



Agri-Food & Biosciences Institute

Annual report for marine biotoxin analysis, official
control monitoring programme for Northern Ireland
1st January 2022 – 31st December 2022

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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 2022

Sample type	Number of samples
Oyster	255
Mussel	266

521 samples in 2022

Table 2: Tests carried out 2022

Test	Oyster	Mussel	Total
Domoic Acid	100	107	207
PSP	97	103	200
Lipophilic Toxins	253	258	511

918 tests carried out in 2022.

Table 3: Positive Northern Ireland monitoring samples 2022 (levels above the regulatory limits)

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

0 positive tests in 2022

Table 4: Turnaround times 2022

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 2022 within NI classified production areas

Maximum toxin levels detected in 2022

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	28	Mussel	28	NEG	NEG	502	<0.3
Belfast Lough	B3-AFFNI 50	28	Mussel	25	NEG	NEG	284	<0.3
Belfast Lough	B12-AFFNI 54	28	Mussel	71	NEG	NEG	115	<0.3
Belfast Lough	B20-AFFNI 53	28	Mussel	34	NEG	NEG	44	<0.3
Carlingford Lough	C1-AFFNI 27	51	Mussel	21	NEG	NEG	NEG	<0.3
Carlingford Lough	C11-AFFNI 84	51	Oyster	48	NEG	NEG	NEG	0.41
Carlingford Lough	C15-AFFNI 89	51	Oyster	NEG	NEG	NEG	NEG	<0.3
Carlingford Lough	NW-Wild Fishery	51	Mussel	NEG	NEG	NEG	NEG	<0.3
Dundrum Bay	DB1-AFFNI 95A	39	Oyster	47	NEG	NEG	NEG	<0.3
Killough	K1-AFFNI 18	18	Oyster	NEG	NEG	NEG	NEG	0.35
Larne Lough	L3-AFFNI 88	38	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA30-Wild Fishery	9	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA40- Wild Fishery	9	Oyster	NEG	NEG	NEG	NEG	<0.3
Strangford Lough	S2-AFFNI 42	52	Mussel	32	NEG	NEG	NEG	<0.3
Strangford Lough	S7-AFFNI 76	40	Oyster	NEG	NEG	NEG	NEG	<0.3

Proficiency test results for methods used for official control purposes in 2022.

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 2022 Proficiency Test Summaries

Table 6: Quasimeme round 2022.1 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
QST314BT	Mussel Tissue	40.39	43.31	mg/kg	0.56
QST315BT	Scallop Tissue	25.45	25.88	mg/kg	0.13
QST316BT	Scallop Tissue	5.88	6.02	mg/kg	0.17

Table 7: Quasimeme round 2022.2 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
QST323BT	Scallop Tissue	16.9	18.71	mg/kg	0.8
QST324BT	Queen scallop adductor Tissue	24.5	33.96	mg/kg	3.0
QST325BT	Razor clam Tissue	7.70	8.09	mg/kg	0.4

Table 8: EURLMB 2022 (NI NRL Wageningen) - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
EURLMB/22/A/01	Mussel Tissue	4.3	3.8	mg/kg	-0.83
EURLMB/22/A/02	Scallop Tissue	16.1	16.9	mg/kg	0.47

PSP 2022 proficiency test summaries

Table 9: Quasimeme round 2022.1 - HPLC

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-score
QST320BT	Mussel tissue	dc-STX	1.720	1.008	µmol/kg	-2.50
QST320BT	Mussel tissue	GTX-5	0.064	<0.1	µmol/kg	N/A
QST320BT	Mussel tissue	GTX-2,3	0.084	0.048	µmol/kg	-0.60
QST320BT	Mussel tissue	STX	0.092	0.064	µmol/kg	-0.40
QST320BT	Mussel tissue	Total toxicity	751	411	µgSTXdiHCleq./kg	-3.10
QST321BT	Mussel tissue	C1C2	6.86	0.562	µmol/kg	-6.2
QST321BT	Mussel tissue	dc-STX	0.467	0.312	µmol/kg	-1.4
QST321BT	Mussel tissue	GTX-1,4	0.889	1.496	µmol/kg	2.3
QST321BT	Mussel tissue	GTX-2,3	1.96	1.32	µmol/kg	-2.0
QST321BT	Mussel tissue	GTX-5	7.80	6.184	µmol/kg	-1.50
QST321BT	Mussel tissue	STX	0.662	0.552	µmol/kg	-0.80
QST321BT	Mussel tissue	Total toxicity	1558	1622	µgSTXdiHCleq./kg	0.3
QST322BT	Mussel tissue	C-1,2	0.899	0.648	µmol/kg	-1.4
QST322BT	Mussel tissue	dc-STX	0.123	0.072	µmol/kg	-0.80
QST322BT	Mussel tissue	GTX-1,4	0.396	0.640	µmol/kg	1.8

QST322BT	Mussel tissue	GTX-2,3	1.200	0.832	µmol/kg	-1.8
QST322BT	Mussel tissue	GTX-5	0.201	0.176	µmol/kg	-0.3
QST322BT	Mussel tissue	STX	1.23	0.992	µmol/kg	-1.20
QST322BT	Mussel tissue	Total toxicity	916	850	µgSTXdiHCleq./kg	-0.5

Table 10: Quasimeme round 2022.2 - HPLC

Sample ID	Sample description	Determinant	Assigned Value	Reported Value	Units	z-score
QST329BT	Mussel tissue	GTX-1,4	0.256	0.293	µmol/kg	0.40
QST329BT	Mussel tissue	GTX-2,3	6.502	5.602	µmol/kg	-0.90
QST329BT	Mussel tissue	STX	1.830	1.763	µmol/kg	-0.20
QST329BT	Mussel tissue	Total toxicity	2228	2016	µgSTXdiHCl eq./kg	-0.90
QST330BT	Mussel tissue	GTX-2,3	4.220	3.664	µmol/kg	-0.90
QST330BT	Mussel tissue	STX	0.648	0.602	µmol/kg	-0.30
QST330BT	Mussel tissue	Total toxicity	1190	1042	µgSTXdiHCl eq./kg	-0.90
QST331BT	Patagonian scallop	GTX-1,4	0.360	0.331	µmol/kg	-0.30
QST331BT	Patagonian scallop	GTX-2,3	0.398	0.388	µmol/kg	-0.10
QST331BT	Patagonian scallop	STX	0.102	0.101	µmol/kg	0.00
QST331BT	Patagonian scallop	Total toxicity	None assigned	248	µgSTXdiHCl eq./kg	N/A

Table 11: EURLMB 2022 (NI NRL Wageningen) - HPLC

Sample ID	Sample description	Determinant	Assigned Value	Reported Value	Units	z-score
EURLMB/22/P/01	Mussel Tissue	GTX-2,3	284.7	324.8	µgSTXdiHCl eq./kg	0.69
EURLMB/22/P/01	Mussel Tissue	GTX-1,4	866.2	1589.4	µgSTXdiHCl eq./kg	4.75
EURLMB/22/P/01	Mussel tissue	Total toxicity	1138.4	1914	µgSTXdiHCl eq./kg	4.34
EURLMB/22/P/02	Mussel tissue	C1C2	173.3	208.9	µgSTXdiHCl eq./kg	0.63
EURLMB/22/P/02	Mussel tissue	dcSTX	138.3	112.9	µgSTXdiHCl eq./kg	-0.43
EURLMB/22/P/02	Mussel tissue	GTX-5	178.6	171.6	µgSTXdiHCl eq./kg	-0.16
EURLMB/22/P/02	Mussel tissue	GTX-6	178.3	207.8	µgSTXdiHCl eq./kg	0.28
EURLMB/22/P/02	Mussel tissue	C3C4	68.7	34.6	µgSTXdiHCl eq./kg	-1.85
EURLMB/22/P/02	Mussel tissue	Total toxicity	792.2	736	µgSTXdiHCl eq./kg	-0.39

Lipophilic toxins 2022 proficiency test summaries

Table 12: Quasimeme round 2022.1- LC-MS/MS

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST317BT	Mussel tissue	AZA-1	701	592.4	µg/kg	-1.20
QST317BT	Mussel tissue	AZA-2	197	184.1	µg/kg	-0.50
QST317BT	Mussel tissue	AZA-3	123	113.3	µg/kg	-0.6
QST317BT	Mussel tissue	AZA-total	1227	1082.4	µg AZA eq./kg	-0.90
QST317BT	Mussel tissue	Free-DTX1	90.5	82.8	µg/kg	-0.6
QST317BT	Mussel tissue	Free-DTX2	567	560.7	µg/kg	-0.1
QST317BT	Mussel tissue	free-Okadaic-Acid	145	120.6	µg/kg	-1.3
QST317BT	Mussel tissue	Total-free-OA+DTX1+DTX2	590	539.8	µg OA eq./kg	-0.7
QST317BT	Mussel tissue	Total-DTX1	129	111.7	µg/kg	-1
QST317BT	Mussel tissue	Total-DTX2	786	747	µg/kg	-0.4
QST317BT	Mussel tissue	Total-Okadaic-Acid	350	273.7	µg/kg	-1.7
QST317BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	978	833.6	µg OA eq./kg	-1
QST317BT	Mussel tissue	Total-OA+PTX2	955	833.6	µg OA eq./kg	-1.2
QST317BT	Mussel tissue	YTX	0.241	0.251	mg/kg	0.2
QST317BT	Mussel tissue	homo-YTX	1.08	0.996	mg/kg	-0.5

QST317BT	Mussel tissue	45-OH-YTX	0.147	0.158	mg/kg	0.3
QST317BT	Mussel tissue	45-OH-homo-YTX	0.475	0.596	mg/kg	1.4
QST317BT	Mussel tissue	Total-YTX	1.67	1.703	mg YTX eq./kg	-0.3
QST318BT	Mussel extract	AZA-1	218	189	µg/kg	-1.0
QST318BT	Mussel extract	AZA-2	62.3	64.7	µg/kg	0.3
QST318BT	Mussel extract	AZA-3	49.3	50.9	µg/kg	0.3

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST318BT	Mussel tissue	AZA-total	399	376.7	µg AZA eq./kg	-0.4
QST318BT	Mussel tissue	Free-DTX2	318	287.8	µg/kg	-0.7
QST318BT	Mussel tissue	free-Okadaic-Acid	67.4	52.2	µg/kg	-1.7
QST318BT	Mussel tissue	Total-free-OA+DTX1+DTX2	263	224.8	µg OA eq./kg	-1.1
QST318BT	Mussel tissue	Total-DTX2	456	418.3	µg/kg	-0.6
QST318BT	Mussel tissue	Total-Okadaic-Acid	151	118.2	µg/kg	-1.6
QST318BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	421	369.2	µg OA eq./kg	-1
QST318BT	Mussel tissue	Total-OA+PTX2	417	369.2	µg OA eq./kg	-0.9
QST319BT	Mussel tissue	AZA-1	70.8	66.1	µg/kg	-0.5
QST319BT	Mussel tissue	AZA-2	18.8	22.3	µg/kg	1.4
QST319BT	Mussel tissue	AZA-3	15.3	15.1	µg/kg	-0.1
QST319BT	Mussel tissue	AZA-total	123	127.4	µg AZA eq./kg	0.3

QST319BT	Mussel tissue	Total-DTX1	5.64	<10	µg/kg	N/A
QST319BT	Mussel tissue	Free-DTX2	46.1	49.4	µg/kg	0.5
QST319BT	Mussel tissue	free-Okadaic-Acid	112	97.4	µg/kg	-1.0
QST319BT	Mussel tissue	Total-free-OA+DTX1+DTX2	142	127	µg OA eq./kg	-0.8
QST319BT	Mussel tissue	Total-DTX1	7.42	<10	µg/kg	N/A
QST319BT	Mussel tissue	Total-DTX2	61.4	57.8	µg/kg	-0.4
QST319BT	Mussel tissue	Total-Okadaic-Acid	202	154.3	µg/kg	-1.8
QST319BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	238	189	µg OA eq./kg	-1.6
QST319BT	Mussel tissue	Total-OA+PTX2	235	189	µg OA eq./kg	-1.4
QST319BT	Mussel tissue	YTX	0.0372	0.037	mg/kg	0
QST319BT	Mussel tissue	Total-YTX	0.0412	0.037	mg YTX eq./kg	-0.3

Table 13: Quasimeme round 2022.2- LC-MS/MS

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-Score
QST326BT	Mussel tissue	AZA-1	77.9	63.7	µg/kg	-1.4
QST326BT	Mussel tissue	AZA-2	25.5	21.7	µg/kg	-1.1
QST326BT	Mussel tissue	AZA-3	19	17.9	µg/kg	-0.4
QST326BT	Mussel tissue	AZA-total	154	127.8	µg AZA eq./kg	-1.3
QST326BT	Mussel tissue	Free-DTX1	12.9	9.8	µg/kg	-1.7
QST326BT	Mussel tissue	Free-DTX2	44.1	45.8	µg/kg	0.3

QST326BT	Mussel tissue	free-Okadaic-Acid	12.8	10.8	µg/kg	-1.1
QST326BT	Mussel tissue	Total-free-OA+DTX1+DTX2	50.3	48.1	µg OA eq./kg	-0.3
QST326BT	Mussel tissue	Total-DTX1	18.8	12.8	µg/kg	-2.2
QST326BT	Mussel tissue	Total-DTX2	70.2	76.8	µg/kg	0.7
QST326BT	Mussel tissue	Total-Okadaic-Acid	46.4	36.7	µg/kg	-1.5
QST326BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	107	95.6	µg OA eq./kg	-0.7
QST326BT	Mussel tissue	Total-OA+PTX2	104	95.6	µg OA eq./kg	-0.6
QST327BT	Mussel tissue	Free-DTX1	168	123.9	µg/kg	-1.9
QST327BT	Mussel tissue	Free-DTX2	4.55	<8.0	µg/kg	N/A
QST327BT	Mussel tissue	free-Okadaic-Acid	23.8	18.6	µg/kg	-1.5
QST327BT	Mussel tissue	Total-free-OA+DTX1+DTX2	190	142.5	µg OA eq./kg	-1.9
QST327BT	Mussel tissue	Total-DTX1	231	153.9	µg/kg	-2.4
QST327BT	Mussel tissue	Total-DTX2	5.05	<8.0	µg/kg	N/A
QST327BT	Mussel tissue	Total-Okadaic-Acid	41.3	31.1	µg/kg	-1.8
QST327BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	280	185	µg OA eq./kg	-2.4
QST327BT	Mussel tissue	Total-OA+PTX2	275	185	µg OA eq./kg	-2.2
QST327BT	Mussel tissue	YTX	0.23	0.231	mg/kg	0
QST327BT	Mussel tissue	homoYTX	0.989	0.885	mg/kg	-0.8

QST327BT	Mussel tissue	45-OH-homoYTX	0.365	0.424	mg/kg	1.0
QST327BT	Mussel tissue	45-OH-YTX	0.114	0.141	mg/kg	1.1
QST327BT	Mussel tissue	Total-YTX	1.52	1.469	mg YTX eq./kg	-0.2
QST328BT	Mussel tissue	AZA-1	328	228.3	µg/kg	-2.2
QST328BT	Mussel tissue	AZA-2	89.4	69.8	µg/kg	-1.7
QST328BT	Mussel tissue	AZA-3	57.7	49.9	µg/kg	-1.0
QST328BT	Mussel tissue	AZA-total	568	423.8	µg AZA eq./kg	-1.9
QST328BT	Mussel tissue	Free-DTX2	N/A	9.7	µg/kg	N/A
QST328BT	Mussel tissue	free-Okadaic-Acid	50.7	45.6	µg/kg	-0.7
QST328BT	Mussel tissue	Total-free-OA+DTX1+DTX2	50.3	51.4	µg OA eq./kg	0.2
QST328BT	Mussel tissue	Total-DTX2	5.96	9.7	µg/kg	2.0
QST328BT	Mussel tissue	Total-Okadaic-Acid	89	66	µg/kg	-2.0
QST328BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	89.4	71.8	µg OA eq./kg	-1.5
QST328BT	Mussel tissue	Total-OA+PTX2	90.4	71.8	µg OA eq./kg	-1.4

Table 14: EURLMB 2022 (NI NRL Wageningen)- LC-MS/MS

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-Score
EURLMB/22/L/01	Mussel tissue	AZA-1	330.8	311.1	µg/kg	-0.32
EURLMB/22/L/01	Mussel tissue	AZA-2	81.2	86.8	µg/kg	0.29
EURLMB/22/L/01	Mussel tissue	AZA-3	72.5	72.3	µg/kg	-0.01
EURLMB/22/L/01	Mussel tissue	AZA-total	577.7	568.6	µg AZA eq./kg	-0.08
EURLMB/22/L/01	Mussel tissue	Free-DTX2	74.6	65.1	µg/kg	-0.58
EURLMB/22/L/01	Mussel tissue	free-Okadaic-Acid	61.4	47.9	µg/kg	-0.93
EURLMB/22/L/01	Mussel tissue	Total-DTX2	109.8	94.0	µg/kg	-0.62
EURLMB/22/L/01	Mussel tissue	Total-OA	146.0	118.8	µg/kg	-0.83
EURLMB/22/L/01	Mussel tissue	Total-hy-OA+DTX1+DTX2	214.2	175.2	µg OA eq./kg	-0.90
EURLMB/22/L/02	Mussel tissue	free-Okadaic-Acid	403.5	322.8	µg OA eq./kg	-1.09
EURLMB/22/L/02	Mussel tissue	Free-DTX2	20.6	19.1	µg/kg	-0.22
EURLMB/22/L/02	Mussel tissue	Total-OA	643.4	566.5	µg/kg	-0.70
EURLMB/22/L/02	Mussel tissue	Total-DTX2	28.3	34.3	µg/kg	0.90
EURLMB/22/L/02	Mussel tissue	Total-hy-OA+DTX1+DTX2	652.4	403.5	µg OA eq./kg	-0.56

EURLMB/22/L/02	Mussel tissue	YTX	0.39	0.42	mg/kg	0.35
EURLMB/22/L/02	Mussel tissue	45OH YTX	0.32	0.40	mg/kg	1.11
EURLMB/22/L/02	Mussel tissue	Total-YTX group	0.66	0.8	mg YTX eq./kg	1.46

Overall summary of proficiency tests

The proficiency tests for ASP were 87.5% satisfactory (7/8); for PSP were 85% satisfactory (33/39) and for lipophilic toxins were 95% satisfactory (94/99).

All questionable and unsatisfactory z-scores have been investigated and addressed via internal non-conformance reports on our quality assurance system.

Overall performance for the three toxin groups is 92% satisfactory (134/146), 5% questionable and 3% unsatisfactory.