

Agri-Food & Biosciences Institute

Annual report for marine biotoxin analysis, official
control monitoring programme for Northern
Ireland

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Quality statement: This report is a compilation of the information included on the reports provided weekly/monthly to the FSANI and showing the results of the toxin analyses undertaken on samples submitted by third parties. All results were quality checked and approved prior to release to the FSANI and the results compiled in this report have been further checked against a copy of the original reports held on a central database. Information relating to the origin of the samples (place, date and time of collection) is as provided by sampling staff and has not undergone verification checks by AFBI.

Method standard operating procedures used during the course of the programme:

Determination of Domoic Acid in Shellfish by High Performance Liquid Chromatography (CSD 406) Determination of Paralytic Shellfish Poison by High Performance Liquid Chromatography (CSD 408/409)

Determination of Lipophilic Toxins in Shellfish Species by LC-MS/MS (CSD 379)

Results of programme

Table 1: Monitoring samples received 2024.

Sample type	Number of samples
Oyster	126
Mussel	130

256 samples in 2024

Table 2: Tests carried out 2024.

Test	Oyster	Mussel	Total
Domoic Acid	93	98	191
PSP	89	82	171
Lipophilic Toxins	126	114	240

602 biotoxin tests carried out in 2024.

Table 3: Positive Northern Ireland monitoring samples 2023 (levels above the regulatory limits).

Test	Oyster	Mussel	Total
Domoic Acid	0	0	0
PSP	0	0	0
Lipophilic Toxins	0	0	0

0 monitoring samples reported as exceeding regulatory limits in 2024.

Table 4: Turnaround times 2024.

Test	Target	Percentage within target
Domoic Acid	95% in 3 days	100
PSP	95% in 3 days	100
Lipophilic Toxins	95% in 3 days	100

Table 5: Overview of the toxin results obtained for each bed monitored in 2024 within NI classified production areas**Maximum toxin levels detected in 2024.**

Site location	Site Identification Reference (SIR)	No. of samples received	Shellfish Species	DSP: OA/DTX/PTX (µg/kg)	DSP: AZA (µg/kg)	DSP: YTX (mg/kg)	PSP (µg/kg)	ASP (µg/g)
Belfast Lough	B1-AFFNI 55	18	Mussel	20	NEG	NEG	NEG	<0.3
Belfast Lough	B3-AFFNI 50	18	Mussel	19	NEG	NEG	NEG	<0.3
Belfast Lough	B12-AFFNI 54	16	Mussel	26	NEG	NEG	NEG	<0.3
Belfast Lough	B20-AFFNI 53	18	Mussel	40	NEG	NEG	NEG	<0.3
Carlingford Lough	C1-AFFNI 27	22	Mussel	NEG	NEG	NEG	NEG	<0.3
Carlingford Lough	C11-AFFNI 84	22	Oyster	NEG	NEG	NEG	NEG	1.3
Carlingford Lough	C15-AFFNI 89	22	Oyster	NEG	NEG	NEG	NEG	0.4
Carlingford Lough	NW-Wild Fishery	22	Mussel	13	NEG	NEG	NEG	<0.3
Dundrum Bay	DB1-AFFNI 95A	26	Oyster	30	NEG	NEG	NEG	4.1
Killough	K1-AFFNI 18	12	Oyster	NEG	NEG	NEG	NEG	2.8
Larne Lough	L3-AFFNI 88	13	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA3O-Wild Fishery	8	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA4O- Wild Fishery	8	Oyster	NEG	NEG	NEG	NEG	<0.3
Strangford Lough	S2-AFFNI 42	16	Mussel	14	NEG	NEG	NEG	<0.3
Strangford Lough	S7-AFFNI 76	15	Oyster	NEG	NEG	NEG	72	0.8

Proficiency test results for methods used for official control purposes in 2024.

Performance in proficiency tests is assessed using a z-score. This is calculated for each participant's data.

Each sample /matrix combination is given an assigned value and z-score is calculated thus:

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

The total error is based on values set for the proportional error and the constant error. These values are set by the organisers. The numbers in the z column indicate:

$|z| < 2$ satisfactory performance

$2 < |z| < 3$ questionable performance

$|z| > 3$ unsatisfactory performance

Domoic Acid 2023 Proficiency Test Summaries

Table 6: Quasimeme round 2024.1 - HPLC.

Sample ID	Sample description	Assigned value (mg/kg)	Reported value (mg/kg)	z-score
QST350BT	Mussel Tissue	39.3	44.9	1.4
QST351BT	Razor clam Tissue	21.0	22.2	0.6
QST352BT	Mussel tissue spiked with king scallop	33.0	35.8	0.8

Table 7: Quasimeme round 2024.2 - HPLC.

Sample ID	Sample description	Assigned value (mg/kg)	Reported value (mg/kg)	z-score
QST359BT	Scallop Tissue	25.5	32.5	2.7
QST360BT	Cockle Tissue	16.4	16.4	0
QST361BT	Mussel Tissue	6.2	6.3	0.1

Table 8: EURLMB 2024 (NI NRL Wageningen) - HPLC.

Sample ID	Sample description	Assigned value (mg/kg)	Reported value (mg/kg)	z-score
EURLMB/24/A/01	Mussel Tissue	8.9	7.7	-1.20
EURLMB/24/A/02	Scallop Tissue	11.2	10.1	-0.85

PSP 2024 proficiency test summaries

Table 9: Quasimeme round 2024.1 - HPLC.

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-score
QST356BT	Oyster tissue	STX	0.669	0.602	µmol/kg	-0.4
QST356BT	Oyster tissue	GTX-2,3	4.36	3.83	µmol/kg	-0.5
QST356BT	Oyster tissue	Total toxicity	1192	1079	µgSTXdiHCl eq./kg	-0.4
QST357BT	Mussel tissue	C1C2	0.894	0.607	µmol/kg	-1.0
QST357BT	Mussel tissue	GTX-1,4	N/A	0.354	µmol/kg	N/A
QST357BT	Mussel tissue	dcSTX	0.113	<0.24	µmol/kg	C
QST357BT	Mussel tissue	GTX-5	0.197	<0.24	µmol/kg	C
QST357BT	Mussel tissue	STX	1.340	1.139	µmol/kg	-0.7
QST357BT	Mussel tissue	GTX-2,3	1.240	1.019	µmol/kg	-0.7
QST357BT	Mussel tissue	Total toxicity	891	806	µgSTXdiHCl eq./kg	-0.4
QST358BT	Blue mussel tissue	C1C2	2.570	2.035	µmol/kg	-0.7
QST358BT	Blue mussel tissue	dcGTX-2,3	0.481	0.387	µmol/kg	-0.6
QST358BT	Blue mussel tissue	dcSTX	1.600	1.372	µmol/kg	-0.6
QST358BT	Blue mussel tissue	GTX-5	8.100	7.249	µmol/kg	-0.4
QST358BT	Blue mussel tissue	STX	0.959	0.852	µmol/kg	-0.5
QST358BT	Blue mussel tissue	GTX-2,3	0.825	0.680	µmol/kg	-0.7
QST358BT	Blue mussel tissue	GTX-1,4	N/A	0.469	µmol/kg	N/A
QST358BT	Blue mussel tissue	Total toxicity	1748	1557	µgSTXdiHCl eq./kg	-0.4

Table 10: Quasimeme round 2024.2 - HPLC.

Sample ID	Sample description	Determinant	Assigned Value	Reported Value	Units	z-score
QST365BT	Mussel tissue	dcSTX	0.575	0.401	µmol/kg	-1.1
QST365BT	Mussel tissue	GTX-2,3	5.280	4.230	µmol/kg	-0.8
QST365BT	Mussel tissue	GTX-5	5.110	4.604	µmol/kg	-0.4
QST365BT	Mussel tissue	STX	0.975	0.786	µmol/kg	-0.8
QST365BT	Mussel tissue	dcGTX-2,3	0.666	0.844	µmol/kg	0.8
QST365BT	Mussel tissue	C1C2	4.570	4.085	µmol/kg	-0.3
QST365BT	Mussel tissue	Total toxicity	2340	1836	µgSTXdiHCl eq./kg	-0.8
QST366BT	Mussel tissue	GTX-1,4	N/A	0.308	µmol/kg	N/A
QST366BT	Mussel tissue	dcSTX	0.662	0.462	µmol/kg	-1.1
QST366BT	Mussel tissue	GTX-2,3	1.030	0.753	µmol/kg	-1.0
QST366BT	Mussel tissue	GTX-5	1.140	0.946	µmol/kg	-0.7
QST366BT	Mussel tissue	STX	0.762	0.607	µmol/kg	-0.8
QST366BT	Mussel tissue	dcGTX-2,3	0.906	0.940	µmol/kg	0.1
QST366BT	Mussel tissue	C1C2	1.200	0.958	µmol/kg	-0.8
QST366BT	Mussel tissue	Total toxicity	1057	891	µgSTXdiHCl eq./kg	-0.6
QST367BT	Oyster tissue	GTX-2,3	2.000	1.649	µmol/kg	-0.7
QST367BT	Oyster tissue	STX	0.273	0.204	µmol/kg	-0.8
QST367BT	Oyster tissue	Total toxicity	565	444	µgSTXdiHCl eq./kg	-0.8

Table 11: EURLMB 2024 (NI NRL Wageningen) - HPLC.

Sample ID	Sample description	Determinant	Assigned Value	Reported Value	Units	z-score
EURLMB/24/P/01	Med. mussel Tissue	GTX-2,3	148	117	µgSTXdiHCl eq./kg	-0.90
EURLMB/24/P/01	Med. mussel Tissue	STX	48	47	µgSTXdiHCl eq./kg	-0.04
EURLMB/24/P/01	Med. mussel Tissue	GTX-1,4	282	365	µgSTXdiHCl eq./kg	1.34
EURLMB/24/P/01	Med. mussel Tissue	Total toxicity	478	529	µgSTXdiHCl eq./kg	0.57
EURLMB/24/P/02	Manila clam Tissue	GTX-2,3	202	140	µgSTXdiHCl eq./kg	-1.52
EURLMB/24/P/02	Manila clam Tissue	STX	29	19	µgSTXdiHCl eq./kg	-1.15
EURLMB/24/P/02	Manila clam Tissue	GTX-1,4	380	618.6	µgSTXdiHCl eq./kg	3.06
EURLMB/24/P/02	Manila clam Tissue	Total toxicity	602	777	µgSTXdiHCl eq./kg	1.58

Lipophilic toxins 2024 proficiency test summaries

Table 12: Quasimeme round 2024.1- LC-MS/MS.

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST353BT	Mussel tissue	AZA-1	418	403	µg/kg	-0.2
QST353BT	Mussel tissue	AZA-2	111	109	µg/kg	-0.1
QST353BT	Mussel tissue	AZA-3	80.6	87.9	µg/kg	0.4
QST353BT	Mussel tissue	AZA-total	737	723	µg AZA eq./kg	-0.1
QST353BT	Mussel tissue	Free-DTX2	101	108	µg/kg	0.3
QST353BT	Mussel tissue	free-Okadaic-Acid	46.5	44.1	µg/kg	-0.2
QST353BT	Mussel tissue	Total-free-OA+DTX1+DTX2	108	109	µg OA eq./kg	0
QST353BT	Mussel tissue	Total-DTX2	138	140	µg/kg	0
QST353BT	Mussel tissue	Total-Okadaic-Acid	92.5	94.6	µg/kg	0.1
QST353BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	173	179	µg OA eq./kg	0.1
QST353BT	Mussel tissue	Total-OA+PTX2	178	179	µg OA eq./kg	0
QST354BT	Mussel tissue	Free-DTX2	213	235	µg/kg	0.5
QST354BT	Mussel tissue	free-Okadaic-Acid	109	119	µg/kg	0.4
QST354BT	Mussel tissue	Total-free-OA+DTX1+DTX2	238	260	µg/kg	0.4
QST354BT	Mussel tissue	Total-DTX2	298	323	µg/kg	0.4
QST354BT	Mussel tissue	Total-Okadaic-Acid	252	277	µg/kg	0.5
QST354BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	433	471	µg OA eq./kg	0.4
QST354BT	Mussel tissue	Total-OA+PTX2	454	471	µg OA eq./kg	0.2
QST355BT	Queen scallop tissue	Free-DTX1	78.6	111	µg/kg	1.5
QST355BT	Queen scallop tissue	Free-PTX2	14.1	14.6	µg/kg	0.1
QST355BT	Queen scallop tissue	free-Okadaic-Acid	14.0	13.6	µg/kg	-0.1
QST355BT	Queen scallop tissue	Total-free-OA+DTX1+DTX2	89.4	125	µg OA eq./kg	1.7

QST355BT	Queen scallop tissue	Total-DTX1	588	965	µg/kg	2.3
QST355BT	Queen scallop tissue	Total-Okadaic-Acid	82.8	88.6	µg/kg	0.3
QST355BT	Queen scallop tissue	Total-hy-OA+DTX1+DTX2	None assigned	1053	µg OA eq./kg	N/A
QST355BT	Queen scallop tissue	Total-OA+PTX2	None assigned	1068	µg OA eq./kg	N/A
QST355BT	Queen scallop tissue	YTX	1.61	2.028	mg/kg	1.1
QST355BT	Queen scallop tissue	homo-YTX	0.042	0.031	mg/kg	-0.8
QST355BT	Queen scallop tissue	45 -OH-YTX	0.122	0.164	mg/kg	1.0
QST355BT	Queen scallop tissue	Total-YTX	1.72	2.223	mg YTX eq./kg	1.4

Table 13: Quasimeme round 2024.2- LC-MS/MS.

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-Score
QST362BT	Mussel tissue	Free-DTX1	130	115	µg/kg	-0.4
QST362BT	Mussel tissue	Free-DTX2	117	111	µg/kg	-0.3
QST362BT	Mussel tissue	Free-Okadaic-Acid	58.8	59.0	µg/kg	0
QST362BT	Mussel tissue	Total-free-OA+DTX1+DTX2	267	241	µg OA eq./kg	-0.5
QST362BT	Mussel tissue	Total-DTX1	154	148	µg/kg	-0.2
QST362BT	Mussel tissue	Total-DTX2	346	333	µg/kg	-0.2
QST362BT	Mussel tissue	Total-Okadaic-Acid	303	319	µg/kg	0.3
QST362BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	664	666	µg OA eq./kg	0
QST362BT	Mussel tissue	Total-OA+PTX2	660	666	µg OA eq./kg	0.1
QST362BT	Mussel tissue	YTX	0.054	0.061	mg/kg	0.4
QST362BT	Oyster tissue	45 -OH-YTX	0.023	0.0256	mg/kg	0.4
QST362BT	Mussel tissue	Total-YTX	0.071	0.0866	mg YTX eq./kg	0.7
QST363BT	Oyster tissue	AZA-1	329	275.9	µg/kg	-0.8
QST363BT	Oyster tissue	AZA-2	86.2	63.9	µg/kg	-1.3
QST363BT	Oyster tissue	AZA-3	62.8	43.5	µg/kg	-1.5
QST363BT	Oyster tissue	AZA-total	573	451.2	µg AZA eq./kg	-1.2
QST363BT	Oyster tissue	Free-Okadaic-Acid	52.2	43.4	µg/kg	-0.8
QST363BT	Oyster tissue	Total-free-OA+DTX1+DTX2	56.4	43.4	µg OA eq./kg	-1.0
QST363BT	Oyster tissue	Total-Okadaic-Acid	79.7	71.5	µg/kg	-0.5
QST363BT	Oyster tissue	Total-hy-OA+DTX1+DTX2	82.8	71.5	µg OA eq./kg	-0.6
QST363BT	Oyster tissue	Total-OA+PTX2	86.3	71.5	µg OA eq./kg	-0.8
QST364BT	Mussel tissue	Free-Okadaic-Acid	109	99.8	µg/kg	-0.4

QST364BT	Mussel tissue	Total-free-OA+DTX1+DTX2	122	99.8	µg OA eq./kg	-0.9
QST364BT	Mussel tissue	Total-DTX-1	27.6	22.1	µg/kg	-0.8
QST364BT	Mussel tissue	Total-Okadaic-Acid	391	384.1	µg/kg	-0.1
QST364BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	424	406.2	µg OA eq./kg	-0.2
QST364BT	Mussel tissue	Total-OA+PTX2	427	406.2	µg OA eq./kg	-0.3
QST364BT	Mussel tissue	YTX	1.08	0.9765	mg/kg	-0.4
QST364BT	Oyster tissue	45 -OH-YTX	0.02	0.026	mg/kg	0.8
QST364BT	Mussel tissue	Total-YTX	1.06	1.0025	mg YTX eq./kg	-0.3

Table 14: EURLMB 2024 (NI NRL Wageningen)- LC-MS/MS.

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-Score
EURLMB/24/L/01	Mussel tissue	free-Okadaic-Acid	128	103	µg/kg	-0.86
EURLMB/24/L/01	Mussel tissue	Total-OA	206	200	µg/kg	-0.13
EURLMB/24/L/01	Mussel tissue	Total-hy-OA+DTX1+DTX2	211	200	µg OA eq./kg	-0.25
EURLMB/24/L/01	Mussel tissue	YTX	0.30	0.32	mg/kg	0.34
EURLMB/24/L/01	Mussel tissue	45 -OH-YTX	0.11	0.11	mg/kg	0.12
EURLMB/24/L/01	Mussel tissue	Total-YTX	0.38	0.44	mg YTX eq./kg	0.72
EURLMB/24/L/02	Mussel tissue	AZA-1	673	699	µg/kg	0.22
EURLMB/24/L/02	Mussel tissue	AZA-2	171	158	µg/kg	-0.37
EURLMB/24/L/02	Mussel tissue	AZA-3	131	128	µg/kg	-0.08
EURLMB/24/L/02	Mussel tissue	AZA-total	1147	1163	µg AZA eq./kg	0.09
EURLMB/24/L/02	Mussel tissue	free-Okadaic-Acid	71.2	56.3	µg/kg	-0.90
EURLMB/24/L/02	Mussel tissue	Total-OA	137	135	µg/kg	-0.06
EURLMB/24/L/02	Mussel tissue	Total-DTX-2	28.0	11.0	µg/kg	-2.19
EURLMB/24/L/02	Mussel tissue	Total-hy-OA+DTX1+DTX2	154	142	µg OA eq./kg	-0.34

Overall summary of proficiency tests

The proficiency tests results obtained for ASP were 87.5% satisfactory (7/8), 12.5% questionable (1/8), 0% unsatisfactory (0/8); for PSP were 98% satisfactory (43/44), 0% questionable (0/44) and 2% unsatisfactory (1/44); and for lipophilic toxins were 97% satisfactory (72/74), 3% questionable (2/74), 0% unsatisfactory (0/74).

All questionable and unsatisfactory z-scores have been investigated and addressed via internal non-conformance reports employing AFBI quality management system.

Overall performance for the three toxin groups is 96.8% satisfactory (122/126), 2.4% questionable (3/126) and 0.8% unsatisfactory (1/126).