Investigation of Sediment Toxicity in White Lake for Removing the Degradation of Benthos Beneficial Use Impairment

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1. BACKGROUND

White Lake (Muskegon County, Michigan) is a 10.4 km^2 drowned river mouth lake located in western Michigan. The lake was listed as an Area of Concern (AOC) by the International Joint Commission (IJC) in 1987 because of severe environmental impairments related to the historic discharge of municipal and industrial wastes. The Beneficial Use Impairment (BUI), Degradation of Benthos, was listed because sediment toxicity related to heavy metals and organic chemicals and impacts to species diversity from the discharge of municipal sewage. In consideration of the improvements to the benthic community in the lake and the remediation/restoration of Tannery Bay and Hooker Chemical, the White Lake Public Advisory Council (PAC) voted in 2005 to establish numerical criteria to delist the Degradation of Benthos BUI. The targets are required a post remediation survey of sediment toxicity in the area of the Occidental Chemical Outfall, a sediment toxicity investigation of the deep basin near the channel, and a survey of benthic macroinvertebrates in White Lake. The survival of amphipods in the sediment toxicity experiments needed to be >60% for delisting the BUI. This report covers the sediment toxicity survey. Samples were collected using a PONAR dredge and analyzed for standard physical characteristics. Solid phase toxicity was measured using EPA (2000) methods with amphipods. A total of two samples were collected from the deep basin near the channel (WL-1 and WL-2) and three near the Occidental outfall (WL-21A, WL-21B, and WL-21C). In 2003, 12,000 cubic yards of contaminated sediment were removed from an area surrounding the Occidental Outfall. Three samples were collected from this location to encompass the remediation area. A negative control sample From Duck Lake State Park (DL-1) also was included. The results of this investigation will determine if the delisting criteria have been met and confirm that no sediment toxicity is present in these areas of White Lake

2. PROJECT/TASK DESCRIPTION

Specific objectives and task elements to evaluate the sediment toxicity in selected areas of White Lake are summarized below:

- Surface samples were collected from White Lake with a PONAR.
- Critical measurements were the concentration of total organic carbon and grain size.
- Sediment toxicity evaluations were performed with *Hyalella azteca*.
- Toxicity measurements in White Lake sediments were evaluated and compared to a control location (Duck Lake). These measurements will determine the presence and degree of toxicity associated with sediments in the deep basin and Occidental area of White Lake.
- Critical measurements were the determination of lethality and weight during the toxicity tests and the monitoring of water quality indicators during exposure (ammonia, dissolved oxygen, temperature, conductivity, pH, and alkalinity).

3. SAMPLING LOCATIONS AND METHODS

SAMPLING LOCATIONS

Sampling sites for the assessment of contaminated sediments in White Lake were selected based on previous results from Rediske et al. (2004). A map of the locations is provided in Figure 1. The location of the Duck Lake control sample is given on Figure 2.

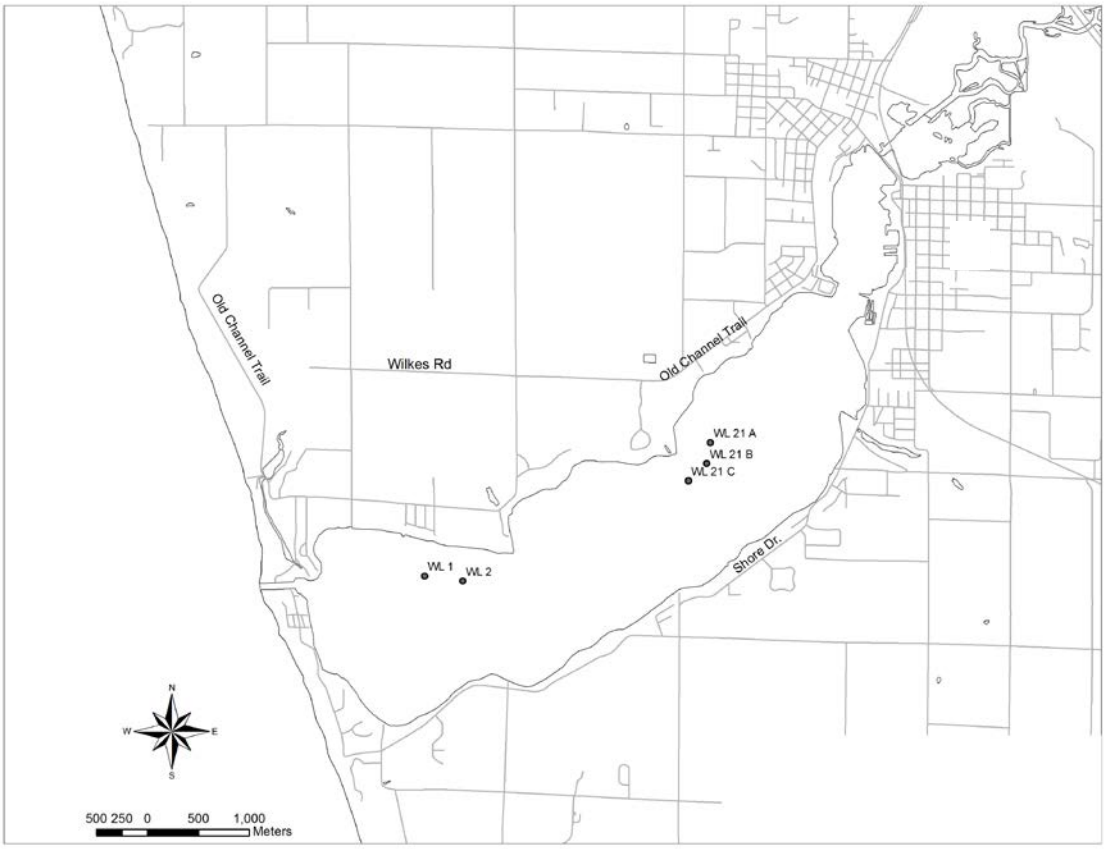

FIGURE 1. WHITE LAKE SAMPLING STATIONS.

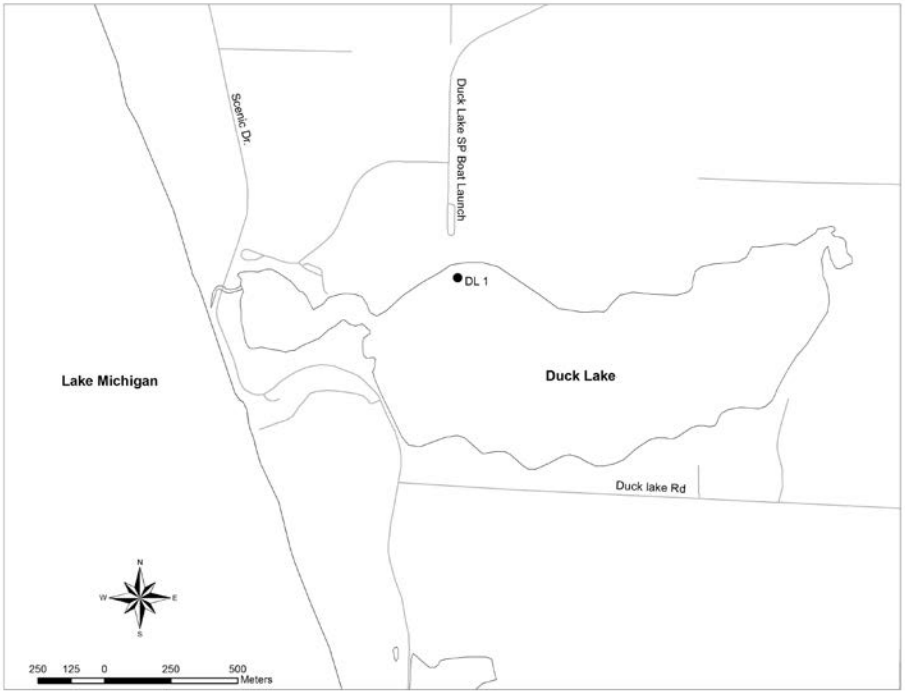

FIGURE 2. DUCK LAKE CONTROL LOCATION.

SAMPLING METHODS

A PONAR dredge was used to collect samples for laboratory toxicity and related chemical analyses. For sediment chemistry and toxicity testing, the samples was deposited into a stainless steel pan and split into two sub-samples. The PONAR was washed with water in between stations. The GPS locations and Depth are summarized below:

Deep Basin	North	West	Depth (m)
WL-1	N 43 22.58'	W 086 24.55'	18.06
WL-2	N 43 22.45'	W 086 24.15'	20.06
Occidental Outfall			
WL-21 A	N 43 23.15'	W 086 22.57'	17.15
WL-21 B	N 43 23.00'	W 086 22.97'	18.77
WL-21 C	N 43 23.04'	W 086 23.10'	17.22
Duck Lake Control			
DL-1	N 43 20.33'	W 086 23.50'	1.80

SAMPLE CONTAINERS, PRESERVATIVES, AND VOLUME REQUIREMENTS

Requirements for sample volumes, containers, and holding times are listed in Table 1. All sample containers for sediment chemistry and toxicity testing were purchased precleaned and certified as Level II by I-CHEM, Inc. Specifications for each lot of bottles were verified by checking the supplier's certification statement. This information was retained in the project files.

TABLE 1. SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

				Hold Times	
Matrix	***Parameter***	***Container***	***Preservation***	***Extraction***	***Analysis***
Sediment	TOC	250 ml Wide Mouth Plastic	Freeze -10^{0}C	---	6 months
Sediment	Grain Size	1 Quart Zip-Lock Plastic Bag	Cool to 4^{o}C	---	6 months
Sediment	Toxicity	4 liter Wide Mouth Glass	Cool to 4^{o}C	---	45 days
Culture Water	Alkalinity Ammonia Hardness Conductivity pH	250 ml Wide Mouth Plastic	Cool to 4^{o}C	---	24 hrs.

SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Chain-of-Custody procedures are essential to establish the relevance and authenticity of the data. Sample custody was addressed in three parts: field collection, laboratory analysis, and the final project file. The chain-of-custody was initiated in the field and maintained through the laboratory

4. ANALYTICAL PROCEDURES

LABORATORY ANALYSIS

Tables 2 and 3 summarize the analyte group and method from which each method is derived and list all analyte detection limits.

TABLE 2. ANALYTICAL METHODS AND DETECTION LIMITS

Parameter	SOP Name	Analytical Method	Detection Limit
Grain Size	Grain Size by Wet Sieve	WRI Method PHY-010	1 %
TOC	TOC by Combustion/IR	WRI Method PHY-020	0.5 %

Grain size was performed by wet sieving the sediments. The following mesh sizes were used: 2 mm (granule), 1 mm (very coarse sand), 0.5 mm (coarse sand), 0.25 mm (medium sand), 0.125 mm (fine sand), 0.063 mm (very fine sand), and < 0.63 mm (silt and clay).

TABLE 3. ANALYTICAL METHODS AND DETECTION LIMITS FOR TOXICITY EXPERIMENTS

Parameter	Method	Detection Limit
Specific Conductance	Standard Methods 2510 B.	NA
Alkalinity	Standard Methods 2320 B.	10 mg/l
Temperature	Standard Methods 2550	NA
Dissolved Oxygen	Standard Methods 4500-O G.	0.5 mg/l
Ammonia Electrode	Standard Methods 4500-NH_3 F.	0.05 mg/l
Hardness	Standard Methods 2340 C.	10 mg/l

TOXICITY TESTING

All toxicity testing for the *Hyalella azteca* 10 day sediment toxicity test followed procedures outlined in EPA-823-F-00-002; Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminates with Freshwater Invertebrates. Performance criteria for toxicity testing are itemized in Table 4. The specific test organisms and procedures are listed below.

TABLE 4. METHOD-SPECIFIC DATA QUALITY OBJECTIVES TOXICITY TESTING

1.	Test Type:	Whole-sediment toxicity test with renewal of overlying water
2.	Temperature (°C):	23 ± 1°C
3.	Light quality:	Wide-spectrum fluorescent lights
4.	Luminance:	About 500 to 1000 lux
5.	Photoperiod:	16 h light, 8 h darkness
6.	Test chamber size:	300 mL high-form lipless beaker
7.	Sediment volume:	100 mL
8.	Overlying water volume:	175 mL
9.	Renewal of overlying water:	2 volume additions per day (e.g., one volume addition every 12 hours)
10.	Age of test organisms:	7 to 14 days old at the start of the test
11.	Number of organisms per chamber:	10
12.	Number of replicate chambers per treatment:	8
13.	Feeding:	Tetrafin® fish food, fed 1.5 mL daily to each test chamber
14.	Aeration:	None, unless dissolved oxygen in overlying water drops below 2.5 ppm of saturation
15.	Overlying water:	Well water
16.	Overlying water quality:	Hardness, alkalinity, conductivity, pH, and ammonia measured at the beginning and end of a test. Temperature and dissolved oxygen measured daily.
17.	Test duration:	10 days
18.	End point (toxicity):	Survival, with greater than 80% in the control
	End Point (growth):	Weight increase from initial measurement

Stocks of *H. azteca* were obtained from Aquatic Biosystems, Boulder, Colorado. Organism stocks were acclimated in the moderately hard well water for 48 hrs prior to initiation of the solid phase toxicity tests. The *H. azteca* were 7-14 days old. A reference test with KCl also was conducted on this lot of test organisms and survival in the control treatment exceeded the required 90%. The Trimmed Spearman-Karber 96-hour EC50

estimate was 433 mg/L KCL with a 95% confidence interval ranging from 410 to 456 mg/L

For the solid phase testing, eight replicates per sediment sample were set up for *H. azteca* exposures, with a sample from Duck Lake used as a control. Duck Lake was used as a reference sediment (negative control) to document the survival of the test organisms in a similar matrix as the White Lake sites.

One day prior to the start of the test (day -1), the sediment from each site was mixed thoroughly and 100 mL aliquots were transferred to each of the eight test chambers. Additionally, visual observations of the sediments were made. Moderately hard well water was also added at this time. On day 0, the overlying water was renewed once before the test organisms were introduced into each of the glass beakers. Measurement of water quality parameters was also initiated on this day. Ten 7-14 day old *H. azteca* were randomly added to their respective test chambers. At this time the organisms were fed 1.5 mL of Tetrafin® suspension. The glass beakers were placed in a rack and transferred to a temperature controlled chamber ($23 \pm 1^{o}C$). The light cycle was 16 hours on and 8 hours off. Temperature and dissolved oxygen measurements were taken from one randomly selected beaker for each sediment sample every 12 hours, after which the overlying water was renewed in all the beakers. Feeding occurred after the morning renewal. This procedure was repeated daily through day 10, at which point the test was terminated. On day 0, the overlying water from the beakers was composited from each sediment sample and 250 mls were retained for alkalinity, pH, conductance, hardness, and ammonia analysis. On the last day, the same procedure was carried out. On day 10, the sediments were sieved, and the surviving test organisms were removed and counted. The biological endpoint for these sediment tests was mortality. The validity of the test was based on greater than 80% survival for amphipods. In addition, growth measurements (weight gain) were made for each organism. Amphipods were placed in tared aluminum pans, dried for 48 hrs at $105^{0}C$, and cooled in a desiccator for one hour prior to weighing. .

6. RESULTS

Chemical Measurements

Sediments from the deep basin near the channel (WL-1 and WL-2) and Duck Lake consisted of black organic silts. Samples for the Occidental Outfall were a mixture of sand and organic silts. This location was remediated in 2003 and covered with a sand cap. The results of grain size and TOC analyses are shown in Table 5.

TABLE 5. RESULTS OF GRAIN SIZE AND TOC ANALYSES OF WHITE LAKE AND DUCK LAKE SEDIMENTS (2011).

White Lake and Duck Lake Grain Size and TOC 2011									
ID	% gravel	% Sand						% Fines	TOC %
	2000 µm	1000 µm	500 µm	250 µm	125 µm	63 µm	1000-63 µm	>63 µm	
WL-1	0	0	0	4	9	6	20	80	12
WL-2	0	0	0	5	7	4	16	84	11
WL-21A	0	0	5	11	14	22	52	48	8
WL-21B	0	0	6	13	15	32	66	34	9
WL-21C	0	0	8	12	18	26	64	36	8
Duck Lake DL 1	0	0	1	5	7	2	15	85	12

Conductivity, hardness, alkalinity, ammonia, and pH were determined on the culture water at the beginning and on the tenth day of each test (Appendix A: Table A-1). With the exception of ammonia, all water quality parameters remained relatively constant (< 50% variation from start to end of test). Variations of greater than 50%, from initial to final measurements for both test species were observed for ammonia. Based on the initial pH values (all < 8.00) and the fact that the overlying water was exchanged prior to adding the organisms, toxicity related to unionized ammonia was not anticipated to be a factor in these experiments. Temperature and dissolved oxygen measurements were recorded daily throughout the duration of the tests (Appendix A: Table A-2). Very little variation was noted with respect to temperature. The results of chemical monitoring indicated that the test criteria in Table 4 were achieved.

Toxicity Testing

Test criteria for temperature (23 $\pm$ 1°C), dissolved oxygen (> 2.5 ppm), and survival in the control (>80%) were met. The initial weight of amphipods was 0.088 mg/individual and all replicates in the control showed increased weight gain (growth). Survival and growth data for *Hyalella azteca* are presented in Table 6. Un-transformed survival and growth data were evaluated for normality with Chi Square at $\alpha = 0.01$ and the data were consistent with a normal distribution. Survival and growth data also passed Hartley's Test for homogeneity of variance at $\alpha = 0.01$. Dunnett's Test showed no statistical difference between the control (Duck Lake) with respect survival and growth (α=0.05). All samples White Lake samples had *Hyalella azteca* survival rates exceeding the > 60% survival Delisting Target.

TABLE 6. SURVIVAL AND GROWTH DATA FOR SOLID PHASE TOXICITY TESTS IN WHITE LAKE AND DUCK LAKE USING *HYALELLA AZTECA*.

Sample ID	Rep.	# of organisms		Pan Wt.	Pan Wt.	Org.	Mean wt. per survivor (mg)	%	Mean test wt. per survivor (mg)
		Added	Alive	Initial (g)	Dry (g)	Wt. (g)		Mean Survival	
Control Duck Lake D-1	A	10	8	1.1096	1.1118	0.0022	0.275	84	0.144
	B	10	9	1.1400	1.1411	0.0011	0.122		
	C	10	8	1.1190	1.1198	0.0008	0.100		
	D	10	8	1.1405	1.1413	0.0008	0.100		
	E	10	8	1.1029	1.1038	0.0009	0.112		
	F	10	8	1.1253	1.1261	0.0008	0.100		
	G	10	8	1.1245	1.1261	0.0016	0.200		
	H	10	10	1.1169	1.1183	0.0014	0.140		
WL-1	A	10	9	1.1123	1.1136	0.0013	0.144	90	0.134
	B	10	8	1.1390	1.1400	0.0010	0.125		
	C	10	7	1.1089	1.1098	0.0009	0.129		
	D	10	10	1.1059	1.1072	0.0013	0.130		
	E	10	9	1.1242	1.1260	0.0018	0.200		
	F	10	10	1.0890	1.0900	0.0010	0.100		
	G	10	10	1.1446	1.1458	0.0012	0.120		
	H	10	9	1.1053	1.1064	0.0011	0.122		
WL-2	A	10	7	1.1200	1.1207	0.0007	0.100	78	0.136
	B	10	6	1.0936	1.0944	0.0008	0.133		
	C	10	10	1.1213	1.1230	0.0017	0.170		
	D	10	9	1.1299	1.1311	0.0012	0.133		
	E	10	7	1.1076	1.1089	0.0013	0.186		
	F	10	9	1.1151	1.1164	0.0013	0.144		
	G	10	9	1.1503	1.1514	0.0011	0.122		
	H	10	5	1.1142	1.1147	0.0005	0.100		
WL-21 A	A	10	10	1.1160	1.1175	0.0015	0.146	91	0.190
	B	10	8	1.0829	1.0846	0.0017	0.212		
	C	10	9	1.1071	1.1096	0.0025	0.278		
	D	10	10	1.1372	1.1399	0.0027	0.270		
	E	10	8	1.1198	1.1210	0.0012	0.150		
	F	10	10	1.1316	1.1332	0.0016	0.160		
	G	10	9	1.1377	1.1388	0.0011	0.122		
	H	10	9	1.1288	1.1304	0.0016	0.178		
WL-21 B	A	10	10	1.1235	1.1264	0.0029	0.290	88	0.186
	B	10	9	1.1140	1.1149	0.0009	0.100		
	C	10	9	1.1106	1.1131	0.0025	0.278		
	D	10	9	1.1471	1.1483	0.0012	0.133		
	E	10	7	1.1201	1.1212	0.0011	0.157		
	F	10	10	1.1446	1.1457	0.0011	0.110		
	G	10	8	1.1165	1.1176	0.0011	0.135		
	H	10	8	1.1304	1.1327	0.0023	0.287		
WL-21 C	A	10	7	1.1316	1.1320	0.0004	0.057	83	0.138
	B	10	10	1.1165	1.1175	0.0010	0.100		
	C	10	7	1.1328	1.1338	0.0010	0.143		
	D	10	8	1.1119	1.1125	0.0006	0.075		
	E	10	9	1.1281	1.1293	0.0012	0.133		
	F	10	8	1.1336	1.1346	0.0010	0.125		
	G	10	8	1.1534	1.1545	0.0011	0.138		
	H	10	9	1.1351	1.1381	0.0030	0.333		

7. DISCUSSION

A sediment toxicity survey of two locations in White Lake was conducted in October 2011. The White lake Public Advisory Council developed Delisting Targets for the Degradation of Benthos Beneficial Use Impairment that required a post remediation survey of sediment toxicity in the area of the Occidental Chemical Outfall and a sediment toxicity investigation of the deep basin near the channel be conducted. Sediment samples were collected using a PONAR dredge from these locations and analyzed for standard physical characteristics and solid phase toxicity was measured using EPA (2000) methods with amphipods. A total of two samples were collected from the deep basin near the channel and three near the Occidental outfall. A negative control sample From Duck Lake State Park also was included. There was no statistically significant difference in amphipod growth and survival between the control and the five samples collected form White Lake. The results indicate that no toxicity to amphipods was present at the sampling locations and that the delisting target of > 60% amphipod survival was achieved.

8. REFERENCES

EPA, 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates Second Edition. EPA 600/R-99/064.

Rediske, R., M, Chu,, D. Uzarski, G. Peaslee, J. Gabrosek. 2004. Phase II Investigation of Sediment Contamination in White Lake. EPA-905-R-04-001.

Appendix A

Summary of Chemical Measurements for the Toxicity Test with Sediments from White Lake (2011)

TABLE A-1. RESULTS OF CHEMICAL ANALYSIS OF THE CULTURE WATER DURING TOXICITY EXPERIMENTS WHITE LAKE SEDIMENTS OCTOBER 2011.

Sample	Parameter	Day		Difference
		0	10	(%)
Duck Lake	pH	8.09	8.20	1
	Conductivity (umhos/cm)	718.6	678.5	6
	Alkalinity (mg/l $CaCO_3$)	179.0	193.0	8
	Hardness (mg/l $CaCO_3$)	163	140	14
	Ammonia (mg/l NH_3)	1.79	0.53	70
WL-1	pH	8.18	8.17	0
	Conductivity (umhos/cm)	718.6	705.7	2
	Alkalinity (mg/l $CaCO_3$)	191.0	181.0	5
	Hardness (mg/l $CaCO_3$)	151	140	7
	Ammonia (mg/l NH_3)	5.86	0.26	96
WL-2	pH	8.49	7.97	6
	Conductivity (umhos/cm)	661.7	741.7	12
	Alkalinity (mg/l $CaCO_3$)	181.3	192.3	6
	Hardness (mg/l $CaCO_3$)	136	150	10
	Ammonia (mg/l NH_3)	1.87	0.84	55
WL-21A	pH	8.18	8.14	0
	Conductivity (umhos/cm)	677.7	630.4	7
	Alkalinity (mg/l $CaCO_3$)	186.0	180.0	3
	Hardness (mg/l $CaCO_3$)	147	133	10
	Ammonia (mg/l NH_3)	1.75	0.14	92
WL-21B	pH	8.24	8.12	1
	Conductivity (umhos/cm)	674.4	673.7	0
	Alkalinity (mg/l $CaCO_3$)	176.0	180.0	2
	Hardness (mg/l $CaCO_3$)	153	136	11
	Ammonia (mg/l NH_3)	2.37	0.38	84
WL-21C	pH	8.25	8.09	2
	Conductivity (umhos/cm)	677.7	682.5	1
	Alkalinity (mg/l $CaCO_3$)	181.0	191.0	6
	Hardness (mg/l $CaCO_3$)	153	140	8
	Ammonia (mg/l NH_3)	2.44	0.15	94

TABLE A-2. SUMMARY OF DAILY DISSOLVED OXYGEN AND TEMPERATURE MEASUREMENTS DURING TOXICITY TESTS WITH WHITE LAKE SEDIMENT OCTOBER 2011.

Sample		Time	Day 0		Day 1		Day 2		Day 3		Day 4		Day 5		Day 6		Day 7		Day 8		Day 9		Day 10	
ID			DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp	DO	Temp
Duck Lake	AM	830	5.38	20.1	5.43	22.9	8.31	23.1	6.22	22.1	5.36	23.2	5.70	23.6	5.33	23.4	5.58	23.9	5.81	23.2	5.57	23.2	5.59	23.4
	PM	1600	4.66	23.9	5.09	24.4	5.25	24.5	5.63	22.2	5.5	23.0	7.09	24.4	4.21	24.9	5.94	24.2	7.82	21.9	5.56	23.5	5.72	23.1
WL-1	AM	830	6.23	19.9	6.23	23.0	7.21	22.3	6.01	22.0	5.25	23.2	6.12	24.0	5.34	23.6	5.44	24.1	6.17	23.2	5.76	23.3	6.04	23.7
	PM	1600	5.45	24.2	5.84	24.8	5.87	24.9	5.78	22.1	5.56	24.7	4.66	23.7	4.72	25.0	6.02	23.8	7.56	22.5	4.86	24.2	6.28	22.7
WL-2	AM	830	6.10	19.9	6.12	22.9	7.91	21.8	6.92	21.8	5.68	23.3	5.66	23.9	5.13	23.7	5.53	24.2	5.55	23.5	5.33	23.5	5.60	23.8
	PM	1600	5.01	24.1	5.96	24.5	6.07	24.6	6.39	22.2	5.92	22.9	7.58	23.2	4.42	24.9	5.55	23.1	7.25	22.5	5.83	24.4	5.88	21.6
WL-21A	AM	830	6.31	19.5	6.44	22.1	7.61	21.3	6.93	20.4	5.95	22.5	6.02	23.0	5.80	22.6	5.95	23.0	5.58	24.0	5.42	23.8	5.46	24.3
	PM	1600	5.33	24.9	6.19	23.2	6.12	23.4	6.34	21.5	6.05	21.9	6.04	22.9	4.61	23.6	6.13	21.6	6.25	23.5	4.32	25.0	5.95	21.2
WL-21B	AM	830	5.54	19.7	5.94	22.6	7.96	21.1	6.33	20.9	5.36	22.5	5.52	22.6	5.24	22.2	5.37	22.5	6.47	23.8	5.69	23.7	6.08	23.9
	PM	1600	5.45	23.0	5.77	23.4	5.96	23.3	5.48	22.3	5.55	21.2	6.25	22.2	4.57	23.0	5.69	20.4	6.90	23.2	5.77	24.6	7.03	22.3
WL-21C	AM	830	6.74	20.0	6.47	23.2	7.89	21.0	6.47	20.1	5.72	23.4	6.07	23.4	5.40	23.2	5.55	23.5	6.14	23.8	5.11	23.5	5.87	23.9
	PM	1600	5.52	23.4	6.24	23.7	6.49	23.7	6.30	22.5	6.16	22.2	7.75	22.7	4.57	23.7	6.23	21.4	6.21	23.1	5.86	22.9	6.21	23.1