

Bioassays of

Millenium Speciality Chemicals

Jacksonville, Duval County, Florida

NPDES #FL0000884

Sampled 2/23/98

July 1998

Biology Section

Division of Administrative & Technical Services

Comprehensive Quality Assurance Plan #870346G

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Introduction

Millenium Speciality Chemicals, 601 Crestwood Street, Jacksonville, Duval County, Florida, NPDES #FL0000884, tests performed on 24 to 26 February, 1998.

This chemical processing facility's wastewater treatment system has a design flow of 5.0 MGD. Wastewater includes: contaminated stormwater; non-contact cooling water; boiler, softener, and cooling tower blowdown; cooling water discharges; and steam condensate. Steam condensate is treated with sodium zeolite water-treatment units to remove heavy metals, specifically copper and iron, from the condensate. The combined effluent passes through an oil skimmer prior to discharge to Moncrief Creek (facility summary prepared by Heather Hayek, FDEP, Jacksonville).

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. The sample of effluent had a sweet, lemon-like odor. The odor diminished in intensity during the duration of the test. The test species used for these bioassays were the water flea, *Ceriodaphnia dubia*, and the bannerfin shiner, *Cyprinella leedsi*. Test results did not show acute toxicity; however, the *Cyprinella leedsi* started exhibiting loss of coordination within 30 minutes of being loaded. At 24 hours the fish appeared normal (Table 1).

The odor detected by laboratory personnel, and the behavior of the fish when exposed to the fresh sample, suggests that an unidentified volatile substance may be present in the sample. The static acute test methods used for this sample are not designed to properly assess the toxicity of unstable substances. A more rigorous test method which uses a static-renewal or flow-through test design may be required to determine whether the effluent is toxic. A biological assessment of the receiving water may also help to determine whether or not this substance has affected the receiving water biota.

Total residual chlorine was reported as being below the detection limit in the field and was <0.03 mg/L in the laboratory. Total ammonia and unionized ammonia concentrations were 0.27 mg/L and 0.014 mg/L, respectively. Further chemical analyses were performed on the sample and several metals were identified (Table 2). The mercury concentration (0.16 µg/L) exceeded the State of Florida Class III freshwater surface water criterion for mercury (≤0.012 µg/L) (FAC 62-302.530).

Conclusion

The sample of effluent collected from this facility on February 23, 1998, was not acutely toxic to the *Ceriodaphnia dubia* or *Cyprinella leedsi* after a 48-hour exposure, despite the temporary loss of coordination exhibited by the *Cyprinella leedsi* test organisms. It is possible that an undetected volatile component in the sample was responsible for the odor and effect on the fish, but the 48-hour static acute toxicity test may not have been able to detect toxicity caused by volatile or rapidly degrading compounds whereas a more rigorous toxicity test method may. It is suggested that a follow-up toxicity test be performed using a static-renewal or flow-through test design. A biological assessment of the receiving water may indicate whether the effluent has had any effects on the instream biota.

Table 1. Data recorded during the 48-hour acute screening bioassays of a sample of effluent from the Millenium Speciality Chemicals facility, Jacksonville, Duval County, Florida, NPDES# FL0000884, performed on 24 to 26 February, 1998.

Facility:	**Millenium Speciality Chemicals**	**NPDES #**	**FL0000884**	Facility Type:	Chemical Processing
Location:	601 Crestwood Street, Jacksonville	Contact/District:	Tim McKelvey/Northeast		
County:	Duval	Test type:	static acute screen		
Sample Collection Date:	2/23/98 Time: 1230	# tests:	2	Receiving Water:	Moncrief Creek
Test Beginning Date:	2/24/98 Time: 1430	Chlorination Type:	non-chlorinated		
Test Ending Date:	2/26/98 Time: 1330				Page 1 of 1

Total Residual CL2	mg/L	Method
Field:	BDL*	-
Lab:	<0.03	DR-100

*Below Detection Limit

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:	0.270	0.014

Alk & Hardness	Alkalinity (mg/L)	Hardness (mg/L)
Control Water (fish):	125	128
Control Water (20% DMW) (cladocerans):	75	86
100% Sample:	126	188

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	8.2	-	8.2	23.0	-	24.1	7.7	-	8.1	170	200
Control B	0/20	5	5	5	-	-	8.2	-	-	24.1	-	-	8.1	-	185
Control C	0/20	5	5	5	-	-	8.2	-	-	24.0	-	-	8.1	-	190
Control D	0/20	5	5	5	-	-	8.2	-	-	23.8	-	-	8.1	-	205
100% A	20/0	5	5	5	8.0	-	8.4	23.2	-	24.0	7.7	-	8.1	565	590
100% B	20/0	5	5	5	-	-	8.4	-	-	23.9	-	-	8.0	-	620
100% C	20/0	5	5	5	-	-	8.4	-	-	23.9	-	-	8.0	-	630
100% D	20/0	5	5	5	-	-	8.4	-	-	23.8	-	-	8.0	-	700

[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 24.5-25.5°C.

Organism: ***Cyprinella leedsi*** Age: 12 days

	Sample/Diluent	SURVIVAL # Alive			pH			Temperature[B]			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	5	7.9	8.2	8.3	22.3	25.2	24.8	8.3	7.7	8.1	240	260
Control B	0/500	5	5	5	7.9	8.2	8.3	22.1	26.3	25.2	8.2	7.8	8.0	240	265
Control C	0/500	5	5	5	7.9	8.2	8.3	22.1	25.8	25.7	8.3	7.7	7.8	240	265
Control D	0/500	5	5	5	7.9	8.2	8.3	22.1	25.7	25.1	8.3	7.8	7.9	240	260
100% A	500/0	5	4	4	7.9	7.9	8.1	24.5	26.0	25.1	6.4	4.8	6.3	605	615
100% B	500/0	5	5	5	7.9	7.9	8.1	24.6	26.1	25.5	6.5	4.9	6.3	605	635
100% C	500/0	5	5	5	7.9	7.9	8.1	24.6	25.5	24.8	6.5	4.9	6.4	605	620
100% D	500/0	5	5	5	7.9	7.9	8.1	24.7	25.6	25.3	6.5	4.9	6.4	605	625

[B] Temperature of test incubator and test room were continuously recorded on strip chart recorders.
Incubator temperature range for the test period was 25.0-26.0°C.
Test room temperature range for the test period was 24.5-25.5°C.

Table 2. Results of chemical analyses on the effluent from the Millenium Speciality Chemicals Facility sampled on February 23, 1998.

Metals

Calcium	46.4	mg/L [A]
Copper	3.7	µg/L [A]
Iron	226	µg/L [A]
Lead	0.3	µg/L [I]
Magnesium	17.1	mg/L [A]
Mercury	0.16	µg/L [I]
Zinc	36	µg/L [J]

Base, Neutral, & Acid Extractable Organics

none detected

Pesticides and Herbicides

none detected

[A] Value reported is the mean of two or more determinations.
[I] Value reported is less than the minimum quantitation limit and greater than or equal to the minimum detection limit.
[J] Estimated value.

FDEP Biology Section — Acute Bioassay Bench Sheet

Sample Source: Millenium Specialty Chemicals
County: Duval
Contact / District: Tim McKelvey / Northeast
NPDES Permit #: FL0000884
LIMS Sample #: 309333 LIMS Job #: TLH 1998-02-24-27
sample log: 3120/98 PW

Sample Collection: Date 2/23/98 Time 1230
Test Beginning: Date 2/24/98 Time 14:30
Test Ending: Date 2/26/98 Time 13:30
Organism Batch #: 10 Diluent Batch #: 9
Organism Age: < 24 hrs.
Test Organism: Ceriodaphnia dubia

Test Type: (Screening) Definitive
Static | Static Renewal | Flow-through
Temperature range: room 24.5-25.5°C
incubator 23.9-25.0°C
Test Number: 1 of 2
Remarks: D = dead, M = missing
Sample has a sweet, lemon-like smell. Fish started exhibiting loss of coordination within ½ hour of being loaded.

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	21.1 @ 21.2	8.2 @ 24.9 °C	94.6 @ 96.3 978/9410 @ 1005/1008.9 @ 24.2 °C
24 hr	7.0 @ 7.0 9.0 @ 9.0	22.0 @ 22.0	8.2 @ 25.1 °C	96.7 @ 96.3 984/9750 @ 1005/1008.9 @ 24.5 °C
48 hr	7.0 @ 7.0 9.0 @ 9.0	22.4 @ 22.4	8.2 @ 25.3 °C	98.3 @ 96.3 987 @ 1005 @ 24.6 °C

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 Cond. (mmhos/cm) Cond. (µmhos/cm) 0 hr	Cond. 24 h	Cond. 48 h
Ctl	A	5	5	5	8.2		8.2	23.0		24.1	7.7		8.1	170		200
Ctl	B	5	5	5			8.2			24.1			8.1			185
Ctl	C	5	5	5			8.2			24.0			8.1			190
Ctl	D	5	5	5			8.2			23.8			8.1			205
100%	A	5	5	5	8.0		8.4	23.2		24.0	7.7		8.1	565		590
100%	B	5	5	5			8.4			23.9			8.0			620
100%	C	5	5	5			8.4			23.9			8.0			630
100%	D	5	5	5			8.4			23.8			8.0			700
Measured/Loaded by:		MF	MF	MF	MF		MF	MF		MF	MF		MF	MF		MF
Recorded by:		DW	MF	MF	PW		PW	PW		PW	PW		PW	PW		PW

Investigators' Signatures

Marshall Faircloth

Reviewer

form updated 4/01/96

Salt Water Water Quality Parameters verified by MF

	Well Water	20% Min Water	Sample	Method	Measured by
Field Total Residual Cl2 (mg/L):			BDL	—	Heather Hayek
Lab Total Residual Cl2 (mg/L):		<0.03	<0.03	DR-100	DW
Alkalinity (mg/L as $CaCO_3$)		75	126	Hach	DAW
Hardness (mg/L as $CaCO_3$)		86	164	Hach	DAW
Total ammonia (mg/L as N)		<0.017	0.27	Orion	MF

Ammonia Meter #98136 Ammonia Meter Slope -56.5 Ammonia Blank <0.017 Control Salinity 0 ppt Sample Salinity: 0 ppt

FDEP Biology Section — Acute Bioassay Bench Sheet

Sample Source: Millennium Specialty Chemicals
County: Duval
Contact / District: Tim McKelvey / Northeast
NPDES Permit #: FL0000884
LIMS Sample #: 309333 LIMS Job #: 1998-02-24.27 T&T
sample log: 3/20/98 ow

Sample Collection: Date 2/23/98 Time 1230
Test Beginning: Date 2/24/98 Time 14.30
Test Ending: Date 2/26/98 Time 13:30
Organism Batch #: 12 Diluent Batch #: Well
Organism Age: 12 days
Test Organism: Cyprinella leedsi

Test Type: Screening / Definitive
Static / Static Renewal / Flow-through
Temperature range: room 24.5-25.5 °C
incubator 25.0-26.0°C
Test Number: 2 of 2

Remarks: D = dead, M = missing
Sample has a sweet, lemon-like smell. Fish started exhibiting loss of coordination w/in ½ hour of being loaded.
@ 24 hours, odor still present; however, fish seem normal. MF 2-25-98 @ 48 hours just a trace of original odor. MF 2/26/98

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	21.1 @ 21.2	8.2 @ 24.9 °C	98.6 @ 96.3 978 @ 1005 @ 24.2°C 9810 1005.9
24 hr	7.0 @ 7.0 9.0 @ 9.0	22.0 @ 22.0	8.2 @ 25.1 °C	96.7 @ 96.3 984 @ 1005 @ 24.5°C 97506 100594
48 hr	7.0 @ 7.0 9.0 @ 9.0	22.4 @ 22.4	8.2 @ 25.3 °C	98.3 @ 96.3 987 @ 1005 @ 24.6 °C

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 Cond. (mmhos/cm) Cond. (µmhos/cm) 0 hr	24 h	48 h
Ctl A	61	5	5	5	7.9	8.2	8.3	22.3	25.2	24.6	8.3	7.7	8.1	240	260	260
Ctl B	62	5	5	5	7.9	8.2	8.3	22.1	26.3	25.2	8.2	7.8	8.0	240	270	265
Ctl C	63	5	5	5	7.9	8.2	8.3	22.1	25.4	25.7	8.3	7.7	7.8	240	260	265
Ctl D	64	5	5	5	7.9	8.2	8.3	22.1	25.7	25.1	8.3	7.8	7.9	240	265	260
100% A	65	5	4[D]	4	7.9	7.9	8.1	24.5	26.0	25.1	6.4	4.8	6.3	605	625	615
100% B	66	5	5	5	7.9	7.9	8.1	24.6	26.1	25.5	6.5	4.9	6.3	605	630	635
100% C	67	5	5	5	7.9	7.9	8.1	24.6	25.5	24.6	6.5	4.9	6.4	605	628	620
100% D	68	5	5	5	7.9	7.9	8.1	24.7	25.6	25.3	6.5	4.9	6.4	605	620	625
Measured/Loaded by:		MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF	MF
Recorded by:		DAW	MF	MF	FW	FW	FW	FW	FW	FW	FW	FW	FW	FW	FW	FW

Investigators' Signatures

Tim McKelvey
Dan Pelt
Marshall Faircloth
David Warmington

reviewer

form updated 4/01/96

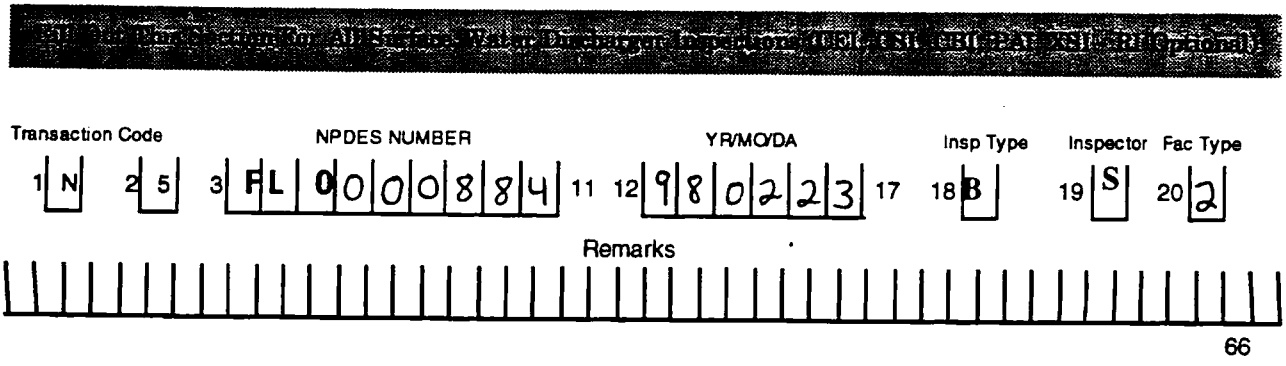
Salt Water / Well Water

Water Quality Parameters verified by MF

	20% Min Water	Sample	Method	Measured by
Field Total Residual Cl2 (mg/L):		BDL	—	Heather Hayek
Lab Total Residual Cl2 (mg/L):	<0.03	<0.03	DR-100	DW
Alkalinity (mg/L as $CaCO_3$):	125	126	Hach	DAW
Hardness (mg/L as $CaCO_3$):	128	168	Hach	DAW
Total ammonia (mg/L as N):	<0.017	0.27	Orion	MF

Ammonia Meter #98136 Ammonia Meter Slope: -56.5 Ammonia Blank: <0.017 Control Salinity: 0 ppt Sample Salinity: 0 ppt

The Bioassay of the Millenium Speciality Chemicals effluent sampled on February 23, 1998, NPDES #FL0000884.



The Priority Pollutants Analysis for the Bioassay of the Millenium Speciality Chemicals effluent sampled on February 23, 1998, NPDES #FL0000884.

