

Chemical Analysis Report

WQAP-2021-04-21-01

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-2021-04-19-47**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

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

For additional information please contact
Colin Wright, Ph.D.
Liang-Tsair Lin, Ph.D.
Kerry Tate, Ph.D.
Dr. rer. nat. Bettina Steinbock
Thekkekalathil Chandrasekhar, Ph.D, QA Officer
Phone (850) 245-8085

Certified by: Kerry Tate, Ph.D., Environmental Administrator

Date Certified: 11-MAY-2021 12:43

A handwritten signature in black ink that reads "Kerry Tate". The signature is written in a cursive, flowing style with a horizontal line above the first name.

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: Clear Lake - 4680 Clear Lake Dr.

Collection Date/Time: 04/20/2021 14:55

Field ID: NEHAB0018

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2241211	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P396904	
		Cylindrospermopsin	0.25	U	ug/L	P396904	
		Desmethyl microcystin LR	0.25	U	ug/L	P396904	
		Microcystin HIR**	0.25	U	ug/L	P396904	
		Microcystin HtyR**	0.25	U	ug/L	P396904	
		Microcystin LA	0.25	U	ug/L	P396904	
		Microcystin LF	0.25	U	ug/L	P396904	
		Microcystin LR	1.2		ug/L	P396904	
		Microcystin LW	0.25	U	ug/L	P396904	
		Microcystin LY	0.25	U	ug/L	P396904	
		Microcystin RR	0.25	U	ug/L	P396904	
		Microcystin WR	0.50	U	ug/L	P396904	
		Microcystin YR	0.25	U	ug/L	P396904	
		Nodularin-R**	0.25	U	ug/L	P396904	
2241212	EPA 350.1 Rev. 2.0	Ammonia-N	0.39		mg N/L	P397510	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	4.6		mg N/L	P397446	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.008	I	mg N/L	P397176	
	EPA 365.1 Rev. 2.0	Total-P	0.16		mg P/L	P397014	
2241213	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.017		mg P/L	P396925	

Ref. Method and Comment:

EPA 351.2 Rev. 2.0: Batch matrix spike recoveries are unavailable because of high analyte concentration in the QC sample.

Quality Assurance Report

Method Blank Results

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P397510

Component	Result	Code	Units
Ammonia-N	0.002	U	mg N/L

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P397446

Component	Result	Code	Units
Kjeldahl Nitrogen	0.080	U	mg N/L

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P397176

Component	Result	Code	Units
NO2NO3-N	0.004	U	mg N/L

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P397014

Component	Result	Code	Units
Total-P	0.002	U	mg P/L

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P396925

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

Reference Method: EPA 8321B
Batch ID: P396904

Component	Result	Code	Units
Anatoxin-a	0.25	U	ug/L
Cylindrospermopsin	0.25	U	ug/L
Desmethyl microcystin LR	0.25	U	ug/L
Microcystin H11R	0.25	U	ug/L
Microcystin HtyR	0.25	U	ug/L
Microcystin LA	0.25	U	ug/L
Microcystin LF	0.25	U	ug/L
Microcystin LR	0.25	U	ug/L
Microcystin LW	0.25	U	ug/L
Microcystin LY	0.25	U	ug/L
Microcystin RR	0.25	U	ug/L
Microcystin WR	0.50	U	ug/L
Microcystin YR	0.25	U	ug/L
Nodularin-R	0.25	U	ug/L

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P397510

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Ammonia-N	95.8		P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P397446

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Kjeldahl Nitrogen	103		P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P397176

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
NO2NO3-N	100		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P397014

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Total-P	107		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P396925

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
O-Phosphate-P	97.9		P	90 - 110

Reference Method: EPA 8321B
Batch ID: P396904

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	114		P	40 - 150
Cylindrospermopsin	103		P	40 - 150
Desmethyl microcystin LR	98.7		P	40 - 150
Microcystin HIR	116		P	40 - 150
Microcystin HtyR	100		P	40 - 150
Microcystin LA	126		P	40 - 150
Microcystin LF	124		P	40 - 150
Microcystin LR	116		P	40 - 150
Microcystin LW	121		P	40 - 150
Microcystin LY	136		P	40 - 150
Microcystin RR	98.8		P	40 - 150
Microcystin WR	110		P	40 - 150
Microcystin YR	124		P	40 - 150
Nodularin-R	112		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P397510

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2241209	Ammonia-N	96.8	98.4	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P397176

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2241212	NO2NO3-N	99.8	97.8	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P397014

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2241186	Total-P	107	109	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P396925

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2241300	O-Phosphate-P	97.0	96.4	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P396904

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2241211	Anatoxin-a	92.6	81.8	P/P	40 - 150
2241211	Cylindrospermopsin	101	91.7	P/P	40 - 150
2241211	Desmethyl microcystin LR	102	103	P/P	40 - 150
2241211	Microcystin H1LR	115	98.1	P/P	40 - 150
2241211	Microcystin HtyR	106	100	P/P	40 - 150
2241211	Microcystin LA	121	116	P/P	40 - 150
2241211	Microcystin LF	102	115	P/P	40 - 150
2241211	Microcystin LR	120	115	P/P	40 - 150
2241211	Microcystin LW	110	99.2	P/P	40 - 150
2241211	Microcystin LY	125	116	P/P	40 - 150
2241211	Microcystin RR	93.9	89.5	P/P	40 - 150
2241211	Microcystin WR	101	102	P/P	40 - 150
2241211	Microcystin YR	120	114	P/P	40 - 150
2241211	Nodularin-R	121	116	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0

Batch ID: P397510

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2241209	Ammonia-N	0.985	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0

Batch ID: P397446

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2241927	Kjeldahl Nitrogen	0.0382	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0

Batch ID: P397176

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2241212	NO2NO3-N	1.94	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0

Batch ID: P397014

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2241186	Total-P	1.59	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Batch ID: P396925

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2241300	O-Phosphate-P	0.477	Spike	P	0 - 20

Reference Method: EPA 8321B

Batch ID: P396904

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2241211	Anatoxin-a	12.3	Spike	P	0 - 30
2241211	Cylindrospermopsin	9.57	Spike	P	0 - 30
2241211	Desmethyl microcystin LR	0.477	Spike	P	0 - 30
2241211	Microcystin HtIR	15.9	Spike	P	0 - 30
2241211	Microcystin HtyR	5.73	Spike	P	0 - 30
2241211	Microcystin LA	4.09	Spike	P	0 - 30
2241211	Microcystin LF	12.0	Spike	P	0 - 30
2241211	Microcystin LR	3.95	Spike	P	0 - 30
2241211	Microcystin LW	10.0	Spike	P	0 - 30
2241211	Microcystin LY	7.49	Spike	P	0 - 30
2241211	Microcystin RR	4.77	Spike	P	0 - 30
2241211	Microcystin WR	1.73	Spike	P	0 - 30
2241211	Microcystin YR	5.31	Spike	P	0 - 30
2241211	Nodularin-R	4.00	Spike	P	0 - 30

Quality Assurance Report Precision

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

Quality Assurance Report Surrogates

Lab Sample ID: 2241211

Field Sample ID: NEHAB0018

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	140	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	130	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Run ID: A104886

Included Lab Sample IDs: 2241213

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

Reference Method: EPA 8321B

Run ID: A104912

Included Lab Sample IDs: 2241211

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	113	123	P/P	50 - 160
Cylindrospermopsin	109	114	P/P	50 - 160
Desmethyl microcystin LR	109	111	P/P	50 - 160
Microcystin HIR	126	119	P/P	50 - 160
Microcystin HtyR	118	118	P/P	50 - 160
Microcystin LA	134	139	P/P	50 - 160
Microcystin LF	132	126	P/P	50 - 160
Microcystin LR	126	130	P/P	50 - 160
Microcystin LW	116	119	P/P	50 - 160
Microcystin LY	114	134	P/P	50 - 160
Microcystin RR	100	104	P/P	50 - 160
Microcystin WR	107	106	P/P	50 - 160
Microcystin YR	134	127	P/P	50 - 160
Nodularin-R	120	127	P/P	50 - 160

Reference Method: EPA 365.1 Rev. 2.0

Run ID: A104942

Included Lab Sample IDs: 2241212

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	103	105	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0

Run ID: A104995

Included Lab Sample IDs: 2241212

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
NO2NO3-N	102	99.5	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0

Run ID: A105110

Included Lab Sample IDs: 2241212

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	100	101	P/P	90 - 110

Reference Method: EPA 350.1 Rev. 2.0

Run ID: A105117

Included Lab Sample IDs: 2241212

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	98.5	97.3	P/P	90 - 110

Quality Assurance Report

Calibration Verification

* 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		LCS	Precision	MS
						SMP	
EPA 350.1 Rev. 2.0	Ammonia-N	95.8	96.8	98.4			0.985
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	103					0.0382
EPA 353.2 Rev. 2.0	NO ₂ NO ₃ -N	100	99.8	97.8			1.94
EPA 365.1 Rev. 2.0	Total-P	107	107	109			1.59
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	97.9	97.0	96.4			0.477
EPA 8321B	Anatoxin-a	114	92.6	81.8			12.3
	Cylindrospermopsin	103	101	91.7			9.57
	Desmethyl microcystin LR	98.7	102	103			0.477
	Microcystin HiLR	116	115	98.1			15.9
	Microcystin HtyR	100	106	100			5.73
	Microcystin LA	126	121	116			4.09
	Microcystin LF	124	102	115			12.0
	Microcystin LR	116	120	115			3.95
	Microcystin LW	121	110	99.2			10.0
	Microcystin LY	136	125	116			7.49
	Microcystin RR	98.8	93.9	89.5			4.77
	Microcystin WR	110	101	102			1.73
	Microcystin YR	124	120	114			5.31
	Nodularin-R	112	121	116			4.00

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 350.1 Rev. 2.0	Ammonia in aqueous matrices as mg N/L	2241212
EPA 351.2 Rev. 2.0	Total Kjeldahl Nitrogen in aqueous matrices	2241212
EPA 353.2 Rev. 2.0	Nitrite/Nitrate in aqueous matrices as mg N/L	2241212
EPA 365.1 Rev. 2.0	Total Phosphorus in aqueous matrices as mg P/L	2241212
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2241213
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2241211

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 350.1 Rev. 2.0	04/21/2021			05/01/2021 13:23	Ping Hua	2241212
EPA 351.2 Rev. 2.0	04/21/2021	04/29/2021 13:24	Benjamin T. Nordmann	04/30/2021 11:59	Virginia P. Brown	2241212
EPA 353.2 Rev. 2.0	04/21/2021			04/26/2021 16:11	Touraj Touran	2241212
EPA 365.1 Rev. 2.0	04/21/2021	04/22/2021 16:03	Yen Ta	04/23/2021 14:07	Shengyu Ding	2241212
EPA 365.1 Rev. 2.0 dissolved	04/21/2021			04/21/2021 14:30	Blanca Fach	2241213
EPA 8321B	04/21/2021	04/22/2021 09:00	Pramila Ghimire	04/22/2021 11:48	Pramila Ghimire	2241211