

Chemical Analysis Report

WQAP-2020-06-24-01

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

Event Description: **Algal Bloom Response - SE ROC**

Request ID: **RQ-2020-03-23-45**

Customer: **WQAP**

Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
2199 South Rock Road
Fort Pierce, FL 34945
Attn: Jordan Skaggs

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

Certified by: Thekkekalathil Chandrasekhar, Chemist Administrator

Date Certified: 08-JUL-2020 16:04



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 Okeechobee

Collection Date/Time: 06/22/2020 11:08

Field ID: LakeO_West

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2178143	EPA 8321B	Anatoxin-a**	0.25	U	ug/L	P383615	
		Cylindrospermopsin**	0.25	U	ug/L	P383615	
		Desmethyl microcystin LR**	0.25	U	ug/L	P383615	
		Microcystin LA**	0.25	U	ug/L	P383615	
		Microcystin LF**	0.25	U	ug/L	P383615	
		Microcystin LR**	0.25	U	ug/L	P383615	
		Microcystin LW**	0.25	U	ug/L	P383615	
		Microcystin LY**	0.25	U	ug/L	P383615	
		Microcystin RR**	0.25	U	ug/L	P383615	
		Microcystin WR**	0.50	U	ug/L	P383615	
		Microcystin YR**	0.25	U	ug/L	P383615	
2178144	EPA 350.1 Rev. 2.0	Ammonia-N	0.01		mg N/L	P383764	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	0.94		mg N/L	P383653	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.004	U	mg N/L	P383641	
	EPA 365.1 Rev. 2.0	Total-P	0.060		mg P/L	P383668	
2178145	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.033	Q	mg P/L	P383633	

Ref. Method and Comment:

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

Non-Conformance Report

NCR ID: 8317

Event(s)

WQAP-2020-06-24-01

Job(s)

Sample(s)

Test(s)

NCR Type: SHIPPING/RECEIVING

NCR Category: Samples arrived late

Observation: Samples arrived one or more days late by common carrier or hand delivery.

Resolution: Samples will be qualified as appropriate.

Authorized by/Date: Kerry Tate, Ph.D. 6/25/2020

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: P383764

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

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P383653

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P383641

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P383668

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

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P383633

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

Reference Method: EPA 8321B
Batch ID: P383615

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 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

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: EPA 350.1 Rev. 2.0
 Batch ID: P383764

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

Reference Method: EPA 351.2 Rev. 2.0
 Batch ID: P383653

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: P383641

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: P383668

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Total-P	97.3		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
 Batch ID: P383633

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
O-Phosphate-P	97.7		P	90 - 110

Reference Method: EPA 8321B
 Batch ID: P383615

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	90.3		P	40 - 140
Cylindrospermopsin	109		P	40 - 140
Desmethyl microcystin LR	109		P	40 - 140
Microcystin LA	105		P	40 - 140
Microcystin LF	123		P	40 - 140
Microcystin LR	106		P	40 - 140
Microcystin LW	116		P	40 - 140
Microcystin LY	116		P	40 - 140
Microcystin RR	115		P	40 - 140
Microcystin WR	108		P	40 - 140
Microcystin YR	110		P	40 - 140

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P383764

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2178030	Ammonia-N	99.7	99.1	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P383653

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2178019	Kjeldahl Nitrogen	105	106	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P383641

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2178029	NO2NO3-N	100	100	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P383668

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2178029	Total-P	99.6	100	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P383633

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2178199	O-Phosphate-P	98.9	98.6	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P383615

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2178135	Anatoxin-a	78.7	79.2	P/P	40 - 140
2178135	Cylindrospermopsin	91.2	90.0	P/P	40 - 140
2178135	Desmethyl microcystin LR	106	106	P/P	40 - 140
2178135	Microcystin LA	104	108	P/P	40 - 140
2178135	Microcystin LF	109	108	P/P	40 - 140
2178135	Microcystin LR	93.3	97.3	P/P	40 - 140
2178135	Microcystin LW	102	106	P/P	40 - 140
2178135	Microcystin LY	104	112	P/P	40 - 140
2178135	Microcystin RR	110	116	P/P	40 - 140
2178135	Microcystin WR	105	105	P/P	40 - 140
2178135	Microcystin YR	101	104	P/P	40 - 140

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0
 Batch ID: P383764

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2178030	Ammonia-N	0.554	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0
 Batch ID: P383653

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2178019	Kjeldahl Nitrogen	1.19	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0
 Batch ID: P383641

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2178029	NO2NO3-N	0.0	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0
 Batch ID: P383668

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2178029	Total-P	0.888	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved
 Batch ID: P383633

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2178199	O-Phosphate-P	0.304	Spike	P	0 - 20

Reference Method: EPA 8321B
 Batch ID: P383615

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2178135	Anatoxin-a	0.585	Spike	P	0 - 30
2178135	Cylindrospermopsin	1.30	Spike	P	0 - 30
2178135	Desmethyl microcystin LR	0.316	Spike	P	0 - 30
2178135	Microcystin LA	4.42	Spike	P	0 - 30
2178135	Microcystin LF	0.112	Spike	P	0 - 30
2178135	Microcystin LR	4.02	Spike	P	0 - 30
2178135	Microcystin LW	3.77	Spike	P	0 - 30
2178135	Microcystin LY	7.15	Spike	P	0 - 30
2178135	Microcystin RR	5.54	Spike	P	0 - 30
2178135	Microcystin WR	0.239	Spike	P	0 - 30
2178135	Microcystin YR	2.39	Spike	P	0 - 30

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

Quality Assurance Report Surrogates

Lab Sample ID: 2178143
Field Sample ID: LakeO_West

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	96.3	P	40 - 150

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B
Run ID: A99498
Included Lab Sample IDs: 2178143

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	90.8	93.9	P/P	50 - 160
Cylindrospermopsin	104	111	P/P	60 - 160
Desmethyl microcystin LR	110	111	P/P	60 - 160
Microcystin LA	114	108	P/P	50 - 160
Microcystin LF	109	112	P/P	60 - 160
Microcystin LR	106	106	P/P	60 - 160
Microcystin LW	107	113	P/P	60 - 160
Microcystin LY	112	118	P/P	60 - 160
Microcystin RR	107	115	P/P	60 - 160
Microcystin WR	109	105	P/P	60 - 160
Microcystin YR	101	98.5	P/P	60 - 160

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Run ID: A99500
Included Lab Sample IDs: 2178145

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	99.2	100	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Run ID: A99508
Included Lab Sample IDs: 2178144

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

Reference Method: EPA 351.2 Rev. 2.0
Run ID: A99554
Included Lab Sample IDs: 2178144

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	94.6	98.1	P/P	90 - 110

Reference Method: EPA 350.1 Rev. 2.0
Run ID: A99556
Included Lab Sample IDs: 2178144

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

Reference Method: EPA 365.1 Rev. 2.0
Run ID: A99563
Included Lab Sample IDs: 2178144

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

* 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	101	99.7	99.1			0.554
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	103	105	106			1.19
EPA 353.2 Rev. 2.0	NO2NO3-N	100	100	100			0.0
EPA 365.1 Rev. 2.0	Total-P	97.3	99.6	100			0.888
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	97.7	98.9	98.6			0.304
EPA 8321B	Anatoxin-a	90.3	78.7	79.2			0.585
	Cylindrospermopsin	109	91.2	90.0			1.30
	Desmethyl microcystin LR	109	106	106			0.316
	Microcystin LA	105	104	108			4.42
	Microcystin LF	123	109	108			0.112
	Microcystin LR	106	93.3	97.3			4.02
	Microcystin LW	116	102	106			3.77
	Microcystin LY	116	104	112			7.15
	Microcystin RR	115	110	116			5.54
	Microcystin WR	108	105	105			0.239
	Microcystin YR	110	101	104			2.39

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 350.1 Rev. 2.0	Ammonia in aqueous matrices as mg N/L	2178144
EPA 351.2 Rev. 2.0	Total Kjeldahl Nitrogen in aqueous matrices	2178144
EPA 353.2 Rev. 2.0	Nitrite/Nitrate in aqueous matrices as mg N/L	2178144
EPA 365.1 Rev. 2.0	Total Phosphorus in aqueous matrices as mg P/L	2178144
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2178145
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2178143

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	06/24/2020			06/27/2020 14:48	Ping Hua	2178144
EPA 351.2 Rev. 2.0	06/24/2020	06/25/2020 10:55	Sima Feizi	06/26/2020 16:07	Jhamieka S. Greenwood	2178144
EPA 353.2 Rev. 2.0	06/24/2020			06/25/2020 08:48	Beverly Y. Sanders	2178144
EPA 365.1 Rev. 2.0	06/24/2020	06/25/2020 11:41	Yen Ta	06/29/2020 08:15	Benjamin T. Nordmann	2178144
EPA 365.1 Rev. 2.0 dissolved	06/24/2020			06/24/2020 16:36	Julia Storbeck	2178145
EPA 8321B	06/24/2020	06/24/2020 15:00	Juyoung Kim	06/24/2020 23:14	Juyoung Kim	2178143