

Chemical Analysis Report

WQAP-2021-06-08-01

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

Event Description: **Algal Bloom Response - SWD**
Request ID: **RQ-2021-05-24-38**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
13051 N. Telecom Parkway
Temple Terrace, FL 33637
Attn: Matthew Bearden

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Certified by: Liang Lin, Environmental Administrator

Date Certified: 24-JUN-2021 14:44

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

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: Withlacoochee River @ Nobleton

Collection Date/Time: 06/07/2021 13:55

Field ID: 670034

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2252711	EPA 8321B	Anatoxin-a	0.25	U	ug/L	P399346	
		Cylindrospermopsin	0.10	U	ug/L	P399346	
		Desmethyl microcystin LR	0.25	U	ug/L	P399346	
		Microcystin HilR**	0.25	U	ug/L	P399346	
		Microcystin HtyR**	0.25	U	ug/L	P399346	
		Microcystin LA	0.10	U	ug/L	P399346	
		Microcystin LF	0.10	U	ug/L	P399346	
		Microcystin LR	0.25	U	ug/L	P399346	
		Microcystin LW	0.25	U	ug/L	P399346	
		Microcystin LY	0.10	U	ug/L	P399346	
		Microcystin RR	0.10	U	ug/L	P399346	
		Microcystin WR	0.50	U	ug/L	P399346	
		Microcystin YR	0.25	U	ug/L	P399346	
		Nodularin-R**	0.10	U	ug/L	P399346	
2252712	EPA 350.1 Rev. 2.0	Ammonia-N	0.003	I	mg N/L	P399795	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	1.5		mg N/L	P399469	MS
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.011		mg N/L	P399555	
	EPA 365.1 Rev. 2.0	Total-P	0.088		mg P/L	P399591	
2252713	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.004	U	mg P/L	P399368	

Quality Assurance Report Method Blank Results

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P399795

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

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P399469

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P399555

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P399591

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

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P399368

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

Reference Method: EPA 8321B
Batch ID: P399346

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: P399795

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Ammonia-N	99.2		P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P399469

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P399555

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P399591

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: P399368

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

Reference Method: EPA 8321B
Batch ID: P399346

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	106		P	40 - 150
Cylindrospermopsin	117		P	40 - 150
Desmethyl microcystin LR	80.2		P	40 - 150
Microcystin HIR	98.5		P	40 - 150
Microcystin HtyR	96.3		P	40 - 150
Microcystin LA	103		P	40 - 150
Microcystin LF	98.4		P	40 - 150
Microcystin LR	92.5		P	40 - 150
Microcystin LW	97.9		P	40 - 150
Microcystin LY	110		P	40 - 150
Microcystin RR	109		P	40 - 150
Microcystin WR	89.3		P	40 - 150
Microcystin YR	101		P	40 - 150
Nodularin-R	106		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0

Batch ID: P399795

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2252618	Ammonia-N	96.0	98.0	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0

Batch ID: P399469

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2252617	Kjeldahl Nitrogen	113	112	F/F	90 - 110

Reference Method: EPA 353.2 Rev. 2.0

Batch ID: P399555

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2252682	NO2NO3-N	98.0		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0

Batch ID: P399591

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2252712	Total-P	102	104	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Batch ID: P399368

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2252713	O-Phosphate-P	97.4	99.2	P/P	90 - 110

Reference Method: EPA 8321B

Batch ID: P399346

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2252694	Anatoxin-a	108	113	P/P	40 - 150
2252694	Cylindrospermopsin	99.7	111	P/P	40 - 150
2252694	Desmethyl microcystin LR	89.5	92.6	P/P	40 - 150
2252694	Microcystin HiLR	98.0	101	P/P	40 - 150
2252694	Microcystin HtyR	91.7	98.0	P/P	40 - 150
2252694	Microcystin LA	97.5	107	P/P	40 - 150
2252694	Microcystin LF	90.2	104	P/P	40 - 150
2252694	Microcystin LR	88.5	94.3	P/P	40 - 150
2252694	Microcystin LW	91.5	94.2	P/P	40 - 150
2252694	Microcystin LY	98.0	112	P/P	40 - 150
2252694	Microcystin RR	106	116	P/P	40 - 150
2252694	Microcystin WR	88.9	99.6	P/P	40 - 150
2252694	Microcystin YR	106	108	P/P	40 - 150
2252694	Nodularin-R	109	111	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0

Batch ID: P399795

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2252618	Ammonia-N	2.06	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0

Batch ID: P399469

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2252617	Kjeldahl Nitrogen	0.327	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0

Batch ID: P399555

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2252682	NO2NO3-N	0.0	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0

Batch ID: P399591

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2252712	Total-P	1.50	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Batch ID: P399368

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2252713	O-Phosphate-P	1.83	Spike	P	0 - 20

Reference Method: EPA 8321B

Batch ID: P399346

Replicated

Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2252694	Anatoxin-a	4.51	Spike	P	0 - 30
2252694	Cylindrospermopsin	11.1	Spike	P	0 - 30
2252694	Desmethyl microcystin LR	3.39	Spike	P	0 - 30
2252694	Microcystin HtIR	3.03	Spike	P	0 - 30
2252694	Microcystin HtyR	6.60	Spike	P	0 - 30
2252694	Microcystin LA	8.85	Spike	P	0 - 30
2252694	Microcystin LF	14.5	Spike	P	0 - 30
2252694	Microcystin LR	6.32	Spike	P	0 - 30
2252694	Microcystin LW	2.94	Spike	P	0 - 30
2252694	Microcystin LY	13.3	Spike	P	0 - 30
2252694	Microcystin RR	8.93	Spike	P	0 - 30
2252694	Microcystin WR	11.3	Spike	P	0 - 30
2252694	Microcystin YR	1.91	Spike	P	0 - 30
2252694	Nodularin-R	1.70	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: 2252711
Field Sample ID: 670034

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	90.3	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	96.5	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Run ID: A105940

Included Lab Sample IDs: 2252713

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

Reference Method: EPA 8321B

Run ID: A105974

Included Lab Sample IDs: 2252711

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	81.1	80.0	P/P	50 - 160
Cylindrospermopsin	111	114	P/P	50 - 160
Desmethyl microcystin LR	92.4	99.7	P/P	50 - 160
Microcystin HIR	99.7	110	P/P	50 - 160
Microcystin HtyR	95.6	106	P/P	50 - 160
Microcystin LA	116	113	P/P	50 - 160
Microcystin LF	109	108	P/P	50 - 160
Microcystin LR	93.4	98.9	P/P	50 - 160
Microcystin LW	97.4	95.0	P/P	50 - 160
Microcystin LY	108	111	P/P	50 - 160
Microcystin RR	108	119	P/P	50 - 160
Microcystin WR	94.7	97.2	P/P	50 - 160
Microcystin YR	103	109	P/P	50 - 160
Nodularin-R	114	106	P/P	50 - 160

Reference Method: EPA 353.2 Rev. 2.0

Run ID: A106016

Included Lab Sample IDs: 2252712

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

Reference Method: EPA 351.2 Rev. 2.0

Run ID: A106020

Included Lab Sample IDs: 2252712

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	99.9	104	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0

Run ID: A106033

Included Lab Sample IDs: 2252712

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	106	105	P/P	90 - 110

Reference Method: EPA 350.1 Rev. 2.0

Run ID: A106115

Included Lab Sample IDs: 2252712

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	99.3	98.3	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		Precision LCS	SMP	MS
EPA 350.1 Rev. 2.0	Ammonia-N	99.2	96.0	98.0			2.06
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	107	113	112			0.327
EPA 353.2 Rev. 2.0	NO ₂ NO ₃ -N	102	98.0				0.0
EPA 365.1 Rev. 2.0	Total-P	104	102	104			1.50
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	100	97.4	99.2			1.83
EPA 8321B	Anatoxin-a	106	108	113			4.51
	Cylindrospermopsin	117	99.7	111			11.1
	Desmethyl microcystin LR	80.2	89.5	92.6			3.39
	Microcystin HiLR	98.5	98.0	101			3.03
	Microcystin HtyR	96.3	91.7	98.0			6.60
	Microcystin LA	103	97.5	107			8.85
	Microcystin LF	98.4	90.2	104			14.5
	Microcystin LR	92.5	88.5	94.3			6.32
	Microcystin LW	97.9	91.5	94.2			2.94
	Microcystin LY	110	98.0	112			13.3
	Microcystin RR	109	106	116			8.93
	Microcystin WR	89.3	88.9	99.6			11.3
	Microcystin YR	101	106	108			1.91
	Nodularin-R	106	109	111			1.70

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 350.1 Rev. 2.0	Ammonia in aqueous matrices as mg N/L	2252712
EPA 351.2 Rev. 2.0	Total Kjeldahl Nitrogen in aqueous matrices	2252712
EPA 353.2 Rev. 2.0	Nitrite/Nitrate in aqueous matrices as mg N/L	2252712
EPA 365.1 Rev. 2.0	Total Phosphorus in aqueous matrices as mg P/L	2252712
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2252713
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2252711

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	06/08/2021			06/16/2021 12:14	Roberta Tatti	2252712
EPA 351.2 Rev. 2.0	06/08/2021	06/09/2021 13:12	Benjamin T. Nordmann	06/10/2021 13:18	Virginia P. Brown	2252712
EPA 353.2 Rev. 2.0	06/08/2021			06/11/2021 10:38	Beverly Y. Sanders	2252712
EPA 365.1 Rev. 2.0	06/08/2021	06/11/2021 14:33	Madeline Gruver	06/14/2021 09:31	Shengyu Ding	2252712
EPA 365.1 Rev. 2.0 dissolved	06/08/2021			06/08/2021 14:06	Lipi Saha	2252713
EPA 8321B	06/08/2021	06/09/2021 08:00	Pramila Ghimire	06/09/2021 13:00	Pramila Ghimire	2252711