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water resources / environmental consultants

CORRECTIVE ACTION PLAN
INTERIM FINAL REPORT
GERDAU MACSTEEL, INC.

DECEMBER 31, 2019

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Prepared for

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1.0 INTRODUCTION

In its letter dated August 22, 2017, the Arkansas Department of Energy and Environment, Division of Environmental Quality (DEQ) noted that from July 1, 2014, through July 31, 2017, Gerdau MacSteel Inc. (Gerdau) had reported five violations of the permit effluent limitations (i.e., Gerdau exceeded its permitted total recoverable lead limit in 2 reporting periods and its permitted chemical oxygen demand [COD] limit in 3 reporting periods) as well as 12 Whole Effluent Toxicity (WET) testing violations. Based on the DEQ review, the August 22, 2017 letter further requested that Gerdau submit a Corrective Action Plan (CAP) to include the following information:

- Causes of the effluent, sanitary sewer overflow (SSO), and bypass violations[1];
- Evaluation of the current permitted treatment system for compliance within the requirements of the permit;
- Actions to be taken to correct and prevent the recurrence of the violations; and
- A milestone schedule for compliance with the permit effluent limits, with a final date of compliance not to exceed December 31, 2017[2].

Accordingly, Gerdau submitted the CAP on September 20, 2017, which DEQ approved in writing on October 17, 2017. The conditions of the CAP include submittal of monthly progress reports by the 15th of each month until completion of the CAP. Upon completion of the CAP, a final report, stamped by a P.E. licensed in the state of Arkansas, certifying compliance with the permit upon completion of actions taken to correct, and prevent, the recurrence of the effluent violations shall be submitted to DEQ.

[1] Gerdau assumed that the mention of "SSO and bypass" violations in the DEQ letter was a misprint as there are no storm sewer or bypass aspects to the facility.
[2] In its letter dated October 17, 2017, DEQ corrected the misprinted December 31, 2017 date referenced in the August 22, 2017 letter to December 31, 2019.

This document provides a summary of CAP activities and findings during the CAP action period (October 2017 through December 2019). However, the CAP activities did not identify any actions that, when implemented, would correct and prevent the recurrence of the effluent violations. Therefore, no certification of permit compliance is provided herein. Instead, this document proposes to continue efforts to identify the cause(s) of effluent toxicity and/or actions to prevent it.

2.0 EFFLUENT PERMIT LIMITS EXCEEDANCES: EVALUATION AND CORRECTIVE ACTION

2.1 Effluent Violations for Lead

Gerdau exceeded its permitted lead limit in 2 of 37 monthly reporting periods between July 1, 2014, and July 31, 2017, prior to the implementation of the CAP. These two violations exceeded the permit limit of 5.4 µg/L by 0.8 and 1.6 µg/L. Following implementation of the CAP, Gerdau exceeded its permitted lead limit during 2 reporting periods; November 2018 and November 2019. These two violations exceeded the permit limit of 5.4 µg/L by 2.0 and 1.8 µg/L, respectively. The most likely cause of these infrequent and extremely small exceedance margins is sample contamination during collection or analysis.

2.2 Effluent Violations for Copper

Exceedances of the permitted copper limit did not occur during DEQ's review of certified Discharge Monitoring Reports from July 1, 2014, through July 31, 2017; however, following implementation of the CAP, Gerdau exceeded its permitted copper limit during 2 reporting periods; December 2017 and November 2019. These two violations exceeded the permit limit of 18.5 µg/L by 7.6 and 18.5 µg/L, respectively. The cause of these infrequent exceedances is unknown but may be the result of sample contamination during collection or analysis.

2.3 Corrective Action for Effluent Violations for Lead and Copper

On November 8, 2017, FTN personnel visited Gerdau to observe metal sampling techniques utilized by Gerdau's contract laboratory for collecting composite lead samples. FTN personnel returned on November 9, 2017, to observe sample packaging and transport to the contract laboratory. The following observations were made:

- An automatic composite sampler was used to collect samples for metal analysis. The composite sampler was set to collect a 200 mL sample every hour for 24 hours. The samples were composited upon collection into a single container located within the sampler. Cubed ice was placed around the container to chill the samples during collection.

- Sampling was performed by a single representative from the contract laboratory. As a result, the “clean hands, dirty hands” technique described in "clean sampling" protocols was not followed.
- The laboratory representative stated that the tubing between the sampler and the sample collection point was not new but was only used at the Gerdau site. New tubing was placed within the automatic sampling unit.
- The weighted end of the sample tubing was lowered into the water from the gantry at the outfall structure. The laboratory representative stated that at times the water level is rather shallow and the sampler will inadvertently strike the bottom of the ditch. Once in position below the water level, the sample tubing is secured to maintain that position. At that point, the weighted end of the tubing rests against the gantry structure.
- The area around the outfall structure is surrounded with slag and is located near a haul road used to transport slag. The haul road is covered with slag as well.

Based on these observations and a review of the permit, FTN offered the following recommendations for improving compliance with lead limitations:

- The permit requires the collection of composite samples for monitoring of copper and lead concentrations in the outfall. However, the use of a composite sampler prohibits the use of clean sampling techniques. Changing the sample type required in the permit to a grab sample would allow implementation of clean sampling techniques which would reduce the potential for contamination of samples collected for metal analyses.
- Use of new tubing each time samples are collected.
- Use of a second sampling person to allow implementation of “clean hands, dirty hands” techniques to reduce likelihood of contamination.
- Although composite samples should be flow-weighted, current sampling practices collect a time-weighted sample. This procedure is acceptable provided Gerdau maintains documentation of constant flows during the sampling period.
- The weighted end of the sampling tube is cast into the water sometimes striking the bottom of the ditch and disturbing sediments. The sampling tube also rests against the gantry structure during sample collection. These are potential sources of copper and lead contamination. A device, such as a moveable or extendable boom, that can allow the sampling tube to be suspended away from the gantry structure and avoid disturbing the bottom of the ditch should help reduce potential sample contamination.

Per National Pollution Discharge Elimination System (NPDES) Permit No. AR0039730, sampling with a composite sampler has continued at the designated Outfall location. Composite samples are collected as a time-weighted sample. New tubing is utilized for each composite sample. The weighted end of the sampling tube is carefully lowered into the water to avoid striking the bottom of the ditch and disturbing sediments though still rests against the gantry structure during sample collection. It is unclear if these actions have reduced the potential for sample contamination due to recent (though infrequent) exceedances of copper and lead.

2.4 Effluent Violations for Chemical Oxygen Demand

Gerdau exceeded its permitted COD limit in 3 of 37 monthly reporting periods between July 1, 2014, and July 31, 2017, prior to the implementation of the CAP. The three violations occurred in consecutive months (December 2016 and January and February 2017) and exceeded the permit limit of 100 mg/L during those months by 901, 41, and 8 mg/L, respectively. Following implementation of the CAP, Gerdau has not observed exceedances in the permitted COD limit. The most likely cause of the exceedances during the 3-month period was runoff from the onsite roads due to dust control activities during low-flow conditions.

2.5 Corrective Action for Effluent Violations for Chemical Oxygen Demand

In March of 2017, Gerdau conducted a study of potential sources and causes of the COD exceedances that occurred during the preceding months. That study (provided as Attachment 1) concluded that runoff into the ditch from road surfaces due to dust control activities during low-flow conditions was a likely cause of the COD concentrations exceeding permit limits and accumulation of algal growth. High COD levels were the likely result of a combination of COD that solubilized from the material applied to settle the dust at the site and algal biomass due to the input of nutrients.

Gerdau evaluated the current and alternative dust control products for their potential to elevate COD concentrations (see Section 4.1). Following confirmation that dust control products have the potential to elevate COD concentrations, Gerdau implemented closer management of runoff from the roads during road watering activities during low-flow conditions.

3.0 TOXICOLOGICAL EVALUATIONS

3.1 Routine Biomonitoring

Gerdau completed WET testing[3] at Outfall 001 on 14 occasions during the CAP action period. Samples were tested for toxicity to Ceriodaphnia dubia and Pimephales promelas per NPDES requirements according to EPA methods 1002.0 and 1000.0, respectively (USEPA 2002a). Results of the 14 WET tests conducted during the CAP action period are summarized in Table 1. All 14 tests were conducted using P. promelas while 11 tests were conducted using C. dubia. The results showed sub-lethal toxicity to C. dubia in 1 of 11 tests and sub-lethal toxicity to P. promelas in 3 of 14 tests. No lethal toxicity to either species was observed in the tests.

[3] WET testing conducted by American Interplex Corporation, 8600 Kanis Rd. Little Rock, AR, 72204

Table 1. Summary of No Observed Effect Concentration values (% effluent) from definitive chronic tests at Outfall 001 during the CAP action period.

Biomonitoring		***Ceriodaphnia dubia***		***Pimephales promelas***	
Begin[1]	**End[2]**	**Survival**	**Reproduction**	**Survival**	**Growth**
01/30/18	02/06/18	100	100	100	100
05/15/18	05/22/18[a]	100	100	100	100
08/14/18	08/21/18[b]	100	100	100	100
11/13/18	11/20/18[a]	100	100	100	45
12/11/18	12/18/18	No Test		100	<32
01/15/19	01/22/19[b]	100	100	100	100
02/26/19	03/05/19[b]	100	100	100	100
03/19/19	03/26/19	No Test		100	100
06/04/19	06/11/19	No Test		100	100
07/16/19	07/23/19	100	100	100	80
09/24/19	10/01/19[b]	100	32	100	<32
10/15/19	10/22/19[b]	100	100	100	100
11/19/19	11/26/19	100	100	100	100
12/10/19	12/17/19	100	100	100	100

1 – First day of analysis; 2 – Last day of analysis; a – Test ended 1 day later for *Ceriodaphnia dubia*; b – Test ended 1 day earlier for *Ceriodaphnia dubia*.

Sublethal toxicity to P. promelas was observed at Outfall 001 during November 2018 WET testing. Per the requirements of NPDES Permit No. AR0039730, Gerdau increased the frequency of WET testing to monthly until such time compliance with the No Observed Effect Concentration (NOEC) effluent limitation (lethal limit = 100%; sublethal limit = 80%) was demonstrated for a period of three consecutive months. Sublethal toxicity to P. promelas was again observed during December 2018 WET testing. Based on the persistent sublethal toxicity, Gerdau attempted to conduct Toxicity Identification Evaluation (TIE) follow-up testing during the required retesting period if the effluent sample failed WET testing. A screening sample collected in January 2018 for the purpose of TIE follow-up testing was not successful due to loss of sample toxicity during the one week of storage that occurred while the initial test (to determine if the sample was toxic) was performed (see Section 3.2.2). In response to this problem, the testing was modified such that a grab of sufficient volume was collected from the outfall immediately following collection of the third composite used for WET testing. This modification was intended to circumvent the need to delay TIE testing for one week while the initial test was conducted and reduce the chance of conducting TIE testing on a sample that was not toxic to begin with; however, no toxicity was observed at Outfall 001 during the January, February, or March 2019 WET testing (Table 1) and no TIE follow-up was conducted. Gerdau resumed quarterly testing following demonstration of compliance with the NOEC effluent limitation for three consecutive months.

Sublethal toxicity to C. dubia and P. promelas was observed at Outfall 001 during September 2019 WET testing (Table 1); however, adequate sample volume was not collected to conduct follow-up TIE testing. As with the November 2018 WET testing failure, Gerdau increased the frequency of WET testing to monthly until such time compliance with the NOEC effluent limitation was demonstrated for a period of three consecutive months and attempted to conduct TIE follow-up testing using a grab sample collected immediately following collection of the third composite sample used for WET testing during the required retesting period. No toxicity was observed at Outfall 001 during the October, November, or December 2019 WET testing (Table 1) and no TIE follow-up was conducted. Gerdau will resume quarterly testing in 2020.

3.2 **Effluent Screening**

Chronic screening tests were conducted[4] using *C. dubia* and *P. promelas* during November 2017 and January and February 2018. The purpose of the screenings was to obtain a sample showing a sufficient level of toxicity for TIE analyses per USEPA (1991) and to determine if the properties of the toxicity observed previously were still present. Two types of tests based on USEPA (2002a) were conducted: A 3-brood survival and reproduction test using *C. dubia* with untreated (i.e., “as received” Outfall 001 sample and a paired (i.e., “side by side”) 7-day survival and growth test using *P. promelas* with untreated sample and sample treated by

[4] Toxicity testing conducted by American Interplex Corporation, 8600 Kanis Rd. Little Rock, AR, 72204

exposure to UV light[5]. Exposure to UV light has been shown to reduce or remove lethal toxicity in previous testing (Attachment 2).

3.2.1 Sample Collected on November 27, 2017

On November 27, 2017, Gerdau collected a single grab sample from Outfall 001 to screen for toxicity to *C. dubia* and *P. promelas*. Results of these tests are provided in Tables 2, 3 and 4. There were no lethal or sub-lethal effects to *C. dubia* (Table 2). Lethality, though not significant, to *P. promelas* was removed by UV treatment (Table 3) and there were no sub-lethal effects in either untreated or UV-treated sample (Table 4). Other results from the testing using *P. promelas* (not shown) such as the onset of mortality at day 7 and similarity of response among replicates suggests that the lethal toxicity in untreated sample is not due to pathogen interference. This is the same pattern seen in the past testing (see Attachment 1).

Chemical analysis of the sample (Table 5) indicated no toxicologically significant concentrations of metals or inorganic ions.

[5] Sample treated by exposure to UV light was not tested during February 2018.

Table 2. Results of the 3-brood survival and reproduction test using *Ceriodaphnia dubia* with Outfall 001 sample collected on November 27, 2017.

Exposure (% Sample)	***Ceriodaphnia dubia***	
	% Survival (N= 10)	**Mean Reproduction**
Control	100	15.3
25	100	17.7
50	100	18.8
100	100	17.4

Table 3. Results of 7-day survival and growth test using *Pimephales promelas* with Outfall 001 sample collected on November 27, 2017: Percent survival in untreated and UV-treated sample.

***Pimephales promelas* % Survival (N = 20) on Day 7**		
Exposure (% Sample)	Untreated	UV-treated
Control	100	100
25	95	100
50	90	100
100	60	90

Table 4. Results of 7-day survival and growth test using *Pimephales promelas* with Outfall 001 sample collected on November 27, 2017: Average dry weight of fish in untreated and UV-treated sample.

***Pimephales promelas* Growth: Average dry weight (mg/fish) of surviving fish**		
Exposure (% Sample)	Untreated	UV-treated
Control	0.539	0.429
25	0.451	0.428
50	0.384	0.447
100	0.311	0.427

Table 5. Results of chemical analysis of Outfall 001 sample collected on November 27, 2017.

Analyte	Result		DL	Units
	Total Recoverable	Dissolved		
Aluminum	< 40	< 40	40	ug/l
Antimony	< 5	< 5	5	ug/l
Arsenic	< 5	< 5	5	ug/l
Barium	98	98	2	ug/l
Beryllium	< 0.5	< 0.5	0.5	ug/l
Boron	270	280	100	ug/l
Cadmium	< 0.5	< 0.5	0.5	ug/l
Calcium	74000	74000	100	ug/l
Chromium	< 5	< 5	5	ug/l
Cobalt	< 10	< 10	10	ug/l
Copper	4.8	3.9	0.5	ug/l
Iron	670	490	50	ug/l
Lead	< 0.5	< 0.5	0.5	ug/l
Magnesium	24000	24000	50	ug/l
Manganese	2200	2100	2	ug/l
Molybdenum	75	75	10	ug/l
Nickel	8.7	8.7	0.5	ug/l
Potassium	4200	4000	1000	ug/l
Selenium	< 2	< 2	2	ug/l
Silver	< 0.5	< 0.5	0.5	ug/l
Sodium	91000	91000	1000	ug/l
Thallium	< 0.5	< 0.5	0.5	ug/l
Vanadium	< 10	< 10	10	ug/l
Zinc	< 10	< 10	10	ug/l
Bromide	< 0.2	NA	0.2	mg/l
Chloride	70	NA	2	mg/l
Nitrite as N	< 0.05	NA	0.05	mg/l
Nitrate as N	< 0.05	NA	0.05	mg/l
Sulfate	110	NA	2	mg/l

DL = Detection limit

3.2.2 Sample Collected on January 3, 2018

On January 3, 2018, Gerdau collected a single grab sample from Outfall 001 to screen for toxicity to *C. dubia* and *P. promelas.* There were no lethal or sub-lethal effects to *C. dubia*; however, lethal and sub-lethal toxicity to *P. promelas* were observed (Table 6) and follow-up TIE testing using *P. promelas* was conducted using the TIE manipulations summarized in Table 7. Only lethality was considered in the *P. promelas* follow-up testing. Each sample treatment included a negative control (blank) which consisted of laboratory culture water treated in the same manner as the sample. Although the initial screening test showed lethal effects at the 100% exposure, the baseline test associated with the TIE treatments, which are by necessity begun one week after sample collection, did not. The laboratory results associated with this testing are provided in Table 8.

Table 6. Results of the 3-brood survival and reproduction test using *Ceriodaphnia dubia* and 7-day growth and survival test using *Pimephales promelas* with Outfall 001 sample collected on January 3, 2018.

	Ceriodaphnia dubia		***Pimephales promelas***	
Exposure (% Sample)	**% Survival (N= 10)**	**Mean Reproduction**	**% Survival (N = 40)**	**Mean Growth (mg/fish)**
Control	100	15.1	100	0.387
32	100	20.3	95.0	0.257*
45	100	19.9	95.0	0.235*
56	100	20.5	92.5	0.191*
80	100	19.6	62.5*	--
100	100	20.3	52.5*	--

* Significantly less than the control.

Table 7. Summary of Toxicity Identification Evaluation treatments conducted on sample collected on January 3, 2018.

Treatment Description	Effect on Sample
Baseline; No treatment	Positive control
UV - Exposure to ultraviolet light.	Kills pathogens; Degrades some organic compounds and enhances the toxicity of others.
pH3 adjustment - Adjust pH to 3 su with HCl and readjust to pH*i* with NaOH.	Degrades or hydrolyzes some toxicants such as anionic organic compounds.
pH11 adjustment - Adjust pH to 11 su with NaOH; filter precipitate and readjust to pH*i* with HCl.	Precipitate metals and other substances with pH sensitive solubility. Adds additional ions to sample due to readjustment to pH*i* which can assist in diagnosing toxicity due to ionic strength/composition.
EDTA - Addition of ethylenediaminetetraacetic acid (EDTA)	Chelate metals and some organic compounds.
STS - Addition of sodium thiosulfate (STS)	Neutralize (reduce) oxidizing substances and can chelate some metals and metaloids.
SPE (C18) - 0.45 µm filter/solid phase extraction (SPE). Filter sample and pass filtrate through a C18 SPE column at 25 mL/min. Conduct acute test on methanol eluate if toxicity is removed.	Remove non-polar or weakly polar organic compounds and provides additional filtration of sample. Toxicity removal with subsequent toxicity recovery in the column elutriate suggests further follow-up focused on non-polar organic toxicants. Toxicity removal without subsequent recovery with methanol elution can be due to enhanced filtration or highly non-polar toxicants.

Table 8. Percent survival (N = 40, except as noted) in toxicity tests conducted on samples manipulated as given in Table 7.

Test Exposure (%)	Sample Manipulation								
	Lab control	Baseline	UV	pH 3	pH 11	EDTA	STS	SPE	SPE Eluate*
50	NA	100	88	88	85	82	92	88	NA
100	100	75	90	92	90	85	90	88	100**

* N = 20; ** Exposure corresponds to theoretical 400X concentration of non-polar organic toxicants in sample.

3.2.3 Sample Collected on February 19, 2018

On February 19, 2018, Gerdau collected a single grab sample from Outfall 001 to screen for toxicity to *C. dubia* and *P. promelas.* No TIE follow-up testing was conducted because the results of the screening test (Table 9) indicated no lethal or sub-lethal toxicity to *C. dubia* or *P. promelas*.

Table 9. Results of the 3-brood survival and reproduction test using *Ceriodaphnia dubia* and 7-day growth and survival test using *Pimephales promelas* with Outfall 001 sample collected on February 19, 2018.

Exposure (% Sample)	*Ceriodaphnia dubia*		*Pimephales promelas*	
	% Survival (N= 10)	**Mean Reproduction**	**% Survival (N = 40)**	**Mean Growth (mg/fish)**
Control	100	19.4	100	0.538
32	100	23.3	97.5	0.560
45	100	23.6	92.5	0.549
56	100	24.6	95.0	0.572
80	100	26.0	92.5	0.537
100	100	25.3	95.0	0.563

3.3 Non-Effluent Screening: Samples collected on December 4, 2017

On December 4, 2017, Gerdau staff notice unusual coloration in one pool of the storm water collection ditch that drains the Gerdau facility. A sample of the discolored water was collected along with a sample of unaffected water from a location immediately upstream (Figure 1) and shipped to the laboratory for toxicity testing. Each sample was tested using a 3-brood survival and reproduction test using *C. dubia* and a 7-day survival and growth test with *P. promelas.* Results of the tests are provided in Tables 10 and 11. The unaffected sample collected immediately upstream was not toxic to *C. dubia*; however, the discolored sample was sub-lethally toxic to *C. dubia* (Table 10). Both samples were lethally toxic to *P. promelas* with 100% mortality occurring by the end of the tests (7 days). Percent survival on each day of the test was similar for the 2 samples (Table 11) which indicates that both samples were equally toxic to *P. promelas.*

Episodes of toxicity occurring at Outfall 001 typically affect *P. promelas* more than *C. dubia.* Therefore, the discoloration observed in the storm water ditch during early

December 2017 is probably not related to episodes of toxicity to *P. promelas*. However, the discoloration might be relevant to occasional episodes of toxicity to *C. dubia* that have occurred in the past.

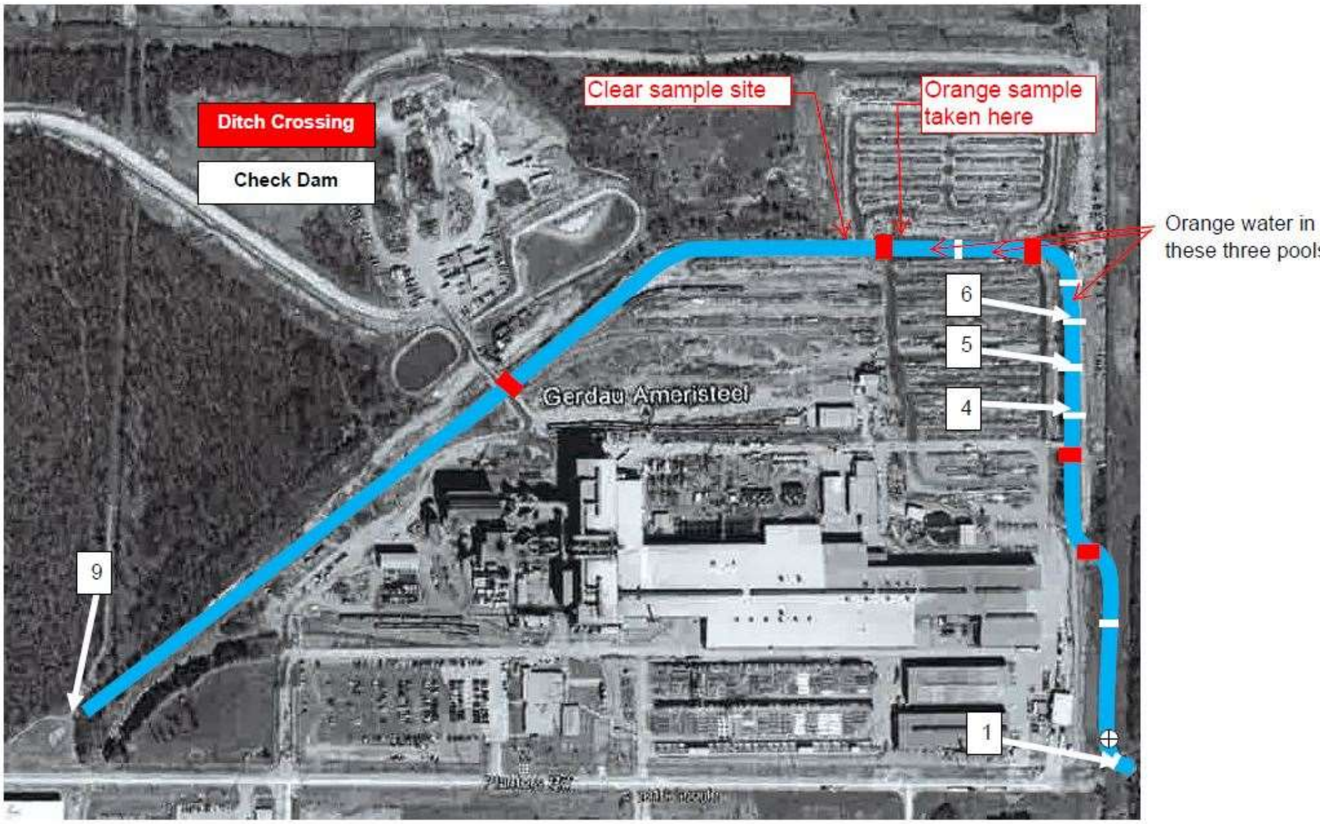

Figure 1. Sample locations of December 4, 2017 samples collected for toxicity testing.

Table 10. Summary of chronic screening tests using samples collected on December 4, 2017.

Test species	Test Endpoint	Exposure (% Sample)	Normal Color	Discolored	Control
Ceriodaphnia dubia	% Survival (N= 10)	100	100	70	100
	Reproduction (Average number of neonates)	100	14.7	1.0	16.2
Pimephales promelas	% Survival (N= 40)	100	0	0	100
	Growth (Average dry weight (mg) per fish)	100	NA	NA	0.302

NA = Not applicable

Table 11. Summary of *Pimephales promelas* percent survival on each day of tests using samples collected on December 4, 2017.

Sample	*Pimephales promelas* % Survival by Day						
	1	2	3	4	5	6	7
Control	100	100	100	100	100	100	100
Clear	100	100	70	35	7.5	0	-
Orange	100	100	75	35	0	-	-

4.0 SOURCE EVALUATION: TOXICOLOGICAL EVALUATION OF DUST CONTROL PRODUCTS AND ONSITE SLAG

Based on the non-persistent toxicity observed in the effluent and, therefore, unsuccessful TIE follow-up, Gerdau modified the testing protocol for TIE such that the TIE tests would begin upon sample arrival without an intervening initial test to determine if the sample was toxic. This modification would circumvent the need to delay TIE testing for 1 week while the initial test was conducted, but might also result in conducting TIE testing on a sample that was not toxic to begin with and wasted resources. Gerdau also elected to focus on potential sources of toxicity such as slag and dust control products rather than relying solely on TIE evaluations of Outfall 001. Gerdau would continue to monitor Outfall 001 through routine biomonitoring with the intention of conducting TIE follow-up in the event toxicity was observed.

4.1 Toxicological Evaluation of Dust Control Products – March 2018

The current dust control product (Haul-EZ) was evaluated as a potential factor in observed toxicity. Gerdau developed an experimental protocol to evaluate the effects of Haul-EZ on toxicity as well as evaluate an alternate dust control product that could potentially remove toxicity.

During March 2018, eight dust control products (Table 12) were evaluated for potential replacement of Haul-EZ which is currently used at the Gerdau facility. The choice of the eight products tested was based on products used at other Gerdau facilities and similar products suggested by vendor representatives.

The experimental approach to evaluating toxicity of dust control products involved simulating the application of dust control product on a dusty surface that is subsequently disrupted and suspended in water from rain events and addition of water to the surfaces for additional dust control. This process was simulated in the laboratory by constructing small dusty soil surfaces, treating each surface with a dust control product per manufacturer's recommendations, allowing the treated surfaces to "cure" in warm dry conditions and preparing an aqueous extract of each treated surface material for toxicity/chemical testing. It is recognized that this experimental procedure does not precisely capture the operations at the Gerdau facility.

However, the procedure does simulate the combination of dust control product with fine "dusty" material and water in a way that is comparable among different products. Therefore, the results of the toxicity tests on the aqueous extracts are valid and useful for comparing *relative* toxicity among the various products under the test conditions.

Table 12. Dust control products used in the March 2018 evaluation and their general chemical composition.

Product	Composition
Haul-EZ 81201 (used at Jackson, TN plant)	Glycerol: 60-100% (CAS 56-81-5); Methanol: 0.1-1% (CAS 67-56-1)
Borresperse AM 870L	Ammonium lignosulfate: 32% (CAS 8061-53-8); Sodium lignosulfate: 17% (CAS 8061-51-6); Water: 51%
Dustabate 6123 (used at Gerdau in the past)	Heavy petroleum extracts (naphthenic distillate; cyclic aliphatic hydrocarbon [cyclo-alkane]): 60% (CAS 64742-11-6); Emulsifiers & water: 40%
Dust Bond (similar or identical to Dustabate 6123)	Heavy petroleum extracts (naphthenic distillate; cyclic aliphatic hydrocarbon [cyclo-alkane]): 60-65% (CAS 64742-11-6); Water: 35-40%; Proprietary ingredients: < 5%
Earth Armour 2100	Severely hydrotreated[1] high viscosity, synthetic iso-alkane: >50% (CAS 72623-86-0)
EnviroKleen 2800	Severely hydrotreated[1], high viscosity, synthetic iso-alkane: >10% (CAS 72623-86-0); Polyolefin: <60% (CAS 9003-27-4)
NORLIG A 58% (Recommendation of NALCO)	Calcium Lignosulfonate (polymer): 50% (CAS 8061-52-7); Water: 50%
SOKLEEN (Used at Rancho Cucamonga, CA plant)	Severely hydrotreated, high viscosity, synthetic iso-alkane (non-cyclic, saturated): >50% (CAS 72623-86-0); C15- C30 lubricating oil: 50%
DVSLC010[2] (Recommendation of NALCO)	Fatty acid ester: 30-60% (CAS Proprietary) Oxyalkylated fatty acid derivative: 30-60% (CAS Proprietary) Inorganic solvent: 1-5% (CAS Proprietary)

1 – Impurities removed by treating with hydrogen; 2 – Used in 2019 evaluation only.

4.1.1 Sample Preparation

Soil used in the evaluation was collected from a hay barn that was not contaminated with equipment fluids. The soil was sifted through a 600-micrometer sieve to produce a fine dust for testing. For each treatment, approximately 3 liters of dust was evenly distributed in a 24" diameter water heater drain pan resulting in an approximate depth of 0.4 inches of dust. Each of eight dust control products was diluted with tap water (if needed) per manufacturer's recommendations and applied to an experimental pan using a squirt bottle at a coverage rate per the manufacturer's recommendations (Table 13). One experimental pan was not treated with a dust control product to serve as a negative control.

Table 13. Dilution ratios, coverage rates, and application volumes of dust control products used during the March 2018 study.

Product	Dilution Ratio (product to water)	Coverage Rate	Volume of Product Applied (oz)
Dustabate 6123	1:7	1.00 gal / sq yd	44.7
Norlig A 58%	1:1	0.40 gal / sq yd	17.9
Borresperse AM 870L	1:1	0.40 gal / sq yd	17.9
Haul-EZ	No Dilution	0.38 gal / sq yd	16.8
SoKleen	No Dilution	0.11 gal / sq yd	5.0
EnviroKleen 2800	No Dilution	0.11 gal / sq yd	5.0
Earth Armour 2100	No Dilution	0.11 gal / sq yd	5.0
Dust Bond	1:7	1.50 gal / sq yd	67.0

The 9 experimental pans (8 dust control product treatments and 1 untreated control) were placed on shelves approximately 14 inches below heat lamps that consisted of 10.5-inch aluminum reflectors and 250-watt clear bulbs. All nine pans were exposed to 12 hour on/off cycles for 10 days. Following the 10-day heat lamp exposure, contents of each pan were emptied into 5-gallon buckets, each containing 1.5 gallons of laboratory water[6] obtained from American Interplex Corporation (AIC). A lid was placed on each bucket and they were shaken on an industrial paint bucket shaker for approximately 30 seconds. The sediment was allowed to settle for 44 to 48 hours following shaking.

[6] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).

After the contents of the buckets were allowed to settle, the supernatant was slowly poured off the precipitate through cheesecloth as a pre-filter treatment to remove larger particles. Additional culture water was added to each sample, as required, to a total volume of 1 gallon[7]. The 1-gallon volume from each of the nine treatments was then filtered through a 0.45-micron filter to remove smaller particles. A 1-gallon aliquot of the laboratory water used to prepare the extracts was also filtered to provide a water control. The resulting 10 samples (8 dust control treatments, 1 untreated soil control, and 1 filtered laboratory water control) were submitted to the laboratory for toxicity and analytical testing.

4.1.2 Toxicity and Analytical Testing

The 10 samples were tested for acute toxicity to *C. dubia* and *P. promelas* in 48-hour static non-renewal tests based on the USEPA (2002b) procedure and analyzed for COD (Table 14).

Although acute toxicity was present in the dust control (Control-Dust) sample, the results of the toxicity tests on the aqueous extracts are valid and useful for comparing *relative* toxicity among the various products tested. Three dust control products, Norlig A 58%, Borresperse AM 870L, and Haul-EZ, increased toxicity relative to the dust control, greatly increased COD, and reduced pH. The remaining dust control products showed similar or improved toxicity relative to the dust control as well as similar COD and pH levels.

Based on the results of the acute testing, Norlig A 58% and Borresperse AM 870L were excluded from additional testing based on the observed toxicity and the remaining eight products were further tested using a chronic (7-day) test protocol[8]. Although Haul-EZ increased toxicity, it was included in chronic testing because it is the current product being used for dust control. It is worth noting that, in this experimental context, the most toxic products were the ligno sulfate (Norlig A 58% and Borresperse AM 870L) and glycerol (Haul-EZ) based products while the least toxic were all cyclo- and iso-alkane based products.

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[7] The volume of culture water added to the pre-filtered treatment and dust control samples ranged from 0 to 16 ounces.

[8] Due to limited sample volume the chronic tests using *P. promelas* were conducted with 5 replicates each with 50 mL test solution and 5 test organisms.

Table 15 provides chemistry and chronic toxicity results for the eight samples retained for additional testing following the acute toxicity results. *P. promelas* was selected as the test species due to a historical pattern of greater toxicity to fish than *C. dubia* at Outfall 001. Results from the chronic tests were similar to the results from the acute tests. With the exception of Haul-EZ, the dust control products identified in Table 15 showed similar or improved toxicity relative to the dust control. Haul-EZ was observed to increase toxicity relative to the dust control.

A review of the chemistry for the eight samples during chronic testing determined that the non-native soil produced ammonia levels in the samples that interfered with the results. Ammonia levels at the IC50 effect level ranged from 0.04 mg/L in Haul EZ sample to 13.69 mg/L in Earth Armour 2100 sample, with an average level of 9.92 mg/L. While ammonia levels interfered with the results for most products, the ammonia level in Haul-EZ at the IC50 effect level would not be expected to interfere with toxicity. Additionally, although the pH in the 100% eluate generated from Haul-EZ exposure could interfere with toxicity results, the initial and final pH values at the IC50 effect level were between 7.0 and 8.0 su; levels not expected to interfere with toxicity.

While five of the dust control products did not increased toxicity compared to the control, not all of these products were equally effective at eliminating dusty conditions. Three of the six products, Dustabate 6123, Earth Armour 2100, and Dust Bond, applied to the dust in the experimental pans evenly and completely. Of these three products, only Earth Armour 2100 appeared to completely conglomerate the fine dust particles after initial disturbance[9]. Dustabate 6123 and Dust Bond completely conglomerated the top ¼ to ½ of the dust layer and slightly conglomerated the remaining dust at the bottom of the layer. These two products reduced airborne dust approximate 25 to 50% during initial disturbance which was further reduced to no airborne dust after complete mixing by hand.

Earth Armour 2100 is an iso-alkane based dust control product that was observed to apply evenly and completely to a treatment area, eliminated airborne dust, reduced COD, had no obvious effect on pH, and reduced toxicity relative to the control. This dust control product was

[9] The initial disturbance consisted of gentle hand mixing in preparation for the transfer of the treated material from the pans to the buckets used to combine the samples with water.

further evaluated as a potential dust control product to combat future dust control issues (See Section 4.5).

Table 14. 48-hour acute survival and water chemistry data.

Water	*P. promelas*		*C. dubia*		COD (mg/L)	pH (s.u.)	Dissolved oxygen (mg/L)	Alkalinity (mg/L as CaCO3)	Hardness (mg/L as CaCO3)	Conductivity (umhos/cm)
	NOEC*	LC50*	NOEC*	LC50*						
Dilution Water	-	-	-	-	-	7.6	8.1	61	84	320
Dustabate 6123	50	70.7	50	70.7	3000	6.9	-	-	-	-
Norlig A 58%	12.5	13.4	6.25	8.1	42000	4.2	-	-	-	-
Borresperse AM 870L	12.5	17.7	6.25	8.1	33000	4.4	-	-	-	-
Haul-EZ	25	30.8	<6.25	<6.25	94000	5.6	-	-	-	-
SoKleen	100	>100	6.25	8.8	2200	7.0	-	-	-	-
EnviroKleen 2800	100	89.1	25	26.8	2000	6.8	-	-	-	-
Earth Armour 2100	50	70.7	25	35.4	210	7.0	-	-	-	-
Dust Bond	50	70.7	50	53.6	2800	6.8	-	-	-	-
Control - Dust	50	70.7	25	25.8	2300	7.1	-	-	-	-
Control - Water	100	>100	100	>100	10	7.2	-	-	-	-

* Percent sample

Table 15. 7-day chronic survival and water chemistry data.

Elutriate	*Pimephales promelas* NOEC* (lethality)	*Pimephales promelas* NOEC* (sub-lethality)	*Pimephales promelas* LC50* (linear interp)	pH (su)	Dissolved oxygen (mg/L)	Ammonia as N (mg/L)	Alkalinity (mg/L as CaCO3)	Hardness (mg/L as CaCO3)	Conductivity (umhos/cm)
Dilution Water	-	-	-	8.0	6.4	-	61	84	360
Dustabate 6123	<20	<20	21	7.2	0.84	61	500	370	3100
Haul-EZ	<20	<20	<20	5.4	2.2	0.35	230	1200	16000
SoKleen	<20	<20	28	7.4	0.99	36	520	560	2400
Envirokleen 2800	20	<20	35	7.1	0.81	39	380	310	2200
Earth Armour 2100	20	20	37	7.3	1.3	37	510	460	2500
Dust Bond	<20	<20	20	7.0	0.84	57	400	620	2600
Control - Dust	<20	<20	18	7.5	2.8	43	580	480	2500
Control - Water	80	80	>80	7.9	6.5	0.13	66	120	300

4.2 Toxicological Evaluation of Onsite "Unweathered Slag" – September 2018

Due to the presence of artifactual toxicity introduced through the use of non-native soil, the toxicological evaluation of dust control products was repeated using onsite slag material to reduce to potential for external factors that could potentially influence the toxicity results. Prior to repeating the experiment, Gerdau tested the slag material for toxicity to determine its suitability for use in the experiment.

4.2.1 Sample Preparation

Onsite slag used in the evaluation was newly produced slag with minimal environmental exposure ("unweathered"). The slag was sifted through a 600-micrometer sieve to produce a fine dust for testing. Approximately 2 liters of slag fines was placed into each of two 5-gallon buckets containing 2 gallons of laboratory water[10] obtained from AIC. A lid was placed on each bucket and they were shaken on an industrial paint bucket shaker for approximately 30 seconds. The sediment was allowed to settle for approximately 24 hours following shaking.

After the contents of the buckets were allowed to settle, the supernatant from the two buckets was slowly poured off the supernatant through cheesecloth as a filter treatment to remove any larger particles and composited into a single sample. The resulting elutriate and an aliquot of the laboratory water were submitted to the laboratory for toxicity and analytical testing.

4.2.2 Toxicity and Analytical Testing

The elutriate and laboratory water were tested for chronic toxicity to *P. promelas*[11] based on the USEPA (2002a) procedure. Additionally, the elutriate was analyzed for COD; total and dissolved copper, nickel, and zinc; alkalinity (as calcium carbonate [$CaCO_3$]); and hardness (as $CaCO_3$). Tables 16 and 17 provide the elutriate toxicity and analytical results, respectively.

Toxicity testing was initially conducted as a 5-concentration dilution series with a dilution factor of 0.5 (resulting in test concentrations of 6.25, 12.5, 25, 50, and 100% sample) using moderately hard (MH) laboratory water as a control and a diluent. The results of the 7-day

[10] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).
[11] *Pimephales promelas* was selected as the test species due to a historical pattern of greater toxicity to fish than *Ceriodaphnia dubia* at Outfall 001.

chronic test indicated a significant level of acute toxicity in the elutriate. A review of the analytical data identified pH at 12 su and a high ionic concentration. Additionally, the review suggested that metals were not present in significant concentrations to cause toxicity. Since significant toxicity is expected at a pH of 12 su, the pH of the elutriate was adjusted to 8 su and 100% sample was screened for toxicity to evaluate the potential for toxicity due to the high ionic concentration.

The pH adjusted elutriate screening test indicated a significant level of acute toxicity. The analytical results of the unadjusted elutriate showed a total alkalinity concentration of 1800 mg/L (as $CaCO_3$). At pH 12 su, alkalinity will be present primarily as carbonate (CO_3^{-2}). Upon adjusting the pH to 8 su, the alkalinity as carbonate would convert to alkalinity as bicarbonate (HCO_3^-). The toxicity in the pH adjusted elutriate was therefore likely due to toxic levels of bicarbonate originally present as carbonate. Gerdau treats the slag with calcium hydroxide ($Ca[OH]_2$) prior to storage. The high ionic concentration is likely due to bicarbonate as a byproduct of the calcium hydroxide treatment.

Table 16. Toxicity summary for the elutriate and fish culture water submitted September 25, 2018.

Pimephales promelas				
	Initial pH (12 su)			
	Elutriate		Fish Culture Water	
Exposure (% Sample)	**% Survival (N=40)**	**Mean Growth (mg/fish)**	**% Survival (N=40)**	**Mean Growth (mg/fish)**
Control	100	0.343	100	0.341
6.25	20*	--	100	0.331
12.5	0*	--	100	0.333
25	0*	--	100	0.359
50	0*	--	97.5	0.335
100	0*	--	95	0.33
	Adjusted pH (8 su)			
	Slag Exposed Water			
Exposure (% Sample)	**% Survival (N=40)**		**Mean Growth (mg/fish)**	
Control	100		0.321	
100	65*		--	

*Significantly less than the control (P < 0.05).

Table 17. Analytical summary for the elutriate and fish culture water submitted September 25, 2018.

Parameter	Results (mg/L unless otherwise noted)	
	Elutriate	Fish Culture Water
COD	44	47
Copper (total)	0.028	--
Copper (dissolved)	<0.01	--
Nickel (total)	<0.01	--
Nickel (dissolved)	<0.01	--
Zinc (total)	0.64	--
Zinc (dissolved)	0.23	--
Dissolved Oxygen	5.6	6.7
pH (su)	12	7.7
Alkalinity (as CaCO3)	1800	32
Hardness (as CaCO3)	990	45
Conductivity (µS/cm)	6900	110
Residual Chlorine	1.9	<0.05
Ammonia as N	1.9	<0.1

4.3 Toxicological Evaluation of Haul-EZ on Onsite "Weathered" Slag – January 2019

Gerdau uses calcium hydroxide (hydrated lime) in their blast furnace to create slag aggregates during the production of iron and steel. Calcium hydroxide on the slag surface quickly converts to calcium carbonate when exposed to the atmosphere. Because calcium carbonate is removed from the slag surface by rainwater containing carbon dioxide (CO_2), bicarbonate levels are expected to be significantly lower in runoff exposed to slag with environmental exposure ("weathered"), thus reducing the toxicity observed in the September toxicological evaluation of unweathered slag. However, breaking or crushing slag into smaller sizes (e.g., driving over them with heavy equipment) could cause elevated bicarbonate levels in the short-term. An experiment was conducted to evaluate the potential effects of the current dust control product, Haul-EZ, by comparing toxicity results from elutriate prepared from weathered slag not treated with dust control product (untreated) to elutriate prepared from weathered slag treated with dust control product (treated).

4.3.1 Sample Preparation

Weathered slag was collected from two locations at the Gerdau site. One sample consisted of weathered slag that had been applied to a roadway but had not been treated with dust control product (untreated). A second sample consisted of weathered slag that had been applied to a roadway and had subsequently been treated with dust control (treated). For each sample, approximately 3 liters of slag as collected[12] was placed into each of two 5-gallon buckets containing 3 gallons of laboratory water[13] obtained from AIC. A lid was placed on each bucket and they were shaken on an industrial paint bucket shaker for approximately 30 seconds. The sediment was allowed to settle for approximately 72 hours following shaking.

After the contents of the buckets were allowed to settle, the supernatant from the two buckets of each exposure (treated and untreated) was removed using a peristaltic pump and composited into a single sample. The resulting two samples (untreated slag elutriate and treated slag elutriate) and an aliquot of the laboratory water were submitted to the laboratory for toxicity and analytical testing.

4.3.2 Toxicity and Analytical Testing

The untreated and treated slag elutriates and laboratory water were tested for chronic toxicity to *P. promelas*[14] based on the USEPA (2002a) procedure. Additionally, the untreated and treated elutriates were analyzed for COD, total suspended solids (TSS), total and dissolved arsenic, copper, nickel, and zinc; oil and grease; alkalinity (as $CaCO_3$); and hardness (as $CaCO_3$). Tables 18 and 19 provide toxicity and analytical results, respectively, from the untreated slag elutriate, treated slag elutriate, and laboratory water testing.

The two elutriate samples and laboratory water were tested for chronic toxicity as a 7-day, 5-concentration dilution series with a dilution factor of 0.5 (resulting in test concentrations of 6.25, 12.5, 25, 50, and 100% sample) using MH laboratory water as a control and a diluent. The results of the 7-day chronic tests indicated a significant level of acute toxicity in the untreated slag elutriate. The treated slag elutriate indicated a significant level of chronic toxicity but no acute toxicity was observed. A review of the analytical data suggested that elutriate

[12] Slag material was not sieved down to ≤ 600 µm to produce a fine dust.
[13] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).
[14] *Pimephales promelas* was selected as the test species due to a historical pattern of greater toxicity to fish than *Ceriodaphnia dubia* at Outfall 001.

prepared from the unweathered slag had much higher ionic strength (mainly due to bicarbonate alkalinity) and was correspondingly more toxic than elutriate prepared from weathered slag.

Ionic strength (as indicated by conductivity), concentration of metals, and bicarbonate (as indicated by total alkalinity) were relatively low in the untreated slag elutriate and do not account for the observed toxicity. A reduction in the level of toxicity observed in the treated slag elutriate compared to the untreated slag elutriate suggests that the dust control product may reduce the toxic effects of the weathered slag. A review of the dissolved metals data from the treated slag exposed water identified that the dissolved copper level exceeded acute and chronic criteria (0.0327 and 0.0205 mg/L, respectively) based on site-specific hardness (200 mg/L as $CaCO_3$) which may have influenced the observed toxicity.

Testing of alternate dust control products in September 2018 suggested that iso-alkane based dust control products showed improved toxicity relative to Haul-EZ, a glycerol-based product, and January 2019 testing suggested that Haul-EZ improve toxicity relative to untreated slag.

Table 18. Toxicity summary (7-day chronic using *Pimephales promelas*) for the untreated slag elutriate, treated slag elutriate, and fish culture water submitted January 14, 2019.

Pimephales promelas						
	Untreated Slag Elutriate		**Treated Slag Elutriate**		**Fish Culture Water**	
Exposure (% Sample)	**% Survival (N=40)**	**Mean Growth (mg/fish)**	**% Survival (N=40)**	**Mean Growth (mg/fish)**	**% Survival (N=40)**	**Mean Growth (mg/fish)**
Control	100	0.26	97.5	0.26	100	0.267
6.25	100	0.208	97.5	0.241	92.5	0.275
12.5	85	0.173*	100	0.244*	87.5	0.234
25	85	0.164*	95	0.219*	90	0.256
50	67.5*	--	82.5	0.227*	95	0.247
100	0*	--	82.5	0.185*	97.5	0.266

*Significantly less than the control ($P < 0.05$)

Table 19. Analytical summary for the untreated slag elutriate, treated slag elutriate, and fish culture water submitted January 14, 2019.

Parameter	Results (mg/L unless otherwise noted)		
	Untreated Slag Elutriate	Treated Slag Elutriate	Fish Culture Water
COD	28	250	--
TSS	<10	95	--
Arsenic (total)	<0.05	<0.05	--
Arsenic (dissolved)	<0.05	<0.05	--
Copper (total)	<0.01	0.13	--
Copper (dissolved)	<0.01	0.043	--
Nickel (total)	<0.01	0.047	--
Nickel (dissolved)	<0.01	<0.01	--
Zinc (total)	0.011	0.38	--
Zinc (dissolved)	<0.01	0.11	--
Oil & Grease	<5	<5	--
Dissolved Oxygen	6.8	7.3	7.0
pH (su)	8.1*	8.2	7.5
Alkalinity (as CaCO3)	30	280	49
Hardness (as CaCO3)	91	200	85
Conductivity (µS/cm)	250	520	210
Residual Chlorine	<0.05	<0.05	<0.05
Ammonia as N	0.12	<0.1	<0.1

*The pH was adjusted from 9.5 su (initial pH).

4.4 Toxicological Evaluation of Onsite "Weathered" Slag – March 2019

September 2018 and January 2019 testing of onsite slag suggested that elutriates from unweathered and weathered slag were lethally toxic to *P. promelas*. January 2019 testing also suggested that elutriate from weathered slag subsequently treated with Haul-EZ was sub-lethally (but not lethally) toxic to *P. promelas*, suggesting that Haul-EZ may reduce the toxic effects of the slag. Finally, March 2018 testing of multiple dust control products suggested that iso-alkane based dust control products showed improved toxicity relative to Haul-EZ, a glycerol-based product. Based on these results, Gerdau developed an experimental approach to evaluate the effects of dust control products composed of glycerol (Haul-EZ) and iso-alkane (Earth Armour 2100 and EnviroKleen 2800) on the toxicity of weathered slag. Additionally, a previously untested dust control product composed of a fatty acid ester and oxyalkylated fatty acid derivative (DVSLC010) would be evaluated.

Weathered slag used in previous testing was not collected in sufficient volume to conduct the toxicological evaluation of the dust control products. Therefore, additional weathered slag was collected on February 25, 2019, from the same general area where weathered slag was collected for the January 2019 weathered slag toxicological evaluation in order to complete the dust control experiment and any future experiments. Gerdau tested the weathered slag elutriate for acute toxicity and chemistry to determine its suitability for use in the dust control experiment.

4.4.1 Sample Preparation

Approximately 2 liters of weathered slag collected on February 25, 2019, was placed into a 5-gallon bucket containing 2 gallons of laboratory water[15] obtained from AIC. A lid was placed on the bucket and it was shaken on an industrial paint bucket shaker for approximately 30 seconds. The sediment was allowed to settle for approximately 24 hours following shaking.

After the contents of the bucket were allowed to settle, 1 gallon of the supernatant was pumped into a plastic container using a peristaltic pump. The resulting sample and an aliquot of the laboratory water were submitted to the laboratory for toxicity and analytical testing.

4.4.2 Toxicity and Analytical Testing

The weathered slag elutriate and laboratory water were tested at 100% for acute toxicity to *P. promelas*[16] in a 48-hr static non-renewal test based on the USEPA (2002b) procedure. Additionally, the weathered slag elutriate was analyzed for COD; TSS; total and dissolved arsenic, copper, nickel, and zinc; oil and grease; alkalinity (as $CaCO_3$); and hardness (as $CaCO_3$). Tables 20 and 21 provide the weathered slag elutriate toxicity and analytical results, respectively.

Similar to previous testing, the pH was above 9 su in the weathered slag elutriate and, following pH adjustment, the acute test indicated a significant level of toxicity to *P. promelas*. A review of the analytical data identified similar chemistry in the weathered slag elutriate as

[15] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).
[16] *Pimephales promelas* was selected as the test species due to a historical pattern of greater toxicity to fish than *Ceriodaphnia dubia* at Outfall 001.

previously tested weathered slag. Based on the results of the toxicity testing and chemistry, the "weathered" slag collected on February 25, 2019, was suitable for use in the experiment.

Table 20. 48-hour acute survival data for the weathered slag elutriates submitted January 14 and March 11, 2019.

Exposure	*Pimephales promelas* % Survival (N=40)	
	January 16-18, 2019**	March 12-14, 2019
Moderately hard control	100	100
Weathered slag - 100%	5*	50*
Laboratory water	100	100

*Significantly less than the control; **Data interpolated from a 7-day chronic test

Table 21. Analytical summary for the weathered slag elutriates submitted January 14 and March 11, 2019.

Parameter	Results (mg/L unless otherwise noted)	
	Weathered Slag Elutriate January 14, 2019	Weathered Slag Elutriate March 11, 2019
COD	28	<10
TSS	<10	76
Arsenic (total)	<0.05	0.0017
Arsenic (dissolved)	<0.05	0.00059
Copper (total)	<0.01	0.0097
Copper (dissolved)	<0.01	0.006
Nickel (total)	<0.01	0.0073
Nickel (dissolved)	<0.01	0.007
Zinc (total)	0.011	0.049
Zinc (dissolved)	<0.01	<0.01
Oil & Grease	<5	<5
Dissolved Oxygen	6.8	7.1
Initial pH (su)	9.5	11.4
Toxicity Test pH (su)[1]	8.1	7.7
Alkalinity (as CaCO3)	30	NM
Hardness (as CaCO3)	91	NM
Conductivity (µS/cm)	250	290
Residual Chlorine	<0.05	<0.05
Ammonia as N	0.12	NM

1 – Adjusted from initial pH with sulfuric acid; NM – Not Measured

4.5 Toxicological Evaluation of Dust Control Products – March 2019

September 2018 and January 2019 testing of onsite slag suggested that elutriates from unweathered and weathered slag were lethally toxic to *P. promelas*. January 2019 testing also suggested that elutriate from weathered slag subsequently treated with Haul-EZ was sub-lethally (but not lethally) toxic to *P. promelas*, suggesting that Haul-EZ may reduce the toxic effects of the slag. Finally, March 2018 testing of multiple dust control products suggested that iso-alkane based dust control products showed improved toxicity relative to Haul-EZ, a glycerol-based product.

Based on these results, Gerdau developed an experimental approach to evaluate the effects of dust control products composed of glycerol (Haul-EZ) and iso-alkane (Earth Armour 2100 and EnviroKleen 2800) on the toxicity of weathered slag. Additionally, a previously untested dust control product composed of a fatty acid ester and oxyalkylated fatty acid derivative (DVSLC010) was evaluated. The general chemical composition of each of the four dust control products is provided in Table 12.

Similar to previous experimentation, the experimental approach to evaluating toxicity of dust control products involved simulating the application of dust control product on a dusty surface that is subsequently disrupted and suspended in water from rain events and addition of water to the surfaces for additional dust control. This process was simulated in the laboratory by constructing a small weathered slag surface, treating the surface with dust control product (a total of four separate products) per manufacturer's recommendations, allowing the treated surface to "cure" in warm dry conditions, and preparing an aqueous extract of the treated surface material for toxicity/chemical testing. It is recognized that this experimental procedure does not precisely capture the operations at the Gerdau facility. However, the procedure does simulate the combination of dust control product with weathered slag and water in a way that is comparable among different dust control products. Therefore, the results of the toxicity tests on the aqueous extracts are valid and useful for comparing *relative* toxicity among the various products tested.

4.5.1 Sample Preparation

Four dust control products with 3 different compositions were tested to evaluate the effects of dust control products on the toxicity of onsite slag. For each treatment approximately 3 liters of weathered slag[17] was evenly distributed in each of two 24" diameter water heater drain pans resulting in an approximate depth of 0.4 inches of slag in each pan. One of the 4 dust control products was diluted with tap water per manufacturer's recommendations for application; the remaining 3 products were not diluted per manufacturer's recommendations for application (Table 22). Each of the 4 dust control products was applied to 2 experimental pans using a squirt bottle at a coverage rate per the manufacturer's recommendations. Two experimental pans were not treated with a dust control product to serve as a negative control.

Table 22. Dilution ratios and application rates of dust control products used during the study.

Product	Dilution Ratio (product to water)	Recommended Coverage Rate	Volume of Product Applied (oz)
Haul-EZ	No Dilution	0.38 gal / sq yd	16.8
EnviroKleen 2800	No Dilution	0.11 gal / sq yd	5.0
Earth Armour 2100	No Dilution	0.11 gal / sq yd	5.0
DVLS010	1:12.33 (7.5% solution)	0.25 gal / sq yd	11.2

The 10 experimental pans (2 each for the 4 dust control product treatments and 1 untreated slag control) were placed on shelves approximately 14 inches below heat lamps that consisted of 10.5-inch aluminum reflectors and 250-watt clear bulbs. All 10 pans were continuously exposed to the heat lamps for 10 days. Following the 10-day heat lamp exposure, contents of each pan were emptied into 5-gallon buckets, each containing 3 gallons of laboratory water[18] obtained from AIC. A lid was placed on each bucket and they were shaken on an industrial paint bucket shaker for approximately 2 minutes. The sediment was allowed to settle for approximately 24 hours following shaking.

[17] Onsite slag collected on February 25, 2019. The slag had long-term environmental exposure and had not been treated with dust control product.

[18] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).

After the contents of the buckets were allowed to settle, 2 gallons of the supernatant was pumped from each of the 2 buckets corresponding to each treatment into a plastic container using a peristaltic pump. Additionally, an aliquot of the laboratory water used to prepare the extracts was prepared to provide a water control. The resulting 6 samples (4 dust control treatments, 1 untreated slag control, and 1 laboratory water control) were submitted to the laboratory for toxicity and analytical testing.

4.5.2 Toxicity and Analytical Testing

The 6 samples were tested for chronic toxicity to *P. promelas* based on the USEPA (2002a) procedure; tested for chronic toxicity as a 7-day, 5-concentration dilution series with a dilution factor of 0.5 (resulting in test concentrations of 6.25, 12.5, 25, 50, and 100% sample) using MH laboratory water as a control and a diluent. Each sample was analyzed for COD, TSS, total and dissolved metals (arsenic, copper, nickel, and zinc), oil and grease, alkalinity (as $CaCO_3$), and hardness (as $CaCO_3$). Tables 23 and 24 provide chronic toxicity and analytical results, respectively.

Although sub-lethal toxicity was present in the laboratory water sample, previous and current testing of the weathered slag material has identified that the slag is lethally toxic to *P. promelas*. The results of the toxicity tests on the aqueous extracts are valid and useful for comparing *relative* toxicity among the four products tested.

With the exception of Haul-EZ, the dust control products showed similar toxicity relative to the untreated slag control. Haul-EZ was observed to increase toxicity relative to the untreated slag control. In addition to increased toxicity, Haul-EZ greatly increased COD. The remaining dust control products showed similar or improved parameter levels relative to the untreated slag control though DVLSC010 increased COD above the maximum daily limit at Outfall 001.

Table 23. Toxicity summary (7-day chronic using *Pimephales promelas*) for the four dust control product elutriates, weathered slag elutriate, and laboratory water submitted March 25, 2019.

Exposure (% Sample)	*Pimephales promelas*											
	Haul-EZ		Earth Armour 2100		EnviroKleen 2800		DVLSC010		Weathered Slag		Fish Culture Water	
	% Survival (N=40)	Mean Growth (mg/fish)	% Survival (N=40)	Mean Growth (mg/fish)	% Survival (N=40)	Mean Growth (mg/fish)	% Survival (N=40)	Mean Growth (mg/fish)	% Survival (N=40)	Mean Growth (mg/fish)	% Survival (N=40)	Mean Growth (mg/fish)
Control	100	0.444	100	0.444	100	0.444	100	0.444	100	0.444	100	0.444
6.25	97.5	0.403	92.5	0.440	95	0.489	95	0.399	100	0.444		
12.5	90	0.303*	100	0.454	97.5	0.476	95	0.366	100	0.541		
25	60*	--	95	0.386	97.5	0.491	100	0.448	100	0.494		
50	17.5*	--	90*	--	100	0.449	95	0.352*	92.5	0.447		
100	0*	--	27.5*	--	35*	--	2.5*	--	45*	--	95	0.367*

*Significantly less than the control ($P < 0.05$).

Table 24. Analytical summary for the four dust control product elutriates, weathered slag elutriate, and laboratory water submitted March 25, 2019.

Parameter	Results (mg/L unless otherwise noted)					
	Haul-EZ	Earth Armour 2100	EnviroKleen 2800	DVLSC010	Weathered Slag	Laboratory Water[1]
COD	40000	49	76	160	65	<10
TSS	13	<10	<10	39	14	<10
Arsenic (total)	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Arsenic (dissolved)	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Copper (total)	0.037	<0.01	<0.01	<0.01	<0.01	0.01
Copper (dissolved)	0.036	<0.01	<0.01	<0.01	<0.01	<0.01
Nickel (total)	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Nickel (dissolved)	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zinc (total)	<0.01	<0.01	<0.01	<0.01	<0.01	0.012
Zinc (dissolved)	<0.01	<0.01	<0.01	<0.01	<0.01	0.012
Oil & Grease	<5	<5	6.2	<5	<5	<5
Dissolved Oxygen	6.2	7.4	7.1	7.0	7.8	7.1
Initial pH (su)	11.1	11.2	11.5	11.1	11.4	8.0
Toxicity Test pH (su)[2]	7.3	7.1	6.3	7.6	7.2	7.5
Alkalinity (as CaCO3)	510	940	870	500	910	71
Hardness (as CaCO3)	980	660	570	480	670	90
Conductivity (µS/cm)	8500	1600	1600	1000	2200	220
Residual Chlorine	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Ammonia as N	<0.1	0.14	0.13	<0.1	0.14	0.11

* Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR); Adjusted from initial pH with sulfuric acid

4.6 Toxicological Evaluation of Onsite Slag Persistent Toxicity – July 2019

Previous toxicity testing has demonstrated that onsite slag is a potential source of intermittent toxicity to *P. promelas* observed at Outfall 001. Test results from untreated slag elutriate indicate that, when present, toxicity is not persistent with nearly all effects occurring in the first 48 to 72 hours of the tests (Table 25). Based on these results, Gerdau developed an experimental approach to evaluate the persistence of toxicity of runoff from onsite slag.

Table 25. Survival by day in 7-day chronic tests using *Pimephales promelas* conducted on untreated slag elutriate in January and March 2019.

January 2019							
	Survival (N=40)						
Exposure (% Sample)	**Day 1 (24 hrs)**	**Day 2 (48 hrs)**	**Day 3 (72 hrs)**	**Day 4 (96 hrs)**	**Day 5 (120 hrs)**	**Day 6 (144 hrs)**	**Day 7 (168 hrs)**
Control	100	100	100	100	100	100	100
6.25	100	100	100	100	100	100	100
12.5	90	85	85	85	85	85	85
25	97.5	90	87.5	87.5	87.5	87.5	85
50	70	67.5	67.5	67.5	67.5	67.5	67.5
100	5	5	5	0	0	0	0
March 2019							
	Survival (N=40)						
Exposure (% Sample)	**Day 1 (24 hrs)**	**Day 2 (48 hrs)**	**Day 3 (72 hrs)**	**Day 4 (96 hrs)**	**Day 5 (120 hrs)**	**Day 6 (144 hrs)**	**Day 7 (168 hrs)**
Control	100	100	100	100	100	100	100
6.25	100	100	100	100	100	100	100
12.5	100	100	100	100	100	100	100
25	100	100	100	100	100	100	100
50	100	100	100	95	95	90	90
100	92.5	70	55	55	45	48	48

Similar to previous experimentation, the experimental approach to evaluating toxicity of onsite slag involved exposing the slag to laboratory water as a surrogate for rainwater. It is recognized this experimental procedure does not precisely mimic rain events. However, the procedure does simulate the combination of slag material and water in a way that is comparable across different tests.

4.6.1 Sample Preparation

Approximately 4 liters of weathered slag[19] was placed into each of two 5-gallon buckets containing 2 gallons of laboratory water[20] obtained from AIC. A lid was placed on each bucket and they were shaken on an industrial paint bucket shaker for approximately 2 minutes. The sediment was allowed to settle for approximately 5.5 hours following shaking. After the contents of the buckets were allowed to settle, 1.5 gallons of the supernatant was pumped from each of the two buckets into a plastic container using a peristaltic pump. Additionally, an aliquot of the laboratory water used to prepare the extracts was prepared to provide a water control. The weathered slag elutriate sample and laboratory water were submitted to the laboratory for toxicity and analytical testing.

4.6.2 Toxicity and Analytical Testing

The weathered slag elutriate was tested for acute toxicity to *P. promelas* based on the USEPA (2002b) procedure; tested for acute toxicity as a 48-hr static non-renewal, 5-concentration dilution series with a dilution factor of 0.5 (resulting in test concentrations of 6.5, 12.5, 25, 50, and 100% sample). Additionally, the weathered slag elutriate was analyzed for a suite of inorganic ions. The aliquot of the laboratory water used to prepare the extracts was tested at 100% for acute toxicity to *P. promelas* in a 48-hr static non-renewal test based on the USEPA (2002b) procedure. Table 26 provides acute toxicity results from the weathered slag elutriate and laboratory water testing. Table 27 provides analytical results for the weathered slag elutriate.

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[19] Onsite slag collected on February 25, 2019. The slag had long-term environmental exposure and had not been treated with dust control product.
[20] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).

Table 26. Toxicity summary (48-hr acute using *Pimephales promelas*) for the weathered slag elutriate and fish culture water submitted July 8, 2019.

Weathered Slag Elutriate								
Test	% Survival (N=40)							
	Day 1 - 7/9/19		Day 2 - 7/10/19		Day 3 - 7/11/19		Day 4 - 7/12/19	
Exposure (% Sample)	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs
Control	100	100	100	97.5	100	100	100	100
6.25	100	100	100	95	100	100	100	97.5
12.5	97.5	97.5	100	92.5	100	100	100	95
25	97.5	92.5	97.5	92.5	100	100	100	95
50	100	55*	97.5	82.5	100	100	97.5	82.5
100	12.5*	12.5*	97.5	72.5*	100	100	97.5	90
Fish Culture Water								
Test	% Survival (N=40)							
	Day 1 - 7/9/19		Day 2 - 7/10/19		Day 3 - 7/11/19		Day 4 - 7/12/19	
Exposure (% Sample)	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs
Control	100	100	100	97.5	100	100	100	100
100	100	100	100	100	100	100	97.5	97.5

*Significantly less than the control (P < 0.05).

Table 27. Analytical summary for the weathered slag elutriate submitted July 8, 2019.

Parameter	Result (mg/L)	Parameter	Result (mg/L)	Parameter	Result (mg/L)
Aluminum	2.1	Lead	<0.04	Sulfur	8.0
Antimony	0.27	Magnesium	1.3	Thallium	0.16
Arsenic	<0.05	Manganese	0.070	Tin	<0.1
Barium	0.28	Molybdenum	0.073	Titanium	0.010
Beryllium	<0.0005	Nickel	0.035	Vanadium	<0.01
Boron	<0.1	Phosphorus	<0.1	Zinc	0.22
Cadmium	<0.004	Potassium	2.2	Bromide	<0.2
Calcium	590	Selenium	<0.07	Chloride	8.5
Chromium	0.052	Silicon	1.4	Fluoride	2.3
Cobalt	<0.01	Silver	<0.005	Nitrate as N	5.5
Copper	0.016	Sodium	11	Nitrite as N	<0.05
Iron	0.30	Strontium	4.8	Sulfate	2.3

Similar to previous testing, the pH was above 9 su in the weathered slag elutriate and, following pH adjustment, acute testing indicated a significant level of lethal toxicity; however, lethal toxicity was not persistent and was not observed in the weathered slag elutriate after approximately 3 days (72 hours) following initial exposure of slag to laboratory water. Inorganic ions were measured; however, the concentrations were relatively low and the lack of persistent toxicity suggests that an inorganic ion(s) is not the cause of the observed toxicity.

4.7 Toxicity Identification Evaluation of Onsite Slag

Previous toxicity testing has demonstrated that onsite slag is a potential source of intermittent toxicity to *P. promelas* observed at Outfall 001. Test results indicate that, when present, toxicity is not persistent with nearly all effects occurring in the first 48 to 72 hours of the tests. Based on these results, Gerdau pursued an acute TIE approach to obtain information regarding the physical properties of the toxicant(s) that is present in the supernatant created from exposure of laboratory water to onsite slag. TIE testing was conducted in August, early-October, and late-October 2019.

Similar to previous experimentation, the experimental approach to conducting the TIE involved exposing the slag to laboratory water as a surrogate for rainwater. It is recognized this experimental procedure does not precisely mimic rain events. However, the procedure does simulate the combination of slag material and water in a way that is comparable across different tests.

4.7.1 Sample Preparation

Sample preparation for the three TIE attempts were prepared as follows. Approximately 4 liters of weathered slag[21] was placed into each of two 5-gallon buckets containing 2 gallons of laboratory water[22] obtained from AIC. A lid was placed on each bucket and they were vigorously shaken by hand (August 13 sample) or on an industrial paint shaker (October 1 and 23 samples) for approximately 2 minutes. The sediment was allowed to settle for approximately 15 hours following shaking. After the contents of the buckets were allowed to settle, 1 gallon of the supernatant was pumped from each of the two buckets into a plastic container using a peristaltic pump. The resulting elutriate sample was submitted to the laboratory for TIE testing. Additionally, an aliquot of the laboratory water used to prepare the extracts was submitted to the laboratory to provide a water control.

[21] Onsite slag collected on February 25, 2019. The slag had long-term environmental exposure and had not been treated with dust control product.
[22] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).

4.7.2 Toxicity Testing

For all three TIE attempts, slag elutriate aliquots manipulated with TIE treatments (Table 28) were tested for acute toxicity to *P. promelas* in 3-dilution series tests (100, 50, and 25%). Test organism performance (survival) in each TIE treatment was compared to organism performance in the baseline (no TIE treatment). Interpretation of acute TIE results does not typically involve formal statistical comparisons between manipulated samples and the baseline. Rather a "weight of evidence" approach is used to evaluate whether or not a particular treatment reduced or removed toxic effects. Tests on manipulated samples included negative controls or "blank" treatments conducted on laboratory water, i.e., MH laboratory water and water obtained from the *P. promelas* rearing tanks at AIC. Results from the blanks can indicate the presence of experimental artifacts.

Table 28. Summary of Toxicity Identification Evaluation treatments conducted on slag elutriate.

Treatment Description	Effect on Sample
Baseline; No treatment	Positive control
EDTA - Addition of ethylenediaminetetraacetic acid (EDTA)	Chelate metals and some organic compounds.
STS - Addition of sodium thiosulfate (STS)	Neutralize (reduce) oxidizing substances and can chelate some metals and metaloids.
SPE (C18) - 0.45 µm filter/solid phase extraction (SPE). Filter sample and pass filtrate through a C18 SPE column at 25 mL/min. Conduct acute test on methanol eluate if toxicity is removed.	Remove non-polar or weakly polar organic compounds and provides additional filtration of sample. Toxicity removal with subsequent toxicity recovery in the column elutriate suggests further follow-up focused on non-polar organic toxicants. Toxicity removal without subsequent recovery with methanol elution can be due to enhanced filtration or highly non-polar toxicants.
pH10 adjustment - Adjust pH to 10 su with NaOH; filter precipitate and readjust to pH*i* with H_2SO_4.	Precipitate metals and other substances with pH sensitive solubility. Adds additional ions to sample due to readjustment to pH*i* which can assist in diagnosing toxicity due to ionic strength/composition.
Filtration - Pass sample through a 1 µm glass fiber filter without organic binder.	Provides information on whether the toxicity is filterable yet provides relatively little specific information about which class of toxicant may be causing the toxicity.

Following the August 13, 2019 TIE attempt, the lack of acute toxicity to *P. promelas* in the baseline exposure and, therefore, the inability to interpret TIE results was attributed to the sample preparation method where the slag/laboratory water supernatant was generated by vigorous shaking by hand rather than shaking on an industrial paint shaker. Additional testing of the sample preparation methods was evaluated during the week of August 26, 2019 (see Section 4.8), prior to reattempting TIE testing.

Although some treatments (solid phase extraction [C18], pH 10 adjustment, and filtration) appeared to reduce toxicity in the two October TIE attempts[23], the insufficient toxicity to *P. promelas* in the baseline exposures precluded definitive conclusions of the type of toxicant that was present to justify additional follow-up testing (Table 29).

Table 29. Results of acute toxicity tests at the 100% concentration using *Pimephales promelas* in manipulated samples from Toxicity Identification Evaluation testing conducted on slag elutriate submitted on August 13, October 1, and October 23, 2019.

Exposure	% Mortality at 48 hrs (N=10)		
	Aug 13-15, 2019[4]	**Oct 1-3, 2019[5]**	**Oct 23-25, 2019[5]**
Moderately Hard Control	0	0	0
Laboratory Water Control[1]	0	0	0
Baseline	10	30	40
Addition of EDTA[2] (0.025 g/L)	0	40	0
Addition of EDTA[2] (0.05 g/L)	0	40	20
Addition of STS[3] (0.025 g/L)	20	30	10
Addition of STS[3] (0.05 g/L)	10	30	10
Solid phase extraction (C18)	10	0	10
pH 10 adjustment (filtered)	0	10	0
pH 10 adjustment (unfiltered)	10	0	0
Filtration (1 µm)	20	10	0

1 – Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR); 2 – Ethylenediaminetetraacetic acid; 3 – Sodium thiosulfate; 4 – Elutriate prepared by vigorously shaking the slag/laboratory water mixture by hand; 5 – Elutriate prepared by shaking the slag/laboratory water mixture on an industrial paint shaker.

[23] Attempts on elutriate samples prepared by shaking on an industrial paint shaker.

4.8 Toxicological Evaluation of Supernatant Preparation

Previous toxicity testing demonstrated that the slag/laboratory water supernatant produced by shaking the mixture on an industrial paint shaker was acutely toxic to *P. promelas*; however, the August 13, 2019 slag/laboratory water supernatant produced for TIE testing by vigorously shaking the mixture by hand was not acutely toxic to *P. promelas.* The slag used for the August 13, 2019 TIE testing was from the same source of slag collected on February 25, 2019 and used in all toxicological evaluations since the date of collection. The two methods for producing the supernatant (i.e., shaking by hand vs. shaking on an industrial paint shaker) were further evaluated for acute toxicity to *P. promelas*.

4.8.1 Sample Preparation

Approximately 4 liters of weathered slag[24] was placed into each of four 5-gallon buckets containing 2 gallons of laboratory water[25] obtained from AIC. Following lid placement on the buckets, 2 buckets were shaken on an industrial paint shaker for approximately 2 minutes and 2 buckets were vigorously shaken by hand for approximately 2 minutes. The sediment was allowed to settle for approximately 7 hours following shaking. After the contents of the buckets were allowed to settle, a peristaltic pump was used to pump 1 gallon of the supernatant from each of the 2 buckets shaken on an industrial paint shaker into a plastic container and 1 gallon of supernatant was pumped from each of the 2 buckets vigorously shaken by hand into a separate plastic container. The resulting elutriate samples were submitted to the laboratory for toxicity testing. Additionally, an aliquot of the laboratory water used to prepare the extracts was submitted to the laboratory to provide a water control.

[24] Onsite slag collected on February 25, 2019. The slag had long-term environmental exposure and had not been treated with dust control product.
[25] Water obtained from the *Pimephales promelas* rearing tanks at American Interplex Corporation (Little Rock, AR).

4.8.2 Toxicity Testing

The slag elutriates were tested for acute toxicity to *P. promelas* based on the USEPA (2002b) procedure; tested for acute toxicity as a 48-hr static non-renewal, 5-concentration dilution series with a dilution factor of 0.5 (resulting in test concentrations of 6.25, 12.5, 25, 50, and 100% sample). The aliquot of laboratory water used to prepare the extracts was tested at 100% for acute toxicity to *P. promelas* in a 48-hr static non-renewal test based on the USEPA (2002b) procedure. Table 30 provides acute toxicity results from the slag elutriate and laboratory water testing.

Similar to previous testing, the slag elutriate prepared by shaking on an industrial paint shaker was acutely toxic to *P. promelas* at 100% and the slag elutriate prepared by vigorously shaking by hand was not acutely toxic to *P. promelas*. Follow-up acute TIE testing using slag elutriate prepared by shaking on an industrial paint shaker is recommended for all future testing to characterize the toxicant(s) in the slag elutriate.

Table 30. 48-hour acute survival data for weathered slag elutriates submitted August 28, 2019.

Weathered Slag Elutriate				
Test	**% Survival (N=40)**			
	Slag Elutriate Prepared by Shaking on an Industrial Paint Shaker		**Slag Elutriate Prepared by Vigorously Shaking by Hand**	
Exposure (% Sample)	**24 hrs**	**48 hrs**	**24 hrs**	**48 hrs**
Control	100	100	100	100
6.25	100	100	100	100
12.5	100	100	100	100
25	100	100	100	100
50	90	90	100	100
100	0*	0*	80	70

Fish Culture Water		
	% Survival (N=40)	
Exposure (% Sample)	**24 hrs**	**48 hrs**
Control	100	100
100	100	100

*Significantly less than the control.

4.9 Chemical Composition of Onsite Slag

Previous measurements of inorganic ions in slag elutriate created by exposing laboratory water to slag were relatively low (see Section 4.6). In addition, acute toxicity does not appear to be persistent, i.e., is not observed in initially toxic sample after approximately 72 hours. The relatively low inorganic ion concentrations and lack of persistent toxicity suggest that an inorganic ion(s) is not the cause of the observed toxicity.

Weathered slag[26] was analyzed to determine the chemical composition in an effort to identify a potential cause of toxicity. Results of the chemical composition analysis suggest that slag is comprised of approximately 11% volatile substances (Table 31). The presence of 11% volatile substances in the slag further supports the theory that an inorganic ion(s) is not the cause of the observed toxicity.

Table 31. Chemical composition summary of weathered slag collected on February 25, 2019.

Analyte	Result (% weight unless otherwise noted)	Analyte	Result (% weight unless otherwise noted)
Total Solids	96	Strontium as SrO_2	0.03
Loss on Ignition	11	Sulfur	0.64
Aluminum as Al_2O_3	6.9	Thallium as Tl_2O	0.01
Antimony as Sb_2O_3	0.08	Titanium as TiO_2	0.16
Barium as B_2O_3	0.06	Vanadium as V_2O_4	0.07
Calcium as $CaCO_3$	57	Zinc as ZnO	0.33
Chromium as Cr_2O_3	0.79	Sulfur	0.58
Copper as CuO	0.34	Bromide (mg/Kg)	<2
Iron as Fe_2O_3	15	Chloride (mg/Kg)	7.6
Magnesium as MgO	9.5	Nitrate as N (mg/Kg)	<0.5
Manganese as MnO_2	2.4	Nitrite as N (mg/Kg)	<0.5
Phosphorus as P_2O_5	0.34	Sulfate (mg/Kg)	120
Silicon as SiO_2	9.3		

[26] Onsite slag collected on February 25, 2019. The slag had long-term environmental exposure and had not been treated with dust control product.

5.0 DISCUSSION

Due to violations of the Outfall 001 effluent limitations and WET testing from July 1, 2014, through July 31, 2017, Gerdau was requested to take corrective action and provide documentation, in the form of a final report stamped by a P.E. licensed in the State of Arkansas, that certifies Outfall 001 compliance with NPDES Permit No. AR0039730 effluent limitations and monitoring requirements. Corrective action began in October 2017 following DEQ's approval of Gerdau's CAP.

Beginning with implementation of the CAP through the present (approximately 27 months), Gerdau has exceeded its permitted lead limit during 2 reporting periods, its permitted copper limit during 2 reporting periods, and its WET limit during 3 reporting periods.

Due to the infrequent exceedances of effluent limitations for lead and copper, action could not be taken and the cause of the exceedances could not be identified; however, the cause of the infrequent exceedances may be the result of sample contamination during collection due to the process by which the sample is collected. Additionally (however less likely), the exceedances may be the result of sample contamination during analysis.

Infrequent episodes of effluent toxicity and loss of sample toxicity with storage has so far prevented identification of the toxicant(s) responsible for the WET failures. Attempts to evaluate the potential roles of dust control products and onsite slag material have so far been unsuccessful due to the difficulty in developing repeatable experimental protocols.

The toxicant(s) show the following properties which are consistent with an organic compound:

1. Effluent exposure to ultraviolet light, which degrades some organic compounds, has been shown to eliminate or reduce lethal and sub-lethal toxicity to *P. promelas* when compared to untreated effluent.
2. Chemical analysis of effluent and slag eluate observed to have toxic effects has indicated no toxicologically significant concentrations of metals or inorganic ions.
3. The lack of persistent toxicity in effluent and slag eluate suggests that an inorganic ion(s) is not the cause of observed toxicity.

4. Chemical composition of onsite slag identified the presence of 11% volatile substances, suggesting that an inorganic ion(s) is not the cause of the observed toxicity if slag is the source.

WET violations have become less frequent and the level of toxicity is reduced compared to historical toxicity; only sublethal effects have been observed in more recent testing compared to lethal effects in historical testing (Table 32). Therefore, intermittent toxicity will continue to hamper efforts to identify toxicants using a TIE-based approach during periods of toxicity. The reduction in frequency and level of toxicity (and no recent exceedances of COD) may be attributed to closer management of runoff from the roads during road watering activities during low-flow conditions, more judicious use of dust control product, complete containment of runoff from unweathered slag during storage, and more judicious reapplication of unweathered slag to onsite roads.

Table 32. Summary of Whole Effluent Toxicity testing at Outfall 001 pre- and post-Corrective Action Plan implementation.

Period	***Ceriodaphnia dubia***				
	# of WET Tests	**Lethal**		**Sub-lethal**	
		# Failed	**% Failed**	**# Failed**	**% Failed**
2014 Q1 - 2017 Q3	19*	1	5%	4	21%
2017 Q4 - 2019 Q4 (CAP Action Period)	11	0	0%	1	9%
Period	***Pimephales promelas***				
	# of WET Tests	**Lethal**		**Sub-lethal**	
		# Failed	**% Failed**	**# Failed**	**% Failed**
2014 Q1 - 2017 Q3	19	6	32%	12	63%
2017 Q4 - 2019 Q4 (CAP Action Period)	14	0	0%	3	21%

Evaluation of the current permitted treatment system for compliance within the requirements of NPDES Permit No. AR0039730 could not be completed since the source(s) of effluent violations for lead and copper and the source(s) or toxicant(s) causing WET testing violations could not be identified.

Gerdau's milestone schedule for submittal of a final report documenting compliance with effluent and WET limitations was December 31, 2019; however, due to the intermittency of WET failures and lack of toxicity persistence that prevent TIE follow-up, and inconclusive source evaluations, the milestone date of December 31, 2019 for a final report documenting compliance cannot be achieved. Gerdau will continue working with DEQ and taking action to identify sources, correct, and prevent the recurrence of effluent limitation and WET testing violations.

6.0 LITERATURE CITED

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USEPA. 2002b. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. EPA-821-R-02-012. Office of Water. Washington, DC.

ATTACHMENT 1

Evaluation of potential sources of COD in Outfall 001

FTN Associates Ltd.
water resources / environmental consultants
3 Innwood Circle, Suite 220 • Little Rock, AR 72211 • (501) 225-7779 • Fax (501) 225-6738

TECHNICAL MEMORANDUM

DATE: April 19, 2017

TO: **Lisa Overton**
Gerdau Special Steel North America

FROM: **Pat Downey** PDD
FTN Associates, Ltd. (FTN)

SUBJECT: Evaluation of potential sources of COD (chemical oxidation demand) in Outfall 001: Gerdau Special Steel North America (AR0039730)
FTN No. R06335-0009-003

1.0 INTRODUCTION

Gerdau Special Steel North America (AR0039730) discharges stormwater runoff, air conditioner condensate, and natural springs to an unnamed tributary of Massard Creek from its Fort Smith (Sebastian County) facility. Stormwater runoff from the facility enters a ditch that passes through the western, northern, and eastern portions of the facility (Figure 1). Stormwater and other drainage enters the upstream portion of the ditch from offsite (Location 9 in Figure 1). A number of pools are present in the ditch, which are formed by check dams and culverts for road crossings. During January through March of 2017, COD concentrations at Outfall 001 (Location 1 in Figure 1) consistently exceeded the daily maximum permit of 100 mg/L. During this time, Gerdau staff noted algal growths accumulating in some of the pools formed by the check dams. In March 2017, Gerdau contacted FTN to assist in evaluating potential causes and sources of the accumulated algal material and elevated COD measurements.

2.0 METHODS

On March 23, 2017, technical staff from FTN visited the Gerdau site to evaluate potential sources and causes of elevated COD. After an initial reconnaissance, an approach to the evaluation was to collect water samples and in situ measurements of temperature (^{o}C), dissolved oxygen ((DO); mg/L), pH (S.U.) and conductivity (μS/cm) at selected locations beginning at the downstream end of the ditch (Location 1 near Outfall 001; Figure 1) and proceeding upstream to the inflow from offsite (Location 9; Figure 1).

Sampling locations are shown in Figure 3. Grab samples and in situ measurements were taken at the locations 1, 2, 3, 4, 5, 6, 8, and 9; Only in situ measurements were taken at Location 7. In general, grab samples and in situ measurements were taken at the upstream edge of selected check dams and culverts based on the appearance of the water upstream or downstream of the structure. Water samples were collected as grab samples, labeled, placed immediately on ice and delivered to the laboratory (American Interplex Corp., 8600 Kanis Rd., Little Rock, AR 72202) for analysis of the parameters listed in Table 1.

Table 1 Analytes, analytical methods and detection limits (reporting limits) for chemical analyses of samples collected on March 23, 2017.

Analyte	Analytical Method	Detection Limit (Reporting Limit) (mg/L)
Total Kjeldahl Nitrogen (TKN)	EPA 351.2	1.0
Ammonia as N (NH_3)	SM 4500-NH3 G 1997	0.1
Nitrate + Nitrite as N (NO_3/NO_2)	EPA 300.0	0.5
Total organic carbon (TOC)	SM 5310 C 2000	5
Dissolved organic carbon (DOC)	SM 5310 C 2000	5
Total phosphorus (TP)	EPA 200.0	0.02
Chemical oxygen demand (COD)	HACH 8000	10

3.0 RESULTS AND DISCUSSION

Average measured flow rate from Outfall 001 during the day of sampling was 20 gpm. During the site visit it was noticed that a water truck was applying water to the haul roads for dust control and that some of this water was draining into the ditch at Location 3 at an estimated flow rate of 0.5 to 1 gal /min. A sample of water that was draining into the ditch from the haul road at Location 3 was collected. Floating and submerged algal growth was observed in the pools associated with Locations 5 and 6 of the ditch (Figure 4).

Results of chemical analyses and in situ measurements are provided in Table 2. Analyte concentrations in the inflow from the road wash (Location 3) were substantially higher than background (Location 9) concentrations. Location 3 concentrations were over 300 times background COD, 100 times background TOC/DOC, 10 times background TP and 3 times background TN. Gerdau staff present at the time of sampling indicated that the runoff to the ditch, similar to that observed at Location 3, is a common feature of daily operations. Evidence of runoff inflow from the roads to the ditch could be seen on the surface of the ground near the ditch at other locations. If analyte concentrations observed at Location 3 are typical and these types of inflows are common, then it is likely that runoff from the road to the ditch has a significant impact on the water quality of the ditch.

Nutrient inputs from sources similar to Location 3 would be sufficient to support high algal biomass and high levels of primary productivity. However, nutrient concentrations (TP, TN and NH_3) at the ditch locations were generally similar to background. This similarity suggests that the nutrient input is rapidly assimilated into algal biomass. Accordingly, the heavy growth of algae observed in the ditch is likely the result of a combination of high nutrient input from dust control activities and low current velocity in the ditch that allows the algal growth to accumulate.

In contrast, all COD values from the ditch locations were generally 20 to 50 times background and TOC (most of which is dissolved) was generally 10 to 30 x background. This result is consistent with an organic carbon source that is not quickly assimilated into biomass, remains dissolved in the water column and is expressed as COD. A strong correlation between DOC and COD (Figure 2) supports this conclusion.

4.0 CONCLUSIONS AND RECOMMENDATIONS

Runoff into the ditch from road surfaces due to dust control activities during low flow conditions is a likely cause of COD concentrations exceeding permit limits and accumulation of algal growth. High COD levels are the likely result of a combination of DOC that is solubilized from the material applied to settle dust at the site and algal biomass due to the input of nutrients. Closer management of runoff from the roads during road watering activities during low-flow conditions might lessen the input of this high-COD water to the ditch. An alternative to an organic dust control agent should be considered.

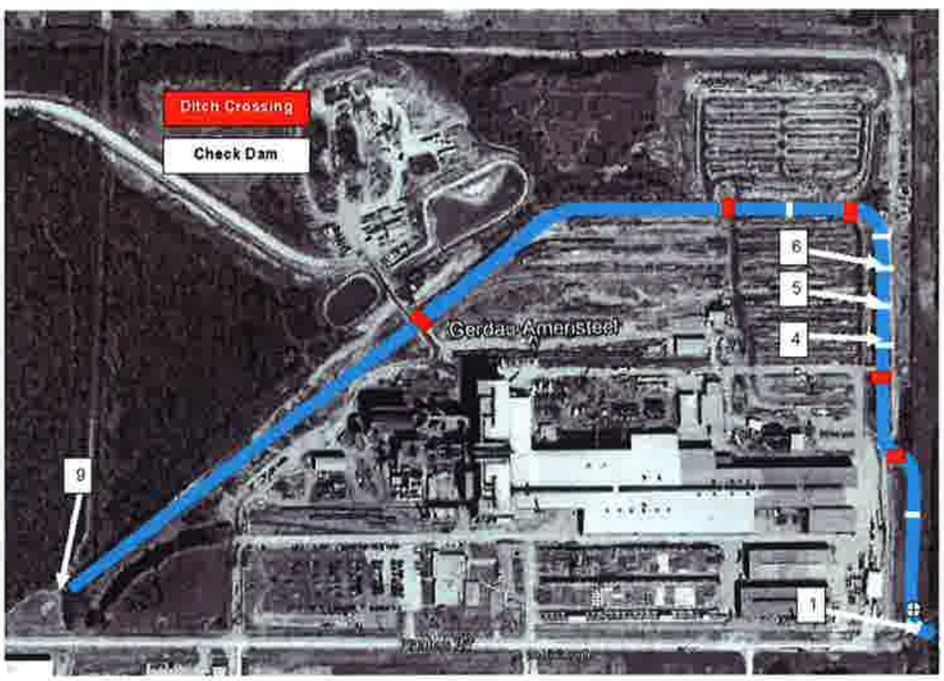

Figure 1 Aerial view of the Gerdau facility showing the locations of the check dams and ditch crossings.

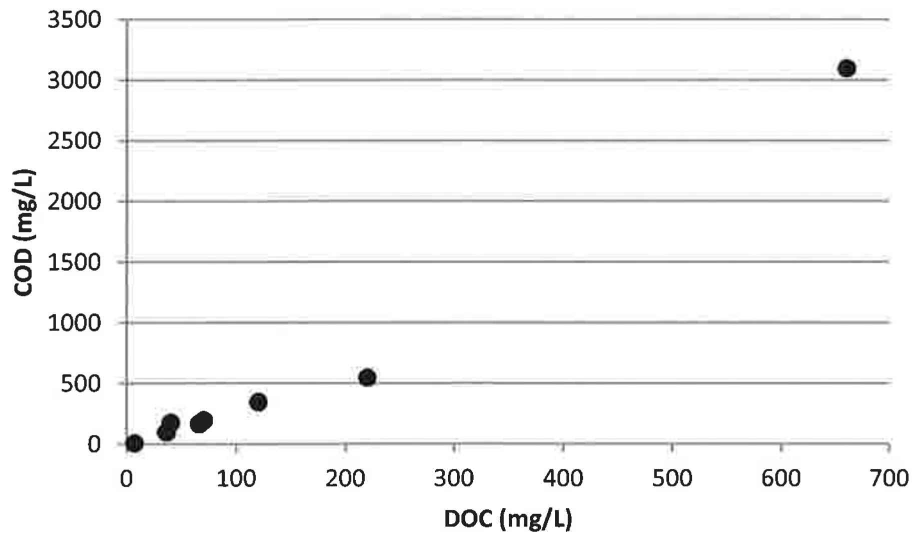

Figure 2 Relationship between COD and DOC at ditch locations samples on March 23, 2017.

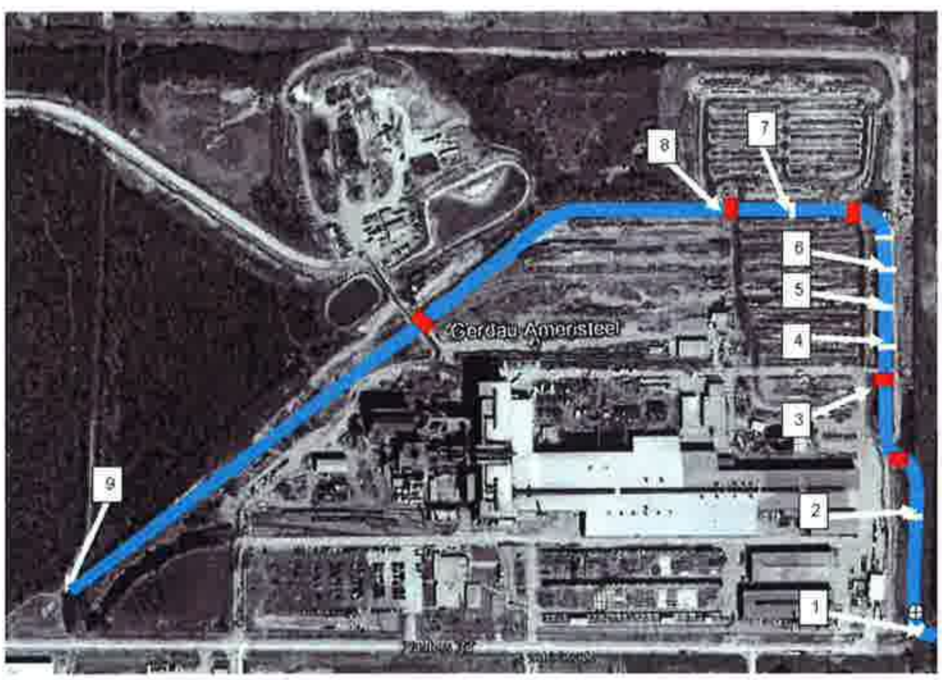

Figure 3. Locations of grab sample collection and in situ measurements.

Figure 4 Photographs of algal growth present at Location 5 and 6 (left and right photograph, respectively).

Table 2 Results of chemical analyses and in situ measurements from March 23 source evaluation.

Analyte*	Location								
	1	2	3	4	5	6	7	8	9
COD	180	200	3100	350	550	170	NS	100	< 10
TOC	77	76	840	130	220	66	NS	41	7.2
DOC	40	70	660	120	220	66	NS	36	7.0
TOC as DOC (%)	52	92	79	92	100	100	NS	88	97
TP	0.042	0.036	5.6	0.02	0.063	0.09	NS	0.052	0.051
TKN	2.0	2.5	7.9	4.7	2.3	2.8	NS	1.9	2.3
NO_3/NO_2	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	NS	< 0.5	< 0.5
Total N (TKN + NO_3/NO_2)	2.0	2.5	7.9	4.7	2.3	2.8	NS	1.9	2.3
NH_3	0.42	0.63	2.5	1.0	0.2	0.31	NS	0.16	0.22
Temperature	14.6	14.8	20.5	15.0	16.1	17.1	16.6	17.1	16.7
DO	4.6	7.8	7.7	5.8	3.0	7.6	4.4	4.0	8.1
pH (S.U.)	7.9	7.7	11.9	11.8	8.9	8	8.7	8.2	7.9
Conductivity (µS/cm)	708	720	2227	910	992	708	679	702	737

* All units as mg/L unless otherwise noted. NS - no sample.

We appreciate the opportunity to work with you on this project. If you have any questions or comments regarding this Technical Memorandum, please do not hesitate to call me or Rex Robbins PE at (501) 225-7779.

PJD/ack

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ATTACHMENT 2

Evaluation of Potential Pathogen Interference in Routine Biomonitoring Tests at Outfall 001

FTN Associates Ltd.
water resources / environmental consultants
3 Innwood Circle, Suite 220 • Little Rock, AR 72211-2449 • (501) 225-7779 • Fax (501) 225-6738

TECHNICAL MEMORANDUM

DATE: May 24, 2017

TO: **Lisa Overton**
Gerdau Special Steel North America

FROM: **Pat Downey** PJD
FTN Associates, Ltd. (FTN)

SUBJECT: Evaluation of Potential Pathogen Interference in Routine Biomonitoring Tests at Outfall 001: Gerdau Special Steel North America (AR0039730)
FTN No. R06335-0009-003

1.0 SUMMARY AND CONCLUSION

Based on the analysis routine biomonitoring, concurrent testing of samples treated with ultraviolet (UV) light and other manipulations (ultra-filtration and alternate test protocols) pathogen interference (PI) may, at times, account for the results of FHM chronic tests at Outfall 001. The strongest evidence in favor of PI as a factor causing toxicity in FHM chronic tests is the occurrence of non-monotonic dose responses, the presence of high variability among test replicates, the effects of sample manipulations such as UV-treatment and ultra-filtration, and the effect of the use of the modified test protocol. However, the lack of seasonality, effects on biomass, inconsistent timing of the onset of mortality and the presence of lethal and/or sub-lethal effects on *C. dubia* suggest the presence of toxicants in addition to PI.

2.0 INTRODUCTION

This document provides an evaluation of biomonitoring conducted at Outfall 001 of Gerdau Special Steel (AR0039730) to assess the occurrence of PI in episodes of toxicity in whole effluent toxicity (WET) test failures conducted as part of routine quarterly monitoring. PI [also referred to as "sporadic mortality" in USEPA (2002)] in 7-day fathead minnow tests as described in Downey et. al. (2000) and recognized in USEPA (2002) is indicated by the following symptoms:

1. Non-monotonic dose response. Rather than decreasing with increasing effluent concentration, survival (when a toxic response occurs) is often (but not always) higher or similar at lower concentration.
2. High variation in survival among replicates within test concentrations. Percent survival at the end of 7 days can range from 0 to 100% among replicates within a single concentration.

3. Sample manipulations such as heating, ultrafiltration (0.2 µm pore size), exposure to UV light and addition of antibiotics that are intended to kill or remove bacteria typically remove PI.

4. Test protocols that minimize contact among fish (e.g., replicates with 1 or 2 fish) often reduce or eliminate PI.

5. Effects on growth but not biomass. The sporadic mortality described in Item 1 above can cause apparent sub-lethal effects on fish growth (as opposed to biomass) because the FHM test protocol (EPA Method 1000.0; USEPA, 2002) calculates the average "growth" endpoint using the final total weight of fish in the replicate divided by the number of fish that were initially present. As a result, the growth endpoint combines effects on survival as well as fish size. Accordingly, PI can result in a statistically significant reduction in the growth end point even if the surviving fish are not smaller than the controls. In contrast, average "biomass" which is based on the number of surviving fish will show no effects in the presence of PI.

6. Timing of the appearance of the effect during the test. Mortality in the test containers generally first occurs between day 3 and 5 of the test.

7. Distinct seasonality. PI is almost completely limited to the colder months and rarely, if ever, occurs during the summer.

8. Fungal growth on dead and dying fish. These growths are not always observed.

9. No effects on survival or reproduction of *Ceriodaphnia dubia.* When a sample is tested using FHM and *C. dubia* in concurrent tests, PI (as described herein) is never seen in the *C. dubia* test even when there is a strong indication of PI in FHM test.

Based on the types of sample manipulation that control PI (i.e., treatments such a sterilization and filtration that would be expected to remove or kill bacteria) it is likely that PI is due to water borne pathogens and should be viewed as a form of interference rather than toxicity. Documentation of PI as the exclusive cause of WET failures in FHM justifies the use of appropriate sample manipulations of modified test protocols in routine testing to eliminate biomonitoring failures due to this interference.

3.0 APPROACH TO THIS EVALUATION

This evaluation includes routine and special tests conducted from January 2014 through March 2017 and focuses on the following three types of data generated during that period:

1. Routine quarterly chronic WET tests using FHM and *C. dubia*.

2. Concurrent tests on FHM using untreated sample and sample exposed to UV light. These comparisons were usually conducted in conjunction with routine quarterly tests.

3. Evaluation of other PI control methods such as ultrafiltration and modified test protocols. These tests were conducted as separate studies apart from routine monitoring.

These test results were evaluated based on characteristics of PI given in the previous section. Historical WET data from Outfall 001 has indicated only slight potential PI effects that nonetheless could have caused the observed indications of the lethal and sublethal toxicity to FHM. Because none of the symptoms given above has been particularly strong in any particular test, this evaluation must use a "weight of evidence" approach. Accordingly, each of the characteristics listed in the preceding section will be evaluated in the available data to assess the evidence as a whole for the role of PI in biomonitoring failures using FHM.

4.0 SUMMARY OF AVAILABLE DATA AND TEST RESULTS

4.1 Routine Biomonitoring and UV Treatments

As mentioned above, during January 2014 through March 2017 routine quarterly biomonitoring often included concurrent testing of the FHM using untreated sample and sample exposed to UV light. Results of these tests and some of their characteristics are summarized in Tables 1 (tests without concurrent tests on UV-treated sample) and 2 (test with concurrent tests on UV-treated sample). These tables provide results of all biomonitoring tests including results of *C. dubia* survival and reproduction tests.

Inspection of Table 1 indicates the following:

1. Twelve of the 16 chronic FHM test conducted during this period showed lethal or sublethal toxicity to FHM.
2. Of the five tests showing lethal toxicity (Test numbers 2, 3, 5, 12 and18), three (test numbers 5, 12 and 18; Figure 1) showed high variability and/or non-monotonic dose response.
3. Six of the 16 tests showed high variability in survival among replicates within exposures (Figures 1 and 2).[1] Note that not all tests with high variability in survival among replicates showed significant lethal toxicity.
4. Seven of the ten tests that showed statistically significant growth effects also showed significant effects on biomass. That is, in these seven tests there was an actual reduction in the size of the fish.

[1] There is no standard criterion for deciding when variation among replicates is "high". This determination was based on professional judgment and the response curves of those test are provided.

Table 1. Summary of chronic toxicity tests using *Pimephales promelas* on untreated Outfall 001 samples.

Test Number	Sampling Date	*P. promelas*						*C. dubia*	
		Lethal	Sublethal		Onset of mortality (day of test)	High variability among replicates?**	Survival response curve monotonic?	Lethal	Sublethal
			Growth	Biomass					
1	1/19/2014	P	F	F	7	N	Y	P	P
2	6/15/2014	F	NA*	NA	4	N	Y	F	F
3	7/20/2014	F	NA*	NA	2	N	Y	P	P
4	9/14/2014	P	F	F	6	Y	Y	P	F
5	10/18/2014	F	F	F	5	Y	N	P	P
6	11/30/2014	P	F	F	3	N	Y	P	P
7	4/20/2015	P	F	F	5	N	Y	P	F
8	6/6/2015	P	F	P	1	N	N	P	P
9	8/23/2015	P	P	P	NA	N	Y	NT	NT
10	11/8/2015	NT	NT	NA	NA	NA	NA	P	P
11	12/6/2015	P	F	F	6	Y	Y	P	P
12	1/31/2016	F	F	P	4	N	Y	P	P
13	5/3/2016	P	P	P	NA	N	Y	P	P
14	7/31/2016	P	P	P	NA	N	N	NT	NT
15	9/22/2016	NT	NT	NA	NA	NA	NA	F	F
16	10/23/2016	P	P	P	NA	N	Y	P	P
17	11/27/2016	NT	NT	NA	NA	NA	NA	P	F
18	2/2/2017	F	F	P	3	N	N	P	P
19	3/11/2017	P	F	F	6	Y	Y	P	P

P - No statistically significant ($P < 0.05$) differences between endpoint values (survival, growth, biomass) in exposures and control; NA - Not applicable; F - Statistically significant ($P < 0.05$) differences between endpoint values (survival, growth, biomass) in exposures and control; NT - No test; Y - Yes; N - No; * 100 % mortality in all test exposures; ** having one exposure in which survival ranged from 62.5 to 100% (i.e., 5 to 8 surviving fish)

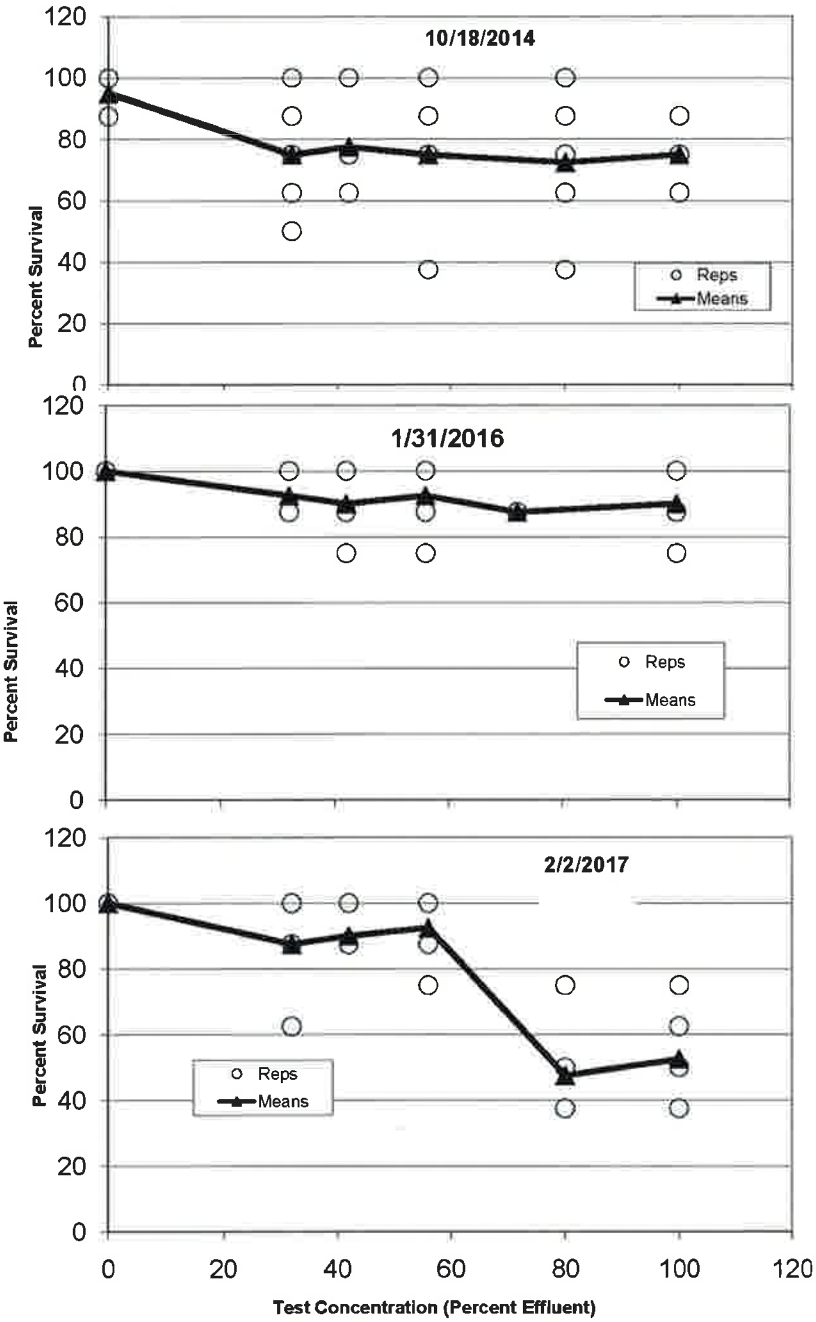

Figure 1. Fathead minnow chronic tests on samples collected October 18, 2014, January 31, 2016 and February 2, 2017 showing non-monotonic dose response and high variability among replicates.

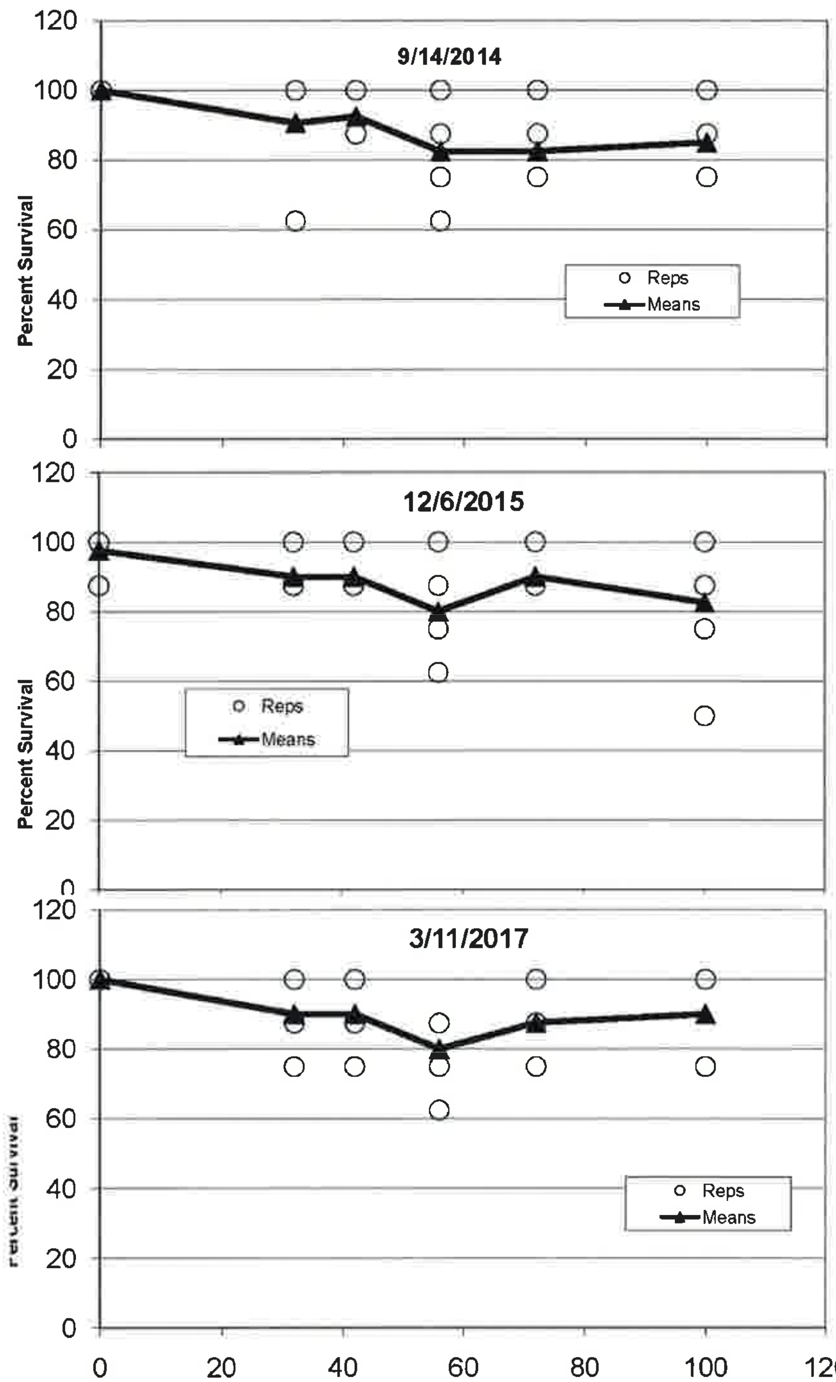

Figure 2. Fathead minnow chronic tests on samples collected September 14, 2014, December 6, 2015 and March 11, 2017 showing high variability among replicates.

Table 2, which summarizes results of the ten concurrent tests on untreated and UV-treated sample, indicates that:

1. UV treatment completely removed lethal effects in the two samples showing lethal toxicity in untreated sample (test number 12 and 18; Table 1).
2. UV treatment removed sublethal effects (reduced growth) in four of the six tests that showed sublethal toxicity in untreated sample.
3. UV treatment increased the level of sublethal toxicity (biomass effect) in the sample collected 3/11/2017. During this test the laboratory reported an orange film of material that floated to the surface of the sample after UV treatment (Figure 3).

Table 2. Comparison of chronic toxicity tests using *Pimephales promelas* with untreated vs UV-treated Outfall 001 samples.

Sampling Date	Test Number (from Table 1)	Untreated		UV Treated	
		Survival	Growth	Survival	Growth
9/14/2014	4	P	F	P	P
11/30/2014	6	P	F	P	P
6/6/2015	8	P	F	P	P
8/23/2015	9	P	P	P	P
12/6/2015	11	P	F	P	P
1/31/2016	12	F	F	P	F
5/3/2016	13	P	P	P	P
10/23/2016	16	P	P	P	P
2/2/2017	18	F	F	P	F
3/11/2017	19	P	P	P	F

P - No statistically significant ($P > 0.05$) differences between endpoint values (survival, growth, biomass) in exposures and control; F - Statistically significant ($P < 0.05$) differences between endpoint values (survival, growth, biomass) in exposures and control; Y - Yes; N - No

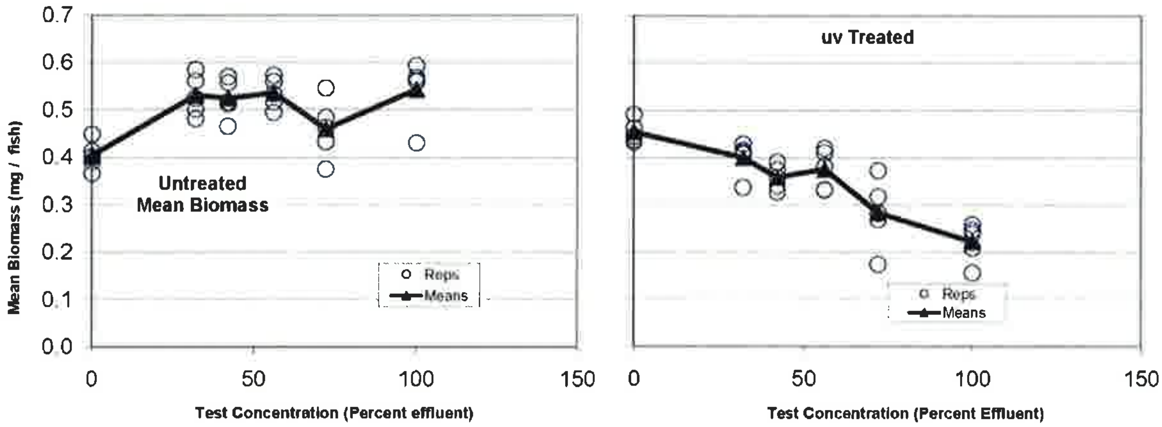

Figure 3. Fathead minnow chronic tests showing increased effect on biomass due to UV treatment in the sample collected on March 11, 2017.

4.2 Other PI Control Methods

Other available methods of PI control include filtration through a 0.1 µm pore-size membrane filter and a modified test protocol described in USEPA (2002). Filtration through a 0.1 µm pore-size membrane filter removes bacteria which are the presumed cause of pathogen interference. The modified test protocol involves the use of replicates with 2 fish per test beaker instead of the usual 8 or 10. Since PI tends to have an "all or none" effect in test beakers, reducing the number of fish per beaker can reduce the impact of PI on the test when it occurs. These 2 methods of PI control were evaluated in samples collected on July 20-24, 2015 (modified protocol) and January 11, 2016 (filtration).

4.2.1 Filtration

For this comparison separate aliquots of a single grab sample collected January 11, 2016 were treated with UV exposure and by filtration through 0.2 µm pore-size membrane filters. Table 3 and Figure 4 compare the effects of filtration and UV treatment. There were statistically significant effects on survival and growth on the test using the untreated sample. Both the UV and filtration treatment removed lethal (survival effects) and sublethal (growth effects) toxicity.

Table 3. Results of toxicity tests on *Pimephales promelas* untreated, filtered (0.1 µm pore-size) and UV-treated sample.

Sample Treatment	Percent Sample	Percent Survival	Growth (mg/fish)		Biomass (mg/fish)	
			Mean	CV	Mean	CV
Untreated	0	100	0.297	7.1	0.297	7.1
	25	92.5	0.273	9.0	0.297	10.5
	50	92.5	0.249*	6.8	0.273	12.4
	100	77.5*	0.229*	12.0	0.296	14.3
UV-treated	0	100	0.297	7.1	0.297	7.1
	25	100	0.316	4.2	0.316	5.5
	50	100	0.301	6.0	0.301	8.2
	100	100	0.289	6.8	0.289	13.0
Filtered	0	100	0.297	7.1	0.297	7.1
	25	100	0.305	4.3	0.305	4.7
	50	100	0.316	3.2	0.316	5.6
	100	100	0.313	3.3	0.313	6.6

* Statistically less than the control (P < 0.05); CV - coefficient of variation

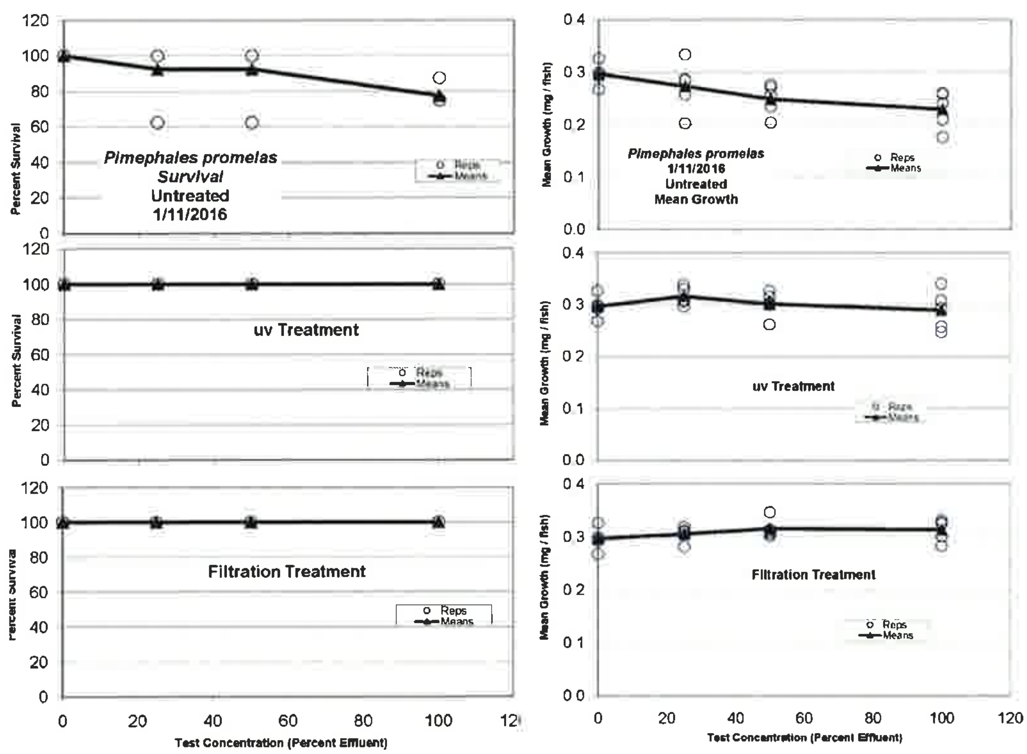

Figure 4. Comparison of survival and growth endpoints in untreated, UV-treated and filtered grab sample collected January 11, 2016.

4.2.2 Modified Test Protocol

Untreated sample collected during July 20 - 24, 2015 was tested using the normal chronic test protocol, (Method 1000.0, USEPA, 2002) and the modified protocols described in Section 11.3 of USEPA (2002). An additional aliquot of samples treated with UV exposure was tested concurrently. Results of the these tests (Table 4 and Figure 5) indicate slight, but statistically significant, effects on survival and growth in the untreated sample which were not apparent in the test using UV-treated sample and the modified protocol. Although UV-treatment in this test series removed statistically significant growth effects, there remained high variability among replicates (Especially at the 25% test concentration) and a suggestion of a sublethal effect. These effects and their occurrences in a sample collected during the summer suggest factors other than PI at play.

Table 4. Results of toxicity tests on *Pimephales promelas* untreated, UV-treated sample and untreated sample using the modified protocol.

Sample Treatment	Percent Sample	Percent Survival	Growth (mg/fish)		Biomass (mg/fish)	
			Mean	CV	Mean	CV
Untreated	0	97.5	0.374	3.9	0.384	7.3
	25	85.0	0.343	5.1	0.412	19.5
	50	85.0	0.288*	9.0	0.339	2.5
	100	82.5*	0.296*	12.7	0.357	6.7
UV-treated	0	100.0	0.326	11.4	0.326	11.4
	25	90.0	0.293	15.3	0.325	24.6
	50	97.5	0.275	16.4	0.281	6.3
	100	90.0	0.276	14.5	0.307	11.2
Modified Protocol	0	87.5	0.261	6.3	0.299	5.5
	25	92.5	0.262	4.6	0.285	8.8
	50	87.5	0.257	5.0	0.296	9.8
	100	87.5	0.252	3.9	0.290	9.0

* Statistically less than the control ($P < 0.05$); CV - coefficient of variation

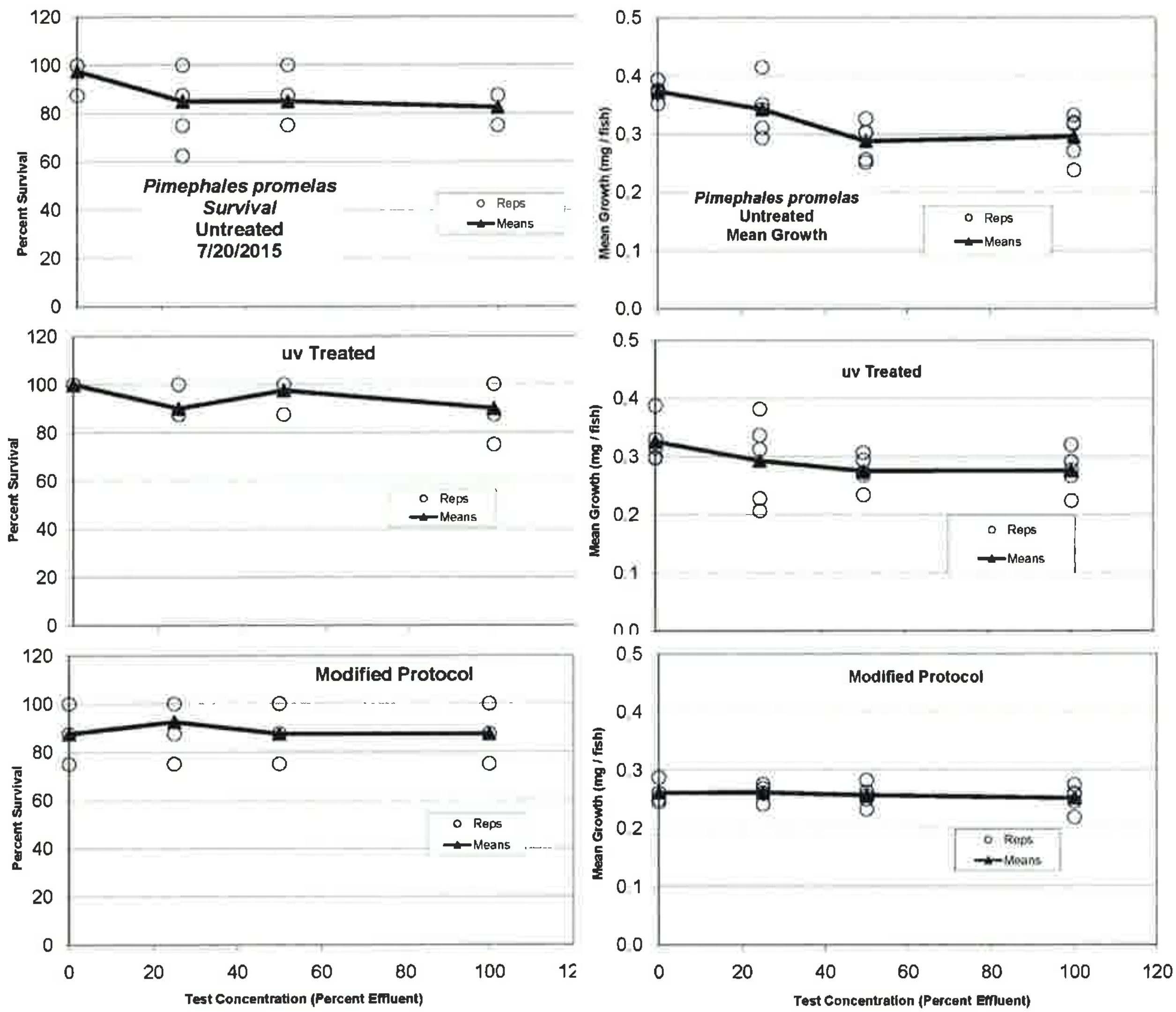

Figure 5. Comparison of survival and growth endpoints in untreated, UV-treated and modified protocol test conducted on samples collected July 20 - 24, 2015.

5.0 EVAUATION OF PI CHARACTERISTICS

This section evaluates the test data presented in Section 3 with respect to the symptoms of PI presented in Section 1.

5.1 Non-monotonic dose response

Of the four chronic FHM tests showing lethal toxicity, three (test numbers 5, 12 and 18; Table 1) showed non-monotonic dose responses (Figure 1). The response curve of the fouth test showing lethal toxicity, conducted on sample collected 7/20/2015 (Test number 3, Table 1), was not strongly monotonic (Figure 4). These observations are consistent with toxicity due to PI.

5.2 High Variation in Survival among Replicates

Based on general experience with normal variability in FHM tests five tests showed high variability in survival among replicates within exposures. Dose response relationships for all 5 tests are shown on Figures 1 and 2. Not all tests with high variability in survival among replicates showed significant lethal toxicity (Test numbers 4, 11 and 19: Table 1). Similarly, not all tests showing significant lethal toxicity showed high variability (test numbers 2 and 3; Table 1). Of the five tests showing lethal toxicity (Test numbers 2, 3, 5, 12 and18), three (test numbers 5, 12 and 18) showed high variability and/or non-monotonic dose response. These observations are consistent with toxicity due to PI in at least some of the five episodes of lethal toxicity to FHM.

5.3 Toxicity Removal/reduction by UV Light and Filtration

5.3.1 Treatment with UV Light

A total of ten paired UV treated vs untreated tests on FHM were conducted during January 2014 through March 2017. Table 2, which summarizes results of the 10 concurrent tests on untreated and UV-treated sample, indicates that:

1. UV treatment completely removed lethal effects in the two samples showing lethal toxicity in untreated sample (test number 12 and 18; Table 1).
2. UV treatment removed sublethal effects (reduced growth) in four of the six tests that showed sublethal toxicity in untreated sample.
3. UV treatment increased the level of sublethal toxicity (biomass effect) in the sample collected 3/11/2017. During this test the laboratory reported an orange film of material that floated to the surface of the sample after UV treatment (Figure 3).

Although only two of ten paired UV-treated/untreated tests showed significant lethal toxicity there often appeared to be a low level of lethal effects present with some significant sub-lethal effects. Inspection of the results suggested that UV-treatment also consistently removed statistically no-significant low-level effects. To evaluate this apparent tendency, differences in % survival and growth and their respective CVs (coefficients of variation) between test concentrations in UV treated and untreated tests were evaluated statistically. Results of this analysis (Table 5) indicate that average differences in % survival, CVs for % survival and CVs for fish weight (growth) among replicates in tests using UV-treated vs untreated samples were small but significantly different from zero. In all cases except for the sample collected on 3/11/2017 there was improved survival and growth or reduced variability among replicates after UV-treatment. Differences in these parameters between UV-treated and untreated samples provide the strongest "signal" of potential PI in the entire data set.

These observations are consistent with toxicity due to PI.

5.3.2 Filtration

Filtration treatment removed lethal (survival effects) and sublethal (growth effects) toxicity observed with sample collected January 11, 2016 (Table 3 and Figure 3).

These observations are consistent with toxicity due to PI.

5.4 Modified Test Protocol

The modified test protocol (Section 11.3 of USEPA, 2002) used on samples collected July 20-24, 2015 removed the slight but statistically significant effects on survival and growth (Table 4 and Figure 4).

These observations are consistent with toxicity due to PI.

Table 5. Summary of statistical evaluation of differences in % survival and growth and their respective CVs (coefficients of variation) between test concentrations in chronic FHM test with UV treated and untreated sample.

Test Statistic	% Survival Treated-untreated	Survival CV Untreated-treated	Mean Growth (mg/fish) Treated-untreated	Growth CV Untreated-treated
Average difference	6.82	7.42	0.012	3.24
Standard Deviation	9.2	8.25	0.0954	7.72
Calculated t	5.55	6.73	1.01	3.12
P (2-tailed; df = 55)*	< 0.0001	< 0.0001	0.32	0.003

* H_o: Average difference =0

5.5 Sub-lethal Effects

Due to the method of calculation, PI can affect the growth endpoint but not the biomass endpoint. As noted in Section 3.1, seven of the ten tests that showed a statistically significant growth effect also showed a significant effect on biomass. That is, in these seven tests there was an actual reduction in the size of the fish.

This observation is not consistent with toxicity due to PI.

5.6 Timing of Mortality

The onset of mortality was between day three and five in 6 of the 12 tests in which some level of mortality was observed (Table 1). Of the 5 tests in which there was statistically significant mortality the onset of mortality was between day three and five in 5 tests.

This result is somewhat consistent with toxicity due to PI.

5.7 Seasonality

PI is almost completely limited to the colder months and rarely, if ever, occurs during the summer. As noted in Section 4.3.1, differences in % survival, survival CV and biomass CV between UV-treated and untreated samples provide the strongest "signal" of potential PI in this data set. Seasonality of potential PI effects was evaluated by analyzing differences in this signal from warm (May - Oct) v cold (Nov - April) months of the year. Average differences are summarized in Table 6. There were no statistically significant differences (Mann-Whitney Z-test) in % survival, survival CV and biomass CV between UV-treated and untreated in warm vs cold months.

This result is strongly incompatible with toxicity due to PI.

Table 6. Average difference between corresponding test dilutions in UV-treated and untreated sample in warm vs cold months.

Summary Statistic	% Survival		% CV - Survival		% CV- Growth	
	Cold	Warm	Cold	Warm	Cold	Warm
Average Difference	8.8	4.9	7.5	7.4	5.0	1.5
Mann-Whitney Z-statistic	1.22		0.51		1.47	
P value (two-tailed)	0.22		0.61		0.14	

5.8 Fungal Growth on Dead and Dying fish

There were no mentions in the laboratory reports of fungal growths on dead and dying fish. This result is not incompatible with PI because these growths are not always noted and the level of PI-like effects in the tests was typically low.

5.9 Effects on *Ceriodaphnia dubia*

The presence of lethal and/or sub-lethal effects in tests using *C. dubia* suggest that factors in addition to PI might be present.

5.10 Summary of PI Characteristics

PI characteristics of test data presented in Section 3 with respect to the symptoms of PI (Section 1) are summarized in Table 7. The strongest evidence in favor of PI as a factor causing toxicity in FHM chronic tests is the occurrence of non-monotonic dose responses, the presence of high variability among test replicates, the effects of sample manipulations such as UV-treatment and ultra-filtration, and the effect of the use of the modified test protocol. However, although PI may, at times, account for the results of FHM chronic tests, the lack of seasonality, effects on biomass, inconsistent timing of the onset of mortality and the presence of lethal and/or sub-lethal effects on *C. dubia* strongly suggest the presence of toxicants not attributable to PI.

Table 7. Summary of PI symptoms from FHM testing.

Symptom	Result from Testing
Non-monitonic dose response	Consistent with PI
High variability among replicates	Consistent in some tests
Effect of manipulation (UV, filtration)	Consistent with PI
Effect of modified protocol	Consistent with PI
Sub-lethal effects	Not consistent with PI
Timing of mortality	Consistent in some tests
Seasonality	Not consistent with PI
Presence of fungal growth	Indeterminate
Effects on *C. dubia*	Suggests presence of other toxicants

We appreciate the opportunity to work with you on this project. If you have any questions or comments regarding this project, please do not hesitate to contact me or Rex Robbins, PE, at (501) 225-7779

PJD/tas

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6.0 LITERATURE CITED

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