



Agri-Food & Biosciences Institute

Annual report for marine biotoxin analysis, official
control monitoring programme for Northern Ireland
1st January 2020 – 31st December 2020

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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 2020

Sample type	Number of samples
Oyster	178
Mussel	229

407 samples in 2020

Table 2: Tests carried out 2020

Test	Oyster	Mussel	Total
Domoic Acid	118	147	265
PSP	115	141	256
Lipophilic Toxins	176	220	396

917 tests carried out in 2020

Table 3: Positive Northern Ireland monitoring samples 2020 (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 2020

Table 4: Turn round times 2020

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 2020 within NI classified production areas

Maximum toxin levels detected in 2020

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	33	Mussel	18	NEG	NEG	NEG	<0.3
Belfast Lough	B3-AFFNI 50	33	Mussel	18	NEG	NEG	NEG	<0.3
Belfast Lough	B12-AFFNI 54	33	Mussel	27	NEG	NEG	NEG	<0.3
Belfast Lough	B20-AFFNI 53	33	Mussel	23	NEG	NEG	NEG	<0.3
Carlingford Lough	C1-AFFNI 27	5	Mussel	NEG	NEG	NEG	NEG	<0.3
Carlingford Lough	C11-AFFNI 84	13	Oyster	31	NEG	NEG	NEG	1.5
Carlingford Lough	C15-AFFNI 89	12	Oyster	NEG	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	NEG	NEG	NEG	NEG	0.6
Carlingford Lough	C9-AFFNI 39	8	Oyster	NEG	NEG	NEG	NEG	0.85
Carlingford Lough	NW-Wild Fishery	12	Mussel	20	NEG	NEG	NEG	<0.3
Dundrum Bay	DB1-AFFNI 95A	37	Oyster	28	NEG	NEG	NEG	<0.3
Dundrum Bay	DB1-AFFNI 95A	30	Mussel	95	NEG	NEG	NEG	1.4
Killough	K1-AFFNI 18	19	Oyster	NEG	NEG	NEG	34	2.0
Larne Lough	L3-AFFNI 88	39	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA3M-Wild Fishery	3	Mussel	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA3O-Wild Fishery	8	Oyster	NEG	NEG	NEG	NEG	<0.3
Lough Foyle	PA4M-Wild Fishery	3	Mussel	NEG	NEG	NEG	NEG	<0.3

Lough Foyle	PA40- Wild Fishery	8	Oyster	NEG	NEG	NEG	NEG	<0.3
Strangford Lough	S7-AFFNI 76	10	Oyster	NEG	NEG	NEG	NEG	0.46
Strangford Lough	S2-AFFNI 42	44	Mussel	29	NEG	NEG	NEG	<0.3

Proficiency test results for methods used for official control purposes in 2020

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 2020 Proficiency Test Summaries

Table 6: EURL 2020 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
EURLMB/20/A/01	Scallop homogenate	9.8	10.2	mg/kg	0.20
EURLMB/20/A/02	Scallop homogenate	16.7	12.6	mg/kg	-1.12

Table 7: Quasimeme round 2020.1 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
QST278 BT	Scallop tissue	59.0	69.8	mg/kg	1.44
QST279 BT	Scallop tissue	27.3	32.3	mg/kg	1.42
QST280 BT	Razor clam tissue	24.7	26.4	mg/kg	0.53

Table 8: Quasimeme round 2020.2 - HPLC

Sample ID	Sample description	Assigned value	Reported value	Units	z-score
QST287 BT	Mussel tissue	42.7	48.9	mg/kg	1.1
QST288 BT	Scallop tissue	17.5	24.7	mg/kg	3.2
QST289 BT	Cockle tissue	17.9	21.1	mg/kg	1.4

PSP 2020 proficiency test summaries

Table 9: EURL 2020 - HPLC

Sample ID	Matrix	Determinant	Assigned value	Reported value	Units	z-Score
EURLMB/20/P/01	mussel homogenate	Total toxicity	1659	1704	µgSTX2HCL eq./kg	0.18
EURLMB/20/P/01	mussel homogenate	GTX-2,3	526.20	679.43	µgSTX2HCL eq./kg	1.48
EURLMB/20/P/01	mussel homogenate	STX	1084.70	1024.67	µgSTX2HCL eq./kg	-0.35
EURLMB/20/P/02	mussel homogenate	Total toxicity	1132.21	1325	µgSTX2HCL eq./kg	1.02
EURLMB/20/P/02	mussel homogenate	C-1,2	252.49	375.13	µgSTX2HCL eq./kg	2.03
EURLMB/20/P/02	mussel homogenate	dcSTX	144.64	166.85	µgSTX2HCL eq./kg	0.65
EURLMB/20/P/02	mussel homogenate	GTX-5	203.23	200.68	µgSTX2HCL eq./kg	-0.06
EURLMB/20/P/02	mussel homogenate	dcNEO	63.19	32.75	µgSTX2HCL eq./kg	1.46
EURLMB/20/P/02	mussel homogenate	C-3,4	72.50	55.12	µgSTX2HCL eq./kg	-0.94
EURLMB/20/P/02	mussel homogenate	GTX-6	314.35	493.98	µgSTX2HCL eq./kg	2.43

Table 10: Quasimeme round 2020.1 - HPLC

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-score
QST 284BT	Mussel tissue	dc-STX	0.55	0.47	µmol/kg	-0.65
QST 284BT	Mussel tissue	GTX-2,3	5.16	4.42	µmol/kg	-0.96
QST 284BT	Mussel tissue	STX	1.29	1.00	µmol/kg	-1.33
QST 284BT	Mussel tissue	Total toxicity	1829.54	1533	µgSTXdiHC l eq./kg	-1.23
QST 285BT	Mussel tissue	GTX-2,3	6.33	5.79	µmol/kg	-0.62
QST 285BT	Mussel tissue	STX	2.03	1.58	µmol/kg	-1.43
QST 285BT	Mussel tissue	Total toxicity	2246.76	1880	µgSTXdiHC l eq./kg	-1.27
QST 286BT	Mussel tissue	C-1,2	2.82	2.89	µmol/kg	0.18
QST 286BT	Mussel tissue	dc-STX	0.34	0.27	µmol/kg	-0.75
QST 286BT	Mussel tissue	GTX-2,3	0.89	0.76	µmol/kg	-0.78
QST 286BT	Mussel tissue	GTX-5	4.08	3.62	µmol/kg	-0.78
QST 286BT	Mussel tissue	STX	0.38	0.28	µmol/kg	-0.96
QST 286BT	Mussel tissue	Total toxicity	705.19	617	µgSTXdiHC l eq./kg	-0.91

Table 11: Quasimeme round 2020.2 - HPLC

Sample ID	Sample description	Determinant	Assigned Value	Reported Value	Units	z-score
QST293BT	Mussel tissue	GTX-2,3	2.94	2.44	µmol/kg	-1.1
QST293BT	Mussel tissue	STX	1.39	1.14	µmol/kg	-1.1
QST293BT	Mussel tissue	Total toxicity	1161	968	µgSTXdiHC l eq./kg	-1.3
QST294BT	Mussel tissue	dc-STX	1.64	1.34	µmol/kg	-1.1
QST294BT	Mussel tissue	STX	0.09	0.06	µmol/kg	-0.4

QST294BT	Mussel tissue	Total toxicity	645	524	µgSTXdiHC l eq./kg	-1.3
QST295BT	Mussel tissue	dc-STX	0.49	0.42	µmol/kg	-0.6
QST295BT	Mussel tissue	GTX-2,3	1.99	1.57	µmol/kg	-1.3
QST295BT	Mussel tissue	GTX-5	7.75	7.06	µmol/kg	-0.6
QST295BT	Mussel tissue	STX	0.67	0.54	µmol/kg	-1.0
QST295BT	Mussel tissue	dc-GTX-2,3	0.58	0.46	µmol/kg	-0.6
QST295BT	Mussel tissue	C-1,2	6.54	6.03	µmol/kg	-0.5
QST295BT	Mussel tissue	Total toxicity	1521	1265	µgSTXdiHC l eq./kg	-1.3

Lipophilic toxins 2020 proficiency test summaries

Table 12: EURL 2020 - LC-MS/MS

Sample ID	Matrix	Determinant	Assigned Value	Reported Value	Units	z-score
EURLMB/20/L/01	Mussel homogenate	Total OA group	497.0	533.9	µg OA eq./kg	0.38
EURLMB/20/L/01	Mussel homogenate	Free OA	29.9	26.5	µg/kg	-0.48
EURLMB/20/L/01	Mussel homogenate	Free DTX1	120.5	121.3	µg/kg	0.03
EURLMB/20/L/01	Mussel homogenate	Total OA	59.26	62.9	µg/kg	0.25
EURLMB/20/L/01	Mussel homogenate	Total DTX1	480.33	471.0	µg/kg	-0.11
EURLMB/20/L/01	Mussel homogenate	AZA group	228.07	230.3	µg AZA eq./kg	0.05
EURLMB/20/L/01	Mussel homogenate	AZA1	130.61	132.1	µg/kg	0.05
EURLMB/20/L/01	Mussel homogenate	AZA2	36.79	34.7	µg/kg	-0.25
EURLMB/20/L/01	Mussel homogenate	AZA3	26.78	25.5	µg/kg	-0.22
EURLMB/20/L/02	Mussel homogenate	Total OA group	210.65	219.6	µg OA eq./kg	0.21
EURLMB/20/L/02	Mussel homogenate	Free OA	24.36	20.9	µg/kg	-0.61
EURLMB/20/L/02	Mussel homogenate	Free DTX2	21.53	18.4	µg/kg	-0.57
EURLMB/20/L/02	Mussel homogenate	Total OA	187.39	198.5	µg AZA eq./kg	0.27
EURLMB/20/L/02	Mussel homogenate	Total DTX2	40.12	35.2	µg/kg	-0.53
EURLMB/20/L/02	Mussel homogenate	Total YTX	0.35	0.38	mg YTX eq./kg	0.33
EURLMB/20/L/02	Mussel homogenate	Homo YTX	0.26	0.24	mg/kg	-0.45
EURLMB/20/L/02	Mussel homogenate	45-OH-homo-YTX	0.15	0.16	mg/kg	0.24

Table 13: Quasimeme round 2020.1- LC-MS/MS

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST281B T	Mussel tissue	AZA-1	1327.48	1386.00	µg/kg	0.34
QST281B T	Mussel tissue	AZA-2	340.84	323.90	µg/kg	-0.38
QST281B T	Mussel tissue	AZA-3	305.91	324.30	µg/kg	0.46
QST281B T	Mussel tissue	AZA-total	2348.38	2423.00	µg AZA eq./kg	0.25
QST281B T	Mussel tissue	Free-DTX2	261.30	250.00	µg/kg	-0.33
QST281B T	Mussel tissue	free-Okadaic-Acid	246.94	230.00	µg/kg	-0.51
QST281B T	Mussel tissue	Total-DTX2	340.14	350.00	µg/kg	0.23
QST281B T	Mussel tissue	Total-free-OA+DTX1+DTX2	424.31	380.00	µg OA eq./kg	-0.78
QST281B T	Mussel tissue	Total-hy-OA+DTX1+DTX2	656.80	707.00	µg OA eq./kg	0.58
QST281B T	Mussel tissue	Total-Okadaic-Acid	439.32	497.00	µg/kg	0.99
QST282B T	Mussel extract	AZA-1	91.38	91.70	µg/kg	0.03
QST282B T	Mussel extract	AZA-2	19.52	18.80	µg/kg	-0.28
QST282B T	Mussel extract	AZA-3	38.65	37.40	µg/kg	-0.24
QST282B T	Mussel extract	AZA-total	183.39	177.90	µg AZA eq./kg	-0.23
QST282B T	Mussel extract	Free-DTX2	3.88	2.40	µg/kg	-2.12
QST282B T	Mussel extract	free-Okadaic-Acid	39.02	36.80	µg/kg	-0.40
QST282B T	Mussel extract	Total-DTX2	3.82	3.30	µg/kg	-0.93
QST282B T	Mussel extract	Total-free-OA+DTX1+DTX2	42.13	38.30	µg OA eq./kg	-0.62
QST282B T	Mussel extract	Total-hy-OA+DTX1+DTX2	46.67	46.00	µg OA eq./kg	-0.11
QST282B T	Mussel extract	Total-Okadaic-Acid	44.04	44.00	µg/kg	-0.01

Sample ID	Sample Description	Determinant	Assigned Value	Reported Value	Units	z-Score
QST283B T	Mussel tissue	45-OH-homo-YTX	0.36	0.41	mg/kg	0.71
QST283B T	Mussel tissue	45-OH-YTX	0.16	0.22	mg/kg	1.71
QST283B T	Mussel tissue	Free-DTX1	163.40	162.00	µg/kg	-0.06
QST283B T	Mussel tissue	free-Okadaic-Acid	26.06	15.90	µg/kg	-2.63
QST283B T	Mussel tissue	homo-YTX	0.95	1.00	mg/kg	0.37
QST283B T	Mussel tissue	Total-DTX1	204.03	245.00	µg/kg	1.45
QST283B T	Mussel tissue	Total-free-OA+DTX1+DTX2	191.58	177.90	µg OA eq./kg	-0.50
QST283B T	Mussel tissue	Total-hy-OA+DTX1+DTX2	238.80	281.00	µg OA eq./kg	1.22
QST283B T	Mussel tissue	Total-Okadaic-Acid	41.63	36.00	µg/kg	-0.92
QST283B T	Mussel tissue	Total-YTX	1.62	1.76	mg YTX eq./kg	0.59
QST283B T	Mussel tissue	YTX	0.24	0.33	mg/kg	2.08

Table 14: Quasimeme round 2020.2- LC-MS/MS

Sample ID	Sample description	Determinant	Assigned value	Reported value	Units	z-Score
QST290B T	Mussel extract	Free okadaic acid	12	12.8	µg/kg	0.5
QST290B T	Mussel extract	Free DTX2	68.3	63.9	µg/kg	-0.5
QST290B T	Mussel extract	Total-free-OA+DTX1+DTX2	54	51.2	µg OA eq./kg	-0.4
QST290B T	Mussel extract	Total okadaic acid	19.6	20.8	µg/kg	0.5
QST290B T	Mussel extract	Total DTX2	74.5	71.1	µg/kg	-0.3
QST290B T	Mussel extract	Total-hy-OA+DTX1+DTX2	65.9	63.4	µg OA eq./kg	-0.3
QST290B T	Mussel extract	Total OA group + PTX group	65.8	63.4	µg OA eq./kg	-0.3
QST290B T	Mussel extract	AZA-1	13.6	13.8	µg/kg	0.1
QST290B T	Mussel extract	AZA-2	3.92	4.3	µg/kg	0.7
QST290B T	Mussel extract	AZA-3	3.95	3.6	µg/kg	-0.6
QST290B T	Mussel extract	AZA-total	25.6	26.5	µg AZA eq./kg	0.3
QST290B T	Mussel extract	YTX	0.033	0.046	mg/kg	0.9
QST290B T	Mussel extract	homo-YTX	0.31	0.33	mg/kg	0.5
QST290B T	Mussel extract	45-OH-homo-YTX	0.13	0.22	mg/kg	2.8
QST290B T	Mussel extract	45-OH-YTX	0.04	0.041	mg/kg	0.0
QST290B T	Mussel extract	Total-YTX	0.44	0.53	mg YTX eq./kg	1.3
QST291B T	Mussel tissue	Free okadaic acid	87.4	81.9	µg/kg	-0.4
QST291B T	Mussel tissue	Free DTX2	519	454.3	µg/kg	-0.9
QST291B T	Mussel tissue	Total-free-OA+DTX1+DTX2	411	354.5	µg OA eq./kg	-1.0
QST291B T	Mussel tissue	Total okadaic acid	210	208.9	µg/kg	0.0
QST291B T	Mussel tissue	Total DTX2	774	687.1	µg/kg	-0.8
QST291B T	Mussel tissue	Total-hy-OA+DTX1+DTX2	680	621.1	µg OA eq./kg	-0.6

QST291B T	Mussel tissue	Total OA group + PTX group	689	621.1	µg OA eq./kg	-0.7
QST291B T	Mussel tissue	AZA-1	72.7	59	µg/kg	-1.4
QST291B T	Mussel tissue	AZA-2	22.7	23.3	µg/kg	0.2
QST291B T	Mussel tissue	AZA-3	25.5	22.6	µg/kg	-0.9
QST291B T	Mussel tissue	AZA-total	141	132.6	µg AZA eq./kg	-0.5
QST292B T	Mussel tissue	Free okadaic acid	76.2	73.3	µg/kg	-0.3
QST292B T	Mussel tissue	Free DTX1	156	136.2	µg/kg	-1.0
QST292B T	Mussel tissue	Total-free- OA+DTX1+DTX2	234	209.5	µg OA eq./kg	-0.8
QST292B T	Mussel tissue	Total okadaic acid	355	373.3	µg/kg	0.4
QST292B T	Mussel tissue	Total DTX1	215	188.6	µg/kg	-0.9
QST292B T	Mussel tissue	Total-hy- OA+DTX1+DTX2	573	562	µg OA eq./kg	-0.1
QST292B T	Mussel tissue	Total OA group + PTX group	573	562	µg OA eq./kg	-0.1
QST292B T	Mussel tissue	YTX	0.15	0.17	mg/kg	0.9
QST292B T	Mussel tissue	45-OH-YTX	0.07	0.13	mg/kg	3.3
QST292B T	Mussel tissue	Total-YTX	0.22	0.30	mg YTX eq./kg	2.2

Overall summary of proficiency tests

The proficiency tests for ASP were 87.5% satisfactory (7/8); for PSP were 94% satisfactory (34/36) and for lipophilic toxins were 93% satisfactory (79/85).

There was a single unsatisfactory result for ASP in Quasimeme round 2 due to an interfering peak in the HPLC chromatogram and there was an unsatisfactory result for one of the yessotoxin (YTX) analogues which in turn produced a questionable z-score for the YTX total toxicity.

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

Overall performance for the three toxin groups in the three proficiency tests is 93% satisfactory (120/129), 5.4% questionable and 1.6% unsatisfactory.