

Chemical Analysis Report

WQAP-2016-12-13-02

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

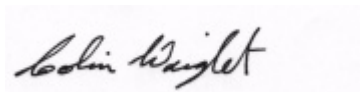
Event Description: **Algal Bloom Response - As needed**
Request ID: **RQ-2016-07-25-93**
Customer: **WQAP**
Project ID: **BLOOM-RESP**

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.
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Phone (850) 245-8085

Certified by: Colin Wright, Environmental Administrator

Date Certified: 15-DEC-2016 12:47



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 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: Little Lake Jackson (WBID1860)

Collection Date/Time: 12/12/2016 13:15

Field ID: East Dock-HAB Monitoring

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1857150	EPA 8321B	Microcystin LR**	0.99	T	ug/L	P316778	
		Microcystin LA**	2.6	IJ	ug/L	P316778	RPD
		Microcystin RR**	1.0	U	ug/L	P316778	
		Microcystin YR**	1.0	U	ug/L	P316778	
		Cylindrospermopsin**	1.0	UJ	ug/L	P316778	RPD

Ref. Method and Comment:

EPA 8321B: Anatoxin-a = 1.0 U ug/L. 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.

Non-Conformance Report

NCR ID: 6154

Event(s)

Job(s)

Sample(s)

Test(s)

WQAP-2016-12-13-02

NCR Type: SHIPPING/RECEIVING

NCR Category: Receiving

Observation: Sample submittal form indicates a container was collected for BLOOM-ID, but no container was received.

Resolution: Sample 1857149 has been cancelled.

Authorised by/Date: Ramsey K. White 12/15/2016

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: P316778

Component	Result	Code	Units
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: P316778

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Cylindrospermopsin	99.0		P	50 - 160
Microcystin LA	96.2		P	40 - 160
Microcystin LR	88.0		P	40 - 160
Microcystin RR	115		P	40 - 160
Microcystin YR	82.9		P	50 - 160

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 8321B
Batch ID: P316778

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1857150	Cylindrospermopsin	86.8	124	P/P	50 - 160
1857150	Microcystin LA	112	74.5	P/P	40 - 160
1857150	Microcystin LR	97.8	108	P/P	40 - 160
1857150	Microcystin RR	107	112	P/P	40 - 160
1857150	Microcystin YR	94.1	90.7	P/P	50 - 160

Quality Assurance Report Precision

Reference Method: EPA 8321B
Batch ID: P316778

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1857150	Cylindrospermopsin	35.3	Spike	F	0 - 30
1857150	Microcystin LA	31.2	Spike	F	0 - 30
1857150	Microcystin LR	9.91	Spike	P	0 - 30
1857150	Microcystin RR	5.14	Spike	P	0 - 30
1857150	Microcystin YR	3.68	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: 1857150
Field Sample ID: East Dock-HAB Monitoring

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Carbamazepine-d10	111	P	40 - 160
EPA 8321B	Microcystin LF-d5	120	P	40 - 150

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B
Run ID: A73382
Included Lab Sample IDs: 1857150

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Cylindrospermopsin	94.3	81.3	P/P	60 - 160
Microcystin LA	88.4	78.5	P/P	50 - 160
Microcystin LR	84.5	91.5	P/P	60 - 160
Microcystin RR	115	110	P/P	60 - 160
Microcystin YR	101	82.9	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	Cylindrospermopsin	99.0	86.8	124		35.3
	Microcystin LA	96.2	112	74.5		31.2
	Microcystin LR	88.0	97.8	108		9.91
	Microcystin RR	115	107	112		5.14
	Microcystin YR	82.9	94.1	90.7		3.68

Reference Method Descriptions

Method / DoH Cert #	Description	<u>Associated Samples</u>
EPA 8321B / E31780	Microcystins in water matrices by HPLC/MS/MS	1857150

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	12/13/2016	12/14/2016	Juyoung Kim	12/14/2016	Juyoung Kim	1857150