of the Z scores less than 2 (satisfactory), 3.7% of the Z scores less than 3 (questionable) and 0.9% of the Z scores as >3 (1 result out of 108 reported as unsatisfactory). The unsatisfactory result was for a toxin analogue (45-OH-Homo-YTX) for which no reference standard is available. Results for this toxin had been quantified using YTX as the calibrant. Retrospective quantification using homo-YTX as calibrant would have returned a satisfactory result, and this approach has now been recommended by the EURL. The questionable results relate to individual toxin analogues whilst all figures for Total Toxicity were less than 2. Overall, performance of the analytical method is in within the expected statistical limits with <5% of results falling at the extremes of a normal distribution.

For PSP, 97% of Z scores were less than 2 and 3% were between 2 and 3 (1 questionable result out of 30). The questionable result related to Total Toxicity with a Z-score of -2.1. In addition, dcNeo was misidentified in a single sample as GTX1,4 due to a complex toxin profile. A follow up investigation and non-conformance report was carried out. The non-conformance report was forwarded to the EURL and internal IQA auditors, was accepted and subsequently closed. In addition, further training of analysts in quantification of toxin profiles was subsequently conducted and an additional follow up of this through analysis of blind samples sent by the EURL for reporting in January 2016 (this was an optional exercise). The results for this were accepted by the EURL as satisfactory.

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

Dissemination of Information from the EURL-MB

& Provision of Scientific and Technical Assistance to the Competent Authority

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

The EURL standard operating procedure (SOP) for lipophilic toxins by LC-MS/MS was revised by the EURL LC-MS/MS working group (as version 5) to harmonise the application of the method to cooked and processed shellfish. Details of the changes and NRL comments to the method SOP were forwarded to Valerie McFarlane prior to the Brussels Bivalve molluscs WG meeting in October 2015.

The UK-NRL provided the EURL with a document on the application of measurement uncertainty in the UK within the eWG. This document was also circulated to the UK network for discussion and comment.

The annual EURL Network workshop was held in Cesenatico by the Italian NRL on the 22nd – 23rd October 2015. Presentations by participants on the work of their NRL were included in the Agenda to encourage more wide-spread participation by the NRLs. The UK-NRL gave an overview of the role of the NRL within the UK, outlined some of the work it had undertaken during the year, and detailed recent restructuring within the NRL due to staff retirements. The UK-NRL and Dutch-NRL asked that TTX as an emerging toxin threat in the EU be added to the list of agenda items for discussion and for inclusion in future EURL activities. It was agreed in the final conclusions that the EURL should raise this with the EU commission. The performance of NRLs in the 2015 EURL PT schemes, a review of the 2015 work programme of the EURL and activities for 2016 were presented by the EURL for discussion.

In November 2015, the technical working group CEN/TC 275/WG14 on marine biotoxins was convened in Berlin with the purpose of agreeing final comments/changes to two standards. The Working Group considered revision of EN 14526 "Foodstuffs - Determination of saxitoxin-group toxins in shellfish – HPLC method using pre- column derivatization with peroxide or periodate oxidation" and EN 14176 "Foodstuffs - Determination of domoic acid in raw shellfish, raw finfish and cooked mussels by HPLC using UV detection". The UK-NRL had previously provided FSA with detailed comments on the methods under discussion. Further comments and

revisions were agreed at the CEN/TC 275/WG14 meeting and a final draft of both standards was recommended to be put forward for formal vote, which was subsequently accepted by member states.

Links

UK-NRL Web page:

The NRL website and associated links have now been updated following migration of the domain and 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.aesan.msssi.gob.es/en/CRLMB/web/home.shtml

EURL-MB Work Programme 2015:

http://aesan.msssi.gob.es/CRLMB/docs/docs/program_de_trabajo_anual/EURLMB_Work_Program_2015_2.pdf

Appendix 1

Agri-Food & Biosciences Institute

VETERINARY SCIENCES DIVISION

Chemical Surveillance Branch

Work Programme
UK National Reference Laboratory
For Marine Biotoxins

2015

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Appendix 2

Domoic Acid 2015 Proficiency Test Summaries

AFBI EURL 2015

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
EURLMB/15/A/01	Scallop homogenate	15.2	16.5	mg/kg	0.79
EURLMB/15/A/02	Mussel homogenate	17.3	17.9	mg/kg	0.29

AFBI Quasimeme Round 2015.1

Sample ID	Sample description	Round	Assigned value	Reported value	Units	z-score
QST182SS	Standard solution	2015.1	0.458	0.46	mg/Kg	-0.03
QST183BT	Mussel Homogenate	2015.1	29.53	30.42	mg/Kg	0.24
QST184BT	Scallop Homogenate	2015.1	62.54	62.05	mg/Kg	-0.06

AFBI Quasimeme Round 2015.2

Sample ID	Sample description	Round	Assigned value	Reported value	Units	z-score
QST194SS	Standard solution	2015.2	1.311	1.36	mg/Kg	0.23
QST195BT	Oyster Homogenate	2015.2	42.46	45.93	mg/Kg	0.65
QST196BT	Mussel Homogenate	2015.2	47.59	51.13	mg/Kg	0.59

PSP 2015 Proficiency Test Summaries

AFBI EURL 2015

Sample ID	Method	Matrix	Determinand	Assigned Value	Reported Value	Units	Z-Score
EURLMB/15/P/01	**HPLC**	**Mussels**	**Total STX**	**673**	**709.3**	**ugSTX2HCL equiv/Kg**	**0.29**
EURLMB/15/P/01	HPLC	Mussels	GTX2,3	284	226.9	umol/kg	-0.92
EURLMB/15/P/01	HPLC	Mussels	STX	95	71.5	umol/kg	-1.05
EURLMB/15/P/01	HPLC	Mussels	GTX1,4	305	410.9	umol/kg	1.59
EURLMB/15/P/02	**MBA**	**Wedge shell clams**		**2321**	**1867**	**ugSTX2HCL equ/Kg**	**-1.05**
EURLMB/15/P/02	**HPLC**	**Wedge shell clams**	**Total STX**	**1965**	**1869.6**	**ugSTX2HCL eq/Kg**	**-0.31**
EURLMB/15/P/02	HPLC	Wedge shell clams	dcGTX2&3	85	66.7	umol/kg	-0.85
EURLMB/15/P/02	HPLC	Wedge shell clams	C1&2	56	56.6	umol/kg	0.08
EURLMB/15/P/02	HPLC	Wedge shell clams	dcSTX	1234	982.6	umol/kg	-1.24
EURLMB/15/P/02	HPLC	Wedge shell clams	GTX5	207	181.6	umol/kg	-0.57
EURLMB/15/P/02	HPLC	Wedge shell clams	dcNEO	141	Not detected	umol/kg	

EURLMB/15/P/02	HPLC	Wedge shell clams	GTX6	185	171.2	umol/kg	-0.32
EURLMB/15/P/03	**MBA**	**Mussels**	**NEGATIVE**	**N/A**	**N/A**	**ugSTX2HCL equiv/Kg**	**N/A**
EURLMB/15/P/03	**HPLC**	**Mussels**	**NEGATIVE**	**N/A**	**N/A**	**ugSTX2HCL equiv/Kg**	**N/A**

AFBI Quasimeme Round 2015.1

Sample ID	Method	Determinand	Sample description	Assigned Value	Reported Value	Units	z-score
QST190BT	HPLC	dc-STX	Mussel Homogenate	0.617	0.480	µmol/kg	-1.08
QST190BT	HPLC	GTX-2,3	Mussel Homogenate	1.618	1.416	µmol/kg	-0.80
QST190BT	HPLC	STX	Mussel Homogenate	1.910	1.768	µmol/kg	-0.49
QST190BT	**HPLC**	**Total toxicity**	**Mussel Homogenate**	**1292.8**	**1152**	**µgSTXdiHCl eq./kg**	**-0.87**
QST191BT	HPLC	GTX-1,4	Oyster Homogenate	0.656	0.696	µmol/kg	0.30
QST191BT	HPLC	GTX-2,3	Oyster Homogenate	1.644	1.448	µmol/kg	-0.77
QST191BT	HPLC	NEO	Oyster Homogenate	0.188	0.168	µmol/kg	-0.28
QST191BT	HPLC	STX	Oyster Homogenate	0.572	0.544	µmol/kg	-0.23
QST191BT	**HPLC**	**Total toxicity**	**Oyster Homogenate**	**839.8**	**849**	**µgSTXdiHCl eq./kg**	**0.09**
QST192BT	HPLC	dc-STX	Mussel Homogenate	0.583	0.464	µmol/kg	-0.97
QST192BT	HPLC	GTX-1,4	Mussel Homogenate	0.542	0.504	µmol/kg	-0.32
QST192BT	HPLC	GTX-2,3	Mussel Homogenate	1.000	0.896	µmol/kg	-0.60
QST192BT	HPLC	STX	Mussel Homogenate	0.313	0.288	µmol/kg	-0.28
QST192BT	**HPLC**	**Total toxicity**	**Mussel Homogenate**	**748.92**	**667**	**µgSTXdiHCl eq./kg**	**-0.87**
QST193BT	HPLC	dc-STX	Mussel Homogenate	2.204	1.592	µmol/kg	-1.88
QST193BT	HPLC	STX	Mussel Homogenate	0.081	0.56	µmol/kg	-0.41
QST193BT	**HPLC**	**Total toxicity**	**Mussel Homogenate**	**837.4**	**613**	**µgSTXdiHCl eq./kg**	**-2.12**

Lipophilic Toxins 2015 Proficiency Test Summaries

AFBI EURL 2015

Sample ID	Method	Matrix	Determinand	Assigned Value	Reported Value	Units	Z-Score
EURLMB/15/L/01	LCMS:MS/MS	Mussel		NEG	NEG		0.0
EURLMB/15/L/02	LCMS:MS-OA	Mussel		240.3	219.4	OA equiv/kg	-0.4
EURLMB/15/L/02	LCMS:MS-OA	Mussel		128.4	117.1	Free OA µg/kg	-0.4
EURLMB/15/L/02	LCMS:MS-OA	Mussel		232.0	209.9	OA equiv/kg	-0.5
EURLMB/15/L/02	LCMS:MS-AZA	Mussel		335.9	362.5	AZA1 equiv/kg	0.4
EURLMB/15/L/02	LCMS:MS-AZA	Mussel		217.4	225.8	AZA1 µg/kg	0.2
EURLMB/15/L/02	LCMS:MS-AZA	Mussel		58.7	67.6	AZA2 µg/kg	0.6
EURLMB/15/L/02	LCMS:MS-YTX	Mussel		2.03	2.15	YTX equiv/kg	0.4
EURLMB/15/L/02	LCMS:MS-YTX	Mussel		0.15	0.15	YTX mg/kg	-0.1
EURLMB/15/L/02	LCMS:MS-YTX	Mussel		1.30	1.10	Homo-YTX mg/kg	-0.9

EURLMB/15/L/02	LCMS:MS-YTX	Mussel		0.18	0.14	45-OH-YTX mg/kg	-0.8
EURLMB/15/L/02	LCMS:MS-YTX	Mussel		0.94	1.52	45-OH-Homo-YTX mg/kg	3.5
EURLMB/15/L/03	LCMS:MS-OA	Mussel		544.3	565.8	OA equiv/kg	0.2
EURLMB/15/L/03	LCMS:MS-OA	Mussel		41.6	35.5	Free OA µg/kg	-0.7
EURLMB/15/L/03	LCMS:MS-OA	Mussel		145.3	143.5	Free DTX-1 µg/kg	-0.1
EURLMB/15/L/03	LCMS:MS-OA	Mussel		160.0	143.3	Free DTX-2 µg/kg	-0.5
EURLMB/15/L/03	LCMS:MS-OA	Mussel		148.0	147.0	Total OA µg/kg	0.0
EURLMB/15/L/03	LCMS:MS-OA	Mussel		251.8	287.6	Total DTX-1 µg/kg	0.7
EURLMB/15/L/03	LCMS:MS-OA	Mussel		220.1	218.6	Total DTX-2 µg/kg	0.0

AFBI Quasimeme Round 2015.1

Sample ID	*Sample Description*	*Determinand*	*Assigned Value*	*Reported Value*	*Units*	*Z Score*
QST185SS	Multi toxin standard solution	AZA1	12.65	13.77	µg/kg	0.69
QST185SS	Multi toxin standard solution	AZA-total	12.68	13.77	µg AZA eq./kg	0.67
QST185SS	Multi toxin standard solution	Free-OA	80.70	68.83	µg/kg	-1.17
QST185SS	Multi toxin standard solution	PTX-2	26.34	29.55	µg/kg	0.96
QST185SS	Multi toxin standard solution	Total free OA-DTX1-DTX2	79.67	68.83	µg OA eq./kg	-1.08
QST186SS	Lipophilic Standard Solution	AZA1	16.67	17.68	µg/kg	0.47
QST186SS	Lipophilic Standard Solution	AZA2	11.16	14.27	µg/kg	2.15
QST186SS	Lipophilic Standard Solution	AZA3	8.44	9.35	µg/kg	0.82
QST186SS	Lipophilic Standard Solution	AZA-total	47.63	56.46	µg AZA eq./kg	1.47
QST186SS	Lipophilic Standard Solution	Free-DTX1	65.54	62.52	µg/kg	-0.37
QST186SS	Lipophilic Standard Solution	Total free OA-DTX1-DTX2	65.13	62.52	µg OA eq./kg	-0.32
QST187BT	DSP/AZP Extract Solution	Free-DTX2	45.55	42.70	µg/kg	-0.50
QST187BT	DSP/AZP Extract Solution	Free-OA	14.38	11.80	µg/kg	-1.40
QST187BT	DSP/AZP Extract Solution	Total OA + PTX	127.30	152.60	µg OA eq./kg	1.58
QST187BT	DSP/AZP Extract Solution	Total-DTX2	94.08	109.20	µg/kg	1.28
QST187BT	DSP/AZP Extract Solution	Total free OA-DTX1-DTX2	43.05	37.50	µg OA eq./kg	-1.02
QST187BT	DSP/AZP Extract Solution	Total-hy-OA-DTX1-DTX2	125.99	152.60	µg OA eq./kg	1.68
QST187BT	DSP/AZP Extract Solution	Total-OA	63.80	87.10	µg/kg	2.90
QST188BT	Mussel homogenate	AZA-1	412.92	302.80	µg/kg	-2.13
QST188BT	Mussel homogenate	AZA-2	92.32	95.10	µg/kg	0.24
QST188BT	Mussel homogenate	AZA-3	51.01	44.70	µg/kg	-0.98
QST188BT	Mussel homogenate	AZA-total	642.15	536.50	µg AZA eq./kg	-1.32
QST188BT	Mussel homogenate	Free-DTX1	124.23	125.20	µg/kg	0.06
QST188BT	Mussel homogenate	Free-DTX2	133.52	120.70	µg/kg	-0.77
QST188BT	Mussel homogenate	free-Okadaic-Acid	167.96	152.70	µg/kg	-0.73
QST188BT	Mussel homogenate	Total OA and PTX	493.83	530.70	µg OA eq./kg	0.60

Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
QST188BT	Mussel homogenate	Total-DTX1	189.57	204.60	µg/kg	0.63
QST188BT	Mussel homogenate	Total-DTX2	151.38	146.00	µg/kg	-0.28
QST188BT	Mussel homogenate	Total-free-OA+DTX1+DTX2	382.23	350.40	µg OA eq./kg	-0.67
QST188BT	Mussel homogenate	Total-hy-OA+DTX1+DTX2	505.40	530.70	µg OA eq./kg	0.40
QST188BT	Mussel homogenate	Total-Okadaic-Acid	232.95	238.50	µg/kg	0.19
QST189BT	Mussel homogenate	45-OH-homo-YTX	0.58	0.83	mg/kg	2.04
QST189BT	Mussel homogenate	45-OH-YTX	0.14	0.14	mg/kg	-0.02
QST189BT	Mussel homogenate	AZA-1	708.26	682.80	µg/kg	-0.29
QST189BT	Mussel homogenate	AZA-2	184.01	210.70	µg/kg	1.16
QST189BT	Mussel homogenate	AZA-3	111.87	107.10	µg/kg	-0.34
QST189BT	Mussel homogenate	AZA-total	1210.68	1212.10	µg AZA eq./kg	0.01
QST189BT	Mussel homogenate	Free-DTX1	83.01	83.10	µg/kg	0.01
QST189BT	Mussel homogenate	Free-DTX2	540.35	518.10	µg/kg	-0.33
QST189BT	Mussel homogenate	free-Okadaic-Acid	145.16	124.00	µg/kg	-1.16
QST189BT	Mussel homogenate	homo-YTX	1.13	0.94	mg/kg	-1.01
QST189BT	Mussel homogenate	Total OA and PTX	876.92	866.50	µg OA eq./kg	-0.10
QST189BT	Mussel homogenate	Total-DTX1	114.85	111.50	µg/kg	-0.23
QST189BT	Mussel homogenate	Total-DTX2	771.70	758.80	µg/kg	-0.13
QST189BT	Mussel homogenate	Total-free-OA+DTX1+DTX2	566.03	517.90	µg OA eq./kg	-0.68
QST189BT	Mussel homogenate	Total-hy-OA+DTX1+DTX2	886.64	866.50	µg OA eq./kg	-0.18
QST189BT	Mussel homogenate	Total-Okadaic-Acid	320.08	299.70	µg/kg	-0.51
QST189BT	Mussel homogenate	Total-YTX	1.83	1.71	mg YTX eq./kg	-0.41
QST189BT	Mussel homogenate	YTX	0.22	0.22	mg/kg	-0.03

AFBI Quasimeme Round 2015.2

Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
QST197SS	DTX-2 standard solution	Free-DTX2	65.75	57.59	µg/kg	-0.99
QST197SS	DTX-2 standard solution	Total free OA-DTX1-DTX2	39.33	34.60	µg OA eq./kg	-0.95
QST198SS	Multi toxin standard solution	Free-DTX2	33.13	30.31	µg/kg	-0.67
QST198SS	Multi toxin standard solution	free-Okadaic-Acid	56.12	50.77	µg/kg	-0.76
QST198SS	Multi toxin standard solution	Total-free-OA+DTX1+DTX2	75.65	68.96	µg OA eq./kg	-0.70
QST198SS	Multi toxin standard solution	YTX	0.032	0.028	mg YTX eq./kg	&
QST198SS	Multi toxin standard solution	Total-YTX	0.031	0.028	mg YTX eq./kg	&
QST199BT	DSP-AZP extract	AZA-1	39.87	34.90	µg/kg	-0.99
QST199BT	DSP-AZP extract	AZA-2	9.61	10.20	µg/kg	0.47
QST199BT	DSP-AZP extract	AZA-3	12.88	12.90	µg/kg	0.01
QST199BT	DSP-AZP extract	AZA-total	74.68	71.30	µg AZA eq./kg	-0.36
QST199BT	DSP-AZP extract	Free-DTX1	1.71	1.40	µg/kg	-1.17
QST199BT	DSP-AZP extract	Free-DTX2	1.99	1.90	µg/kg	-0.29

Sample ID	Sample Description	Determinand	Assigned Value	Reported Value	Units	Z Score
QST199BT	DSP-AZP extract	free-Okadaic-Acid	14.79	13.80	µg/kg	-0.52
QST199BT	DSP-AZP extract	Total-free-OA+DTX1+DTX2	16.40	16.40	µg OA eq./kg	0.00
QST199BT	DSP-AZP extract	Total OA and PTX	20.36	22.00	µg OA eq./kg	0.63
QST199BT	DSP-AZP extract	Total-DTX1	2.71	2.60	µg/kg	-0.29
QST199BT	DSP-AZP extract	Total-DTX2	2.20	1.80	µg/kg	-1.22
QST199BT	DSP-AZP extract	Total-Okadaic-Acid	17.83	18.20	µg/kg	0.16
QST199BT	DSP-AZP extract	Total-hy-OA+DTX1+DTX2	21.22	22.00	µg OA eq./kg	0.29
QST199BT	DSP-AZP extract	YTX	0.007	0.007	mg/kg	&
QST199BT	DSP-AZP extract	45-OH-YTX	0.005	0.004	mg/kg	&
QST199BT	DSP-AZP extract	Total-YTX	0.008	0.012	mg YTX eq./kg	&
QST200BT	Mussel homogenate	AZA-1	116.35	96.20	µg/kg	-1.38
QST200BT	Mussel homogenate	AZA-2	38.55	44.60	µg/kg	1.24
QST200BT	Mussel homogenate	AZA-3	26.84	24.90	µg/kg	-0.57
QST200BT	Mussel homogenate	AZA-total	215.70	211.40	µg AZA eq./kg	-0.16
QST200BT	Mussel homogenate	Free-DTX1	70.87	62.60	µg/kg	-0.93
QST200BT	Mussel homogenate	Free-DTX2	479.35	486.20	µg/kg	0.11
QST200BT	Mussel homogenate	free-Okadaic-Acid	91.85	84.40	µg/kg	-0.65
QST200BT	Mussel homogenate	Total-free-OA+DTX1+DTX2	458.37	438.70	µg OA eq./kg	-0.34
QST200BT	Mussel homogenate	Total-DTX1	100.74	92.70	µg/kg	-0.64
QST200BT	Mussel homogenate	Total-DTX2	725.01	570.70	µg/kg	-1.70
QST200BT	Mussel homogenate	Total-Okadaic-Acid	211.74	174.10	µg/kg	-1.42
QST200BT	Mussel homogenate	Total OA and PTX	767.68	609.20	µg OA eq./kg	-1.65
QST200BT	Mussel homogenate	Total-hy-OA+DTX1+DTX2	778.22	609.20	µg OA eq./kg	-1.74
QST200BT	Mussel homogenate	YTX	0.890	1.016	mg/kg	0.78
QST200BT	Mussel homogenate	45-OH-YTX	0.003	0.017	mg/kg	&
QST200BT	Mussel homogenate	Total-YTX	0.893	1.033	mg YTX eq./kg	0.86
QST201BT	Mussel homogenate	Free-DTX2	405.35	399.00	µg/kg	-0.13
QST201BT	Mussel homogenate	free-Okadaic-Acid	144.59	152.50	µg/kg	0.44
QST201BT	Mussel homogenate	Total-free-OA+DTX1+DTX2	389.13	391.90	µg OA eq./kg	0.06
QST201BT	Mussel homogenate	Total-DTX2	699.37	602.00	µg/kg	-1.11
QST201BT	Mussel homogenate	Total-Okadaic-Acid	460.05	382.20	µg/kg	-1.35
QST201BT	Mussel homogenate	Total OA and PTX	889.02	743.40	µg OA eq./kg	-1.31
QST201BT	Mussel homogenate	Total-hy-OA+DTX1+DTX2	898.90	743.40	µg OA eq./kg	-1.38