

MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
WATER RESOURCES DIVISION
MAY 2012

STAFF REPORT

A SEDIMENT CHEMISTRY OF LAKE SUPERIOR SHORELINE IN THE VICINITY OF GAY,
KEWEENAW AND HOUGHTON COUNTIES, MICHIGAN
AUGUST 26, 27, AND 28, 2008

INTRODUCTION

Staff of the Surface Water Assessment Section conducted a sediment chemistry survey of Lake Superior Shoreline in the vicinity of Gay Michigan, Keweenaw and Houghton County, Michigan. Mining activities in the Village of Gay, Michigan, conducted between the late 1800s and early 1900s, generated approximately 38,000,000 cubic yards of copper mining waste referred to as stamp sands. These stamp sand wastes were deposited in or along Lake Superior in the vicinity of Gay, Michigan. Other stamp sand waste piles in the Keweenaw Peninsula have been shown to pose unacceptable impacts to aquatic organisms. Previous studies of the Gay stamp sand piles (Weston, 2006) measured copper concentrations and toxicity to aquatic life with material collected from the upland portion of the stamp sand piles. Results of that study showed the Gay stamp sand material has the potential to be toxic to aquatic life. The objective of this study was to evaluate the sediment and water column chemical characteristics in Lake Superior and determine potential impacts to designated uses.

SUMMARY

1. Eleven locations were sampled for water column chemistry and analyzed for calcium, magnesium, hardness, mercury, arsenic, barium, cadmium, chromium, copper, lead, nickel, selenium, silver, zinc, and total organic carbon (Appendix I).
2. Nine locations were sampled for discrete sediments and analyzed for mercury, arsenic, barium, cadmium, chromium, copper, lead, nickel, selenium, silver, and zinc (Appendix I).
3. Elevated copper concentrations (>149 mg/kg MacDonald, 2000) were detected in 8 of the 9 sediment samples (300 mg/kg–8500 mg/kg dry). Three water column samples also showed elevated levels of copper (4-22 µg/l).
4. Sediment samples around the Traverse River outfall had the lowest sediment copper concentrations. Concentrations of copper are highest between the pier at the Traverse River outfall and the village of Gay which coincides with the largest deposits of stamp sand.
5. Bulk sediment toxicity testing showed that all sediment samples were acutely toxic to both *Chironomus dilutus* and *Hyalella azteca*.

METHODS

Water and sediment samples were collected via grab samples, preserved, and transported according to procedures contained in the Michigan Department of Environmental Quality (MDEQ), Water Resources Division's (WRD) Quality Assurance Manual (MDNR, 1994).

Bulk sediment toxicity testing was conducted by Great Lakes Environmental Center (GLEC) following procedures outlined by EPA/600/R-99/064 (USEPA, 2000). These tests were 10-day whole sediment survival and growth tests using *Chironomus dilutus* and *Hyalella azteca*. Both water column and sediment samples were analyzed by GLEC. Detailed results of bulk sediment toxicity and sample analysis can be found in Appendix II.

RESULTS AND DISCUSSION

Sediment sampling locations for this study are presented in Figure 1. Results of the water column and sediment chemistry are presented in Table 1. For purposes of evaluation, sediment analysis results were compared to the threshold effect concentration (TEC) and probable effect concentrations (PEC) described in "Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems" (MacDonald et. al, 2000). The TEC and PEC values are used as screening tools to assess the potential for aquatic life effects. The TEC is the sediment concentration below which impacts to aquatic life would be less likely to occur. The PEC is the sediment concentration above which impacts to aquatic life would be more likely to occur. Water chemistry results were evaluated by comparing sample results to MDEQ, WRD's Rule 57 Water Quality Values.

Given the historic use of the area as a dump area for copper mine waste, not surprisingly elevated levels of copper were detected in the stamp sands that engulf the shoreline area between the Village of Gay and the mouth of the Traverse River. Samples results throughout the study area showed concentrations of copper above the MacDonald PEC screening levels suggesting negative impacts to aquatic life are likely occurring. Bulk sediment toxicity testing supports this conclusion based on significant reductions in growth and survival of both test organisms (Table 1).

Remedial alternatives are currently being evaluated by the United States Army Corps of Engineers. The WRD will continue to monitor the stamp sands within the vicinity of Gay throughout the future.

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Eric Alexander, Aquatic Biologist
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Report By: Bill Keiper, Aquatic Biologist
Michael Alexander, Aquatic Biologist
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LITERATURE CITED

- MacDonald, D. D., C. G. Ingersoll, T. A. Berger. 2000. Development and Evaluation of Consensus-based Sediment Quality Guidelines for Freshwater Ecosystems. Archives of Environmental Contamination and Toxicology. 39, 20-31.
- MDNR. 1994. Quality Assurance Manual for Water, Sediment, and Biological Sampling. MDNR. SWQD.
- USEPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates EPA 600/R-99/064
- Weston. 2006. Toxicological Evaluations for the Gay, Michigan Stamp Sand W.O. No. 2008.032.002

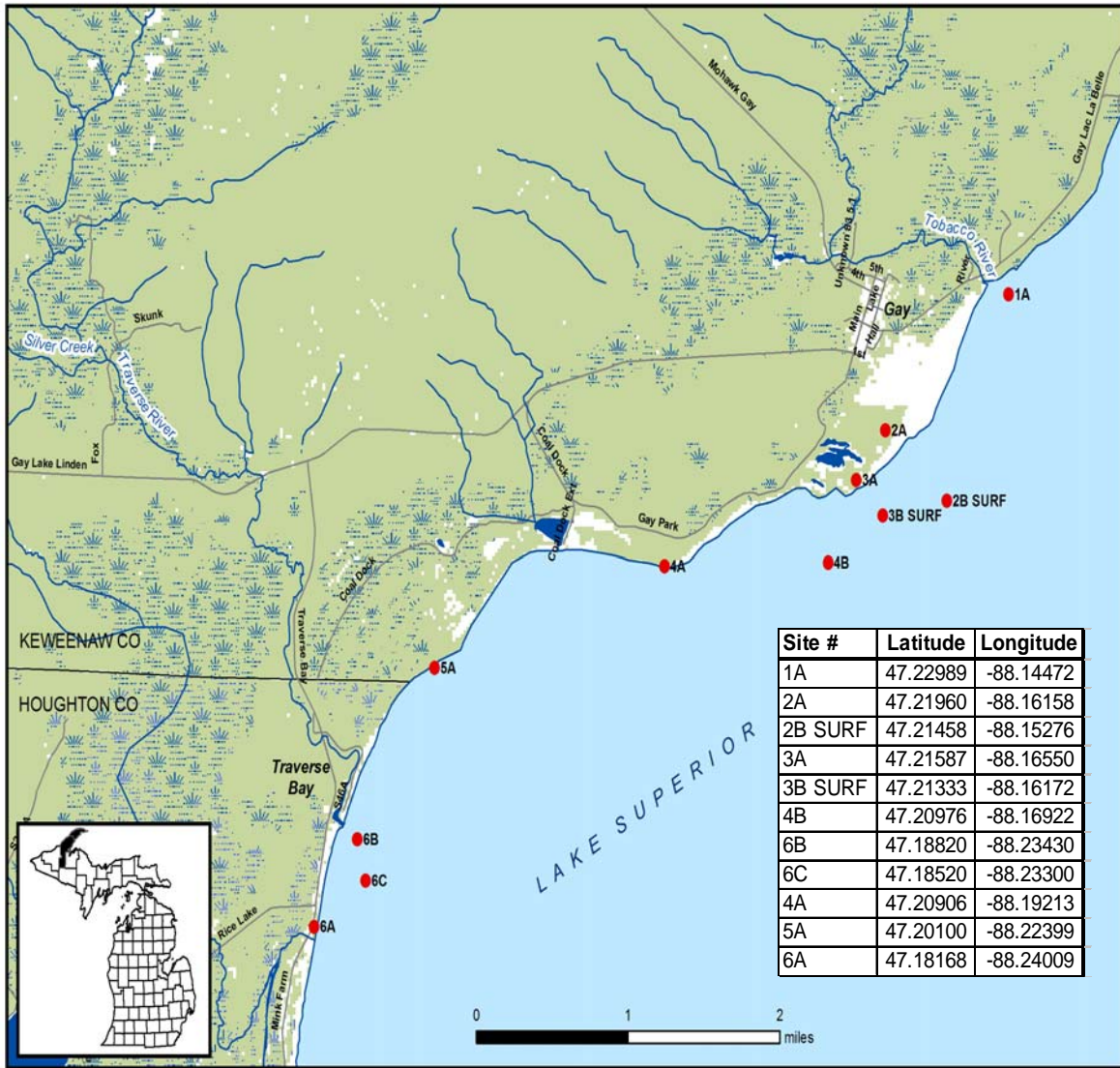


Figure 1. 2008 Sampling locations for sediment and water column analysis of the shoreline within the vicinity of Gay, MI.

Table 1. Results of water column and sediment samples taken August 26-28, 2008 for copper (CU) compared to bulk sediment toxicity results.

Sample location	Lat - Long	Copper H2O ug/L	Hardness ug/L	Copper Sediment mg/kg	Copper PEC mg/kg	<i>C. dilutus</i> % survival	<i>C. dilutus</i> weight mg	<i>H. azteca</i> % survival	<i>H. azteca</i> weight mg
1A	N 47.22989 - W 088.14472	ND	46		149				
2A	N 47.21960 - W 088.16158	4.9*	46	2,100	149	87.5	0.587^a	60.0^a	0.043^a
2B SURF	N 47.21458 - W 088.15276	ND	44		149				
2B BOTTOM		ND	44		149				
3A	N 47.21587 - W 088.16550	2.8	45	1,900	149				
3B SURF	N 47.21333 - W 088.16172	ND	44		149				
3B BOTTOM		ND	44	1,500	149	92.5	0.667^a	57.5^a	0.040^a
4A	N 47.20906 - W 088.19213	22*	47	2,300	149	83.8	0.815^a	85	0.059^a
4B	N 47.20976 - W 088.16922	ND	46	1,500	149	75	0.439^a	58.8^a	0.039^a
5A	N 47.20100 - W 088.22399	21*	46	8,500	149	91.3	0.815^a	65.0^a	0.060^a
6A	N 47.18168 - W 088.24009	3.7	45	79	149	100	1.114^a	91.3	0.059^a
6B	N 47 11.292 - W 88 14.059	1.1	45	400	149	93.8	0.858^a	73.8^a	0.050^a
6C	N 47 11.113 - W 88 13.979			300	149				
Lab control						92.5	1.48	95	0.112

^a Significant difference ($p \leq 0.05$) from laboratory sediment control

* Exceeds water quality standards (WQS) for Copper: Chronic (4.53 ug/L) and Acute (12.70 ug/L) values

Appendix I
WATER COLUMN CHEMISTRY AND
DISCRETE SEDIMENTS
SAMPLE DATA



MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
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P.O. Box 30270
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Division: WB
Report to: MIKE ALEXANDER
 MDEQ-SWAS-LANSING
 CONSTITUTION HALL
 525 W. ALLEGAN, LANSING, MI 48909

Lab Work Order #: 80800357
Work Site ID: LB040649
Site Name: LAKE SUPERIOR STAMP SAND
Received: 08/29/2008
Reported: 09/24/2008
Collected By: MIKE ALEXANDER

Total: \$1,848.00

Samples Received :

No:	Sample ID	Sample Description	Matrix:	Collection Date
01	AB22260	1A	WASTE WATER	08/28/2008
02	AB22261	2A	WASTE WATER	08/28/2008
03	AB22262	2B SURF	WASTE WATER	08/28/2008
04	AB22263	2B BOTTOM	WASTE WATER	08/28/2008
05	AB22264	3A	WASTE WATER	08/28/2008
06	AB22265	3B SURF	WASTE WATER	08/28/2008
07	AB22266	3B BOTTOM	WASTE WATER	08/28/2008
08	AB22267	4B	WASTE WATER	08/28/2008
09	AB22268	6B	WASTE WATER	08/28/2008
10	AB22269	4A	WASTE WATER	08/27/2008
11	AB22270	5A	WASTE WATER	08/27/2008
12	AB22271	6A	WASTE WATER	08/27/2008

I certify that the analysis performed by the MDEQ Environmental Laboratory are accurate and that the laboratory tests were conducted by methods approved by the U.S. Environmental Protection Agency and other appropriate regulatory agencies.

Bob Avery, Laboratory Director



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Sample	AB22260	1A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.8	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.8	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	46	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	.2	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.3	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

Sample	AB22261	2A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.9	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	46	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.7	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	4.9	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

CAS# : Chemical Abstract Service Registry Number
 RL : Reporting Limit
 ND : Not Detected

ug / L : microgram / liter (ppb)
 mg / L : milligram / liter (ppm)
 ug / Kg : microgram / kilogram (ppb)
 mg / Kg : milligram / kilogram (ppm)

Laboratory Contacts
 Inorganic Unit Mgr: Sandy Gregg
 Organic Unit Mgr: Carol Smith
 Systems Mgmt Unit: George Krisztian



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Sample	AB22262	2B SURF						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.1	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	44	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.7	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

Sample	AB22263	2B BOTTOM						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.2	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	44	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.3	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.3	mg/L	0.5		09/03/2008	415.1	MB

CAS# : Chemical Abstract Service Registry Number
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ug / L : microgram / liter (ppb)
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Sample	AB22264	3A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.6	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	45	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	1.9	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.5	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	2.8	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.3	mg/L	0.5		09/03/2008	415.1	MB

Sample	AB22265	3B SURF						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.0	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	44	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.2	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

CAS# : Chemical Abstract Service Registry Number
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Sample	AB22266	3B BOTTOM						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.2	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	44	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.2	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.3	mg/L	0.5		09/03/2008	415.1	MB

Sample	AB22267	4B						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.8	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	46	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	.3	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	9.4	µg/L	5		09/09/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/09/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

CAS# : Chemical Abstract Service Registry Number
 RL : Reporting Limit
 ND : Not Detected

ug / L : microgram / liter (ppb)
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Sample	AB22268	6B						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.4	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	45	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	8.4	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	1.1	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.5	mg/L	0.5		09/03/2008	415.1	MB

Sample	AB22269	4A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	14.1	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.8	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	47	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	9.0	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	22	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.3	mg/L	0.5		09/03/2008	415.1	MB

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Sample	AB22270	5A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.7	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.8	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/08/2008	3010/200	TK2
	Hardness - Calculated	46	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	10	µg/L	5		09/09/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	21	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/09/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

Sample	AB22271	6A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
7440-70-2	Calcium - Total	13.4	mg/L	1		09/03/2008	7140/215.1	LAV
7439-95-4	Magnesium - Total	2.7	mg/L	1		09/03/2008	7450/242.1	LAV
	Digestion Metals Water	Completed				09/09/2008	3010/200	TK2
	Hardness - Calculated	45	mg/L	5		09/03/2008	Calculated	LV
	Digestion Mercury Water	Completed				09/15/2008	7470/245.1	TK2
7439-97-6	Mercury - Total	ND	µg/L	0.2		09/18/2008	7470/245.1	TS
7440-38-2	Arsenic - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-39-3	Barium - Total	9.3	µg/L	5		09/10/2008	6020/200.8	KS
7440-43-9	Cadmium - Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-47-3	Chromium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-50-8	Copper - Total	3.7	µg/L	1		09/10/2008	6020/200.8	KS
7439-92-1	Lead - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-02-0	Nickel - Total	ND	µg/L	2.0		09/10/2008	6020/200.8	KS
7782-49-2	Selenium - Total	ND	µg/L	1		09/10/2008	6020/200.8	KS
7440-22-4	Silver -Total	ND	µg/L	0.2		09/10/2008	6020/200.8	KS
7440-66-6	Zinc - Total	ND	µg/L	10		09/10/2008	6020/200.8	KS
7440-44-0	TOC	1.4	mg/L	0.5		09/03/2008	415.1	MB

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<u>Qualifier Code</u>	<u>Qualifier Description</u>
1	Result(s) and RL(s) are estimated due to low surrogate recovery.
2	Result is estimated due to high surrogate recovery.
3	Result(s) and RL(s) are estimated due to low matrix spike recovery.
4	Result is estimated due to high matrix spike recovery.
5	Result and RL are estimated due to low continuing calibration standard criteria failure.
6	Result is estimated due to high continuing calibration standard criteria failure.
7	Result(s) and RL(s) are estimated due to poor precision.
8	Result(s) and RL(s) are estimated due to low recovery of batch QC.
9	Result outside QC acceptance criteria.
A	Value reported is the mean of two or more determinations.
C	Value calculated from other independent parameters.
D	Analyte value quantified from a dilution(s); reporting limit (RL) raised.
E	Result is estimated due to high recovery of batch QC.
F	Amenable cyanide was not analyzed due to low level of total cyanide.
G	Result and RL are estimated due to initial calibration standard criteria failure.
H	Recommended laboratory holding time was exceeded.
I	Dilution required due to matrix interference; reporting limit (RL) raised.
J	Analyte was positively identified. Value is an estimate.
JA	Result is estimated due to multiple Aroclors present.
JC	Result is estimated since confirmation analysis did not meet acceptance criteria
JD	Due to severe degradation, specific Aroclor identification is difficult and quantitation is estimated.
K	RL(s) raised due to matrix interferences.
KR	RL(s) raised due to low sample volume submitted.
KS	RL(s) raised due to low total solids.
KW	RL(s) raised due to light sample weight.
LB	Reported library search compounds are tentative identifications with estimated concentrations.
M	The level of the method preparation blank (MPB) is reported in the qualifier column.
N	Non-homogeneous sample made analysis of sample questionable.
O	Result and RL estimated due to analysis from an open vial.
P	Recommended sample collection/preservation technique not used; reported result(s) is an estimate.
Q	Quantity of sample insufficient to perform analyses requested.
R	Result confirmed by re-extraction and analysis.
S	Supernatant analyzed.
T	Reported value is less than the reporting limit (RL). Result is estimated.
V	Value not available due to dilution.
W	Reported value is less than the method detection limit (MDL).
X	Methods 8260 & 624 are used to analyze volatile organics that have boiling points below 200°C. 2-Methylnaphthalene & naphthalene have boiling points above 200°C and are better suited to analysis by methods 8270 or 625 as semivolatile organics.
PI	Possible interference may have affected the accuracy of the laboratory result
Z	Result reported below the RL to meet the TDL in RRD Op Memo 2 (10/22/04) multiplied by applicable dilution factor.

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Division: WB
Report to: MIKE ALEXANDER
 MDEQ-SWAS-LANSING
 CONSTITUTION HALL
 525 W. ALLEGAN, LANSING, MI 48909

Lab Work Order #: 80800358
Work Site ID: LB040649
Site Name: LAKE SUPERIOR STAMP SAND
Received: 08/29/2008
Reported: 09/26/2008
Collected By: MIKE ALEXANDER

Total: \$1,084.50

Samples Received :

No:	Sample ID	Sample Description	Matrix:	Collection Date
01	AB22272	2A	SEDIMENT	08/26/2008
02	AB22273	3A	SEDIMENT	08/26/2008
03	AB22274	3B	SEDIMENT	08/26/2008
04	AB22275	4B	SEDIMENT	08/26/2008
05	AB22276	6B	SEDIMENT	08/26/2008
06	AB22277	6C	SEDIMENT	08/26/2008
07	AB22278	4A	SEDIMENT	08/27/2008
08	AB22279	5A	SEDIMENT	08/27/2008
09	AB22280	6A	SEDIMENT	08/27/2008

I certify that the analysis performed by the MDEQ Environmental Laboratory are accurate and that the laboratory tests were conducted by methods approved by the U.S. Environmental Protection Agency and other appropriate regulatory agencies.

Bob Avery, Laboratory Director



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Sample	AB22272	2A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	2.2	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	7.3	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	31	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	2100	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.6	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	1.2	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	62	mg/Kg dry	1		09/22/2008	6020	KS
	% Total Solids	79.8	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

Sample	AB22273	3A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	1.9	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	7.9	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	0.31	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	36	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	1900	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.5	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	1.3	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	77	mg/Kg dry	1		09/22/2008	6020	KS
	% Total Solids	77.7	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

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Sample	AB22274	3B						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	1.9	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	6.6	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	33	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	1500	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.6	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	1.6	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	67	mg/Kg dry	1		09/22/2008	6020	KS
	% Total Solids	78.9	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

Sample	AB22275	4B						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	1.7	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	7.6	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	34	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	1500	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.7	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	1.4	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	69	mg/Kg dry	1		09/22/2008	6020	KS
	% Total Solids	78.4	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

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Sample	AB22276	6B						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	1.2	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	7.1	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	21	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	400	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.8	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	0.57	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	39	mg/Kg dry	1		09/19/2008	6020	KS
	% Total Solids	80.7	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

Sample	AB22277	6C						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	1.5	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	9.0	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	0.34	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	19	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	300	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	3.0	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	0.74	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	42	mg/Kg dry	1		09/19/2008	6020	KS
	% Total Solids	75.2	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

CAS# : Chemical Abstract Service Registry Number
 RL : Reporting Limit
 ND : Not Detected

ug / L : microgram / liter (ppb)
 mg / L : milligram / liter (ppm)
 ug / Kg : microgram / kilogram (ppb)
 mg / Kg : milligram / kilogram (ppm)

Laboratory Contacts
 Inorganic Unit Mgr: Sandy Gregg
 Organic Unit Mgr: Carol Smith
 Systems Mgmt Unit: George Krisztian



MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY
 ENVIRONMENTAL LABORATORY

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 TEL: (517) 335-9800
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Sample	AB22278	4A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	2.1	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	8.5	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	37	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	2300	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.9	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	1.7	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	78	mg/Kg dry	1		09/22/2008	6020	KS
	% Total Solids	79.4	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

Sample	AB22279	5A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	3.1	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	8.0	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	39	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	8500	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	2.1	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	1.6	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	79	mg/Kg dry	1		09/22/2008	6020	KS
	% Total Solids	80.5	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

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Sample	AB22280	6A						
CAS#	Analyte Name	Result	Unit	RL	Qualifier	Date Tested	Method	Analyst
	Digest Mercury - Sediment	Completed				09/02/2008	7471	TK2
7439-97-6	Mercury - Sediment	ND	mg/Kg dry	0.05		09/03/2008	7471	TS
7440-38-2	Arsenic - Sediment	0.77	mg/Kg dry	0.5		09/19/2008	6020	KS
7440-39-3	Barium - Sediment	2.7	mg/Kg dry	1		09/19/2008	6020	KS
7440-43-9	Cadmium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-47-3	Chromium - Sediment	3.9	mg/Kg dry	2		09/19/2008	6020	KS
7440-50-8	Copper - Sediment	79	mg/Kg dry	1		09/22/2008	6020	KS
	Digest Metals - Sediment	Completed				09/15/2008	3050	RG
7439-92-1	Lead - Sediment	ND	mg/Kg dry	1		09/19/2008	6020	KS
7782-49-2	Selenium - Sediment	ND	mg/Kg dry	0.2		09/19/2008	6020	KS
7440-22-4	Silver - Sediment	0.21	mg/Kg dry	0.1		09/19/2008	6020	KS
7440-66-6	Zinc - Sediment	9.1	mg/Kg dry	1		09/19/2008	6020	KS
	% Total Solids	83.3	%	0.1		09/02/2008		DB
	Drying and Grinding - Sediment	Completed				09/10/2008		DB

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<u>Qualifier Code</u>	<u>Qualifier Description</u>
1	Result(s) and RL(s) are estimated due to low surrogate recovery.
2	Result is estimated due to high surrogate recovery.
3	Result(s) and RL(s) are estimated due to low matrix spike recovery.
4	Result is estimated due to high matrix spike recovery.
5	Result and RL are estimated due to low continuing calibration standard criteria failure.
6	Result is estimated due to high continuing calibration standard criteria failure.
7	Result(s) and RL(s) are estimated due to poor precision.
8	Result(s) and RL(s) are estimated due to low recovery of batch QC.
9	Result outside QC acceptance criteria.
A	Value reported is the mean of two or more determinations.
C	Value calculated from other independent parameters.
D	Analyte value quantified from a dilution(s); reporting limit (RL) raised.
E	Result is estimated due to high recovery of batch QC.
F	Amenable cyanide was not analyzed due to low level of total cyanide.
G	Result and RL are estimated due to initial calibration standard criteria failure.
H	Recommended laboratory holding time was exceeded.
I	Dilution required due to matrix interference; reporting limit (RL) raised.
J	Analyte was positively identified. Value is an estimate.
JA	Result is estimated due to multiple Aroclors present.
JC	Result is estimated since confirmation analysis did not meet acceptance criteria
JD	Due to severe degradation, specific Aroclor identification is difficult and quantitation is estimated.
K	RL(s) raised due to matrix interferences.
KR	RL(s) raised due to low sample volume submitted.
KS	RL(s) raised due to low total solids.
KW	RL(s) raised due to light sample weight.
LB	Reported library search compounds are tentative identifications with estimated concentrations.
M	The level of the method preparation blank (MPB) is reported in the qualifier column.
N	Non-homogeneous sample made analysis of sample questionable.
O	Result and RL estimated due to analysis from an open vial.
P	Recommended sample collection/preservation technique not used; reported result(s) is an estimate.
Q	Quantity of sample insufficient to perform analyses requested.
R	Result confirmed by re-extraction and analysis.
S	Supernatant analyzed.
T	Reported value is less than the reporting limit (RL). Result is estimated.
V	Value not available due to dilution.
W	Reported value is less than the method detection limit (MDL).
X	Methods 8260 & 624 are used to analyze volatile organics that have boiling points below 200°C. 2-Methylnaphthalene & naphthalene have boiling points above 200°C and are better suited to analysis by methods 8270 or 625 as semivolatile organics.
PI	Possible interference may have affected the accuracy of the laboratory result
Z	Result reported below the RL to meet the TDL in RRD Op Memo 2 (10/22/04) multiplied by applicable dilution factor.

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 RL : Reporting Limit
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 mg / L : milligram / liter (ppm)
 ug / Kg : microgram / kilogram (ppb)
 mg / Kg : milligram / kilogram (ppm)

Laboratory Contacts
 Inorganic Unit Mgr: Sandy Gregg
 Organic Unit Mgr: Carol Smith
 Systems Mgmt Unit: George Krisztian

Appendix II

**GREAT LAKES ENVIRONMENTAL CENTER INC. FINAL REPORT:
CHIRONOMUS DILUTUS AND *HYALELLA AZTECA* 10-DAY WHOLE SEDIMENT
TOXICITY TESTING RESULTS FOR STAMP SANDS SEDIMENT SAMPLES
COLLECTED NEAR THE VILLAGE OF GAY, MICHIGAN**



December 10, 2008

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**RE: FINAL REPORT: *CHIRONOMUS DILUTUS* AND *HYALELLA AZTECA*
10-DAY WHOLE SEDIMENT TOXICITY TESTING RESULTS FOR
STAMP SANDS SEDIMENT SAMPLES COLLECTED NEAR THE
VILLAGE OF GAY, MICHIGAN**

Dear Mr. Alexander:

Great Lakes Environmental Center Inc. (GLEC) has completed our analysis of the *Chironomus dilutus* (*tentans*) and *Hyaella azteca* 10-day whole sediment survival and growth toxicity tests performed with seven sediment samples, collected by Michigan Department of Environmental Quality (MDEQ) personnel for whole sediment toxicity assessment. The sample identification numbers, average percent survival and average dry weight test results are summarized in Tables 1 and 2 for the *C. dilutus* tests and in Tables 3 and 4 for the *H. azteca* tests. Water quality data for the overlying water is summarized for each sediment sample in Table 5 for the *C. dilutus* test and Table 6 for the *H. azteca* test. A detailed summary of the overlying water quality measurements is provided in Appendix B. A summary of the statistical analyses conducted on the whole sediment toxicity test data is provided in Table 7 for the *C. dilutus* test and Table 8 for the *H. azteca* test. (The daily laboratory bench data sheets are kept on file at GLEC and can be provided to you upon your request.) Chain of Custody forms and reference toxicant data are provided in Appendix A and F, respectively.

METHODS

The sediment samples were analyzed at our Traverse City, Michigan laboratory following GLEC's written protocols which are based on the procedures outlined by: EPA/600/R-99/064 *Methods for Measuring the Toxicity and Bioaccumulation of*

Sediment-Associated Contaminants with Freshwater Invertebrates, Second Edition; ASTM 1706-95B, *Standard Test Methods for Measuring the Toxicity of Sediment Associated Contaminants with Freshwater Invertebrates* (ASTM 2000); and GLEC Standard Operating Procedures (SOPs).

The seven sediment samples were hand delivered by MDEQ personnel and were received at GLEC, where they were assigned a unique GLEC laboratory identification number and stored at 4°C until test initiation (see table below).

Sample I.D.	GLEC Number	Date Sampled	Date Delivered	Date Received
3B	7430	August 28, 2008	August 28, 2008	August 28, 2008
2A	7431	August 28, 2008	August 28, 2008	August 28, 2008
6B	7432	August 28, 2008	August 28, 2008	August 28, 2008
4B	7433	August 28, 2008	August 28, 2008	August 28, 2008
6A	7434	August 27, 2008	August 28, 2008	August 28, 2008
5A	7435	August 27, 2008	August 28, 2008	August 28, 2008
4A	7436	August 27, 2008	August 28, 2008	August 28, 2008

The 10-day *C. dilutus* and *H. azteca* whole sediment toxicity tests were initiated on October 14 and October 17, 2008, respectively.

Second to third instar *C. dilutus* (10 days old at test initiation) and *H. azteca* (10 days old at test initiation) were used to initiate the whole sediment toxicity tests. *C. dilutus* and *H. azteca* were continuously exposed for 10 days to each of the sediment samples and to a laboratory control sediment. There were eight replicate beakers for each sediment sample and laboratory control; each replicate contained 10 animals. The laboratory control sediment is a reference sediment collected locally from the Boardman River, a blue ribbon trout stream, located in the Pere Marquette State Forest.

The *C. dilutus* and *H. azteca* were exposed in 300 mL high form beakers, each containing 100 mL of whole sediment and 175 mL of overlying water. Overlying water was supplied to each test chamber at least twice daily (once every 12-hour period) via a static-renewal water delivery system. The overlying water consisted of de-chlorinated municipal (Lake Michigan) water of moderate hardness (160-180 mg/L). The *C. dilutus* test chambers were fed 1.5 mL of Tetrafin® goldfish food slurry (4mg/mL dry solids) daily. The *H. azteca* test chambers were fed 1.0 mL of YTC (1800 mg/L solids) daily.

The test chambers were placed in a temperature controlled water bath under the specified conditions of 23 ± 1 °C; photoperiod of 16 hours light and 8 hours dark; and ambient lighting. Water temperature was monitored continuously in the water bath using an electronic data logger (Appendix E) while temperatures in the test chambers were measured daily in two alternating replicates for each test sample. Alkalinity, hardness, pH and total ammonia were measured at test initiation and at test termination (Tables 5 and 6). Observations on organism behavior were made daily in each test chamber and recorded on the laboratory bench data sheets. The number of *C. dilutus* and *H. azteca* surviving was recorded at test termination (10 days) and a summary of the percent survival is provided in Tables 1 and 3, respectively. The average ash free dry weight (AFDW) (mg) for each *C. dilutus* replicate and the average dry weight (mg) for each *H. azteca* replicate was also determined at test termination and summarized in Tables 2 and 4, respectively.

A statistical analysis using TOXSTAT (3.5, 1996) and statistical guidelines provided in EPA/600/R-99/064 *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates*, Second Edition; and ASTM 1706-95B, *Standard Test Methods for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates* (ASTM 2000), was used to compare the 10-day survival and growth endpoints. Initially, all survival data were transformed using an arc sine-square root transformation prior to analysis. The transformed data were then tested for normality and homogeneity of variances. Next, an analysis of variance (ANOVA) was conducted using the most appropriate parametric or nonparametric *t* test. If the data failed to meet the assumptions of normality or homogeneity, then the nonparametric tests were used to analyze the data. The percent survival in each investigative sample was considered statistically different when it was significantly lower ($p \leq 0.05$) than in the control sediments.

RESULTS

Chironomus dilutus

The organisms in the laboratory control sediment exceeded the minimum survival (70 percent or greater) and growth (0.48 mg or greater AFDW at test termination) criteria for an acceptable control for the *C. dilutus* test (Tables 1 and 2). The acceptable requirements for survival and growth for the *C. dilutus* test can be found in EPA /600/R-99/064, Table 12.1. The overlying water quality measurements (Table 5) were also within the acceptable limits following the EPA testing protocol (i.e., daily mean temperatures were $23^\circ\text{C} \pm 1$ °C; dissolved oxygen (D.O.) was maintained above 2.5 mg/L in the overlying water; and there were no variations greater than 50% in overlying water hardness,

alkalinity or ammonia measurements within each test type). Consequently, the *C. dilutus* whole sediment toxicity tests were conducted following the standard protocols and are valid assessments of sediment toxicity, with the following exception. On October 22, 2008, the D.O. in the overlying water of laboratory control sediment fell below 2.5 mg/L. In response, a third overlying water renewal was added to all of the controls and investigative test sediments. The brief drop in D.O. was unlikely to have affected the test results (see EPA /600/R-99/064 manual, section 12.3.6.2.2). All test chambers were observed daily to assess organism behavior and no unusual observations were noted with the test organisms.

Laboratory Control Compared to Investigative Sediment Samples

There was no statistically significant reduction ($p \geq 0.05$) in *C. dilutus* survival for the seven investigative samples after 10 days of exposure, when compared to the laboratory control sediment (Table 1). However, growth (measured as AFDW in mg) was significantly reduced ($p \leq 0.05$) in the seven investigative sediment samples (3B, 2A, 6B, 4B, 6A, 5A, and 4A) when compared to the laboratory control sediment (Table 2).

A summary of the survival and growth statistical analysis for the *C. dilutus* whole sediment toxicity tests are provided in Table 7 and Appendix C.

Hyaella azteca

The *H. azteca* in the laboratory control sediment exceeded the minimum survival criteria (80%) and had measurable growth relative to the weight of organisms at test initiation (Tables 3 and 4). The requirements for acceptable survival and growth for the *H. azteca* can be found in EPA /600/R-99/064, Table 11.2. The overlying water quality measurements (Table 6) were also within the acceptable limits following the EPA testing protocol (i.e., daily mean temperatures were 23 ± 1 °C; D.O. was maintained above 2.5 mg/L in the overlying water; and there were no variations greater than 50% in overlying water hardness, alkalinity or ammonia measurements within each test type). All test chambers were checked daily to assess organism behavior and no unusual observations were noted. Consequently, the *H. azteca* whole sediment toxicity tests are valid assessments of sediment toxicity.

Laboratory Control Compared to Investigative Sediment Samples

There was a statistically significant reduction ($p \leq 0.05$) in *H. azteca* survival after 10 days of exposure in five of the seven investigative samples (3B, 2A, 6B, 4B, and 5A) when compared to the laboratory control sediment (Table 3). *H. azteca* growth (measured as average dry weight in mg) was significantly reduced in the seven investigative samples (3B, 2A, 6B, 4B, 6A, 5A, and 4A) when compared to the laboratory control sediment (Table 4).

Mr. Michael Alexander
Michigan Department of Environmental Quality
Water Bureau

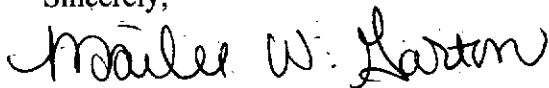
5

December 10, 2008

A summary of the survival and growth statistical analysis for the *H. azteca* whole sediment toxicity tests are provided in Table 8 and Appendix D.

If you have any questions regarding the results of these sediment toxicity tests, or if you would like additional information, please contact either me or Jamie Saxton at (231) 941-2230. Thank you for the opportunity to provide this service to the MDEQ and we look forward to continuing to provide environmental services to you in the future.

Sincerely,



Mailee W. Garton
Laboratory Coordinator

MWG:mg



TABLE 1. Comparison of Percent Survival Between the Laboratory Control and Investigative Sediment Samples (3B, 2A, 6B, 4B, 6A, 5A, and 4A) for the MDEQ Stamp Sands *Chironomus dilutus* 10-Day Whole Sediment Toxicity Tests Conducted October 14-24, 2008

REPLICATE #	Laboratory Control	3B GLC# 7430	2A GLC# 7431	6B GLC# 7432	4B GLC# 7433	6A GLC# 7434	5A GLC# 7435	4A GLC# 7436
1	9	6	9	10	7	10	9	6
2	10	6	10	6	1	10	10	7
3	9	9	8	10	9	10	8	10
4	9	9	10	10	9	10	10	9
5	8	10	7	9	10	10	8	10
6	9	9	10	10	10	10	10	9
7	10	9	6	10	5	10	9	9
8	10	7	10	10	9	10	9	7
10-Day Percent Survival	92.5	81.3	87.5	93.8	75.0	100.0	91.3	83.8

^a Significantly different ($p \leq 0.05$) from laboratory sediment control
 Note: Replicates initiated with 10 organisms



TABLE 2. Comparison of Average Ash-Free Dry Weight (mg) and Percent Survival Between the Laboratory Control and Investigative Sediment Samples (3B, 2A, 6B, 4B, 6A, 5A, and 4A) for the MDEQ Stamp Sands *Chironomus dilutus* 10-Day Whole Sediment Toxicity Tests Conducted October 14-24, 2008

REPLICATE #	Laboratory Control	3B GLC# 7430	2A GLC# 7431	6B GLC# 7432	4B GLC# 7433	6A GLC# 7434	5A GLC# 7435	4A GLC# 7436
1	1.630	0.768	0.781	1.002	0.454	1.291	1.138	0.937
2	1.414	0.662	0.647	0.930	0.100	1.061	0.713	0.696
3	1.418	0.513	0.729	0.801	0.521	1.049	0.985	0.752
4	1.516	0.630	0.463	1.066	0.510	1.006	0.683	0.730
5	1.546	0.486	0.731	0.753	0.309	1.015	0.984	0.906
6	1.420	0.726	0.474	0.653	0.375	0.715	0.633	0.679
7	1.390	0.488	0.368	0.675	0.552	1.010	0.692	0.823
8	1.508	1.060	0.506	0.982	0.688	1.767	0.688	0.997
Average Ash-Free Dry Weight (mg)	1.480	0.667^a	0.587^a	0.858^a	0.439^a	1.114^a	0.815^a	0.815^a
10-Day Percent Survival	92.5	81.3	87.5	93.8	75.0	100.0	91.3	83.8

^a Significantly different ($p \leq 0.05$) from laboratory sediment control
 Note: Average weight of *Chironomus dilutus* at day 0: 0.284 mg



TABLE 3. Comparison of Percent Survival Between the Laboratory Control and Investigative Sediment Samples (3B, 2A, 6B, 4B, 6A, 5A, and 4A) for the MDEQ Stamp Sands *Hyalella azteca* 10-Day Whole Sediment Toxicity Tests Conducted October 17-27, 2008

REPLICATE #	Laboratory Control	3B GLC# 7430	2A GLC# 7431	6B GLC# 7432	4B GLC# 7433	6A GLC# 7434	5A GLC# 7435	4A GLC# 7436
1	9	7	6	9	6	9	8	8
2	10	9	10	8	5	10	6	10
3	9	5	3	5	9	10	7	10
4	10	8	3	7	9	10	7	7
5	10	4	6	5	3	9	7	10
6	10	3	9	9	4	8	7	8
7	8	6	3	7	7	9	5	5
8	10	4	8	9	4	8	5	10
10-Day Percent Survival	95.0	57.5^a	60.0^a	73.8^a	58.8^a	91.3	65.0^a	85.0

^a Significantly different ($p \leq 0.05$) from laboratory sediment control
 Note: Replicates initiated with 10 organisms



TABLE 4. Comparison of Average Dry Weight (mg) and Percent Survival Between the Laboratory Control and Investigative Sediment Samples (3B, 2A, 6B, 4B, 6A, 5A, and 4A) for the MDEQ Stamp Sands *Hyalella azteca* 10-Day Whole Sediment Toxicity Tests Conducted October 17-27, 2008

REPLICATE #	Laboratory Control	3B GLC# 7430	2A GLC# 7431	6B GLC# 7432	4B GLC# 7433	6A GLC# 7434	5A GLC# 7435	4A GLC# 7436
1	0.129	0.041	0.035	0.040	0.025	0.069	0.091	0.056
2	0.125	0.039	0.036	0.048	0.060	0.075	0.052	0.056
3	0.097	0.046	0.057	0.054	0.029	0.057	0.046	0.059
4	0.082	0.059	0.063	0.069	0.030	0.056	0.106	0.056
5	0.115	0.038	0.038	0.052	0.043	0.046	0.027	0.054
6	0.099	0.053	0.028	0.038	0.055	0.046	0.053	0.048
7	0.131	0.008	0.043	0.049	0.031	0.054	0.030	0.078
8	0.114	0.033	0.045	0.048	0.038	0.070	0.076	0.062
Average Dry Weight (mg)	0.112	0.040^a	0.043^a	0.050^a	0.039^a	0.059^a	0.060^a	0.059^a
10-Day Percent Survival	95.0	57.5^a	60.0^a	73.8^a	58.8^a	91.3	65.0^a	85.0

^a Significantly different ($p \leq 0.05$) from laboratory sediment control
 Note: Average weight of *Hyalella azteca* at day 0: 0.024 mg



TABLE 5. Summary of Mean Water Quality Parameters of Overlying Water Samples Collected Immediately Prior to Renewal for the MDEQ Stamp Sands *Chironomus dilutus* 10-Day Whole Sediment Toxicity Tests Conducted October 14-24, 2008.

Sample ID GLC #	Temperature °C <i>n</i> =22	pH (s.u.) <i>n</i> =4	Dissolved Oxygen (mg/L) <i>n</i> =22	Specific Conductivity (μmhos) <i>n</i> =4	Alkalinity (mg/L CaCO ₃) <i>n</i> =2	Hardness (mg/L CaCO ₃) <i>n</i> =2	Total Ammonia (mg/L) <i>n</i> =2
Laboratory Control	22.9 (22.6-23.5)	7.48	4.6 (1.6-6.5)	294.5	94.0	140.0	0.27
3B GLC# 7430	23.0 (22.5-23.4)	7.68	6.4 (3.8-7.9)	291.8	104.0	130.0	0.21
2A GLC# 7431	22.9 (22.5-23.4)	7.71	6.5 (4.4-7.6)	294.5	109.0	130.0	0.22
6B GLC# 7432	22.9 (22.3-23.5)	7.74	6.1 (2.8-7.4)	290.3	102.0	130.0	0.16
4B GLC# 7433	23.0 (22.5-23.4)	7.76	6.5 (3.6-7.6)	289.3	109.0	130.0	0.19
6A GLC# 7434	23.0 (22.4-23.5)	7.73	6.6 (3.9-7.7)	285.8	108.0	126.0	0.19
5A GLC# 7435	23.0 (22.4-23.5)	7.73	6.6 (5.3-7.6)	289.3	93.0	130.0	0.18
4A GLC# 7436	22.9 (22.3-23.4)	7.76	6.3 (5.0-7.7)	290.5	107.0	130.0	0.17

Note: Ranges for temperature and dissolved oxygen are shown in parentheses.



TABLE 6. Summary of Mean Water Quality Parameters of Overlying Water Samples Collected Immediately Prior to Renewal for the MDEQ Stamp Sands *Hyaella azteca* 10-Day Whole Sediment Toxicity Tests Conducted October 17-27, 2008.

Sample ID GLC #	Temperature °C <i>n</i> =22	pH (s.u.) <i>n</i> =4	Dissolved Oxygen (mg/L) <i>n</i> =22	Specific Conductivity (µmhos) <i>n</i> =4	Alkalinity (mg/L CaCO ₃) <i>n</i> =2	Hardness (mg/L CaCO ₃) <i>n</i> =2	Total Ammonia (mg/L) <i>n</i> =2
Laboratory Control	22.7 (22.3-23.8)	7.91	6.5 (4.5-8.2)	293.8	107.0	132.0	0.09
3B GLC# 7430	22.7 (22.3-23.8)	8.19	7.9 (6.9-8.7)	284.8	103.0	132.0	0.01
2A GLC# 7431	22.8 (22.3-23.8)	8.28	7.9 (6.7-8.7)	287.3	110.0	130.0	0.00
6B GLC# 7432	22.8 (22.3-23.8)	8.16	7.9 (6.7-8.9)	286.0	105.0	126.0	0.00
4B GLC# 7433	22.8 (22.3-23.8)	8.19	7.9 (7.0-8.7)	284.3	102.0	130.0	0.00
6A GLC# 7434	22.8 (22.3-23.8)	8.19	8.0 (6.5-8.8)	285.0	104.0	130.0	0.00
5A GLC# 7435	22.8 (22.3-23.8)	8.19	7.9 (6.9-8.9)	287.3	104.0	128.0	0.00
4A GLC# 7436	22.8 (22.3-23.9)	8.16	8.0 (6.9-8.7)	284.8	104.0	126.0	0.00

Note: Ranges for temperature and dissolved oxygen are shown in parentheses.



TABLE 7. Summaries of Statistically Significant Differences ($p \leq 0.05$) Between the Investigative Sediment Samples and the Laboratory Control Sediment for the MDEQ Stamp Sands 10-day *Chironomus dilutus* Sediment Toxicity Tests Conducted October 14-24, 2008

Test Material	GLC #	Survival with Laboratory Control	Growth with Laboratory Control
3B	7430		X
2A	7431		X
6B	7432		X
4B	7433		X
6A	7434		X
5A	7435		X
4A	7436		X

X-Significantly different from control sediment ($\alpha \leq 0.05$)



TABLE 8.

Summaries of Statistically Significant Differences ($p \leq 0.05$) Between the Investigative Sediment Samples and the Laboratory Control Sediment for the MDEQ Stamp Sands 10-day *Hyaella azteca* Sediment Toxicity Tests Conducted October 17-27, 2008

Test Material	GLC #	Survival with Laboratory Control	Growth with Laboratory Control
3B	7430	X	X
2A	7431	X	X
6B	7432	X	X
4B	7433	X	X
6A	7434		X
5A	7435	X	X
4A	7436		X

X-Significantly different from control sediment ($\alpha \leq 0.05$)