

Chemical Analysis Report

WQAP-2020-09-03-03

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-2020-08-31-60**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
DEP SO ROC
2295 Victoria Ave
Fort Myers, FL 33901
Attn: Kirby Wolfe

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: Kerry Tate, Ph.D., Environmental Administrator

Date Certified: 08-SEP-2020 09:42

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.

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 group are included in this report: 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: Upstream NW

Collection Date/Time: 09/02/2020 10:26

Field ID: CC-090220-001

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2194164	EPA 8321B	Anatoxin-a**	0.25	UY	ug/L	P386886	MS
		Cylindrospermopsin**	0.25	UY	ug/L	P386886	
		Desmethyl microcystin LR**	0.25	UY	ug/L	P386886	
		Microcystin LA**	0.25	UY	ug/L	P386886	
		Microcystin LF**	0.25	UY	ug/L	P386886	
		Microcystin LR**	0.25	UY	ug/L	P386886	
		Microcystin LW**	0.25	UY	ug/L	P386886	
		Microcystin LY**	0.25	UY	ug/L	P386886	
		Microcystin RR**	0.25	UY	ug/L	P386886	
		Microcystin WR**	0.50	UY	ug/L	P386886	
		Microcystin YR**	0.25	UY	ug/L	P386886	

Ref. Method and Comment:

EPA 8321B: Y - Sample was received with inadequate sample preservation; see NCR report.

Non-Conformance Report

NCR ID: 8410

Event(s)

WQAP-2020-09-03-03

Job(s)

Sample(s)

Test(s)

NCR Type: SHIPPING/RECEIVING

NCR Category: Preservation Not Intact

Observation: Sample(s) received outside the acceptable temperature range (7.6 Celsius).

Resolution: Data will be qualified as appropriate.

Customer notified.

Supervisors notified. The algae were not degraded and were able to be analyzed. The result will not be qualified.-AGR

Authorized by/Date: Kerry Tate, Ph.D. 9/8/2020

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 8321B
Batch ID: P386886

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 8321B
Batch ID: P386886

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	106		P	40 - 140
Cylindrospermopsin	126		P	40 - 140
Desmethyl microcystin LR	129		P	40 - 140
Microcystin LA	93.8		P	40 - 140
Microcystin LF	115		P	40 - 140
Microcystin LR	110		P	40 - 140
Microcystin LW	96.2		P	40 - 140
Microcystin LY	107		P	40 - 140
Microcystin RR	83.6		P	40 - 140
Microcystin WR	104		P	40 - 140
Microcystin YR	112		P	40 - 140

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 8321B
Batch ID: P386886

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2194012	Anatoxin-a	105	108	P/P	40 - 140
2194012	Cylindrospermopsin	116	118	P/P	40 - 140
2194012	Desmethyl microcystin LR	140	142	P/F	40 - 140
2194012	Microcystin LA	90.4	97.5	P/P	40 - 140
2194012	Microcystin LF	110	116	P/P	40 - 140
2194012	Microcystin LR	119	127	P/P	40 - 140
2194012	Microcystin LW	116	113	P/P	40 - 140
2194012	Microcystin LY	116	116	P/P	40 - 140
2194012	Microcystin RR	84.6	83.2	P/P	40 - 140
2194012	Microcystin WR	124	116	P/P	40 - 140
2194012	Microcystin YR	120	125	P/P	40 - 140

Quality Assurance Report Precision

Reference Method: EPA 8321B
Batch ID: P386886

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2194012	Anatoxin-a	2.81	Spike	P	0 - 30
2194012	Cylindrospermopsin	1.96	Spike	P	0 - 30
2194012	Desmethyl microcystin LR	1.34	Spike	P	0 - 30
2194012	Microcystin LA	7.60	Spike	P	0 - 30
2194012	Microcystin LF	4.94	Spike	P	0 - 30
2194012	Microcystin LR	6.95	Spike	P	0 - 30
2194012	Microcystin LW	2.27	Spike	P	0 - 30
2194012	Microcystin LY	0.0712	Spike	P	0 - 30
2194012	Microcystin RR	1.75	Spike	P	0 - 30
2194012	Microcystin WR	6.86	Spike	P	0 - 30
2194012	Microcystin YR	4.03	Spike	P	0 - 30

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

Quality Assurance Report Surrogates

Lab Sample ID: 2194164

Field Sample ID: CC-090220-001

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Microcystin LR-d7	110	P	30 - 160
EPA 8321B	Microcystin LR ethyl-d5	90.0	P	30 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B

Run ID: A100806

Included Lab Sample IDs: 2194164

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	109	117	P/P	50 - 160
Cylindrospermopsin	120	120	P/P	60 - 160
Desmethyl microcystin LR	122	131	P/P	60 - 160
Microcystin LA	94.3	100	P/P	50 - 160
Microcystin LF	115	116	P/P	50 - 160
Microcystin LR	118	118	P/P	60 - 160
Microcystin LW	86.9	114	P/P	60 - 160
Microcystin LY	121	114	P/P	60 - 160
Microcystin RR	82.9	89.3	P/P	60 - 160
Microcystin WR	110	109	P/P	60 - 160
Microcystin YR	109	114	P/P	60 - 160

* 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 SMP	MS
			LCS			
EPA 8321B	Anatoxin-a	106	105	108		2.81
	Cylindrospermopsin	126	116	118		1.96
	Desmethyl microcystin LR	129	140	142		1.34
	Microcystin LA	93.8	90.4	97.5		7.60
	Microcystin LF	115	110	116		4.94
	Microcystin LR	110	119	127		6.95
	Microcystin LW	96.2	116	113		2.27
	Microcystin LY	107	116	116		0.0712
	Microcystin RR	83.6	84.6	83.2		1.75
	Microcystin WR	104	124	116		6.86
	Microcystin YR	112	120	125		4.03

Reference Method Descriptions

Method	Description	Associated Samples
EPA 8321B	Microcystins in water matrices by HPLC/MS/MS	2194164

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	09/03/2020	09/03/2020 13:00	Mohammad Ghaffari	09/03/2020 14:30	Pramila Ghimire	2194164