

Effluent Quality Assessment

DuPont North Maxville Expansion

Baker County

NPDES #FL0435490

Sampled March 15, 2010

May 2010
(Resubmitted March 2011 with corrected tables)

Biology Section
Bureau of Laboratories
Division of Environmental Assessment and Restoration

Quality Manual No. 870346G
NELAC Certification No. E31780

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DuPont North Maxville Expansion, US Highway 301 near Macclenny, Baker County, Florida. NPDES #FL0435490. Effluent grab samples for bioassay and chemical analyses for this facility were collected on March 15, 2010.

Introduction

DuPont North Maxville Expansion is a dredge mining facility which uses humate settling ponds to treat industrial wastewater. (See Appendix F for detailed facility information provided by Anthony Vigliotti of FDEP Northeast District.)

Methods

Samples were collected from the EFF-1 NPDES sampling point for Outfall D-001 by Eesa Ali, Brian Kirts, Anthony Vigliotti, and Sharon Piltz (FDEP Northeast District) following DEP-SOP-001/01 FS 2400 Wastewater Sampling. All DEP SOPs are available on the web at: http://www.floridadep.org/labs/qa/sops.htm

See Appendix A for the completed chain of custody form.

The 96-hour acute definitive and acute screening toxicity tests and algal growth potential test were performed following internal DEP SOPs TA07_01, TA07_02, and TA08_01 through TA08_09. All internal DEP SOPs are available on the web at: http://www.floridadep.org/labs/cgi-bin/sop/biosop.asp.

An aliquot of sample was adjusted for pH by the addition of 3.0 mL of 0.1 N sodium hydroxide per liter of sample; increasing the pH from 4.3 SU to 6.9 SU. The pH-adjusted sample was used in the acute definitive bioassays.

The sample was aerated for 10 minutes at a rate of approximately 100 bubbles per minute to decrease the dissolved oxygen (DO) from 9.2 mg/L to 7.5 mg/L.

Results and Discussion

Toxicity Test Results

Tests were performed April 16 - 20, 2010.

Original Sample:

Ceriodaphnia dubia 96-hour acute screening bioassay – LC_{50} < 100%, 100%* mortality in 100% sample at 24 hours

Cyprinella leedsi 96-hour acute screening bioassay – LC_{50} < 100%, 100%* mortality in 100% sample at 24 hours

These bioassay samples were acutely toxic to the test organisms. The cause of toxicity was primarily attributed to the low pH in the 100% sample.

pH-Adjusted Sample:

Ceriodaphnia dubia 96-hour pH-adjusted acute definitive bioassay – LC_{50} > 100%, 0%* mortality in 100% sample at 96 hours

Cyprinella leedsi 96-hour pH-adjusted acute definitive bioassay – LC_{50} > 100%, 10%* mortality in 100% sample at 96 hours

The pH-adjusted bioassay samples were not acutely toxic to the test organisms.

*NOTE: In a 96-hour acute screening or definitive test of 100% effluent, mortality of <20% provides confidence that the effluent meets the acute toxicity criterion of Florida's Surface Water Quality Standards (Rule 62-302.500 (1) (a) 4, F.A.C.). Mortality between 20-50% indicates low to moderate levels of toxicity, and further action may be required. Mortality of >50% indicates that the effluent fails to meet the minimum requirement to discharge to waters of the state (Rule 62-4.244(3) (a), F.A.C.).

See Appendix B for bioassay bench sheets. See Appendix C for results of the last 20 standard reference toxicant (SRT) tests and the bench sheets and statistical analyses of the SRT tests that correspond to these facility tests. See Appendix D for test organism receipts.

Algal Growth Potential

The effluent Algal Growth Potential (AGP) was 3.6 mg dry wt/L of the unicellular green alga, *Pseudokirchneriella subcapitata*, formerly known as *Selenastrum capricornutum*. Raschke and Schultz (1987) found that AGP values above 5.0 mg dry weight/L represent a "problem" threshold for fresh receiving waters, implying nutrient enrichment. The analytical chemistry suggests that the effluent is phosphorus-limited. The predicted AGP based on orthophosphate concentrations was 1.7 (±20%) mg dry wt/L (Table 1; Miller et al. 1978).

The AGP sample was adjusted for pH by the addition of ~ 3.0 mL of 0.1 N sodium hydroxide per liter of sample; increasing the pH from 4.5 SU to 7.1 SU.

See Table 1 for AGP results.

Chemistry Results

Total residual chlorine and total ammonia were not detected in the bioassay sample in the laboratory. The total ammonia concentration in the composite sample collected and preserved for chemical analysis was 0.035 mg N/L. Based on the pH, salinity, and temperature of the effluent as collected, the calculated unionized ammonia concentration was < 0.02 mg/L.

Nickel, total alpha, and radium 226 + 228 were detected in the effluent at levels that comply with Class III fresh water quality criteria (62-302.530, F.A.C.). Cadmium, chromium-III, iron, and lead were detected between the laboratory method detection limits (MDL) and practical quantitation limits (PQL). Zinc (89 µg/L; limit ≤ 38.3 µg/L, 62-302.500 (70), F.A.C.) exceeded the Class III criteria for fresh water.

See Table 2 for results of analytes detected in the effluent, and corresponding limits. See Appendix E for a complete list of chemical analyses performed.

Conclusion

The effluent sample collected from the EFF-1 NPDES sampling point for Outfall D-001 for this facility on March 15, 2010, was acutely toxic to both test species during the 96-hour acute screening bioassays conducted with original sample (effluent at a pH of 4.3 SU). The cause of toxicity was primarily attributed to the low pH in the 100% sample. The 96-hour acute definitive bioassays conducted with pH-adjusted effluent were not acutely toxic to either test species. The effluent AGP result was less than the "problem" threshold for fresh receiving waters. Zinc (89 µg/L; limit ≤ 38.3 µg/L, 62-302.500 (70), F.A.C.) exceeded the Class III criteria for fresh water.

Literature Cited

Raschke, R. L. and D. A. Schultz. 1987. The use of the algal growth potential test for data assessment. J. Wat. Poll. Cont. Fed. 59(4): 222-227.

Miller, W. E., T. E. Maloney, and J. C. Greene. 1978. The *Selenastrum capricornutum* Printz algal assay bottle test. EPA-600/9-78-018. U. S. EPA, Cincinnati, Ohio. 126 p.

Table 1. Measured and predicted algal growth potential (AGP; mg dry weight/L) for orthophosphate (OP) and total phosphorus (TP) limitation of the freshwater species *Pseudokirchneriella subcapitata* and ratios of nitrogen to phosphorus for samples collected from the DuPont North Maxville Expansion on March 15, 2010.

DuPont North Maxville Expansion NPDES# FL0435490					
Location	**AGP (measured)**	**Predicted AGP (OP) ± 20%**	**Predicted AGP (TP) ± 20%**	**Inorganic N:P ratio**	**Total N:P ratio**
D-001	3.6 *	1.7 ± 0.3	4.3 ± 0.9	46.3	62.0

* Sample was pH-adjusted

Table 2. Effluent limits, Class III Freshwater Criteria and chemical data for samples collected from the DuPont North Maxville Expansion on March 15, 2010.

DuPont North Maxville Expansion NPDES# FL0435490	Class III Criteria	Effluent Limits	Effluent	
Organic Constituents (µg/L)				
None Detected	-	-	None Detected	
Metals (µg/L unless otherwise noted)				
Aluminum	-	-	4420	
Arsenic	≤ 50	-	0.25	U
Cadmium	≤ 0.1 b	-	0.047	I
Calcium (mg/L)	-	-	6.88	
Chromium-III	≤ 28.6 b	-	0.76	I
Copper	≤ 3.0 b	-	0.5	U
Iron	≤ 1,000	≤ 1,000 s	98	I
Lead	≤ 0.6 b	-	0.38	I
Magnesium (mg/L)	-	-	2.15	
Mercury	≤ 0.012	-	0.12	U
Nickel	≤ 16.7 b	-	1.75	
Selenium	≤ 5.0	-	0.2	U
Silver	≤ 0.07	-	0.025	U
Zinc	≤ 38.3 b	-	89	
Nutrients (mg/L)				
Ortho-phosphate	-	-	0.004	U
Total Phosphorus	-	-	0.01	I
Total Ammonia	-	-	0.035	
Un-ionized Ammonia	≤ 0.02 s	-	≤ 0.02	c
Nitrate and Nitrite	-	-	0.15	
Total Kjeldahl Nitrogen	-	-	0.47	
Organic Nitrogen	-	-	0.435	c
Total Nitrogen	-	-	0.62	c
General Physical and Chemical Parameters				
Dissolved Oxygen (mg/L)	≥ 5.0	-	9.1	
pH (SU)	6.0 - 8.5	Report	4.3	
Conductivity (µmhos/cm)	≤ 1,275	-	264	
Temperature (°C)	-	-	17.0	
Salinity	-	-	≤ 0.15	c
Total Residual Chlorine (mg/L)	≤ 0.01	-	0.03	UQ£
Chlorophyll *a* (µg/L) - Corrected	-	-	6.8	
Phaeophytin (µg/L)	-	-	0.4	U
Total Suspended Solids (mg/L)	-	≤ 30.0 s	6	I
Turbidity (NTU)	< 29 t	Report	3.6	*
Alpha, Total (pCi/L)	≤ 15	≤ 15.0 r	3.6	
Alpha-Counting Error (pCi/L)	-	-	1.3	
Radium 226 (pCi/L)	-	-	1.3	
Radium 226-Counting Error (pCi/L)	-	-	0.2	
Radium 228 (pCi/L)	-	-	2.5	
Radium 228-Counting Error (pCi/L)	-	-	0.7	
Radium 226 + 228 (pCi/L)	≤ 5	≤ 5.0 r	3.8	c
Flow (MGD)	-	≤ 5.0 a	0.394	d
Hardness (mg $CaCO_3$)	-	-	26.03	c

Value exceeds the Class III Water Quality Criteria or permit limits

a - Annual average
b - Value is calculated based on hardness
c - Value is calculated
d - Average daily flow from May-Sep. '09
r - Rolling 12-month average
s - Single sample
t - Shall not exceed 29 NTUs above background
A - Value reported is the mean of two or more determinations
I - The reported value is between the laboratory method detection limit and the laboratory practical quantitation limit
Q - Sample held beyond normal holding time
U - Material analyzed for but not detected; value reported is the method detection limit
£ - measurement taken in the FDEP laboratory
* - Background turbidity measurment 1.9 A (NTU)

Florida Department of Environmental Protection

Central Laboratory Sample Submittal Form

Request Number: RQ-2010-03-15-30
Dupont North Maxville Expansion

Event ID *

Requester: Michael Tanski

Field Report Prepared By: S. Piltz

Customer: NE-DIST

Collected By: E.Ali/B.Kicts/A.Vigliotti/S.Piltz

Send Final Report To Tom Kallemeyn

Project ID: FYI3

Sampling Agency: DEP NED

PMAS: 1143

Lab ID *	Location / Field ID	Matrix (Include type e.g. Salt, Fresh, etc)	Temp (C)	pH	Latitude	Longitude	Collection (begin) Eastern Central	Collection (end) Eastern Central	Tot Res Chlorine (mg/L)	Diss Oxygen (mg/L)	Sample Depth	Salinity (PPTh)	Sp Conductance (umho/cm)	NPDES Number	Storet Station Number	Comments	Bottle Group(s)**
	Dupont N. Maxville Exp. Background	Fresh	13.56	4.17	o ' "	o ' "	☐ Comp ☑ Grab Date 0930 Time 3/15/10 Central	Date Time Central		6.99	0.1 m ☐ ft	004	86				B
	Dupont N. Maxville Exp D001 Final Eff	Eff	16.96	4.31	o ' "	o ' "	☐ Comp ☑ Grab Date 3/15/10 Time 1010 Central	Date Time Central		9.13	0.2 m ☐ ft	0.13	264				A+B
	Blank	DI			o ' "	o ' "	☐ Comp ☑ Grab Date 3/15/10 Time 1020 Central	Date Time Central			☐ m ☐ ft	☐ Salinity (PPTh) ☐ Sp Conductance (umho/cm)					C
					o ' "	o ' "	☐ Comp ☐ Grab Date Time Eastern Central	Date Time Central			☐ m ☐ ft	☐ Salinity (PPTh) ☐ Sp Conductance (umho/cm)				3 bio assay Reid in Tox by JD 9:55 3/16/10	

Relinquished By:	Date/Time	Shipping Method:	Received By:	Date/Time	Relinquished By:	Date/Time	Received By:	Date/Time
	3/15/10	Fed Ex					SL	3.16.10 9:15

* Shaded Areas for Lab use only.

** Please see reverse side for Bottle Group information.

last revised October 1, 2003

FDEP Biology Laboratory - Acute Bioassay Bench Sheets

Facility: DuPont N. Maxville mine

Address: US 301 near Macclenny, FL

City: Macclenny **County:** Baker

Contact/District: Anthony Vigliotti / NED

NPDES Permit #: FL 0435490

LIMS Job #: TLH-2010-03-16-38 **LIMS Sample #:** 1257194

Lims Data Entry: 3-24-10 MF **Data Entry Verification:** BA 3-24-10

Sample Collection: Date: 3-15-10 Time: 10:10

Hold Time Start: Date: 3-15-10 Time: 10:10

Comments: 100% sample was pH adjusted for the definitive test. the screeny test was original (no pH-adjustment) sample, MF 3/24/10

Initial sample handling:

Instructions: Check yes or no. If yes is checked, complete blanks

PH adjustment: ☑ yes ☐ no Initial pH: 4.3 NaOH 0.1 N HCl ____ N ~30 Drops (ml) Final pH: 6.7

Aeration: ☑ yes ☐ no Initial DO: 9.2 mg/L Final DO: 7.5 mg/L Duration: 10 minutes Rate: ~ 100 bubbles/min

Salinity adjusted (Test 1): ☐ yes ☑ no Initial Salinity: ____ Final Salinity: ____ Salts Hypersaline brine

Salinity adjusted (Test 2): ☐ yes ☒ no Initial Salinity: ____ Final Salinity: ____ Salts Hypersaline brine

Dechlorination: ☐ yes ☑ no ____ mL of 0.025N Sodium Thiosulfate per liter of sample. Final TRC: ____

Sample Validation:

Instructions: Check yes or no. Fill in temperatures.

Temperature: Shipped ≤ 6°C ☑ Yes ☐ No — IF Hand Delivered: Cooling ☐ Yes ☐ No (received ____ °C collected ____ °C)

Holding Time: ≤ 36 Hours ☑ Yes ☐ No (Composite-end of collection; grab-when collected; 4 in 24 - time last sample collected)

Investigators' Signatures:	Initials:
Josh Woods	JW
Marshall Faircloth	MF
Brian Alexander	BA
James Daniels	JD
REVIEWER: Elizabeth Miller	

Diluent/Control Batch#:	3-8-10	3-15-10							
Water Quality Parameters	20% DMW	Well Water	Moderately Hard Water	Salt Water ASW NSW Test 1	Salt Water ASW NSW Test 2	Other:	Original Sample	Measured by	Verified by
Field Total Residual Cl_2 (mg/L):	N/A	N/A	N/A	N/A	N/A	N/A	not measured		
Lab Total Residual Cl_2 (mg/L):	<0.03	<0.03					<0.03	BA	MF
Alkalinity (mg/L as $CaCO_3$):	78	171					26	BA	MF
Hardness (mg/L as $CaCO_3$):	81	140					49	BA	MF
Total Ammonia (mg/L as N):	<0.07	<0.07					<0.07	BA / MF	MF / BA
Conductivity: ☑ µmhos ☐ mmhos	189	323					331	BA/FW/JD	MF
Salinity (ppth):	—	—					0.0	JD	BA

V 1.8 1/13/10

Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Survival Bench Sheet

LIMS Sample # 1257194 SOP#: TA 07 01 Test # 1 of 2

Test Species: BA
Batch #: 6-10 Age: <24hrs
- [x] *Ceriodaphnia dubia*
- [ ] *Cyprinella leedsi*
- [ ] *Pimephales promelas*
- [ ] *Menidia beryllina*
- [ ] *Americamysis bahia*
- [ ] Other:

Test Validation
Control Survival ≥ 90%: [x] Yes [ ] No
Temperature Range ≤ 3° C: [x] Yes [ ] No

Diluent (Batch # 3-8-10C)
[ ] ASW [x] 20% DMW
[ ] MHW [ ] Well

Test Type: [x] Acute [x] Definitive
[ ] Static [x] Static Renewal [ ] Flow-Through

Test Duration:
[ ] 48 hr
[x] 96 hr
[ ] ___hr

Test Started: Date 3-16-10 Time 13:10

Test Ended: Date 3-20-10 Time 0900

Chamber Size 30 ml
Test Volume: 20 ml

Concentration	Replicate	Chamber #	Test Hour 0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
Ctrl	A	A1	5	5	5	5	5	5
	B	B1	5	5	5	5	5	5
	C	C1	5	5	5	5	5	5
	D	D1	5	5	5	5	5	5
6.25	A	A2	5	5	5	5	5	5
	B	B2	5	5	5	5	5	5
	C	C2	5	5	5	5	5	5
	D	D2	5	5	5	5	5	5
12.5	A	A3	5	5	5	5	5	5
	B	B3	5	5	5	5	5	5
	C	C3	5	5	5	5	5	5
	D	D3	5	5	5	5	5	5
25	A	A4	5	5	5	5	5	5
	B	B4	5	5	5	5	5	5
	C	C4	5	5	5	5	5	5
	D	D4	5	5	5	5	5	5
50	A	A5	5	5	5	5	5	5
	B	B5	5	5	4 ID	4	4	4
	C	C5	5	5	5	5	4 ID	4
	D	D5	5	5	4 ID	4	4	4
100	A	A6	5	0 SD	-	-	-	-
	B	B6	5	0 SD	-	-	-	-
	C	C6	5	0 SD	-	-	-	-
	D	D6	5	0 SD	-	-	-	-
pH adj. 100%	A	A7	5	5	5	5	5	5
	B	B7	5	5	5	5	5	5
	C	C7	5	5	5	5	5	5
	D	D7	5	5	5	5	5	5
Checked or Verified by:			BA	BA	BA	BA	BA	MF

Food
- [x] YCT Batch# 10-30-09
- [x] P. subcapitata Batch# 11-9-09
- [ ] Artemia Lot#_____

Feeding and Artemia Batch #
- [x] 0 hr -- [x] Prior to Test
- [ ] 24 hr -
- [x] 48 hr - [x] Prior to Renewal
- [ ] 72 hr -

Temperature Range (°C)
Incubator # 2
min. 25.5 max. 27.6 mean 26.6
Incubator #___
min.___max.___mean___
Waterbath #___
min.___max.___mean___
Room B246
min. 24.2 max. 25.3 mean 24.7
Light Intensity (ft. candles): 50-100
Photoperiod (hours): 16 light, 8 dark

Loaded/ Verified by: [illegible] / SP
m = missing, d = dead, BR/AR Before/ After Renewal

Test Results:
Screening Tests: Report LC50 as >100%, =100%, or <100%.
% mortality in 100% sample: 100% LC50: <100%
Calculated LC50 (Definitive) 95% CI_____ Method_____

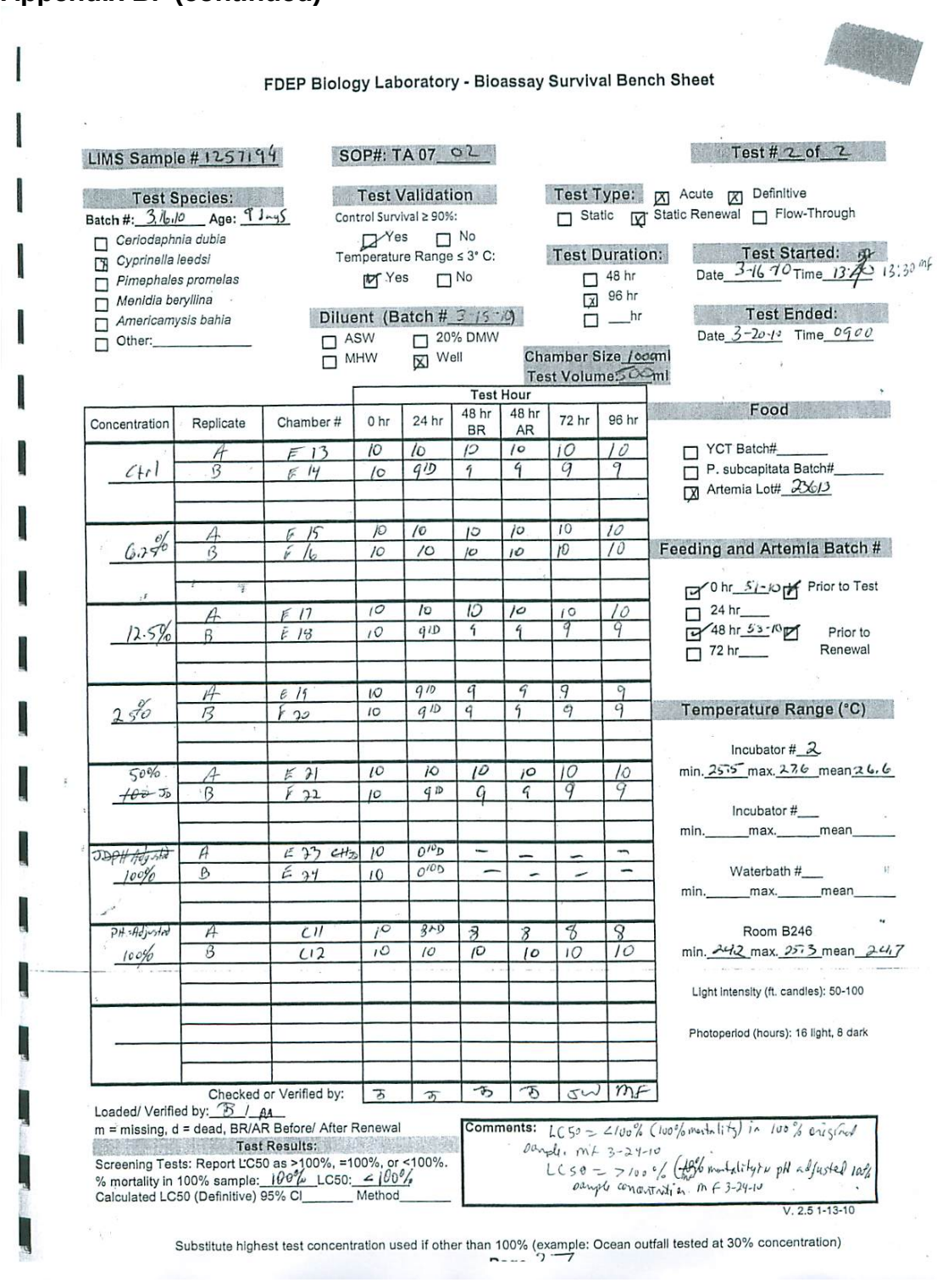
Comments: original Sample Screening Test LC50 = <100% 100% mortality, MF 3-24-10
pH adjusted sample 0% mortality in 100% concentration LC50 = >100% MF 3-24-10

V. 2.5 1-13-10

Pg 26

Substitute highest test concentration used if other than 100% (example: Ocean outfall tested at 30% concentration)

Appendix B. (continued)



FDEP Biology Laboratory - Bioassay Survival Bench Sheet

LIMS Sample # 1257194 | SOP#: TA 07 02 | Test # 2 of 2

Test Species: Batch #: 3.16.10 Age: 9 1-4 days
- ☐ Ceriodaphnia dubia
- ☒ Cyprinella leedsi
- ☐ Pimephales promelas
- ☐ Menidia beryllina
- ☐ Americamysis bahia
- ☐ Other:_____

Test Validation
Control Survival ≥ 90%: ☑ Yes ☐ No
Temperature Range ≤ 3° C: ☑ Yes ☐ No

Diluent (Batch # 3.15.10)
☐ ASW ☐ 20% DMW ☐ MHW ☒ Well

Test Type: ☒ Acute ☒ Definitive ☐ Static ☒ Static Renewal ☐ Flow-Through

Test Duration: ☐ 48 hr ☒ 96 hr ☐ ___hr

Test Started: Date 3-16-10 Time 13:40 13:30 MF

Test Ended: Date 3-20-10 Time 0900

Chamber Size 100ml
Test Volume 500ml

Concentration	Replicate	Chamber #	Test Hour 0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
Ctrl	A	E 13	10	10	10	10	10	10
	B	E 14	10	9 ID	9	9	9	9
6.25%	A	E 15	10	10	10	10	10	10
	B	E 16	10	10	10	10	10	10
12.5%	A	E 17	10	10	10	10	10	10
	B	E 18	10	9 ID	9	9	9	9
25%	A	E 19	10	9 ID	9	9	9	9
	B	F 20	10	9 ID	9	9	9	9
50%	A	E 21	10	10	10	10	10	10
~~100%~~ 50	B	F 22	10	9 ID	9	9	9	9
DDPH Adjusted 100%	A	E 23 CH2	10	0 ID	—	—	—	—
	B	E 24	10	0 ID	—	—	—	—
PH-Adjusted 100%	A	C11	10	8 AD	8	8	8	8
	B	C12	10	10	10	10	10	10
Checked or Verified by:			B	B	B	B	JW	MF

Loaded/ Verified by: B / AA
m = missing, d = dead, BR/AR Before/ After Renewal

Test Results:
Screening Tests: Report LC50 as >100%, =100%, or <100%.
% mortality in 100% sample: 100% LC50: < 100%
Calculated LC50 (Definitive) 95% CI_____ Method_____

Food
☐ YCT Batch#_____
☐ P. subcapitata Batch#_____
☒ Artemia Lot# 2X13

Feeding and Artemia Batch #
☑ 0 hr 3-19-10 ☑ Prior to Test
☐ 24 hr____
☑ 48 hr 3-18-10 ☑ Prior to Renewal
☐ 72 hr____

Temperature Range (°C)
Incubator # 2
min. 25.5 max. 27.6 mean 26.6
Incubator #___
min.____max.____mean____
Waterbath #___
min.____max.____mean____
Room B246
min. 24.2 max. 25.3 mean 24.7
Light Intensity (ft. candles): 50-100
Photoperiod (hours): 16 light, 8 dark

Comments: LC50 = <100% (100% mortality) in 100% original sample. MF 3-24-10
LC50 = >100% (10% mortality in pH adjusted 100% sample concentration. MF 3-24-10

V. 2.5 1-13-10

Substitute highest test concentration used if other than 100% (example: Ocean outfall tested at 30% concentration)

Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Parameter Sheet

LIMS Sample # 125 7194 Test # 1 of 2

Test Species:

☒ *Ceriodaphnia dubia* ☐ *Pimephales promelas* ☐ *Americamysis bahia*
☐ *Cyprinella leedsi* ☐ *Menidia beryllina* ☐ Other:_____

Test aerated: (if DO is < 4.0 mg/L) ☐ Yes ☑ No

Concentration	Replicate	Test Hour	pH (SU)	Temperature (°C)	Dissolved Oxygen (mg/L)	Conductivity ☐ µmhos ☑ mmhos	Salinity* (ppth)
	A	0 hr	7.6	24.0	7.1	0.210	~~0.01~~ JW
	—	24 hr	—	—	—	~~0.188~~ BA	—
CTRL	M	48 hr BR	7.7	24.0	7.4	0.189	—
	M	48 hr AR	7.7	24.0	6.7	0.207	
		72 hr					
	A	96 hr	8.1	25.1	8.0	0.207	
	A	0 hr	~~7.7~~ 8.0 JW	24.0	7.2	0.193	~~0.01~~ JW
	—	24 hr	—	—	—	—	—
6.25	M	48 hr BR	7.8	24.0	7.4	0.201	—
	M	48 hr AR	8.0	24.0	6.9	0.233	
		72 hr					
	A	96 hr	8.1 JW	24.9	8.0	0.214	
	A	0 hr 7.1	~~8.0~~ JW	24.0	7.3	0.198	—
	—	24 hr	—	—	—	—	—
12.5	M	48 hr BR	7.8	24.0	7.5	0.203	—
	M	48 hr AR	8.0	24.0	7.1	0.229	
		72 hr					
	A	96 hr	8.1	24.5	7.9	0.220	
	A	0 hr	7.9	24.0	7.3	0.189	—
	—	24 hr	—	—	—	—	—
25	M	48 hr BR	7.7	24.0	7.5	0.208	—
	M	48 hr AR	8.0	24.0	7.2	0.230	
		72 hr	~~7.9~~ 8.0 BA				
	A	96 hr	8.1 JW	24.6	7.9	0.216	
	A	0 hr 7.3	~~7.7~~ JW	24.0	7.4	0.213	—
	—	24 hr	—	—	—	—	—
50	M	48 hr BR	7.7	24.0	7.5	0.219	—
	M	48 hr AR	7.9	24.0	7.3	0.258	
		72 hr					
	A	96 hr	8.0	24.4	7.9		
	A	0 hr	4.4	~~24.0~~	7.5	0.284	—
	—	24 hr *	—	—	—	—	—
100%	—	48 hr BR	—	—	—	—	—
	—	48 hr AR	—	—	—	—	—
		72 hr					
	~~MF~~	96 hr					
pH adjusted 100%	A	0 hr	6.9	24.0	8.2	0.352	—
	—	24 hr	—	—	—	—	—
	M	48 hr BR	7.5	24.0	7.8	0.346	—
	M	48 hr AR	7.8	24.0	7.4	0.364	
		72 hr					
	A	96 hr	7.9	24.3	7.9	MF 0.339	
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					

*Measure/ record salinity for marine tests

Comments: * 24 hour Aeration initially not measured. MF for TD 3-8-2010

Measured By:
0 hr: JW
24 hr: —
48 hr BR: BA
48 hr AR: BA
72 hr: —
96 hr: MF

Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Parameter Sheet

LIMS Sample # 1257194 Test # 2 of 2

Test Species:

☐ *Ceriodaphnia dubia* ☐ *Pimephales promelas* ☐ *Americamysis bahia*
☒ *Cyprinella leedsi* ☐ *Menidia beryllina* ☐ Other:_____

Test aerated: (if DO is < 4.0 mg/L) ☐ Yes ☑ No

Concentration	Replicate	Test Hour	pH (SU)	Temperature (°C)	Dissolved Oxygen (mg/L)	Conductivity ☐ µmhos ☑ mmhos	Salinity* (ppth)
Ctrl	A	0 hr	8.4	24.0	7.0	0.324	—
	B	24 hr	8.1	24.0	7.3	0.323	—
	A	48 hr BR	7.8	25.0	6.9	0.324	—
	A	48 hr AR	8.0	24.0	7.3	0.320	—
	B	72 hr	8.2	25.7	7.3	0.300	
	A	96 hr	8.2	25.5	7.2		
6.25	A	0 hr	8.0	24.0	6.9	0.301	—
	B	24 hr	8.2	24.0	7.4	0.290	—
	A	48 hr BR	8.3	25.5	6.9	0.361	—
	A	48 hr AR	8.0	24.1	7.4	0.360	—
	B	72 hr	8.2	25.7	7.0	0.291	
	A	96 hr	8.2	25.5	7.9	0.305	
12.5	A	0 hr	8.0	24.0	7.0	0.289	—
	B	24 hr	8.2	24.0	7.3	0.285	—
	A	48 hr BR	8.2	24.7	7.2	0.346	—
	A	48 hr AR	8.0	24.1	7.4	0.346	—
	B	72 hr	8.2	25.7	7.0	0.289	
	A	96 hr	8.2	25.1	7.9	0.298	
25	A	0 hr	7.9	24.1	7.2	0.277	—
	B	24 hr	8.0	24.0	7.4	0.278	—
	A	48 hr BR	8.1	25.5	7.1	0.346	—
	A	48 hr AR	7.8	24.2	7.5	0.343	—
	B	72 hr	8.1	25.8	7.0	0.284	
	A	96 hr	8.0	25.3	7.9	0.293	
50	A	0 hr	7.6	24.2	7.2	0.279	—
	B	24 hr	7.8	24.0	7.4	0.284	—
	A	48 hr BR	8.0	25.6	7.3	0.347	—
	A	48 hr AR	7.7	24.5	7.5	0.343	—
	B	72 hr	7.9	25.6	7.1	0.270	
	A	96 hr	7.8	26.0	7.9	0.285	
100%	A	0 hr	4.5	24.3	7.2	0.296	—
	B	24 hr	4.8	24.0	7.5	0.270	—
		48 hr BR					
		48 hr AR					
		72 hr	—	—	—	—	—
		96 hr					
pH Adjusted 100%	A	0 hr	6.9	24.0	8.3	0.352	—
	B	24 hr	6.9	24.0	7.6	0.347	—
	A	48 hr BR	7.6	25.9	7.3	0.434	—
	A	48 hr AR	7.5	24.0	7.6	0.427	—
	B	72 hr	7.7	25.7	7.0	0.358	
	A	96 hr	7.8	25.8	7.9	~~0.385~~ 0.385 mf	
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					

*Measure/ record salinity for marine tests

Comments:

Measured By:
0 hr: JW
24 hr: JS
48 hr BR: JW
48 hr AR: JW
72 hr: BA
96 hr: MF

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V. 2.3 1-13-10

Bioassay Instrument ID

Test ID: Dupont Mine Dates: 3-16-10 · 3-20-10

METER / DEVICE	ID	SOP	Parameter	Method	v used
YSI Model 58 Dissolved Oxygen Meter	90H018262	TA06_04	Dissolved Oxygen	SM 4500 O G	✓
Accumet AR-10 pH Meter	93312105	TA06_23	pH	SM 4500 H+	✓
Mettler-Toledo SevenMulti Conductivity Meter	SN 1225117049 Probe H04257	TA06_25	Conductivity	SM 2510 B	✓
			Salinity	SM 2520 B	—
Denver Model 225 ISE with ammonia probe	SN K01377	TA06_17	Ammonia	SM 4500 NH3 F	✓
Thermometer (DO probe)	YSI 5750 SN 05J1720	TA06_05	Temperature	SM-2530B modified DEP SOP-001/01 FT1400	✓
Hach Pocket Colorimeter II	05050C026378	TA06_22	Total Residual Chlorine	EPA 330.5 SM 4500 CL G	✓
Alkalinity (Hach Kit)	No SN	TA06_08	Alkalinity	SM 2320 B	✓
Hardness (Hach Kit)	No SN	TA06_09	Hardness	SM 2340 C	✓
OHAUS Balance 250D	1125481084	TA06_21 TA06_27	Weight	-	
S-Weights	BIO-S-WEIGHTS-01	NA	Balance check	-	

SM-2530B modified to allow use of electronic and non-mercury filled thermometers.

Insert other device information in blank spaces(if used). Test ID = Facility or SRTs (acute SRT–Chronic SRT)

BA on 3-24-10

V 1.1 6/12/08 MF

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Appendix C. SRT Bench Sheets and Statistical Analysis

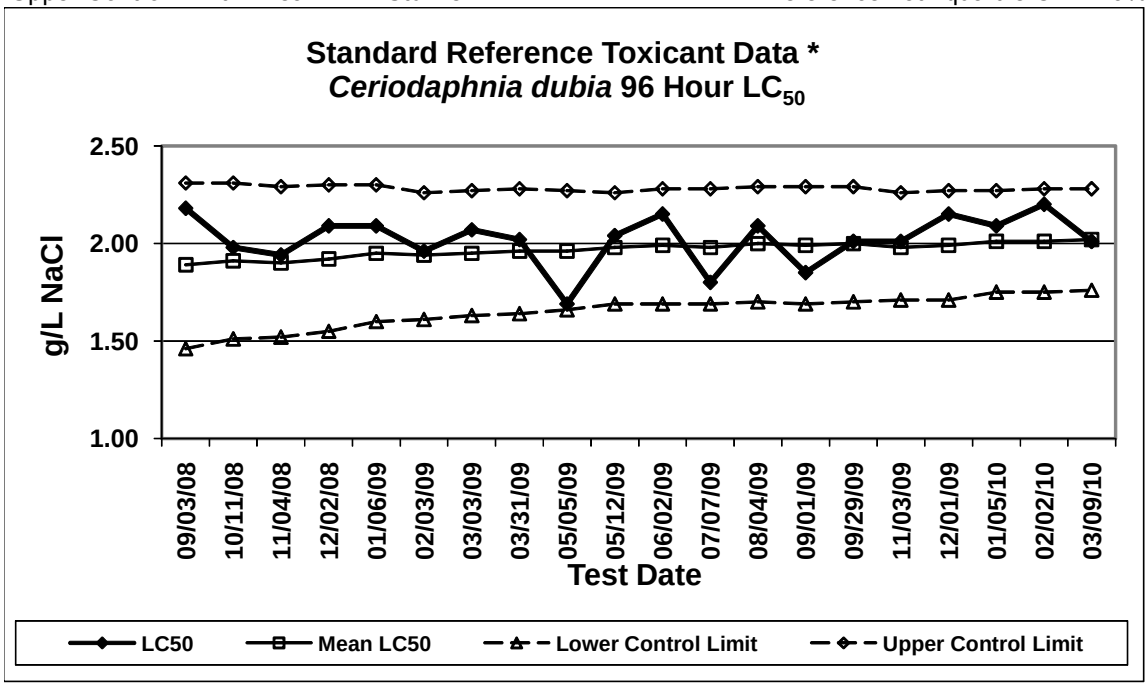
Florida Department of Environmental Protection
Bureau of Laboratories

Standard Reference Toxicant (SRT) Test Data - 96-hour Acute Toxicity
Ceriodaphnia dubia (water flea) - Sodium Chloride (NaCl)

Date	LC50	Mean LC50	Std Dev	Lower Control Limit	Upper Control Limit
09/03/08	2.18	1.89	0.21	1.46	2.31
10/11/08	1.98	1.91	0.20	1.51	2.31
11/04/08	1.94	1.90	0.19	1.52	2.29
12/02/08	2.09	1.92	0.19	1.55	2.30
01/06/09	2.09	1.95	0.17	1.60	2.30
02/03/09	1.96	1.94	0.16	1.61	2.26
03/03/09	2.07	1.95	0.16	1.63	2.27
03/31/09	2.02	1.96	0.16	1.64	2.28
05/05/09	1.69	1.96	0.15	1.66	2.27
05/12/09	2.04	1.98	0.14	1.69	2.26
06/02/09	2.15	1.99	0.15	1.69	2.28
07/07/09	1.80	1.98	0.15	1.69	2.28
08/04/09	2.09	2.00	0.15	1.70	2.29
09/01/09	1.85	1.99	0.15	1.69	2.29
09/29/09	2.01	2.00	0.15	1.70	2.29
11/03/09	2.01	1.98	0.14	1.71	2.26
12/01/09	2.15	1.99	0.14	1.71	2.27
01/05/10	2.09	2.01	0.13	1.75	2.27
02/02/10	2.20	2.01	0.13	1.75	2.28
03/09/10	2.01	2.02	0.13	1.76	2.28

Lower Control Limit = mean - 2 * Std Dev
Upper Control Limit = mean + 2 * Std Dev

CV = 6.34
EPA reference 75th quartile CV = 29%



Appendix C. (continued)

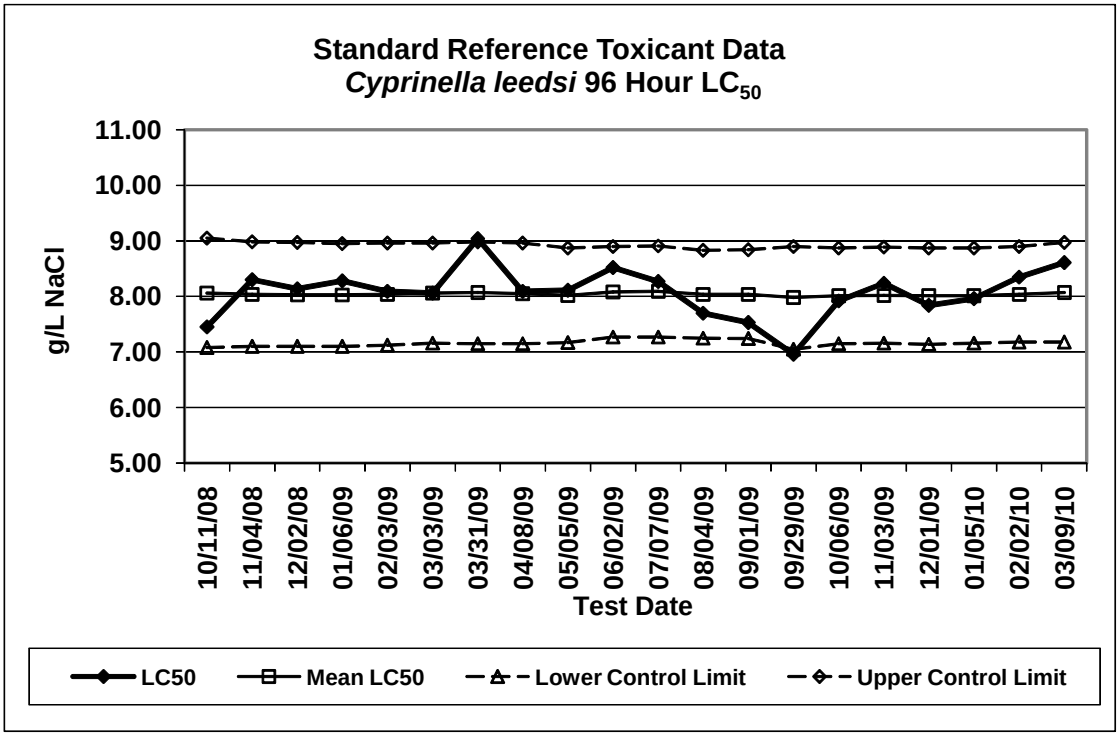
Florida Department of Environmental Protection
Bureau of Laboratories

Standard Reference Toxicant (SRT) Test Data - 96-hour Acute Toxicity
Cyprinella leedsi (bannerfin shiner) - Sodium Chloride (NaCl)

Date	LC50	Mean LC50	Std Dev	Lower Control Limit	Upper Control Limit
10/11/08	7.45	8.06	0.49	7.08	9.05
11/04/08	8.30	8.04	0.47	7.10	8.98
12/02/08	8.14	8.03	0.47	7.10	8.97
01/06/09	8.28	8.03	0.46	7.10	8.95
02/03/09	8.09	8.04	0.46	7.12	8.96
03/03/09	8.06	8.06	0.45	7.16	8.96
03/31/09	9.04	8.07	0.46	7.15	8.98
04/08/09	8.09	8.05	0.45	7.15	8.96
05/05/09	8.11	8.02	0.43	7.17	8.87
06/02/09	8.52	8.08	0.41	7.27	8.90
07/07/09	8.27	8.09	0.41	7.27	8.91
08/04/09	7.70	8.04	0.40	7.25	8.83
09/01/09	7.53	8.04	0.40	7.24	8.84
09/29/09	6.96	7.98	0.46	7.05	8.90
10/06/09	7.92	8.01	0.43	7.15	8.87
11/03/09	8.24	8.02	0.43	7.16	8.89
12/01/09	7.84	8.01	0.43	7.14	8.87
01/05/10	7.96	8.01	0.43	7.16	8.87
02/02/10	8.35	8.04	0.43	7.18	8.90
03/09/10	8.61	8.07	0.45	7.18	8.97

Lower Confidence Limit = mean - 2 * Std Dev
Upper Confidence Limit = mean + 2 * Std Dev

CV = 5.55
EPA reference 75th quartile CV = 19%



Appendix C. (continued)

FDEP Biology Laboratory - Acute SRT Bench Sheet

Method from EPA-821-R-02-012 except *H. azteca*

Test Organism: (check one)
☐ *Americamysis bahia* Method 2007.0 TA07.03 ☐ *Menidia Beryllina* Method 2006.0 TA07.04 ☐ *Hyalella azteca* Method 100.1 EPA-600-R-99-064
☒ *Ceriodaphnia dubia* Method 2002.0 TA07.01 ☐ *Cyprinella leedsi* Method 2000.0 TA07.02 ☐ *Pimephales promelas* Method 2000.0 TA07.02

Test Type: ☐ non-renewal ☒ renewal
Test Page 1 of 2

Diluent/ Batch #: DMW / 3-1-10
Toxicant/ Batch#: NaCl / 13-7-10
Organism Batch: 5-10 Age: <24hr
Test Beginning: Date: 3-9-10 Time: 12:40
Test Ending: Date: 3-13-10 Time: 0900

Water Quality Parameters:	Diluent	SOP	Initials
Total Residual Cl_2 (mg/L):	<0.03	TA06_22	BA
Alkalinity (mg/L as $CaCO_3$):	78	TA06_08	CF
Hardness (mg.L as $CaCO_3$):	38835	TA06_09	CF
Total Ammonia (mg/L as N):	<0.017	TA06_17	MF
Conductivity (mmhos) (mmhos):	0.171	TA06_25	BA
(A) Salinity (ppth):	—	TA06_25	—

Food:	Batch
☒ YCT	10-30-09
☒ *P. subcapitata*	11-9-09
☐ Artemia Lot #	

Artemia Batch:
0 hr:
24 hr:
48 hr:
72 hr:

Chamber size: 30 mL
Test volume: 20 mL per replicate

Concentrations prepared by: BA
Test Incubated in: Incubator # 2 Waterbath # ____
Light Intensity: 50-100 Ft. candles
Photoperiod: 16 hours Light / 8 hours dark

Temperature Range °C:(if used)
Incubator # 2 min. 26.5 max. 27.9 mean 27.2
Waterbath # ___ min. ____ max. ____ mean ____
Room B246 min. 25.2 max. 26.0 mean 25.6

Feeding: √as needed
☑ Before test
☐ 0 hr
☐ 24 hr
☑ 48 hr
☐ 72 hr

all species 2 hrs prior to test
mysids daily
all species except *H. azteca* 2 hr prior to renewal
H. azteca 0 hr and 48 hr

Concentration µg/L mg/L (A)	Replicate	Chamber #	Test Hour 0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
Control	A	A	5	5	5	5	5	5
	B	B	5	5	5	5	5	5
	C	C	5	5	5	5	5	5
	D	D	5	5	5	5	5	5
1.5	A	A	5	5	5	5	5	5
	B	B	5	5	5	5	5	5
	C	C	5	5	5	5	4 1D	4
	D	D	5	5	5	5	5	5
2.0	A	A	5	5	5	5	5	4 1D
	B	B	5	5	5	5	5	4 1D
	C	C	5	5	2 3D	2	1 1D	1
	D	D	5	5	4 1D	4	4	3 1D
2.5	A	A	5	5	0 4D	0	—	—
	B	B	5	5	1 4D	1	1	0 1D
	C	C	5	4 1D	1 3D	1	0 1D	—
	D	D	5	5	0 5D	0	—	—
3.0	A	A	5	4 1D	0 4D	—	—	—
	B	B	5	5	0 5D	—	—	—
	C	C	5	2 3D	0 4D	—	—	—
	D	D	5	3 2D	0 3D	—	—	—
4.0	A	A	5	0 5D	—	—	—	—
	B	B	5	1 4D	0 1D	—	—	—
	C	C	5	2 3D	0 2D	—	—	—
	D	D	5	0 5D	—	—	—	—
Organisms loaded by:		BA Checked by:	BA	BA	BA	BA	BA	MF

Loading Verified by: JG
AR = after renewal
m = missing d = dead BR = before renewal hr = hour

H. azteca		
0 hr	Alk	Hrd
Well		
100%		
96 hr	Alk	Hrd
Well		
100%		

Comments: (A) Transcription error BA

Investigators' Signatures
Bruce Alexander
Marshall Faircloth
John Francis
Jason Daniel
Chelsea French
Reviewer: Walter Wolfe

Method: TSK

Statistics: LC50	95% confidence intervals Lower	Upper
48 hour: 2.168	2.056	2.287
96 hour: 1.013	1.887	2.116

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Revised 7/15/2008 mf

Appendix C. (continued)

FDEP Biology Laboratory - Acute SRT Bench Sheet
0-96 hour parameters

Methods from EPA-821-R-02-012 except *H. azteca*

Test Page 2 of 2

Test Organism: (check one)
- [ ] *Americamysis bahia* Method 2007.0 TA07.03
- [ ] *Menidia Beryllina* Method 2006.0 TA07.04
- [ ] *Hyalella azteca* Method 100.1 EPA-600-R-99-064
- [X] *Ceriodaphnia dubia* Method 2002.0 TA07.01
- [ ] *Cyprinella leedsi* Method 2000.0 TA07.02
- [ ] *Pimephales promelas* Method 2000.0 TA07.02

Test Beginning: Date: 3-9-10 Time: 12 40
Test Ending: Date: 3-13-10 Time: 0900

Test Type: [ ] non-renewal [X] renewal

hr = hour AR = after renewal BR = before renewal

Concentration µg/L mg/L (circle)	Replicate	Test Hour	pH (SU)	Temperature (°C)	Dissolved Oxygen (mg/L)	Conductivity [ ] µmhos [X] mmhos	Salinity (ppth)
Control	M	0 hr	8.0	24.5	7.2	0.171	—
	—	24 hr	—	—	—	—	—
	A	48 hr BR	8.0	24.0	7.1	0.206	—
	M	48 hr AR	8.0	24.6	6.9	0.177	—
	—	72 hr	—	—	—	—	—
	A	96 hr	8.1	24.7	7.4	0.178	—
1.5	M	0 hr	8.1	24.8	7.3	2.93	—
	—	24 hr	—	—	—	—	—
	A	48 hr BR	8.1	24.0	7.2	3.43	—
	M	48 hr AR	8.0	25.2	6.9	3.05	—
	—	72 hr	—	—	—	—	—
	A	96 hr	8.1	24.3	7.6	3.30	—
2.0	M	0 hr	8.1	24.8	7.4	3.80	—
	—	24 hr	—	—	—	—	—
	A	48 hr BR	8.1	24.0	7.3	4.45	—
	M	48 hr AR	8.0	25.3	7.0	3.98	—
	—	72 hr	—	—	—	—	—
	A	96 hr	8.1	24.0	7.7	4.49	—
2.5	M	0 hr	8.1	24.7	7.3	4.65	—
	—	24 hr	—	—	—	—	—
	A	48 hr BR	8.1	24.0	7.3	5.46	—
	M	48 hr AR	8.1	25.3	7.0	4.84	—
	—	72 hr	—	—	—	—	—
	A	96 hr	8.1	24.4	7.7	5.03	—
3.0	M	0 hr	8.1	25.2	7.4	5.59	—
	—	24 hr	—	—	—	—	—
	A	48 hr BR	8.1	24.0	7.4	6.39	—
	M	48 hr AR	8.1	25.0	7.0	5.74	—
	—	72 hr	—	—	—	—	—
	—	96 hr	—	—	—	—	—
4.6	M	0 hr	8.0	24.3	7.4	7.28	—
	—	24 hr	—	—	—	—	—
	A	48 hr BR	8.1	24.0	6.9	9.58	—
	—	48 hr AR	—	—	—	—	—
	—	72 hr	—	—	—	—	—
	—	96 hr	—	—	—	—	—

Investigators Signatures
Marshall Faircloth
Jones Frank
Chelsea Frenah
Reviewed By: Loretta Wolfe

Measured by:
0 hr: BA
24 hr: —
48 hr BR: BA
48 hr AR: BA
72 hr: —
96 hr: MF

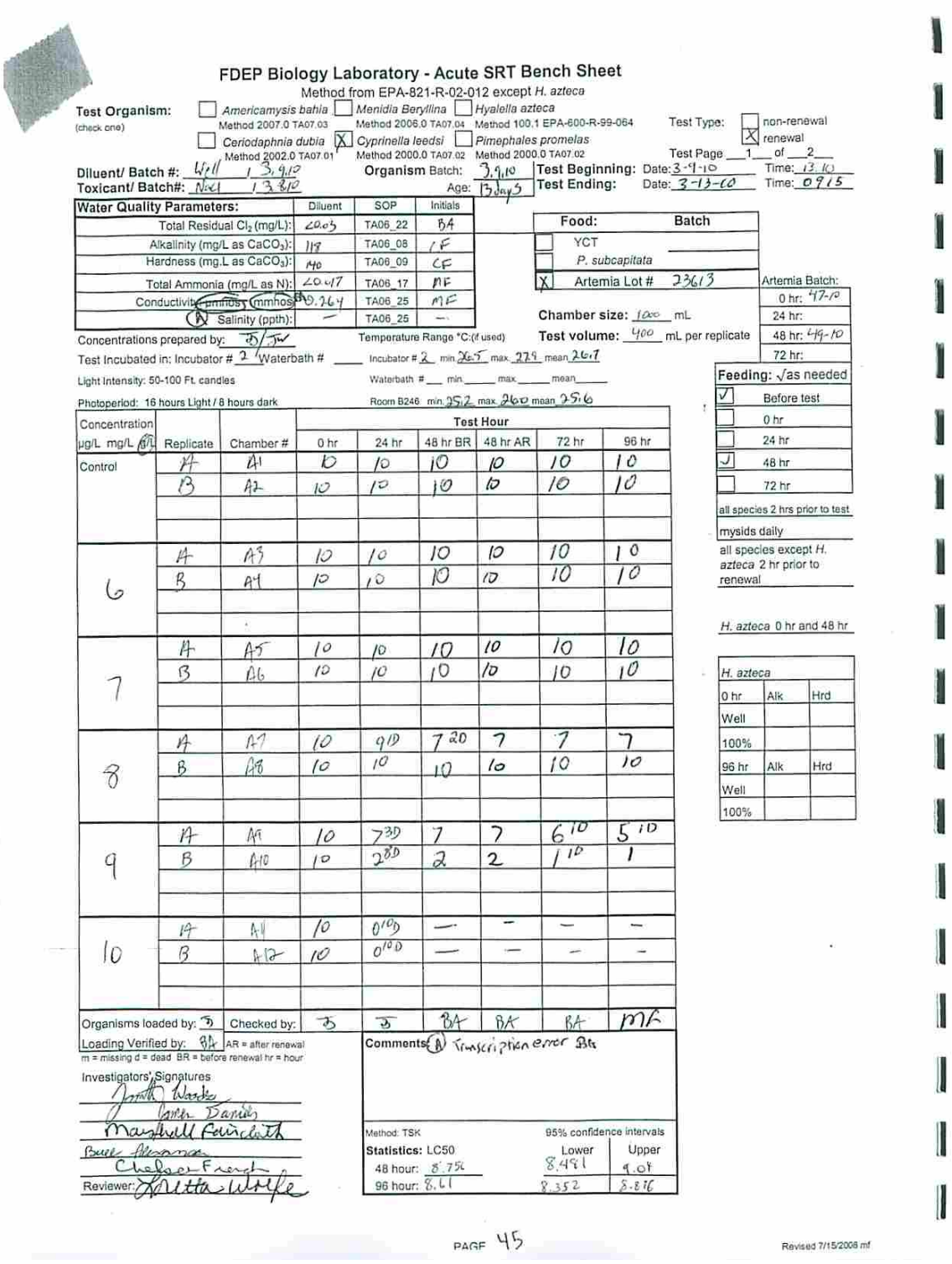
Measure/record salinity for marine tests

Comments:

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Revised 07/15/2008 MF

Appendix C. (continued)



FDEP Biology Laboratory - Acute SRT Bench Sheet

Method from EPA-821-R-02-012 except *H. azteca*

Test Organism: (check one)
☐ *Americamysis bahia* Method 2007.0 TA07.03 ☐ *Menidia Beryllina* Method 2006.0 TA07.04 ☐ *Hyalella azteca* Method 100.1 EPA-600-R-99-064
☐ *Ceriodaphnia dubia* Method 2002.0 TA07.01 ☒ *Cyprinella leedsi* Method 2000.0 TA07.02 ☐ *Pimephales promelas* Method 2000.0 TA07.02

Test Type: ☐ non-renewal ☒ renewal
Test Page 1 of 2

Diluent/ Batch #: Well 1 3.9.10
Toxicant/ Batch#: NaCl 1 3.8.10
Organism Batch: 3.9.10
Age: 13 days
Test Beginning: Date: 3-9-10 Time: 13:10
Test Ending: Date: 3-13-10 Time: 09:15

Water Quality Parameters:	Diluent	SOP	Initials
Total Residual Cl_2 (mg/L):	<0.03	TA06_22	BA
Alkalinity (mg/L as $CaCO_3$):	118	TA06_08	CF
Hardness (mg.L as $CaCO_3$):	140	TA06_09	CF
Total Ammonia (mg/L as N):	<0.017	TA06_17	MF
Conductivity ~~µmhos~~ (mmhos)	0.264	TA06_25	MF
(R) Salinity (ppth):	—	TA06_25	—

Food:	Batch
☐ YCT	
☐ *P. subcapitata*	
☒ Artemia Lot #	23613

Artemia Batch:
0 hr: 47-10
24 hr:
48 hr: 49-10
72 hr:

Chamber size: 1000 mL
Test volume: 400 mL per replicate

Concentrations prepared by: JS/JW
Temperature Range °C:(if used)
Test Incubated in: Incubator # 2 Waterbath # ____ Incubator # 2 min 26.5 max 27.9 mean 26.7
Light Intensity: 50-100 Ft. candles Waterbath # ___ min ____ max ____ mean ____
Photoperiod: 16 hours Light / 8 hours dark Room B246 min 25.2 max 26.0 mean 25.6

Feeding: √as needed
☑ Before test
☐ 0 hr
☐ 24 hr
☑ 48 hr
☐ 72 hr

all species 2 hrs prior to test
mysids daily
all species except *H. azteca* 2 hr prior to renewal
H. azteca 0 hr and 48 hr

Concentration µg/L mg/L (R)	Replicate	Chamber #	Test Hour 0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
Control	A	A1	10	10	10	10	10	10
	B	A2	10	10	10	10	10	10
6	A	A3	10	10	10	10	10	10
	B	A4	10	10	10	10	10	10
7	A	A5	10	10	10	10	10	10
	B	A6	10	10	10	10	10	10
8	A	A7	10	9D	7 20	7	7	7
	B	A8	10	10	10	10	10	10
9	A	A9	10	7 3D	7	7	6 1D	5 1D
	B	A10	10	2 3D	2	2	1 1D	1
10	A	A11	10	0 10D	—	—	—	—
	B	A12	10	0 10D	—	—	—	—
Organisms loaded by: JS		Checked by:	JS	JS	BA	BA	BA	MF

H. azteca		
0 hr	Alk	Hrd
Well		
100%		
96 hr	Alk	Hrd
Well		
100%		

Loading Verified by: BA AR = after renewal
m = missing d = dead BR = before renewal hr = hour

Comments: (A) Transcription error BA

Investigators' Signatures
Jonathan Wade
Carter Daniels
Marshall Faircloth
Brett Thomas
Chelsea French
Reviewer: Martha Wolfe

Method: TSK

Statistics: LC50	95% confidence intervals Lower	Upper
48 hour: 8.75	8.491	9.01
96 hour: 8.61	8.352	8.876

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Revised 7/15/2008 mf

Appendix C. (continued)

FDEP Biology Laboratory - Acute SRT Bench Sheet
0-96 hour parameters

Methods from EPA-821-R-02-012 except *H. azteca*

Test Page 2 of 2

Test Organism: (check one)
- ☐ *Americamysis bahia* Method 2007.0 TA07.03
- ☐ *Menidia Beryllina* Method 2006.0 TA07.04
- ☐ *Hyalella azteca* Method 100.1 EPA-600-R-99-064
- ☐ *Ceriodaphnia dubia* Method 2002.0 TA07.01
- ☒ *Cyprinella leedsi* Method 2000.0 TA07.02
- ☐ *Pimephales promelas* Method 2000.0 TA07.02

Test Beginning: Date: 3-9-10 Time: 13:10
Test Ending: Date: 3-13-10 Time: 0915

Test Type: ☐ non-renewal ☒ renewal

hr = hour AR = after renewal BR = before renewal

Concentration µg/L mg/L (circle)	Replicate	Test Hour	pH (SU)	Temperature (° C)	Dissolved Oxygen (mg/L)	(A) Conductivity ☐ µmhos ☒ mmhos	Salinity (ppth)
Control	A	0 hr	7.96	24.0	7.7	0.264	—
	B	24 hr	8.2	24.1	7.0	0.266	—
	A	48 hr BR	8.1	25.0	6.9	0.294	—
	A	48 hr AR	8.2	24.3	7.5	0.416	—
	B	72 hr	8.2	24.1	7.2	0.330	—
	A	96 hr	8.2	24.9	6.6	0.307	—
6	A	0 hr	7.9	25.1	7.5	9.61	—
	B	24 hr	8.0	24.9	6.6	9.95	—
	A	48 hr BR	8.0	25.0	6.7	10.0	—
	A	48 hr AR	8.0	24.4	7.2	10.5	—
	B	72 hr	8.0	24.0	7.0	10.6	—
	A	96 hr	7.9	25.4	6.1	11.2	—
7	A	0 hr	8.0	25.5	7.3	11.36	—
	B	24 hr	8.1	24.9	6.8	11.90	—
	A	48 hr BR	8.0	24.8	6.7	12.0	—
	A	48 hr AR	8.0	24.5	7.0	12.4	—
	B	72 hr	8.0	24.1	6.9	12.4	—
	A	96 hr	8.0	25.2	6.0	12.8	—
8	A	0 hr	8.0	25.7	7.2	13.62	—
	B	24 hr	8.1	24.9	6.7	13.98	—
	A	48 hr BR	8.0	24.9	6.8	14.5	—
	A	48 hr AR	8.0	24.5	6.6	14.7	—
	B	72 hr	8.1	24.0	7.0	14.1	—
	A	96 hr	8.1	25.3	6.5	14.5	—
9	A	0 hr	7.9	25.9	7.1	14.81	—
	B	24 hr	8.0	24.8	6.5	15.25	—
	A	48 hr BR	8.1	25.1	6.8	15.7	—
	A	48 hr AR	8.0	24.6	7.0	15.8	—
	B	72 hr	8.2	24.4	7.0	15.6	—
	A	96 hr	8.1	25.3	6.8	16.2	—
10	A	0 hr	7.9	25.9	7.1	17.04	—
	B	24 hr	8.0	24.7	6.6	17.25	—
—	—	48 hr BR	—	—	—	—	—
—	—	48 hr AR	—	—	—	—	—
—	—	72 hr	—	—	—	—	—
—	—	96 hr	—	—	—	—	—

Investigators Signatures
Reviewed By:

Measured by:
0 hr: JW
24 hr: JW
48 hr BR: JS
48 hr AR: JS
72 hr: JW
96 hr: MF

Measure/record salinity for marine tests

Comments: (A) Transcription error 30

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Revised 07/15/2008 MF

Appendix C. (continued)

***Ceriodaphnia dubia* Acute Standard Reference Toxicant Test**

DATE: 3-9-10 DURATION: 96 h TOXICANT : NaCl
SPECIES: ***Ceriodaphnia dubia***

RAW DATA: Concentration (g/L)	Number Exposed	Mortalities
.00	20	0
1.50	20	1
2.00	20	8
2.50	20	20
3.00	20	20
4.00	20	20

SPEARMAN-KARBER TRIM: 5.00%

SPEARMAN-KARBER ESTIMATES: LC50: 2.01
95% LOWER CONFIDENCE: 1.89
95% UPPER CONFIDENCE: 2.15

***Cyprinella leedsi* Acute Standard Reference Toxicant Test**

DATE: 3-9-10 DURATION: 96 h TOXICANT : NaCl
SPECIES: ***Cyprinella leedsi***

RAW DATA: Concentration (g/L)	Number Exposed	Mortalities
.00	20	0
6.00	20	0
7.00	20	0
8.00	20	3
9.00	20	14
10.00	20	20

SPEARMAN-KARBER TRIM: .00%

SPEARMAN-KARBER ESTIMATES: LC50: 8.61
95% LOWER CONFIDENCE: 8.35
95% UPPER CONFIDENCE: 8.88

Appendix D. Test Organism Receipts

MARINCO BIOASSAY LABORATORY
The Aquatic Toxicology Specialists

NELAP Certification # E84191

Shipment Record

State of Florida Aquaculture Certificate Number AQ0668007

Shipping Date: 3/8/10

Ship to: FDEP

P.O. No:

Species	Quantity	Age	Brood/Lot Number	Temp. (°C)	pH (S.U.)	Salinity (‰)
Mysidopsis bahia	100	4 DAYS	MS100304	25	7.9	≈ 14
Menidia beryllina	100	12 DAYS	SS100224	25	7.9	≈ 14
						Hardness mg/L
Cyprinella leedsi	~~200~~ 150	12 DAYS	CL100224	25	7.8	80
Pimephales promelas	150 ac	7days	FM100301	25	7.8	80
Ceriodaphnia dubia						
Daphnia magna						
YCT						
P. subcapitata						

Packed by:

Shipped Via: FedEx

Notes:

Thank you for your order.

4569 Samuel Street · Sarasota, FL 34233 · Tel: 941-925-3594 · Fax: 941-922-3874 · Web: www.biologylab.com

Appendix D. (continued)

MARINCO BIOASSAY LABORATORY
The Aquatic Toxicology Specialists

NELAP Certification # E84191

Shipment Record

State of Florida Aquaculture Certificate Number AQ0668007

Shipping Date: 3/15/10

Ship to: FDEP

P.O. No: _____

Species	Quantity	Age	Brood/Lot Number	Temp. (°C)	pH (S.U.)	Salinity (‰)
Mysidopsis bahia	150	4 DAY	MS100311	25	8.0	25
Menidia beryllina	150	12 DAYS	SS100303	25	8.0	25
						Hardness mg/L
Cyprinella leedsi	150	8 DAYS	CL100307	25	7.8	80
Pimephales promelas						
Ceriodaphnia dubia						
Daphnia magna						
YCT						
P. subcapitata						

Packed by: PMC

Shipped Via: FedEx

Notes: _____

Thank you for your order.

4569 Samuel Street · Sarasota, FL 34233 · Tel: 941-925-3594 · Fax: 941-922-3874 · Web: www.biologylab.com

Appendix E. Chemical Analyses performed on the effluent from the DuPont North Maxville Expansion Plant Outfall D-001, sampled on March 15, 2010.

Field ID	Analysis Group	Components	Result	Remark	Units	MDL	PQL
BLANK	Metals	Aluminum	60	U	ug/L	60	240
BLANK	Metals	Magnesium	0.04	U	mg/L	0.04	0.16
BLANK	Metals	Iron	30	U	ug/L	30	120
BLANK	Metals	Selenium	0.2	U	ug/L	0.2	0.8
BLANK	Metals	Nickel	0.25	U	ug/L	0.25	1
BLANK	Metals	Copper	0.5	U	ug/L	0.5	0.5
BLANK	Metals	Chromium	0.3	U	ug/L	0.3	1.2
BLANK	Metals	Cadmium	0.03	U	ug/L	0.03	0.12
BLANK	Metals	Silver	0.025	U	ug/L	0.025	0.1
BLANK	Metals	Arsenic	0.25	U	ug/L	0.25	1
BLANK	Metals	Lead	0.2	U	ug/L	0.2	0.8
BLANK	Metals	Calcium	0.075	U	mg/L	0.075	0.3
BLANK	Metals	Mercury	0.12	U	ug/L	0.12	0.5
BLANK	Metals	Zinc	5	U	ug/L	5	20
BLANK	Nutrients	Ammonia-N	0.01	U	mg N/L	0.01	0.02
BLANK	Nutrients	Kjeldahl Nitrogen	0.08	U	mg N/L	0.08	0.2
BLANK	Nutrients	O-Phosphate-P	0.004	U	mg P/L	0.004	0.01
BLANK	Nutrients	NO2NO3-N	0.004	U	mg N/L	0.004	0.01
BLANK	Nutrients	Total-P	0.004	U	mg P/L	0.004	0.01
BLANK	Overflow	Radium 226	0.2	U	pCi/L		
BLANK	Overflow	Radium 228-Counting Error	0.4		pCi/L		
BLANK	Overflow	Alpha-Counting Error	0.3		pCi/L		
BLANK	Overflow	Radium 228	0.7	U	pCi/L		
BLANK	Overflow	Alpha, Total	0.5	U	pCi/L		
BLANK	Overflow	Radium 226-Counting Error	0.1		pCi/L		
BLANK	Priority Organic Pollutants	2,4-Dinitrotoluene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2-Chloronaphthalene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Bis(2-ethylhexyl)phthalate	14	U	ug/L	14	57
BLANK	Priority Organic Pollutants	Di-n-octyl phthalate	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Dibenzo(a,h)anthracene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Diethyl phthalate	0.95	U	ug/L	0.95	3.8

Appendix E. (continued)

Field ID	Analysis Group	Components	Result	Remark	Units	MDL	PQL
BLANK	Priority Organic Pollutants	Benzidine	95	UJ	ug/L	95	380
BLANK	Priority Organic Pollutants	Benzo(k)fluoranthene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2,4,6-Trichlorophenol	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2,4-Dichlorophenol	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2,6-Dinitrotoluene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	3,3'-Dichlorobenzidine	38	UJ	ug/L	38	150
BLANK	Priority Organic Pollutants	Dimethyl phthalate	9.5	U	ug/L	9.5	38
BLANK	Priority Organic Pollutants	Hexachlorobenzene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	N-Nitrosodi-n-propylamine	1.9	U	ug/L	1.9	7.6
BLANK	Priority Organic Pollutants	Naphthalene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2-Chlorophenol	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2-Nitrophenol	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Benzo(b)fluoranthene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Butyl benzyl phthalate	4.7	U	ug/L	4.7	19
BLANK	Priority Organic Pollutants	Hexachlorocyclopentadiene	2.8	UJ	ug/L	2.8	11
BLANK	Priority Organic Pollutants	Phenol	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Pyrene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2-Methyl-4,6-dinitrophenol	2.8	U	ug/L	2.8	11
BLANK	Priority Organic Pollutants	4-Bromophenyl phenyl ether	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	4-Chlorophenyl phenyl ether	1.9	U	ug/L	1.9	7.6
BLANK	Priority Organic Pollutants	Anthracene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Benzo(a)anthracene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Bis(2-chloroethoxy)methane	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Isophorone	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	1,2,4-Trichlorobenzene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	4-Chloro-3-methylphenol	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	4-Nitrophenol	14	U	ug/L	14	57
BLANK	Priority Organic Pollutants	Acenaphthene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Bis(2-chloroisopropyl)ether	2.8	U	ug/L	2.8	11
BLANK	Priority Organic Pollutants	Chrysene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Di-n-butyl phthalate	4.7	U	ug/L	4.7	19
BLANK	Priority Organic Pollutants	N-Nitrosodiphenylamine/ Diphenylamine	2.8	U	ug/L	2.8	11
BLANK	Priority Organic Pollutants	Nitrobenzene	1.9	U	ug/L	1.9	7.6
BLANK	Priority Organic Pollutants	Pentachlorophenol	2.8	U	ug/L	2.8	11

Appendix E. (continued)

Field ID	Analysis Group	Components	Result	Remark	Units	MDL	PQL
BLANK	Priority Organic Pollutants	Phenanthrene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	2,4-Dimethylphenol	9.5	U	ug/L	9.5	38
BLANK	Priority Organic Pollutants	Benzo(g,h,i)perylene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Fluorene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Hexachlorobutadiene	2.8	UJ	ug/L	2.8	11
BLANK	Priority Organic Pollutants	Hexachloroethane	2.8	UJ	ug/L	2.8	11
BLANK	Priority Organic Pollutants	2,4-Dinitrophenol	14	U	ug/L	14	57
BLANK	Priority Organic Pollutants	Acenaphthylene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Benzo(a)pyrene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Bis(2-chloroethyl)ether	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Fluoranthene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	Indeno(1,2,3-cd)pyrene	0.95	U	ug/L	0.95	3.8
BLANK	Priority Organic Pollutants	N-Nitrosodimethylamine	1.9	U	ug/L	1.9	7.6
DUPONT N. BCKD	Nutrients	Turbidity	1.9	A	NTU	0.1	0.1
DUPONT N. BCKD	Field Parameter	pH	4.17				
DUPONT N. BCKD	Field Parameter	Sample Depth	0.1		m		
DUPONT N. BCKD	Field Parameter	Temperature	13.56		C		
DUPONT N. BCKD	Field Parameter	Specific Conductance	86		umhos/cm		
DUPONT N. BCKD	Field Parameter	Dissolved Oxygen	6.99		mg/L		
DUPONT N. D001	AGP/LN	Algal Growth Potential	3.64		mg DryWt/L	0.3	0.9
DUPONT N. D001	Chlorophyll/Grain Size/BOD	Phaeophytin-a	0.4	U	ug/L	0.4	1.2
DUPONT N. D001	Chlorophyll/Grain Size/BOD	Chlorophyll-a, Corrected	6.8		ug/L	0.55	1.7
DUPONT N. D001	Toxicology	BSY-AS-FW-C.dubia, LC50	100	K	LC50		
DUPONT N. D001	Toxicology	BSY-AD-FW-Fish, LC50-pHadj	100	L	LC50		
DUPONT N. D001	Toxicology	BSY-AS-FW-Fish, LC50	100	K	LC50		
DUPONT N. D001	Toxicology	BSY-AD-FW-C.dubia,LC50-pHadj	100	L	LC50		
DUPONT N. D001	Metals	Arsenic	0.25	U	ug/L	0.25	1
DUPONT N. D001	Metals	Nickel	1.75		ug/L	0.25	1
DUPONT N. D001	Metals	Cadmium	0.047	I	ug/L	0.03	0.12
DUPONT N. D001	Metals	Lead	0.38	I	ug/L	0.2	0.8
DUPONT N. D001	Metals	Aluminum	4420		ug/L	60	240
DUPONT N. D001	Metals	Magnesium	2.15		mg/L	0.04	0.16
DUPONT N. D001	Metals	Zinc	89		ug/L	5	20
DUPONT N. D001	Metals	Copper	0.5	U	ug/L	0.5	0.5

Appendix E. (continued)

Field ID	Analysis Group	Components	Result	Remark	Units	MDL	PQL
DUPONT N. D001	Metals	Chromium	0.76	I	ug/L	0.3	1.2
DUPONT N. D001	Metals	Silver	0.025	U	ug/L	0.025	0.1
DUPONT N. D001	Metals	Selenium	0.2	U	ug/L	0.2	0.8
DUPONT N. D001	Metals	Mercury	0.12	U	ug/L	0.12	0.5
DUPONT N. D001	Metals	Calcium	6.88		mg/L	0.075	0.3
DUPONT N. D001	Metals	Iron	98	I	ug/L	30	120
DUPONT N. D001	Nutrients	TSS	6	I	mg/L	4	16
DUPONT N. D001	Nutrients	Total-P	0.01	I	mg P/L	0.004	0.01
DUPONT N. D001	Nutrients	NO2NO3-N	0.15		mg N/L	0.004	0.01
DUPONT N. D001	Nutrients	O-Phosphate-P	0.004	U	mg P/L	0.004	0.01
DUPONT N. D001	Nutrients	Kjeldahl Nitrogen	0.47		mg N/L	0.08	0.2
DUPONT N. D001	Nutrients	Ammonia-N	0.035		mg N/L	0.01	0.02
DUPONT N. D001	Nutrients	Turbidity	3.6		NTU	0.1	0.1
DUPONT N. D001	Overflow	Radium 228	2.5		pCi/L		
DUPONT N. D001	Overflow	Radium 228-Counting Error	0.7		pCi/L		
DUPONT N. D001	Overflow	Alpha-Counting Error	1.3		pCi/L		
DUPONT N. D001	Overflow	Radium 226-Counting Error	0.2		pCi/L		
DUPONT N. D001	Overflow	Alpha, Total	3.6		pCi/L		
DUPONT N. D001	Overflow	Radium 226	1.3		pCi/L		
DUPONT N. D001	Priority Organic Pollutants	Nitrobenzene	1.9	U	ug/L	1.9	7.8
DUPONT N. D001	Priority Organic Pollutants	Pentachlorophenol	2.9	U	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	2,4-Dinitrophenol	15	U	ug/L	15	58
DUPONT N. D001	Priority Organic Pollutants	2-Chlorophenol	0.97	UJ	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2-Methyl-4,6-dinitrophenol	2.9	U	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	2-Nitrophenol	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Bis(2-chloroethoxy)methane	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Fluorene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Hexachlorobutadiene	2.9	UJ	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	Pyrene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	1,2,4-Trichlorobenzene	0.97	UJ	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2-Chloronaphthalene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	3,3'-Dichlorobenzidine	39	UJ	ug/L	39	160
DUPONT N. D001	Priority Organic Pollutants	Acenaphthene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Anthracene	0.97	U	ug/L	0.97	3.9

Appendix E. (continued)

Field ID	Analysis Group	Components	Result	Remark	Units	MDL	PQL
DUPONT N. D001	Priority Organic Pollutants	Dimethyl phthalate	9.7	U	ug/L	9.7	39
DUPONT N. D001	Priority Organic Pollutants	Hexachlorocyclopentadiene	2.9	UJ	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	Naphthalene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2,4,6-Trichlorophenol	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2,6-Dinitrotoluene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	4-Chloro-3-methylphenol	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Benzidine	97	UJ	ug/L	97	390
DUPONT N. D001	Priority Organic Pollutants	Benzo(a)anthracene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Benzo(k)fluoranthene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Isophorone	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2,4-Dichlorophenol	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2,4-Dimethylphenol	9.7	UJ	ug/L	9.7	39
DUPONT N. D001	Priority Organic Pollutants	4-Nitrophenol	15	U	ug/L	15	58
DUPONT N. D001	Priority Organic Pollutants	Acenaphthylene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Benzo(a)pyrene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Benzo(g,h,i)perylene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Bis(2-chloroisopropyl)ether	2.9	U	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	Di-n-butyl phthalate	4.9	U	ug/L	4.9	19
DUPONT N. D001	Priority Organic Pollutants	Bis(2-chloroethyl)ether	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Butyl benzyl phthalate	4.9	U	ug/L	4.9	19
DUPONT N. D001	Priority Organic Pollutants	Chrysene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Diethyl phthalate	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	N-Nitrosodi-n-propylamine	1.9	U	ug/L	1.9	7.8
DUPONT N. D001	Priority Organic Pollutants	N-Nitrosodimethylamine	1.9	U	ug/L	1.9	7.8
DUPONT N. D001	Priority Organic Pollutants	4-Bromophenyl phenyl ether	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	4-Chlorophenyl phenyl ether	1.9	U	ug/L	1.9	7.8
DUPONT N. D001	Priority Organic Pollutants	Benzo(b)fluoranthene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Bis(2-ethylhexyl)phthalate	15	U	ug/L	15	58
DUPONT N. D001	Priority Organic Pollutants	Dibenzo(a,h)anthracene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Phenanthrene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Di-n-octyl phthalate	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Hexachlorobenzene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Hexachloroethane	2.9	UJ	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	Indeno(1,2,3-cd)pyrene	0.97	U	ug/L	0.97	3.9

Appendix E. (continued)

Field ID	Analysis Group	Components	Result	Remark	Units	MDL	PQL
DUPONT N. D001	Priority Organic Pollutants	N-Nitrosodiphenylamine/ Diphenylamine	2.9	U	ug/L	2.9	12
DUPONT N. D001	Priority Organic Pollutants	Phenol	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	2,4-Dinitrotoluene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Priority Organic Pollutants	Fluoranthene	0.97	U	ug/L	0.97	3.9
DUPONT N. D001	Field Parameter	Dissolved Oxygen	9.13		mg/L		
DUPONT N. D001	Field Parameter	Sample Depth	0.2		m		
DUPONT N. D001	Field Parameter	Temperature	16.96		C		
DUPONT N. D001	Field Parameter	Specific Conductance	264		umhos/cm		
DUPONT N. D001	Field Parameter	pH	4.31				

Appendix F. Facility Summary, Effluent Limits, and Permit Violations Recorded on Discharge Monitoring Report

State of Florida
Department of Environmental Protection
Facility Introduction & Summary

PART I: Information gathered prior to sampling event	
Prepared By: Anthony Vigliotti	Contact Number:(904) 807-3337
Facility Name (as it appears on permit): DuPont North Maxville Expansion	Former Names:
NPDES Permit Number: FL0435490	Permit Expiration Date: February 27, 2011
Physical Address: US 301 near Macclenny, Macclenny, FL	
District: NED	County: Baker
Function of Facility: Dredge mining for heavy minerals.	
Facility Type: Mineral Mining	Permitted Capacity (MGD): 5.0MGD
Mean Flow for previous 12 months for all EXTERNAL outfalls listed in permit (list date range including months): May 09 – Sep 09 : D-001 @ 0.3944 MGD Average	
If applicable, Mean Flow for previous 12 months for REUSE outfall(s) (list date range including months): N/A	
Description of type of Discharge: ☐ Continuous ☒ Intermittent ☐ Seasonal ☒ Rainfall Dependent ☐ Other, please specify	

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Appendix F. (continued)

Description of Sampling Location(s) - actual permit designation of permitted sampling point(s):

Prior to each discharge from each outfall, the wastewater shall be sampled in the final polishing pond (s) for turbidity and pH to verify compliance with the pH and turbidity standard required. If the quality of wastewater in the polishing pond(s) is such that water quality standards will not be met, the wastewater from the impoundment shall be treated prior to discharge into the surface water.

(1) Routine and additional tests shall be conducted on a single grab sample of final effluent. If the duration of the discharge is less than 24-hours, the duration of discharge shall be documented on the chain of custody.
(2) Results for each additional test shall include the determination of LC50 values with 95% confidence limits.

Description of Permitted Outfall(s):

A new 5 MGD maximum discharge to wetlands to Turkey Creek (Class III Fresh waters), D-001. The D-001 outfall is located approximately at latitude 30° 12' 34.74" N, longitude 82° 04' 11.19" W.

A new 5 MGD maximum discharge to wetland to Turkey Creek (Class III Fresh waters), D-002. The D-002 outfall is located approximately at latitude 30° 13' 34" N, longitude 82° 04' 11" W.

A new 5 MGD maximum discharge to wetlands to Turkey Creek (Class III Fresh waters), D-003. The D-003 outfall is located approximately at latitude 30° 14' 16.73" N, longitude 82° 04' 35.5" W.

A new 5 MGD maximum discharge to wetlands to Turkey Creek (Class III Fresh waters), D-004. The D-004 outfall is located approximately at latitude 30° 14' 44.66" N, longitude 82° 04' 35.59" W.

A new 5 MGD maximum discharge to wetlands to Turkey Creek (Class III Fresh waters), D-005. The D-005 outfall is located approximately at latitude 30° 15' 46.28" N, longitude 82° 04' 42.43" W.

A new 5 MGD maximum discharge to wetlands to Deep Creek (Class III Fresh waters), D-006. The D-006 outfall is located approximately at latitude 30° 16' 39.92" N, longitude 82° 03' 48.39" W.

A new 5 MGD maximum discharge to wetlands to Deep Creek (Class III Fresh waters), D-007. The D-007 outfall is located approximately at latitude 30° 16' 12.13" N, longitude 82° 03' 40.4" W.

A new 5 MGD maximum discharge to wetlands to Deep Creek (Class III Fresh waters), D-008. The D-008 outfall is located approximately at latitude 30° 15' 52.23" N, longitude 82° 03' 37.15" W.

A new 5 MGD maximum discharge to wetlands to Deep Creek (Class III Fresh waters), D-011. The D-011 outfall is located approximately at latitude 30° 14' 03" N, longitude 83° 03' 22" W.

Appendix F. (continued)

Description of Treatment Process (if multiple discharge points, include a detailed map or diagram of facility):

Process water from the dredge mining operation is collected in humate settling ponds that are constructed as the mining progresses. Stormwater from mining areas and overflow from the humate settling ponds is collected and treated in a set of polishing ponds in series to meet water quality standards prior to discharge through the outfall. There will be a total of 9 sets of polishing ponds with nine outfalls due to the progressive nature of the mining operation. Each set of ponds will be constructed as needed as the footprint of the mine progresses. Ponds that are no longer in use will be properly decommissioned and closed. Currently the only outfall used thus far has been D-001.

Appendix F. (continued)

Effluent Limitations			Monitoring Requirements						
Parameter	Max/Min	Annual Average	Monthly Average	Weekly Average	Single Sample	Monitoring Frequency	Sample Type	Monitoring Location Site Number	Notes
Flow (MGD)	5.0		--		-	Per Discharge	Estimated	EFF-1	
Flow (MGD)	--		5.0		-	Per Discharge	Estimated	CAL-1	
pH (effluent) (SU)	See Cond. I.A.11. & I.A.14		--		See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-1	
pH (Background) (SU)	Report		--		Report	Weekly, When Discharging	Grab	SWB-1	
Solids, Total Suspended (MG/L)	30.0		20.0		--	Weekly, When Discharging	Grab	EFF-1	
Iron, Total Recoverable (MG/L)	1.0		Report		--	Weekly, When Discharging	Grab	EFF-1	
Turbidity (NTU)	See Cond. I.A.11. & I.A.14		--		--	Weekly, When Discharging	Grab	EFF-1	
Turbidity (Background) (NTU)	Report		--		--	Weekly, When Discharging	Grab	SWB-1	
Radium 226+ Radium 228, Total (PCI/L)	5.0		5.0 (rolling 12 month average)		--	Monthly (see Cond. I.C.1)	Grab	EFF-1	
Alpha, Gross Particle Activity (PCI/L)	15.0		15.0 (rolling 12 month average)		--	Monthly (see Cond. I.C.1)	Grab	EFF-1	
Acute Whole Effluent Toxicity	Per discharge or once per quarter		See Condition I.A.15		Grab	EFF-1			

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-2
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-2
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-1

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Appendix F. (continued)

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-2
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-2
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-2
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-1
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-2
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-2
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-2

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-3
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-3
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-2
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-3
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-3
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-3
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-2
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-3

Appendix F. (continued)

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-3
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-3

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-4
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-4
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-2
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-4
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-4
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-4
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-2
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-4
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-4
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-4

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-5
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-5

Appendix F.(continued)

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-1
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-5
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-5
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-5
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-1
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-5
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-5
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-5

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-6
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-6
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-2
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-6
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-6
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-6
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-2

Appendix F. (continued)

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-6
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-6
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-6

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-7
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-7
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-2
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-7
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-7
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-7
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-2
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-7
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-7
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-7

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-8

Appendix F. (continued)

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-8
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-2
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-8
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-8
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-8
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-2
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-8
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-8
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-8

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Flow (MGD)	--	5.0	--	Per Discharge	Estimated	EFF-11
pH (effluent) (SU)	--	See Cond. I.A.11. & I.A.14	See Cond. I.A.11. & I.A.14	Weekly, When Discharging	Grab	EFF-11
pH (background) (SU)	--	Report	Report	Weekly, When Discharging	Grab	SWB-2
Solids, Total Suspended (MG/L)	20.0	30.0	--	Weekly, When Discharging	Grab	EFF-11
Iron, Total Recoverable (MG/L)	Report	1.0	--	Weekly, When Discharging	Grab	EFF-11
Turbidity (NTU)	--	See Cond. I.A.11. & I.A.14	--	Weekly, When Discharging	Grab	EFF-11

Appendix F. (continued)

Parameters (units)	Monthly Average	Daily Maximum	Daily Minimum	Monitoring Frequency	Sample Type	Sample Point
Turbidity (Background) (NTU)	--	Report	--	Weekly, When Discharging	Grab	SWB-2
Radium 226 + Radium 228, Total (PCI/L)	5.0 (rolling 12 month average)	5.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-11
Alpha, Gross Particle Activity (PCI/L)	15.0 (rolling 12 month average)	15.0	--	Monthly (see Cond. I.C.1)	Grab	EFF-11
Acute Whole Effluent Toxicity	See Condition I.A.15			Per discharge or once per quarter	Grab	EFF-11

The Facility is only currently discharging from D-001 (EFF-1).

Appendix F. (continued)

PART I: Continued

Receiving Waters: **Turkey Creek & Deep Creek**	**Classification (indicate whether fresh or marine):** **Class III Fresh Waters**

Toxicity Test Requirements (routine and/or additional test language, test species, salinity adjustment, etc.):

Test Requirements

(1) Routine Tests: All routine tests shall be conducted using a control (0% effluent) and one test concentration of 100% final effluent.

(2) Additional Follow-up Tests, if required:
   (a) If a routine test does not meet the acute toxicity limitation in 15.a.(1) and (2) (Permit), the permittee shall conduct three additional follow-up tests on each species that failed the test.
   (b) The first additional test shall be conducted using a control and a minimum of five dilutions: 100%, 75%, 50%, 25%, and 12.5% effluent. The dilution series may be modified in the second and third tests to more accurately bracket the toxicity, such that at least two dilutions above (not to exceed 100% effluent) and two dilutions below the target concentration and a control are run. All test results shall be statistically analyzed according to the Appendices in EPA-821-R-02-012.
   (c) The first test shall be initiated within two weeks of the end of the failed routine test. The remaining additional tests shall be conducted weekly thereafter until a total of three valid additional tests are completed.

(3) The permittee shall conduct 96-hour acute static renewal toxicity tests using the daphnid, *Ceriodaphnia dubia*, and the bannerfin shiner, *Cyprinella leedsi*, concurrently.

(4) All test species, procedures and quality assurance criteria used shall be in accordance with Methods for Measuring Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, EPA-821-R-02-012. Any deviation of the bioassay procedures outlined herein shall be submitted in writing to the Department for review and approval prior to use. In the event the above method is revised, the permittee shall conduct acute toxicity testing in accordance with the revised method.

(5) The control water and dilution water used will be moderately hard water as described in EPA-821-R-02-012, Table 7.

Administrative or Consent Orders (date executed, specific requirements, interim limit dates-if applicable, etc.): N/A

Facility Mixing Zone Details (if applicable): N/A

List Permit Violations (DMR data) and plant upsets that occurred at the plant within the last year:

Date	Parameter and Units	Limit	Result
05/31/09	**Turbidity**	**0.0**	**19.5**
06/30/09	**Turbidity**	**0.0**	**<1.6**
09/30/09	**Ceriodaphnia**	**100 min**	**<100**
12/31/09	**Ceriodaphnia**	**100 min**	**<100**

Describe previous FYI and WQBEL study:
First FYI3 to be performed.

Appendix F. (continued)

Describe previous or current Enforcement Actions (including WLs): N/A
Describe any violations or problems noted in previous inspection: N/A
Discuss whether DMR trends within previous five years are improving or declining: N/A
Additional Comments (this should include a trend of the Effluent, i.e. if the facility is now Reuse):

PART II: Sampling Event Information	
Date(s) sample collected:	Sampler(s):
Temperature (Deg C):	pH (SU):
Dissolved Oxygen (mg/L):	Conductivity (umhos/cm):
Total Residual Chlorine (mg/L):	Total Residual Chlorine - after Disinfection (mg/L):
Method of Chlorination:	Method of De-chlorination:
Flow During Sample Collection for each outfall(MGD):	
Mean Flow for each outfall for previous 12 months based on sampling date (If samples are taken on more than one day, indicate Mean Flow for each day):	
Describe Discharge during Sampling Event: ☐ Continuous ☐ Intermittent ☐ Seasonal ☐ Rainfall Dependent ☐ Other, please specify	
Describe any conditions, difficulties or abnormal events that occurred during the sample collection and any other additional comments here:	

Appendix G. EPA End Sheet

The Bioassay of DuPont North Maxville Expansion effluent sampled on March 15, 2011. NPDES #FL0435490.

Fill Out This Section For All Surface Water Discharger Inspections(CEI, CSI, CBI, PAI, XSI-RI Optional)

Transaction Code	NPDES NUMBER	YR/MO/DA	Insp Type	Inspector	Fac Type
1 N 2 5	3 F L 0 4 3 5 4 9 0 11	12 1 0 0 3 1 5	18 B	19 S	20 2

Remarks

66

The Priority Pollutants Analysis of DuPont North Maxville Expansion effluent sampled on March 15, 2011. NPDES #FL0435490.

Fill Out This Section For All Surface Water Discharger Inspections(CEI, CSI, CBI, PAI, XSI-RI Optional)

Transaction Code	NPDES NUMBER	YR/MO/DA	Insp Type	Inspector	Fac Type
1 N 2 5	3 F L 0 4 3 5 4 9 0 11	12 1 0 0 3 1 5	18 X	19 S	20 2

Remarks

66

Biological Analyses of DuPont North Maxville Expansion effluent sampled on March 15, 2011. NPDES #FL0435490.

Fill Out This Section For All Surface Water Discharger Inspections(CEI, CSI, CBI, PAI, XSI-RI Optional)

Transaction Code	NPDES NUMBER	YR/MO/DA	Insp Type	Inspector	Fac Type
1 N 2 5	3 F L 0 4 3 5 4 9 0 11	12 1 0 0 3 1 5	18 S	19 S	20 2

Remarks

66