

Chemical Analysis Report

WQAP-2021-08-04-02

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-2021-07-26-26**
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
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Phone (850) 245-8085

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

Date Certified: 27-AUG-2021 13:12

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.

NON-CONFORMANCE REPORT INCLUDED

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: Clapboard Creek at Boat Ramp

Collection Date/Time: 08/02/2021 12:20

Field ID: NEHAB0023

Matrix: W-SURF-SLT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2267029	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P401920	
		Cylindrospermopsin	0.10	U	ug/L	P401920	
		Desmethyl microcystin LR	0.25	U	ug/L	P401920	
		Microcystin HilR**	0.25	U	ug/L	P401920	
		Microcystin HtyR**	0.25	U	ug/L	P401920	
		Microcystin LA	0.10	U	ug/L	P401920	
		Microcystin LF	0.10	U	ug/L	P401920	RPD
		Microcystin LR	0.25	U	ug/L	P401920	
		Microcystin LW	0.25	U	ug/L	P401920	RPD
		Microcystin LY	0.10	U	ug/L	P401920	
		Microcystin RR	0.10	U	ug/L	P401920	
		Microcystin WR	0.50	U	ug/L	P401920	
		Microcystin YR	0.25	U	ug/L	P401920	
		Nodularin-R**	0.10	U	ug/L	P401920	
2267030	EPA 350.1 Rev. 2.0	Ammonia-N	0.012		mg N/L	P402259	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	0.62		mg N/L	P402688	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.14		mg N/L	P402162	
	EPA 365.1 Rev. 2.0	Total-P	0.088		mg P/L	P402236	
2267031	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.052		mg P/L	P401958	

Non-Conformance Report

NCR ID: 8967

Event(s)

WQAP-2021-08-04-02

Job(s)

Sample(s)

Test(s)

NCR Type: SHIPPING/RECEIVING

NCR Category: Samples arrived late

Observation: Samples arrived late, samples received within the acceptable temperature range.

Resolution: Samples will be qualified as appropriate.

Authorized by/Date: Kerry Tate, Ph.D. 8/5/2021

The Non-Conformance Report details exceptions or problems encountered with the events/jobs/samples/test.
Please address questions to:

Chemistry	Colin Wright	(850) 245-8085
Biology	Cheryl Swanson	(850) 245-8177

Quality Assurance Report Method Blank Results

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P402259

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

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P402688

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P402162

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P402236

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

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P401958

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

Reference Method: EPA 8321B
Batch ID: P401920

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 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

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P402259

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Ammonia-N	98.8		P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P402688

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Kjeldahl Nitrogen	105		P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P402162

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: P402236

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: P401958

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
O-Phosphate-P	98.2		P	90 - 110

Reference Method: EPA 8321B
Batch ID: P401920

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	104		P	40 - 150
Cylindrospermopsin	115		P	40 - 150
Desmethyl microcystin LR	89.0		P	40 - 150
Microcystin HIR	97.5		P	40 - 150
Microcystin HtyR	96.7		P	40 - 150
Microcystin LA	91.5		P	40 - 150
Microcystin LF	93.7		P	40 - 150
Microcystin LR	83.6		P	40 - 150
Microcystin LW	88.3		P	40 - 150
Microcystin LY	89.9		P	40 - 150
Microcystin RR	104		P	40 - 150
Microcystin WR	96.0		P	40 - 150
Microcystin YR	102		P	40 - 150
Nodularin-R	109		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P402259

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2266968	Ammonia-N	101	103	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P402688

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2267639	Kjeldahl Nitrogen	104	106	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P402162

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2266971	NO2NO3-N	102	101	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P402236

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2267659	Total-P	106	106	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P401958

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2267031	O-Phosphate-P	99.3	99.0	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P401920

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2266865	Anatoxin-a	107	97.3	P/P	40 - 150
2266865	Cylindrospermopsin	128	117	P/P	40 - 150
2266865	Desmethyl microcystin LR	94.8	83.6	P/P	40 - 150
2266865	Microcystin HiLR	105	95.7	P/P	40 - 150
2266865	Microcystin HtyR	104	87.8	P/P	40 - 150
2266865	Microcystin LA	107	90.6	P/P	40 - 150
2266865	Microcystin LF	93.1	65.2	P/P	40 - 150
2266865	Microcystin LR	95.8	89.1	P/P	40 - 150
2266865	Microcystin LW	104	70.8	P/P	40 - 150
2266865	Microcystin LY	91.7	89.6	P/P	40 - 150
2266865	Microcystin RR	106	95.5	P/P	40 - 150
2266865	Microcystin WR	98.5	88.3	P/P	40 - 150
2266865	Microcystin YR	112	98.4	P/P	40 - 150
2266865	Nodularin-R	114	99.7	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0

Batch ID: P402259

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2266968	Ammonia-N	1.69	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0

Batch ID: P402688

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2267639	Kjeldahl Nitrogen	1.91	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0

Batch ID: P402162

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2266971	NO2NO3-N	0.494	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0

Batch ID: P402236

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2267659	Total-P	0.161	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Batch ID: P401958

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2267031	O-Phosphate-P	0.198	Spike	P	0 - 20

Reference Method: EPA 8321B

Batch ID: P401920

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2266865	Anatoxin-a	9.30	Spike	P	0 - 30
2266865	Cylindrospermopsin	9.49	Spike	P	0 - 30
2266865	Desmethyl microcystin LR	12.6	Spike	P	0 - 30
2266865	Microcystin HtIR	8.89	Spike	P	0 - 30
2266865	Microcystin HtyR	17.0	Spike	P	0 - 30
2266865	Microcystin LA	17.0	Spike	P	0 - 30
2266865	Microcystin LF	35.2	Spike	F	0 - 30
2266865	Microcystin LR	7.24	Spike	P	0 - 30
2266865	Microcystin LW	37.6	Spike	F	0 - 30
2266865	Microcystin LY	2.27	Spike	P	0 - 30
2266865	Microcystin RR	10.8	Spike	P	0 - 30
2266865	Microcystin WR	10.9	Spike	P	0 - 30
2266865	Microcystin YR	13.2	Spike	P	0 - 30
2266865	Nodularin-R	13.8	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: 2267029

Field Sample ID: NEHAB0023

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	90.6	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	73.9	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B
Run ID: A107011
Included Lab Sample IDs: 2267029

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	119	119	P/P	50 - 160
Cylindrospermopsin	139	135	P/P	60 - 160
Desmethyl microcystin LR	97.6	102	P/P	60 - 160
Microcystin HiLR	111	111	P/P	60 - 160
Microcystin HtyR	106	113	P/P	60 - 160
Microcystin LA	115	101	P/P	50 - 160
Microcystin LF	94.4	95.3	P/P	60 - 160
Microcystin LR	93.4	97.5	P/P	60 - 160
Microcystin LW	103	101	P/P	60 - 160
Microcystin LY	98.9	112	P/P	60 - 160
Microcystin RR	114	113	P/P	60 - 160
Microcystin WR	103	109	P/P	60 - 160
Microcystin YR	108	110	P/P	60 - 160
Nodularin-R	121	121	P/P	60 - 160

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Run ID: A107035
Included Lab Sample IDs: 2267031

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	99.4	99.7	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Run ID: A107114
Included Lab Sample IDs: 2267030

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
NO2NO3-N	100	101	P/P	90 - 110

Reference Method: EPA 350.1 Rev. 2.0
Run ID: A107153
Included Lab Sample IDs: 2267030

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	99.9	98.4	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Run ID: A107180
Included Lab Sample IDs: 2267030

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	99.5	100	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Run ID: A107363
Included Lab Sample IDs: 2267030

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	105	103	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		Precision LCS	SMP	MS
EPA 350.1 Rev. 2.0	Ammonia-N	98.8	101	103			1.69
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	105	104	106			1.91
EPA 353.2 Rev. 2.0	NO ₂ NO ₃ -N	100	102	101			0.494
EPA 365.1 Rev. 2.0	Total-P	107	106	106			0.161
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	98.2	99.3	99.0			0.198
EPA 8321B	Anatoxin-a	104	107	97.3			9.30
	Cylindrospermopsin	115	128	117			9.49
	Desmethyl microcystin LR	89.0	94.8	83.6			12.6
	Microcystin HiLR	97.5	105	95.7			8.89
	Microcystin HtyR	96.7	104	87.8			17.0
	Microcystin LA	91.5	107	90.6			17.0
	Microcystin LF	93.7	93.1	65.2			35.2
	Microcystin LR	83.6	95.8	89.1			7.24
	Microcystin LW	88.3	104	70.8			37.6
	Microcystin LY	89.9	91.7	89.6			2.27
	Microcystin RR	104	106	95.5			10.8
	Microcystin WR	96.0	98.5	88.3			10.9
	Microcystin YR	102	112	98.4			13.2
	Nodularin-R	109	114	99.7			13.8

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 350.1 Rev. 2.0	Ammonia in aqueous matrices as mg N/L	2267030
EPA 351.2 Rev. 2.0	Total Kjeldahl Nitrogen in aqueous matrices	2267030
EPA 353.2 Rev. 2.0	Nitrite/Nitrate in aqueous matrices as mg N/L	2267030
EPA 365.1 Rev. 2.0	Total Phosphorus in aqueous matrices as mg P/L	2267030
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2267031
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2267029

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	08/04/2021			08/10/2021 14:08	Roberta Tatti	2267030
EPA 351.2 Rev. 2.0	08/04/2021	08/19/2021 13:51	Benjamin T. Nordmann	08/20/2021 11:55	Virginia P. Brown	2267030
EPA 353.2 Rev. 2.0	08/04/2021			08/09/2021 09:31	Beverly Y. Sanders	2267030
EPA 365.1 Rev. 2.0	08/04/2021	08/10/2021 13:06	Simonne.V. Calhoun	08/11/2021 11:41	Shengyu Ding	2267030
EPA 365.1 Rev. 2.0 dissolved	08/04/2021			08/04/2021 11:43	Patrick M. Roche	2267031
EPA 8321B	08/04/2021	08/04/2021 12:30	Pramila Ghimire	08/04/2021 17:06	Pramila Ghimire	2267029