

Chemical Analysis Report

WQAP-2019-09-25-03

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

Event Description: **Algal Bloom Response - As needed**
Request ID: **RQ-2019-08-05-55**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
3319 Maguire Blvd
Suite 232
Orlando, FL 32806
Attn: Mike Linger

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: Dr. rer. nat. Bettina Steinbock, Environmental Administrator

Date Certified: 07-OCT-2019 18:10



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: Sykes Creek HAB

Collection Date/Time: 09/24/2019 11:30

Field ID: Sykes Creek HAB

Matrix: W-SURF-SLT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2122110	EPA 8321B	Anatoxin-a**	0.25	U	ug/L	P371286	
		Cylindrospermopsin**	0.25	U	ug/L	P371286	
		Microcystin LA**	0.25	U	ug/L	P371286	
		Microcystin LF**	0.25	UJ	ug/L	P371286	LCS
		Microcystin LR**	0.25	UJ	ug/L	P371286	LCS
		Microcystin LY**	0.25	U	ug/L	P371286	
		Microcystin RR**	0.25	U	ug/L	P371286	
		Microcystin YR**	0.25	UJ	ug/L	P371286	LCS, MS
2122111	EPA 350.1 Rev. 2.0	Ammonia-N	0.011		mg N/L	P371695	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	2.2		mg N/L	P371410	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.004	U	mg N/L	P371538	
	EPA 365.1 Rev. 2.0	Total-P	0.065		mg P/L	P371495	
2122112	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.040	U	mg P/L	P371440	

Ref. Method and Comment:

EPA 8321B: Refer to the narrative for an explanation of QC Codes.

EPA 365.1 Rev. 2.0 dissolved: O-Phosphate-P: The MDL was elevated because of sample matrix interference.

Quality Assurance Report Method Blank Results

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P371695

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

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P371410

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

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P371538

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

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P371495

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

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P371440

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

Reference Method: EPA 8321B
Batch ID: P371286

Component	Result	Code	Units
Anatoxin-a	0.25	U	ug/L
Cylindrospermopsin	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 LY	0.25	U	ug/L
Microcystin RR	0.25	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: P371695

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Ammonia-N	95.3		P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
 Batch ID: P371410

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Kjeldahl Nitrogen	95.2		P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
 Batch ID: P371538

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: P371495

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Total-P	96.9		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
 Batch ID: P371440

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

Reference Method: EPA 8321B
 Batch ID: P371286

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	107		P	40 - 140
Cylindrospermopsin	105		P	40 - 140
Microcystin LA	119		P	40 - 140
Microcystin LF	142		F	40 - 140
Microcystin LR	147		F	40 - 140
Microcystin LY	134		P	40 - 140
Microcystin RR	124		P	40 - 140
Microcystin YR	149		F	40 - 140

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P371695

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2122032	Ammonia-N	97.4	98.6	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P371410

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2121960	Kjeldahl Nitrogen	93.0	92.3	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P371538

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2122031	NO2NO3-N	99.9	99.9	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P371495

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2121969	Total-P	92.5	91.2	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P371440

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2122112	O-Phosphate-P	90.5	93.6	P/P	90 - 110

Reference Method: EPA 8321B
Batch ID: P371286

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2122068	Anatoxin-a	99.5	102	P/P	40 - 140
2122068	Cylindrospermopsin	98.1	94.1	P/P	40 - 140
2122068	Microcystin LA	105	116	P/P	40 - 140
2122068	Microcystin LF	123	127	P/P	40 - 140
2122068	Microcystin LR	139	139	P/P	40 - 140
2122068	Microcystin LY	129	130	P/P	40 - 140
2122068	Microcystin RR	112	108	P/P	40 - 140
2122068	Microcystin YR	151	153	F/F	40 - 140

Quality Assurance Report Precision

Reference Method: EPA 350.1 Rev. 2.0
 Batch ID: P371695

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2122032	Ammonia-N	0.878	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0
 Batch ID: P371410

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2121960	Kjeldahl Nitrogen	0.624	Spike	P	0 - 20

Reference Method: EPA 353.2 Rev. 2.0
 Batch ID: P371538

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2122031	NO2NO3-N	0.0	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0
 Batch ID: P371495

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2121969	Total-P	1.39	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved
 Batch ID: P371440

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2122112	O-Phosphate-P	3.37	Spike	P	0 - 20

Reference Method: EPA 8321B
 Batch ID: P371286

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2122068	Anatoxin-a	2.41	Spike	P	0 - 30
2122068	Cylindrospermopsin	4.02	Spike	P	0 - 30
2122068	Microcystin LA	9.58	Spike	P	0 - 30
2122068	Microcystin LF	3.20	Spike	P	0 - 30
2122068	Microcystin LR	0.235	Spike	P	0 - 30
2122068	Microcystin LY	0.149	Spike	P	0 - 30
2122068	Microcystin RR	3.73	Spike	P	0 - 30
2122068	Microcystin YR	1.75	Spike	P	0 - 30

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

Quality Assurance Report Surrogates

Lab Sample ID: 2122110
Field Sample ID: Sykes Creek HAB

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	100	P	30 - 165

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B
 Run ID: A94524
 Included Lab Sample IDs: 2122110

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	69.1	84.5	P/P	50 - 160
Cylindrospermopsin	86.8	105	P/P	60 - 160
Microcystin LA	99.7	117	P/P	50 - 160
Microcystin LF	93.0	119	P/P	60 - 160
Microcystin LR	115	141	P/P	60 - 160
Microcystin LY	115	135	P/P	60 - 160
Microcystin RR	99.9	113	P/P	60 - 160
Microcystin YR	129	141	P/P	60 - 160

Reference Method: EPA 365.1 Rev. 2.0 dissolved
 Run ID: A94551
 Included Lab Sample IDs: 2122112

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	97.1	96.5	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
 Run ID: A94584
 Included Lab Sample IDs: 2122111

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	99.8	99.5	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
 Run ID: A94589
 Included Lab Sample IDs: 2122111

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
NO2NO3-N	99.5	99.0	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
 Run ID: A94616
 Included Lab Sample IDs: 2122111

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	99.8	100	P/P	90 - 110

Reference Method: EPA 350.1 Rev. 2.0
 Run ID: A94647
 Included Lab Sample IDs: 2122111

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	96.1	97.5	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 SMP	MS
EPA 350.1 Rev. 2.0	Ammonia-N	95.3	97.4	98.6			0.878
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	95.2	93.0	92.3			0.624
EPA 353.2 Rev. 2.0	NO ₂ NO ₃ -N	100	99.9	99.9			0.0
EPA 365.1 Rev. 2.0	Total-P	96.9	92.5	91.2			1.39
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	98.2	90.5	93.6			3.37
EPA 8321B	Anatoxin-a	107	99.5	102			2.41
	Cylindrospermopsin	105	98.1	94.1			4.02
	Microcystin LA	119	105	116			9.58
	Microcystin LF	142	123	127			3.20
	Microcystin LR	147	139	139			0.235
	Microcystin LY	134	129	130			0.149
	Microcystin RR	124	112	108			3.73
	Microcystin YR	149	151	153			1.75

Reference Method Descriptions

Method / DoH Cert #	Description	Associated Samples
EPA 350.1 Rev. 2.0 / E31780	Ammonia in aqueous matrices as mg N/L	2122111
EPA 351.2 Rev. 2.0 / E31780	Total Kjeldahl Nitrogen in aqueous matrices	2122111
EPA 353.2 Rev. 2.0 / E31780	Nitrite/Nitrate in aqueous matrices as mg N/L	2122111
EPA 365.1 Rev. 2.0 / E31780	Total Phosphorus in aqueous matrices as mg P/L	2122111
EPA 365.1 Rev. 2.0 dissolved / E31780	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	2122112
EPA 8321B / E31780	Microcystins in water matrices by HPLC/MS/MS	2122110

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	09/25/2019			10/01/2019 14:28	Ping Hua	2122111
EPA 351.2 Rev. 2.0	09/25/2019	09/26/2019 13:26	Sima Feizi	09/27/2019 19:41	Julia Storbeck	2122111
EPA 353.2 Rev. 2.0	09/25/2019			09/30/2019 11:04	Beverly Y. Sanders	2122111
EPA 365.1 Rev. 2.0	09/25/2019	09/27/2019 15:05	Lipi Saha	09/30/2019 11:39	Lipi Saha	2122111
EPA 365.1 Rev. 2.0 dissolved	09/25/2019			09/26/2019 11:07	Ping Hua	2122112
EPA 8321B	09/25/2019	09/26/2019 09:00	Juyoung Kim	09/26/2019 12:54	Juyoung Kim	2122110