

Biological Analysis Report

NW-DIST-2023-07-25-01

Florida DEP Laboratory
2600 Blair Stone Road
Tallahassee, FL 32399-2400
DOH Accreditation E31780

Overflow Analyses Performed By:
University of West Florida
11000 University Parkway
Pensacola, FL 32514
DOH Accreditation E71969

Eurofins Environmental Testing, Orlando
481 Newburyport Ave
Altamonte Springs, Florida 32701
DOH Accreditation E83012

Event Description: **FY15 International Paper Company - Pensacola Mill
FL0002526 NWD**
Request ID: **RQ-2023-07-10-53**
Customer: **NW-DIST**
Project ID: **FY15**

Send Reports to:
FL Dept. of Environmental Protection
160 Governmental Center
Suite 302
Pensacola, FL 32501
Attn: Mike Brumfield, Larry Couch, Savanna Harrison

For additional information please contact
Cheryl Swanson - Administrator
Sarah Menz, Ph.D. - Bench Biology & Aquatic
Toxicity
Puja Jasrotia, Ph.D. - Molecular Biology & Taxonomy
Thekkekalathil Chandrasekhar, Ph.D, QA Officer
Phone (850) 245-8177

Certified by: Puja Jasrotia, Environmental Administrator

Date Certified: 01-SEP-2023 12:20



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 groups are included in this report: AGP/LN, Chlorophyll/Grain Size/BOD, Overflow and Toxicology.

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 by the Florida Department of Health Environmental Laboratory Certification Program 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: International Paper Company- Pensacola Mill

Collection Date/Time: 07/24/2023 10:55

Field ID: EFF-3GRAB

Matrix: W-EFFLUENT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2426317	Colilert-18	Fecal Coliform-Quanti-Tray	10		MPN/100 mL		
	SM 9223 B Quanti-Tray	Escherichia Coli-Quanti-Tray	10		MPN/100 mL		
2426318	SM 5210 B	Biochemical Oxygen Demand	4.4		mg/L		
2426631	EPA 600/9-78-018 (mod.)	Algal Growth Potential**	0.538		mg DryWt/L	P433972	
		Algal Growth Potential-Salinity Adjusted**	3.0	U	mg DryWt/L	P433972	
2426653	SM 10150 B	Chlorophyll-a, Corrected	2.1	UYJQ	ug/L	P433241	
		Phaeophytin-a	5.9	IYJQ	ug/L	P433241	
		Chlorophyll-a, Uncorrected	3.0	IYJQ	ug/L	P433241	

Ref. Method and Comment:

EPA 600/9-78-018 (mod.): The AGP value is from day 10 of the test. Test species used, *P. subcapitata*. A salinity adjusted test was also performed. See NCR #9995. AGP samples not effected by unacceptable temperature range at sample receipt.

EPA 600/9-78-018 (mod.): The AGP values are from day 10 of the test. Test species used, *D. tertiolecta*. A fresh water test was also performed. See NCR #9995. AGP samples not effected by unacceptable temperature range at sample receipt.

SM 10150 B: See Non-Conformance Report #'s 10009 and 9999. Precision data is not available for at least one component due to the small amount of analyte in the QC sample. Refer to QA report for available precision data.

Sample Location: International Paper Company- Pensacola Mill

Collection Date/Time: 07/24/2023 10:14

Field ID: EFF-3 Composite

Matrix: W-EFFLUENT

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2426305	EPA-821-R-02-013, Method 1000	Bioassay-Chron-Def-FW-Fish, NOEC	100		NOEC		
		Bioassay-Chron-Def-FW-Fish, IC25	100	L	IC25		
	EPA-821-R-02-013, Method 1002.0	Bioassay-Chron-Def-FW-C.dubia, NOEC	100		NOEC		
		Bioassay-Chron-Def-FW-C.dubia, IC25	100	L	IC25		
2426319	SM 5210 B	Biochemical Oxygen Demand	11		mg/L		

Ref. Method and Comment:

EPA-821-R-02-013, Method 1000: The 25% Inhibition Concentration (IC25) for growth in the *Pimephales promelas* bioassay was >100% effluent. The No Observed Effect Concentration (NOEC) for survival and growth was 100% effluent. See NCR #9995. Toxicity samples arrived at 5.9 and 6.0°C.

EPA-821-R-02-013, Method 1002.0: The 25% Inhibition Concentration (IC25) for reproduction in the *Ceriodaphnia dubia* bioassay was >100% effluent. The No Observed Effect Concentration (NOEC) for survival and reproduction was 100% effluent. See NCR #9995. Toxicity samples arrived at 5.9 and 6.0°C.

Sample Location: IP Control Wetland

Collection Date/Time: 07/24/2023 11:20

Field ID: G5IPCONTROL

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2426509	EPA 600/9-78-018 (mod.)	Algal Growth Potential**	0.300	U	mg DryWt/L	P433972	
2426517	SM 10150 B	Chlorophyll-a, Corrected	13		ug/L	P433241	
		Phaeophytin-a	5.1	I	ug/L	P433241	
		Chlorophyll-a, Uncorrected	17		ug/L	P433241	
2426519	SM 9223 B Quanti-Tray	Escherichia Coli-Quanti-Tray	10	U	MPN/100 mL		
2426521	SM 5210 B	Biochemical Oxygen Demand	2.0	U	mg/L		

Ref. Method and Comment:

EPA 600/9-78-018 (mod.): The AGP value is from day 10 of the test. Test species used, *P. subcapitata*.

SM 10150 B: Precision data is not available for at least one component due to the small amount of analyte in the QC sample. Refer to QA report for available precision data.

Sample Location: G5NWIPTEST

Collection Date/Time: 07/24/2023 09:45

Field ID: G5IPTTEST

Matrix: W-SURF-FRH

Sample ID	Ref. Method	Component	Result	Code	Units	Batch ID	QC Codes
2426510	EPA 600/9-78-018 (mod.)	Algal Growth Potential**	0.300	U	mg DryWt/L	P433972	
2426518	SM 10150 B	Chlorophyll-a, Corrected	3.9	I	ug/L	P433160	
		Phaeophytin-a	1.8	U	ug/L	P433160	
		Chlorophyll-a, Uncorrected	3.6		ug/L	P433160	
2426520	SM 9223 B Quanti-Tray	Escherichia Coli-Quanti-Tray	98		MPN/100 mL		
2426522	SM 5210 B	Biochemical Oxygen Demand	2.0	U	mg/L		

Ref. Method and Comment:

EPA 600/9-78-018 (mod.): The AGP value is from day 10 of the test. Test species used, *P. subcapitata*. The laboratory duplicate result was 0.300 U.
 SM 10150 B: Lab dup result is 3.2 I for Chl-a Corrected, 2.5 I for Chl-a Uncorrected, and 1.8 U for Phaeophytin-a. Precision data is unavailable due to the small amount of analyte in the QC sample.
 SM 5210 B: Within sample results increased with increasing dilutions; concentration may be biased low.

Non-Conformance Report

NCR ID: 9995

<u>Event(s)</u>	<u>Job(s)</u>	<u>Sample(s)</u>	<u>Test(s)</u>
NW-DIST-2023-07-25-01	TLH-2023-07-25-26	2426305	
NW-DIST-2023-07-25-01	TLH-2023-07-26-36	2426631	

NCR Type: SHIPPING/RECEIVING **NCR Category:** Preservation Not Intact

Observation: Samples received outside the acceptable temperature range.

Resolution: Data will be qualified as appropriate.

Authorized by/Date: Kyle Klug 7/27/2023

NCR ID: 9999

<u>Event(s)</u>	<u>Job(s)</u>	<u>Sample(s)</u>	<u>Test(s)</u>
NW-DIST-2023-07-25-01	TLH-2023-07-26-39	2426653	

NCR Type: SHIPPING/RECEIVING **NCR Category:** Samples arrived late

Observation: Samples arrived late, samples received outside the acceptable temperature range.

Resolution: Samples will be qualified as appropriate.

Customer contacted, email correspondence attached to sample submittal forms.

Authorized by/Date: Kyle Klug 7/27/2023

NCR ID: 10009

<u>Event(s)</u>	<u>Job(s)</u>	<u>Sample(s)</u>	<u>Test(s)</u>
NW-DIST-2023-07-25-01	TLH-2023-07-26-39	2426653	CHLSUITE-W

NCR Type: ANALYSIS **NCR Category:** Analytical Measurement

Observation: Sample didnt respond to acidification as expected, values increased instead of decreased.

Resolution: Results will be J-qualified and customers, Mike Brumfield, Larry Couch, and Savanna Harrison, will be notified.

Authorized by/Date: Cheryl A. Swanson 7/31/2023

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: SM 10150 B
Batch ID: P433160

Component	Result	Code	Units
Chlorophyll-a, Corrected	0.82	U	ug/L
Chlorophyll-a, Uncorrected	0.60	U	ug/L
Phaeophytin-a	0.90	U	ug/L

Reference Method: SM 10150 B
Batch ID: P433241

Component	Result	Code	Units
Chlorophyll-a, Corrected	0.82	U	ug/L
Chlorophyll-a, Uncorrected	0.60	U	ug/L
Phaeophytin-a	0.90	U	ug/L

Quality Assurance Report Laboratory Control Sample Accuracy

Reference Method: SM 10150 B
Batch ID: P433160

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Chlorophyll-a, Corrected	87.6		P	84 - 116

Reference Method: SM 10150 B
Batch ID: P433241

Component	% Rec.1	% Rec.2	Pass/Fail	Control Limits
Chlorophyll-a, Corrected	90.0		P	84 - 116

Quality Assurance Report Precision

Reference Method: SM 10150 B
Batch ID: P433241

Replicated Lab Sample	Component	% RSD/RPD	Sample/Spike/LCS*	Pass/Fail	Control Limits
2426749	Chlorophyll-a, Corrected	6.01	Sample	P	0 - 25
2426749	Chlorophyll-a, Uncorrected	8.50	Sample	P	0 - 25

* Sample, spike and/or laboratory control sample precision (LCS) is reported.

Quality Assurance Report Summary

Ref. Method	Analyte	LCS % Recovery	MS % Recovery	LCS	Precision SMP	MS
SM 10150 B	Chlorophyll-a, Corrected	87.6				
	Chlorophyll-a, Corrected	90.0			6.01	
	Chlorophyll-a, Uncorrected				8.50	

Reference Method Descriptions

Method	Description	<u>Associated Samples</u>
Colilert-18	Fecal coliforms (WW only) by Colilert-18/QT by UWF in Pensacola	2426317
EPA-821-R-02-013, Method 1000	Chronic toxicity test, definitive, FW--freshwater fish.	2426305
EPA-821-R-02-013, Method 1000	Chronic toxicity test, definitive, FW--freshwater fish.	2426305
EPA-821-R-02-013, Method 1002.0	Chronic toxicity test, definitive, FW--water flea.	2426305
EPA-821-R-02-013, Method 1002.0	Chronic toxicity test, definitive, FW--water flea.	2426305
EPA 600/9-78-018 (mod.)	Potential algal growth determination-salinity adjusted sample.	2426631
EPA 600/9-78-018 (mod.)	Potential algal growth determination.	2426509, 2426510, 2426631
SM 10150 B	Phytoplankton chlorophyll-a corrected, uncorrected, and phaeophytin by spectrophotometry	2426517, 2426518, 2426653
SM 5210 B	BOD, NOT nitrogen-inhibited at Eurofins Testamerica	2426318, 2426319, 2426521, 2426522
SM 9223 B Quanti-Tray	E. coli by Colilert-18/QT by UWF in Pensacola	2426317, 2426519, 2426520

Preparation and Analysis Log

Ref. Method	Received Date	Prep Date/Time	Prepared By	Analysis Date/Time	Analyzed By	Associated Samples
Colilert-18	07/24/2023	07/24/2023 15:00		07/25/2023 11:49	UWF Lab	2426317
EPA-821-R-02-013, Method 1000	07/25/2023			07/25/2023 12:10	Devon Witczak	2426305
EPA-821-R-02-013, Method 1002.0	07/25/2023			07/25/2023 10:45	Lucas Sasnett	2426305
EPA 600/9-78-018 (mod.)	07/25/2023	07/26/2023 16:30	Lucas Sasnett	07/28/2023 15:00	Lucas Sasnett	2426509, 2426510
	07/26/2023	07/26/2023 16:30	Lucas Sasnett	07/28/2023 15:00	Lucas Sasnett	2426631
SM 10150 B	07/25/2023	07/25/2023 14:25	Keanu Thijm	07/27/2023 11:05	Megan Higbee	2426518
	07/25/2023	07/25/2023 14:25	Keanu Thijm	07/28/2023 10:33	Callie Williams	2426517
	07/26/2023	07/26/2023 13:37	Megan Higbee	07/28/2023 10:33	Callie Williams	2426653
SM 5210 B	07/25/2023			07/25/2023 14:32	Eurofins Orlando	2426318
	07/25/2023			07/25/2023 14:52	Eurofins Orlando	2426319
	07/25/2023			07/25/2023 15:13	Eurofins Orlando	2426522
	07/25/2023			07/25/2023 15:29	Eurofins Orlando	2426521
SM 9223 B Quanti-Tray	07/24/2023	07/24/2023 14:25		07/25/2023 11:18	UWF Lab	2426317