

Chemical Analysis Report

WQAP-2017-04-13-03

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

Event Description: **Algal Bloom Response**

Request ID: **RQ-2017-02-20-48**

Customer: **WQAP**

Project ID: **BLOOM-RESP**

Send Reports to:
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Certified by: Colin Wright, Program Administrator

Date Certified: 17-APR-2017 14:04

A handwritten signature in black ink, appearing to read "Colin Wright", is written over a light gray 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: Algal Bloom @ Orienta Lake

Collection Date/Time: 04/12/2017 09:50

Field ID: Orienta 1

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1888802	EPA 8321B	Microcystin LR**	1.0	U	ug/L	P323402	
		Microcystin LA**	1.0	U	ug/L	P323402	
		Microcystin RR**	1.0	U	ug/L	P323402	
		Microcystin YR**	1.0	U	ug/L	P323402	
		Anatoxin-a**	1.0	UJ	ug/L	P323402	MS
		Cylindrospermopsin**	1.0	U	ug/L	P323402	

Ref. Method and Comment:

EPA 8321B: Microcystin LF = 1.0 U ug/L and microcystin LY = 1.0 U ug/L. Results for microcystins LF and LY are qualitative only and their validation is pending. Refer to the narrative for an explanation of QC Codes.

Quality Assurance Report Method Blank Results

Reference Method: EPA 8321B
Batch ID: P323402

Component	Result	Code	Units
Anatoxin-a	1.0	U	ug/L
Cylindrospermopsin	1.0	U	ug/L
Microcystin LA	1.0	U	ug/L
Microcystin LR	1.0	U	ug/L
Microcystin RR	1.0	U	ug/L
Microcystin YR	1.0	U	ug/L

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: EPA 8321B
Batch ID: P323402

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Anatoxin-a	65.6		P	40 - 140
Cylindrospermopsin	71.4		P	40 - 140
Microcystin LA	74.2		P	40 - 160
Microcystin LR	99.3		P	40 - 160
Microcystin RR	82.0		P	40 - 160
Microcystin YR	96.2		P	40 - 160

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 8321B
Batch ID: P323402

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1888802	Anatoxin-a	166	142	F/F	40 - 140
1888802	Cylindrospermopsin	96.2	92.6	P/P	40 - 140
1888802	Microcystin LA	78.8	81.9	P/P	40 - 160
1888802	Microcystin LR	120	113	P/P	40 - 160
1888802	Microcystin RR	91.7	90.6	P/P	40 - 160
1888802	Microcystin YR	129	114	P/P	40 - 160

Quality Assurance Report Precision

Reference Method: EPA 8321B
Batch ID: P323402

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1888802	Anatoxin-a	15.6	Spike	P	0 - 30
1888802	Cylindrospermopsin	3.81	Spike	P	0 - 30
1888802	Microcystin LA	3.86	Spike	P	0 - 30
1888802	Microcystin LR	6.01	Spike	P	0 - 30
1888802	Microcystin RR	1.26	Spike	P	0 - 30
1888802	Microcystin YR	12.3	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: 1888802
Field Sample ID: Orienta 1

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	91.3	P	40 - 160
EPA 8321B	Microcystin LF-d5	81.0	P	40 - 150

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B
Run ID: A75800
Included Lab Sample IDs: 1888802

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Anatoxin-a	82.4	72.7	P/P	50 - 160
Cylindrospermopsin	82.4	89.7	P/P	60 - 160
Microcystin LA	84.0	80.7	P/P	50 - 160
Microcystin LR	106	110	P/P	60 - 160
Microcystin RR	92.7	87.6	P/P	60 - 160
Microcystin YR	113	99.3	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	LCS	Precision SMP	MS
EPA 8321B	Anatoxin-a	65.6	166 142			15.6
	Cylindrospermopsin	71.4	96.2 92.6			3.81
	Microcystin LA	74.2	78.8 81.9			3.86
	Microcystin LR	99.3	120 113			6.01
	Microcystin RR	82.0	91.7 90.6			1.26

Quality Assurance Report Summary

Ref. Method	Analyte	LCS % Recovery	MS % Recovery		Precision		MS
					LCS	SMP	
EPA 8321B	Microcystin YR	96.2	129	114			12.3

Reference Method Descriptions

Method / DoH Cert #	Description	<u>Associated Samples</u>
EPA 8321B / E31780	Microcystins in water matrices by HPLC/MS/MS	1888802

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	04/13/2017	04/14/2017	Juyoung Kim	04/14/2017	Juyoung Kim	1888802