

Effluent Quality Assessment

CF Industries, Inc., Hardee Complex I (North Pasture Mine)

Hardee County

NPDES #FL0035271

Sampled February 7, 2011

April 2011

Biology Section
Bureau of Laboratories
Division of Environmental Assessment and Restoration

Quality Manual No. 870346G
NELAC Certification No. E31780

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CF Industries, Inc., Hardee Complex I (North Pasture Mine), 6209 N. County Rd. 663, Wauchula, Hardee County, Florida, 33873. NPDES# FL0035271. Effluent grab samples for biological and chemical analyses for this facility were collected on February 7, 2011.

Introduction

CF Industries, Inc., Hardee Complex I, is a phosphate mine which has not been in operation since the early 1990's. (See Appendix F for detailed facility information provided by Jacki Champion of BMMR Phosphate Management FDEP Southwest District.)

Methods

Samples were collected from the EFF-03 NPDES sampling point for Outfall D-003 by Jacki Champion, Melani Dickenson, and Scott Rose (FDEP Southwest District) following DEP-SOP-001/01 FS 2400 Wastewater Sampling. All DEP SOPs are available on the web at: http://www.floridadep.org/labs/bars/sas/sop/index.htm.

See Appendix A for the completed chain of custody form.

The 48-hour 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.

Results and Discussion

- **Toxicity Test Results**
  Tests were performed February 8 - 10, 2011.

  Ceriodaphnia dubia 48-hour acute screening bioassay – LC_{50} > 100%, 5%* mortality in 100% sample at 48 hours

  Cyprinella leedsi 48-hour acute screening bioassay – LC_{50} > 100%, 0%* mortality in 100% sample at 48 hours

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

  *NOTE: In a 48-hour acute screening 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 5.8 mg dry wt/L of the unicellular green alga, *Pseudokirchneriella subcapitata*, formerly known as *Selenastrum capricornutum*. Raschke and Schultz (1987) determined 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 nitrogen-limited. The predicted AGP based on inorganic nitrogen concentrations was 1.6 (±20%) mg dry wt/L (Table 1; Miller et al. 1978).

  See Table 1 for AGP results.

- **Chemistry Results**
  Total residual chlorine and total ammonia were not detected in the bioassay sample in the bioassay laboratory. The total ammonia concentration in the composite sample collected and preserved for chemical analysis was 0.032 mg N/L. Based on the pH, salinity, and temperature of the effluent as collected, the calculated unionized ammonia concentration was ≤ 0.02 mg N/L.

  Chloride, fluoride, sulfate and iron were detected in the effluent at levels that comply with Class III fresh water quality criteria (62-302.530, F.A.C.). Aluminum, arsenic, nickel, and selenium were detected between the laboratory method detection limits (MDL) and practical quantitation limits (PQL). Chlorophyll *a* was also detected in the effluent.

  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. See Appendix B for bioassay lab equipment.

Conclusion

The effluent sample collected from the EFF-03 NPDES sampling point for Outfall D-003 for this facility on February 7, 2011, was not acutely toxic to either test species during the 48-hour acute screening bioassays. The effluent AGP result was slightly greater than the "problem" threshold for fresh receiving waters suggesting that nutrient enrichment may be an issue. Effluent water quality samples collected on February 7, 2011, did not exceed any applicable water quality standards or violate any permit conditions.

Literature Cited

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.

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.

Table 1. Measured and predicted algal growth potential (AGP; mg dry weight/L) for total soluble inorganic nitrogen (TSIN) and total nitrogen (TN) limitation of the freshwater species *Pseudokirchneriella subcapitata* and ratios of nitrogen to phosphorus for samples collected from the CF Industries, Inc., Hardee Complex I on February 7, 2011.

CF Industries, Inc. Hardee Complex I (North Pasture Mine) NPDES# FL0035271									
Location	AGP (measured)	Predicted AGP (TSIN) ± 20%			Predicted AGP (TN) ± 20%			Inorganic N:P ratio	Total N:P ratio
Effluent	5.8	1.6	±	0.3	42.1	±	8.4	0.1	1.6

Table 2. Effluent limits, Class III Freshwater Criteria and chemical data for samples collected from the CF Industries, Inc., Hardee Complex I on February 7, 2011.

CF Industries, Inc., Hardee Complex I (North Pasture Mine) NPDES# FL0035271	Class III Criteria	Effluent Limits	Effluent	
Metals (µg/L unless otherwise noted)				
Aluminum	-	-	71	I
Arsenic	≤ 50	-	0.92	I
Cadmium	≤ 0.5 b	-	0.05	U
Calcium (mg/L)	-	-	43.9	
Chromium-III	≤ 157.5 b	-	0.3	U
Copper	≤ 17.5 b	-	0.5	U
Iron	≤ 1,000	-	160	
Lead	≤ 8.1 b	-	0.2	U
Magnesium (mg/L)	-	-	24.1	
Nickel	≤ 97.3 b	-	0.28	I
Selenium	≤ 5.0	-	0.35	I
Silver	≤ 0.07	-	0.025	U
Zinc	≤ 223.6 b	-	5	U
Nutrients (mg/L)				
Ortho-phosphate	-	-	0.6	
Total Phosphorus	-	≤ 5.0 s	0.69	
Total Ammonia	-	-	0.032	
Un-ionized Ammonia	≤ 0.02 s	-	≤ 0.02	c
Nitrate and Nitrite	-	-	0.009	I
Total Kjeldahl Nitrogen	-	-	1.1	
Organic Nitrogen	-	-	1.068	c
Total Nitrogen	-	Report	1.109	c
General Physical and Chemical Parameters				
Dissolved Oxygen (mg/L)	≥ 5.0	≥ 5.0 s	8.8	
pH (SU)	6.0 - 8.5	6.0 - 8.5 s	7.6	
Conductivity (µmhos/cm)	≤ 1,275	Report	430	
Temperature (°C)	-	Report	20.2	
Salinity	-	-	< 0.23	c
Total Residual Chlorine (mg/L)	≤ 0.01	-	0.03	UQ£
Chlorophyll *a* (µg/L) - Corrected	-	Report	2.9	
Phaeophytin (µg/L)	-	-	2.7	
Total Suspended Solids (mg/L)	-	≤ 60.0 s	4	U
Turbidity (NTU)	< 29 t	Report	1.6	A
CBOD, 5 day (mg/L), N - inhibited	-	-	2	
Chloride (mg/L)	-	-	13	
Fluoride (mg/L)	≤ 10.0	-	1.2	
Sulfate (mg/L)	-	-	20	
Flow (MGD)	-	Report	0.303	a*
Hardness (mg $CaCO_3$)	-	-	208.86	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
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
* - Data measured from 11/2009 - 11/2010

Florida Department of Environmental Protection

Central Laboratory Sample Submittal Form

Event ID *

Request Number: RQ-2011-02-07-19
FYI3 for CF Industries - Hardee Complex I North Pasture

Requester: Cari Butler
Field Report Prepared By: Jachi Champion
Collected By: Jachi Champion, Melani Dickenson, Scott Rose
Send Final Report To: Vishwas Sathe
Sampling Agency: FDEP

Customer: BMR-TAMPA
Project ID: FYI5
PMAS: 1143

Lab ID *	Location	Collection (begin)	Collection (end)	Bottle Group(s)**
	CF Hardee North Mine	☐ Comp ☒ Grab Date 2/7/11 Time 10:30 ☒ Eastern ☐ Central	Date Time ☐ Eastern ☐ Central	A
	Field ID: D-003	Tot Res Chlorine (mg/L)	Diss Oxygen (mg/L): 8.82	Storet Station Number
	Matrix (include type e.g. Salt, Fresh, etc): Fresh Water; Temp (C): 20.16; pH: 7.62	Sample Depth ☐ m ☐ ft	430 ☐ Salinity (PPTh) ☒ Sp Conductance (umho/cm)	NPDES Number: FL0035271
	Latitude ☐ dd ☐ dms	Longitude ☐ dd ☐ dms	Comments	

Lab ID *	Location	Collection (begin)	Collection (end)	Bottle Group(s)**
	CF Hardee North Mine	☐ Comp ☒ Grab Date 2/7/11 Time ☒ Eastern ☐ Central	Date Time ☐ Eastern ☐ Central	A
	Field ID: D-003	Tot Res Chlorine (mg/L)	Diss Oxygen (mg/L): 8.82	Storet Station Number
	Matrix (include type e.g. Salt, Fresh, etc): Fresh Water; Temp (C): 20.16; pH: 7.62	Sample Depth ☐ m ☐ ft	430 ☐ Salinity (PPTh) ☒ Sp Conductance (umho/cm)	NPDES Number: FL0035271
	Latitude ☐ dd ☐ dms	Longitude ☐ dd ☐ dms	Comments: Bioassay Jugs (2)	

Lab ID *	Location	Collection (begin)	Collection (end)	Bottle Group(s)**
	CF Hardee North Mine	☐ Comp ☒ Grab Date 2/7/11 Time 10:15 ☒ Eastern ☐ Central	Date Time ☐ Eastern ☐ Central	B
	Field ID: Equipment Blank	Tot Res Chlorine (mg/L)	Diss Oxygen (mg/L)	Storet Station Number
	Matrix (include type e.g. Salt, Fresh, etc): Fresh Water; Temp (C); pH	Sample Depth ☐ m ☐ ft	☐ Salinity (PPTh) ☐ Sp Conductance (umho/cm)	NPDES Number: FL0035271
	Latitude ☐ dd ☐ dms	Longitude ☐ dd ☐ dms	Comments	

Lab ID *	Location	Collection (begin)	Collection (end)	Bottle Group(s)**
		☐ Comp ☐ Grab Date Time ☐ Eastern ☐ Central	Date Time ☐ Eastern ☐ Central	
	Field ID	Tot Res Chlorine (mg/L)	Diss Oxygen (mg/L)	Storet Station Number
	Matrix (include type e.g. Salt, Fresh, etc); Temp (C); pH	Sample Depth ☐ m ☐ ft	☐ Salinity (PPTh) ☐ Sp Conductance (umho/cm)	NPDES Number
	Latitude ☐ dd ☐ dms	Longitude ☐ dd ☐ dms	Comments: 2 Bioassay Recd in Tox by JD 2.8.11 10:20	

Relinquished By:	Date/Time	Shipping Method:	Received By:	Date/Time	Relinquished By:	Date/Time	Received By:	Date/Time
[signature]	2/7/11 10:45	FedEx					[signature]	2/8/11 9207

* Shaded Areas for Lab use only.

** Please see reverse side for Bottle Group information.

last revised October 1, 2003

1 bottle Tues. 2/8/11

FDEP Biology Laboratory - Acute Bioassay Bench Sheets

Facility: CF Industries – Hardee

Address: 6209 N. Country Rd. 663

City: Wauchula County: Hardee

Contact/District: Jacki Champion / SW

NPDES Permit #: FL 0035271

LIMS Job #: TLH-2011-02-08-52 LIMS Sample #: 1328532

Lims Data Entry: MF 2-15-11 Data Entry Verification: BA 2.15.11

Sample Collection: Date: 2-7-11 Time: 10:30

Hold Time Start: Date: 2-7-11 Time: 10:30

Comments:

Initial sample handling:

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

PH adjustment: ☐ yes ☑ no Initial pH: ____ NaOH ____ N HCl ____ N ____ Drops mL Final pH: ____

Aeration: ☐ yes ☑ no Initial DO: ____ mg/L Final DO: ____ mg/L Duration: ____ minutes Rate: ~ ____ 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:
Kara Coy	KC
Brea Abernathy	BA
Mahabal Fairchild	MF
Jess David	JD
REVIEWER: Kim Hellen Liz	

Diluent/Control Batch#:	1.29.11	1.31.11							
Water Quality Parameters	20% DMW	Well Water	Moderately Hard Water	Salt Water ASW NSW Test 1	Salt Water ASW NSW Test 2	Other: DMW 2.2.11	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	n/a		
Lab Total Residual Cl_2 (mg/L):	<0.03	<0.03				<0.03	<0.03	BA/KC	KC
Alkalinity (mg/L as $CaCO_3$):	83	150				86	200	KC	KC
Hardness (mg/L as $CaCO_3$):	87	139				86	209	KC	KC
Total Ammonia (mg/L as N):	<0.017	<0.017				<0.017	<0.017	MF	KC
Conductivity: ☑ µmhos ☐ mmhos	172	263				180	410	JD/KC	KC
Salinity (ppth):	—	—				—	—	—	—

V 1.8 1/13/10

Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Survival Bench Sheet

LIMS Sample # 1328532   SOP#: TA 07 01   Test # 1 of 2

Test Species:
Batch #: 2-7-11   Age: ≤24 hr
- [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 # 1.29.11 (A) KC 2.2.11)
- [ ] ASW [x] 20% DMW
- [ ] MHW [ ] Well

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

Test Duration:
- [x] 48 hr
- [ ] 96 hr
- [ ] __hr

Chamber Size 30 ml
Test Volume: 10 ml

Test Started: Date 2-8-11 Time 1230

Test Ended: Date 2-10-11 Time 1300

Concentration	Replicate	Chamber #	0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
Ctrl	A	A	5	5	5			
	B	B	5	5	5			
	C	C	5	5	5			
	D	D	5	5	5			
100%	A	A	5	5	4 1D			
	B	B	5	5	5			
	C	C	5	5	5			
	D	D	5	5	5			
Checked or Verified by:			BA	BA	BA			

Food
- [x] YCT Batch# 11.7.10
- [x] P. subcapitata Batch# 10.22.10
- [ ] Artemia Lot#______

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

Temperature Range (°C)

Incubator # 2
min. 26.5 max. 27.6 mean —

Incubator #___
min.____ max.____ mean____

Waterbath #___
min.____ max.____ mean____

Room B246
min. 22.5 max. 24.0 mean —

Light intensity (ft. candles): 50-100

Photoperiod (hours): 16 light, 8 dark

Loaded/ Verified by: BA / MF

m = missing, d = dead, BR/AR Before/ After Renewal

Test Results:

Screening Tests: Report LC50 as ≥100%, =100%, or ≤100%.
% mortality in 100% sample: 5% LC50: >100%
Calculated LC50 (Definitive) 95% CI______ Method______

Comments:
(A) ~~1.29.11 used~~ on 2.8.11
~~2.2.11 used 2.10.11~~ KC

V. 2.5 1-13-10

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

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Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Survival Bench Sheet

LIMS Sample # 1328532 SOP#: TA 07 02 Test # 2 of 2

Test Species:
Batch #: 2.8.11 Age: 13days

- ☐ *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 # 2.31.11)
☐ ASW ☐ 20% DMW
☐ MHW ☒ Well

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

Test Duration:
☒ 48 hr
☐ 96 hr
☐ __hr

Chamber Size 1000 ml
Test Volume: 1000 ml

Test Started: Date 2-8-11 Time 1300

Test Ended: Date 2-10-11 Time 1300

Concentration	Replicate	Chamber #	0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
	A	G16	5	5	5			
ctrl	B	G17	5	5	5			
	C	G18	5	5	5			
	D	G19	5	5	5			
	A	G20	5	5	5			
100%	B	G21	5	5	5			
	C	G22	5	5	5			
	D	G23	5	5	5			
Checked or Verified by:			JA	JD	KC			

Food

- ☐ YCT Batch#______
- ☐ P. subcapitata Batch#______
- ☒ Artemia Lot# 23613

Feeding and Artemia Batch #

- ☒ 0 hr 2-4-11 ☒ Prior to Test
- ☐ 24 hr___
- ☐ 48 hr___ ☐ Prior to Renewal
- ☐ 72 hr___

Temperature Range (°C)

Incubator # 2
min. 26.5 max. 27.6 mean —

Incubator #___
min.____ max.____ mean____

Waterbath #___
min.____ max.____ mean____

Room B246
min. 22.5 max. 24.0 mean —

Light Intensity (ft. candles): 50-100

Photoperiod (hours): 16 light, 8 dark

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

Test Results:
Screening Tests: Report LC50 as >100%, =100%, or <100%.
% mortality in 100% sample: 0% LC50: >100%
Calculated LC50 (Definitive) 95% CI______ Method______

Comments:

V. 2.5 1-13-10

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

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Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Parameter Sheet

LIMS Sample # 1328532 Test # 1 of 2

Test Species:

- ☑ *Ceriodaphnia dubia*
- ☐ *Cyprinella leedsi*
- ☐ *Pimephales promelas*
- ☐ *Menidia beryllina*
- ☐ *Americamysis bahia*
- ☐ 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)
	M	0 hr	8.0	26.0	8.6	172	
	–	24 hr	–	–	–	–	
Ctrl	A	48 hr BR	7.8	24.0	7.2	193	
		48 hr AR					
		72 hr					
		96 hr					
	M	0 hr	7.8	26.0	8.4	412	
	–	24 hr	–	–	–	–	
100%	A	48 hr BR	8.3	24.0	7.4	436	
		48 hr AR					
		72 hr					
		96 hr					
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					
		0 hr					
		24 hr					
		48 hr BR					
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		72 hr					
		96 hr					
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					
		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: BA / KC
24 hr: –
48 hr BR: ~~BA~~ BA
48 hr AR:
72 hr:
96 hr:

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Appendix B. (continued)

FDEP Biology Laboratory - Bioassay Parameter Sheet

LIMS Sample #1328532 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	8.1	24.0	6.9	263	
	B	24 hr	8.0	24.2	7.5	275	
	C	48 hr BR	8.1	24.3	7.1	291	
		48 hr AR					
		72 hr					
		96 hr					
100%	A	0 hr	7.7	25.9	~~8.2~~ 7.4	417	
	B	24 hr	8.1	24.2	7.4	419	
	C	48 hr BR	8.3	24.1	7.4	439	
		48 hr AR					
		72 hr					
		96 hr					
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
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		72 hr					
		96 hr					
		0 hr					
		24 hr					
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					
		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: DD
24 hr: DD/KC
48 hr BR: KC
48 hr AR:
72 hr:
96 hr:

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V. 2.3 1-13-10

Appendix B. (continued)

Test ID: C F Industries Dates: Feb 8-10, 2011

METER / DEVICE	ID	SOP	Parameter	Method	v used
YSI Model 58 Dissolved Oxygen Meter	90H018262	TA06_04	Dissolved Oxygen	SM 4500 O G	✓
YSI DO Probe	5905	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 Salinity	SM 2510 B SM 2520 B	✓
Denver Model 225 ISE with ammonia probe	SN K01377	TA06_17	Ammonia	SM 4500 NH3 F	✓
Traceable Thermometer	#1-SN101686412 #2-SN221295527 #3-SN240116020 #4-SN80361795 (#5) SN61990624	TA06_05	Temperature	SM-2530B modified DEP SOP-001/01 FT1400	✓ EC #5
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	-	✓ MF 2-10-11
S-Weights	BIO-S-WEIGHTS-01	NA	Balance check	-	✓ MF 2-10-11

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)

02/10/11
- KC -

V 1.1 9/21/10 MF/jd

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Appendix C. SRT Bench Sheets and Statistical Analysis

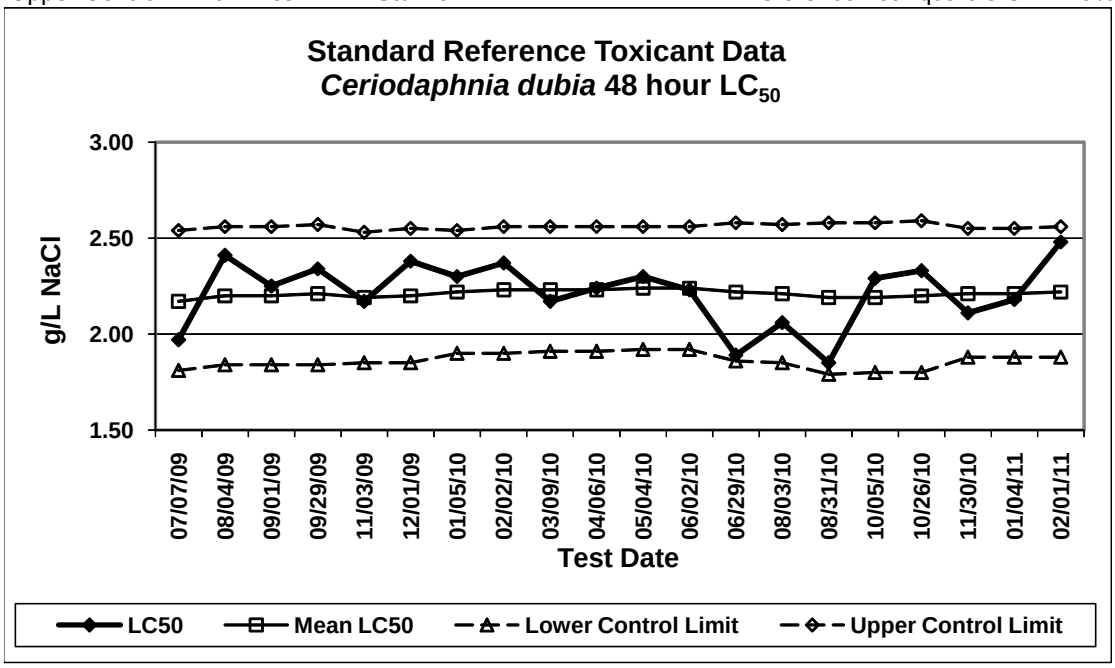
Florida Department of Environmental Protection
Bureau of Laboratories

Standard Reference Toxicant (SRT) Test Data - 48-hour Acute Toxicity
Ceriodaphnia dubia (water flea) - Sodium Chloride (NaCl)

Date	LC50	Mean LC50	Std Dev	Lower Control Limit	Upper Control Limit
07/07/09	1.97	2.17	0.18	1.81	2.54
08/04/09	2.41	2.20	0.18	1.84	2.56
09/01/09	2.25	2.20	0.18	1.84	2.56
09/29/09	2.34	2.21	0.18	1.84	2.57
11/03/09	2.17	2.19	0.17	1.85	2.53
12/01/09	2.38	2.20	0.17	1.85	2.55
01/05/10	2.30	2.22	0.16	1.90	2.54
02/02/10	2.37	2.23	0.17	1.90	2.56
03/09/10	2.17	2.23	0.16	1.91	2.56
04/06/10	2.24	2.23	0.16	1.91	2.56
05/04/10	2.30	2.24	0.16	1.92	2.56
06/02/10	2.23	2.24	0.16	1.92	2.56
06/29/10	1.89	2.22	0.18	1.86	2.58
08/03/10	2.06	2.21	0.18	1.85	2.57
08/31/10	1.85	2.19	0.20	1.79	2.58
10/05/10	2.29	2.19	0.20	1.80	2.58
10/26/10	2.33	2.20	0.20	1.80	2.59
11/30/10	2.11	2.21	0.17	1.88	2.55
01/04/11	2.18	2.21	0.17	1.88	2.55
02/01/11	2.48	2.22	0.17	1.88	2.56

Means are of the last 20 SRT tests.
Lower Control Limit = mean - 2 * Std Dev
Upper Control Limit = mean + 2 * Std Dev

CV = 7.68
EPA reference 75th quartile CV = 29%



Appendix C. (continued)

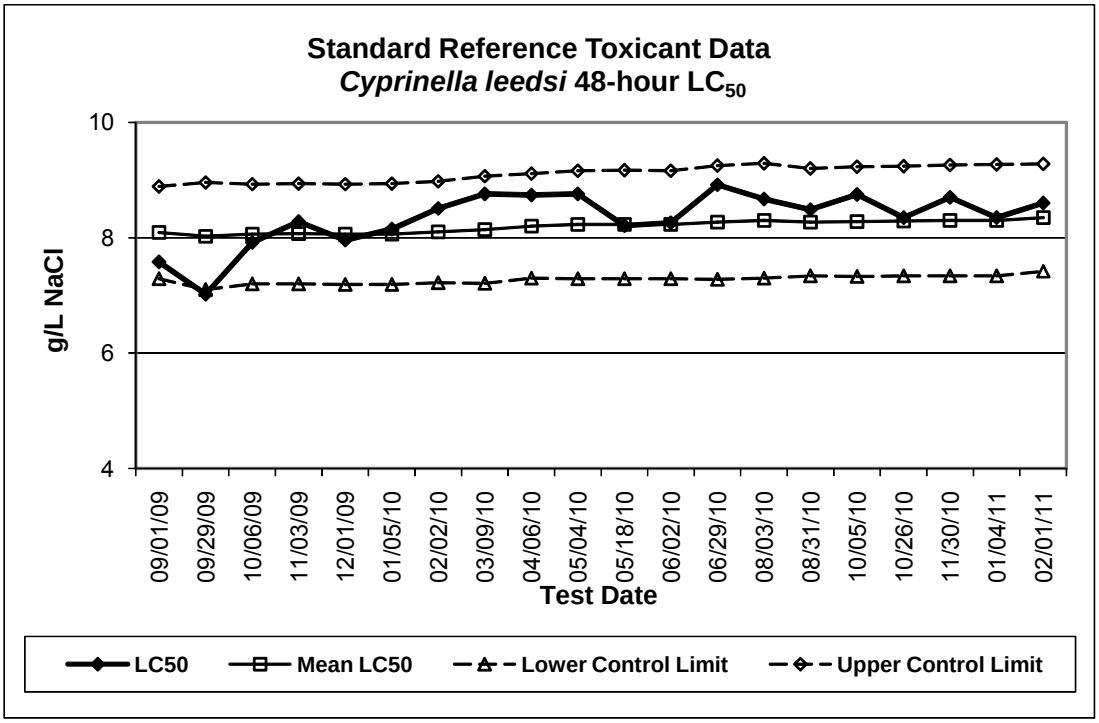
Florida Department of Environmental Protection
Bureau of Laboratories

Standard Reference Toxicant (SRT) Test Data - 48-hour Acute Toxicity
Cyprinella leedsi (bannerfin shiner) - Sodium Chloride (NaCl)

Date	LC50	Mean LC50	Std Dev	Lower Control Limit	Upper Control Limit
09/01/09	7.58	8.09	0.40	7.29	8.89
09/29/09	7.02	8.03	0.46	7.10	8.96
10/06/09	7.92	8.06	0.43	7.20	8.93
11/03/09	8.28	8.07	0.44	7.20	8.94
12/01/09	7.96	8.06	0.44	7.19	8.93
01/05/10	8.15	8.06	0.44	7.19	8.94
02/02/10	8.51	8.10	0.44	7.22	8.98
03/09/10	8.76	8.14	0.46	7.21	9.07
04/06/10	8.74	8.20	0.45	7.30	9.11
05/04/10	8.76	8.23	0.47	7.29	9.16
05/18/10	8.21	8.23	0.47	7.29	9.17
06/02/10	8.26	8.23	0.47	7.29	9.16
06/29/10	8.92	8.27	0.49	7.28	9.25
08/03/10	8.67	8.30	0.50	7.30	9.29
08/31/10	8.49	8.27	0.47	7.34	9.20
10/05/10	8.75	8.28	0.48	7.33	9.23
10/26/10	8.35	8.29	0.48	7.34	9.24
11/30/10	8.7	8.30	0.48	7.34	9.26
01/04/11	8.35	8.30	0.48	7.34	9.27
02/01/11	8.6	8.35	0.46	7.42	9.28

Means are of the last 20 SRT tests.
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 #: DRW 11.29.11
Toxicant/ Batch#: NaCl 11.31.11
Organism Batch: 2.1.11 Age: <24hr
Test Beginning: Date: 2.1.11 Time: 1300
Test Ending: Date: 2.5.11 Time: 1020

Water Quality Parameters:	Diluent	SOP	Initials
Total Residual Cl_2 (mg/L):	<0.03	TA06_22	KC
Alkalinity (mg/L as $CaCO_3$):	83	TA06_08	KC
Hardness (mg.L as $CaCO_3$):	87	TA06_09	KC
Total Ammonia (mg/L as N):	<0.017	TA06_17	MF
Conductivity: µmhos <mmhos>	0.173	TA06_25	BA
Salinity (ppth):	–	TA06_25	–

Food:	Batch
☑ YCT	11-17-10
☑ *P. subcapitata*	10-22-10
☐ 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
Temperature Range °C (if used)
Test incubated in: Incubator # 2 Waterbath # ____ Incubator # 2 min 26.3 max 28.1 mean ____
Light Intensity: 50-100 Ft. candles Waterbath # ___ min ____ max ____ mean ____
Photoperiod: 16 hours Light / 8 hours dark Room B246 min 25.1 max 25.3 mean ____

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 g/L	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	5	4 1D
	D	D	5	5	5	5	5	5
2.0	A	A	5	5	5	5	5	3 2D
	B	B	5	5	5	5	5	5
	C	C	5	5	5	5	5	5
	D	D	5	5	5	5	5	4 1D
2.5	A	A	5	5	2 3D	2	2	1 1D
	B	B	5	5	2 3D	2	1 1D	0 1D
	C	C	5	5	2 2D	2	0 2D	–
	D	D	5	5	3 2D	3	3	1 2D
3.0	A	A	5	4 1D	0 1D	–	–	–
	B	B	5	4 1D	1 3D	1	0 1D	–
	C	C	5	5	0 CD	–	–	–
	D	D	5	5	0 CD	–	–	–
4.0	A	A	5	0 5D	–	–	–	–
	B	B	5	0 5D	–	–	–	–
	C	C	5	0 5D	–	–	–	–
	D	D	5	0 5D	–	–	–	–
Organisms loaded by: BA		Checked by:	BA	BA	BA	BA	BA	MF

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

Investigators' Signatures
Bruce Nuckles
Tina Cox
Marshall Faircloth
James Daniels
Reviewer: Liz Miller

Comments:

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

Method: TSK

Statistics: LC50	95% confidence intervals Lower	Upper
48 hour: 2.48	2.36	2.61
96 hour: 2.19	2.08	2.32

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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: 2-1-11 Time: 13:00
Test Ending: Date: 2-5-11 Time: 1020

Test Type: ☐ non-renewal ☒ renewal

hr = hour AR = after renewal BR = before renewal

Concentration µg/L mg/L@	Replicate	Test Hour	pH (SU)	Temperature (°C)	Dissolved Oxygen (mg/L)	Conductivity ☐ µmhos ☒ mmhos	Salinity (ppth)
Control	M	0 hr	7.9	24.0	6.9	0.173	
	—	24 hr	—	—	BA —	—	
	A	48 hr BR	7.8	24.0	6.3 ~~7.8~~	0.191	
	M	48 hr AR	7.8	24.2	7.3	0.180	
	—	72 hr	—	—	—	—	
	A	96 hr	8.0	25.1	7.7	0.190	
1.5	M	0 hr	7.9	24.0	7.1	2.94	
	—	24 hr	—	—	—	—	
	A	48 hr BR	7.9	24.0	6.5	3.06	
	M	48 hr AR	7.8	24.2	7.3	2.91	
	—	72 hr	—	—	—	—	
	A	96 hr	8.0	25.1	7.6	2.96	
2.0	M	0 hr	8.0	24.0	6.9	~~4.67~~ 3.80	BA
	—	24 hr	—	—	—	—	
	A	48 hr BR	7.9	24.0	6.5	4.07	
	M	48 hr AR	7.9	24.3	7.3	3.77	
	—	72 hr	—	—	—	—	
	A	96 hr	8.0	25.2	7.8	3.84	
2.5	M	0 hr	8.0	24.0	7.0	4.67	
	—	24 hr	—	—	—	—	
	A	48 hr BR	7.9	24.0	6.8	4.84	
	M	48 hr AR	7.9	24.2	7.3	4.73	
	—	72 hr	—	—	—	—	
	A	96 hr	8.0	25.0	8.0	4.91	
3.0	M	0 hr	8.0	24.0	7.1	5.53	
	—	24 hr	—	—	—	—	
	A	48 hr BR	8.0	24.0	7.1	5.87	
	M	48 hr AR	8.0	24.1	7.4	5.13	
	B	72 hr	7.7	24.0	6.9	5.45	
	—	96 hr	—	—	—	—	
4.0	M	0 hr	8.0	24.0	6.7	7.21	
	A	24 hr	7.8	24.0	7.0	7.20	
	—	48 hr BR	—	—	—	—	
	—	48 hr AR	—	—	—	—	
	—	72 hr	—	—	—	—	
	—	96 hr	—	—	—	—	

Investigators Signatures: Sue [illegible], Kara [illegible], Marshall Brimfield, James Daniel

Reviewed By: Liz Miller

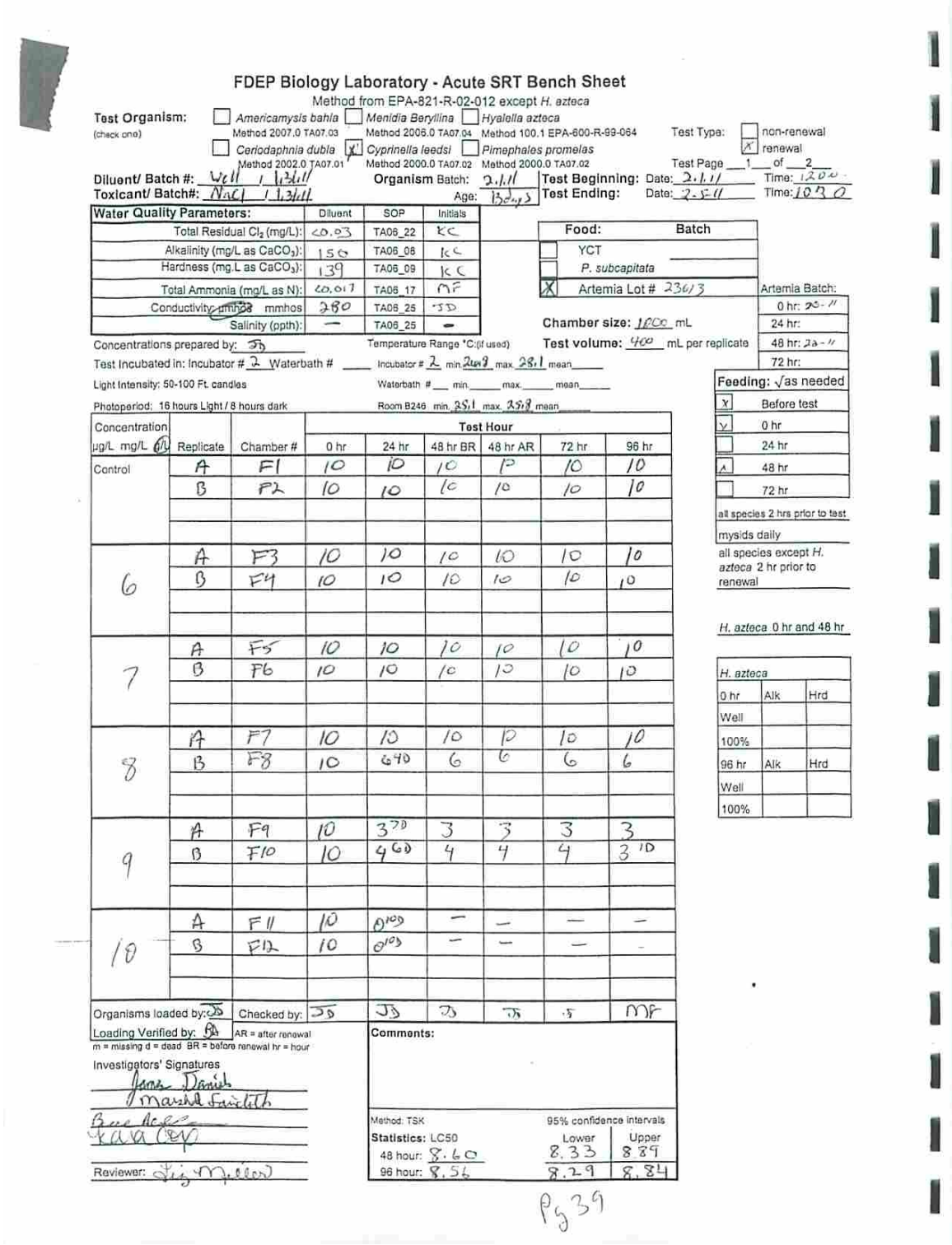
Measured by: 0 hr: BA; 24 hr: BA; 48 hr BR: BA; 48 hr AR: BA; 72 hr: BA; 96 hr: BA

Measure/record salinity for marine tests

Comments: Thermometers used 0-96 hr - EC#5

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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 / 1.13.11 **Organism Batch:** 2.1.11 **Test Beginning:** Date: 2.1.11 Time: 1200

Toxicant/ Batch#: NaCl / 1.13.11 Age: 30 hrs **Test Ending:** Date: 2.5.11 Time: 1030

Water Quality Parameters:	Diluent	SOP	Initials
Total Residual Cl_2 (mg/L):	<0.03	TA06_22	KC
Alkalinity (mg/L as $CaCO_3$):	150	TA06_08	KC
Hardness (mg.L as $CaCO_3$):	139	TA06_09	KC
Total Ammonia (mg/L as N):	<0.017	TA06_17	MF
Conductivity ~~umhos~~ mmhos	280	TA06_25	SD
Salinity (ppth):	—	TA06_25	—

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

Artemia Batch:
- 0 hr: 2.5-11
- 24 hr:
- 48 hr: 2.8-11
- 72 hr:

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

Concentrations prepared by: SD

Test Incubated in: Incubator # 2 Waterbath # ____ Temperature Range °C:(if used) Incubator # 2 min. 28.0 max. 28.1 mean

Light Intensity: 50-100 Ft. candles Waterbath # ___ min. ___ max. ___ mean ___

Photoperiod: 16 hours Light / 8 hours dark Room B246 min. 28.1 max. 28.3 mean

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

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

Concentration µg/L mg/L	Replicate	Chamber #	Test Hour 0 hr	24 hr	48 hr BR	48 hr AR	72 hr	96 hr
Control	A	F1	10	10	10	10	10	10
	B	F2	10	10	10	10	10	10
6	A	F3	10	10	10	10	10	10
	B	F4	10	10	10	10	10	10
7	A	F5	10	10	10	10	10	10
	B	F6	10	10	10	10	10	10
8	A	F7	10	10	10	10	10	10
	B	F8	10	6d0	6	6	6	6
9	A	F9	10	3d0	3	3	3	3
	B	F10	10	4d0	4	4	4	3d0
10	A	F11	10	0d03	—	—	—	—
	B	F12	10	0d03	—	—	—	—
Organisms loaded by: SD		Checked by:	SD	JD	JD	JD	S	MF
Loading Verified by: BA		AR = after renewal						

m = missing d = dead BR = before renewal hr = hour

Comments:

Investigators' Signatures

Jane Daniels

Marshall Faircloth

Bree Acree

Kara Coy

Reviewer: Liz Miller

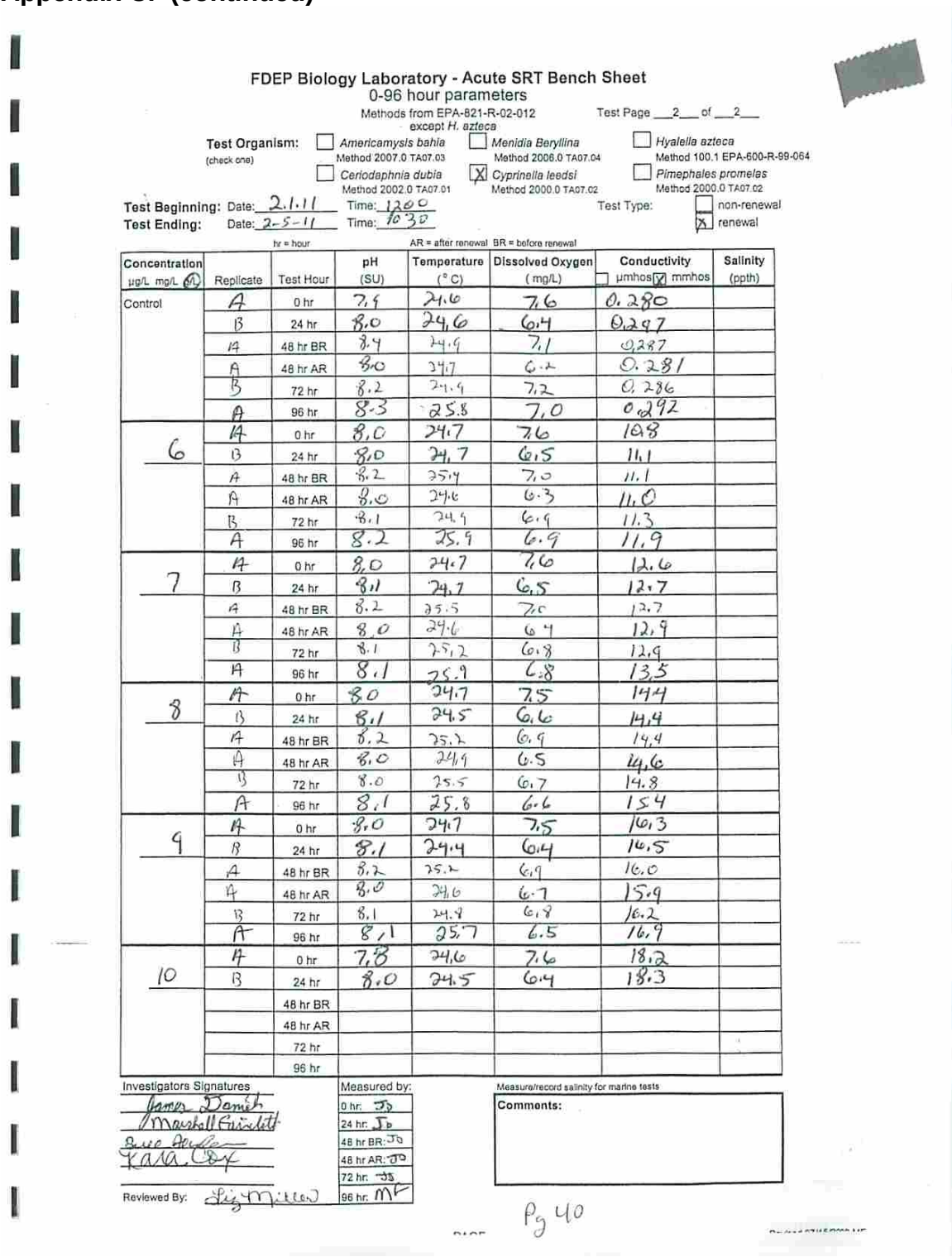
Method: TSK

Statistics: LC50

	95% confidence intervals Lower	Upper
48 hour: 8.60	8.33	8.89
96 hour: 8.56	8.29	8.84

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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: 2.1.11 Time: 1200
Test Ending: Date: 2-5-11 Time: 1030
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)	Conductivity ☐ µmhos ☒ mmhos	Salinity (ppth)
Control	A	0 hr	7.9	24.6	7.6	0.280	
	B	24 hr	8.0	24.6	6.4	0.297	
	A	48 hr BR	8.4	24.9	7.1	0.287	
	A	48 hr AR	8.0	24.7	6.2	0.281	
	B	72 hr	8.2	24.9	7.2	0.286	
	A	96 hr	8.3	25.8	7.0	0.292	
6	A	0 hr	8.0	24.7	7.6	108	
	B	24 hr	8.0	24.7	6.5	11.1	
	A	48 hr BR	8.2	25.4	7.0	11.1	
	A	48 hr AR	8.0	24.6	6.3	11.0	
	B	72 hr	8.1	24.9	6.9	11.3	
	A	96 hr	8.2	25.9	6.9	11.9	
7	A	0 hr	8.0	24.7	7.6	12.6	
	B	24 hr	8.1	24.7	6.5	12.7	
	A	48 hr BR	8.2	25.5	7.0	12.7	
	A	48 hr AR	8.0	24.6	6.4	12.9	
	B	72 hr	8.1	25.2	6.8	12.9	
	A	96 hr	8.1	25.9	6.8	13.5	
8	A	0 hr	8.0	24.7	7.5	144	
	B	24 hr	8.1	24.5	6.6	14.4	
	A	48 hr BR	8.2	25.2	6.9	14.4	
	A	48 hr AR	8.0	24.9	6.5	14.6	
	B	72 hr	8.0	25.5	6.7	14.8	
	A	96 hr	8.1	25.8	6.6	15.4	
9	A	0 hr	8.0	24.7	7.5	16.3	
	B	24 hr	8.1	24.4	6.4	16.5	
	A	48 hr BR	8.2	25.2	6.9	16.0	
	A	48 hr AR	8.0	24.6	6.7	15.9	
	B	72 hr	8.1	24.8	6.8	16.2	
	A	96 hr	8.1	25.7	6.5	16.9	
10	A	0 hr	7.8	24.6	7.6	18.2	
	B	24 hr	8.0	24.5	6.4	18.3	
		48 hr BR					
		48 hr AR					
		72 hr					
		96 hr					

Investigators Signatures
James Daniels
Marshall Faircloth
Bruce Hayden
Kara Cox

Reviewed By: Liz Miller

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

Measure/record salinity for marine tests

Comments:

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Appendix C. (continued)

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

DATE: 2-1-11 DURATION: 48 h TOXICANT: NaCl
SPECIES: *Ceriodaphnia dubia*

RAW DATA: Concentration (g/L)	Number Exposed	Mortalities
.00	20	0
1.50	20	0
2.00	20	0
2.50	20	11
3.00	20	19
4.00	20	20

SPEARMAN-KARBER TRIM: .00%

SPEARMAN-KARBER ESTIMATES: LC50: 2.48
95% LOWER CONFIDENCE: 2.36
95% UPPER CONFIDENCE: 2.61

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

DATE: 2-1-11 DURATION: 48 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	4
9.00	20	13
10.00	20	20

SPEARMAN-KARBER TRIM: .00%

SPEARMAN-KARBER ESTIMATES: LC50: 8.60
95% LOWER CONFIDENCE: 8.33
95% UPPER CONFIDENCE: 8.89

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: 1/31/11

Ship to: FDEP

P.O. No: _____

Species	Quantity	Age	Brood/Lot Number	Temp. (°C)	pH (S.U.)	Salinity (‰)
Mysidopsis bahia	150 ac	4 days	MS 110127	25	8.0	20.0
Menidia beryllina	150 ac	12 days	SS 110119	25	8.0	20.0
						Hardness mg/L
Cyprinella leedsi	150 ac	12 days	CL 110119	25	7.8	80
Pimephales promelas	150 ac	7 days	FM 110124	25	7.8	80
Ceriodaphnia dubia						
Daphnia magna						
YCT						
P. subcapitata						

Packed by: PM

Shipped Via: Fed Ex

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: 2/7/11

Ship to: FDEP

P.O. No: _____

Species	Quantity	Age	Brood/Lot Number	Temp. (°C)	pH (S.U.)	Salinity (‰)
Mysidopsis bahia	150	4 DAYS	MS 110203	25	8.0	20
Menidia beryllina						
						Hardness mg/L
Cyprinella leedsi	100	12 days	CL 110126	25	7.8	80
Pimephales promelas	150 ac	8 days	FM 110130	25	7.8	80
Ceriodaphnia dubia						
Daphnia magna						
YCT						
P. subcapitata						

Packed by: PM

Shipped Via: Fed Ex

Notes: _____

Thank you for your order.

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Appendix E. Chemical Analyses performed on the effluent from the CF Industries, Inc., Hardee Complex 1 (North Pasture Mine) Plant Outfall D-003, sampled on February 7, 2011.

Date Sampled	Field ID	Analysis Group	Component	Result	Remark	Units	MDL	PQL
2/7/2011 10:30	Effluent	AGP/LN	Algal Growth Potential	5.75		mg DryWt/L	0.3	0.9
2/7/2011 10:30	Effluent	Chlorophyll/Grain Size/BOD	Chlorophyll-a, Corrected	2.9		ug/L	0.68	2.1
2/7/2011 10:30	Effluent	Chlorophyll/Grain Size/BOD	Phaeophytin-a	2.7		ug/L	0.49	1.5
2/7/2011 10:30	Effluent	Chlorophyll/Grain Size/BOD	Biochemical Oxygen Demand-5 Day,N-Inhib	2		mg/L	0.2	2
2/7/2011 10:30	Effluent	Toxicology	Bioassay-Acute-Screen-FW-C.dubia, LC50	100	L	LC50		
2/7/2011 10:30	Effluent	Toxicology	Bioassay-Acute-Screen-FW-Fish, LC50	100	L	LC50		
2/7/2011 10:30	Effluent	Metals	Selenium	0.35	I	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Metals	Cadmium	0.05	U	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Metals	Nickel	0.28	I	ug/L	0.25	1
2/7/2011 10:30	Effluent	Metals	Zinc	5	U	ug/L	5	20
2/7/2011 10:30	Effluent	Metals	Silver	0.025	U	ug/L	0.025	0.1
2/7/2011 10:30	Effluent	Metals	Lead	0.2	U	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Metals	Copper	0.5	U	ug/L	0.5	1
2/7/2011 10:30	Effluent	Metals	Chromium	0.3	U	ug/L	0.3	1.2
2/7/2011 10:30	Effluent	Metals	Aluminum	71	I	ug/L	60	240
2/7/2011 10:30	Effluent	Metals	Magnesium	24.1		mg/L	0.04	0.16
2/7/2011 10:30	Effluent	Metals	Iron	160		ug/L	30	120
2/7/2011 10:30	Effluent	Metals	Arsenic	0.92	I	ug/L	0.25	1
2/7/2011 10:30	Effluent	Metals	Calcium	43.9		mg/L	0.075	0.3
2/7/2011 10:30	Effluent	Metals	Sodium	10.1		mg/L	0.5	2
2/7/2011 10:30	Effluent	Nutrients	Turbidity	1.6	A	NTU	0.1	0.1
2/7/2011 10:30	Effluent	Nutrients	TSS	4	U	mg/L	4	16
2/7/2011 10:30	Effluent	Nutrients	O-Phosphate-P	0.6		mg P/L	0.02	0.05
2/7/2011 10:30	Effluent	Nutrients	Ammonia-N	0.032		mg N/L	0.01	0.02
2/7/2011 10:30	Effluent	Nutrients	Kjeldahl Nitrogen	1.1		mg N/L	0.08	0.2
2/7/2011 10:30	Effluent	Nutrients	NO2NO3-N	0.009	I	mg N/L	0.004	0.01
2/7/2011 10:30	Effluent	Nutrients	Total-P	0.69		mg P/L	0.005	0.05
2/7/2011 10:30	Effluent	Nutrients	Chloride	13		mg Cl/L	0.2	0.5
2/7/2011 10:30	Effluent	Nutrients	Sulfate	20		mg SO4/L	0.2	0.5
2/7/2011 10:30	Effluent	Nutrients	Fluoride	1.2		mg F/L	0.035	0.1
2/7/2011 10:30	Effluent	Pesticides	Ametryn	0.05	UJ	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Atrazine	0.05	UJ	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Ethion	0.05	U	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Ethoprop	0.099	U	ug/L	0.099	0.4

Appendix E.

Date Sampled	Field ID	Analysis Group	Component	Result	Remark	Units	MDL	PQL
2/7/2011 10:30	Effluent	Pesticides	Naled	0.79	UJ	ug/L	0.79	3.2
2/7/2011 10:30	Effluent	Pesticides	Alachlor	0.59	UJ	ug/L	0.59	2.4
2/7/2011 10:30	Effluent	Pesticides	Chlorpyrifos Ethyl	0.05	U	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Parathion Ethyl	0.15	U	ug/L	0.15	0.6
2/7/2011 10:30	Effluent	Pesticides	Parathion Methyl	0.099	U	ug/L	0.099	0.4
2/7/2011 10:30	Effluent	Pesticides	Bromacil	0.2	UJ	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Pesticides	Metalaxyl	0.25	U	ug/L	0.25	1
2/7/2011 10:30	Effluent	Pesticides	Phorate	0.05	U	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Metribuzin	0.099	UJ	ug/L	0.099	0.4
2/7/2011 10:30	Effluent	Pesticides	Prometryn	0.15	UJ	ug/L	0.15	0.6
2/7/2011 10:30	Effluent	Pesticides	Azinphos Methyl	0.2	U	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Pesticides	Butylate	0.2	UJ	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Pesticides	Diazinon	0.05	U	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Fenamiphos	0.2	U	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Pesticides	Hexazinone	0.099	U	ug/L	0.099	0.4
2/7/2011 10:30	Effluent	Pesticides	Metolachlor	0.5	UJ	ug/L	0.5	2
2/7/2011 10:30	Effluent	Pesticides	Malathion	0.15	U	ug/L	0.15	0.6
2/7/2011 10:30	Effluent	Pesticides	Simazine	0.05	U	ug/L	0.05	0.2
2/7/2011 10:30	Effluent	Pesticides	Fonofos	0.099	U	ug/L	0.099	0.4
2/7/2011 10:30	Effluent	Pesticides	Chlorpyrifos Methyl	0.099	U	ug/L	0.099	0.4
2/7/2011 10:30	Effluent	Pesticides	Mevinphos	0.2	U	ug/L	0.2	0.8
2/7/2011 10:30	Effluent	Pesticides	Norflurazon	0.15	U	ug/L	0.15	0.6
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Benzo(g,h,i)perylene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Isophorone	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Butyl benzyl phthalate	4.9	U	ug/L	4.9	20
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Fluoranthene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Phenanthrene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Bis(2-ethylhexyl)phthalate	15	U	ug/L	15	59
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Indeno(1,2,3-cd)pyrene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Benzo(b)fluoranthene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	3,3'-Dichlorobenzidine	39	UJ	ug/L	39	160
2/7/2011 10:30	Effluent	Priority Organic Pollutants	N-Nitrosodimethylamine	2	U	ug/L	2	7.8
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Acenaphthene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2-Methyl-4,6-dinitrophenol	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	1,2,4-Trichlorobenzene	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2,4-Dimethylphenol	9.8	U	ug/L	9.8	39

Appendix E.

Date Sampled	Field ID	Analysis Group	Component	Result	Remark	Units	MDL	PQL
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2,4-Dinitrotoluene	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	4-Chloro-3-methylphenol	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Acenaphthylene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Anthracene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Benzo(k)fluoranthene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Di-n-butyl phthalate	4.9	U	ug/L	4.9	20
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Diethyl phthalate	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Hexachlorocyclopentadiene	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Hexachloroethane	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Naphthalene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Phenol	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Bis(2-chloroethoxy)methane	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	N-Nitrosodiphenylamine/ Diphenylamine	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Pentachlorophenol	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2-Chlorophenol	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2-Nitrophenol	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	4-Chlorophenyl phenyl ether	2	U	ug/L	2	7.8
2/7/2011 10:30	Effluent	Priority Organic Pollutants	4-Nitrophenol	15	U	ug/L	15	59
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Benzo(a)anthracene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Benzo(a)pyrene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Chrysene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Di-n-octyl phthalate	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Dimethyl phthalate	9.8	U	ug/L	9.8	39
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Fluorene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Hexachlorobenzene	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2,4,6-Trichlorophenol	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2,4-Dichlorophenol	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2,4-Dinitrophenol	15	U	ug/L	15	59
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2,6-Dinitrotoluene	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Bis(2-chloroethyl)ether	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Bis(2-chloroisopropyl)ether	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Hexachlorobutadiene	2.9	U	ug/L	2.9	12
2/7/2011 10:30	Effluent	Priority Organic Pollutants	N-Nitrosodi-n-propylamine	2	U	ug/L	2	7.8
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Nitrobenzene	2	U	ug/L	2	7.8
2/7/2011 10:30	Effluent	Priority Organic Pollutants	4-Bromophenyl phenyl ether	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Benzidine	98	UJ	ug/L	98	390

Appendix E.

Date Sampled	Field ID	Analysis Group	Component	Result	Remark	Units	MDL	PQL
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Dibenzo(a,h)anthracene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	Pyrene	0.2	U	ug/L	0.2	0.78
2/7/2011 10:30	Effluent	Priority Organic Pollutants	2-Chloronaphthalene	0.98	U	ug/L	0.98	3.9
2/7/2011 10:30	Effluent	Field Parameter	Specific Conductance	430		umhos/cm		
2/7/2011 10:30	Effluent	Field Parameter	pH	7.62				
2/7/2011 10:30	Effluent	Field Parameter	Temperature	20.16		C		
2/7/2011 10:30	Effluent	Field Parameter	Dissolved Oxygen	8.82		mg/L		
2/7/2011 10:15	Blank	Metals	Magnesium	0.04	U	mg/L	0.04	0.16
2/7/2011 10:15	Blank	Metals	Copper	0.5	U	ug/L	0.5	1
2/7/2011 10:15	Blank	Metals	Zinc	5	U	ug/L	5	20
2/7/2011 10:15	Blank	Metals	Aluminum	60	U	ug/L	60	240
2/7/2011 10:15	Blank	Metals	Nickel	0.25	U	ug/L	0.25	1
2/7/2011 10:15	Blank	Metals	Sodium	0.5	U	mg/L	0.5	2
2/7/2011 10:15	Blank	Metals	Cadmium	0.05	U	ug/L	0.05	0.2
2/7/2011 10:15	Blank	Metals	Arsenic	0.25	U	ug/L	0.25	1
2/7/2011 10:15	Blank	Metals	Silver	0.025	U	ug/L	0.025	0.1
2/7/2011 10:15	Blank	Metals	Selenium	0.2	U	ug/L	0.2	0.8
2/7/2011 10:15	Blank	Metals	Lead	0.2	U	ug/L	0.2	0.8
2/7/2011 10:15	Blank	Metals	Calcium	0.075	U	mg/L	0.075	0.3
2/7/2011 10:15	Blank	Metals	Iron	30	U	ug/L	30	120
2/7/2011 10:15	Blank	Metals	Chromium	0.3	U	ug/L	0.3	1.2
2/7/2011 10:15	Blank	Nutrients	O-Phosphate-P	0.004	U	mg P/L	0.004	0.01
2/7/2011 10:15	Blank	Nutrients	Turbidity	0.1	U	NTU	0.1	0.1
2/7/2011 10:15	Blank	Nutrients	Fluoride	0.035	U	mg F/L	0.035	0.1
2/7/2011 10:15	Blank	Nutrients	Kjeldahl Nitrogen	0.08	U	mg N/L	0.08	0.2
2/7/2011 10:15	Blank	Nutrients	Sulfate	0.2	U	mg SO4/L	0.2	0.5
2/7/2011 10:15	Blank	Nutrients	Ammonia-N	0.01	U	mg N/L	0.01	0.02
2/7/2011 10:15	Blank	Nutrients	Chloride	0.2	U	mg Cl/L	0.2	0.5
2/7/2011 10:15	Blank	Nutrients	NO2NO3-N	0.004	U	mg N/L	0.004	0.01
2/7/2011 10:15	Blank	Nutrients	Total-P	0.004	U	mg P/L	0.004	0.01

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: Jacki Champion	Contact Number: 813-632-7600 X133
Facility Name (as it appears on permit): CF Industries, Inc. Hardee Complex I (North Pasture Mine)	Former Names:
NPDES Permit Number: FL0035271	Permit Expiration Date: 11/16/11
Physical Address: 6209 N. County Road 663 Wauchula, FL	
District: Bureau of Mining and Minerals Regulation-Phosphate Management located in the SW district	County: Hardee
Function of Facility: Former Phosphate Mine	
Facility Type: Phosphate Mine	Permitted Capacity (MGD):
Mean Flow for previous 12 months for all EXTERNAL outfalls listed in permit (list date range including months): 0.303 MGD from 11/09 through 11/10	
If applicable, Mean Flow for previous 12 months for REUSE outfall(s) (list date range including months):	
Description of type of Discharge: ☐ Continuous ☒ Intermittent ☐ Seasonal ☒ Rainfall Dependent ☐ Other, please specify	
Description of Sampling Location(s) - actual permit designation of permitted sampling point(s): EFF-003 discharge at Outfall D-003	
Description of Permitted Outfall(s): Half Moon Riser Weir Structure	
Description of Treatment Process (if multiple discharge points, include a detailed map or diagram of facility): Clarification in the clay settling area	

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PART I: Continued										
List current Effluent Limits of Outfall(s) to be Sampled: This should include any AO or CO limits the facility may be under (Leave cell blank if not applicable)										
			Effluent Limitations				Monitoring Requirements			
Parameter	Units	Max/Min	Annual Average	Monthly Average	Weekly Average	Single Sample	Monitoring Frequency	Sample Type	Monitoring Location Site Number	Notes

water body.

	Discharge Limitations			Monitoring Requirements		
Parameters (units)	**Daily Minimum**	**Monthly Average**	**Daily Maximum**	**Monitoring Frequency**	**Sample Type**	**Sample Point**
Flow (MGD)	N/A	Report	Report	Continuous	Recorder	EFF-003
Solids, Total Suspended (MG/L)	N/A	30	60	Weekly	Grab	EFF-003
Solids, Fixed Suspended (MG/L)*	N/A	12	25	Weekly	Grab	EFF-003

Appendix F. (continued)

Parameter (Units)	Minimum	Average	Maximum	Frequency	Type	Point
Phosphorus, Total (as P) (MG/L)**	N/A	3.0	5.0	Weekly	Grab	EFF-003
Phosphorus, Total (as P) (LBS/DAY)***	N/A	Report	See Condition I.A.8.	Weekly	Calculated	EFF-003
Nitrogen, Total (as N) (MG/L)***	N/A	Report	Report	Weekly	Grab	EFF-003
Nitrogen, Total (as N) (LBS/DAY)***	N/A	Report	See Condition I.A.8.	Weekly	Calculated	EFF-003
Oxygen, Dissolved (DO) (MG/L)	5.0	Report	N/A	Weekly	Grab	EFF-003
pH (SU)	6.0	Report	8.5	Weekly	Grab	EFF-003
Specific Conductance (UMHO/CM)	N/A	Report	See Condition I.A.4.	Weekly	Grab	EFF-003
Specific Conductance (background) (UMHO/CM)	N/A	Report	See Condition I.A.4.	Weekly when discharging	Grab	SWB-003
Temperature (C), Water (DEG.C)	Report	Report	Report	Weekly	Grab	EFF-003
Turbidity (NTUs)	N/A	Report	See Condition I.A.9.	Weekly	Grab	EFF-003
Turbidity (background) (NTUs)	N/A	Report	See Condition I.A.9.	Weekly when discharging	Grab	SWB-003
Chlorophyll-a (MG/L)	N/A	N/A	Report	Monthly	Grab	EFF-003
Whole Effluent Toxicity - Acute	See Permit Condition I.A.3.			Annually	Grab	EFF-003

Appendix F. (continued)

PART I: Continued	
Receiving Waters: Payne Creek a tributary of the Peace River	**Classification (indicate whether fresh or marine): Class III Fresh**
Toxicity Test Requirements (routine and/or additional test language, test species, salinity adjustment, etc.): **Annual acute screening test, fresh water**	
Administrative or Consent Orders (date executed, specific requirements, interim limit dates-if applicable, etc.): None	
Facility Mixing Zone Details (if applicable):none	
List Permit Violations (DMR data) and plant upsets that occurred at the plant within the last year: none	

Date	Parameter and Units	Limit	Result

Describe previous FYI and WQBEL study: In the previous FYI Aluminum, arsenic and iron were detected in the effluent at low levels. Oil and grease was detected during the FYI but subsequent sampling did not detect oil and grease. The macroinvertebrate and phytoplankton communities were similar in the test and control sites. Due to the low discharge volume and inconsistent flows in stream sampling is not being done at this time.
Describe previous or current Enforcement Actions (including WLs):   **None**
Describe any violations or problems noted in previous inspection: **In my previous inspection at the facility no deficiencies were noted.**
Discuss whether DMR trends within previous five years are improving or declining: static

Appendix F. (continued)

Additional Comments (this should include a trend of the Effluent, i.e. if the facility is now Reuse): This facility has not operated since the early 1990s.	
PART II: Sampling Event Information	
Date(s) sample collected: 2/7/11	Sampler(s): Jacki Champion; Melani Dickenson; Scott Rose
Temperature (Deg C):20.16	pH (SU):7.62
Dissolved Oxygen (mg/L):8.82[J]	Conductivity (umhos/cm):430
Total Residual Chlorine (mg/L):na	Total Residual Chlorine - after Disinfection (mg/L):na
Method of Chlorination: na	Method of De-chlorination: na
Flow During Sample Collection for each outfall(MGD): 0.432	
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): 0.303 MGD from 11/09 through 11/10 (waiting for December and January DMR flow data)	
Describe Discharge during Sampling Event: ☒ Continuous ☐ Intermittent ☐ Seasonal ☐ Rainfall Dependent ☐ Other, please specify	

Appendix F. (continued)

Describe any conditions, difficulties or abnormal events that occurred during the sample collection and any other additional comments here:
[J]Dissolved Oxygen post-calibration was slightly high (106%). Very windy out during sampling.

Appendix G. EPA End Sheet

The Bioassay of CF Industries, Inc., Hardee Complex 1 (North Pasture Mine) efffluent sampled on February 7, 2011. NPDES #FL0035271.

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 0 3 5 2 7 1 11	12 1 1 0 2 0 7	18 B	19 S	20 2

Remarks

66

The Priority Pollutants Analysis of CF Industries, Inc., Hardee Complex 1 (North Pasture Mine) efffluent sampled on February 7, 2011. NPDES #FL0035271.

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 0 3 5 2 7 1 11	12 1 1 0 2 0 7	18 X	19 S	20 2

Remarks

66

Biological Analyses of CF Industries, Inc., Hardee Complex 1 (North Pasture Mine) efffluent sampled on February 7, 2011. NPDES #FL0035271.

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 0 3 5 2 7 1 11	12 1 1 0 2 0 7	18 S	19 S	20 2

Remarks

66