

Chemical Analysis Report

SO-DIST-2016-01-07-01

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-2016-01-04-74**

Customer: **SO-DIST**

Project ID: **BLOOM-RESP**

Send Reports to:
FL Dept. of Environmental Protection
FLDEP South District Office
2295 Victoria Ave. Suite 364
Fort Myers, FL 33901-3381
Attn: Kirby Wolfe

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Certified by: Thekkekalathil Chandrasekhar, Chemist Administrator

Date Certified: 21-JAN-2016 14:14



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 groups are included in this report: Nutrients and 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: HUCKLEBERRY LAKE ALGAE BLOOM #1

Collection Date/Time: 01/06/2016 12:00

Field ID: SD010616-9

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1761359	DEP SOP LC-001-2 (based on EPA 8321B)	Microcystin LR**	14		ug/L	P296749	
		Microcystin RR**	5.1		ug/L	P296749	
1761360	EPA 350.1 Rev. 2.0	Ammonia-N	0.047		mg N/L	P296985	
	EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	1.5		mg N/L	P296888	
	EPA 353.2 Rev. 2.0	NO2NO3-N	0.16		mg N/L	P296915	
	EPA 365.1 Rev. 2.0	Total-P	0.13		mg P/L	P296911	
1761361	EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	0.080	J	mg P/L	P296765	

Ref. Method and Comment:

DEP SOP LC-001-2 (based on EPA 8321B): Results confirmed in re-extraction.

EPA 365.1 Rev. 2.0 dissolved: O-Phosphate-P: The sample contained algae and was filtered in the laboratory.

Quality Assurance Report Method Blank Results

Reference Method: DEP SOP LC-001-2 (based on EPA 8321B)

Batch ID: P296749

Component	Result	Code	Units
Microcystin LR	1.0	U	ug/L
Microcystin RR	1.0	U	ug/L

Reference Method: EPA 350.1 Rev. 2.0

Batch ID: P296985

Component	Result	Code	Units
Ammonia-N	0.002	U	mg N/L

Reference Method: EPA 351.2 Rev. 2.0

Batch ID: P296888

Component	Result	Code	Units
Kjeldahl Nitrogen	0.080	U	mg N/L

Reference Method: EPA 353.2 Rev. 2.0

Batch ID: P296915

Component	Result	Code	Units
NO2NO3-N	0.004	U	mg N/L

Reference Method: EPA 365.1 Rev. 2.0

Batch ID: P296911

Component	Result	Code	Units
Total-P	0.002	U	mg P/L

Reference Method: EPA 365.1 Rev. 2.0 dissolved

Batch ID: P296765

Component	Result	Code	Units
O-Phosphate-P	0.004	U	mg P/L

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: DEP SOP LC-001-2 (based on EPA 8321B)
Batch ID: P296749

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Microcystin LR	105	103	P/P	50 - 150
Microcystin RR	82.1	80.6	P/P	50 - 150

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P296985

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Ammonia-N	97.8		P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Batch ID: P296888

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Kjeldahl Nitrogen	106		P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P296915

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
NO2NO3-N	101		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P296911

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Total-P	97.3		P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P296765

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
O-Phosphate-P	102		P	90 - 110

Quality Assurance Report Matrix Spike Accuracy

Reference Method: DEP SOP LC-001-2 (based on EPA 8321B)
Batch ID: P296749

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1761352	Microcystin LR	97.8	115	P/P	50 - 150
1761352	Microcystin RR	79.1	81.5	P/P	50 - 150

Reference Method: EPA 350.1 Rev. 2.0
Batch ID: P296985

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1761280	Ammonia-N	96.0	98.0	P/P	90 - 110

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 351.2 Rev. 2.0
 Batch ID: P296888

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1761319	Kjeldahl Nitrogen	104	107	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
 Batch ID: P296915

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1761360	NO2NO3-N	97.5	98.5	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0
 Batch ID: P296911

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1761277	Total-P	95.2	99.0	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0 dissolved
 Batch ID: P296765

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1761434	O-Phosphate-P	94.9	95.6	P/P	90 - 110

Quality Assurance Report Precision

Reference Method: DEP SOP LC-001-2 (based on EPA 8321B)
 Batch ID: P296749

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1761352	Microcystin LR	16.2	Spike	P	0 - 30
1761352	Microcystin RR	2.99	Spike	P	0 - 30
LFB	Microcystin LR	1.92	LCS	P	0 - 30
LFB	Microcystin RR	1.84	LCS	P	0 - 30

Reference Method: EPA 350.1 Rev. 2.0
 Batch ID: P296985

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1761280	Ammonia-N	1.71	Spike	P	0 - 20

Reference Method: EPA 351.2 Rev. 2.0
 Batch ID: P296888

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1761319	Kjeldahl Nitrogen	2.46	Spike	P	0 - 20

Quality Assurance Report Precision

Reference Method: EPA 353.2 Rev. 2.0
Batch ID: P296915

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1761360	NO2NO3-N	0.567	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0
Batch ID: P296911

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1761277	Total-P	2.76	Spike	P	0 - 20

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Batch ID: P296765

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1761434	O-Phosphate-P	0.685	Spike	P	0 - 20

* 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: 1761359
Field Sample ID: SD010616-9

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
DEP SOP LC-001-2 (based on EPA 8321B)	Carbamazepine-d10	139	P	40 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 365.1 Rev. 2.0 dissolved
Run ID: A66963
Included Lab Sample IDs: 1761361

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
O-Phosphate-P	98.3	96.2	P/P	90 - 110

Reference Method: EPA 353.2 Rev. 2.0
Run ID: A67023
Included Lab Sample IDs: 1761360

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
NO2NO3-N	100	100	P/P	90 - 110

Reference Method: EPA 351.2 Rev. 2.0
Run ID: A67057
Included Lab Sample IDs: 1761360

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
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Quality Assurance Report Calibration Verification

Reference Method: EPA 351.2 Rev. 2.0

Run ID: A67057

Included Lab Sample IDs: 1761360

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Kjeldahl Nitrogen	103	103	P/P	90 - 110

Reference Method: DEP SOP LC-001-2 (based on EPA 8321B)

Run ID: A67059

Included Lab Sample IDs: 1761359

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Microcystin LR	87.7	110	P/P	60 - 160
Microcystin RR	94.1	75.0	P/P	60 - 160

Reference Method: EPA 350.1 Rev. 2.0

Run ID: A67060

Included Lab Sample IDs: 1761360

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Ammonia-N	100	101	P/P	90 - 110

Reference Method: EPA 365.1 Rev. 2.0

Run ID: A67062

Included Lab Sample IDs: 1761360

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Total-P	102	103	P/P	90 - 110

* 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
DEP SOP LC-001-2 (based on EPA 8321B)	Microcystin LR	105	103	97.8	115	1.92		16.2
	Microcystin RR	82.1	80.6	79.1	81.5	1.84		2.99
EPA 350.1 Rev. 2.0	Ammonia-N	97.8		96.0	98.0			1.71
EPA 351.2 Rev. 2.0	Kjeldahl Nitrogen	106		104	107			2.46
EPA 353.2 Rev. 2.0	NO2NO3-N	101		97.5	98.5			0.567
EPA 365.1 Rev. 2.0	Total-P	97.3		95.2	99.0			2.76
EPA 365.1 Rev. 2.0 dissolved	O-Phosphate-P	102		94.9	95.6			0.685

Reference Method Descriptions

Method / DoH Cert #	Description	Associated Samples
DEP SOP LC-001-2 (based on EPA 8321B) / E31780	Microcystins in water matrices by HPLC/MS/MS	1761359
EPA 350.1 Rev. 2.0 / E31780	Ammonia in aqueous matrices as mg N/L	1761360

Reference Method Descriptions

Method / DoH Cert #	Description	<u>Associated Samples</u>
EPA 351.2 Rev. 2.0 / E31780	Total Kjeldahl Nitrogen in aqueous matrices	1761360
EPA 353.2 Rev. 2.0 / E31780	Nitrite/Nitrate in aqueous matrices as mg N/L	1761360
EPA 365.1 Rev. 2.0 / E31780	Total Phosphorus in aqueous matrices as mg P/L	1761360
EPA 365.1 Rev. 2.0 dissolved / E31780	Ortho-phosphate, dissolved, in filtered, aqueous matrices as mg P/L	1761361

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
DEP SOP LC-001-2 (based on EPA 8321B)	01/07/2016	01/12/2016	Juyoung Kim	01/12/2016	Juyoung Kim	1761359
EPA 350.1 Rev. 2.0	01/07/2016			01/12/2016	Diana M. Turner	1761360
EPA 351.2 Rev. 2.0	01/07/2016	01/11/2016	Sofia Elnemr	01/12/2016	Bryan A. Werth	1761360
EPA 353.2 Rev. 2.0	01/07/2016			01/11/2016	Diana M. Turner	1761360
EPA 365.1 Rev. 2.0	01/07/2016	01/11/2016	Lipi Saha	01/12/2016	Lipi Saha	1761360
EPA 365.1 Rev. 2.0 dissolved	01/07/2016			01/07/2016 13:20	Julia Storbeck	1761361