

Chemical Analysis Report

WQAP-2018-07-10-02

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-2018-07-09-63**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
2295 Victoria AVE
Suite 364
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: Colin Wright, Program Administrator

Date Certified: 12-JUL-2018 08:44

A handwritten signature in black ink, appearing to read "Colin Wright", is displayed on a light-colored rectangular 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 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 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 RR BRIDGE CALOOSAHATCHEE Collection Date/Time: 07/09/2018 09:50

Field ID: UPSTREAM RR BRIDGE CALOOSAHAT

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2005345	EPA 8321B	Microcystin LR**	15		ug/L	P348123	
		Microcystin LA**	0.25	U	ug/L	P348123	
		Microcystin RR**	0.25	U	ug/L	P348123	
		Microcystin YR**	0.25	U	ug/L	P348123	
		Anatoxin-a**	0.25	U	ug/L	P348123	
		Cylindrospermopsin**	0.25	U	ug/L	P348123	
		Microcystin LF**	0.25	U	ug/L	P348123	
		Microcystin LY**	0.25	U	ug/L	P348123	

Quality Assurance Report

Method Blank Results

Reference Method: EPA 8321B
Batch ID: P348123

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 8321B
Batch ID: P348123

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	115		P	40 - 160
Cylindrospermopsin	114		P	40 - 160
Microcystin LA	109		P	40 - 160
Microcystin LF	107		P	40 - 150
Microcystin LR	96.9		P	40 - 160
Microcystin LY	108		P	40 - 150
Microcystin RR	113		P	40 - 160
Microcystin YR	101		P	40 - 160

Quality Assurance Report

Matrix Spike Accuracy

Reference Method: EPA 8321B
Batch ID: P348123

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2005332	Anatoxin-a	103	106	P/P	40 - 160
2005332	Cylindrospermopsin	106	108	P/P	40 - 160
2005332	Microcystin LA	96.3	97.8	P/P	40 - 160
2005332	Microcystin LF	102	104	P/P	40 - 150
2005332	Microcystin LR	108	112	P/P	40 - 160
2005332	Microcystin LY	103	103	P/P	40 - 150
2005332	Microcystin RR	111	113	P/P	40 - 160
2005332	Microcystin YR	102	111	P/P	40 - 160

Quality Assurance Report Precision

Reference Method: EPA 8321B
Batch ID: P348123

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2005332	Anatoxin-a	2.45	Spike	P	0 - 30
2005332	Cylindrospermopsin	1.74	Spike	P	0 - 30
2005332	Microcystin LA	1.61	Spike	P	0 - 30
2005332	Microcystin LF	1.58	Spike	P	0 - 30
2005332	Microcystin LR	3.51	Spike	P	0 - 30
2005332	Microcystin LY	0.550	Spike	P	0 - 30
2005332	Microcystin RR	2.01	Spike	P	0 - 30
2005332	Microcystin YR	8.87	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: 2005345

Field Sample ID: UPSTREAM RR BRIDGE CALOOSAHAT

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	108	P	30 - 165

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B

Run ID: A85148

Included Lab Sample IDs: 2005345

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	117	115	P/P	50 - 160
Cylindrospermopsin	113	113	P/P	60 - 160
Microcystin LA	110	107	P/P	50 - 160
Microcystin LF	109	104	P/P	60 - 160
Microcystin LR	102	95.2	P/P	60 - 160
Microcystin LY	108	98.9	P/P	60 - 160
Microcystin RR	116	115	P/P	60 - 160
Microcystin YR	98.7	91.4	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	115	103	106		2.45
	Cylindrospermopsin	114	106	108		1.74
	Microcystin LA	109	96.3	97.8		1.61
	Microcystin LF	107	102	104		1.58
	Microcystin LR	96.9	108	112		3.51
	Microcystin LY	108	103	103		0.550
	Microcystin RR	113	111	113		2.01
	Microcystin YR	101	102	111		8.87

Reference Method Descriptions

Method / DoH Cert #	Description	<u>Associated Samples</u>
EPA 8321B / E31780	Microcystins in water matrices by HPLC/MS/MS	2005345

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	07/10/2018	07/11/2018 11:30	Juyoung Kim	07/11/2018 13:03	Juyoung Kim	2005345