

Chemical Analysis Report

WQAP-2022-05-03-01

Florida Department of Environmental Protection
Central Laboratory
2600 Blair Stone Road
Tallahassee, FL 32399-2400
DOH Accreditation E31780

Event Description: **Algal Bloom Response- NEROC**
Request ID: **RQ-2022-04-11-48**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
8800 Baymeadows Way West
Jacksonville, FL 32256
Attn: Jeremy Parrish

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Certified by: Colin Wright, Program Administrator

Date Certified: 10-MAY-2022 09:12

A handwritten signature in black ink, appearing to read "Colin Wright", is displayed on a light gray rectangular background.

Case Narrative

Unless otherwise noted, all samples included in this report were received in accordance with protocols referenced in Chapter 62-160, Florida Administrative Code (F.A.C.). Results published in this report pertain only to the samples as submitted to, and received by the laboratory. All times in this report are adjusted to the applicable Eastern Time Zone (EST or EDT).

Results for the following analytical groups are included in this report: Nutrients and Pesticides.

Scientific notation may be used in reporting very large or small values. Values reported using scientific notation will take the form of the following example: 1.3E+03, which is equivalent to 1.3×10^3 or 1300.

Unless otherwise noted, analytical values for soil and sediment samples are reported on a dry weight basis, and analytical values for waste and tissue samples are reported on a wet weight basis.

Results for TNI accredited tests met requirements established by The NELAC Institute. A double asterisk (**) is used to indicate an analyte/matrix/method for which the laboratory is not TNI accredited by the Florida Department of Health Environmental Laboratory Certification Program or where accreditation for that field of testing is not applicable.

Any significant anomalies or deviations from established protocols are documented in Non-Conformance Reports, which, where appropriate, are included within this analytical report. Additional comments related to specific analytical tests may be included as remarks following the analytical results for each sample. Such comments and remarks are for informational purposes only and are not intended to convey judgement about the usability of the reported data.

A quality control report on the performance of the test method for the submitted samples is included. Uncertainty associated with the analytical results contained in this report can be estimated from the reported quality assurance results and from published quality control acceptance limits for each analytical test. Matrix quality control results (matrix spike recoveries and matrix sample precision) pertain only to the matrix sample tested and do not necessarily reflect test method performance for other samples.

Typical matrix quality control (QC) measurements may include matrix spike recovery, matrix spike duplicate recovery, matrix spike precision and matrix sample precision. Not all matrix QC results may be available or reportable; where they are not an explanation is provided. Typical reasons for unavailable QC results include, but are not limited to, a) insufficient matrix sample to perform some or all QC measurements; b) analyte concentration in the sample replicated was too low for a meaningful measurement of precision and c) analyte concentration in the matrix sample spiked was too high (relative to the amount of analyte spiked) for a meaningful measurement of recovery. Where matrix QC results are unavailable, other method performance metrics (e.g., LCS recovery, LCS precision, surrogate recovery) may be used to assess performance of the method. Comments explaining any missing QC measurements are not intended to convey any adverse conclusions about the quality of the reported data.

Precision is reported as relative percent difference unless otherwise noted.

Quality Control codes as defined below may be used in this report to indicate results that are associated with one or more quality control elements which did not fall within established test method criteria. Such results may be qualified as estimates using a J qualifier as required by 62-160 F.A.C. Explanations are included in the report for any results that were reported as estimates for other reasons.

QC Codes used in this report may include:

- LCS – Recovery for the batch Laboratory Control Sample (LCS) was outside existing control limits;
- MS – Recovery for the batch matrix spike (MS) was outside existing control limits;
- CCV – Recovery for a continuing calibration verification (CCV) standard was outside existing control limits;
- SUR – Recovery of a surrogate (SUR) for associated analytes was outside existing control limits;
- RPD – The precision, measured as relative percent difference (RPD), of batch replicate measurements was outside existing control limits;
- RSD – The precision, measured as relative standard deviation (RSD), of batch replicate measurements was outside existing control limits;
- SMP – Sample - used precision derived from replicate analyses of a sample;

The following data qualifiers are used, where applicable, in this report as specified in 62-160 F.A.C.

- A - Value reported is the mean of two or more determinations.
- B - Results based on colony counts outside the acceptable range.
- I - The reported value is between the laboratory method detection limit and the laboratory practical quantitation limit.
- J - Estimated value and/or the analysis did not meet established quality control criteria.
- K - Actual value is known to be less than value given.
- L - Actual value is known to be greater than value given.
- N - Presumptive evidence of presence of material.
- O - Sampled, but analysis lost or not performed.
- Q - Sample held beyond normal holding time.
- T - Value reported is less than the criterion of detection.
- U - Material was analyzed for but not detected. The reported value is the method detection limit for the sample analyzed.
- V - Analyte was detected in both sample and method blank.
- X - Too few individuals to calculate SCI value.
- Y - The laboratory analysis was from an unpreserved or improperly preserved sample. The data may not be accurate.
- Z - Colonies were too numerous to count (TNTC).

Quality control information from overflow laboratories may not be included in this report. Please refer to the associated report from the overflow laboratory for additional information.

Sample Location: Drs. Lake end of Lawrence Rd

Collection Date/Time: 05/02/2022 11:00

Field ID: NEHAB0073

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2323406	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P413340	
		Cylindrospermopsin	0.10	U	ug/L	P413331	
		Desmethyl microcystin LR	0.25	U	ug/L	P413331	
		Microcystin HIR**	0.25	U	ug/L	P413331	
		Microcystin HtyR**	0.25	U	ug/L	P413331	
		Microcystin LA	0.10	U	ug/L	P413331	
		Microcystin LF	0.10	U	ug/L	P413331	
		Microcystin LR	0.25	U	ug/L	P413331	
		Microcystin LW	0.25	U	ug/L	P413331	
		Microcystin LY	0.10	U	ug/L	P413331	
		Microcystin RR	0.10	U	ug/L	P413331	
		Microcystin WR	0.50	U	ug/L	P413331	
		Neosaxitoxin**	0.50	U	ug/L	P413340	
		Microcystin YR	0.25	U	ug/L	P413331	
		Saxitoxin	0.50	U	ug/L	P413340	
		Nodularin-R**	0.10	U	ug/L	P413331	
2323408	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.004	U	mg P/L	P413396	

Sample Location: Swimming Pen CR @ Whiteys Fish Camp

Collection Date/Time: 05/02/2022 10:15

Field ID: NEHAB0074

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2323407	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P413340	
		Cylindrospermopsin	0.10	U	ug/L	P413331	
		Desmethyl microcystin LR	0.25	U	ug/L	P413331	
		Microcystin HilR**	0.25	U	ug/L	P413331	
		Microcystin HtyR**	0.25	U	ug/L	P413331	
		Microcystin LA	0.10	U	ug/L	P413331	
		Microcystin LF	0.10	U	ug/L	P413331	
		Microcystin LR	0.25	U	ug/L	P413331	
		Microcystin LW	0.25	U	ug/L	P413331	
		Microcystin LY	0.10	U	ug/L	P413331	
		Microcystin RR	0.10	U	ug/L	P413331	
		Microcystin WR	0.50	U	ug/L	P413331	
		Neosaxitoxin**	0.50	U	ug/L	P413340	
		Microcystin YR	0.25	U	ug/L	P413331	
		Saxitoxin	0.50	U	ug/L	P413340	
		Nodularin-R**	0.10	U	ug/L	P413331	
2323409	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.004	U	mg P/L	P413396	

Quality Assurance Report Method Blank Results

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P413396

Component	Result	Code	Units
O-Phosphate-P	0.004	U	mg P/L

Reference Method: EPA 8321B
Batch ID: P413331

Component	Result	Code	Units
Cylindrospermopsin	0.10	U	ug/L
Desmethyl microcystin LR	0.25	U	ug/L
Microcystin HIR	0.25	U	ug/L
Microcystin HtyR	0.25	U	ug/L
Microcystin LA	0.10	U	ug/L
Microcystin LF	0.10	U	ug/L
Microcystin LR	0.25	U	ug/L
Microcystin LW	0.25	U	ug/L
Microcystin LY	0.10	U	ug/L
Microcystin RR	0.10	U	ug/L
Microcystin WR	0.50	U	ug/L
Microcystin YR	0.25	U	ug/L
Nodularin-R	0.10	U	ug/L

Reference Method: EPA 8321B
Batch ID: P413340

Component	Result	Code	Units
Anatoxin-a	0.25	U	ug/L
Neosaxitoxin	0.50	U	ug/L
Saxitoxin	0.50	U	ug/L

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P413396

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
O-Phosphate-P	98.3		P	90 - 110

Reference Method: EPA 8321B
Batch ID: P413331

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Cylindrospermopsin	87.0		P	40 - 150
Desmethyl microcystin LR	85.6		P	40 - 150
Microcystin HllR	82.3		P	40 - 150
Microcystin HtyR	88.3		P	40 - 150
Microcystin LA	86.7		P	40 - 150
Microcystin LF	73.3		P	40 - 150
Microcystin LR	80.4		P	40 - 150
Microcystin LW	94.6		P	40 - 150
Microcystin LY	88.6		P	40 - 150
Microcystin RR	78.3		P	40 - 150
Microcystin WR	81.0		P	40 - 150
Microcystin YR	79.0		P	40 - 150
Nodularin-R	76.7		P	40 - 150

Reference Method: EPA 8321B
Batch ID: P413340

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	104		P	40 - 150
Neosaxitoxin	104		P	40 - 150
Saxitoxin	81.0		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P413396

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2323359	O-Phosphate-P	91.6	90.7	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P413331

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2323101	Cylindrospermopsin	105	100	P/P	40 - 150
2323101	Desmethyl microcystin LR	105	104	P/P	40 - 150
2323101	Microcystin HiLR	103	99.9	P/P	40 - 150
2323101	Microcystin HtyR	103	105	P/P	40 - 150
2323101	Microcystin LA	105	99.9	P/P	40 - 150
2323101	Microcystin LF	85.8	87.4	P/P	40 - 150
2323101	Microcystin LR	109	116	P/P	40 - 150
2323101	Microcystin LW	103	128	P/P	40 - 150
2323101	Microcystin LY	105	110	P/P	40 - 150
2323101	Microcystin RR	89.6	86.8	P/P	40 - 150
2323101	Microcystin WR	92.5	96.2	P/P	40 - 150
2323101	Microcystin YR	94.7	99.6	P/P	40 - 150
2323101	Nodularin-R	91.5	90.3	P/P	40 - 150

Reference Method: EPA 8321B
Batch ID: P413340

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2323105	Anatoxin-a	105	109	P/P	40 - 150
2323105	Neosaxitoxin	48.8	48.2	P/P	40 - 150
2323105	Saxitoxin	49.3	50.9	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P413396

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2323359	O-Phosphate-P	0.554	Spike	P	0 - 20

Reference Method: EPA 8321B
Batch ID: P413331

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2323101	Cylindrospermopsin	4.78	Spike	P	0 - 30
2323101	Desmethyl microcystin LR	0.307	Spike	P	0 - 30
2323101	Microcystin HiLR	3.15	Spike	P	0 - 30
2323101	Microcystin HtyR	2.03	Spike	P	0 - 30
2323101	Microcystin LA	4.84	Spike	P	0 - 30
2323101	Microcystin LF	1.90	Spike	P	0 - 30
2323101	Microcystin LR	6.48	Spike	P	0 - 30
2323101	Microcystin LW	22.0	Spike	P	0 - 30
2323101	Microcystin LY	4.87	Spike	P	0 - 30
2323101	Microcystin RR	3.21	Spike	P	0 - 30
2323101	Microcystin WR	3.93	Spike	P	0 - 30
2323101	Microcystin YR	5.02	Spike	P	0 - 30
2323101	Nodularin-R	1.39	Spike	P	0 - 30

Reference Method: EPA 8321B
Batch ID: P413340

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2323105	Anatoxin-a	3.98	Spike	P	0 - 30
2323105	Neosaxitoxin	1.24	Spike	P	0 - 30
2323105	Saxitoxin	3.12	Spike	P	0 - 30

* Sample, spike and/or laboratory control sample precision (LCS) is reported.

Quality Assurance Report Surrogates

Lab Sample ID: 2323406
Field Sample ID: NEHAB0073

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR ethyl-d5	95.2	P	30 - 160
EPA 8321B	Neosaxitoxin-15N7	74.4	P	30 - 160

Lab Sample ID: 2323407
Field Sample ID: NEHAB0074

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR ethyl-d5	87.1	P	30 - 160
EPA 8321B	Neosaxitoxin-15N7	80.9	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B

Run ID: A111921

Included Lab Sample IDs: 2323406, 2323407

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Cylindrospermopsin	100	102	P/P	50 - 160
Desmethyl microcystin LR	91.6	93.1	P/P	50 - 160
Microcystin HILR	90.9	87.3	P/P	50 - 160
Microcystin HtyR	97.0	106	P/P	50 - 160
Microcystin LA	102	111	P/P	50 - 160
Microcystin LF	72.4	67.7	P/P	50 - 160
Microcystin LR	89.9	92.3	P/P	50 - 160
Microcystin LW	102	84.9	P/P	50 - 160
Microcystin LY	106	97.1	P/P	50 - 160
Microcystin RR	93.6	90.5	P/P	50 - 160
Microcystin WR	85.2	90.5	P/P	50 - 160
Microcystin YR	81.5	84.9	P/P	50 - 160
Nodularin-R	89.2	90.4	P/P	50 - 160

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Run ID: A111929

Included Lab Sample IDs: 2323408, 2323409

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	99.7	100	P/P	90 - 110

Reference Method: EPA 8321B

Run ID: A111977

Included Lab Sample IDs: 2323406, 2323407

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	103	104	P/P	50 - 160
Neosaxitoxin	99.4	101	P/P	50 - 160
Saxitoxin	91.1	98.7	P/P	50 - 160

* Pass/Fail determinations are made for each bracketing calibration verification check.

Control limits for initial calibration checks may be different from those for continuing checks, depending on method requirements.

Where they are different, both control limits are provided.

Quality Assurance Report Summary

Ref. Method	Analyte	LCS % Recovery	MS % Recovery		Precision LCS	SMP	MS
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	98.3	91.6	90.7			0.554
EPA 8321B	Anatoxin-a	104	105	109			3.98
	Cylindrospermopsin	87.0	105	100			4.78
	Desmethyl microcystin LR	85.6	105	104			0.307
	Microcystin HlIR	82.3	103	99.9			3.15
	Microcystin HtyR	88.3	103	105			2.03
	Microcystin LA	86.7	105	99.9			4.84
	Microcystin LF	73.3	85.8	87.4			1.90
	Microcystin LR	80.4	109	116			6.48
	Microcystin LW	94.6	103	128			22.0
	Microcystin LY	88.6	105	110			4.87
	Microcystin RR	78.3	89.6	86.8			3.21
	Microcystin WR	81.0	92.5	96.2			3.93
	Microcystin YR	79.0	94.7	99.6			5.02
	Neosaxitoxin	104	48.8	48.2			1.24
	Nodularin-R	76.7	91.5	90.3			1.39
	Saxitoxin	81.0	49.3	50.9			3.12

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2323408, 2323409
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2323406, 2323407
EPA 8321B	Saxitoxin in water matrices by HPLC/MS/MS	2323406, 2323407

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 365.1 Rev. 2.0 dissolved	05/03/2022			05/03/2022 16:47	Nathaniel J Jones	2323408
	05/03/2022			05/03/2022 16:48	Nathaniel J Jones	2323409
EPA 8321B	05/03/2022	05/04/2022 09:00	Manjita Shrestha	05/04/2022 18:47	Mohammad Ghaffari	2323406
	05/03/2022	05/04/2022 09:00	Manjita Shrestha	05/04/2022 19:04	Mohammad Ghaffari	2323407
	05/03/2022	05/05/2022 08:00	Manjita Shrestha	05/06/2022 20:01	Manjita Shrestha	2323406
	05/03/2022	05/05/2022 08:00	Manjita Shrestha	05/06/2022 20:15	Manjita Shrestha	2323407