

Chemical Analysis Report

WQAP-2017-09-07-03

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

Event Description: **Pinellas LR3**
Request ID: **RQ-2017-09-04-30**
Customer: **WQAP**
Project ID: **SMP**

Send Reports to:
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13051 N. Telecom Parkway
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Certified by: Liang Lin, Environmental Administrator

Date Certified: 29-SEP-2017 14:36

A handwritten signature in black ink, appearing to read "Liang Lin", on a light-colored 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: Alligator Crk

Collection Date/Time: 09/06/2017 11:52

Field ID: G1SW0057

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1928756	EPA 8321B	Sucralose**	0.64		ug/L	P331506	MS
	USGS O-2060-01	2,4-D	0.016		ug/L	P331608	
		Diuron	0.0082		ug/L	P331608	
		Fenuron	0.0080	U	ug/L	P331608	
		Imidacloprid	0.087		ug/L	P331608	
		Bentazon	0.016	J	ug/L	P331608	MS
		Linuron	0.0040	UJ	ug/L	P331608	RPD
		Acetaminophen**	0.0080	U	ug/L	P331608	
		MCP	0.0021	I	ug/L	P331608	
		Carbamazepine**	0.0030		ug/L	P331608	
		Triclopyr**	0.0040	U	ug/L	P331608	
		Primidone**	0.0051	I	ug/L	P331608	
		Fluridone**	0.093		ug/L	P331608	

Ref. Method and Comment:

USGS O-2060-01: Refer to the narrative for an explanation of QC Codes.

USGS O-2060-01: MS accuracy for imidacloprid and fluridone could not be assessed due to high concentration of parameter in the spiked sample. MS precision for fluridone could not be assessed due to it exceeding the instrument calibration range. Refer to the narrative for an explanation of QC Codes.

Sample Location: Bishop Crk

Collection Date/Time: 09/06/2017 10:18

Field ID: G1SW0061

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1928757	EPA 8321B	Sucralose**	0.22		ug/L	P331506	MS
	USGS O-2060-01	2,4-D	0.032		ug/L	P331608	
		Diuron	0.0029	I	ug/L	P331608	
		Fenuron	0.0080	U	ug/L	P331608	
		Imidacloprid	0.081		ug/L	P331608	
		Bentazon	0.021		ug/L	P331608	MS
		Linuron	0.0040	U	ug/L	P331608	RPD
		Acetaminophen**	0.0080	U	ug/L	P331608	
		MCP	0.0020	U	ug/L	P331608	
		Carbamazepine**	0.0010	I	ug/L	P331608	
		Triclopyr**	0.0040	U	ug/L	P331608	
		Primidone**	0.0040	U	ug/L	P331608	
		Fluridone**	1.1		ug/L	P331608	

Ref. Method and Comment:

EPA 8321B: Results confirmed in re-extraction.

USGS O-2060-01: MS accuracy for imidacloprid and fluridone could not be assessed due to high concentration of parameter in the spiked sample. MS precision for fluridone could not be assessed due to it exceeding the instrument calibration range.

Sample Location: Bishop Crk @ Oak

Collection Date/Time: 09/06/2017 10:57

Field ID: G1SW0063

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1928758	EPA 8321B	Sucralose**	0.042		ug/L	P331506	MS
	USGS O-2060-01	2,4-D	0.018		ug/L	P331608	

Field ID: G1SW0063

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
1928758	USGS O-2060-01	Diuron	0.0053		ug/L	P331608	
		Fenuron	0.0080	U	ug/L	P331608	
		Imidacloprid	0.16		ug/L	P331608	
		Bentazon	0.063		ug/L	P331608	MS
		Linuron	0.0040	U	ug/L	P331608	RPD
		Acetaminophen**	0.0085	I	ug/L	P331608	
		MCPP	0.0050	I	ug/L	P331608	
		Carbamazepine**	4.0E-04	U	ug/L	P331608	
		Triclopyr**	0.0040	U	ug/L	P331608	
		Primidone**	0.0040	U	ug/L	P331608	
		Fluridone**	0.0062		ug/L	P331608	

Ref. Method and Comment:
 USGS O-2060-01: Results confirmed in re-extraction.
 USGS O-2060-01: MS accuracy for imidacloprid and fluridone could not be assessed due to high concentration of parameter in the spiked sample. MS precision for fluridone could not be assessed due to it exceeding the instrument calibration range.

Quality Assurance Report Method Blank Results

Reference Method: EPA 8321B
 Batch ID: P331506

Component	Result	Code	Units
Sucralose	0.010	U	ug/L

Reference Method: USGS O-2060-01
 Batch ID: P331608

Component	Result	Code	Units
2,4-D	0.0020	U	ug/L
Acetaminophen	0.0080	U	ug/L
Bentazon	8.0E-04	U	ug/L
Carbamazepine	4.0E-04	U	ug/L
Diuron	8.0E-04	U	ug/L
Fenuron	0.0080	U	ug/L
Fluridone	4.0E-04	U	ug/L
Imidacloprid	8.0E-04	U	ug/L
Linuron	0.0040	U	ug/L
MCPP	0.0020	U	ug/L
Primidone	0.0040	U	ug/L
Triclopyr	0.0040	U	ug/L

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: EPA 8321B
 Batch ID: P331506

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Sucralose	92.2		P	40 - 150

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: USGS O-2060-01
 Batch ID: P331608

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
2,4-D	58.5		P	40 - 150
Acetaminophen	127		P	30 - 150
Bentazon	46.4		P	40 - 150
Carbamazepine	133		P	40 - 150
Diuron	110		P	40 - 150
Fenuron	110		P	40 - 150
Fluridone	111		P	40 - 150
Imidacloprid	118		P	40 - 160
Linuron	96.2		P	40 - 150
MCPP	58.8		P	40 - 150
Primidone	110		P	40 - 150
Triclopyr	61.0		P	40 - 150

Quality Assurance Report Matrix Spike Accuracy

Reference Method: EPA 8321B
 Batch ID: P331506

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1928742	Sucralose	38.7	64.2	F/P	40 - 150

Reference Method: USGS O-2060-01
 Batch ID: P331608

Spiked Sample	Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
1928756	2,4-D	48.3	50.9	P/P	40 - 150
1928756	Acetaminophen	68.6	67.6	P/P	30 - 150
1928756	Bentazon	25.4	24.4	F/F	40 - 150
1928756	Carbamazepine	91.6	93.5	P/P	40 - 150
1928756	Diuron	69.6	75.5	P/P	40 - 150
1928756	Fenuron	91.3	99.7	P/P	40 - 150
1928756	Linuron	59.1	80.0	P/P	40 - 150
1928756	MCPP	50.0	52.0	P/P	40 - 150
1928756	Primidone	96.4	105	P/P	40 - 150
1928756	Triclopyr	40.7	41.9	P/P	40 - 150

Quality Assurance Report Precision

Reference Method: EPA 8321B
 Batch ID: P331506

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1928742	Sucralose	22.0	Spike	P	0 - 30

Quality Assurance Report Precision

Reference Method: USGS O-2060-01
 Batch ID: P331608

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
1928756	2,4-D	3.64	Spike	P	0 - 30
1928756	Acetaminophen	1.53	Spike	P	0 - 30
1928756	Bentazon	1.94	Spike	P	0 - 30
1928756	Carbamazepine	1.92	Spike	P	0 - 30
1928756	Diuron	6.59	Spike	P	0 - 30
1928756	Fenuron	8.73	Spike	P	0 - 30
1928756	Imidacloprid	4.68	Spike	P	0 - 30
1928756	Linuron	30.1	Spike	F	0 - 30
1928756	MCP	3.92	Spike	P	0 - 30
1928756	Primidone	8.88	Spike	P	0 - 30
1928756	Triclopyr	2.90	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: 1928756
 Field Sample ID: G1SW0057

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Acetaminophen-d4	97.4	P	30 - 160
EPA 8321B	Carbamazepine-d10	95.4	P	30 - 160
EPA 8321B	Diuron-d6	71.3	P	30 - 160
EPA 8321B	Primidone-d5	114	P	30 - 160
EPA 8321B	Sucralose-d6	62.7	P	40 - 160
USGS O-2060-01	2,4-D-d3	76.8	P	40 - 160
USGS O-2060-01	Acetaminophen-d4	97.4	P	30 - 160
USGS O-2060-01	Carbamazepine-d10	95.4	P	30 - 160
USGS O-2060-01	Diuron-d6	71.3	P	30 - 160
USGS O-2060-01	Primidone-d5	114	P	30 - 160
USGS O-2060-01	Sucralose-d6	62.7	P	40 - 160

Lab Sample ID: 1928757
 Field Sample ID: G1SW0061

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Acetaminophen-d4	101	P	30 - 160
EPA 8321B	Carbamazepine-d10	101	P	30 - 160
EPA 8321B	Diuron-d6	76.9	P	30 - 160
EPA 8321B	Primidone-d5	123	P	30 - 160
EPA 8321B	Sucralose-d6	54.9	P	40 - 160
EPA 8321B	Sucralose-d6	78.5	P	40 - 160
USGS O-2060-01	2,4-D-d3	79.4	P	40 - 160
USGS O-2060-01	Acetaminophen-d4	101	P	30 - 160
USGS O-2060-01	Carbamazepine-d10	101	P	30 - 160
USGS O-2060-01	Diuron-d6	76.9	P	30 - 160
USGS O-2060-01	Primidone-d5	123	P	30 - 160
USGS O-2060-01	Sucralose-d6	54.9	P	40 - 160

Quality Assurance Report Surrogates

Lab Sample ID: 1928757
 Field Sample ID: G1SW0061

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
USGS O-2060-01	Sucralose-d6	78.5	P	40 - 160

Lab Sample ID: 1928758
 Field Sample ID: G1SW0063

Reference Method	Surrogate	% Rec.	Pass/Fail	Control Limits
EPA 8321B	Acetaminophen-d4	106	P	30 - 160
EPA 8321B	Carbamazepine-d10	98.2	P	30 - 160
EPA 8321B	Diuron-d6	77.0	P	30 - 160
EPA 8321B	Primidone-d5	127	P	30 - 160
EPA 8321B	Sucralose-d6	57.4	P	40 - 160
USGS O-2060-01	2,4-D-d3	79.8	P	40 - 160
USGS O-2060-01	Acetaminophen-d4	106	P	30 - 160
USGS O-2060-01	Carbamazepine-d10	98.2	P	30 - 160
USGS O-2060-01	Diuron-d6	77.0	P	30 - 160
USGS O-2060-01	Primidone-d5	127	P	30 - 160
USGS O-2060-01	Sucralose-d6	57.4	P	40 - 160

Quality Assurance Report Calibration Verification

Reference Method: EPA 8321B
 Run ID: A78939
 Included Lab Sample IDs: 1928756, 1928757, 1928758

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
Sucralose	109	110	P/P	60 - 150

Reference Method: USGS O-2060-01
 Run ID: A79179
 Included Lab Sample IDs: 1928756, 1928757, 1928758

Component	% Rec.1	% Rec.2	Pass/Fail*	Control Limits
2,4-D	104	120	P/P	50 - 150
Acetaminophen	96.5	101	P/P	60 - 150
Bentazon	119	128	P/P	50 - 150
Carbamazepine	112	112	P/P	60 - 150
Diuron	106	103	P/P	60 - 150
Fenuron	101	103	P/P	60 - 150
Fluridone	116	115	P/P	60 - 160
Fluridone	117	116	P/P	60 - 160
Imidacloprid	96.4	99.9	P/P	60 - 150
Linuron	97.0	107	P/P	60 - 150
MCP	107	97.1	P/P	50 - 150
Primidone	95.4	103	P/P	60 - 150
Triclopyr	103	109	P/P	50 - 150

* 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 LCS	SMP	MS
EPA 8321B	Sucralose	92.2	38.7	64.2			22.0
USGS O-2060-01	2,4-D	58.5	48.3	50.9			3.64
	Acetaminophen	127	68.6	67.6			1.53
	Bentazon	46.4	25.4	24.4			1.94
	Carbamazepine	133	91.6	93.5			1.92
	Diuron	110	69.6	75.5			6.59
	Fenuron	110	91.3	99.7			8.73
	Fluridone	111					
	Imidacloprid	118					4.68
	Linuron	96.2	59.1	80.0			30.1
	MCP	58.8	50.0	52.0			3.92
	Primidone	110	96.4	105			8.88
	Triclopyr	61.0	40.7	41.9			2.90

Reference Method Descriptions

Method / DoH Cert #	Description	Associated Samples
EPA 8321B / E31780	Analysis of sucralose in water matrices by HPLC/MS/MS	1928756, 1928757, 1928758
USGS O-2060-01 / E31780	Chlorinated (phenoxy acid) herbicides in water matrices by HPLC/MS/MS	1928756, 1928757, 1928758
USGS O-2060-01 / E31780	Urea herbicides and other chemicals in water matrices by HPLC/MS/MS	1928756, 1928757, 1928758

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
EPA 8321B	09/07/2017	09/13/2017	Hoor Shaik	09/13/2017	Julie Michelle Frank	1928756
	09/07/2017	09/13/2017	Hoor Shaik	09/14/2017	Julie Michelle Frank	1928757, 1928758
USGS O-2060-01	09/07/2017	09/13/2017	Hoor Shaik	09/27/2017	Julie Michelle Frank	1928756, 1928757, 1928758