

Chemical Analysis Report

SW-DIST-2019-05-10-01

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-2019-05-06-74**
Customer: **SW-DIST**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
FL Dept. of Environmental Protection
13051 N. Telecom Parkway
Temple Terrace, FL 33637-0926
Attn: Matthew Bearden

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: Colin Wright, Program Administrator

Date Certified: 15-MAY-2019 11:12



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: Anna Maria Island Bloom Response

Collection Date/Time: 05/09/2019 10:40

Field ID: ANNA MARIA ISLAND

Matrix: W-SURF-SLT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2085529	EPA 8321B	Anatoxin-a**	0.25	U	ug/L	P363858	
		Cylindrospermopsin**	0.25	U	ug/L	P363858	
		Microcystin LA**	0.25	U	ug/L	P363858	
		Microcystin LF**	0.25	U	ug/L	P363858	
		Microcystin LR**	0.25	U	ug/L	P363858	
		Microcystin LY**	0.25	U	ug/L	P363858	
		Microcystin RR**	0.25	U	ug/L	P363858	
		Microcystin YR**	0.25	U	ug/L	P363858	
		Saxitoxin**	0.25	UJ	ug/L	P363858	MS

Ref. Method and Comment:

EPA 8321B: Method validation for saxitoxin is still in progress. Second source was not analyzed.

Sample Location: Holmes Beach Bloom Response

Collection Date/Time: 05/09/2019 11:25

Field ID: HOLMES BEACH

Matrix: W-SURF-SLT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2085530	EPA 8321B	Anatoxin-a**	0.25	U	ug/L	P363858	
		Cylindrospermopsin**	0.25	U	ug/L	P363858	
		Microcystin LA**	0.25	U	ug/L	P363858	
		Microcystin LF**	0.25	U	ug/L	P363858	
		Microcystin LR**	0.25	U	ug/L	P363858	
		Microcystin LY**	0.25	U	ug/L	P363858	
		Microcystin RR**	0.25	U	ug/L	P363858	
		Microcystin YR**	0.25	U	ug/L	P363858	
		Saxitoxin**	0.25	UJ	ug/L	P363858	MS

Ref. Method and Comment:

EPA 8321B: Method validation for saxitoxin is still in progress. Second source was not analyzed.

Sample Location: Palma Sola Bay Bloom Response

Collection Date/Time: 05/09/2019 12:20

Field ID: PALMA SOLA BAY

Matrix: W-SURF-SLT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2085531	EPA 8321B	Anatoxin-a**	0.25	U	ug/L	P363858	
		Cylindrospermopsin**	0.25	U	ug/L	P363858	
		Microcystin LA**	0.25	U	ug/L	P363858	
		Microcystin LF**	0.25	U	ug/L	P363858	
		Microcystin LR**	0.25	U	ug/L	P363858	
		Microcystin LY**	0.25	U	ug/L	P363858	
		Microcystin RR**	0.25	U	ug/L	P363858	
		Microcystin YR**	0.25	U	ug/L	P363858	
		Saxitoxin**	0.25	UJ	ug/L	P363858	MS

Ref. Method and Comment:

EPA 8321B: Method validation for saxitoxin is still in progress. Second source was not analyzed.

Quality Assurance Report

Method Blank Results

Reference Method: EPA 8321B
Batch ID: P363858

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
Saxitoxin	0.25	U	ug/L

Quality Assurance Report

Laboratory Control Sample Accuracy

Reference Method: EPA 8321B
Batch ID: P363858

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	86.7		P	40 - 140
Cylindrospermopsin	91.6		P	40 - 140
Microcystin LA	90.6		P	40 - 140
Microcystin LF	93.8		P	40 - 140
Microcystin LR	94.0		P	40 - 140
Microcystin LY	95.4		P	40 - 140
Microcystin RR	89.5		P	40 - 140
Microcystin YR	85.1		P	40 - 140
Saxitoxin	79.9		P	40 - 140

Quality Assurance Report

Matrix Spike Accuracy

Reference Method: EPA 8321B

Batch ID: P363858

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2085469	Anatoxin-a	90.7	93.2	P/P	40 - 140
2085469	Cylindrospermopsin	90.6	91.4	P/P	40 - 140
2085469	Microcystin LA	98.1	97.9	P/P	40 - 140
2085469	Microcystin LF	102	106	P/P	40 - 140
2085469	Microcystin LR	102	108	P/P	40 - 140
2085469	Microcystin LY	90.6	96.1	P/P	40 - 140
2085469	Microcystin RR	99.9	99.9	P/P	40 - 140
2085469	Microcystin YR	94.7	97.3	P/P	40 - 140
2085469	Saxitoxin	11.2	12.5	F/F	40 - 140

Quality Assurance Report Precision

Reference Method: EPA 8321B
Batch ID: P363858

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2085469	Anatoxin-a	2.70	Spike	P	0 - 30
2085469	Cylindrospermopsin	0.913	Spike	P	0 - 30
2085469	Microcystin LA	0.188	Spike	P	0 - 30
2085469	Microcystin LF	3.48	Spike	P	0 - 30
2085469	Microcystin LR	6.04	Spike	P	0 - 30
2085469	Microcystin LY	5.87	Spike	P	0 - 30
2085469	Microcystin RR	0.0186	Spike	P	0 - 30
2085469	Microcystin YR	2.65	Spike	P	0 - 30
2085469	Saxitoxin	11.1	Spike	P	0 - 30

* Sample, spike and/or laboratory control sample precision (LCS) is reported.
Replicate spike precision may be reported when sample results are below quantifiable levels.

Quality Assurance Report Surrogates

Lab Sample ID: 2085529

Field Sample ID: ANNA MARIA ISLAND

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	84.2	P	30 - 165

Lab Sample ID: 2085530

Field Sample ID: HOLMES BEACH

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	87.1	P	30 - 165

Lab Sample ID: 2085531

Field Sample ID: PALMA SOLA BAY

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	91.4	P	30 - 165

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B

Run ID: A91426

Included Lab Sample IDs: 2085529, 2085530, 2085531

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	96.3	97.3	P/P	50 - 160
Cylindrospermopsin	95.3	95.5	P/P	60 - 160
Microcystin LA	94.1	96.5	P/P	50 - 160
Microcystin LF	99.5	94.0	P/P	60 - 160
Microcystin LR	101	96.0	P/P	60 - 160
Microcystin LY	96.2	96.1	P/P	60 - 160
Microcystin RR	95.0	92.8	P/P	60 - 160
Microcystin YR	93.0	88.2	P/P	60 - 160
Saxitoxin	102	103	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	MS		
EPA 8321B	Anatoxin-a	86.7	90.7	93.2		2.70
	Cylindrospermopsin	91.6	90.6	91.4		0.913
	Microcystin LA	90.6	98.1	97.9		0.188
	Microcystin LF	93.8	102	106		3.48
	Microcystin LR	94.0	102	108		6.04
	Microcystin LY	95.4	90.6	96.1		5.87
	Microcystin RR	89.5	99.9	99.9		0.0186
	Microcystin YR	85.1	94.7	97.3		2.65
	Saxitoxin	79.9	11.2	12.5		11.1

Reference Method Descriptions

Method / DoH Cert #	Description	<u>Associated Samples</u>
EPA 8321B / E31780	Microcystins in water matrices by HPLC/MS/MS	2085529, 2085530, 2085531

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	05/10/2019	05/14/2019 12:00	Juyoung Kim	05/14/2019 16:00	Juyoung Kim	2085529
	05/10/2019	05/14/2019 12:00	Juyoung Kim	05/14/2019 16:17	Juyoung Kim	2085530
	05/10/2019	05/14/2019 12:00	Juyoung Kim	05/14/2019 16:34	Juyoung Kim	2085531