



Agri-Food & Biosciences Institute

Annual report for marine biotoxin analysis, official
control monitoring programme for Northern Ireland
1st January 2021 – 31st December 2021

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

Results of programme

Table 1: Monitoring samples received 2021

Sample type	Number of samples
Oyster	228
Mussel	229

457 samples in 2021

Table 2: Tests carried out 2021

Test	Oyster	Mussel	Total
Domoic Acid	133	113	246
PSP	130	112	242
Lipophilic Toxins	226	219	445

933 tests carried out in 2021

Table 3: Positive Northern Ireland monitoring samples 2021 (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 2021

Table 4: Turnaround times 2021

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 2021 within NI classified production areas

Maximum toxin levels detected in 2021

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	29	Mussel	NEG	NEG	NEG	502	<0.3
Belfast Lough	B3-AFFNI 50	29	Mussel	NEG	NEG	NEG	284	<0.3
Belfast Lough	B12-AFFNI 54	29	Mussel	54	NEG	NEG	115	<0.3
Belfast Lough	B20-AFFNI 53	29	Mussel	NEG	NEG	NEG	44	<0.3
Carlingford Lough	C1-AFFNI 27	13	Mussel	NEG	NEG	NEG	NEG	<0.3
Carlingford Lough	C11-AFFNI 84	14	Oyster	48	NEG	NEG	NEG	<0.3
Carlingford Lough	C15-AFFNI 89	12	Oyster	15	NEG	NEG	NEG	<0.3
Carlingford Lough	C17-AFFNI 92	12	Oyster	NEG	NEG	NEG	NEG	<0.3
Carlingford Lough	C7-AFFNI 73	12	Oyster	17	NEG	NEG	NEG	<0.3
Carlingford Lough	C9-AFFNI 39	12	Oyster	NEG	NEG	NEG	NEG	<0.3
Carlingford Lough	NW-Wild Fishery	13	Mussel	NEG	NEG	NEG	NEG	<0.3
Dundrum Bay	DB1-AFFNI 95A	38	Oyster	38	NEG	NEG	NEG	<0.3
Dundrum Bay	DB1-AFFNI 95A	35	Mussel	158	NEG	NEG	NEG	<0.3
Killough	K1-AFFNI 18	18	Oyster	22	NEG	NEG	NEG	0.35
Larne Lough	L3-AFFNI 88	45	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA3O-Wild Fishery	9	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA4O- Wild Fishery	9	Oyster	NEG	NEG	NEG	NEG	<0.3
Strangford Lough	S2-AFFNI 42	52	Mussel	36	NEG	NEG	NEG	<0.3
Strangford Lough	S6-AFFNI 93	1	Oyster	NEG	NEG	NEG	NEG	<0.3
Strangford Lough	S7-AFFNI 76	46	Oyster	NEG	NEG	NEG	NEG	<0.3

Proficiency test results for methods used for official control purposes in 2021

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 2021 Proficiency Test Summaries

Table 6: Quasimeme round 2021.1 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
QST296BT	Scallop Tissue	26.7	30.5	mg/kg	1.10
QST297BT	Mussel Tissue	34.9	37.6	mg/kg	0.60
QST298BT	Scallop Tissue	43.0	46.2	mg/kg	0.58

Table 7: Quasimeme round 2021.2 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
QST305BT	Scallop Tissue	56.4	67.2	mg/kg	1.52
QST306BT	Mussel Tissue	34.6	38.3	mg/kg	0.83
QST307BT	Scallop Tissue	20.6	21.7	mg/kg	0.42

PSP 2021 proficiency test summaries

Table 8: Quasimeme round 2021.1 - HPLC

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-score
QST302BT	Mussel tissue	dc-STX	0.61	0.5	µmol/kg	-0.84
QST302BT	Mussel tissue	GTX-1,4	0.24	0.23	µmol/kg	-0.07
QST302BT	Mussel tissue	GTX-2,3	5.29	5.18	µmol/kg	-0.13
QST302BT	Mussel tissue	STX	1.20	1.0	µmol/kg	-0.93
QST302BT	Mussel tissue	Total toxicity	1779.62	1800	µgSTXdiHCl eq./kg	0.09
QST303BT	Mussel tissue	GTX-1,4	0.39	0.36	µmol/kg	-0.20
QST303BT	Mussel tissue	GTX-2,3	6.95	6.42	µmol/kg	-0.48
QST303BT	Mussel tissue	STX	1.84	1.46	µmol/kg	-1.25
QST303BT	Mussel tissue	Total toxicity	2221.60	2110	µgSTXdiHCl eq./kg	-0.38
QST304BT	Mussel tissue	C-1,2	5.01	5.06	µmol/kg	0.07
QST304BT	Mussel tissue	dc-GTX-2,3	0.74	0.84	µmol/kg	0.54
QST304BT	Mussel tissue	dc-STX	0.80	0.72	µmol/kg	-0.45
QST304BT	Mussel tissue	GTX-2,3	6.09	5.74	µmol/kg	-0.37
QST304BT	Mussel tissue	GTX-5	5.68	5.33	µmol/kg	-0.38
QST304BT	Mussel tissue	STX	1.05	0.84	µmol/kg	-1.05
QST304BT	Mussel tissue	Total toxicity	2770.61	2371	µgSTXdiHCl eq./kg	-1.03

Table 9: Quasimeme round 2021.2 - HPLC

Sample ID	Sample description	Determinant	Assigned Value	Reported Value	Units	z-score
QST311BT	Mussel tissue	GTX-2,3	3.00	2.37	µmol/kg	-1.25
QST311BT	Mussel tissue	STX	1.36	0.84	µmol/kg	-2.32
QST311BT	Mussel tissue	Total toxicity	1158.36	841	µgSTXdiHCl eq./kg	-2.05
QST312BT	Mussel tissue	GTX-1,4		0.56	µmol/kg	
QST312BT	Mussel tissue	dc-STX	0.35	0.25	µmol/kg	-1.02
QST312BT	Mussel tissue	GTX-2,3	0.94	0.78	µmol/kg	-0.87
QST312BT	Mussel tissue	GTX-5	3.92	4.01	µmol/kg	0.16
QST312BT	Mussel tissue	STX	0.34	0.23	µmol/kg	-1.15
QST312BT	Mussel tissue	C-1,2	3.21	2.69	µmol/kg	-1.04
QST312BT	Mussel tissue	Total toxicity	791.46	804	µgSTXdiHCl eq./kg	0.11
QST313BT	Oyster tissue	GTX-2,3	4.46	3.64	µmol/kg	-1.18
QST313BT	Oyster tissue	STX	0.65	0.42	µmol/kg	-1.71
QST313BT	Oyster tissue	Total toxicity	1156.23	969	µgSTXdiHCl eq./kg	-1.22

Lipophilic toxins 2021 proficiency test summaries

Table 10: Quasimeme round 2021.1- LC-MS/MS

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST299BT	Mussel tissue	AZA-1	96.2	75.4	µg/kg	-1.60
QST299BT	Mussel tissue	AZA-2	19.8	18.6	µg/kg	-0.47
QST299BT	Mussel tissue	AZA-3	34.9	36.1	µg/kg	0.27
QST299BT	Mussel tissue	AZA-total	179.9	159.5	µg AZA eq./kg	-0.86
QST299BT	Mussel tissue	Free-DTX2	4.33	4.5	µg/kg	0.24
QST299BT	Mussel tissue	free-Okadaic-Acid	36.8	36.3	µg/kg	-0.10
QST299BT	Mussel tissue	Total-DTX2	4.08	4.4	µg/kg	0.53
QST299BT	Mussel tissue	Total-free-OA+DTX1+DTX2	38.6	39	µg OA eq./kg	0.07
QST299BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	47.1	47.1	µg OA eq./kg	0.00
QST299BT	Mussel tissue	Total-Okadaic-Acid	44.7	44.5	µg/kg	-0.03
QST300BT	Mussel extract	AZA-1	444.2	406	µg/kg	-0.66
QST300BT	Mussel extract	AZA-2	118.2	117.3	µg/kg	-0.06
QST300BT	Mussel extract	AZA-3	77.6	80.4	µg/kg	0.28
QST300BT	Mussel extract	AZA-total	765.6	729.7	µg AZA eq./kg	-0.37
QST300BT	Mussel extract	free-Okadaic-Acid	34.1	35.3	µg/kg	0.26
QST300BT	Mussel extract	Total-DTX2	11.9	10.3	µg/kg	-0.89
QST300BT	Mussel extract	Total-free-OA+DTX1+DTX2	37.7	35.3	µg OA eq./kg	-0.46
QST300BT	Mussel extract	Total-hy-OA+DTX1+DTX2	72.7	67.6	µg OA eq./kg	-0.54
QST300BT	Mussel extract	Total-Okadaic-Acid	65.8	61.4	µg/kg	-0.52
QST301BT	Mussel extract	45-OH-YTX	0.03	0.0476	mg/kg	1.00

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST301BT	Mussel tissue	Free-DTX1	123.5	122.8	µg/kg	-0.04
QST301BT	Mussel tissue	Free-DTX2	124	103.5	µg/kg	-1.25
QST301BT	Mussel tissue	free-Okadaic-Acid	63.5	61.3	µg/kg	-0.27
QST301BT	Mussel tissue	Total-DTX1	158	128	µg/kg	-1.34
QST301BT	Mussel tissue	Total-DTX2	370	316.2	µg/kg	-1.10
QST301BT	Mussel tissue	Total-free-OA+DTX1+DTX2	264	246.1	µg OA eq./kg	-0.52
QST301BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	720	628.2	µg OA eq./kg	-0.96
QST301BT	Mussel tissue	Total-Okadaic-Acid	345	310.5	µg/kg	-0.78
QST301BT	Mussel tissue	Total-YTX	0.09	0.1228	mg YTX eq./kg	1.39
QST301BT	Mussel tissue	YTX	0.06	0.0752	mg/kg	0.56

Table 11: Quasimeme round 2021.2- LC-MS/MS

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-Score
QST308BT	Mussel extract	AZA-1	75.29	73	µg/kg	-0.23
QST308BT	Mussel extract	AZA-2	25.8	24.6	µg/kg	-0.35
QST308BT	Mussel extract	AZA-3	18.57	18.6	µg/kg	0.01
QST308BT	Mussel extract	AZA-total	147.45	143.3	µg AZA eq./kg	-0.22
QST308BT	Mussel extract	Free-DTX1	12.028	10.5	µg/kg	-0.93
QST308BT	Mussel extract	Free DTX2	43.20	39.4	µg/kg	-0.68
QST308BT	Mussel extract	Free okadaic acid	13.06	13.9	µg/kg	0.48
QST308BT	Mussel extract	Total OA group + PTX group	109.73	111.4	µg OA eq./kg	0.11
QST308BT	Mussel extract	Total-DTX1	16.77	15.4	µg/kg	-0.56
QST308BT	Mussel extract	Total DTX2	73.92	70.6	µg/kg	-0.35
QST308BT	Mussel extract	Total-free-OA+DTX1+DTX2	49.72	48.0	µg OA eq./kg	-0.27

QST308BT	Mussel extract	Total-hy-OA+DTX1+DTX2	111.46	111.4	µg OA eq./kg	0.00
QST308BT	Mussel extract	Total okadaic acid	48.80	53.7	µg/kg	0.75
QST309BT	Mussel tissue	AZA-1	387.69	338.7	µg/kg	-0.97
QST309BT	Mussel tissue	AZA-2	110.59	111.3	µg/kg	0.05
QST309BT	Mussel tissue	AZA-3	85.03	80.9	µg/kg	-0.37
QST309BT	Mussel tissue	AZA-total	721.42	652.2	µg AZA eq./kg	-0.74
QST309BT	Mussel tissue	Free DTX2	101.8	84.7	µg/kg	-1.27
QST309BT	Mussel tissue	Free okadaic acid	45.88	43.7	µg/kg	-0.36
QST309BT	Mussel tissue	Total OA group + PTX group	172.40	174.20	µg OA eq./kg	0.08
QST309BT	Mussel tissue	Total DTX2	136.25	126.9	µg/kg	-0.50
QST309BT	Mussel tissue	Total-free-OA+DTX1+DTX2	108.06	94.5	µg OA eq./kg	-0.96
QST309BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	170.04	174.20	µg OA eq./kg	0.18
QST309BT	Mussel tissue	Total okadaic acid	87.22	98.1	µg/kg	0.93
QST310BT	Mussel tissue	Free DTX2	212.69	178.5	µg/kg	-1.22
QST310BT	Mussel tissue	Free okadaic acid	105.88	110.5	µg/kg	0.33
QST310BT	Mussel tissue	Total OA group + PTX group	408.79	420.9	µg OA eq./kg	0.22
QST310BT	Mussel tissue	Total DTX2	287.33	274.8	µg/kg	-0.33
QST310BT	Mussel tissue	Total-free-OA+DTX1+DTX2	237.58	217.6	µg OA eq./kg	-0.64
QST310BT	Mussel tissue	Total-hy-OA+DTX1+DTX2	424.42	420.9	µg OA eq./kg	-0.06
QST310BT	Mussel tissue	Total okadaic acid	240.11	256	µg/kg	0.52

Overall summary of proficiency tests

The proficiency tests for ASP were 100% satisfactory (6/6); for PSP were 93% satisfactory (26/28) and for lipophilic toxins were 100% satisfactory (61/61).

A questionable z-score for saxitoxin in sample QST311BT led to an overall questionable z-score for total toxicity of the same sample.

For sample QST312BT GTX1,4 was incorrectly identified instead of dc-GTX2,3. However the overall toxicity of the sample was satisfactory.

The questionable z-scores and misidentification of dc-GTX2,3 have been addressed via an internal non-conformance report on our quality assurance system.

Overall performance for the three toxin groups is 98% satisfactory (93/95), 2% questionable and 0% unsatisfactory.