

Chemical Analysis Report

WQAP-2020-03-25-01

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-2020-02-10-30**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

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

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

Date Certified: 10-APR-2020 08:59

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: Hillsborough River-Near Yukon

Collection Date/Time: 03/24/2020 10:55

Field ID: HAB-SW-0015

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2167584	EPA 8321B	Anatoxin-a**	0.25	U	ug/L	P381267	
		Cylindrospermopsin**	0.25	U	ug/L	P381267	
		Desmethyl microcystin LR**	0.25	U	ug/L	P381267	
		Microcystin LA**	0.25	U	ug/L	P381267	
		Microcystin LF**	0.25	U	ug/L	P381267	
		Microcystin LR**	0.25	U	ug/L	P381267	
		Microcystin LW**	0.25	U	ug/L	P381267	
		Microcystin LY**	0.25	U	ug/L	P381267	
		Microcystin RR**	0.25	U	ug/L	P381267	
		Microcystin WR**	0.50	U	ug/L	P381267	
		Microcystin YR**	0.25	U	ug/L	P381267	
2167585	EPA 350.1 Rev. 2.0	Ammonia-N	0.052		mg N/L	P381430	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	1.4		mg N/L	P381702	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.005	I	mg N/L	P381591	
	EPA 365.1 Rev. 2.0	Total-P	0.18		mg P/L	P381367	
2167586	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.060		mg P/L	P381295	

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: P381430

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

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P381702

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P381591

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P381367

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

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P381295

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

Reference Method: EPA 8321B
Batch ID: P381267

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: P381430

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: P381702

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P381591

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
NO2NO3-N	98.5		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P381367

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: P381295

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

Reference Method: EPA 8321B
Batch ID: P381267

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	112		P	40 - 140
Cylindrospermopsin	111		P	40 - 140
Desmethyl microcystin LR	83.7		P	40 - 140
Microcystin LA	98.1		P	40 - 140
Microcystin LF	96.2		P	40 - 140
Microcystin LR	77.6		P	40 - 140
Microcystin LW	88.9		P	40 - 140
Microcystin LY	97.4		P	40 - 140
Microcystin RR	105		P	40 - 140
Microcystin WR	86.1		P	40 - 140
Microcystin YR	80.3		P	40 - 140

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P381430

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2167591	Ammonia-N	99.3	99.6	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P381591

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2167540	NO2NO3-N	97.2		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P381367

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2167560	Total-P	107	107	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P381295

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2167623	O-Phosphate-P	97.1	98.1	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P381267

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2167173	Anatoxin-a	129	131	P/P	40 - 140
2167173	Cylindrospermopsin	103	102	P/P	40 - 140
2167173	Desmethyl microcystin LR	86.5	88.1	P/P	40 - 140
2167173	Microcystin LA	86.4	90.6	P/P	40 - 140
2167173	Microcystin LF	87.0	88.9	P/P	40 - 140
2167173	Microcystin LR	80.2	87.0	P/P	40 - 140
2167173	Microcystin LW	88.9	86.1	P/P	40 - 140
2167173	Microcystin LY	89.3	89.3	P/P	40 - 140
2167173	Microcystin RR	97.7	102	P/P	40 - 140
2167173	Microcystin WR	85.2	88.6	P/P	40 - 140
2167173	Microcystin YR	83.8	90.0	P/P	40 - 140

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P381430

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2167591	Ammonia-N	0.237	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P381702

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2167585	Kjeldahl Nitrogen	4.64	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P381591

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2167540	NO2NO3-N	0.0	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P381367

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2167560	Total-P	0.656	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P381295

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2167623	O-Phosphate-P	0.968	Spike	P	0 - 20

Reference Method: EPA 8321B
Batch ID: P381267

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2167173	Anatoxin-a	1.70	Spike	P	0 - 30
2167173	Cylindrospermopsin	0.484	Spike	P	0 - 30
2167173	Desmethyl microcystin LR	1.89	Spike	P	0 - 30
2167173	Microcystin LA	4.71	Spike	P	0 - 30
2167173	Microcystin LF	2.13	Spike	P	0 - 30
2167173	Microcystin LR	8.14	Spike	P	0 - 30
2167173	Microcystin LW	3.24	Spike	P	0 - 30
2167173	Microcystin LY	0.0349	Spike	P	0 - 30
2167173	Microcystin RR	4.32	Spike	P	0 - 30
2167173	Microcystin WR	3.95	Spike	P	0 - 30
2167173	Microcystin YR	7.12	Spike	P	0 - 30

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

Quality Assurance Report Surrogates

Lab Sample ID: 2167584
Field Sample ID: HAB-SW-0015

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	127	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Run ID: A98436

Included Lab Sample IDs: 2167586

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	100	98.0	P/P	90 - 110

Reference Method: EPA 8321B

Run ID: A98461

Included Lab Sample IDs: 2167584

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	90.1	100	P/P	50 - 160
Cylindrospermopsin	95.8	109	P/P	60 - 160
Desmethyl microcystin LR	79.2	85.7	P/P	60 - 160
Microcystin LA	91.5	97.6	P/P	50 - 160
Microcystin LF	92.5	97.7	P/P	60 - 160
Microcystin LR	77.8	80.5	P/P	60 - 160
Microcystin LW	84.5	90.3	P/P	60 - 160
Microcystin LY	96.6	105	P/P	60 - 160
Microcystin RR	90.5	99.4	P/P	60 - 160
Microcystin WR	86.5	86.4	P/P	60 - 160
Microcystin YR	90.8	85.3	P/P	60 - 160

Reference Method: EPA 350.1 Rev. 2.0

Run ID: A98491

Included Lab Sample IDs: 2167585

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	102	103	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0

Run ID: A98546

Included Lab Sample IDs: 2167585

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

Reference Method: EPA 365.1 Rev. 2.0

Run ID: A98602

Included Lab Sample IDs: 2167585

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	103	102	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0

Run ID: A98637

Included Lab Sample IDs: 2167585

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	101	95.1	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	99.3	99.6			0.237
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	101					4.64
EPA 353.2 Rev. 2.0	NO ₂ NO ₃ -N	98.5	97.2				0.0
EPA 365.1 Rev. 2.0	Total-P	104	107	107			0.656
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	99.3	97.1	98.1			0.968
EPA 8321B	Anatoxin-a	112	129	131			1.70
	Cylindrospermopsin	111	103	102			0.484
	Desmethyl microcystin LR	83.7	86.5	88.1			1.89
	Microcystin LA	98.1	86.4	90.6			4.71
	Microcystin LF	96.2	87.0	88.9			2.13
	Microcystin LR	77.6	80.2	87.0			8.14
	Microcystin LW	88.9	88.9	86.1			3.24
	Microcystin LY	97.4	89.3	89.3			0.0349
	Microcystin RR	105	97.7	102			4.32
	Microcystin WR	86.1	85.2	88.6			3.95
	Microcystin YR	80.3	83.8	90.0			7.12

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
EPA 350.1 Rev. 2.0	Ammonia in aqueous matrices as mg N/L	2167585
EPA 351.2 Rev. 2.0	Total Kjeldahl Nitrogen in aqueous matrices	2167585
EPA 353.2 Rev. 2.0	Nitrite/Nitrate in aqueous matrices as mg N/L	2167585
EPA 365.1 Rev. 2.0	Total Phosphorus in aqueous matrices as mg P/L	2167585
EPA 365.1 Rev. 2.0 dissolved	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2167586
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2167584

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	03/25/2020			03/28/2020 16:28	Ping Hua	2167585
EPA 351.2 Rev. 2.0	03/25/2020	04/02/2020 09:25	Sima Feizi	04/03/2020 09:11	Jhamieka S. Greenwood	2167585
EPA 353.2 Rev. 2.0	03/25/2020			03/31/2020 09:39	Beverly Y. Sanders	2167585
EPA 365.1 Rev. 2.0	03/25/2020	03/26/2020 17:15	Yen Ta	04/01/2020 17:11	Benjamin T. Nordmann	2167585
EPA 365.1 Rev. 2.0 dissolved	03/25/2020			03/25/2020 17:22	Julia Storbeck	2167586
EPA 8321B	03/25/2020	03/26/2020 09:00	Mohammad Ghaffari	03/26/2020 13:38	Mohammad Ghaffari	2167584