

Chemical Analysis Report

WQAP-2021-09-17-01

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

Event Description: **Algal Bloom Response - CEROC**
Request ID: **RQ-2021-09-06-61**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
3319 Maguire Blvd. Suite 232
Orlando, FL 32803
Attn: Matt Bearden

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: Liang Lin, Environmental Administrator

Date Certified: 11-OCT-2021 13:57

A handwritten signature in black ink, appearing to read "Liang Lin", on a light-colored background.

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: Lake Haines- Four Lakes Dock

Collection Date/Time: 09/15/2021 11:40

Field ID: HABCE0078_LakeHaines

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2276340	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P403871	
		Cylindrospermopsin	0.10	U	ug/L	P403871	
		Desmethyl microcystin LR	0.25	U	ug/L	P403871	
		Microcystin HIR**	0.25	U	ug/L	P403871	
		Microcystin HtyR**	0.25	U	ug/L	P403871	
		Microcystin LA	0.10	U	ug/L	P403871	
		Microcystin LF	0.10	U	ug/L	P403871	
		Microcystin LR	0.31	I	ug/L	P403871	
		Microcystin LW	0.25	U	ug/L	P403871	
		Microcystin LY	0.10	U	ug/L	P403871	
		Microcystin RR	0.10	U	ug/L	P403871	
		Microcystin WR	0.50	U	ug/L	P403871	
		Microcystin YR	0.25	U	ug/L	P403871	
		Nodularin-R**	0.10	U	ug/L	P403871	
2276341	EPA 350.1 Rev. 2.0	Ammonia-N	0.007		mg N/L	P403992	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	1.3		mg N/L	P403935	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.004	U	mg N/L	P404038	
	EPA 365.1 Rev. 2.0	Total-P	0.061		mg P/L	P404100	
2276342	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.004	UQ	mg P/L	P403877	

Ref. Method and Comment:

EPA 365.1 Rev. 2.0 dissolved: O-Phosphate-P: The sample expired upon receipt.

Non-Conformance Report

NCR ID: 9036

Event(s)

WQAP-2021-09-17-01

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: Joshua Ayres 9/22/2021

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: P403992

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

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P403935

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P404038

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P404100

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

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P403877

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

Reference Method: EPA 8321B
Batch ID: P403871

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: P403992

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: P403935

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Kjeldahl Nitrogen	92.8		P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P404038

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
NO2NO3-N	103		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P404100

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Total-P	104		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P403877

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

Reference Method: EPA 8321B
Batch ID: P403871

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	73.3		P	40 - 150
Cylindrospermopsin	84.2		P	40 - 150
Desmethyl microcystin LR	70.4		P	40 - 150
Microcystin HIR	69.0		P	40 - 150
Microcystin HtyR	72.4		P	40 - 150
Microcystin LA	68.3		P	40 - 150
Microcystin LF	79.1		P	40 - 150
Microcystin LR	62.5		P	40 - 150
Microcystin LW	80.5		P	40 - 150
Microcystin LY	73.3		P	40 - 150
Microcystin RR	72.7		P	40 - 150
Microcystin WR	74.1		P	40 - 150
Microcystin YR	84.0		P	40 - 150
Nodularin-R	76.8		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P403992

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2276348	Ammonia-N	97.0	98.2	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P403935

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2276504	Kjeldahl Nitrogen	103	109	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P404038

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2276341	NO2NO3-N	103	102	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P404100

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2276403	Total-P	107	107	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P403877

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2276365	O-Phosphate-P	102	100	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P403871

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2276340	Anatoxin-a	95.3	91.0	P/P	40 - 150
2276340	Cylindrospermopsin	93.1	88.8	P/P	40 - 150
2276340	Desmethyl microcystin LR	86.8	94.4	P/P	40 - 150
2276340	Microcystin HiLR	87.1	85.2	P/P	40 - 150
2276340	Microcystin HtyR	88.5	95.6	P/P	40 - 150
2276340	Microcystin LA	80.2	82.4	P/P	40 - 150
2276340	Microcystin LF	94.5	93.7	P/P	40 - 150
2276340	Microcystin LR	87.5	79.2	P/P	40 - 150
2276340	Microcystin LW	83.4	83.0	P/P	40 - 150
2276340	Microcystin LY	80.4	87.1	P/P	40 - 150
2276340	Microcystin RR	81.9	82.5	P/P	40 - 150
2276340	Microcystin WR	82.5	81.9	P/P	40 - 150
2276340	Microcystin YR	101	95.6	P/P	40 - 150
2276340	Nodularin-R	90.2	89.6	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0

Batch ID: P403992

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2276348	Ammonia-N	1.13	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0

Batch ID: P403935

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2276504	Kjeldahl Nitrogen	3.66	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0

Batch ID: P404038

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2276341	NO2NO3-N	0.487	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0

Batch ID: P404100

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2276403	Total-P	0.416	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Batch ID: P403877

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2276365	O-Phosphate-P	0.833	Spike	P	0 - 20

Reference Method: EPA 8321B

Batch ID: P403871

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2276340	Anatoxin-a	4.57	Spike	P	0 - 30
2276340	Cylindrospermopsin	4.74	Spike	P	0 - 30
2276340	Desmethyl microcystin LR	8.40	Spike	P	0 - 30
2276340	Microcystin H1LR	2.20	Spike	P	0 - 30
2276340	Microcystin HtyR	7.69	Spike	P	0 - 30
2276340	Microcystin LA	2.67	Spike	P	0 - 30
2276340	Microcystin LF	0.861	Spike	P	0 - 30
2276340	Microcystin LR	9.65	Spike	P	0 - 30
2276340	Microcystin LW	0.553	Spike	P	0 - 30
2276340	Microcystin LY	8.05	Spike	P	0 - 30
2276340	Microcystin RR	0.671	Spike	P	0 - 30
2276340	Microcystin WR	0.827	Spike	P	0 - 30
2276340	Microcystin YR	5.21	Spike	P	0 - 30
2276340	Nodularin-R	0.711	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: 2276340

Field Sample ID: HABCE0078_LakeHaines

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	115	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	117	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Run ID: A107855

Included Lab Sample IDs: 2276342

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	99.2	98.8	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0

Run ID: A107906

Included Lab Sample IDs: 2276341

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	95.9	99.0	P/P	90 - 110

Reference Method: EPA 350.1 Rev. 2.0

Run ID: A107911

Included Lab Sample IDs: 2276341

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	99.8	98.5	P/P	90 - 110

Reference Method: EPA 8321B

Run ID: A107917

Included Lab Sample IDs: 2276340

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	105	114	P/P	50 - 160
Cylindrospermopsin	120	117	P/P	60 - 160
Desmethyl microcystin LR	103	102	P/P	60 - 160
Microcystin H1R	90.1	96.3	P/P	60 - 160
Microcystin HtyR	115	113	P/P	60 - 160
Microcystin LA	94.8	85.8	P/P	50 - 160
Microcystin LF	96.2	108	P/P	60 - 160
Microcystin LR	111	101	P/P	60 - 160
Microcystin LW	92.8	82.4	P/P	60 - 160
Microcystin LY	104	93.5	P/P	60 - 160
Microcystin RR	96.4	90.5	P/P	60 - 160
Microcystin WR	93.5	93.9	P/P	60 - 160
Microcystin YR	118	106	P/P	60 - 160
Nodularin-R	99.8	84.8	P/P	60 - 160

Reference Method: EPA 353.2 Rev. 2.0

Run ID: A107927

Included Lab Sample IDs: 2276341

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
NO2NO3-N	102	104	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0

Run ID: A107988

Included Lab Sample IDs: 2276341

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	100	100	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	98.8	97.0	98.2			1.13
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	92.8	103	109			3.66
EPA 353.2 Rev. 2.0	NO ₂ NO ₃ -N	103	103	102			0.487
EPA 365.1 Rev. 2.0	Total-P	104	107	107			0.416
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	98.3	102	100			0.833
EPA 8321B	Anatoxin-a	73.3	95.3	91.0			4.57
	Cylindrospermopsin	84.2	93.1	88.8			4.74
	Desmethyl microcystin LR	70.4	86.8	94.4			8.40
	Microcystin HiLR	69.0	87.1	85.2			2.20
	Microcystin HtyR	72.4	88.5	95.6			7.69
	Microcystin LA	68.3	80.2	82.4			2.67
	Microcystin LF	79.1	94.5	93.7			0.861
	Microcystin LR	62.5	87.5	79.2			9.65
	Microcystin LW	80.5	83.4	83.0			0.553
	Microcystin LY	73.3	80.4	87.1			8.05
	Microcystin RR	72.7	81.9	82.5			0.671
	Microcystin WR	74.1	82.5	81.9			0.827
	Microcystin YR	84.0	101	95.6			5.21
	Nodularin-R	76.8	90.2	89.6			0.711

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 350.1 Rev. 2.0	Ammonia in aqueous matrices as mg N/L	2276341
EPA 351.2 Rev. 2.0	Total Kjeldahl Nitrogen in aqueous matrices	2276341
EPA 353.2 Rev. 2.0	Nitrite/Nitrate in aqueous matrices as mg N/L	2276341
EPA 365.1 Rev. 2.0	Total Phosphorus in aqueous matrices as mg P/L	2276341
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2276342
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2276340

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	09/17/2021			09/21/2021 13:02	Ping Hua	2276341
EPA 351.2 Rev. 2.0	09/17/2021	09/20/2021 17:00	Benjamin T. Nordmann	09/21/2021 12:56	Alexandra J Mattheus	2276341
EPA 353.2 Rev. 2.0	09/17/2021			09/22/2021 08:55	Beverly Y. Sanders	2276341
EPA 365.1 Rev. 2.0	09/17/2021	09/23/2021 12:05	Simonne.V. Calhoun	09/24/2021 12:00	Sarah A Nixon	2276341
EPA 365.1 Rev. 2.0 dissolved	09/17/2021			09/17/2021 12:17	James L Waggerby	2276342
EPA 8321B	09/17/2021	09/21/2021 09:00	Manjita Shrestha	09/21/2021 11:55	Manjita Shrestha	2276340