

Bioassays of

Mulberry Phosphates, Inc.

Mulberry, Polk County, Florida

NPDES #FL0000671

Sampled 1/5/98

July 1998

Biology Section

Division of Administrative & Technical Services

Comprehensive Quality Assurance Plan #870346G

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Mulberry Phosphates, Inc.

Mulberry, Polk County, Florida

NPDES #FL0000671

Biology Section
Bureau of Laboratories
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Introduction

Mulberry Phosphates, Inc., State Road 60, Mulberry, Polk County, Florida, NPDES #FL0000671, tests performed on 6 to 8 January, 1998.

This fertilizer manufacturing facility's wastewater treatment system has several outfalls. Outfall 001 is an internal (NPDES) structure which would discharge double lime-treated process wastewater. This structure has never discharged. Outfall 001F discharges excess stormwater that collects in a series of five on-site holding/settling ponds. Outfall 001F has a design flow of 45 MGD and an average annual flow of 0.21 MGD. Discharge from Outfall 001F goes through a discharge ditch to Skinned Sapling Creek. The sample of effluent from this facility was collected from Outfall 001F (facility information provided by Vishwas Sathe and Jay Spencer, FDEP, Tampa).

On December 7, 1997, there was a large spill (approximately 50M gallons) of low pH water (2.1 S.U.) from the South Gypsum Stack at this facility to Skinned Sapling Creek which flows into the Alafia River (Figure 1. Taken from Amundsen and Moore, 1998. *Preliminary Report Biological Assessment Associated with Accidental Process Water Discharge, Mulberry Phosphates, Inc., Mulberry, Florida.* p. 3). The spill resulted in a large fishkill from Skinned Sapling Creek to the mouth of the Alafia River in Hillsborough Bay.

The toxicity tests discussed in this report were performed in accordance with methods described by Weber, 1993, *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*, 4th Edition, EPA/600/4-90/027F.

Results and Discussion

The FDEP Biology Section performed two 48-hour static acute screening toxicity bioassays on a sample of effluent from this facility. Unlike the water which was discharged from the South Gypsum Stack on December 7, 1997, the water sample from Outfall 001F was not acidic, having a pH of 7.6. The test species used for these bioassays were the water flea, *Ceriodaphnia dubia*, and the bannerfin shiner, *Cyprinella leedsi*. The *Ceriodaphnia dubia* test results did not show toxicity by 48 hours. The *Cyprinella leedsi* test was invalidated due to excess control mortality in one of the control replicates (Table 1); however, there was no deaths among organisms in the effluent chambers.

Total residual chlorine was not measured in the field and was <0.03 mg/L in the laboratory. Total ammonia and unionized ammonia concentrations were 0.908 mg/L and 0.024 mg/L, respectively. Further chemical analyses were performed on the sample and several metals and fluoride were identified (Table 2).

Conclusion

The sample of effluent collected from this facility on January 5, 1998, did not show toxicity to the water flea, *Ceriodaphnia dubia,* by 48 hours.

Table 1. Data recorded during the 48-hour acute screening bioassays of a sample of effluent from the Mulberry Phosphates, Inc., Mulberry, Polk County, Florida, NPDES# FL0000671, performed on 6 to 8 January,1998.

Facility:	**Mulberry Phosphates, Inc.**		**NPDES #**	**FL0000671**	Facility Type: Fertilizer Manufacturing
Location:	County Road 60, Mulberry		Contact/District:	J. Spencer/Southwest	
County:	Polk		Test type:	static acute screen	
Sample Collection Date:	1/5/98	Time: 1130	# tests:	2	Receiving Water: Moncrief Creek
Test Beginning Date:	1/6/98	Time: 1415	Chlorination Type:	Non- chlorinated	
Test Ending Date:	1/8/98	Time: 1440			Page 1 of 1

Total Residual CL2	mg/L	Method
Field:	not measured	-
Lab:	<0.03	DR-100

Ammonia	Total (mg/L)	Unionized (mg/L)
Control Water (fish):	<0.017	<0.017
Control Water (20% DMW) (cladocerans):	<0.017	<0.017
100% Sample:	0.908	0.024

Alk & Hardness	Alkalinity (mg/L)	Hardness (mg/L)
Control Water (fish):	113	123
Control Water (20% DMW) (cladocerans):	64	80
100% Sample:	48	113

Salinity	(ppt)
Control Water (fish):	0
Control Water (20% DMW) (cladocerans):	0
100% Sample:	0

Organism: ***Ceriodaphnia dubia*** Life stage: < 24 hours old

	Sample/Diluent	SURVIVAL # Alive			pH			Temperature[A]			Dissolved Oxygen			Conductivity Uncorrected µmhos/cm	
Concentrations	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
Control A	0/20	5	5	5	8.1	-	8.1	24.8	-	24.7	8.4	-	7.8	170	180
Control B	0/20	5	5	5	-	-	8.1	-	-	24.6	-	-	7.8	-	180
Control C	0/20	5	5	5	-	-	8.1	-	-	24.5	-	-	7.9	-	180
Control D	0/20	5	5	5	-	-	8.1	-	-	24.4	-	-	7.8	-	185
100% A	20/0	5	5	5	7.6	-	8.1	25.0	-	24.3	8.5	-	8.5	365	390
100% B	20/0	5	5	5	-	-	8.1	-	-	24.2	-	-	8.5	-	395
100% C	20/0	5	5	5	-	-	8.1	-	-	24.0	-	-	8.7	-	400
100% D	20/0	5	5	5	-	-	8.1	-	-	24.1	-	-	8.7	-	400

[A] Temperature of test incubator and test room were continuously recorded on strip chart recorders.
Incubator temperature range for the test period was 23.9-25.0°C.
Test room temperature range for the test period was 24.0-25.5°C.

Organism: ***Cyprinella leedsi*** [B] Age: 11 days

[B] Test invalidated due to excessive control mortality.

	Sample/Diluent	SURVIVAL # Alive			pH			Temperature[C]			Dissolved Oxygen			Conductivity Uncorrected µmhos/cm	
Concentrations	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
Control A	0/500	5	5	0[D]	7.9	8.2	8.2	24.9	24.2	25.2	8.1	7.7	6.8	255	275
Control B	0/500	5	5	5	7.9	8.2	8.3	24.9	24.5	24.9	8.2	7.7	7.4	255	270
Control C	0/500	5	5	5	7.9	8.2	8.3	24.8	25.0	24.8	8.3	7.6	7.5	255	265
Control D	0/500	5	5	5	7.9	8.2	8.3	24.8	24.8	24.3	8.3	7.5	7.5	255	265
100% A	500/0	5	5	5	7.6	7.8	8.0	25.0	24.5	25.4	8.2	8.0	8.4	395	410
100% B	500/0	5	5	5	7.6	7.8	8.0	25.0	24.7	24.3	8.2	8.1	8.6	395	415
100% C	500/0	5	5	5	7.6	7.7	8.0	25.0	24.8	24.8	8.2	7.5	7.9	395	410
100% D	500/0	5	5	5	7.6	7.7	7.9	25.0	25.2	24.7	8.2	7.7	7.9	395	405

[D] Contamination from pH probe was probable cause of mortality. All other control fish appeared healthy.

[C] Temperature of test incubator and test room were continuously recorded on strip chart recorders.
Incubator temperature range for the test period was 23.9-25.0°C.
Test room temperature range for the test period was 24.0-25.5°C.

Table 2. Results of chemical analyses on the effluent from the Mulberry Phosphates, Inc., facility sampled on January 5, 1998.

Metals

Aluminum	214	µg/L
Cadmium	0.03	µg/L[I]
Calcium	27.1	mg/L
Copper	0.6	µg/L[I]
Iron	214	µg/L
Lead	0.3	µg/L[I]
Magnesium	12.5	mg/L

Base, Neutral, & Acid Extractable Organics

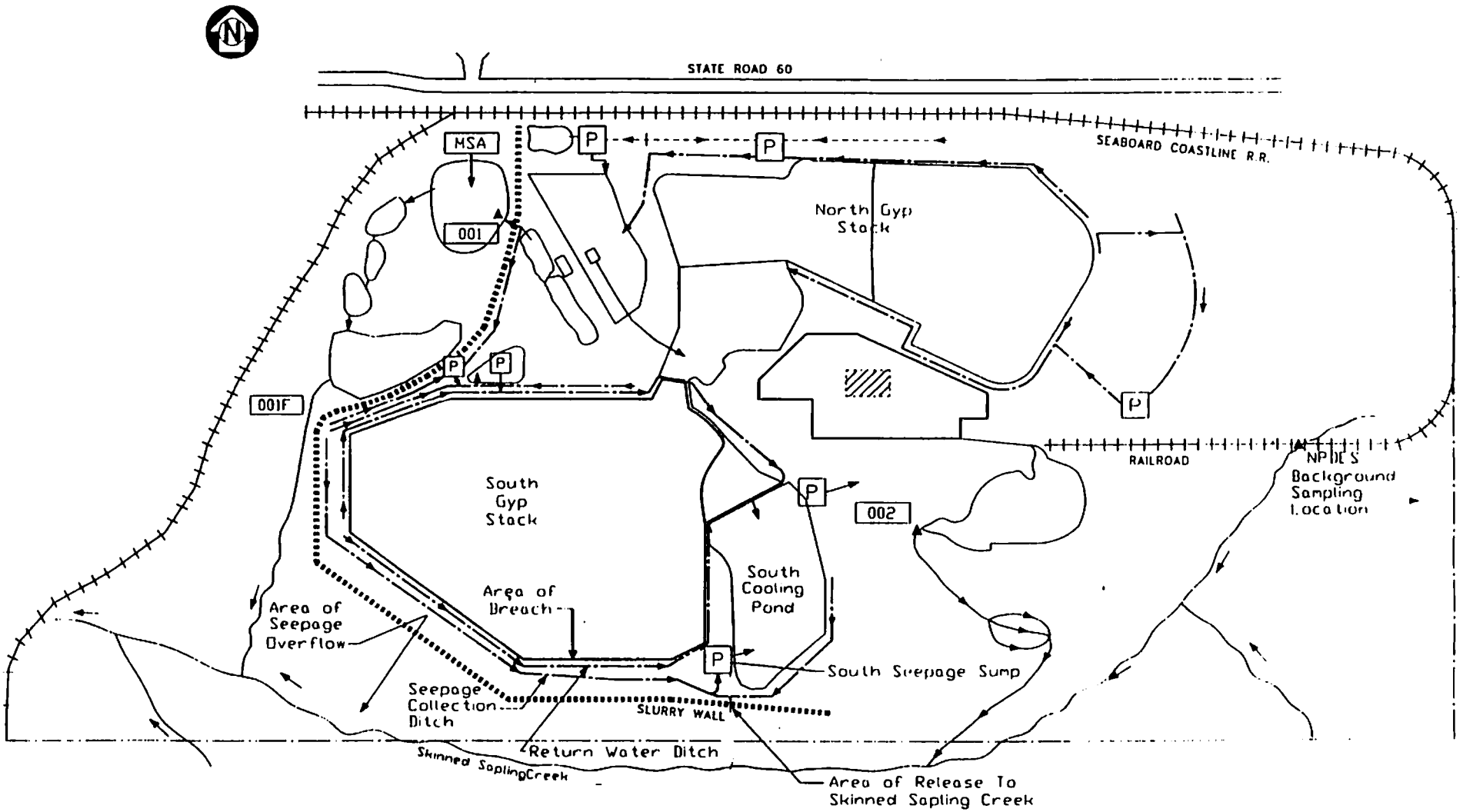
none detected

Fluoride

Fluoride	2.3	mg/L

[I] Value reported is less than the minimum quantitation limit and greater than or equal to the minimum detection limit.

Figure 1



Mulberry Phosphates Inc.

FDEP Biology Section — Acute Bioassay Bench Sheet

Sample Source: Mulberry Phosphate
County: Polk
Contact / District: J. Spencer / Southwest
NPDES Permit #: FL0000671
LIMS Sample #: 300024 LIMS Job #: 98-01-06-12
sample log: 2/23/98 DW

Sample Collection: Date 1-5-98 Time 1130
Test Beginning: Date 1-6-98 Time 1415
Test Ending: Date 1-8-98 Time 1440
Organism Batch #: 1 Diluent Batch #: 58
Organism Age: < 24 hours
Test Organism: Ceriodaphnia dubia

Test Type: (Screening) / Definitive
(Static) / Static Renewal / Flow-through
Temperature range: room 24.0-25.5°C
incubator 23.9-25.0°C
Test Number: 1 of 2
Remarks: D = dead, M = missing

Detected unidentified zooplankton in sample. Removed zooplankton by filtering sample through a 60 µm mesh screen. MF 1/6/98

Instrument Calibrations:	pH	Temperature °C	D.O. mg/L	Conductivity µmhos/cm
meter #	7851	90H018262	90H018262	G9005749
0 hr	7.0 @ 7.0	23.7 @ 23.7	8.2 @ 25.1 °C	104.7 @ 103.2
	9.0 @ 9.0			985 @ 1005 @ 25.0 °C
24 hr	7.0 @ 7.0	24.0 @ 24.0	8.3 @ 24.5 °C	101.6 @ 103.2
	9.0 @ 9.0			973 @ 1005 @ 24.7 °C
48 hr	7.0 @ 7.0	23.8 @ 23.8	8.1 @ 25.7 °C	104.3 @ 103.2
	9.0 @ 9.0			996 @ 1005 @ 25.7 °C

A Incorrect selection procedure used. Corrected 1/28/98 MF

Conc.	Chamber #	Number Live 0 hr	Number Live 24 h	Number Live 48 h	pH 0 hr	pH 24 h	pH 48 h	Temperature (°C) 0 hr	Temperature (°C) 24 h	Temperature (°C) 48 h	D.O. (mg/L) 0 hr	D.O. (mg/L) 24 h	D.O. (mg/L) 48 h	A UNCORRECTED Cond ~~(mmhos/cm)~~ Cond. (µmhos/cm) 0 hr	24 h	48 h
Control A	A	5	5	5	8.1		8.1	24.4		24.7	8.40		7.4	170		180
Control B	B	5	5	5			8.1			24.6			7.8			180
Control C	C	5	5	5			8.1			24.5			7.9			180
Control D	D	5	5	5			8.1			24.4			7.8			185
100 % A	A	5	5	5	7.6		8.1	25.0		24.3	8.50		8.5	365		390
100 % B	B	5	5	5			8.1			24.2			8.5			395
100 % C	C	5	5	5			8.1			24.0			8.7			400
100 % D	D	5	5	5			8.1			24.1			8.7			400
Measured/Loaded by		MF	MF	MF	MF		MF	MF		MF	MF		MF	MF		MF
Recorded by		MF	MF	MF	FW		FW	FW		FW	FW		FW	FW		FW

Investigators' Signatures
Marshall Faircloth
Tom Mitchell
Dan Wilcox

Water Quality Parameters verified by MF

	Salt Water Well Water	20% Min Water	Sample	Method	Measured by
Field Total Residual Cl2 (mg/L)			not measured		
Lab Total Residual Cl2 (mg/L)		<0.03	<0.03	DR-100	DW/FW
Alkalinity (mg/L as $CaCO_3$)		64	48	Hach	DW/FW
Hardness (mg/L as $CaCO_3$)		80	113	Hach	DW/FW
Total ammonia (mg/L as N)		<0.017	0.908	Orion	MF

Ammonia Meter #98136 Ammonia Meter Slope -56.7 Ammonia Blank <0.017 Control Salinity: 0 ppt Sample Salinity 0 ppt

reviewer

form updated 4/01/96

FDEP Biology Section — Acute Bioassay Bench Sheet

Sample Source: Mulberry Phosphate
County: Polk
Contact / District: J. Spencer / Southwest
NPDES Permit #: FL00 00671
LIMS Sample #: 300024 LIMS Job #: 1998-01-06-12
sample log: 2/23/98 pw

Sample Collection: Date 1-5-98 Time 1130
Test Beginning: Date 1-6-98 Time 1430
Test Ending: Date 1-8-98 Time 1440
Organism Batch #: 10.1 Diluent Batch #: well H_2O
Organism Age: 11 days
Test Organism: Cyprinella leedsi

Test Type: Screening / Definitive
Static / Static Renewal / Flow-through
Temperature range: room 24.0-25.5°C
incubator 23.9-25.0°C
Test Number: 2 of 2
Remarks: D = dead, M = missing

excessive control mortality at 48 hrs. All dead in Rep. A. (D1). Possibly pH probe contamination was cause of death. all other control fish appeared to be in good health. mf 1/8/98

Instrument Calibrations:

	pH	Temperature °C	D.O. mg/L	Conductivity µmhos/cm
meter #	7851	90H018262	90H018262	G9005749
0 hr	7.0 @ 7.0 9.0 @ 9.0	23.7 @ 23.7	8.2 @ 25.1 °C	104.7 @ 103.2 985 @ 1005 @ 25.0 °C
24 hr	7.0 @ 7.0 9.0 @ 9.0	24.0 @ 24.0	8.3 @ 24.5 °C	101.6 @ 103.2 973 @ 1005 @ 24.7 °C
48 hr	7.0 @ 7.0 9.0 @ 9.0	23.8 @ 23.8	8.1 @ 25.7 °C	104.3 @ 103.2 976 @ 1005 @ 25.7 °C

A Inverse selection procedure used (corrected) 5/28/98 mf

Conc.	Chamber #	Number Live 0 hr	Number Live 24 h	Number Live 48 h	pH 0 hr	pH 24 h	pH 48 h	Temperature (°C) 0 hr	Temperature (°C) 24 h	Temperature (°C) 48 h	D.O. (mg/L) 0 hr	D.O. (mg/L) 24 h	D.O. (mg/L) 48 h	UNCORRECTED A Cond. ~~(mmhos/cm)~~ Cond. (µmhos/cm) 0 hr	24 h	48 h
Control A	D1	5	5	0 5D	7.9	8.2	8.2	24.9	24.2	25.2	8.10	7.7	6.4	~~255~~	260	275
Control B	D2	5	5	5	7.9	8.2	8.3	24.9	24.5	24.9	8.20	7.7	7.4	~~255~~	260	270
Control C	D3	5	5	5	7.9	8.2	8.3	24.8	25.0	24.8	8.30	7.6	7.5	255	260	265
Control D	D4	5	5	5	7.9	8.2	8.3	24.8	24.8	24.3	8.30	7.5	7.5	255	260	265
100 A	D5	5	5	5	7.6	7.8	8.0	25.0	24.5	25.4	8.20	8.0	8.4	395	395	410
100 B	D6	5	5	5	7.6	7.8	8.0	25.0	24.7	24.3	8.20	8.1	8.6	395	415	415
100 C	D7	5	5	5	7.6	7.7	8.0	25.0	24.8	24.6	8.20	7.5	7.9	395	405	410
100 D	D8	5	5	5	7.6	7.7	7.9	25.0	25.2	24.7	8.20	7.7	7.9	395	410	405
Measured/Loaded by:		MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF
Recorded by:		MF	MF	MF	FW	FW	FW	FW	FW	FW	FW	FW	FW	FW	FW	FW

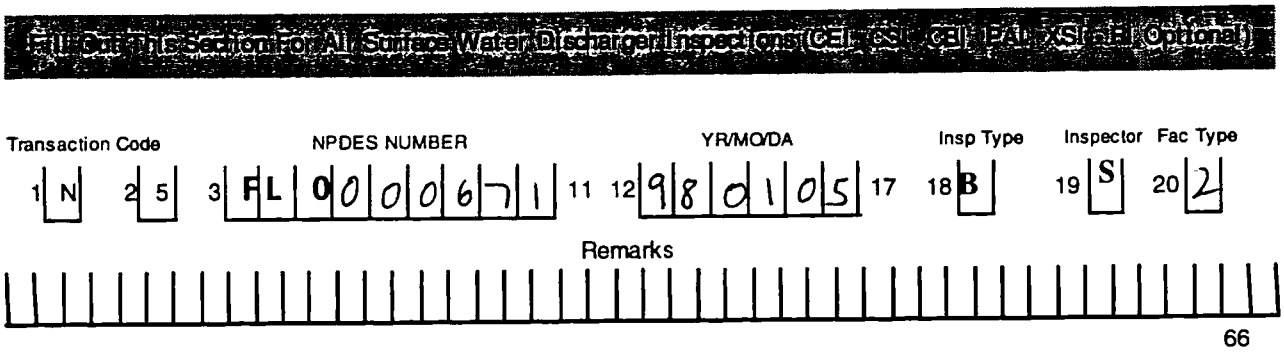
Investigators' Signatures
Marshall Fairfoth
Lacey Nordell
reviewer
form updated 4/01/96

Water Quality Parameters verified by MF

	Salt Water / Well Water	20% Min Water	Sample	Method	Measured by
Field Total Residual Cl2 (mg/L):			not measured		
Lab Total Residual Cl2 (mg/L):	<0.03		<0.03	DR-100	DW/FW
Alkalinity (mg/L as $CaCO_3$):	113		48	Hach	DW/FW
Hardness (mg/L as $CaCO_3$):	123		113	Hach	DW/FW
~~Total~~ ammonia (mg/L as N):	<0.017		0.903	Orion	MF

Ammonia Meter #98136 Ammonia Meter Slope: -56.7 Ammonia Blank: <0.017 Control Salinity: 0 ppt Sample Salinity: 0 ppt

The Bioassay of the Mulberry Phosphates, Inc., effluent sampled on January 5, 1998, NPDES #FL0000671.



The Priority Pollutants Analysis for the Bioassay of the Mulberry Phosphates, Inc., effluent sampled on January 5, 1998, NPDES #FL0000671.

