



September 12, 2019

Mr. Les Arnold  
ALS Environmental  
3352 128<sup>th</sup> Avenue  
Holland, MI 49424

Reference: 0501867.0152

**Subject: Whole Effluent Toxicity Test Results**

Dear Les,

Enclosed please find the final results of the following Chronic Toxicity Tests performed on samples of the ArcelorMittal Burns Harbor Outfall 001 effluent.

- 19 August 2019, Chronic *Ceriodaphnia dubia* Toxicity Test
- 19 August 2019, Chronic *Pimephales promelas* Toxicity Test

If you have any questions concerning this report or if I can be of any further assistance to you, please feel free to contact me at (616) 738-7308 or via e-mail at [bruce.rabe@erm.com](mailto:bruce.rabe@erm.com).

Yours sincerely,

Bruce A. Rabe  
Director, Aquatic Toxicology Laboratory

BAR:km

Enclosure: Whole Effluent Toxicity Test Report

cc: Amanda Grzybowski  
Brandon Frye  
File

<b>Permittee/Location:</b> ArcelorMittal Burns Harbor LLC 250 West U.S. Hwy 12 Burns Harbor, IN 46304				<b>Permit number:</b> IN0000175		<b>Outfall number:</b> 001	
<b>Laboratory Name and Contact:</b> Environmental Resources Management 3352 128 <sup>th</sup> Avenue Holland, MI 49424				<b>Report <u>Due</u> Date:</b> N/A		<b>Report Date:</b> September 12, 2019	
<b>WETT Reporting Frequency or Type:</b>	Monthly	Quarterly	Semi-annual	Annual	TRE	Post TRE	<u>First</u> (per Reporting Frequency)?
	<u>Re-take</u> (per Reporting Frequency)?						

Test Organism	Test Type	Endpoint	Units	Result	Pass/Fail	Limit	Reporting
<i>Ceriodaphnia dubia</i>	7-day Survival and Reproduction	NOEC Survival	%	100		N/A	Laboratory Report
			TU <sub>c</sub>	1.0		1.0	
	Definitive Static-Renewal	NOEC Reproduction	%	100		N/A	
			TU <sub>c</sub>	1.0		1.0	
		IC <sub>25</sub> Reproduction	%	>100		N/A	
			TU <sub>c</sub>	1.0		1.0	
	48 hr. LC <sub>50</sub>		%	>100		N/A	
			TU <sub>a</sub>	1.0		1.0	
	<b>Toxicity (chronic)</b>		TU <sub>c</sub>	1.0	Pass	1.0	Laboratory Report and <b>NetDMR</b> (Parameter Code 61426)
	<b>Toxicity (acute)</b>		TU <sub>a</sub>	1.0	Pass	1.0	Laboratory Report and <b>NetDMR</b> (Parameter Code 61425)
<i>Pimephales promelas</i>	7-day Larval Survival and Growth	NOEC Survival	%	100		N/A	Laboratory Report
			TU <sub>c</sub>	1.0		1.0	
	Definitive Static-Renewal	NOEC Growth	%	100		N/A	
			TU <sub>c</sub>	1.0		1.0	
		IC <sub>25</sub> Growth	%	>100		N/A	
			TU <sub>c</sub>	1.0		1.0	
	96 hr. LC <sub>50</sub>		%	>100		N/A	
			TU <sub>a</sub>	1.0		1.0	
	<b>Toxicity (chronic)</b>		TU <sub>c</sub>	1.0	Pass	1.0	Laboratory Report and <b>NetDMR</b> (Parameter Code 61428)
	<b>Toxicity (acute)</b>		TU <sub>a</sub>	1.0	Pass	1.0	Laboratory Report and <b>NetDMR</b> (Parameter Code 61427)

# FINAL REPORT

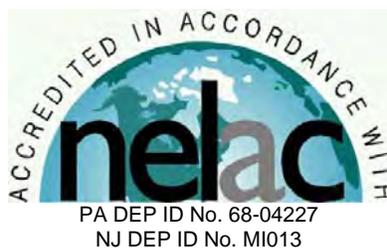
Chronic Toxicity Test  
Freshwater Invertebrate,  
*Ceriodaphnia dubia*  
EPA Test Method 1002.0

*Submitted To:*  
*ALS Environmental*  
*3352 128<sup>th</sup> Avenue*  
*Holland, MI 49424*

Sample: ArcelorMittal Burns Harbor, LLC - Outfall 001

Testing Period: 19 – 25 August 2019

Laboratory I.D. Number: 081919-2



Conducted By:  
**Environmental Resources Management, Inc.**  
3352 128th Avenue  
Holland, Michigan 49424



# Test Overview



Permittee: ArcelorMittal Burns Harbor, LLC  
Location: 250 West U.S. Hwy 12  
Burns Harbor, IN 46304  
Contact: Robert Maciel  
Telephone #: 219.787.2120  
NPDES Permit #: IN0000175  
Permit Requirements: Acute Toxicity Limit = 1.0 TUa  
Chronic Toxicity Limit = 1.0 TUc  
Test Sample: Outfall 001  
Receiving Water: East Branch, Little Calumet River  
Testing Date: 19 – 25 August 2019  
Sample Date(s): 19 August 2019  
21 August 2019  
22 August 2019  
Test/Method: Daphnid, *Ceriodaphnia dubia*,  
Survival and Reproduction  
Test EPA 821-R-02-013  
Method 1002.0.  
QC Objectives: Test data met all test  
acceptability criteria, except  
where noted below.  
Data Qualifiers: None

## DATA SUMMARY

Effluent Concentrations (%)	Survival (%)	Reproduction (Average Young/Female)
Control	100	38.2
6	100	42.2
13	100	43.8
25	100	44.1
50	100	43.3
100	100	43.2

## TEST RESULTS

48-Hour LC <sub>50</sub>	>100%
NOEC (Survival & Reproduction)	100%
LOEC (Survival & Reproduction)	>100%
IC <sub>25</sub>	>100%
MSDp (Reproduction)	12.6%
TUa (100/LC <sub>50</sub> )	1.0
TUc (100/IC <sub>25</sub> )	1.0

## TEST CONCLUSION

In accordance with the NPDES permit requirements for ArcelorMittal Burns Harbor, LLC, this toxicity test did not exceed either the acute or the chronic toxicity limit.



Bruce A. Rabe  
Director, Aquatic  
Toxicology Laboratory  
ERM Project No. 0501867.0152

Environmental Resources Management  
3352 128<sup>th</sup> Avenue  
Holland, Michigan 49424-9263  
Phone: 616.399.3500  
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## ERM Testing Method

### *Ceriodaphnia dubia* – Survival and Reproduction Toxicity Test



Upon sample receipt, each effluent sample was analyzed for a suite of water quality parameters (Appendix A - Table 1). Where indigenous organisms were present, the sample was filtered through a 60 micron ( $\mu\text{m}$ ) NITEX® screen. All samples were maintained at 0 – 6 degrees Celsius ( $^{\circ}\text{C}$ ) until needed for testing.

A series of five effluent concentrations and a control solution were established for testing. All test solutions were prepared by mixing appropriate volumes of dilution water and effluent in the test containers. Dilution water consisted of reconstituted moderately hard water. The control solution consisted of 100 percent dilution water.

*Ceriodaphnia dubia* used to initiate this test were obtained from individual, in-house cultures and were less than 24-hours old, and had an age range of 0 to 8 hours at test initiation. Test organisms used to initiate this test were released from adults which met acceptable performance criteria (i.e.,  $\geq 15$  young/surviving female within 3 broods and obtained from a brood of at least 8 young) and were maintained in reconstituted moderately hard water prior to test initiation.

The *Ceriodaphnia dubia* test was conducted using 30-milliliter (mL) disposable polystyrene containers containing 15 mL of control water or test solution. One *Ceriodaphnia dubia* was added to each test chamber with ten replicate chambers per treatment. Each *Ceriodaphnia dubia* test chamber was fed a 0.2-mL suspension consisting of yeast-Cerophyll-trout chow (YCT) and green algae (*Raphidocelis subcapitata*) mixture daily.

The test solutions were renewed daily during the exposure by transferring the adult daphnid, by way of a wide bore pipette, into fresh control water or test solution.

Percent survival of exposed *Ceriodaphnia dubia* was determined by inspecting for adult mortality daily. Mortality was defined as no body or appendage movement after gentle prodding. Production of young was also determined by daily inspections and enumeration. When 60 percent of the surviving females in the control treatment produced three broods, mean reproduction was determined by calculating the average number of live young produced per female for each treatment.

The test was conducted at a temperature of  $25 \pm 1^{\circ}\text{C}$  under fluorescent lighting with a photoperiod of 16 hours light and 8 hours dark. Water quality measurements were performed on all control and test solutions prior to test initiation and on selected treatments daily thereafter, as indicated in the raw data (Appendix A - Table 2).

Following termination of the chronic toxicity test, No Observed Effect Concentrations (NOEC) and Lowest Observed Effect Concentrations (LOEC) were determined for *Ceriodaphnia dubia* survival and reproduction, and a 25 percent Inhibition Concentration ( $\text{IC}_{25}$ ) was determined for *Ceriodaphnia dubia* reproduction. An NOEC is defined as the highest effluent concentration that does not produce any observed adverse effect to the exposed test organism. An LOEC is defined as the lowest effluent concentration that does produce an observed adverse effect to the exposed test organism. An adverse effect is determined as a statistically significant difference between the control and a given effluent concentration. Significant differences in *Ceriodaphnia dubia* survival were determined using the Fisher's Exact Test.

Prior to the determination of any significant differences in *Ceriodaphnia dubia* reproduction, the data were evaluated for normal distribution and homogeneity characteristics. Depending on the result and the number of test replicates per concentration, an analysis of variance test was performed followed by one of the following mean comparison tests: Dunnett's Procedure, Bonferroni t-Test, Steel's Many-One Rank Test, Wilcoxon Rank Sum Test, or the T-Test. For reporting purposes, a chronic toxic unit (TU<sub>c</sub>) is calculated and is defined as the most conservative of either 100/NOEC based on the more sensitive test endpoint or 100/IC<sub>25</sub>.

To evaluate acute toxicity, a 48-hour LC<sub>50</sub> and corresponding 95 percent confidence interval was also calculated, where possible. The LC<sub>50</sub> value estimate was determined by using one of the following statistical methods: graphical, Spearman-Kärber, Trimmed Spearman-Kärber, or Probit. The method selected for reporting test results was determined by the characteristics of the data; that is, the presence or absence of 0 and 100 percent mortality and the number of concentrations in which mortalities between 0 and 100 percent occurred. For reporting purposes, the 48-hour LC<sub>50</sub> value was converted to an acute toxic unit (TU<sub>a</sub>) by 100/LC<sub>50</sub>. All statistical analyses were performed using the CETIS™ Version 1.9.4.3 software program.

The reference toxicant, sodium chloride, was used to monitor the sensitivity of the test organisms and the precision of the testing procedure. Chronic reference toxicant tests are performed at least monthly and the resulting IC<sub>25</sub> are plotted to determine if the results are within prescribed limits (Appendix A - Standard Reference Toxicant Data). If the IC<sub>25</sub> of a particular reference toxicant test does not fall within the expected range of ± two standard deviations from the mean for a given test organism, the sensitivity of that organism and the overall credibility of the test system is suspect.

#### Reference:

USEPA. 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4<sup>th</sup> Ed. U.S. Environmental Protection Agency, Office of Water, Washington, D.C., EPA-821-R-02-013.

# Case Narrative



## **1.0 TEST PERFORMANCE CRITERIA**

The quality control results achieved laboratory specifications.

## **2.0 MODIFICATIONS TO ERM'S STANDARD TEST METHOD**

Test was performed in accordance with ERM's standard test method (see page 3).

*Appendix A*  
*Supporting Documents*

- *Raw Test Data*
- *Statistical Analysis (if necessary)*
- *Chain-of-Custody Forms*
- *Standard Reference Toxicant Data*

**Environmental Resources Management**

**Ceriodaphnia dubia - Chronic Toxicity Test  
Initial Water Quality and Test Solution Preparation**

Table 1  
Page 1 of 1

Permittee/Client:	ArcelorMittal Burns Harbor, LLC	Control/Dilution Water:	RMHW
Effluent/Location:	Outfall 001	Organism Batch #:	144-19
Lab I.D.#:	081919-2	Organism Age:	19 - 23.5 hrs
Beginning Date:	08/19/19	QC Review:	KM
Ending Date:	08/25/19	QC Review Date:	09/09/19

Time: ~~1500~~ 1700  
Time: 1530

Initial Water Quality:

Parameter	Units	Effluent			Synthetic Water		
		1	2	3	--	--	--
Sample #	--	1	2	3	--	--	--
Lab I.D.#/ Batch #	--	081919-2	082119-1	082319-3	96-19	99-19	-
Temperature	° C	5	3	2	--	--	--
Dissolved Oxygen	mg / L	12.3	11.0	10.7	--	--	--
pH	S.U.	7.9	7.1	7.7	7.6	7.6	-
Conductivity	umhos/cm	394	417	407	321	315	-
Alkalinity	mg / L CaCO <sub>3</sub>	110	108	104	60	60	-
Hardness	mg / L CaCO <sub>3</sub>	156	160	160	84	80	-
Total Ammonia	mg / L NH <sub>3</sub>	0.43	0.24	0.26	--	--	--
Total Residual Chlorine	mg / L Cl <sub>2</sub>	20.01	20.01	20.01	20.01	20.01	-
Total mls of 7.0 g/L Sodium Thiosulfate added per liter	mL / L	--	--	--	--	--	--
Initials	--	SRR	RH	SRR	SRR	SRR	-

Test Solution Preparation:

Test Solution Prepared For Both Species.

Treatment (% Effluent)	Effluent (mL)	Dilution (mL)	Test Day	Initials	Effluent Sample #	Synthetic Batch #
Control	0	1200	0	RWM	1	96-19
6%	72	1128	1	RWM	1	96-19
13%	156	1044	2	RH	2	96-19
25%	300	900	3	MB	2	96-19
50%	600	600	4	RW	3	96-19
100%	1200	0	5	KMRH	3	99-19
			6	KM	3	99-19
			7			

Permittee/Client: ArcelorMittal Burns Harbor, LLC  
Effluent/Location: Outfall 001  
Lab I.D.#: 081919-2

Water Quality Data:

Dissolved Oxygen (mg/L)															
Day															
Meter #	5	3	5	5	5	5	5	5	3	3	5	3	5		
Treatment (% Effluent)	0		1		2		3		4		5		6		7
	I	F	I	F	I	F	I	F	I	F	I	F	I	F	
Control	7.9	8.1	7.9	7.4	8.0	7.8	8.0	7.9	8.4	8.1	8.1	8.1	8.0		
6%	7.9	8.1	7.9	7.3	8.0	7.7	8.1	7.9	8.4	8.2	8.1	8.2	8.0		
13%	8.0	8.2	8.0	7.2	8.0	7.7	8.1	7.8	8.4	8.2	8.2	8.1	8.0		
25%	8.1	8.2	8.0	7.2	8.0	7.6	8.1	7.9	8.4	8.2	8.2	8.0	8.1		
50%	8.1	8.2	8.1	7.1	8.1	7.6	8.1	7.9	8.4	8.1	8.2	7.9	8.2		
100%	8.4	8.1	8.2	7.0	8.1	7.5	8.1	7.9	8.5	8.2	8.3	7.9	8.2		

pH (S.U.)															
Day															
Meter #	10	8	10	9	10	9	10	10	9	8	10	8	10		
Treatment (% Effluent)	0		1		2		3		4		5		6		7
	I	F	I	F	I	F	I	F	I	F	I	F	I	F	
Control	7.9	7.8	7.8	7.6	7.8	7.5	7.8	7.4	8.0	7.6	7.8	7.8	7.9		
6%	--	7.8	--	7.6	--	7.6	--	7.6	--	7.8	--	8.0	--		
13%	--	7.8	--	7.7	--	7.6	--	7.7	--	7.8	--	8.0	--		
25%	--	7.8	--	7.7	--	7.7	--	7.8	--	7.9	--	8.1	--		
50%	--	7.9	--	7.8	--	7.7	--	7.8	--	7.9	--	8.1	--		
100%	8.0	7.9	7.8	7.9	7.7	7.8	7.6	7.9	7.9	8.1	7.9	8.2	7.9		

Conductivity (umhos / cm)															
Day															
Meter #	4	--	4	--	4	--	4	--	4	--	4	--	4	--	
Treatment (% Effluent)	0		1		2		3		4		5		6		7
	I	F	I	F	I	F	I	F	I	F	I	F	I	F	
Control	323	--	322	--	323	--	326	--	323	--	315	--	313	--	
6%	326	--	327	--	326	--	332	--	328	--	320	--	322	--	
13%	332	--	332	--	332	--	338	--	331	--	326	--	329	--	
25%	340	--	341	--	342	--	349	--	343	--	335	--	336	--	
50%	363	--	362	--	366	--	372	--	364	--	357	--	359	--	
100%	405	--	404	--	414	--	421	--	409	--	402	--	404	--	

Temperature (°C)															
Day															
Meter #	5	3	5	5	5	5	5	5	3	3	5	3	5		
Treatment (% Effluent)	0		1		2		3		4		5		6		7
	I	F	I	F	I	F	I	F	I	F	I	F	I	F	
Control	24	24	24	24	24	25	24	24	24	24	24	24	24	24	
6%	24	24	24	24	24	25	24	24	24	24	24	24	24	24	
13%	24	24	24	24	24	25	24	24	24	24	24	24	24	24	
25%	24	24	24	24	24	25	24	24	24	24	24	24	24	24	
50%	24	24	24	24	25	25	24	24	24	24	24	24	24	24	
100%	24	24	24	24	25	25	24	24	24	25	24	24	24	24	

I = Initial Chemistry      F = Final Chemistry

Note: D.O. meter also used for temperature measurement unless otherwise noted.

Permittee/Client: ArcelorMittal Burns Harbor, LLC  
Effluent/Location: Outfall 001  
Lab I.D.#: 081919-2

Treatment (% Effluent)	Day No.	Replicate										Average Young/Female	Number of Live Adults (% Sur.)	Average Young/Female % CV	
		1	2	3	4	5	6	7	8	9	10				
Control	1	--	--	--	--	--	--	--	--	--	--	--	50/2 0/3/2/1/1/1/1 38.2	10	
	2	--	--	--	--	--	--	--	--	--	--	10			
	3	-	6	7	-	-	5	-	6	-	-	10			
	4	6	-	-	8	6	-	6	-	4	6	10			
	5	13	14	13	17	11	17	14	14	11	11	10			
	6	21	24	20	21	21	23	20	20	16	19	10			
	7						50/2, 0/3/2/1/1/1/1								
Totals:		40	44	40	46	38	44	45	40	22	31	36	38.2	(100)	18.9
# Broods (% 3rd Brood)		3	3	3	3	3	3	3	3	3	3	3	(100)		
6%	1	--	--	--	--	--	--	--	--	--	--		10		
	2	--	--	--	--	--	--	--	--	--	--		10		
	3	-	5	7	-	5	5	6	5	-	7		10		
	4	6	-	-	7	-	-	-	-	7	-		10		
	5	14	13	15	16	14	17	15	19	16	19		10		
	6	21	18	20	14	19	23	24	23	23	19		10		
	7														
Totals:		41	36	42	37	38	45	45	47	46	45	42.2	(100)	9.9	
13%	1	--	--	--	--	--	--	--	--	--	--		10		
	2	--	--	--	--	--	--	--	--	--	--		10		
	3	-	7	5	6	5	4	6	5	-	6		10		
	4	7	-	-	-	-	-	-	-	7	-		10		
	5	15	12	14	13	15	15	15	16	16	18		10		
	6	27	16	20	22	24	19	26	26	24	27		10		
	7														
Totals:		49	35	39	41	44	38	47	47	47	51	43.8	(100)	12.1	
25%	1	--	--	--	--	--	--	--	--	--	--		10		
	2	--	--	--	--	--	--	--	--	--	--		10		
	3	-	5	6	6	7	4	5	4	-	6		10		
	4	7	-	-	-	-	-	-	-	6	-		10		
	5	15	17	13	15	16	18	17	16	19	15		10		
	6	19	25	22	20	24	23	24	21	25	21		10		
	7														
Totals:		41	47	41	41	47	45	47	41	50	42	44.1	(100)	7.5	
50%	1	--	--	--	--	--	--	--	--	--	--		10		
	2	--	--	--	--	--	--	--	--	--	--		10		
	3	-	6	6	5	5	-	6	6	-	7		10		
	4	6	-	-	-	-	9	-	-	6	-		10		
	5	14	15	15	12	17	15	17	18	13	13		10		
	6	21	24	26	23	22	22	25	22	19	22		10		
	7														
Totals:		41	45	47	40	44	42	48	46	38	42	43.3	(100)	7.5	
100%	1	--	--	--	--	--	--	--	--	--	--		10		
	2	--	--	--	--	--	--	--	--	--	--		10		
	3	-	5	7	6	-	6	6	6	-	7		10		
	4	7	-	-	-	8	-	-	-	6	7		10		
	5	17	14	13	13	14	18	16	14	15	16		10		
	6	25	21	22	18	23	24	25	20	20	20		10		
	7														
Totals:		49	40	42	37	45	48	47	40	41	43	43.2	(100)	9.1	

X = DEAD ADULT      1X = DEAD ADULT, ONE YOUNG PRODUCED BEFORE DEATH      -- = NO YOUNG RECORDED  
(E) = ABORTED EMBRYOS /EGGS      (1) = ONE DEAD YOUNG      (S) = SPLIT BROOD      \* = 4th BROOD EXCLUDED FROM TOTAL



**CETIS Analytical Report**

Report Date: 28 Aug-19 12:31 (p 1 of 2)  
 Test Code/ID: 510AB0A7 / 13-5965-5079

Ceriodaphnia 7-d Survival and Reproduction Test						ERM
Analysis ID: 02-7071-9699	Endpoint: Reproduction	CETIS Version: CETISv1.9.4				
Analyzed: 28 Aug-19 12:31	Analysis: Parametric-Control vs Treatments	Status Level: 1				
Batch ID: 03-4677-8939	Test Type: Reproduction-Survival (7d)	Analyst: Lab Tech				
Start Date: 19 Aug-19 17:00	Protocol: EPA/821/R-02-013 (2002)	Diluent: Reconstituted Water				
Ending Date: 25 Aug-19 15:30	Species: Ceriodaphnia dubia	Brine:				
Test Length: 5d 22h	Taxon: Branchiopoda	Source: In-House Culture	Age: <24			
Sample ID: 17-2993-0268	Code: 671CA41C	Project: WET Testing				
Sample Date: 19 Aug-19 07:15	Material: Industrial Effluent	Source: ArcelorMittal Burns Harbor, LLC				
Receipt Date: 19 Aug-19 15:30	CAS (PC):	Station: Outfall 001				
Sample Age: 10h (5 °C)	Client: ArcelorMittal Burns Harbor, LLC					

Data Transform	Alt Hyp	NOEL	LOEL	TOEL	TU	PMSD
Untransformed	C > T	100	>100	n/a	1	12.63%

**Dunnett Multiple Comparison Test**

Control	vs	Conc-%	Test Stat	Critical	MSD	DF	P-Type	P-Value	Decision(α:5%)
Lab Water		6	-1.898	2.289	4.825	18	CDF	0.9993	Non-Significant Effect
		13	-2.657	2.289	4.825	18	CDF	1.0000	Non-Significant Effect
		25	-2.799	2.289	4.825	18	CDF	1.0000	Non-Significant Effect
		50	-2.42	2.289	4.825	18	CDF	0.9999	Non-Significant Effect
		100	-2.372	2.289	4.825	18	CDF	0.9999	Non-Significant Effect

**Test Acceptability Criteria**

Attribute	Test Stat	TAC Limits		Overlap	Decision
		Lower	Upper		
Control Resp	38.2	15	>>	Yes	Passes Criteria

**ANOVA Table**

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Between	239.533	47.9067	5	2.157	0.0725	Non-Significant Effect
Error	1199.4	22.2111	54			
Total	1438.93		59			

**Distributional Tests**

Attribute	Test	Test Stat	Critical	P-Value	Decision(α:1%)
Variances	Bartlett Equality of Variance Test	9.051	15.09	0.1071	Equal Variances
Distribution	Shapiro-Wilk W Normality Test	0.9554	0.9459	0.0282	Normal Distribution

**Reproduction Summary**

Conc-%	Code	Count	Mean	95% LCL	95% UCL	Median	Min	Max	Std Err	CV%	%Effect
0	L	10	38.2	33.03	43.37	40	22	46	2.284	18.91%	0.00%
6		10	42.2	39.32	45.08	43.5	36	47	1.272	9.53%	-10.47%
13		10	43.8	40.02	47.58	45.5	35	51	1.672	12.07%	-14.66%
25		10	44.1	41.73	46.47	43.5	41	50	1.048	7.52%	-15.45%
50		10	43.3	40.99	45.61	43	38	48	1.023	7.47%	-13.35%
100		10	43.2	40.38	46.02	42.5	37	49	1.245	9.12%	-13.09%

**Reproduction Detail**

Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10
0	L	40	44	40	46	38	45	40	22	31	36
6		41	36	42	37	38	45	45	47	46	45
13		49	35	39	41	44	38	47	47	47	51
25		41	47	41	41	47	45	46	41	50	42
50		41	45	47	40	44	42	48	46	38	42
100		49	40	42	37	45	48	47	40	41	43

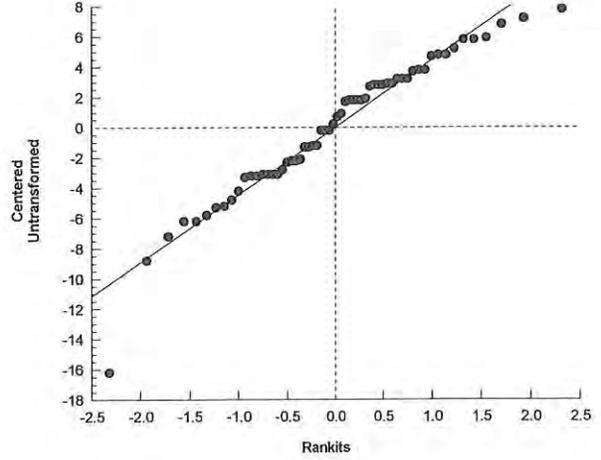
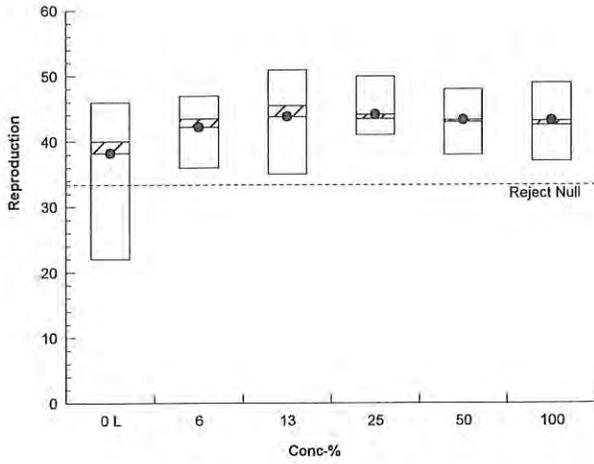
Ceriodaphnia 7-d Survival and Reproduction Test

ERM

Analysis ID: 02-7071-9699      Endpoint: Reproduction  
Analyzed: 28 Aug-19 12:31      Analysis: Parametric-Control vs Treatments

CETIS Version: CETISv1.9.4  
Status Level: 1

Graphics



**CETIS Analytical Report**

Report Date: 28 Aug-19 12:32 (p 1 of 2)  
 Test Code/ID: 510AB0A7 / 13-5965-5079

Ceriodaphnia 7-d Survival and Reproduction Test				ERM
Analysis ID: 10-7912-2024	Endpoint: Reproduction	CETIS Version: CETISv1.9.4		
Analyzed: 28 Aug-19 12:31	Analysis: Linear Interpolation (ICPIN)	Status Level: 1		
Batch ID: 03-4677-8939	Test Type: Reproduction-Survival (7d)	Analyst: Lab Tech		
Start Date: 19 Aug-19 17:00	Protocol: EPA/821/R-02-013 (2002)	Diluent: Reconstituted Water		
Ending Date: 25 Aug-19 15:30	Species: Ceriodaphnia dubia	Brine:		
Test Length: 5d 22h	Taxon: Branchiopoda	Source: In-House Culture	Age: <24	
Sample ID: 17-2993-0268	Code: 671CA41C	Project: WET Testing		
Sample Date: 19 Aug-19 07:15	Material: Industrial Effluent	Source: ArcelorMittal Burns Harbor, LLC		
Receipt Date: 19 Aug-19 15:30	CAS (PC):	Station: Outfall 001		
Sample Age: 10h (5 °C)	Client: ArcelorMittal Burns Harbor, LLC			

Linear Interpolation Options					
X Transform	Y Transform	Seed	Resamples	Exp 95% CL	Method
Log(X+1)	Linear	1089740	200	Yes	Two-Point Interpolation

Test Acceptability Criteria		TAC Limits			
Attribute	Test Stat	Lower	Upper	Overlap	Decision
Control Resp	38.2	15	>>	Yes	Passes Criteria

Point Estimates						
Level	%	95% LCL	95% UCL	TU	95% LCL	95% UCL
IC5	>100	n/a	n/a	<1	n/a	n/a
IC10	>100	n/a	n/a	<1	n/a	n/a
IC15	>100	n/a	n/a	<1	n/a	n/a
IC20	>100	n/a	n/a	<1	n/a	n/a
IC25	>100	n/a	n/a	<1	n/a	n/a
IC40	>100	n/a	n/a	<1	n/a	n/a
IC50	>100	n/a	n/a	<1	n/a	n/a

Reproduction Summary			Calculated Variate						Isotonic Variate	
Conc-%	Code	Count	Mean	Min	Max	Std Dev	CV%	%Effect	Mean	%Effect
0	L	10	38.2	22	46	7.223	18.91%	0.0%	42.47	0.0%
6		10	42.2	36	47	4.022	9.53%	-10.47%	42.47	0.0%
13		10	43.8	35	51	5.287	12.07%	-14.66%	42.47	0.0%
25		10	44.1	41	50	3.315	7.52%	-15.45%	42.47	0.0%
50		10	43.3	38	48	3.234	7.47%	-13.35%	42.47	0.0%
100		10	43.2	37	49	3.938	9.12%	-13.09%	42.47	0.0%

Reproduction Detail											
Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10
0	L	40	44	40	46	38	45	40	22	31	36
6		41	36	42	37	38	45	45	47	46	45
13		49	35	39	41	44	38	47	47	47	51
25		41	47	41	41	47	45	46	41	50	42
50		41	45	47	40	44	42	48	46	38	42
100		49	40	42	37	45	48	47	40	41	43

# CETIS Analytical Report

Report Date: 28 Aug-19 12:32 (p 2 of 2)  
Test Code/ID: 510AB0A7 / 13-5965-5079

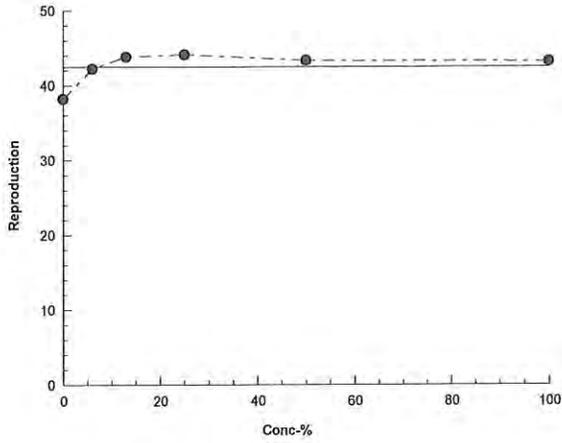
## Ceriodaphnia 7-d Survival and Reproduction Test

ERM

Analysis ID: 10-7912-2024      Endpoint: Reproduction  
Analyzed: 28 Aug-19 12:31      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.9.4  
Status Level: 1

### Graphics





**ERM**

**ENVIRONMENTAL RESOURCES MANAGEMENT**

3352 128<sup>th</sup> Avenue Holland, Michigan 49424-9263

Phone: 616-399-3500 FAX: 616-399-3777

**AQUATIC TOXICITY LAB CHAIN OF CUSTODY FORM \***

CLIENT NAME:		SAMPLER									
ADDRESS:		PHONE NUMBER:									
SAMPLE DESCRIPTION (i.e. Outfall 001)	DATE (Begin/End)	TIME (Begin/End)	GRAB OR COMP	NUMBER AND SIZE OF CONTAINERS	FIELD PARAMETERS	SAMPLE ID NUMBER (Filled in by ERM)	INITIAL WATER QUALITY PARAMETERS UPON RECEIPT BY LABORATORY (filled in by ERM)				
	02/19/19	0715	C	1 cub bag 2.5L	pH= NH <sub>3</sub> =	081919-2	Temp. (°C) <input checked="" type="checkbox"/> On Ice	D.O.	pH	Conduct	umhos/cm
011	02/19/19	0730	G	~	pH= NH <sub>3</sub> =	-3	Temp. (°C) <input checked="" type="checkbox"/> On Ice	D.O.	pH	Conduct	umhos/cm
					pH= NH <sub>3</sub> =		Temp. (°C) <input type="checkbox"/> On Ice	D.O.	pH	Conduct	umhos/cm
					pH= NH <sub>3</sub> =		Temp. (°C) <input type="checkbox"/> On Ice	D.O.	pH	Conduct	umhos/cm
					pH= NH <sub>3</sub> =		Temp. (°C) <input type="checkbox"/> On Ice	D.O.	pH	Conduct	umhos/cm
					pH= NH <sub>3</sub> =		Temp. (°C) <input type="checkbox"/> On Ice	D.O.	pH	Conduct	umhos/cm
ANALYSES REQUESTED [check item(s)]	Test Material:	Test Type:	Test Species:	Test Species: <input type="checkbox"/> <i>Ceriodaphnia dubia</i> <input type="checkbox"/> Rainbow Trout ( <i>Oncorhynchus mykiss</i> ) <input type="checkbox"/> <i>Daphnia magna</i> <input type="checkbox"/> Sheephead minnow ( <i>Cyprinodon variegatus</i> ) <input type="checkbox"/> <i>Daphnia pulex</i> <input type="checkbox"/> Silverside minnow ( <i>Menidia beryllina</i> ) <input type="checkbox"/> Fathead minnow ( <i>Pimephales promelas</i> ) <input type="checkbox"/> Other (write in comments section)							
COMMENT SECTION: See AAS VOC 41501 -51R											

**SAMPLE TRANSFERS**

RELINQUISHED BY: Signature / Organization	DATE	TIME	ACCEPTED BY: Signature / Organization	DATE	TIME
			<i>[Signature]</i>	02/19	1530

\* See Instructions for Sample Collection on Back of Sheet



Everett, WA  
+1 425 356 2600

Seattle, WA  
+1 206 735 5336

Holland, MI  
+1 616 399 6070

# Chain of Custody Form

Page      of       
COC ID: 41581

## Customer Information

Project Name: **Acceler MHAJ BH - WETT**

Project Number: **ALS**

Bill To Company: **ALS**

Invoice Attn: **ALS**

Address: **ALS**

City/State/Zip: **ALS**

Phone: **ALS**

Fax: **ALS**

e-Mail Address: **ALS**

## ALS Project Manager:

Project Information

ALS Work Order # **WETT**

Parameter/Method Requested for Analysis **(Acute + Chronic - Floor)**

Sample Description	Date	Time	Matrix	Pres.	# Bottles	A	B	C	D	E	F	G	H	I	J	Hold
Composite	8-19-19	0715	AB	NEAT	1-2gal.	X										
Grab	8-19-19	0830	AB	NEAT	1-25gal	X										

Signature: *[Signature]*

Turnaround Time in Business Days (BD):  10 BD  5 BD  3 BD  2 BD  1 BD

Results Due Date:     

Received by (Laboratory): *[Signature]*

Received by (Laboratory): *[Signature]*

Checked by (Laboratory): *[Signature]*

QC Package: (Check One Box Below)

Level II Std QC  TRRP Checklist

Level III Std QC/Raw Date  TRRP Level IV

Level IV SW/846/CLP  Other     

1. Any changes must be made in writing once samples and COC Form have been submitted to ALS Environmental.

2. Unless otherwise agreed in a formal contract, services provided by ALS Environmental are expressly limited to the terms and conditions stated on the reverse.

3. The Chain of Custody is a legal document. All information must be completed accurately.

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# Chain of Custody Form

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 Houston, TX +1 281 530 5656  
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 South Charleston, WV +1 304 356 3168  
 Everett, WA +1 425 356 2600  
 Holland, MI +1 616 399 6070  
 Middletown, PA +1 717 944 5541  
 Salt Lake City, UT +1 801 266 7700  
 York, PA +1 717 505 5280

Page      of       
 COC ID: **41865**

Customer Information				Project Information												ALS Work Order #: 9081459			
Project Name				Arcelor Mittal WFT												Parameter/Method Request for Analysis			
Project Number				Round 2 Volume - WFT (sub ERM)															
Bill To Company																			
Invoice Attn																			
Address																			
City/State/Zip																			
Phone																			
Fax																			
e-Mail Address																			
No.	Sample Description	Date	Time	Matrix	Pres.	# Bottles	A	B	C	D	E	F	G	H	I	J	Hold		
1	Outfall 001 - Comp	8-21-19	0625	AQ	8	1-2.5gal	X												
2	Outfall 011 - Comp	8-21-19	0605	AQ	8	2-2gal.	X												
3	001		011																
4	Temp		3																
5	pH		7.1																
6	COND		7.3																
7	417		426																
8																			
9																			
10																			

Turnaround Time in Business Days (BD):  10 BD  5 BD  3 BD  2 BD  1 BD  
 Results Due Date: \_\_\_\_\_

Notes: \_\_\_\_\_

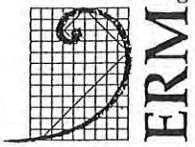
Shipper: B. Frye  
 Date: 8-21-19  
 Time: 1100

Received by: \_\_\_\_\_  
 Date: \_\_\_\_\_  
 Time: \_\_\_\_\_

Checked by (Laboratory): \_\_\_\_\_  
 Date: 8-21-19  
 Time: 1100

Preservative Key: 1-HCl 2-HNO<sub>3</sub> 3-H<sub>2</sub>SO<sub>4</sub> 4-NaOH 5-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 6-NaHSO<sub>4</sub> 7-Other 8-4°C 9-5035

QC Package: (Check One Box Below)  
 Level II Std QC  TRRP Checklist  
 Level III Std QC/Raw Date  TRRP Level IV  
 Level IV SW846/CLP  Other \_\_\_\_\_



# ENVIRONMENTAL RESOURCES MANAGEMENT

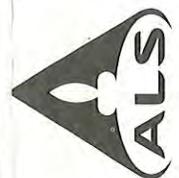
3352 128<sup>th</sup> Avenue Holland, Michigan 49424-9263  
 Phone: 616-399-3500 FAX: 616-399-3777

## AQUATIC TOXICITY LAB CHAIN OF CUSTODY FORM \*

CLIENT NAME:	Arcebor Mittal							SAMPLER							
ADDRESS:															
SAMPLE DESCRIPTION (i.e. Outfall 001)	DATE (Begin/End)	TIME (Begin/End)	GRAB OR COMP	NUMBER AND SIZE OF CONTAINERS	FIELD PARAMETERS	SAMPLE ID NUMBER (filled in by ERM)	INITIAL WATER QUALITY PARAMETERS UPON RECEIPT BY LABORATORY (filled in by ERM)								
	05/22/19	0620			pH= s.u. NH <sub>3</sub> = mg/L	082319-3	Temp. (°C) <input checked="" type="checkbox"/> On Ice	D.O. mg/L	pH	s.u.	Cond	umhos/cm			
011	05/22/19	0635			pH= s.u. NH <sub>3</sub> = mg/L	-4	Temp. (°C) <input checked="" type="checkbox"/> On Ice	D.O. mg/L	pH	s.u.	Cond	umhos/cm			
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH	s.u.	Cond	umhos/cm			
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH	s.u.	Cond	umhos/cm			
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH	s.u.	Cond	umhos/cm			
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH	s.u.	Cond	umhos/cm			
ANALYSES REQUESTED [check item(s)]	Test Material: Water/Wastewater Sediment Product			Test Type: Acute Chronic Other			Test Species: ___ <i>Ceriodaphnia dubia</i> ___ Rainbow Trout ( <i>Oncorhynchus mykiss</i> ) ___ <i>Daphnia magna</i> ___ Sheepshead minnow ( <i>Cyprinodon variegatus</i> ) ___ <i>Daphnia pulex</i> ___ Silverside minnow ( <i>Menidia beryllina</i> ) ___ Fathead minnow ( <i>Pimephales promelas</i> ) ___ <i>Americamysis bahia</i> ___ <i>Hyalella azteca</i> ___ <i>Chironomus dilutus</i> ___ Other (write in comments section)								
COMMENT SECTION: See AHS LOC 4157A -58A															

SAMPLE TRANSFERS					
RELINQUISHED BY: Signature/Organization	DATE	TIME	ACCEPTED BY: Signature/Organization	DATE	TIME
			Arcebor Mittal	05/22/19	1430

\* See Instructions for Sample Collection on Back of Sheet



# Chain of Custody Form

Cincinnati, OH +1 513 733 5336  
 Fort Collins, CO +1 970 490 1511  
 Houston, TX +1 281 530 5656  
 Spring City, PA +1 610 948 4903  
 South Charleston, WV +1 304 356 3168  
 Everett, WA +1 425 356 2600  
 Holland, MI +1 616 399 6070  
 Middletown, PA +1 717 944 5541  
 Salt Lake City, UT +1 801 266 7700  
 York, PA +1 717 505 5280

Page \_\_\_\_\_ of \_\_\_\_\_  
 COC ID: 41578

Customer Information		Project Information		ALS Work Order #: 19081459													
ALS Project Manager:		Parameter/Method Request for Analysis															
Purchase Order	Project Name	Date	Time	Matrix	Pres.	# Bottles	A	B	C	D	E	F	G	H	I	J	Hold
Work Order	Project Number	8-22-19	06 20	AQ	8	(2)-3gal.	X										
Company Name	Bill To Company	8-22-19	06 35	AQ	8	(2)-3gal.	X										
Send Report To	Invoice Attn																
Address	Address																
City/State/Zip	City/State/Zip																
Phone	Phone																
Fax	Fax																
e-Mail Address	e-Mail Address																

ALS Project Manager: \_\_\_\_\_  
 Project Name: AMBH WET #3  
 Project Number: \_\_\_\_\_  
 Bill To Company: Arcelor Mittal  
 Invoice Attn: \_\_\_\_\_  
 Address: \_\_\_\_\_  
 City/State/Zip: \_\_\_\_\_  
 Phone: \_\_\_\_\_  
 Fax: \_\_\_\_\_  
 e-Mail Address: \_\_\_\_\_

Shipment Method		Turnaround Time in Business Days (BD)		Results Due Date:					
Sampler(s) Please Print & Sign		<input type="checkbox"/> 10 BD <input type="checkbox"/> 5 BD <input type="checkbox"/> 3 BD <input type="checkbox"/> 1 BD <input type="checkbox"/> 2 BD <input type="checkbox"/> 1 BD		<input type="checkbox"/> Other _____					
Relinquished by:	Date: 8-23-19	Time:	Received by:	Signature: [Signature]	Notes:				
Relinquished by:	Date:	Time:	Received by (Laboratory):	Signature: [Signature]	Notes: 0821082319 1430				
Logged by (Laboratory):	Date:	Time:	Checked by (Laboratory):	Signature:	Notes:				
Preservative Key:	1-HCl	2-HNO <sub>3</sub>	3-H <sub>2</sub> SO <sub>4</sub>	4-NaOH	5-Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	6-NaHSO <sub>4</sub>	7-Other	8-4°C	9-5035

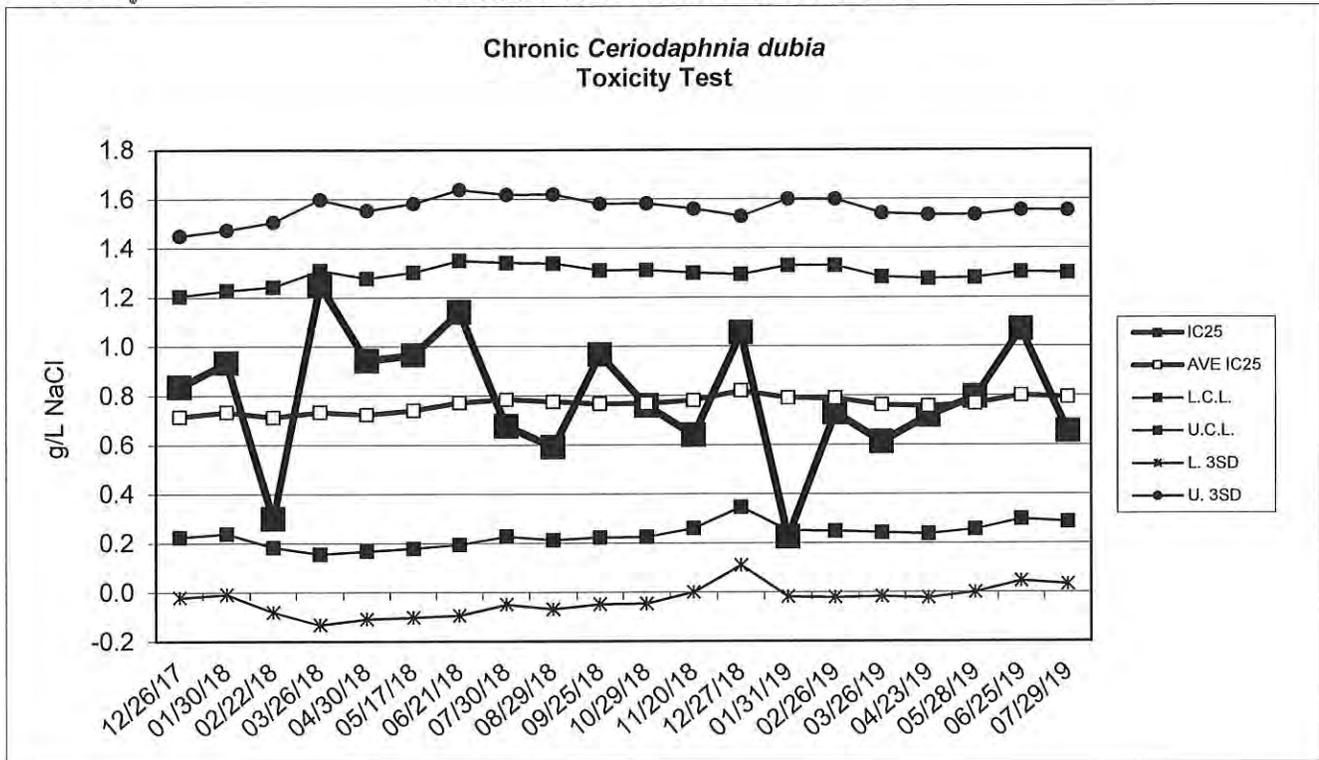
QC Package: (Check One Box Below)  
 Level II Std QC     TRRP Checklist  
 Level III Std QC/Raw Date     TRRP Level IV  
 Level IV SW846/CLP     Other \_\_\_\_\_

Note: 1. Any changes must be made in writing once samples and COC Form have been submitted to ALS Environmental.  
 2. Unless otherwise agreed in a formal contract, services provided by ALS Environmental are expressly limited to the terms and conditions stated on the reverse.  
 3. The Chain of Custody is a legal document. All information must be completed accurately.



# Environmental Resources Management

## Standard Reference Toxicant Data



**Chronic *Ceriodaphnia dubia* Toxicity Test Data**

Date	IC25 (g/L NaCl)	AVE IC25 (g/L NaCl)	CONTROL LIMIT		Survival (%)	CONTROL Reproduction (ave. young)	CV (%)
			Lower	Upper			
12/26/17	0.83	0.7	0.22	1.20	100	21.9	11.9
01/30/18	0.93	0.7	0.24	1.23	100	25.5	36.3
02/22/18	0.30	0.7	0.18	1.24	100	17.8	35.0
03/26/18	1.25	0.7	0.16	1.31	90	32.5	38.5
04/30/18	0.94	0.7	0.17	1.28	100	32.0	25.5
05/17/18	0.97	0.7	0.18	1.30	100	30.0	38.6
06/21/18	1.14	0.8	0.19	1.35	80	35.2	8.2
07/30/18	0.68	0.8	0.23	1.34	100	25.5	16.3
08/29/18	0.59	0.8	0.21	1.34	100	30.1	26.2
09/25/18	0.97	0.8	0.22	1.31	100	27.6	26.7
10/29/18	0.76	0.8	0.22	1.31	100	32.7	24.8
11/20/18	0.64	0.8	0.26	1.30	100	34.8	15.2
12/27/18	1.06	0.8	0.35	1.29	100	26.8	43.7
01/31/19	0.23	0.8	0.25	1.33	100	34.7	14.9
02/26/19	0.73	0.8	0.25	1.33	100	27.9	9.3
03/26/19	0.61	0.8	0.24	1.28	100	40.2	9.9
04/23/19	0.72	0.8	0.24	1.28	100	36.1	25.4
05/28/19	0.79	0.8	0.26	1.28	100	37.6	3.1
06/25/19	1.07	0.8	0.30	1.30	100	29.4	26.7
07/29/19	0.65	0.8	0.29	1.30	100	33.7	14.6

# FINAL REPORT

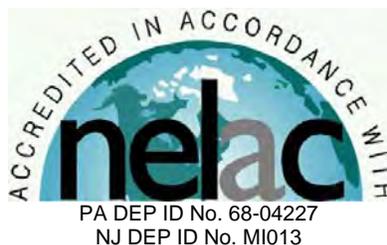
Chronic Toxicity Test  
Freshwater Vertebrate,  
*Pimephales promelas*  
EPA Test Method 1000.0

*Submitted To:*  
*ALS Environmental*  
*3352 128<sup>th</sup> Avenue*  
*Holland, MI 49424*

Sample: ArcelorMittal Burns Harbor, LLC - Outfall 001

Testing Period: 19 – 26 August 2019

Laboratory I.D. Number: 081919-2



Conducted By:  
**Environmental Resources Management, Inc.**  
3352 128th Avenue  
Holland, Michigan 49424



# Test Overview



Permittee: ArcelorMittal Burns Harbor, LLC  
Location: 250 West U.S. Hwy 12  
Burns Harbor, IN 46304  
Contact: Robert Maciel  
Telephone #: 219.787.2120

NPDES Permit #: IN0000175  
Permit Requirements: Acute Toxicity Limit = 1.0 TUa  
Chronic Toxicity Limit = 1.0 TUC  
Test Sample: Outfall 001  
Receiving Water: East Branch, Little Calumet River

Testing Date: 19 – 26 August 2019

Sample Date(s): 19 August 2019  
21 August 2019  
22 August 2019

Test/Method: Fathead Minnow, *Pimephales promelas*, Survival and Growth  
Test EPA 821-R-02-013  
Method 1000.0.

QC Objectives: Test data met all test acceptability criteria, except where noted below.

Data Qualifiers: None

## DATA SUMMARY

Effluent Concentrations (%)	Survival (%)	Growth Average Wt./ Organism (mg)
Control	100	0.521
6	100	0.504
13	95	0.506
25	97.5	0.508
50	95	0.493
100	95	0.522

## TEST RESULTS

96-Hour LC <sub>50</sub>	>100%
NOEC (Survival)	100%
LOEC (Survival)	>100%
IC <sub>25</sub>	>100%
MSDp (Survival)	16.7%
TUa (100/LC <sub>50</sub> )	1.0
TUC (100/ NOEC or IC <sub>25</sub> )	1.0

## TEST CONCLUSION

In accordance with the NPDES permit requirements for ArcelorMittal Burns Harbor, LLC, this toxicity test did not exceed either the acute or the chronic toxicity limit.

Bruce A. Rabe  
Director, Aquatic  
Toxicology Laboratory  
ERM Project No. 0501867.0152

Environmental Resources Management  
3352 128<sup>th</sup> Avenue  
Holland, Michigan 49424-9263  
Phone: 616.399.3500  
Fax: 616.399.3777



# ERM Testing Method

## *Pimephales promelas* – Survival and Growth Toxicity Test



Upon sample receipt, each effluent sample was analyzed for a suite of water quality parameters (Appendix A - Table 1). Where indigenous organisms were present, the sample was filtered through a 60 micron ( $\mu\text{m}$ ) NITEX® screen. All samples were maintained at 0 – 6 degrees Celsius ( $^{\circ}\text{C}$ ) until needed for testing.

A series of five effluent concentrations and a control solution were established for testing. All test solutions were prepared by mixing appropriate volumes of dilution water and effluent in the test containers. Dilution water consisted of reconstituted moderately hard water. The control solution consisted of 100 percent dilution water.

*Pimephales promelas* used to initiate this test were obtained from in-house cultures and were less than 24-hours old at test initiation. Test organisms were maintained in reconstituted moderately hard water prior to test initiation.

The *Pimephales promelas* test was conducted using 300 to 500-milliliter (mL) disposable polypropylene containers containing 250 mL of control water or test solution. Ten fish were randomly added to each test chamber with four replicate chambers per treatment. Each *Pimephales promelas* test chamber was fed 0.2 mL of a concentrated suspension of less than 24-hour old live brine shrimp nauplii (*Artemia* sp.) two times per day. Test solutions were renewed daily during the exposure by replacing approximately 90 percent of the 24-hour old solution with fresh control water or appropriate test solution. Prior to test solution renewal, uneaten and dead brine shrimp, along with other debris, were removed from the bottom of the test chambers.

Percent survival of exposed *Pimephales promelas* was determined daily by enumeration of live organisms. Mortality was defined as no body movement after gentle prodding. At the termination of the chronic test, larvae in each test chamber were counted, dried, and weighed to the nearest 0.01 milligram (mg) on an analytical balance.

The test was conducted at a temperature of  $25 \pm 1^{\circ}\text{C}$  under fluorescent lighting with a photoperiod of 16 hours light and 8 hours dark. Water quality measurements were performed on all control and test solutions prior to test initiation and on selected treatments daily thereafter, as indicated in the raw data (Appendix A - Table 2).

Following termination of the chronic toxicity test, No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC) were determined for both *Pimephales promelas* survival and growth and a 25 percent Inhibition Concentration ( $\text{IC}_{25}$ ) was determined for *Pimephales promelas* growth. The NOEC is defined as the highest effluent concentration which does not produce any observed adverse effect to the exposed test organism whereas the LOEC is defined as the lowest effluent concentration which does produce an observed adverse effect to the exposed test organism. An adverse effect is determined as a statistically significant difference between the control and a given effluent concentration.

Prior to the determination of any significant differences in *Pimephales promelas* survival and growth, the data were evaluated for normal distribution and homogeneity characteristics. Depending on the result and the number of test replicates per concentration, an analysis of variance test was performed, followed by one of the following mean comparison tests: Dunnett's Procedure, Bonferroni t-Test, Steel's Many-One Rank Test, Wilcoxon Rank Sum Test, or the T-Test.

For reporting purposes, a chronic toxic unit (TU<sub>c</sub>) is calculated and is defined as the most conservative of either 100/NOEC based on the most sensitive test endpoint or 100/ $\text{IC}_{25}$ .

To evaluate acute toxicity, a 96-hour LC<sub>50</sub> and corresponding 95 percent confidence interval were also calculated, where possible. The LC<sub>50</sub> value estimate was determined by using one of the following statistical methods: graphical, Spearman-Kärber, Trimmed Spearman-Kärber, or Probit. The method selected for reporting test results was determined by the characteristics of the data; that is, the presence or absence of 0 and 100 percent mortality and the number of concentrations in which mortalities between 0 and 100 percent occurred. For reporting purposes, the 96-hour LC<sub>50</sub> value was converted to an acute toxic unit (TUa) by 100/LC<sub>50</sub>. All statistical analyses were performed using the CETIS™ Version 1.9.4.3 software program.

The reference toxicant, sodium chloride, was used to monitor the sensitivity of the test organisms. Chronic reference toxicant tests are performed at least monthly and the resulting Inhibition Concentrations (IC<sub>25</sub>) are plotted to determine if the results are within prescribed limits (Appendix A - Standard Reference Toxicant Data). If the IC<sub>25</sub> of a particular reference toxicant test does not fall within the expected range of ± two standard deviations from the mean for a given test organism, the sensitivity of that organism and the overall credibility of the test system is suspect.

Reference:

USEPA. 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 4<sup>th</sup> Ed. U.S. Environmental Protection Agency, Office of Water, Washington, D.C., EPA-821-R-02-013.

## Case Narrative



### **1.0 TEST PERFORMANCE CRITERIA**

The quality control results achieved laboratory specifications.

### **2.0 MODIFICATIONS TO ERM'S STANDARD TEST METHOD**

Test was performed in accordance with ERM's standard test method (see page 3).

*Appendix A*  
*Supporting Documents*

- *Raw Test Data*
- *Statistical Analysis (if necessary)*
- *Chain-of-Custody Forms*
- *Standard Reference Toxicant Data*

**Environmental Resources Management**

**Pimephales promelas - Chronic Toxicity Test  
Initial Water Quality and Test Solution Preparation**

Permittee/Client: ArcelorMittal Burns Harbor, LLC  
 Effluent/Location: Outfall 001  
 Lab I.D.#: 081919-2  
 Beginning Date: 08/19/19 Time: 1730  
 Ending Date: 08/26/19 Time: 1630

Control/Dilution Water: RMHW  
 Organism Batch #: 144-19  
 Organism Age: 224 hours old  
 QC Review: KM  
 QC Review Date: 09/09/19

Initial Water Quality:

Parameter	Units	Effluent			Synthetic Water		
Sample #	--	1	2	3	--	--	--
Lab I.D.#/ Batch #	--	081919-2	082119-1	082319-3	96-19	99-19	-
Temperature	° C	5	3	2	--	--	--
Dissolved Oxygen	mg / L	12.3	11.0	10.7	--	--	--
pH	S.U.	7.9	7.1	7.7	7.6	7.6	-
Conductivity	umhos/cm	394	417	407	321	315	-
Alkalinity	mg / L CaCO <sub>3</sub>	110	104	104	60	60	-
Hardness	mg / L CaCO <sub>3</sub>	156	160	160	84	80	-
Total Ammonia	mg / L NH <sub>3</sub>	0.43	0.28	0.26	--	--	--
Total Residual Chlorine	mg / L Cl <sub>2</sub>	20.01	20.01	20.01	20.01	20.01	-
Total mls of 7.0 g/L Sodium Thiosulfate added per liter	mL / L	--	--	--	--	--	--
Initials	--	SPR	RH	SPR	SPR	SPR	-

Test Solution Preparation:

Test Solution Prepared For Both Species.

Treatment (% Effluent)	Effluent (mL)	Dilution (mL)	Test Day	Initials	Effluent Sample #	Synthetic Batch #
Control	0	1200	0	RWM	1	96-19
6%	72	1128	1	RWM	1	96-19
13%	156	1044	2	RH	2	96-19
25%	300	900	3	MB	2	96-19
50%	600	600	4	RH	3	96-19
100%	1200	0	5	RH	3	99-19
			6	KM	3	99-19
			7	SPR	--	--

Environmental Resources Management

Pimephales promelas - Chronic Toxicity Test  
Water Quality Data

Permittee/Client: ArcelorMittal Burns Harbor, LLC  
Effluent/Location: Outfall 001  
Lab I.D.#: 081919-2

Water Quality Data:

Dissolved Oxygen (mg/L)														
Day														
Meter #	5		3		5		5		5		3		3	
Treatment (% Effluent)	0		1		2		3		4		5		6	
	I	F	I	F	I	F	I	F	I	F	I	F	I	F
Control	7.9	7.3	7.9	6.7	8.0	7.6	8.0	6.7	8.4	6.9	8.1	6.6	8.0	7.1
6%	7.9	7.2	7.9	6.2	8.0	6.8	8.1	6.5	8.4	7.0	8.1	6.2	8.0	7.2
13%	8.0	7.2	8.0	6.3	8.0	6.6	8.1	6.4	8.4	7.0	8.2	6.3	8.0	7.4
25%	8.1	7.3	8.0	6.3	8.0	6.6	8.1	6.4	8.4	6.6	8.2	6.2	8.1	7.0
50%	8.1	7.0	8.1	6.0	8.1	6.5	8.1	6.4	8.4	6.3	8.2	5.7	8.2	7.0
100%	7.4	7.1	8.2	5.7	8.1	6.5	8.1	6.1	8.5	6.1	8.3	6.1	8.2	7.1

pH (S.U.)														
Day														
Meter #	10		8		10		10		9		10		9	
Treatment (% Effluent)	0		1		2		3		4		5		6	
	I	F	I	F	I	F	I	F	I	F	I	F	I	F
Control	7.4	7.5	7.8	7.5	7.8	7.5	7.8	7.4	8.0	7.4	7.8	7.4	7.9	7.4
6%	--	7.6	--	7.5	--	7.5	--	7.4	--	7.5	--	7.3	--	7.4
13%	--	7.7	--	7.6	--	7.4	--	7.4	--	7.5	--	7.4	--	7.4
25%	--	7.8	--	7.7	--	7.5	--	7.5	--	7.6	--	7.4	--	7.5
50%	--	7.9	--	7.7	--	7.6	--	7.7	--	7.7	--	7.4	--	7.5
100%	8.0	8.0	7.8	7.8	7.7	7.7	7.6	7.8	7.9	7.8	7.9	7.6	7.9	7.6

Conductivity (umhos / cm)														
Day														
Meter #	4		--		4		--		4		--		4	
Treatment (% Effluent)	0		1		2		3		4		5		6	
	I	F	I	F	I	F	I	F	I	F	I	F	I	F
Control	323	--	322	--	323	--	326	--	323	--	315	--	313	--
6%	326	--	327	--	326	--	332	--	328	--	320	--	322	--
13%	332	--	332	--	332	--	338	--	334	--	326	--	329	--
25%	340	--	341	--	342	--	349	--	343	--	335	--	336	--
50%	363	--	362	--	366	--	372	--	364	--	357	--	359	--
100%	405	--	404	--	414	--	421	--	409	--	402	--	404	--

Temperature (°C)														
Day														
Meter #	5		3		5		5		5		3		3	
Treatment (% Effluent)	0		1		2		3		4		5		6	
	I	F	I	F	I	F	I	F	I	F	I	F	I	F
Control	24	25	24	25	24	25	24	25	24	25	24	24	24	25
6%	24	25	24	25	24	25	24	25	24	25	24	24	24	25
13%	24	25	24	25	24	25	24	25	24	25	24	24	24	25
25%	24	25	24	25	24	25	24	25	24	25	24	24	24	25
50%	24	25	24	25	24	25	24	25	24	25	24	24	24	25
100%	24	25	24	25	24	25	24	25	24	25	24	24	24	25

I = Initial Chemistry F = Final Chemistry

Note: D.O. meter also used for temperature measurement unless otherwise noted.

Permittee/Client: ArcelorMittal Burns Harbor, LLC  
 Effluent/Location: Outfall 001  
 Lab I.D.#: 081919-2

Survival Data:

Treatment (% Effluent)	Rep.	# Live Organisms Day							Rep.	# Live Organisms Day <sup>KM 08/24</sup>							96 Hour Survival Summary				
		0	1	2	3	4	5	6		7	0	1	2	3	4	5	6	7	Initial	Final	% Survival
		Control	A	10	10	10	10	10		10	10	10	B	10	10	10	10	10	10	10	10
6%	A	10	10	10	10	10	10	10	10	B	10	10	10	10	10	10	10	10	40	40	100
13%	A	10	10	10	10	9	9	9	9	B	10	10	10	10	10	10	10	10	40	38	95
25%	A	10	10	10	10	10	10	10	10	B	10	10	10	10	10	10	10	10	40	39	97.5
50%	A	10	10	10	10	9	9	9	9	B	10	10	10	10	10	9	9	9	40	39	97.5
100%	A	10	10	10	10	9	9	9	9	B	10	10	10	9	9	9	9	9	40	39	97.5

Treatment (% Effluent)	Rep.	# Live Organisms Day							Rep.	# Live Organisms Day							7 Day Survival Summary				
		0	1	2	3	4	5	6		7	0	1	2	3	4	5	6	7	Initial	Final	% Survival
		Control	C	10	10	10	10	10		10	10	10	D	10	10	10	10	10	10	10	10
6%	C	10	10	10	10	10	10	10	10	D	10	10	10	10	10	10	10	10	40	40	100
13%	C	10	10	10	10	10	10	10	10	D	10	10	10	10	9	9	9	9	40	36	95
25%	C	10	10	10	10	10	10	10	10	D	10	10	10	10	9	9	9	9	40	39	97.5
50%	C	10	10	10	10	9	9	9	9	D	10	10	10	10	10	10	10	10	40	36	95
100%	C	10	10	10	10	10	10	10	10	D	10	10	10	10	10	10	10	10	40	36	95

Test Information:

	Day 0	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
Time:	1730	1500	1600	1600	1600	1600	1530	1630
Initials:	SPR	KM	PR	KM	KM	KM	PR	SPR
Date:	08/19/19	08/20/19	08/21/19	08/22/19	08/23/19	08/24/19	08/25/19	08/26/19

Feeding:

	Day 0	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7
Batch #:	230-19	231-19	232-19	233-19	234-19	235-19	236-19	--
Initials AM:	--	PR	PR	PR	PR	KM	KM	--
Initials PM:	PR	PR	PR	PR	PR	KM	PR	--

Oven:

Date In	Time In	Initials	Date Out	Time Out	Initials
08/26/19	1630	SPR	08/27/19	1630	KM

Comment Section:

Day	Date	Initials	Comments

Permittee/Client: ArcelorMittal Burns Harbor, LLC  
Effluent/Location: Outfall 001  
Lab I.D.#: 081919-2

Pan #	Conc. (% Effluent)	Replicate	Final Weight (mg)	Initial Weight (mg)	Larvae Weight (mg)	# of Initial Organisms	Avg. Wt./ Organism/ Replicate (mg)	Avg. Wt./ Organism/ Treatment (mg)	Avg. Wt./ Organism/ Treatment % CV
			8/27/2019	8/26/2019					
Date		km							
Analyst		km							
1	Control	A	25.69	20.83	4.86	10	0.486		
2	Control	B	26.73	21.85	4.88	10	0.488		
3	Control	C	24.13	18.81	5.32	10	0.532		
4	Control	D	27.14	21.35	5.79	10	0.579	0.521	8.4
5	6%	A	22.13	16.43	5.70	10	0.570		
6	6%	B	22.39	17.13	5.26	10	0.526		
7	6%	C	28.75	24.30	4.45	10	0.445		
8	6%	D	24.07	19.33	4.74	10	0.474	0.504	11.0
9	13%	A	22.80	18.03	4.77	10	0.477		
10	13%	B	21.99	16.97	5.02	10	0.502		
11	13%	C	23.25	18.11	5.14	10	0.514		
12	13%	D	26.45	21.15	5.30	10	0.530	0.506	4.4
13	25%	A	28.84	23.23	5.61	10	0.561		
14	25%	B	26.87	21.52	5.35	10	0.535		
15	25%	C	23.32	18.47	4.85	10	0.485		
16	25%	D	23.01	18.51	4.50	10	0.450	0.508	9.8
17	50%	A	26.21	21.18	5.03	10	0.503		
18	50%	B	29.80	24.72	5.08	10	0.508		
19	50%	C	26.31	22.11	4.20	10	0.420		
20	50%	D	30.75	25.34	5.41	10	0.541	0.493	10.4
21	100%	A	30.27	24.52	5.75	10	0.575		
22	100%	B	26.31	21.99	4.32	10	0.432		
23	100%	C	33.14	27.33	5.81	10	0.581		
24	100%	D	36.36	31.38	4.98	10	0.498	0.522	13.5

Quality Assurance			Final Wt. (mg)		
25	Blank	A	15.84	15.82	0.02
26	Blank	B	22.13	22.1	0.03

\* Biomass data were transferred directly to the spreadsheet using the data transfer function of the analytical balance.

# CETIS Analytical Report

Report Date: 28 Aug-19 12:31 (p 1 of 2)  
 Test Code/ID: 719A8A12 / 19-0595-3298

## Fathead Minnow 7-d Larval Survival and Growth Test

ERM

Analysis ID: 01-2616-6693	Endpoint: 7d Survival Rate	CETIS Version: CETISv1.9.4
Analyzed: 28 Aug-19 12:31	Analysis: Nonparametric-Control vs Treatments	Status Level: 1
Batch ID: 17-4757-0902	Test Type: Growth-Survival (7d)	Analyst: Lab Tech
Start Date: 19 Aug-19 17:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Reconstituted Water
Ending Date: 26 Aug-19 16:30	Species: Pimephales promelas	Brine:
Test Length: 6d 23h	Taxon: Actinopterygii	Source: In-House Culture Age: <24
Sample ID: 17-9487-7362	Code: 6AFBA7B2	Project: WET Testing
Sample Date: 19 Aug-19 07:15	Material: Industrial Effluent	Source: ArcelorMittal Burns Harbor, LLC
Receipt Date: 19 Aug-19 15:30	CAS (PC):	Station: Outfall 001
Sample Age: 10h (5 °C)	Client: ArcelorMittal Burns Harbor, LLC	

Data Transform	Alt Hyp	NOEL	LOEL	TOEL	TU	PMSD
Angular (Corrected)	C > T	100	>100	n/a	1	7.93%

### Steel Many-One Rank Sum Test

Control	vs	Conc-%	Test Stat	Critical	Ties	DF	P-Type	P-Value	Decision(α:5%)
Lab Water		6	18	10	1	6	Asymp	0.8333	Non-Significant Effect
		13	14	10	1	6	Asymp	0.3451	Non-Significant Effect
		25	16	10	1	6	Asymp	0.6105	Non-Significant Effect
		50	14	10	1	6	Asymp	0.3451	Non-Significant Effect
		100	14	10	1	6	Asymp	0.3451	Non-Significant Effect

### Test Acceptability Criteria

Attribute	Test Stat	TAC Limits		Overlap	Decision
		Lower	Upper		
Control Resp	1	0.8	>>	Yes	Passes Criteria

### ANOVA Table

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Between	0.0320925	0.0064185	5	1.16	0.3663	Non-Significant Effect
Error	0.0995975	0.0055332	18			
Total	0.13169		23			

### Distributional Tests

Attribute	Test	Test Stat	Critical	P-Value	Decision(α:1%)
Variances	Levene Equality of Variance Test	23.4	4.248	2.7E-07	Unequal Variances
Variances	Mod Levene Equality of Variance Test	5.8	4.248	0.0023	Unequal Variances
Distribution	Shapiro-Wilk W Normality Test	0.8745	0.884	0.0064	Non-Normal Distribution

### 7d Survival Rate Summary

Conc-%	Code	Count	Mean	95% LCL	95% UCL	Median	Min	Max	Std Err	CV%	%Effect
0	L	4	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	0.0000	0.00%	0.00%
6		4	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	0.0000	0.00%	0.00%
13		4	0.9500	0.8581	1.0000	0.9500	0.9000	1.0000	0.0289	6.08%	5.00%
25		4	0.9750	0.8954	1.0000	1.0000	0.9000	1.0000	0.0250	5.13%	2.50%
50		4	0.9500	0.8581	1.0000	0.9500	0.9000	1.0000	0.0289	6.08%	5.00%
100		4	0.9500	0.8581	1.0000	0.9500	0.9000	1.0000	0.0289	6.08%	5.00%

### Angular (Corrected) Transformed Summary

Conc-%	Code	Count	Mean	95% LCL	95% UCL	Median	Min	Max	Std Err	CV%	%Effect
0	L	4	1.412	1.412	1.412	1.412	1.412	1.412	0	0.00%	0.00%
6		4	1.412	1.412	1.412	1.412	1.412	1.412	0	0.00%	0.00%
13		4	1.331	1.181	1.48	1.331	1.249	1.412	0.04705	7.07%	5.77%
25		4	1.371	1.242	1.501	1.412	1.249	1.412	0.04074	5.94%	2.89%
50		4	1.331	1.181	1.48	1.331	1.249	1.412	0.04705	7.07%	5.77%
100		4	1.331	1.181	1.48	1.331	1.249	1.412	0.04705	7.07%	5.77%

# CETIS Analytical Report

Report Date: 28 Aug-19 12:31 (p 2 of 2)  
 Test Code/ID: 719A8A12 / 19-0595-3298

## Fathead Minnow 7-d Larval Survival and Growth Test

ERM

Analysis ID: 01-2616-6693      Endpoint: 7d Survival Rate      CETIS Version: CETISv1.9.4  
 Analyzed: 28 Aug-19 12:31      Analysis: Nonparametric-Control vs Treatments      Status Level: 1

### 7d Survival Rate Detail

Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4
0	L	1.0000	1.0000	1.0000	1.0000
6		1.0000	1.0000	1.0000	1.0000
13		0.9000	1.0000	1.0000	0.9000
25		1.0000	1.0000	1.0000	0.9000
50		1.0000	0.9000	0.9000	1.0000
100		0.9000	0.9000	1.0000	1.0000

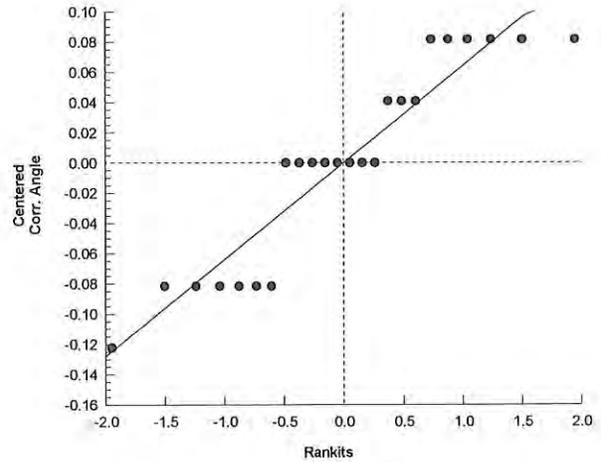
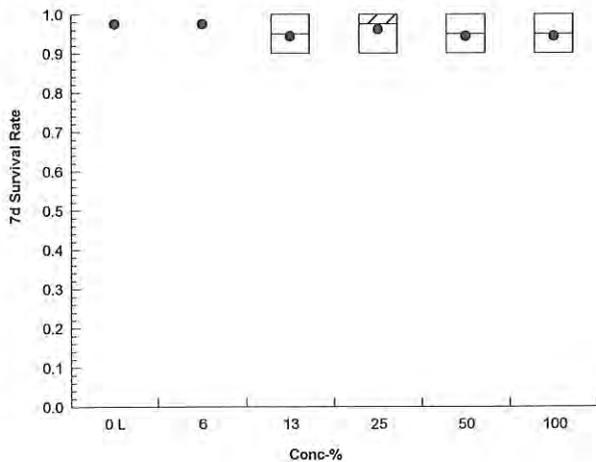
### Angular (Corrected) Transformed Detail

Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4
0	L	1.412	1.412	1.412	1.412
6		1.412	1.412	1.412	1.412
13		1.249	1.412	1.412	1.249
25		1.412	1.412	1.412	1.249
50		1.412	1.249	1.249	1.412
100		1.249	1.249	1.412	1.412

### 7d Survival Rate Binomials

Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4
0	L	10/10	10/10	10/10	10/10
6		10/10	10/10	10/10	10/10
13		9/10	10/10	10/10	9/10
25		10/10	10/10	10/10	9/10
50		10/10	9/10	9/10	10/10
100		9/10	9/10	10/10	10/10

### Graphics



**CETIS Analytical Report**

Report Date: 28 Aug-19 12:30 (p 1 of 2)  
 Test Code/ID: 719A8A12 / 19-0595-3298

**Fathead Minnow 7-d Larval Survival and Growth Test**

ERM

Analysis ID: 09-9546-1868	Endpoint: Mean Dry Biomass-mg	CETIS Version: CETISv1.9.4
Analyzed: 28 Aug-19 12:30	Analysis: Parametric-Control vs Treatments	Status Level: 1
Batch ID: 17-4757-0902	Test Type: Growth-Survival (7d)	Analyst: Lab Tech
Start Date: 19 Aug-19 17:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Reconstituted Water
Ending Date: 26 Aug-19 16:30	Species: Pimephales promelas	Brine:
Test Length: 6d 23h	Taxon: Actinopterygii	Source: In-House Culture Age: <24
Sample ID: 17-9487-7362	Code: 6AFBA7B2	Project: WET Testing
Sample Date: 19 Aug-19 07:15	Material: Industrial Effluent	Source: ArcelorMittal Burns Harbor, LLC
Receipt Date: 19 Aug-19 15:30	CAS (PC):	Station: Outfall 001
Sample Age: 10h (5 °C)	Client: ArcelorMittal Burns Harbor, LLC	

Data Transform	Alt Hyp	NOEL	LOEL	TOEL	TU	PMSD
Untransformed	C > T	100	>100	n/a	1	16.66%

**Dunnett Multiple Comparison Test**

Control	vs	Conc-%	Test Stat	Critical	MSD	DF	P-Type	P-Value	Decision(α:5%)
Lab Water		6	0.485	2.407	0.087	6	CDF	0.6540	Non-Significant Effect
		13	0.4296	2.407	0.087	6	CDF	0.6777	Non-Significant Effect
		25	0.3741	2.407	0.087	6	CDF	0.7007	Non-Significant Effect
		50	0.7829	2.407	0.087	6	CDF	0.5200	Non-Significant Effect
		100	-0.00693	2.407	0.087	6	CDF	0.8354	Non-Significant Effect

**Test Acceptability Criteria**

Attribute	Test Stat	TAC Limits		Overlap	Decision
		Lower	Upper		
Control Resp	0.5212	0.25	>>	Yes	Passes Criteria

**ANOVA Table**

Source	Sum Squares	Mean Square	DF	F Stat	P-Value	Decision(α:5%)
Between	0.0024073	0.0004815	5	0.1849	0.9646	Non-Significant Effect
Error	0.04687	0.0026039	18			
Total	0.0492773		23			

**Distributional Tests**

Attribute	Test	Test Stat	Critical	P-Value	Decision(α:1%)
Variances	Bartlett Equality of Variance Test	3.096	15.09	0.6852	Equal Variances
Distribution	Shapiro-Wilk W Normality Test	0.9534	0.884	0.3198	Normal Distribution

**Mean Dry Biomass-mg Summary**

Conc-%	Code	Count	Mean	95% LCL	95% UCL	Median	Min	Max	Std Err	CV%	%Effect
0	L	4	0.5212	0.4513	0.5912	0.51	0.486	0.579	0.02198	8.43%	0.00%
6		4	0.5037	0.4155	0.592	0.5	0.445	0.57	0.02772	11.01%	3.36%
13		4	0.5057	0.4702	0.5413	0.508	0.477	0.53	0.01117	4.42%	2.97%
25		4	0.5078	0.4286	0.5869	0.51	0.45	0.561	0.02488	9.80%	2.59%
50		4	0.493	0.411	0.575	0.5055	0.42	0.541	0.02575	10.45%	5.42%
100		4	0.5215	0.4091	0.6339	0.5365	0.432	0.581	0.03531	13.54%	-0.05%

**Mean Dry Biomass-mg Detail**

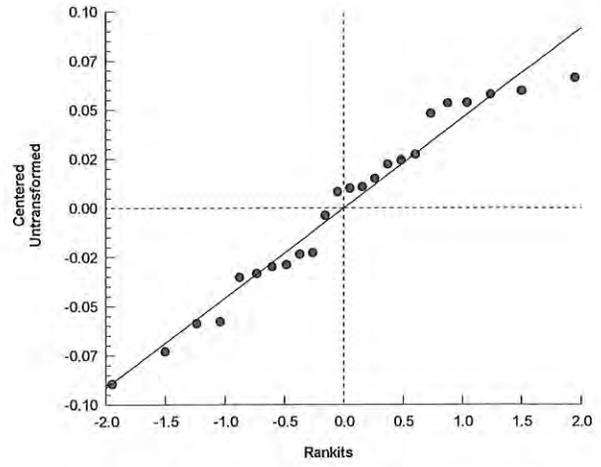
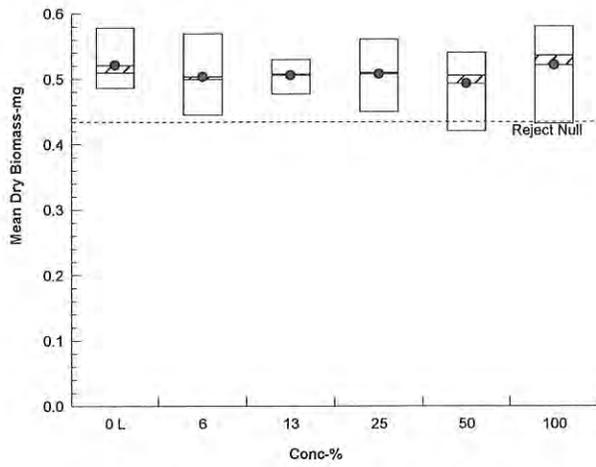
Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4
0	L	0.486	0.488	0.532	0.579
6		0.57	0.526	0.445	0.474
13		0.477	0.502	0.514	0.53
25		0.561	0.535	0.485	0.45
50		0.503	0.508	0.42	0.541
100		0.575	0.432	0.581	0.498

Fathead Minnow 7-d Larval Survival and Growth Test

ERM

Analysis ID: 09-9546-1868      Endpoint: Mean Dry Biomass-mg      CETIS Version: CETISv1.9.4  
Analyzed: 28 Aug-19 12:30      Analysis: Parametric-Control vs Treatments      Status Level: 1

Graphics



**CETIS Analytical Report**

Report Date: 28 Aug-19 12:30 (p 1 of 2)  
 Test Code/ID: 719A8A12 / 19-0595-3298

**Fathead Minnow 7-d Larval Survival and Growth Test**

ERM

Analysis ID: 02-1580-4861	Endpoint: Mean Dry Biomass-mg	CETIS Version: CETISv1.9.4
Analyzed: 28 Aug-19 12:30	Analysis: Linear Interpolation (ICPIN)	Status Level: 1
Batch ID: 17-4757-0902	Test Type: Growth-Survival (7d)	Analyst: Lab Tech
Start Date: 19 Aug-19 17:30	Protocol: EPA/821/R-02-013 (2002)	Diluent: Reconstituted Water
Ending Date: 26 Aug-19 16:30	Species: Pimephales promelas	Brine:
Test Length: 6d 23h	Taxon: Actinopterygii	Source: In-House Culture      Age: <24
Sample ID: 17-9487-7362	Code: 6AFBA7B2	Project: WET Testing
Sample Date: 19 Aug-19 07:15	Material: Industrial Effluent	Source: ArcelorMittal Burns Harbor, LLC
Receipt Date: 19 Aug-19 15:30	CAS (PC):	Station: Outfall 001
Sample Age: 10h (5 °C)	Client: ArcelorMittal Burns Harbor, LLC	

**Linear Interpolation Options**

X Transform	Y Transform	Seed	Resamples	Exp 95% CL	Method
Log(X+1)	Linear	1624462	200	Yes	Two-Point Interpolation

**Test Acceptability Criteria**

Attribute	Test Stat	TAC Limits		Overlap	Decision
		Lower	Upper		
Control Resp	0.5212	0.25	>>	Yes	Passes Criteria

**Point Estimates**

Level	%	95% LCL	95% UCL	TU	95% LCL	95% UCL
IC5	>100	n/a	n/a	<1	n/a	n/a
IC10	>100	n/a	n/a	<1	n/a	n/a
IC15	>100	n/a	n/a	<1	n/a	n/a
IC20	>100	n/a	n/a	<1	n/a	n/a
IC25	>100	n/a	n/a	<1	n/a	n/a
IC40	>100	n/a	n/a	<1	n/a	n/a
IC50	>100	n/a	n/a	<1	n/a	n/a

**Mean Dry Biomass-mg Summary**

Conc-%	Code	Count	Calculated Variate							Isotonic Variate	
			Mean	Min	Max	Std Dev	CV%	%Effect	Mean	%Effect	
0	L	4	0.5212	0.486	0.579	0.04396	8.44%	0.0%	0.5212	0.0%	
6		4	0.5037	0.445	0.57	0.05544	11.01%	3.36%	0.5063	2.86%	
13		4	0.5057	0.477	0.53	0.02234	4.42%	2.97%	0.5063	2.86%	
25		4	0.5078	0.45	0.561	0.04977	9.80%	2.59%	0.5063	2.86%	
50		4	0.493	0.42	0.541	0.0515	10.45%	5.42%	0.5063	2.86%	
100		4	0.5215	0.432	0.581	0.07063	13.54%	-0.05%	0.5063	2.86%	

**Mean Dry Biomass-mg Detail**

Conc-%	Code	Rep 1	Rep 2	Rep 3	Rep 4
0	L	0.486	0.488	0.532	0.579
6		0.57	0.526	0.445	0.474
13		0.477	0.502	0.514	0.53
25		0.561	0.535	0.485	0.45
50		0.503	0.508	0.42	0.541
100		0.575	0.432	0.581	0.498

# CETIS Analytical Report

Report Date: 28 Aug-19 12:30 (p 2 of 2)  
Test Code/ID: 719A8A12 / 19-0595-3298

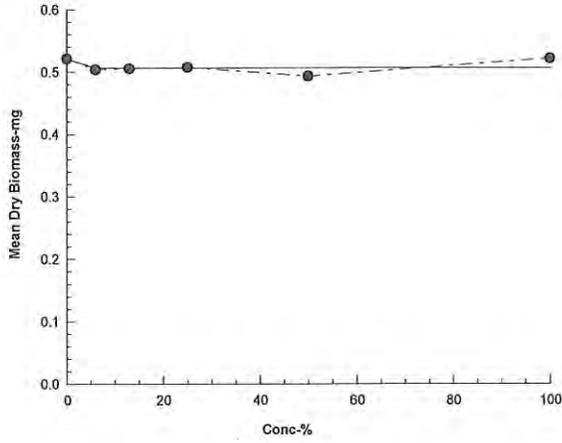
## Fathead Minnow 7-d Larval Survival and Growth Test

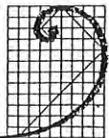
ERM

Analysis ID: 02-1580-4861      Endpoint: Mean Dry Biomass-mg  
Analyzed: 28 Aug-19 12:30      Analysis: Linear Interpolation (ICPIN)

CETIS Version: CETISv1.9.4  
Status Level: 1

### Graphics





**ERM**<sup>®</sup>

**ENVIRONMENTAL RESOURCES MANAGEMENT**

3352 128<sup>th</sup> Avenue Holland, Michigan 49424-9263

Phone: 616-399-3500 FAX: 616-399-3777

**AQUATIC TOXICITY LAB CHAIN OF CUSTODY FORM \***

CLIENT NAME:		SAMPLER								
ADDRESS:		PHONE NUMBER:								
SAMPLE DESCRIPTION (i.e. Outfall 001)	DATE (Begin/End)	TIME (Begin/End)	GRAB OR COMP	NUMBER AND SIZE OF CONTAINERS	FIELD PARAMETERS	SAMPLE ID NUMBER (Filled in by ERM)	INITIAL WATER QUALITY PARAMETERS UPON RECEIPT BY LABORATORY (filled in by ERM)			
	08/19/19	0715	C	1 cubitainer 2-5B	pH= s.u. NH <sub>3</sub> = mg/L	081919-2	Temp. (°C) 5.0 <input checked="" type="checkbox"/> On Ice	D.O. 12.3 mg/L	pH 7.9 s.u.	Cond 394 umhos/cm
G11	08/19/19	0830	G	~	pH= s.u. NH <sub>3</sub> = mg/L	-3	Temp. (°C) 5.0 <input checked="" type="checkbox"/> On Ice	D.O. 5.2 mg/L	pH 7.7 s.u.	Cond 409 umhos/cm
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH s.u.	Cond umhos/cm
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH s.u.	Cond umhos/cm
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH s.u.	Cond umhos/cm
					pH= s.u. NH <sub>3</sub> = mg/L		Temp. (°C) <input type="checkbox"/> On Ice	D.O. mg/L	pH s.u.	Cond umhos/cm
ANALYSES REQUESTED [check item(s)]	Test Material: <input type="checkbox"/> Water/Wastewater <input type="checkbox"/> Sediment <input type="checkbox"/> Product	Test Type: <input type="checkbox"/> Acute <input type="checkbox"/> Chronic <input type="checkbox"/> Other	Test Species: <input type="checkbox"/> <i>Ceriodaphnia dubia</i> <input type="checkbox"/> <i>Daphnia magna</i> <input type="checkbox"/> <i>Daphnia pulex</i> <input type="checkbox"/> Fathead minnow ( <i>Pimephales promelas</i> ) <input type="checkbox"/> Rainbow Trout ( <i>Oncorhynchus mykiss</i> ) <input type="checkbox"/> Sheepshead minnow ( <i>Cyprinodon variegatus</i> ) <input type="checkbox"/> Silverside minnow ( <i>Menidia beryllina</i> ) <input type="checkbox"/> Other (write in comments section)							
COMMENT SECTION: See AAS COC 41501-512										

**SAMPLE TRANSFERS**

RELINQUISHED BY: Signature / Organization	DATE	TIME	ACCEPTED BY: Signature / Organization	DATE	TIME
			<i>[Signature]</i>	8/19/19	1530

\* See Instructions for Sample Collection on Back of Sheet



Everett, WA  
+1 425 356 2600

Holland, MI  
+1 616 399 6070

Chain of Custody Form  
Page      of       
COC ID: 41581

ALS Project Manager:

Project Information

Project Name  
Project Number  
Bill To Company  
Invoice Attn  
Address  
City/State/Zip  
Phone  
Fax  
e-Mail Address

Sample Description  
Date  
Time  
Matrix  
Pres.  
# Bottles

ALS Work Order #

Parameter/Method Requested

ALS Project Manager

ALS Work Order #  
Parameter/Method Requested  
WETT (Acute + Chronic - Pres.)

Project Name

Project Number

Bill To Company

ALS Project Manager

Invoice Attn

Address

City/State/Zip

ALS Project Manager

Phone

Fax

e-Mail Address

ALS Project Manager

Sample Description

Date

Time

ALS Project Manager

Matrix

Pres.

# Bottles

ALS Project Manager

Turnaround Time in Business Days (BD)

Other

Results Due Date:

ALS Project Manager

Received by (Laboratory):

Received by (Laboratory):

Checked by (Laboratory):

ALS Project Manager

Reservative Key: 1-HCl 2-HNO<sub>3</sub> 3-H<sub>2</sub>SO<sub>4</sub> 4-NaOH 5-Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 6-NaHSO<sub>4</sub> 7-Other 8-4°C 9-5035

QC Package: (Check One Box Below)

Level II Std QC  
Level III Std QC/Raw Date  
Level IV SW846/CLP  
Other

ALS Project Manager

1. Any changes must be made in writing once samples and COC Form have been submitted to ALS Environmental.

2. Unless otherwise agreed in a formal contract, services provided by ALS Environmental are expressly limited to the terms and conditions stated on the reverse.

3. The Chain of Custody is a legal document. All information must be completed accurately.

ALS Project Manager

Signature

Date

Time

ALS Project Manager



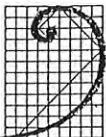
# Chain of Custody Form

Cincinnati, OH +1 513 733 5336  
 Fort Collins, CO +1 970 490 1511  
 Houston, TX +1 281 530 5656  
 South Charleston, WV +1 304 356 3168  
 Everett, WA +1 425 356 2600  
 Holland, MI +1 616 399 6070  
 Middletown, PA +1 717 944 5541  
 Salt Lake City, UT +1 801 266 7700  
 York, PA +1 717 505 5280

Page      of       
**COC ID: 41865**

Customer Information		Project Information		ALS Work Order #: <b>9081459</b>														
Purchase Order	Project Name	Parameter/Method Request for Analysis																
Work Order	Project Number	Round 2 Volume - WET (sub EAM)																
Company Name	Bill To Company	Arcelor Mittal WET																
Send Report To	Invoice Attn																	
Address	Address																	
City/State/Zip	City/State/Zip																	
Phone	Phone																	
Fax	Fax																	
e-Mail Address	e-Mail Address																	
No.	Sample Description	Date	Time	Matrix	Pres.	# Bottles	A	B	C	D	E	F	G	H	I	J	Hold	
1	Outfall 001 - Comp	8-21-19	0625	AQ	8	1=2.5gal.	X											
2	Outfall 011 - Comp	8-21-19	0605	AQ	8	2=2gal.	X											
3	001		011															
4	Temp		3															
5	PH		7.1															
6	PH		7.3															
7	COND		426															
8																		
9																		
10																		
Sampler(s) Please Print & Sign		Shipment Method		Turnaround Time in Business Days (BD) <input type="checkbox"/> 10 BD <input type="checkbox"/> 5 BD <input type="checkbox"/> 3 BD <input type="checkbox"/> 2 BD <input type="checkbox"/> 1 BD														
Received by: <i>B. Frye</i>		Received by (Laboratory):		Results Due Date:														
Date: 8-21-19		Time:		Notes:														
Date: 8-21-19		Time: 1100		Cooler ID														
Date: 8-21-19		Time: 1100		Cooler Temp														
Date: 8-21-19		Time: 1100		Checked by (Laboratory):														
Date: 8-21-19		Time: 1100		QC Package: (Check One Box Below)														
Date: 8-21-19		Time: 1100		<input type="checkbox"/> Level II Std QC <input type="checkbox"/> TRRP Checklist <input type="checkbox"/> Level III Std QC/Raw Date <input type="checkbox"/> TRRP Level IV <input type="checkbox"/> Level IV SW846/CLP <input type="checkbox"/> Other														
Date: 8-21-19		Time: 1100		Preservative Key: 1-HCl 2-HNO <sub>3</sub> 3-H <sub>2</sub> SO <sub>4</sub> 4-NaOH 5-Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 6-NaHSO <sub>4</sub> 7-Other 8-4°C 9-5035														

Note: 1. Any changes must be made in writing once samples and COC Form have been submitted to ALS Environmental.  
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 3. The Chain of Custody is a legal document. All information must be completed accurately.



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**ENVIRONMENTAL RESOURCES MANAGEMENT**

3352 128<sup>th</sup> Avenue Holland, Michigan 49424-9263

Phone: 616-399-3500 FAX: 616-399-3777

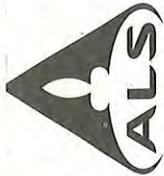
**AQUATIC TOXICITY LAB CHAIN OF CUSTODY FORM \***

CLIENT NAME:		SAMPLER				PHONE NUMBER:		FIELD PARAMETERS		SAMPLE ID NUMBER		INITIAL WATER QUALITY PARAMETERS UPON RECEIPT BY LABORATORY									
ADDRESS:		NUMBER AND SIZE OF CONTAINERS				GRAB OR COMP		TIME (Begin/End)		DATE (Begin/End)		pH= NH <sub>3</sub> =		D.O.		Temp. (°C)		pH		Cond	
SAMPLER		FIELD PARAMETERS		SAMPLE ID NUMBER		INITIAL WATER QUALITY PARAMETERS UPON RECEIPT BY LABORATORY		pH= NH <sub>3</sub> =		D.O.		Temp. (°C)		pH		Cond		pH		Cond	
Arcelor Mittal								06/22/19 0620		06/22/19 0620		77		10.7		7.7		7.7		407	
011								06/22/19 0635		-4		8.9		7.7		4.5		7.7		4.5	
ANALYSES REQUESTED [check item(s)]		Test Material:		Test Type:		Test Species:		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
COMMENT SECTION: See AHS LOC 4157A-59A		Water/Wastewater		Acute		Ceriodaphnia dubia		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
		Sediment		Chronic		Daphnia magna		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
		Product		Other		Daphnia pulex		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
						Fathead minnow (Pimephales promelas)		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
						Rainbow Trout (Oncorhynchus mykiss)		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
						Sheepshead minnow (Cyprinodon variegatus)		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
						Silverside minnow (Menidia beryllina)		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	
						Other (write in comments section)		pH= NH <sub>3</sub> =		D.O. <td colspan="2">Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td></td>		Temp. (°C) <td colspan="2">pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td></td>		pH <td colspan="2">Cond <td colspan="2">pH <td colspan="2">Cond </td></td></td>		Cond <td colspan="2">pH <td colspan="2">Cond </td></td>		pH <td colspan="2">Cond </td>		Cond	

**SAMPLE TRANSFERS**

RELINQUISHED BY: Signature / Organization	DATE	TIME	ACCEPTED BY: Signature / Organization	DATE	TIME
			<i>[Signature]</i>	08/23/19	1430

\* See Instructions for Sample Collection on Back of Sheet



# Chain of Custody Form

Cincinnati, OH +1 513 733 5336  
 Fort Collins, CO +1 970 490 1511  
 Houston, TX +1 281 530 5656  
 Spring City, PA +1 610 948 4903  
 South Charleston, WV +1 304 356 3168  
 Everett, WA +1 425 356 2600  
 Holland, MI +1 616 399 6070  
 Middletown, PA +1 717 944 5541  
 Salt Lake City, UT +1 801 266 7700  
 York, PA +1 717 503 5280

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**COC ID: 41578**

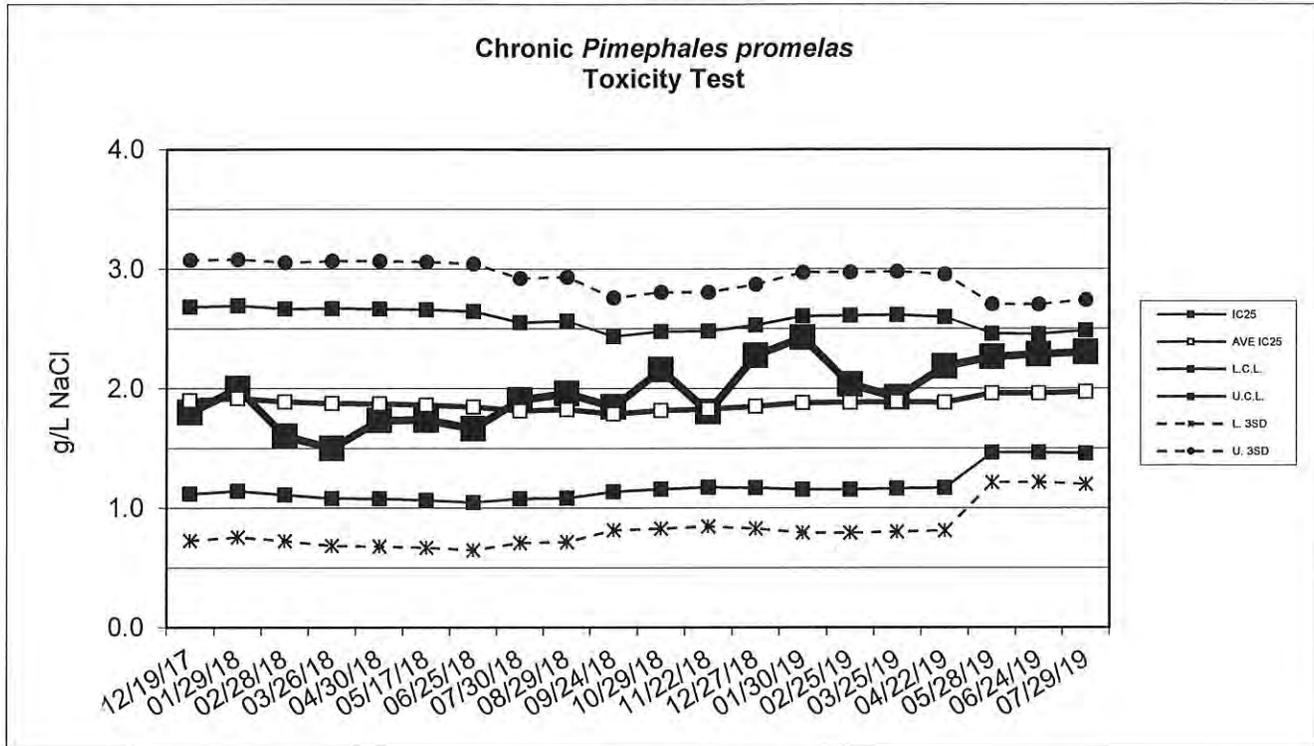
Customer Information		Project Information		ALS Work Order #: 19081459											
Parameter/Method Request for Analysis		ALS Project Manager:		Parameter/Method Request for Analysis											
Purchase Order	Project Name	AMBH WET #3	# Bottles	A	B	C	D	E	F	G	H	I	J	Hold	
Work Order	Project Number														
Company Name	Bill To Company	Arcebor Mital	Pres.												
Send Report To	Invoice Attn														
Address	Address														
City/State/Zip	City/State/Zip														
Phone	Phone														
Fax	Fax														
e-Mail Address	e-Mail Address														
No.	Sample Description	Date	Time	Matrix	Pres.	# Bottles									
1	Outfall 001 Comp	8-22-19	06 20	AQ	8	(2)-3gal.	X								
2	Outfall 011 Comp	8-22-19	06 35	AQ	8	(2)-3gal.	X								
3															
4															
5															
6															
7															
8															
9															
10															
Sampler(s) Please Print & Sign		Shipment Method		Turnaround Time in Business Days (BD)				Results Due Date:							
Relinquished by: <i>B. Boyle</i>		Received by: <i>[Signature]</i>		<input type="checkbox"/> 10 BD <input type="checkbox"/> 5 BD <input type="checkbox"/> 3 BD <input type="checkbox"/> 1 BD <input type="checkbox"/> Other <input type="checkbox"/> 2 BD <input type="checkbox"/> 1 BD											
Relinquished by: <i>[Signature]</i>		Received by (Laboratory): <i>[Signature]</i>		Cooler ID				Cooler Temp							
Logged by (Laboratory):		Checked by (Laboratory):		Cooler ID				Cooler Temp							
Preservative Key: 1-HCl 2-HNO <sub>3</sub> 3-H <sub>2</sub> SO <sub>4</sub> 4-NaOH 5-Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 6-NaHSO <sub>4</sub> 7-Other 8-4°C 9-5035		Notes: <i>082219 1430</i>		QC Package: (Check One Box Below)											
				<input type="checkbox"/> Level II Std OC <input type="checkbox"/> TRRP Checklist <input type="checkbox"/> Level III Std QC/Raw Date <input type="checkbox"/> TRRP Level IV <input type="checkbox"/> Level IV SW/846/CLP <input type="checkbox"/> Other											

Note: 1. Any changes must be made in writing once samples and COC Form have been submitted to ALS Environmental.  
 2. Unless otherwise agreed in a formal contract, services provided by ALS Environmental are expressly limited to the terms and conditions stated on the reverse.  
 3. The Chain of Custody is a legal document. All information must be completed accurately.



# Environmental Resources Management

## Standard Reference Toxicant Data



**Chronic *Pimephales promelas* Toxicity Test Data**

Date	IC25 (g/L NaCl)	AVE IC25 (g/L NaCl)	CONTROL LIMIT		Survival (%)	CONTROL Growth (mg)	CV (%)
			Lower	Upper			
12/19/17	1.8	1.9	1.1	2.7	100	0.58	5.3
01/29/18	2.0	1.9	1.1	2.7	97.5	0.39	4.8
02/28/18	1.6	1.9	1.1	2.7	92.5	0.44	10.7
03/26/18	1.5	1.9	1.1	2.7	97.5	0.47	3.5
04/30/18	1.7	1.9	1.1	2.7	95	0.45	11.4
05/17/18	1.7	1.9	1.1	2.7	100	0.54	10.8
06/25/18	1.7	1.8	1.0	2.6	95	0.56	17.8
07/30/18	1.9	1.8	1.1	2.6	97.5	0.43	4.3
08/29/18	2.0	1.8	1.1	2.6	100	0.58	9.4
09/24/18	1.8	1.8	1.1	2.4	97.5	0.46	8.2
10/29/18	2.2	1.8	1.2	2.5	97.5	0.45	7.7
11/22/18	1.8	1.8	1.2	2.5	95	0.65	5.2
12/27/18	2.3	1.8	1.2	2.5	97.5	0.64	7.4
01/30/19	2.4	1.9	1.2	2.6	100	0.53	10.5
02/25/19	2.0	1.9	1.2	2.6	95	0.53	10.2
03/25/19	1.9	1.9	1.2	2.6	97.5	0.63	6.0
04/22/19	2.2	1.9	1.2	2.6	100	0.57	2.0
05/28/19	2.3	2.0	1.5	2.5	100	0.68	10.4
06/24/19	2.3	2.0	1.5	2.5	92.5	0.48	11.0
07/29/19	2.3	2.0	1.5	2.5	100	0.51	5.6