

Chemical Analysis Report

WQAP-2021-07-30-05

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

Overflow Analyses Performed By:
GreenWater Laboratories
205 Zeagler Drive
Suite 302
Palatka, FL 32177
DOH Accreditation Not NELAC Certified

Event Description: **Algal Bloom Response - NW ROC - Twin Lakes**
Request ID: **RQ-2021-06-21-53**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
2600 Blair Stone Rd
MS #6511
Tallahassee, FL 32399-2400
Attn: Nia Wellendorf

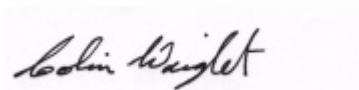
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This Report replaces the previous Report Serial Number: 0133401

Report Comment: The reference method for the dermatotoxins reported by GreenWater Laboratories was corrected.

Revision certified by: Colin Wright, Program Administrator

Date Certified: 04-NOV-2021 11:46



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: Overflow 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: Twin Lake

Collection Date/Time: 07/29/2021 10:50

Field ID: G3NW0215

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2266193	SOP 67 (Greenwater)	Lyngbyatoxin-a (LA)**	0.08	UJQ	ng/mL		
		Aplysia toxin (AT)**	0.07	UJQ	ng/mL		
		Debromoaplysia toxin (DAT)**	0.02	UJQ	ng/mL		

Ref. Method and Comment:

SOP 67 (Greenwater): The Laboratory Fortified Blank recovery failed high for DAT and failed low for LA and AT. One of two matrix spike recoveries failed low for AT. The sample was prepared beyond the holding date/time.

Sample Location: Twin Lake eastern Lake

Collection Date/Time: 07/29/2021 11:30

Field ID: G3NW0449

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2266194	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P401837	
		Cylindrospermopsin	0.10	U	ug/L	P401837	
		Desmethyl microcystin LR	0.25	U	ug/L	P401837	
		Microcystin H1LR**	0.25	U	ug/L	P401837	
		Microcystin HtyR**	0.25	U	ug/L	P401837	
		Microcystin LA	0.10	U	ug/L	P401837	
		Microcystin LF	0.10	U	ug/L	P401837	RPD
		Microcystin LR	0.25	U	ug/L	P401837	
		Microcystin LW	0.25	U	ug/L	P401837	
		Microcystin LY	0.10	U	ug/L	P401837	
		Microcystin RR	0.10	U	ug/L	P401837	
		Microcystin WR	0.50	U	ug/L	P401837	
		Microcystin YR	0.25	U	ug/L	P401837	
		Nodularin-R**	0.10	U	ug/L	P401837	

Sample Location: Coleman Lake

Collection Date/Time: 07/29/2021 12:05

Field ID: G3NW0216

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2266195	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P401837	
		Cylindrospermopsin	0.10	U	ug/L	P401837	
		Desmethyl microcystin LR	0.25	U	ug/L	P401837	
		Microcystin HILR**	0.25	U	ug/L	P401837	
		Microcystin HtyR**	0.25	U	ug/L	P401837	
		Microcystin LA	0.10	U	ug/L	P401837	
		Microcystin LF	0.10	U	ug/L	P401837	RPD
		Microcystin LR	0.29	I	ug/L	P401837	
		Microcystin LW	0.25	U	ug/L	P401837	
		Microcystin LY	0.10	U	ug/L	P401837	
		Microcystin RR	0.10	U	ug/L	P401837	
		Microcystin WR	0.50	U	ug/L	P401837	
		Microcystin YR	0.25	U	ug/L	P401837	
		Nodularin-R**	0.10	U	ug/L	P401837	

Quality Assurance Report

Method Blank Results

Reference Method: EPA 8321B
Batch ID: P401837

Component	Result	Code	Units
Anatoxin-a	0.25	U	ug/L
Cylindrospermopsin	0.10	U	ug/L
Desmethyl microcystin LR	0.25	U	ug/L
Microcystin H1R	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

Quality Assurance Report

Laboratory Control Sample Accuracy

Reference Method: EPA 8321B
Batch ID: P401837

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	98.8		P	40 - 150
Cylindrospermopsin	126		P	40 - 150
Desmethyl microcystin LR	91.7		P	40 - 150
Microcystin HilR	102		P	40 - 150
Microcystin HtyR	104		P	40 - 150
Microcystin LA	85.4		P	40 - 150
Microcystin LF	79.7		P	40 - 150
Microcystin LR	89.0		P	40 - 150
Microcystin LW	97.8		P	40 - 150
Microcystin LY	84.1		P	40 - 150
Microcystin RR	103		P	40 - 150
Microcystin WR	96.4		P	40 - 150
Microcystin YR	88.7		P	40 - 150
Nodularin-R	108		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 8321B

Batch ID: P401837

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2265978	Anatoxin-a	110	112	P/P	40 - 150
2265978	Cylindrospermopsin	126	129	P/P	40 - 150
2265978	Desmethyl microcystin LR	98.6	108	P/P	40 - 150
2265978	Microcystin HilR	107	111	P/P	40 - 150
2265978	Microcystin HtyR	100	107	P/P	40 - 150
2265978	Microcystin LA	95.8	91.8	P/P	40 - 150
2265978	Microcystin LF	69.6	95.2	P/P	40 - 150
2265978	Microcystin LR	88.9	93.3	P/P	40 - 150
2265978	Microcystin LW	77.5	90.0	P/P	40 - 150
2265978	Microcystin LY	93.0	96.9	P/P	40 - 150
2265978	Microcystin RR	105	112	P/P	40 - 150
2265978	Microcystin WR	102	101	P/P	40 - 150
2265978	Microcystin YR	101	113	P/P	40 - 150
2265978	Nodularin-R	108	121	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 8321B

Batch ID: P401837

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2265978	Anatoxin-a	2.29	Spike	P	0 - 30
2265978	Cylindrospermopsin	2.25	Spike	P	0 - 30
2265978	Desmethyl microcystin LR	8.99	Spike	P	0 - 30
2265978	Microcystin H1LR	3.62	Spike	P	0 - 30
2265978	Microcystin HtyR	6.84	Spike	P	0 - 30
2265978	Microcystin LA	4.21	Spike	P	0 - 30
2265978	Microcystin LF	31.1	Spike	F	0 - 30
2265978	Microcystin LR	4.67	Spike	P	0 - 30
2265978	Microcystin LW	14.9	Spike	P	0 - 30
2265978	Microcystin LY	4.13	Spike	P	0 - 30
2265978	Microcystin RR	6.63	Spike	P	0 - 30
2265978	Microcystin WR	1.69	Spike	P	0 - 30
2265978	Microcystin YR	11.4	Spike	P	0 - 30
2265978	Nodularin-R	11.5	Spike	P	0 - 30

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

Quality Assurance Report Surrogates

Lab Sample ID: 2266194
Field Sample ID: G3NW0449

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	119	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	127	P	30 - 160

Lab Sample ID: 2266195
Field Sample ID: G3NW0216

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	112	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	111	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B

Run ID: A107011

Included Lab Sample IDs: 2266194, 2266195

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	108	107	P/P	50 - 160
Cylindrospermopsin	122	122	P/P	60 - 160
Desmethyl microcystin LR	93.8	94.9	P/P	60 - 160
Microcystin HiiR	100	99.9	P/P	60 - 160
Microcystin HtyR	101	102	P/P	60 - 160
Microcystin LA	86.5	98.7	P/P	50 - 160
Microcystin LF	77.1	71.2	P/P	60 - 160
Microcystin LR	84.0	84.8	P/P	60 - 160
Microcystin LW	89.9	93.8	P/P	60 - 160
Microcystin LY	89.8	83.9	P/P	60 - 160
Microcystin RR	103	102	P/P	60 - 160
Microcystin WR	96.5	91.4	P/P	60 - 160
Microcystin YR	97.0	98.0	P/P	60 - 160
Nodularin-R	108	105	P/P	60 - 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 SMP	MS
			LCS			
EPA 8321B	Anatoxin-a	98.8	110	112		2.29
	Cylindrospermopsin	126	126	129		2.25
	Desmethyl microcystin LR	91.7	98.6	108		8.99
	Microcystin HILR	102	107	111		3.62
	Microcystin HtyR	104	100	107		6.84
	Microcystin LA	85.4	95.8	91.8		4.21
	Microcystin LF	79.7	69.6	95.2		31.1
	Microcystin LR	89.0	88.9	93.3		4.67
	Microcystin LW	97.8	77.5	90.0		14.9
	Microcystin LY	84.1	93.0	96.9		4.13
	Microcystin RR	103	105	112		6.63
	Microcystin WR	96.4	102	101		1.69
	Microcystin YR	88.7	101	113		11.4
	Nodularin-R	108	108	121		11.5

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2266194, 2266195
SOP 67 (Greenwater)	Algal Toxins by LC/MS/MS using Greenwater Laboratory	2266193

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	07/30/2021	08/03/2021 07:00	Pramila Ghimire	08/03/2021 16:40	Pramila Ghimire	2266194
	07/30/2021	08/03/2021 07:00	Pramila Ghimire	08/03/2021 16:57	Pramila Ghimire	2266195
SOP 67 (Greenwater)	07/30/2021	08/09/2021 11:29	GreenWater Labs	08/09/2021 14:41	GreenWater Labs	2266193