

Bioassays of

Piney Point Phosphates, Inc.

Manatee County

NPDES #FL0000124

Sampled March 22, 2004

September 2004

Biology Section
Bureau of Laboratories
Division of Resource Assessment and Management

Quality Manual No. 870346G
NELAC Certification No. E31780

Piney Point Phosphates, Inc., 13300 Highway 41 North, Palmetto, Manatee County, Florida, NPDES #FL0000124. Effluent samples for this facility were collected on March 22, 2004.

Introduction

Piney Point Phosphates previously operated a phosphate fertilizer manufacturing complex in Manatee County Florida. After Piney Point Phosphates declared bankruptcy, a Receiver was appointed to maintain the phosphogypsum stack system to prevent a release of untreated wastewater. In an effort to close the stack system, the FDEP has authorized discharge of wastewater after RO treatment. In order to expedite the closing of the stack system, alternative treatments of wastewater for discharge to surface waters are being explored at the site. These toxicity tests were used to determine whether effluent treated with a two-stage liming process, followed by break-point chlorination, then dechlorination would be adequate for discharge. Wastewater treated in this manner is not currently being discharged pending the results of further testing (facility information provided by J. Champion, FDEP, Phosphate Management, Tampa).

Methods

Samples were collected from by Jacquelyn Champion (FDEP Phosphate Management, Tampa) following DEP-SOP-001/01 FS 2400 Wastewater Sampling. The break point chlorination sample was collected from a batch processed at the reverse osmosis plant. The reverse osmosis control sample was a grab sample of the water discharged from the reverse osmosis plant at the facility. The salt control group consisted of a series of test chambers filled with artificial seawater (40 Fathoms ^{tm}). Additionally, the reverse osmosis water sample was tested at 100% concentration. The reverse osmosis sample was used as a diluent in both 96-hour acute definitive tests. The comparison between the salt control and the reverse osmosis 100% concentration can be interpreted as a 96-hour acute screen of the reverse osmosis water sample. All DEP SOP's are available on the web at: http://www.floridadep.org/labs/qa/sops.htm.

The toxicity tests discussed in this report were performed following internal DEP SOP's TA07_03,TA07_04, and TA08_01 through TA08_06. All internal DEP SOP's are available on the web at: http://www.floridadep.org/labs/cgi-bin/sop/biosop.asp.

Toxicity Test Results

Tests were performed March 23 – 27, 2004.

Americamysis bahia 96-hr acute definitive bioassay – LC_{50}=65.74%, 100% mortality in 100% sample at 48 hours

Menidia beryllina 96-hr acute definitive bioassay – LC_{50}=70.09%, 100% mortality in 100% sample at 48 hours

Both bioassays of the break point chlorinated sample showed acute toxicity. The comparison of the salt control with the reverse osmosis water sample indicated acute toxicity (40% vs. 5% mortality) of the reverse osmosis water to the mysid, *A. bahia*, but not for the fish, *M. beryllina*.

Due to a drop in the dissolved oxygen in the 100% sample concentrations in both tests at 24 hours, all test vessels were aerated for the remainder of the test. The pH of the 100% sample concentrations in both tests also dropped below acceptable ranges at 48 hours.

See Table 1 for bioassay bench sheets.

Algal Growth Potential

Algal growth potential samples were not collected.

Chemistry Result

Total residual chlorine (TRC) was not detected in the Break Point bioassay sample in the laboratory; however, TRC was detected in the RO sample at a concentration of 0.25 mg/L, exceeding the Class III Fresh Water Quality Criterion of ≤ 0.01 mg/l (62-302.530 (19) F.A.C.). Total ammonia was detected in both the unpreserved Break Point bioassay sample and the RO sample in the laboratory at a concentration of 0.270 mg/L and 0.728 mg/L, respectively.

Conclusion

The water samples collected from a batch of break point chlorinated effluent on March 22, 2004, showed acute toxicity to both test species during the 96-hour acute definitive bioassays. The reverse osmosis water sample was acutely toxic to the mysid during a 96-hour acute screening bioassay. Samples for chemical analyses were not collected in conjunction with the bioassay samples.

Previous tests for this facility suggest that ion-imbalance may have contributed to the *A. bahia* mortality. The seawater ion-imbalance toxicity testing protocol would need to be performed to confirm ion-imbalance as the sole cause of the toxicity. Samples for chemical analyses were not collected in conjunction with the bioassay samples; therefore, unidentified constituents in the sample cannot be ruled out as contributing to the observed mortality.

Table 1. Data recorded during the 96-hour acute definitive bioassays of a sample of effluent from Piney Point Phosphates, Inc., 13300 Highway 41 North, Palmetto, Manatee County, Florida, NPDES# FL0000124, performed from 23 to 27 March 2004.

Facility:	**Piney Point Phosphates, Inc.**	**NPDES#:**	**FL0000124**	Facility Type:	**Phosphate Plant**	**Analysts:**	Marshall Faircloth
Location:	13300 Highway 41 North, Palmetto	Contact/District:	J. Champion/SW				James Daniels
County:	Manatee	Test type:	acute definitive renewal				Christa Pierini
Sample Collection Date:	3/22/2004 Time: 1200	# tests:	2	Receiving Water:	Class III fresh		Sarah Prichard
Test Beginning Date:	3/23/2004 Time: 0340	Chlorination Type:	dechlorinated		Buckeye Road Ditch to Bishops Harbor		
Test Ending Date:	3/27/2004 Time: 1300	FDEP SOP#:	TA07_03		**Page 1 of 4**	**Reviewer:**	Julie Espy

TRC of RO was 0.25 mg/L. After salting to ~ 11 ppt, TRC was 0.15 mg/L, after salting to ~ 6 ppt, TRC was 0.18 mg/L.
Due to drop in dissolved oxygen (DO) at 24 hours, tests were aerated. All DO's were >6.9 mg/L.
The pH of the 100% sample concentrations in the fish test dropped to pH 3.4 at 48 hours. The mysid 100% sample pH dropped to 5.6 at 48 hours.

Organism: ***Americamysis bahia*** (0-48 hr) Life stage: 4 days Chamber size: 1000 ml [A]Feeding: *Artemia* Food Batch #: 0194025G

		SURVIVAL # Alive			pH			Temperature[B]			Dissolved Oxygen			Conductivity Uncorrected mmhos/cm	
Concentrations	Sample/Diluent Volume(mL)	0 hr	24 hr	48 hr	0 hour	24 hour	48 hour	0 hour	24 hour	48 hour	0 hour	24 hour	48 hour	0 hour	48 hour
R/O A	0/500	5	4	3	8.6	-	-	24.0	-	-	7.8	-	-	17.5	-
R/O B	0/500	5	0	-	-	8.6	-	-	25.0	-	-	7.1	-	-	-
R/O C	0/500	5	5	5	-	-	7.9	-	-	24.6	-	-	7.5	-	17.7
R/O D	0/500	5	5	4	-	-	-	-	-	-	-	-	-	-	-
Salt Cntl A	0/500	5	5	5	8.2	-	-	24.0	-	-	7.3	-	-	18.1	-
Salt Cntl B	0/500	5	4	4	-	8.2	-	-	25.4	-	-	7.1	-	-	-
Salt Cntl C	0/500	5	5	5	-	-	8.2	-	-	24.6	-	-	7.5	-	18.7
Salt Cntl D	0/500	5	5	5	-	-	-	-	-	-	-	-	-	-	-
6.25% A	31.25/468.75	5	5	5	8.5	-	-	24.1	-	-	7.4	-	-	16.9	-
6.25% B	31.25/468.75	5	5	5	-	8.2	-	-	25.1	-	-	6.9	-	-	-
6.25% C	31.25/468.75	5	5	5	-	-	7.8	-	-	24.8	-	-	7.4	-	16.9
6.25% D	31.25/468.75	5	5	5	-	-	-	-	-	-	-	-	-	-	-
12.5% A	62.5/437.5	5	5	5	8.5	-	-	24.4	-	-	7.4	-	-	17.8	-
12.5% B	62.5/437.5	5	5	5	-	8.0	-	-	25.2	-	-	6.5	-	-	-
12.5% C	62.5/437.5	5	5	5	-	-	7.8	-	-	24.9	-	-	7.4	-	17.6
12.5%D	62.5/437.5	5	4	3	-	-	-	-	-	-	-	-	-	-	-
25% A	125/375	5	5	4	8.5	-	-	24.4	-	-	7.5	-	-	17.9	-
25% B	125/375	5	4	4	-	8.0	-	-	25.2	-	-	6.5	-	-	-
25% C	125/375	5	5	5	-	-	7.6	-	-	24.8	-	-	7.4	-	17.9
25% D	125/375	5	5	5	-	-	-	-	-	-	-	-	-	-	-
50% A	250/250	5	5	5	8.5	-	-	24.3	-	-	7.4	-	-	18.0	-
50% B	250/250	5	4	4	-	8.0	-	-	25.2	-	-	6.6	-	-	-
50% C	250/250	5	5	5	-	-	7.4	-	-	24.7	-	-	7.4	-	18.0
50% D	250/250	5	5	5	-	-	-	-	-	-	-	-	-	-	-
100% A	500/0	5	2	0	8.4	-	-	24.2	-	-	7.4	-	-	17.9	-
100% B	500/0	5	3	0	-	7.8	-	-	25.1	-	-	6.6	-	-	-
100% C	500/0	5	2	0	-	-	5.6	-	-	24.8	-	-	7.4	-	18.1
100% D	500/0	5	2	0	-	-	-	-	-	-	-	-	-	-	-

Cntrl = Control

[A] Organisms fed daily, prior to test, and prior to renewal.

[B] Temperatures of room and test incubator were continuously recorded on a strip chart recorder.
Room Temperature range for the test period was 23.0 - 25.0°C.
Waterbath temperature range for the test period was 25.0 - 25.5°C.

LIMS
Job number: TLH-2004-03-23-49
sample number (RO): 743227
sample number (Break Point): 743228

Data Transcription Verification
date: 4/15/04
by: Cathy Oaks/James Daniels

Total Residual CL2	mg/L	Method
Field 100% sample:	not measured	-
Lab 100% sample (RO):	0.25	Hach
Lab 100% sample (Break Point	<0.03	Hach
Control water:	<0.03	Hach

Ammonia	Total (mg/L)	Unionized (mg/L)
Control water (~11 ppt):	<0.034	<0.034
100% sample (Break Point):	0.270	0.038
100% Sample (RO):	0.728	0.102

Alk & Hardness	Alkalinity (mg/L)	Hardness (mg/L)
Control water:	270	-
100% sample (Break Point):	45	1444
100% Sample (RO):	36	9

Salinity[D]	ppt
Control water:	~ 11
[E]Salt Control water:	~ 11
100% sample:	~ 4.5

[D] Salinity based on conductivity and temperature.

[E] Salt control (ASW) was diluted using well water to from 20 ppt to ~4.5 ppt then salted to ~ 11 ppt for Test 1 and ~ 6 ppt for Test 2, using 40 fathoms artificial sea salts.

Light Intensity during the test was 50-100 foot candles.
Photoperiod during the test was 16 hours of light : 8 hours of dark.

Table 1. continued — Data recorded during the 96-hour acute definitive bioassays of a sample of effluent from Piney Point Phosphates, Inc., 13300 Highway 41 North, Palmetto, Manatee County, Florida, NPDES# FL0000124, performed from 23 to 27 March 2004.

Facility:	**Piney Point Phosphates, Inc.**		**NPDES#: FL0000124**	Facility Type:	**Phosphate Plant**	**Analysts:**	Marshall Faircloth
Location:	13300 Highway 41 North, Palmetto		Contact/District:	J. Champion/SW			James Daniels
County:	Manatee		Test type:	acute definitive renewal			Christa Pierini
Sample Collection Date:	3/22/2004	Time: 1200	# tests:	2	Receiving Water:	Class III fresh	Sarah Prichard
Test Beginning Date:	3/23/2004	Time: 0340	Chlorination Type:	dechlorinated		Buckeye Road Ditch to Bishops Harbor	
Test Ending Date:	3/27/2004	Time: 1300	FDEP SOP#:	TA07_03		**Page 2 of 4**	**Reviewer:** Julie Espy

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The pH of the 100% sample concentrations in the fish test dropped to pH 3.4 at 48 hours. The mysid 100% sample pH dropped to 5.6 at 48 hours.

Organism: ***Americamysis bahia*** (48-96 hr) — Life stage: 4 days; Chamber size: 1000 ml; [A]Feeding: *Artemia*; Food Batch #: 0194025G

	Sample/Diluent	SURVIVAL # Alive			pH			Temperature[B]			Dissolved Oxygen			Conductivity Uncorrected mmhos/cm	
Concentrations	Volume(mL)	48 hr	72hr	96 hr	48 hr	72hr	96 hr	48 hr	72hr	96 hr	48 hr	72hr	96 hr	48 hr	96 hr
R/O A	0/500	3	3	3	-	-	8.0	-	-	24.9	-	-	7.0	-	18.9
R/O B	0/500	-	-	-	-	-	-	-	-	-	-	-	-	-	-
R/O C	0/500	5	5	5	8.3	-	-	24.0	-	-	8.0	-	-	17.4	-
R/O D	0/500	4	4	4	-	7.9	-	-	24.7	-	-	8.0	-	-	-
Salt Cntl A	0/500	5	5	5	-	-	8.3	-	-	24.9	-	-	7.0	-	19.1
Salt Cntl B	0/500	4	4	4	-	-	-	-	-	-	-	-	-	-	-
Salt Cntl C	0/500	5	5	5	8.1	-	-	24.1	-	-	8.2	-	-	17.5	-
Salt Cntl D	0/500	5	5	5	-	8.0	-	-	25.2	-	-	8.2	-	-	-
6.25% A	31.25/468.75	5	5	5	-	-	8.0	-	-	24.7	-	-	7.0	-	18.5
6.25% B	31.25/468.75	5	5	4	-	-	-	-	-	-	-	-	-	-	-
6.25% C	31.25/468.75	5	5	5	8.3	-	-	24.0	-	-	8.0	-	-	17.0	-
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25% C	125/375	5	5	5	8.2	-	-	24.1	-	-	7.6	-	-	17.9	-
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50% D	250/250	5	5	5	-	7.7	-	-	25.1	-	-	8.4	-	-	-
100% A	500/0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
100% B	500/0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
100% C	500/0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
100% D	500/0	-	-	-	-	-	-	-	-	-	-	-	-	-	-

Cntrl = Control

[A] Organisms fed daily, prior to test, and prior to renewal.

[B] Temperatures of room and test incubator were continuously recorded on a strip chart recorder.
Room Temperature range for the test period was 23.0 - 25.0°C.
Waterbath temperature range for the test period was 25.0 - 25.5°C.

LIMS
Job number: TLH-2004-03-23-49
sample number (RO): 743227
sample number (Break Point): 743228

Data Transcription Verification
date: 4/15/04
by: Cathy Oaks/James Daniels

Total Residual CL2	mg/L	Method
Field 100% sample:	not measured	-
Lab 100% sample (RO):	0.25	Hach
ab 100% sample (Break Point	<0.03	Hach
Control water:	<0.03	Hach

Ammonia	Total (mg/L)	Unionized (mg/L)
Control water (~11 ppt):	<0.034	<0.034
100% sample (Break Point):	0.270	0.038
100% Sample (RO):	0.728	0.102

Alk & Hardness	Alkalinity (mg/L)	Hardness (mg/L)
Control water:	270	-
100% sample (Break Point):	45	1444
100% Sample (RO):	36	9

Salinity[D]	ppt
Control water:	~ 11
[E]Salt Control water:	~ 11
100% sample:	~ 4.5

[D] Salinity based on conductivity and temperature.

[E] Salt control (ASW) was diluted using well water to from 20 ppt to ~4.5 ppt then salted to ~ 11 ppt for Test 1 and ~ 6 ppt for Test 2, using 40 fathoms artificial sea salts.

Light Intensity during the test was 50-100 foot candles.
Photoperiod during the test was 16 hours of light : 8 hours of dark.

Table 1. continued — Data recorded during the 96-hour acute bioassays of a sample of effluent from Piney Point Phosphates, Inc., 13300 Highway 41 North, Palmetto, Manatee County, Florida, NPDES# FL0000124, performed from 23 to 27 March 2004.

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Test Beginning Date:	3/23/2004	Time: 0320	Chlorination Type:	dechlorinated	Buckeye Road Ditch to Bishops Harbor		
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The pH of the 100% sample concentrations in the fish test dropped to pH 3.4 at 48 hours. The mysid 100% sample pH dropped to 5.6 at 48 hours.

Organism: ***Menidia beryllina*** (0-48 hr) — Life stage: 11 days — Chamber size: 1000 ml — [A]Feeding: *Artemia* — Food Batch #: 0194025G

		SURVIVAL # Alive			pH			Temperature[B]			Dissolved Oxygen			Conductivity Uncorrected mmhos/cm	
Concentrations	Sample/Diluent Volume(mL)	0 hr	24 hr	48 hr	0 hour	24 hour	48 hour	0 hour	24 hour	48 hour	0 hour	24 hour	48 hour	0 hour	48 hour
R/O A	0/500	10	10	10	8.5	-	8.0	24.4	-	24.2	7.8	-	7.8	10.1	10.0
R/O B	0/500	10	10	10	-	7.7	-	-	25.3	-	-	7.4	-	-	-
Salt Cntl A	0/500	10	10	10	8.4	-	8.3	24.2	-	24.7	7.5	-	7.8	10.6	10.2
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6.25% A	31.25/468.75	10	10	10	8.4	-	7.8	24.1	-	24.7	7.6	-	7.8	10.6	10.4
6.25% B	31.25/468.75	10	9	9	-	7.8	-	-	25.5	-	-	7.1	-	-	-
12.5% A	62.5/437.5	10	10	10	8.4	-	7.8	24.1	-	24.6	7.9	-	7.8	10.4	10.4
12.5% B	62.5/437.5	10	10	10	-	7.8	-	-	25.5	-	-	7.0	-	-	-
25% A	125/375	10	10	10	8.4	-	7.6	24.0	-	24.6	7.8	-	7.8	10.4	10.2
25% B	125/375	10	10	10	-	7.6	-	-	25.5	-	-	7.0	-	-	-
50% A	250/250	10	10	10	8.3	-	7.0	24.0	-	24.6	7.8	-	7.8	10.1	10.3
50% B	250/250	10	10	10	-	7.3	-	-	25.6	-	-	6.9	-	-	-
100% A	500/0	10	10	0	8.0	-	3.4	24.1	-	24.5	7.7	-	7.8	10.5	10.4
100% B	500/0	10	10	0	-	6.9	-	-	25.2	-	-	5.4	-	-	-

[A] Organisms fed prior to testing and 2 hours before and after renewal.
[B] Temperatures of room and test incubator were continuously recorded on a strip chart recorder.
Room Temperature range for the test period was 23.0 - 25.0°C.
Waterbath temperature range for the test period was 25.0 - 25.5°C.
[D] Salinity based on conductivity and temperature.
[E] Salt control (ASW) was diluted using well water from ~20 ppt to ~4.5 ppt then salted to v11 ppt for test 1 and ~6 ppt for test 2, using 40 fathoms artificial sea salts.
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sample number (RO): 743227
sample number (Break Point): 743228

Data Transcription Verification
date: 4/15/04
by: Cathy Oaks/James Daniels

Total Residual CL2	mg/L	Method
Field 100% sample:	not measured	-
Lab 100% sample (RO):	0.25	Hach
Lab 100% sample (Break Point):	<0.03	Hach
Control water:	<0.03	Hach

Ammonia	Total (mg/L)	Unionized (mg/L)
Control water (~6 ppt):	<0.017	<0.017
100% sample (Break Point):	0.270	0.038
100% Sample (RO):	0.728	0.102

Alk & Hardness	Alkalinity (mg/L)	Hardness (mg/L)
Control water:	270	-
100% sample (Break Point):	45	1444
100% Sample (RO):	36	9

Salinity[D]	ppt
Control water:	~ 6
[E]Salt Control water:	~ 6
100% sample:	4.5

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	Sample/Diluent	SURVIVAL # Alive			pH			Temperature[B]			Dissolved Oxygen			Conductivity Uncorrected mmhos/cm	
Concentrations	Volume(mL)	48 hr	72hr	96 hr	48 hr	72hr	96 hr	48 hr	72hr	96 hr	48 hr	72hr	96 hr	48 hr	96 hr
R/O A	0/500	10	10	10	8.3	-	8.0	24.0	-	24.9	7.7	-	7.6	9.99	10.9
R/O B	0/500	10	10	10	-	8.0	-	-	25.2	-	-	8.5	-	-	-
Salt Cntl A	0/500	10	10	10	8.1	-	8.3	24.0	-	24.9	7.7	-	7.6	9.39	11.1
Salt Cntl B	0/500	10	10	10	-	8.4	-	-	25.1	-	-	8.4	-	-	-
6.25% A	31.25/468.75	10	10	10	8.2	-	8.0	24.0	-	24.9	7.8	-	7.5	10.2	11.1
6.25% B	31.25/468.75	9	9	9	-	7.9	-	-	25.1	-	-	8.6	-	-	-
12.5% A	62.5/437.5	10	10	10	8.2	-	8.0	24.0	-	24.9	7.7	-	7.4	10.2	11.1
12.5% B	62.5/437.5	10	10	9	-	8.2	-	-	25.2	-	-	8.8	-	-	-
25% A	125/375	10	10	10	8.2	-	7.8	24.0	-	24.9	7.6	-	7.2	10.0	11.0
25% B	125/375	10	10	10	-	7.8	-	-	25.2	-	-	9.1	-	-	-
50% A	250/250	10	10	10	8.0	-	7.2	24.1	-	24.9	7.7	-	7.3	10.2	11.0
50% B	250/250	10	10	10	-	7.2	-	-	25.2	-	-	9.3	-	-	-
100% A	500/0	-	-	-	-	-	-	-	-	-	-	-	-	-	-
100% B	500/0	-	-	-	-	-	-	-	-	-	-	-	-	-	-

[A] Organisms fed prior to testing and 2 hours before and after renewal.
[B] Temperatures of room and test incubator were continuously recorded on a strip chart recorder.
Room Temperature range for the test period was 23.0 - 25.0°C.
Waterbath temperature range for the test period was 25.0 - 25.5°C.
[D] Salinity based on conductivity and temperature.
[E] Salt control (ASW) was diluted using well water from ~20 ppt to ~4.5 ppt then salted to v11 ppt for test 1 and ~6 ppt for test 2, using 40 fathoms artificial sea salts.
Light Intensity during the test was 50-100 foot candles.
Photoperiod during the test was 16 hours of light : 8 hours of dark.
Cntrl = Control

LIMS
Job number: TLH-2004-03-23-49
sample number (RO): 743227
sample number (Break Point): 743228

Data Transcription Verification
date: 4/15/04
by: Cathy Oaks/James Daniels

Total Residual CL2	mg/L	Method
Field 100% sample:	not measured	-
Lab 100% sample (RO):	0.25	Hach
ab 100% sample (Break Point	<0.03	Hach
Control water:	<0.03	Hach

Ammonia	Total (mg/L)	Unionized (mg/L)
Control water (~6 ppt):	<0.017	<0.017
100% sample (Break Point):	0.270	0.038
100% Sample (RO):	0.728	0.102

Alk & Hardness	Alkalinity (mg/L)	Hardness (mg/L)
Control water:	270	-
100% sample (Break Point):	45	1444
100% Sample (RO):	36	9

Salinity[D]	ppt
Control water:	~ 6
[E]Salt Control water:	~ 6
100% sample:	4.5

The Bioassay of Piney Point Phosphates, Inc. effluent sampled on 22 March 2004, NPDES #FL0000124.

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



