

# **Water Effects Ratio and Dissolved Translator Study**

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Prepared for:

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# 1.0 BACKGROUND/APPROACH

The Broken Bow Public Works Authority was issued a renewed OPDES permit (OPDES Permit No. OK0021521) that became effective on September 1, 2010. The permit allows for discharge of treated municipal and industrial wastewater from the city's wastewater treatment system (Facility I.D. No. S10203) to an unnamed tributary of Yanubbe Creek. Among the permit's provisions are limits for the priority pollutants copper, lead, and zinc. The numerical limits for these metals are as follows:

Table 1. Broken Bow OPDES Permit No. OK0021521 permit limits.

Parameter	Concentration Limits	
	Monthly Average (µg/L)	Daily Maximum (µg/L)
Copper, Total Recoverable	3.84	9.47
Lead, Total Recoverable	0.71	1.23
Zinc, Total Recoverable	38.27	66.30

Permit limits for the metals were calculated using the aquatic life criteria of the Oklahoma Water Quality Standards. A water effect ratio of 1.0 was assumed for purposes of the calculations.

Biomonitoring is another required provision of the Broken Bow OPDES permit. The permit specifies that the 7-day chronic *Pimephales promelas* test and the 7-day chronic *Ceriodaphnia dubia* test be completed quarterly. Both lethal and sub-lethal endpoints are reported. The critical (effluent) dilution required in the tests is 100%.

Since the issuance of the current permit, the facility has not consistently achieved concentration limits for metals. Based on these permit excursions, a request to perform a water effects ratio (WER) study to develop site-specific metals criteria was submitted to the Oklahoma Department of Environmental Quality on February 23, 2012. A letter from the agency dated May 29, 2012 granted permission to perform the study. Broken Bow Public Works Authority then submitted a workplan for total and dissolved copper, lead, and zinc WER studies. The workplan was reviewed by the Oklahoma Water Resources Board (OWRB) and EPA and approved via letter on February 11, 2013.

As stated in the workplan, the WER study was conducted using methodology available from the Oklahoma Water Resources Board (OWRB) and the EPA. The OWRB "Guidance Document for the Development of Site-Specific Water Quality Criteria for Metals" (OWRB 2003) provides procedural information for developing site-specific criteria within the State of Oklahoma. This document was used in conjunction with EPA methodology to develop an Oklahoma-defined final WER (fWER) which includes both a water effect ratio (WER) and a total to dissolved translator (f). The fWER is utilized to calculate a final criterion translator (T), which is used to develop site-specific total

recoverable criterion and revise permit limits based on site-specific information while allowing for adequate protection of aquatic life in the receiving stream.

## **2.0 WATER EFFECTS RATIO PROCESS**

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A number of physical and chemical factors in effluent and effluent/receiving stream mixtures affect the toxicity of metals to aquatic life. In most instances, increases in substances such as hardness, total organic carbon, and total suspended solids greatly increase the concentration of metals required to produce a toxic endpoint, such as an LC50. Because of this, there are typically differences between the toxicity of metals in laboratory water compared with site water (effluent). The process used to account for the difference in the toxicity of a metal in laboratory water with that of site water is termed a water effect ratio or WER. A WER is performed by conducting side-by-side toxicity tests using laboratory water and site water. The difference between the two is the "water effect." After the determination of a WER, this information should be used to adjust the instream criteria for the metal (copper, lead, zinc) in accordance with state water quality standards. In turn, the adjusted criteria will then be used for OPDES permitting purposes associated with the Broken Bow Public Works Authority.

Guidance for conducting a WER study is presented by EPA in a document titled "Interim Guidance on Determination and Use of Water-Effect Ratios for Metals" (EPA-823-B-94-001, February 1994). For copper WER studies, more recent guidance is presented in "Streamlined Water-Effect Ratio Procedure for Discharges of Copper" (EPA-822-R-01-005, March 2001). This EPA methodology was utilized in conjunction with OWRB guidance presented in "Guidance Document for the Development of Site-Specific Water Quality Criteria for Metals" (OWRB 2003). For the lead and zinc WER study the OWRB and EPA Interim guidance were utilized. For the copper WER study the OWRB and streamlined method were principally followed.

The OWRB guidance provides three options for conducting studies for the purpose of site-specific criterion development. Option one is the development of a WER for the basis of amending criteria. Option two is the development of a dissolved translator used to translate the applicable statewide total criteria into a site-specific dissolved criteria through the use of a dissolved to total metal ratio. Option three is a combination of the WER and the dissolved translator. This study includes the combined use of a WER and a dissolved translator (Option 3).

### 3.0 SITE DESCRIPTION/DESIGN FLOWS

The Broken Bow Public Works Authority discharges to an unnamed tributary of Yanubbe Creek via Outfall 001, which is located at Latitude 34° 01' 37.165", Longitude 94° 43' 22.270". The receiving stream travels from the unnamed tributary to Yanubbe Creek which enters Stream Segment 410200 of the Lower Red River Basin. The Outfall 001 discharge location is shown in Figure 1.

The Statement of Basis for the NPDES permit currently in effect was reviewed for discharge and receiving stream design flow characteristics. For Outfall 001 the design flow used as the basis for calculation of permit limits including copper, lead, zinc whole effluent toxicity, and others was 1.0 mgd, (1.547 cfs). The upstream flow rate of Yanubbe Creek used in calculation of the permit limits was the default value of 1 cfs. Aquatic life criteria (total) for copper, lead, and zinc for Yanubbe Creek, based on a site-specific average hardness concentration of 34.9 mg/L are shown below.

Table 2. Unnamed tributary of Yanubbe Creek aquatic toxicity criteria.

Parameter	Hardness Dependent Aquatic Toxicity Criteria for Yanubbe Creek	
	Acute Criterion (CMC) µg/L	Chronic Criterion (CCC) µg/L
Copper, Total	7.12	5.20
Lead, Total	21.38	0.83
Zinc, Total	47.96	43.44

The WET test effective in the NPDES permit requires once per quarter chronic biomonitoring with a critical dilution of 100%. Therefore, because the chronic water quality criterion was the basis for the effective permit limit for the metals and because the critical dilution for the effective chronic toxicity testing uses the same flow basis, the site water mix used for WER testing was 100% effluent with no dilution from upstream receiving water.



Figure 1. Aerial photography showing the City of Broken Bow WWTP Outfall 001 discharge location in Broken Bow, OK.

## 4.0 SAMPLING

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Following an initial range finding sampling trip, five subsequent sampling trips were completed to collect samples of effluent from the Broken Bow Public Works Authority Outfall 001 discharge. These trips included sampling for the WER study in addition to the collection of ten samples for the development of a dissolved translator. Sampling was conducted according to the project workplan included as Appendix A and according to GBM<sup>c</sup> Standard Operating Procedures. During each sampling effort for the WER study, four individual samples of effluent were collected from the Outfall 001 discharge during an approximate six hour time period. This time period was chosen to reflect the sampling conducted for routine DMR sampling. The four samples were composited in the laboratory. As the flow from the facility remains relatively constant, the samples were composited at a 1:1 ratio. Samples collected for the dissolved translator included ten grab samples taken on separate days.

To the extent practicable, clean sampling techniques following EPA Method 1669 (modified according to GBM<sup>c</sup> SOP), were used for the collection of water samples. All samples were handled using appropriate techniques and chain of custody procedures. Samples collected for WER development were greater than three weeks apart and included different seasons. Samples were collected during times of normal facility operation, with CBOD and TSS concentrations within permit limits and when relatively unaffected by rainfall inflow or slug loads. The appendices include DMR analytical data and summary, *in-situ* data at the time of sampling, and a summary of rainfall data preceding each sampling event.

### 4.1 Field Measurements

*In-situ* measurements, consisting of temperature, pH, and dissolved oxygen were taken in the effluent each time water samples were collected. Measurements were made with a YSI multi-parameter meter. The meter was calibrated daily in accordance with the GBM<sup>c</sup> Quality Assurance Plan (QAP). Effluent flow, as measured by facility monitoring equipment, was recorded at each sample time. Routine DMR sampling was conducted on the same day as sampling efforts for the various WER studies.

## 4.2 Analytical Methodology

Samples were analyzed in the laboratory according to the procedures outlined in the most current release of Standard Methods for the Examination of Water and Wastewater using specific EPA approved methods. Table 3 summarizes the EPA approved analytical methods used by the laboratory during the study. American Interplex Laboratories, Little Rock, Arkansas completed both the analytical work and the WER toxicity tests. Table 4 provides a summary of analyses conducted during the study.

Table 3. Analytical methods followed during the WER study.

Parameter	Method	Preservative <sup>1</sup>	Holding Time
Total Metal	EPA200.7/200.8	4 °C, HNO <sub>3</sub>	6 Months
Dissolved Metal	EPA200.7/200.8	4 °C, HNO <sub>3</sub>	6 Months
T. Hardness	SM2340B	4 °C, HNO <sub>3</sub>	6 Months
T. Alkalinity	SM 2320B	4 °C	14 Days
TSS	SM2540D	4 °C	7 Days
TOC	SM5310C	4 °C, H <sub>2</sub> SO <sub>4</sub>	28 Days
DOC	SM5310C	4 °C	28 Days
TDS	SM2540C	4 °C	7 Days

<sup>1</sup> All chemical preservatives added after sample composite.

Table 4. Analysis conducted in conjunction with the WER toxicity testing.<sup>1</sup>

Analytical Parameter	Water Sample Source		
	Effluent	Moderately hard lab water	Selected spiked test dilutions <sup>2,3</sup>
Total Metal	X	X	X
Dissolved Metal	X	X	X
Total Hardness	X	X	
Total Alkalinity	X	X	
pH	X	X	X
TSS	X	X	
TOC	X	X	
DOC	X	X	
Specific conductance	X	X	X
TDS	X		
Dissolved oxygen		X	X
Temperature		X	X
Routine DMR parameters	Routine parameters were analyzed by the permittee as part of routine monitoring on the same day as WER study samples are collected. Routine parameters include CBOD, TSS, NH <sub>3</sub> -N, DO, T. Copper, T. Lead, T. Mercury, T. Zinc.. The analytical data from this sampling can be found in Appendix B. Total Residual Chlorine and fecal coliform (found in the permit) were not sampled and analyzed on the days of WER study sampling.		

<sup>1</sup> The normal battery of chemistry completed for routine biomonitoring tests (pH, temperature, conductivity, dissolved oxygen, etc.) was completed during each WER test.

<sup>2</sup> Test treatments that bracket the LC50 must be tested for these parameters. The lowest treatment that exhibited 100% mortality, the highest treatment that exhibited no effect and the control were also tested for these parameters.

<sup>3</sup> Dilutions were prepared using cupric sulfate, zinc sulfate, and lead nitrate.

## 5.0 WER TOXICITY TESTING DESIGN

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### 5.1 Copper

The “Streamlined Water-Effect Ratio Procedure for Discharges of Copper” allows for use of one of two test species for toxicity testing in the WER development. For this study, *Ceriodaphnia dubia* was chosen as the test organism. *Ceriodaphnia dubia* were exposed to a number of copper treatments under static non-renewal conditions for 48 hours in the site water mixture (100% effluent) and in laboratory control water (moderately hard reconstituted). Concentrations of copper for the testing were based on a dilution series of 0.65. Details of the testing procedure can be found in Table 5. A summary of the methodology is presented below.

Ceriodaphnids were exposed to various spiked copper concentrations in lab water tests and site water tests using cupric sulfate 5-hydrate as the spiking agent. Test organisms were exposed to the copper treatments in 30 mL disposable beakers containing 15 mLs of test solution. A summary of test conditions is provided in Table 5. A control (lab water) that was not spiked with copper was run with each test. Spiked copper concentrations in the site water tests generally ranged from 32.3 µg/L to 308 µg/L. Spiked copper concentrations in the lab water tests ranged from 3.7 µg/L to 31 µg/L.

Tests were considered acceptable if control survival was equal to or greater than 90% and if the associated reference toxicity results fell within the upper and lower warning limits developed at the laboratory for each individual species. Test results used for WER development also met the following criteria:

- at least one treatment other than the control should exhibit mortality less than 50%;
- the percent of organisms affected should be greater than 50% in at least one treatment;
- the concentration of dissolved copper in test treatments should not decrease by more than 50% from the initial measurement to the final measurement; and
- the lab water LC50's should be within the range of LC50's determined in lab water at other laboratories under similar conditions.

Table 5. Summary of test conditions for copper WER toxicity testing.

Parameter	Test condition (Lab Water)	Test Condition (Site Water Mbx)
Test Type	48-h Acute static non-renewal	48-h Acute static non-renewal
Chemical Test	Copper	Copper
Temperature	25°C	25°C
Light Quality	Ambient Laboratory	Ambient Laboratory
Light Intensity	50-100 ft-c	50-100 ft-c
Photoperiod	16h light, 8h dark	16h light, 8h dark
Test Chamber Type	30 mL minimum	30 mL minimum
Test Solution Volume	15 mL minimum	15 mL minimum
Solution Renewal	None <sup>1</sup>	None <sup>1</sup>
Age of Test Organisms	<24-h	<24-h
No. Organisms/Chamber	5	5
No. of Replicate Chambers	4	4
Feeding Regime	None	None
Aeration	None	None
Dilution Water (Test Water)	Moderately Hard Reconstituted	Effluent
Dilution Ratio	0.65	0.65
Number of treatments (metal concentrations)	6 treatments (30, 20, 13, 8, 5, 3 µg/L)	6 treatments (300, 195, 127, 82, 54, 35 µg/L)
Highest Copper Concentration	30 µg/L	300 µg/L

<sup>1</sup> Copper concentration were measured in the test solution before test initiation and at the conclusion of the testing.

An LC50 was calculated for each test (site water mix (SWM) and lab water (LW)). To account for hardness differences, the LC50 of the SWM was normalized to the hardness of the lab water. The equation used to normalize the copper LC50 is presented below.

#### Normalization Equation for Copper

$$\text{Copper Normalized LC50} = \text{SWM LC50} * (\text{Lab water Hdns} / \text{SWM Hdns})^{0.9422}$$

The ratio of SWM-LC50/LW-LC50 is the WER for that test. Individual WERs were calculated as the lesser of the site water LC50 divided by the laboratory water LC50, or the site water LC50 divided by the species mean acute value (SMAV). The final WER (fWER) is calculated as the geometric mean from the results of the individual WERs. EPA and OWRB guidance for the calculation of a WER are expressed as the inverse of each other. For the purposes of this study calculations will be shown according to the EPA method as this is the commonly accepted calculation. A conversion to the Oklahoma specific OWRB defined WER will be provided at the conclusion of the report.

**EPA defined WER**  
SWM-LC50 / Lesser of LW-LC50 or SMAV

**OWRB defined WER**  
Lesser of LW-LC50 or SMAV / SWM-LC50

## 5.2 Lead and Zinc

Oklahoma's "Guidance Document for the Development of Site-Specific Water Quality Criteria for Metals" (OWRB 2003) was used in conjunction with the "Interim Guidance on Determination and Use of Water-Effect Ratios for Metals" (EPA 1994) for the lead and zinc WER studies. Methodologies presented in these documents are more stringent than the "streamlined method" allowed for copper, and subsequently additional testing is necessary for the development of a WER for these metals.

Both the OWRB and EPA Interim guidance require that a final WER must be calculated from at least four acceptable WER tests. Two species from different orders (one vertebrate and one an invertebrate) should be used to obtain the four tests. Like the copper WER study, *Ceriodaphnia dubia* was chosen as the primary test organism for the lead and zinc studies. *Pimephales promelas* (fathead minnow) was chosen as the secondary species for the studies. Of the three WER tests required with the primary species, one of these should be comprised of a "high flow" sample proportionally composited with effluent and upstream receiving water.

The workplan submitted for the Broken Bow Public Works Authority WER Study defined an upstream sampling location for the "high flow" sample at the Highway 70 crossing of the unnamed tributary of Yannube Creek. During the study it was determined that the watershed upstream of this point was too small to reasonably provide adequate flow for sampling purposes. GBM<sup>c</sup> & Associates requested to move the sampling point for "high flow" sampling to Yannube Creek, just upstream of the confluence with the unnamed tributary which receives the facility effluent. Throughout discussion with OWRB and EPA personnel, it was agreed upon to forgo the high flow sample and perform another sample of 100% effluent using the primary species.

A summary of WER test conditions for *Ceriodaphnia dubia* is presented in Table 6, and a summary of WER test conditions for fathead minnow is presented in Table 7. Determination of test acceptability was the same for the lead and zinc studies as the copper study.

Table 6. Summary of test conditions for ceriodaphnia lead and zinc WER toxicity testing.

<b>Parameter</b>	<b>Test condition (Lab Water)</b>	<b>Test Condition (Site Water Mix)</b>
Test Type	48-h Acute static non-renewal	48-h Acute static non-renewal
Chemical Test	Lead and Zinc (individually)	Lead and Zinc (individually)
Temperature	25°C	25°C
Light Quality	Ambient Laboratory	Ambient Laboratory
Light Intensity	50-100 ft-c	50-100 ft-c
Photoperiod	16h light, 8h dark	16h light, 8h dark
Test Chamber Type	30 mL minimum	30 mL minimum
Test Solution Volume	15 mL minimum	15 mL minimum
Solution Renewal	None <sup>1</sup>	None <sup>1</sup>
Age of Test Organisms	<24-h	<24-h
No. Organisms/Chamber	5	5
No. of Replicate Chambers	4	4
Feeding Regime	None	None
Aeration	None	None
Dilution Water	Moderately Hard Reconstituted	100% Effluent
Dilution Ratio	0.65	0.65
Lead and Zinc – Number or treatments (concentrations)	6 treatments (Adjusted throughout study based upon results)	6 treatments (Adjusted throughout study based upon results)
Lead and Zinc – Highest Concentration	Adjusted throughout study based upon results	Adjusted throughout study based upon results

<sup>1</sup> The concentration of lead or zinc was measured in the test solution before test initiation and at the conclusion of the testing.

Table 7. Summary of test conditions for lead and zinc fathead minnow WER toxicity testing.

Parameter	Test condition (Lab Water)	Test Condition (Site Water Mix)
Test Type	48-h Acute static non-renewal	48-h Acute static non-renewal
Chemical Test	Lead and Zinc (individually)	Lead and Zinc (individually)
Temperature	25°C	25°C
Light Quality	Ambient Laboratory	Ambient Laboratory
Light Intensity	50-100 ft-c	50-100 ft-c
Photoperiod	16h light, 8h dark	16h light, 8h dark
Test Chamber Type	250 mL minimum	250 mL minimum
Test Solution Volume	200 mL minimum	200 mL minimum
Solution Renewal	None <sup>1</sup>	None <sup>1</sup>
Age of Test Organisms	1-14 days	1-14 days
No. Organisms/Chamber	10	10
No. of Replicate Chambers	2	2
Feeding Regime	None during testing	None during testing
Aeration	None	None
Dilution Water	Moderately Hard Reconstituted	100% Effluent
Dilution Ratio	0.65	0.65
Lead – Number of treatments (concentrations)	10 treatments (40,000; 26,000; 16,900; 10,985; 7,140; 4,641; 3,017; 1,961; 1,275; 828 µg/L )	10 treatments (40,000; 26,000; 16,900; 10,985; 7,140; 4,641; 3,017; 1,961; 1,275; 828 µg/L )
Lead – Highest Concentration	40000 µg/L	40000 µg/L
Zinc - Number of treatments (metal concentrations)	6 treatments (750, 488, 317, 206, 134, 87 µg/L)	6 treatments (1,000, 650, 422, 274, 178, 116 µg/L)
Zinc - Highest Concentration	750 µg/L	1000 µg/L

<sup>1</sup>The concentration of lead or zinc was measured in the test solution before test initiation and at the conclusion of the testing.

<sup>2</sup>After preparation, spiked samples using effluent were allowed to stand for a minimum of 2 hours and spiked lab water samples stood a minimum of 1 hour prior to use.

An LC50 was calculated for each test (site water mix (SWM) and lab water (LW)). To account for hardness differences, the LC50 of the SWM was normalized to the hardness of the lab water. The equations used to normalize the lead and zinc LC50s are presented below.

**Normalization Equation for Lead**

$$\text{Lead Normalized LC50} = \text{SWM LC50} * (\text{Lab water Hdns} / \text{SWM Hdns})^{1.273}$$

**Normalization Equation for Zinc**

$$\text{Zinc Normalized LC50} = \text{SWM LC50} * (\text{Lab water Hdns} / \text{SWM Hdns})^{0.8473}$$

Like the copper tests, WERs for each lead and zinc test were calculated as the ratio of LC50s calculated in SWM and lab water tests.

**EPA defined WER**

$$\text{SWM-LC50} / \text{Lesser of LW-LC50 or SMAV}$$

**OWRB defined WER**

$$\text{Lesser of LW-LC50 or SMAV} / \text{SWM-LC50}$$

## 6.0 DATA HANDLING AND QUALITY ASSURANCE/QUALITY CONTROL

The laboratory's toxicity test results, analytical results, chain of custody forms and laboratory sheets were reviewed and necessary data recorded in a spreadsheet format.

Quality assurance and quality control measures taken during this study followed that of the project workplan (Appendix A).

## 7.0 RESULTS

Five trips were made for the collection of samples for the WER and dissolved translator development. Table 8 summarizes sampling efforts for each of the trips.

Table 8. Summary of WER tests performed for each sampling effort.

Date	Copper Ceriodaphnia	Lead Ceriodaphnia	Zinc Ceriodaphnia	Lead Pimephales	Zinc Pimephales	Synergistic Test
6/25/13	X	X	X			
10/1/13	X	X	X			
11/19/13		X	X	X	X	
3/18/14			X (Retest)			
3/25/14						X

Following the completion of the November 19, 2013 sampling, data collected up to that point was shared with OWRB and EPA. Personnel from EPA provided comments on the data and calculations. The review expressed concern over the high WER results from the 11/19/13 *Ceriodaphnia* test for zinc. The LC50 for the lab water test was lower than expected producing a potentially inflated WER result. After review of the data it was decided to perform an additional zinc *ceriodaphnia* test. Data and calculations for each test are presented in this report, however, the higher WER was not used in calculation of the fWER.

Throughout the study problems with tests for the lead portion became apparent. As is shown in the data, two tests (one *Ceriodaphnia*, one *Pimephales*) were invalid due to a failure to produce enough toxicity to yield an LC50 in both the SWM and lab water tests. In two other tests, the WER results were acceptable. It was determined through discussion with the testing laboratory that problems were most likely due to the low solubility of lead. In producing serial dilutions for the test, a precipitant of lead sulfate would form due to sulfate and chloride ions present in the effluent thereby changing the chemistry of the SWM. Based upon this information and variability of the results, the decision was made to abandon the lead portion of the study. The analytical data from

the lead tests conducted are found in the appendices, however the tests did not produce enough acceptable data and no fWER was calculated.

## **7.1 Water Quality Results Associated with WER Development**

A summary of the analytical results of samples collected during the WER study from the SWM and from the moderately hard lab water are presented in the Water Quality Results summary found in Appendix B. *In-situ* and flow data from Outfall 001 at the time of sampling can be found on the *in-situ* summary also found in Appendix B. Results from composite sampling for routine permit required DMR sampling conducted on the same days as WER study sampling can be found in the DMR Data Summary. Corresponding laboratory reports are located in Appendix C of the report.

## **7.2 Toxicity Test Results Used for WER Development**

### **7.2.1 Copper**

Acute 48-hour static non-renewal toxicity tests were conducted for copper WER development. Two tests using *Ceriodaphnia dubia* were completed for copper. Both tests met acceptance criteria for use in developing the fWER. Complete laboratory reports are presented in Appendix C. A summary of the results of each test utilized in the development of the fWERs is presented in Tables 9 and 10.

Table 9. Ceriodaphnid toxicity test results for Total Copper.

Site Water		Lab Water	
Total Copper (µg/L)	(% Survival at 48-h)	Total Copper (µg/L)	(% Survival at 48-h)
<b>Test Date: 6/25/13</b>			
Control	100	Control	100
40.7	100	4.15	100
60.2	100	5.88	90
87.5	100	8.43	60
133	90	12.2	0
202	65	20.0	0
308	0	31.0	0
LC50	203.3 µg/L	LC50	8.35 µg/L
<b>Test Date: 10/1/13</b>			
Control	100	Control	100
32.3	100	3.72	100
46.6	100	5.22	100
74.4	100	7.36	100
114	100	10.3	75
179	85	16	0
290	0	26.7	0
LC50	212 µg/L	LC50	11.6 µg/L

Table 10. Ceriodaphnid toxicity test results for Dissolved Copper.

Site Water		Lab Water	
Dissolved Copper (µg/L)	(% Survival at 48-h)	Dissolved Copper (µg/L)	(% Survival at 48-h)
<b>Test Date: 6/25/13</b>			
Control	100	Control	100
31.2	100	3.79	100
46.1	100	5.56	90
65.6	100	7.87	60
98.0	90	12.1	5
143	65	19.7	0
212	0	28.4	0
LC50	144.2 µg/L	LC50	8.13 µg/L
<b>Test Date: 10/1/13</b>			
Control	100	Control	100
25.3	100	3.41	100
36.8	100	4.81	100
56.8	100	6.85	100
88.8	100	9.85	75
135	85	15.3	0
217	0	24.9	0
LC50	160.1 µg/L	LC50	11.1 µg/L

## 7.2.2 Zinc

Acute 48-hour static non-renewal toxicity tests were conducted for zinc WER development. Four tests using *Ceriodaphnia dubia* were completed for zinc, however, one was questionable due to the low LC50 in the lab water test and not used in the fWER calculation. One additional test using *Pimephales promelas* was conducted for zinc for a total of four individual WERs for the zinc fWER. Zinc tests met acceptance criteria for use in developing the fWER. Complete laboratory reports are presented in Appendix C. A summary of the results of each test utilized in the development of the fWERs is presented in Tables 11-13.

Table 11. Ceriodaphnid toxicity test results for Total Zinc.

Site Water		Lab Water	
Total Zinc (µg/L)	(% Survival at 48-h)	Total Zinc (µg/L)	(% Survival at 48-h)
<b>Test Date: 6/25/13</b>			
Control	100	Control	100
72.7	100	18.6	90
106	25	27.9	90
155	5	42.6	80
232	0	65.4	85
338	0	100	65
535	0	157	30
LC50	100 µg/L	LC50	127.6 µg/L
<b>Test Date: 10/1/13</b>			
Control	100	Control	100
88	100	24.2	100
119	100	37.2	95
170	60	55.4	85
253	0	85.9	60
370	0	132	50
565	0	209	0
LC50	178.3 µg/L	LC50	102.3 µg/L
<b>Test Date: 3/18/14</b>			
Control	100	Control	100
77.8	100	22.8	100
109	100	35.6	100
160	0	54.6	80
238	0	84.3	0
362	0	133	0
552	0	209	0
LC50	132 µg/L	LC50	62.2 µg/L

Table 12. Ceriodaphnid toxicity test results for Dissolved Zinc.

Site Water		Lab Water	
Dissolved Zinc ( $\mu\text{g/L}$ )	(% Survival at 48-h)	Dissolved Zinc ( $\mu\text{g/L}$ )	(% Survival at 48-h)
<b>Test Date: 6/25/13</b>			
Control	100	Control	100
52.8	100	19.0	90
80.5	25	28.2	90
119	5	43.0	80
182	0	65.9	85
259	0	102	65
379	0	159	30
LC50	75.2 $\mu\text{g/L}$	LC50	129.2 $\mu\text{g/L}$
<b>Test Date: 10/1/13</b>			
Control	100	Control	100
79.7	100	24.2	100
106	100	35.4	95
152	60	56.7	85
220	0	86.6	60
324	0	134	50
526	0	209	0
LC50	158 $\mu\text{g/L}$	LC50	103.2 $\mu\text{g/L}$
<b>Test Date: 3/18/14</b>			
Control	100	Control	100
66.8	100	23.8	100
95.9	100	35.5	100
138	0	54.3	80
210	0	83.5	0
324	0	133	0
501	0	208	0
LC50	115 $\mu\text{g/L}$	LC50	61.8 $\mu\text{g/L}$

Table 13. *Pimephales promelas* toxicity test results for Total Zinc.

Site Water		Lab Water	
Total Zinc (µg/L)	(% Survival at 48-h)	Total Zinc (µg/L)	(% Survival at 48-h)
<b>Test Date: 11/19/13</b>			
Control	100	Control	100
139	100	97.9	100
200	100	147	95
293	100	228	75
436	100	331	55
653	100	500	25
943	55	703	10
LC50	>943 µg/L	LC50	348.2 µg/L

Table 14. *Pimephales promelas* toxicity test results for Dissolved Zinc.

Site Water		Lab Water	
Total Zinc (µg/L)	(% Survival at 48-h)	Total Zinc (µg/L)	(% Survival at 48-h)
<b>Test Date: 11/19/13</b>			
Control	100	Control	100
123	100	97.1	100
178	100	146	95
255	100	218	75
376	100	330	55
571	100	501	25
814	55	707	10
LC50	>814 µg/L	LC50	345 µg/L

## 7.3 Water Effect Ratio Development

### 7.3.1 Copper

WERs are developed as the ratio of the LC50 in the site water mix divided by the greater of the LC50 in the lab water or the SMAV. In order to mitigate the effects of elevated water hardness on the LC50, each SWM LC50 and SMAV were normalized to the hardness of the lab water. Normalizing hardness in this manner eliminates any effect from reduced toxicity the metal may display due to hardness alone. For both tests for copper the hardness normalized SMAV LC50's were greater than the lab water LC50s and were therefore used for WER calculation. WERs were developed for both total and dissolved copper. Table 15 below depicts LC50s and WERs for the copper tests.

Table 15. Summary of Copper LC50s and WERs.

	<b>SWM LC50</b>	<b>SWM LC50 (normalized)</b>	<b>Lab Water LC50</b>	<b>SMAV LC50</b>	<b>SMAV LC50 (normalized)</b>	<b>WER</b>
WER-1 Total	203.3	195	8.35	24	21.3	9.16
WER-1 Dissolved	144	138.1	8.13	22.11	19.6	<b>7.05</b>
WER-2 Total	212	229.7	11.6	24	20.8	11.03
WER-2 Dissolved	160	173.3	11.1	22.11	19.2	<b>9.04</b>

\*Copper Normalized LC50 = SWM LC50 \* (Lab water Hdns/ SWM Hdns)<sup>0.9422</sup>

WER-1 SWM Hardness = 92 mg/L, LW=88 mg/L

WER-2 SWM Hardness = 79 mg/L, LW = 86 mg/L

The fWER for use in recommending amendment of the copper criteria in the Oklahoma WQS was developed from the most stringent of either the hardness normalized lab water WER or the hardness normalized SMAV WER. Additionally, the total and dissolved WERs were compared to find the more stringent. After comparison, it was determined the dissolved copper WERs are the most stringent and will therefore be the focus of the fWER calculation.

The fWER, which is used to adjust the existing copper criteria to create site-specific criteria, is calculated as the geometric mean of all acceptable WERs from the study. A minimum of two acceptable WERs are required for calculation of an fWER according to the "Streamlined" method. For this study the two most stringent WERs were calculated from the ceriodaphnid acute site water tests and the hardness normalized site water mix and SMAV for dissolved copper. **WER-1 was 7.05 and WER-2 was 9.04, resulting in an fWER of 7.98.** The fWER is applicable to either the chronic criterion or the acute criterion.

#### **fWER Geometric Mean Calculation**

$$fWER = \exp [ \sum \ln(WER_i) / n ]$$

Where: n = number of acceptable WERs

WER<sub>i</sub> = WER from ith test

As previously stated, the OWRB guidance defines a WER as the ratio of the lab water LC50 to the site water mix LC50. This is the inverse of a WER as defined by the EPA guidance. The table below provides the WERs and fWERs shown in both formats.

Table 16. EPA and OWRB fWERs for copper.

	EPA WER	OWRB WER
WER-1	7.05	0.142
WER-2	9.04	0.111
fWER	7.98	0.125

### 7.3.2 Zinc

Zinc WERs were developed as the ratio of the LC50 in the site water mix divided by the LC50 in the lab water. LC50s were normalized to the hardness of the lab water using the normalization calculation for zinc. Three WERs were developed for both total and dissolved copper using *Ceriodaphnia dubia*. One WER was developed for both total and dissolved zinc using *Pimephales promelas*. The table below depicts LC50s and WERs for the zinc tests.

Table 17. Summary of zinc LC50s and WERs.

	SWM LC50	SWM LC50 (normalized)	Lab Water LC50	WER
WER-1 Total	100	96.3	128	0.75
WER-1 Dissolved	75.2	72.4	129	<b>0.56</b>
WER-2 Total	178	191.3	102	1.88
WER-2 Dissolved	158	169.8	103	<b>1.65</b>
WER-3 Total (Re-test)	132	143.1	62.2	2.3
WER-3 Dissolved (Re-test)	115	124.7	61.8	<b>2.02</b>
WER-4 Total (Fathead)	943	675.9	348.2	1.94
WER-4 Dissolved (Fathead)	814	583.4	345	<b>1.69</b>

\*Zinc Normalized LC50 = SWM LC50 \* (Lab water Hdns/ SWM Hdns)<sup>0.8473</sup>

WER-1 SWM Hardness = 92 mg/L, LW=88 mg/L

WER-2 SWM Hardness = 79 mg/L, LW = 86 mg/L

WER-3 SWM Hardness = 80 mg/L, LW = 88 mg/L

WER-4 SWM Hardness = 120 mg/L, LW = 81 mg/L

The fWER for use in recommending amendment of the zinc criteria in the Oklahoma WQS was developed from the more stringent of the total or dissolved hardness normalized lab water WER. It was determined the dissolved zinc WERs are the most stringent and will therefore be the focus of the fWER calculation.

The fWER, which is used to adjust the existing zinc criteria to create site-specific criteria, is calculated as the geometric mean of all acceptable WERs from the study. A minimum of four acceptable WERs are required for calculation of an fWER according to the OWRB and EPA Interim Guidance. For this study the most stringent WERs were calculated from the Ceriodaphnid and fathead minnow acute site water tests and the hardness normalized site water mix for dissolved zinc. **WER-1 was 0.56, WER-2 was 1.65, WER-3 was a 2.02, and WER-4 was a 1.69 resulting in an fWER of 1.33.** The fWER is applicable to either the chronic criterion or the acute criterion.

The table below provides a comparison between the EPA and Oklahoma specific defined WERs and fWER for Zinc.

Table 18. EPA and OWRB fWERs for zinc.

	EPA WER	OWRB WER
WER-1	0.56	1.79
WER-2	1.65	0.606
WER-3	2.02	0.495
WER-4	1.69	0.592
<b>fWER</b>	<b>1.33</b>	<b>0.752</b>

## 7.4 Dissolved Translator (f)

A dissolved translator (f) was developed for copper, lead, and zinc to be used in conjunction with the fWERs in amending the Oklahoma WQS criteria for each metal. A dissolved translator is calculated using the following calculation:

$$f = \text{dissolved concentration of metal} / \text{total concentration of metal}$$

Results from ten samples used in calculating a dissolved translator for copper, lead and zinc are found in Table 19. As the data shows, all samples were below detection limit for lead, producing a dissolved translator of 1.0. Based upon this result, amendment of the criteria for lead will not be pursued.

Table 19. Results of clean metal sampling for the dissolved translator.

	Copper (mg/L)		Lead (mg/L)		Zinc (mg/L)	
	Total	Dissolved	Total	Dissolved	Total	Dissolved
2/19/2013	0.00681	0.00431	< 0.001	< 0.001	0.0278	0.0268
6/25/2013	0.00521	0.00322	< 0.001	< 0.001	0.00487	0.002
6/26/2013	0.00536	0.0031	< 0.001	< 0.001	0.00746	0.002
8/6/2013	0.0055	0.0049	< 0.001	< 0.001	0.018	0.015
9/30/2013	0.00372	0.00252	< 0.001	< 0.001	0.0278	0.025
10/1/2013	0.00368	0.0025	< 0.001	< 0.001	0.0274	0.0262
11/19/2013	0.00772	0.00256	< 0.001	< 0.001	0.0277	0.0254
3/18/2014	0.00622	0.00476	< 0.001	< 0.001	0.032	0.0287
3/24/2014	0.00555	0.00438	< 0.001	< 0.001	0.0325	0.0276
3/25/2014	0.00525	0.00407	< 0.001	< 0.001	0.0296	0.0254

The geometric mean of the ratio of the dissolved to total metal concentrations for the ten samples is used as the dissolved translator (f) and is shown in Table 20.

Table 20. Dissolved Translator

	Copper	Lead	Zinc
	(Dissolved/Total)	(Dissolved/Total)	(Dissolved/Total)
2/19/2013	0.63	1.00	0.96
6/25/2013	0.62	1.00	0.41
6/26/2013	0.58	1.00	0.27
8/6/2013	0.89	1.00	0.83
9/30/2013	0.68	1.00	0.90
10/1/2013	0.68	1.00	0.96
11/19/2013	0.33	1.00	0.92
3/18/2014	0.77	1.00	0.90
3/24/2014	0.79	1.00	0.85
3/25/2014	0.78	1.00	0.86
<b>Sum</b>	<b>6.74</b>	<b>10.00</b>	<b>7.85</b>
<b>Geomean (f) (OWRB)</b>	<b>0.65</b>	<b>1.00</b>	<b>0.73</b>
<b>1/f (EPA)</b>	<b>1.53</b>	<b>1.00</b>	<b>1.36</b>

## 7.5 Criterion Translator (T)

A criterion translator was calculated for copper and zinc using the calculated WERs and dissolved translators. Per the OWRB guidance, a criterion translator is defined as:

$$T = WER \times f$$

The table below shows the Criterion Translator (T) for copper and zinc. Results are expressed to correlate with both EPA defined WERs and the OWRB guidance.

Table 21. Criterion translators.

	WER		Dissolved Translator (f)		Criterion Translator (T)	
	EPA	OWRB	EPA	OWRB	EPA	OWRB
Copper	7.98	0.125	1.53	0.65	12.24	0.082
Zinc	1.33	0.752	1.36	0.73	1.81	0.553

## 7.6 Synergistic Test

Following the completion of WER toxicity tests and dissolved translator sampling, an additional toxicity test was conducted to evaluate the potential for toxicity additivity or synergism. A review of the literature provides varied conclusions regarding synergism, additivity, or antagonism of metals mixtures. "A Compilation of Data on the Toxicity of Chemicals to Species in Australia Part 3: Metals" (Markich et. al. 2002) reports in a review of the literature that binary combinations of Cu + Zn had antagonistic effects to unicellular green algae. In contrast, Cooper et. al. (2009) report that a more than additive effect was observed for *C. dubia* exposed to Cu + Zn.

The synergistic test consisted of a *Ceriodaphnia dubia* 48-hour acute static non-renewal test with site water mix and lab water spiked with serial dilutions of copper and zinc combined. The ratio of copper to zinc chosen for the test reflected the ratio of the metals in the proposed site specific instream criteria as adjusted based upon the WER study results. Test conditions for the synergistic test were the same as the toxicity tests conducted for copper and zinc WERs. Dilution series were set to bracket the LC50 results observed for each metal during the WER study such that metal concentrations were higher and lower than the observed LC50's for each metal with metal ratios (copper: zinc) consistent with the ratios in the proposed criteria. Synergistic test dilutions and LC50 results are provided in Table 22 and dissolved LC50 results from each WER used in the final calculations and the synergistic test are shown in Table 23. Complete lab reports are found in Appendix C and corresponding *in-situ*, water quality, and rainfall data are found in respective summaries in the appendices.

Table 22. Synergistic test for copper and zinc. Samples collected 3/25/14.

Site Water		Lab Water	
Total Metal (Cu+Zn) (µg/L)	(% Survival at 48-h)	Total Metal (Cu+Zn) (µg/L)	(% Survival at 48-h)
<b>Test Date: 3/26/2014</b>			
Control	100	Control	100
156	95	45.3	100
196	0	60.0	90
316	0	91.4	45
402	0	139	0
673	0	212	0
LC50	173.8 µg/L	LC50	85.3 µg/L
Site Water		Lab Water	
Dissolved Metal (Cu+Zn) (µg/L)	(% Survival at 48-h)	Dissolved Metal (Cu+Zn) (µg/L)	(% Survival at 48-h)
Control	100	Control	100
144	95	44.8	100
161	0	62.7	90
231	0	85.4	45
326	0	134	0
538	0	211	0
LC50	151.8 µg/L	LC50	82.1 µg/L

Table 23. Comparison of Ceriodaphnia Dissolved Copper LC50s.

	Site Water Mix LC50 (µg/L)	Lab Water LC50 (µg/L)
WER-1	138.1	8.13
WER-2	173.3	11.1
<b>Synergistic</b>	<b>56.7*</b>	<b>7.62*</b>

\*LC50's estimated based upon analytical data of initial metals concentrations.

Table 24. Comparison of Ceriodaphnia Dissolved Zinc LC50s.

	Site Water Mix LC50 (µg/L)	Lab Water LC50 (µg/L)
WER-1	72.4	129
WER-2	169.8	103
WER-3	124.7	61.8
<b>Synergistic</b>	<b>87*</b>	<b>85.9*</b>

\*LC50's estimated based upon analytical data of initial metals concentrations

Synergistic testing provided two key results:

1. Site water tests compared to lab water in the synergistic test exhibited a water effect ratio consistent with those used in development of the fWERs. Individual WERs for copper and zinc calculated using the ratios of copper and zinc from the dilution series are comparable to the WERs presented in this study. Information below shows WERs calculated from the synergistic test using analytical data from the most

conservative dilution series set. Hardness values for the site water and lab water were the same in this test so normalization was not necessary. The synergistic test displayed a dissolved copper WER of 7.4 and a dissolved zinc WER of 1.0 compared to the fWERs 7.98 and 1.33 for dissolved copper and zinc, respectively.

**Total Metal Synergistic WER**

Site Water LC50 = 173.8 µg/L

Total Metal (157 µg/L) = 77 µg/L Copper + 80 µg/L Zinc

Lab Water LC50 = 85.3 µg/L

Total Metal (91.1 µg/L) = 10 µg/L Copper + 81.1 µg/L Zinc

**Total Copper WER = 7.7 (0.129 OWRB)**

**Total Zinc WER = 0.99 (1.01 OWRB)**

**Dissolved Metal Synergistic WER**

Site Water LC50 = 151.8 µg/L

Total Dissolved Metal (143.7 µg/L) = 56.7 µg/L Copper + 87 µg/L Zinc

Lab Water LC50 = 82.1 µg/L

Total Dissolved Metal (98 µg/L) = 7.62 µg/L Copper + 85.9 µg/L Zinc

**Dissolved Copper WER = 7.44 (0.134 OWRB)**

**Dissolved Zinc WER = 1.01 (0.67 OWRB)**

2. The metal combination that represents the synergistic test LC50 has a total dissolved metal concentration of 151.8 µg/L, of which 87 µg/L is attributable to zinc. That zinc concentration fell within the range of the LC50's for zinc measured during the WER study, therefore the toxicity observed in the synergistic test could be attributable to zinc alone with no synergistic or additive effect from copper.

## **8.0 CONCLUSIONS**

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1. Using the final criterion translator (T) of 12.24 (0.082 OWRB) based upon an fWER of 7.98 (0.125 OWRB) and a dissolved translator (f) of 1.53 (0.65 OWRB) results in a site specific WER adjusted copper acute criterion of 83.66 µg/L and a chronic criterion of 61.10 µg/L

2. Using the final criterion translator (T) of 1.81 (0.553 OWRB) based upon an fWER of 1.33 (0.752 OWRB) and a dissolved translator (f) of 1.36 (0.73 OWRB) results in a site specific WER adjusted zinc acute criterion of 84.90 µg/L and a chronic criterion of 77.53 µg/L.
3. Based upon the OWRB WER Guidelines the final permit limits for total copper and zinc for the Broken Bow Public Works Authority could be removed from the permit if the amended criterion results in no reasonable potential; or if reasonable potential is exceeded, the final permit limits for copper and zinc could be amended considering the application of the criterion translator to the instream criteria.

The current and WER adjusted site specific criteria resulting from use of the final criterion translators for copper and zinc are shown in Table 25.

Table 25. Current and WER adjusted aquatic toxicity criteria (at 34.9 mg/L hardness).

Parameter	Existing Hardness Dependent Aquatic Toxicity Criteria		WER adjusted Hardness Dependent Aquatic Toxicity Criteria	
	Acute Criterion (CMC) µg/L	Chronic Criterion (CCC) µg/L	Acute Criterion (CMC) µg/L	Chronic Criterion (CCC) µg/L
Copper	7.12	5.20	83.66	61.10
Zinc	47.96	43.44	84.90	77.53

**Appendix A**

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**Workplan**



**Water Effects Ratio Workplan  
Broken Bow, OK  
Public Works Authority**

**FINAL**  
**January 3, 2013**

# **Water Effects Ratio Workplan**

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Prepared for:

**Broken Bow Public Works Authority  
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Broken Bow, OK 74728**

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**FINAL  
January 3, 2013**

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# 1.0 BACKGROUND/APPROACH

The Broken Bow Public Works Authority was issued a renewed OPDES permit (OPDES Permit No. OK0021521) that became effective on September 1, 2010. The permit allows for discharge of treated municipal and industrial wastewater from the city's wastewater treatment system (Facility I.D. No. S10203) to an unnamed tributary of Yanubbe Creek. Among the permit's provisions are limits for the priority pollutants copper, lead, and zinc. The numerical limits for these metals are as follows:

Parameter	Concentration Limits	
	Monthly Average (µg/L)	Daily Maximum (µg/L)
Copper, Total Recoverable	3.84	9.47
Lead, Total Recoverable	0.71	1.23
Zinc, Total Recoverable	38.27	66.30

Permit limits for the metals were calculated using the aquatic life criteria of the Oklahoma Water Quality Standards. A water-effect ratio of 1.0 was assumed for purposes of the calculations.

Biomonitoring is another required provision of the Broken Bow OPDES permit. The permit specifies that the 7-day chronic *Pimephales promelas* test and the 7-day chronic *Ceriodaphnia dubia* test be completed quarterly. Both lethal and sublethal endpoints are reported. The critical (effluent) dilution required in the tests is 100%.

Since the issuance of the current permit, the facility has repeatedly failed to achieve concentration limits for metals. Based on these repeated permit violations, a request to perform a water effects ratio (WER) study to develop site-specific metals criteria was submitted to the Oklahoma Department of Environmental Quality on February 23, 2012. A letter from the agency dated May 29, 2012 granted permission to perform the study. Broken Bow Public Works Authority has elected to conduct WER studies for copper, lead, and zinc (total and dissolved). Results of the studies will be used to develop site-specific criteria allowing for adequate protection of aquatic life in the receiving stream.

For lead and zinc, the WER study will be conducted following "Interim Guidance on Determination and Use of Water-Effect Ratios for Metals" (EPA 1994). The copper WER

will be conducted following the "Streamlined Water-Effect Procedure for Discharges of Copper" (EPA, 2001) as described further in Section 4.0.

The Oklahoma Water Resources Board "Guidance Document for the Development of Site-Specific Water Quality Criteria for Metals" (OWRB 2003) provides procedural information for developing site-specific criteria within the State of Oklahoma. This document will be used in conjunction with EPA methodology in development of an Oklahoma-defined final WER (fWER). Additionally, the document provides methodology for the development of a dissolved translator (f). This dissolved translator will be used in conjunction with the fWER to calculate a final criterion translator (T) which will be used to develop site-specific total recoverable criterion.

## 2.0 SITE DESCRIPTION/DESIGN FLOWS

The Broken Bow wastewater treatment plant discharges to an unnamed tributary of Yanubbe Creek via Outfall 001, which is located at Latitude 34° 01' 37.165", Longitude 94° 43' 22.270". The receiving stream travels from the unnamed tributary to Yanubbe Creek which enters Stream Segment 410200 of the Lower Red River Basin. The Outfall 001 discharge location is shown in Figure 1.

The Statement of Basis for the NPDES permit currently in effect was reviewed for discharge and receiving stream design flow characteristics. The design flow used as the basis for calculation of permit limits for copper, lead, zinc, WET, and others was 1.0 mgd (1.547 cfs). The upstream flow rate of Yanubbe Creek used in calculation of the permit limits was the default value of 1 cfs.

Aquatic life criteria (total) for copper, lead, and zinc for Yanubbe Creek, based on a site-specific averaged hardness concentration of 34.9 mg/L are shown below.

Parameter	Hardness Dependent Aquatic Toxicity Criteria for Yanubbe Creek	
	Acute Criterion (CMC) µg/L	Chronic Criterion (CCC) µg/L
Copper, Total	7.12	5.20
Lead, Total	21.38	0.83
Zinc, Total	47.96	43.44

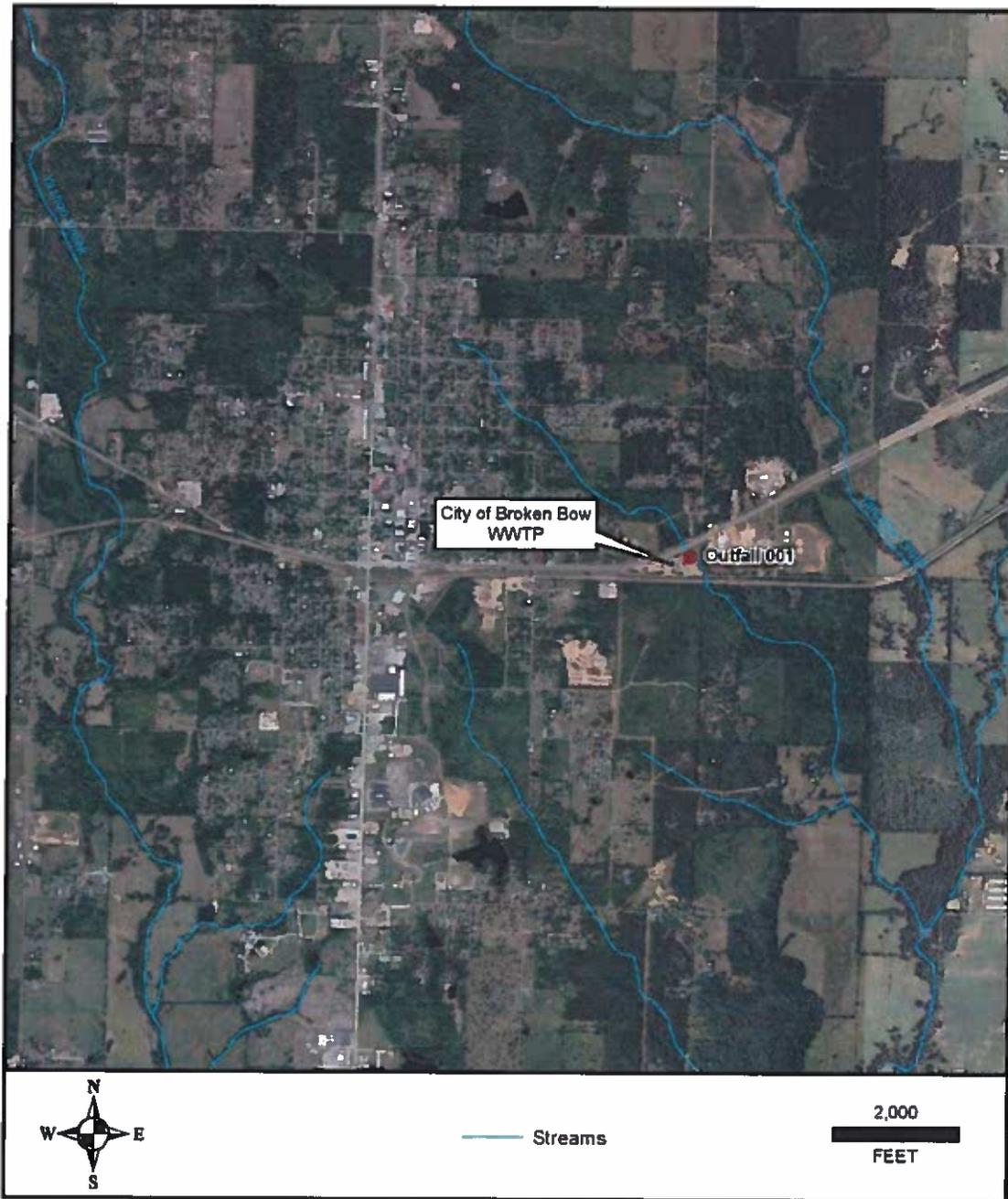


Figure 1. Aerial photography showing the City of Broken Bow WWTP Outfall 001 discharge location in Broken Bow, OK.

The WET test requirements in the NPDES permit are for once per quarter chronic biomonitoring with a critical dilution of 100%. The critical dilution reflects the percent contribution of effluent mixed with receiving water. Therefore, since the chronic water quality criterion was the basis for the effective permit limits for copper, lead, and zinc and because the critical dilution for the chronic toxicity testing uses the same flow basis, the site water mix to be used for WER testing will contain no dilution from upstream receiving water.

Copper WER testing will be completed following the stream-lined protocol where fewer tests are necessary, high flow testing is not required, and tests can be completed closer together. The lead and zinc WER testing will be completed following the more rigorous Interim Guidance. Therefore, more tests will be completed and a minimum of one sample for the lead and zinc WERs will be taken during a period where substantial upstream flow is present allowing for creation of a site water mix of less than 100% effluent. A proportional site water mix based on the flow of effluent from Outfall 001 and upstream receiving water will be used in the testing of this WER for both lead and zinc.

The station which will be utilized to collect upstream receiving water will be located at the Highway 70 crossing of the unnamed tributary of Yannube Creek. If it is determined during sampling that this station is not located far enough upstream of the facility to ensure there is no influence from wastewater discharge due to stream morphology, flow, etc; a station further upstream will be chosen.

## **3.0 SAMPLING**

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A minimum of five sampling trips will be conducted to collect samples of the Broken Bow Outfall for calculation of WERs for copper, lead, and zinc. The initial trip will be performed to gather grab samples which will be used to determine a range of concentrations of each metal to be used in the toxicity tests. Remaining sample trips will involve the collection of composite samples required to complete the WER study including quality control and analysis of routine DMR parameters to ensure effluent conditions are representative of normal discharge. Sampling efforts for the WER study will also

incorporate clean metals sampling and analysis to develop a dissolved translator. A minimum of 10 different samples is required to develop a site-specific translator. Multiple samples for total and dissolved metals analysis may be collected during the scheduled sampling trips. However, samples taken for development of the dissolved translator will not be taken at a frequency greater than one per day.

Composite samples collected for the copper, lead, and zinc WER study will be collected in the following manner. During a single sampling trip, four individual effluent samples will be collected from Outfall 001 over a 6 hour time period (approximately 1 sample per 1.5 hrs). These samples will be composited on a flow-weighted basis in the laboratory to create the site water mix (SWM). Typical practice for composite sampling is done over 24-hours. However, 6-hour composite samples have been approved by ODEQ for the facility's DMR sampling and this reduced sampling time is believed to be representative due to retention time within the wastewater treatment system. Therefore, for purposes of the WER study, composite sampling will still be utilized but over a shortened (6-hour) length of time. For WER samples taken to represent high flow scenarios, the procedures provided in Appendix E of the OWRB guidance document will be utilized to determine the ratio of effluent to receiving water. Grab samples will be utilized for development of the dissolved to total metal translator.

Clean techniques sampling (modified), following EPA Method 1669 will be utilized for the water sample collection. Samples for WER toxicity testing for lead and zinc will be made at a minimum of three weeks apart, with samples taken during a minimum of two different seasons. Samples for WER toxicity testing for copper will be made at a minimum of one month apart. All sampling will occur during times of normal facility operation when CBOD and TSS concentrations are within permit limits, and when relatively unaffected by rainfall inflow or slug loads. The sampling team will coordinate with Broken Bow Public Works Authority to ensure the wastewater treatment systems are operating normally, with no upset conditions. The effluent flows and in-situ measurements at the time of the samples will be recorded and if a site-water mix of effluent and upstream receiving water is tested the upstream flow and in-situ measurements will also be recorded. Sampling will be

conducted during low-flow conditions to the extent practicable, when no recent significant rainfall events have occurred. Rainfall data for the area will be evaluated for the 2 weeks preceding each sampling event. Information necessary for the laboratory to composite all samples will be specified on Chain of Custody (COC) forms prepared for the project.

### **3.1 Sample Handling and Custody**

After the samples have been collected, care will be taken in transporting the samples to the contract laboratory for analyses. All samples will be placed in the appropriate clean containers supplied by the laboratory with no air space in the sample container. Each sample container will be labeled with the sample I.D., date, time, and initials of collector(s). Samples will be placed in ice chests and maintained at 4° C for delivery to the laboratory in a timely manner in order to meet regulatory holding times. COC forms that include information on each sample delivered to the laboratory for analysis will be completed. Each COC form will be signed by each person handling the samples from collection in the field to receipt in the laboratory. The COC form will include all required information and will be checked for completeness prior to submission of samples to the laboratory.

### **3.2 Analytical Methodology**

Samples will be analyzed in the laboratory according to the procedures outlined in the most current release of *Standard Methods for the Examination of Water and Wastewater*. Where specific EPA approved analysis methods exist, the laboratory shall use them. Table 1 summarizes the analytical methods to be used during the study. American Interplex Laboratories, Little Rock, Arkansas will complete both the analytical work and the WER toxicity tests. Analyses required for the WER study are shown in Table 2.

Table 1. Analytical methods to be followed during the WER study.

Parameter	Method	Preservative <sup>1</sup>	Holding Time
Total Metal (copper, lead, zinc)	EPA200.7/200.8	4 °C, HNO <sub>3</sub>	6 Months
Dissolved Metal (copper, lead, zinc)	EPA200.7/200.8	4 °C, HNO <sub>3</sub>	6 Months
T. Hardness	EPA200.8	4 °C, HNO <sub>3</sub>	6 Months
T. Alkalinity	SM 2320B	4 °C	14 Days
TSS	SM2540D	4 °C	7 Days
TOC	SM5310C	4 °C, H <sub>2</sub> SO <sub>4</sub>	28 Days
DOC	SM5310C	4 °C	28 Days
TDS	SM2540C	4 °C	7 Days

All chemical preservatives added after sample composite and/or dilution sub-sampling.

Table 2. Analysis to be conducted in conjunction with the WER toxicity testing.<sup>1</sup>

Analytical Parameter	Water Sample Source		
	Effluent and SWM	Lab water	Selected spiked test dilutions <sup>2,3</sup>
Total metal (copper, lead, zinc)	X	X	X
Dissolved metal (copper, lead, zinc)	X	X	X
Total Hardness	X	X	
Total Alkalinity	X	X	
pH	X	X	X
TSS	X	X	
TOC	X	X	
DOC	X	X	
Specific conductance	X	X	X
TDS	X		
Dissolved oxygen		X	X
Temperature		X	X
Routine DMR parameters	Routine parameters will be analyzed by the permittee as part of routine monitoring on the same day as WER study samples are collected. Routine parameters include CBOD, TSS, NH <sub>3</sub> -N, DO, Total Residual Chlorine, T. Copper, T. Lead, T. Mercury, T. Zinc, pH.		

<sup>1</sup> The normal battery of chemistry completed for routine biomonitoring tests (pH, temperature, conductivity, dissolved oxygen, etc.) should be completed during each WER test.

<sup>2</sup> At a minimum the test treatments that bracket the LC50 must be tested for these parameters. The lowest treatment that exhibited 100% mortality, the highest treatment that exhibited no effect and the control should each also be tested for these parameters.

<sup>3</sup> Dilutions will be prepared using copper sulfate, lead nitrate, and zinc sulfate.

## 4.0 TESTING DESIGN

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### 4.1 Lead and Zinc

The "Interim Guidance on Determination and Use of Water-Effect Ratios for Metals" (EPA 1994) will be followed for development of water-effect ratios for lead and zinc. For each metal, a minimum of three primary WER toxicity tests sets will be conducted using *Ceriodaphnia dubia*. A minimum of one sample for each metal will consist of a proportionally composited sample comprised of effluent and upstream receiving stream water. One secondary WER toxicity test set will be conducted for *Pimephales promelas* for both lead and zinc. A summary of WER test conditions for *Ceriodaphnia dubia* is presented in Table 3, and a summary of WER test conditions for fathead minnow is presented in Table 4.

Table 3. Summary of test conditions for ceriodaphnia lead and zinc WER toxicity testing.

Parameter	Test condition (Lab Water)	Test Condition (Site Water Mix)
Test Type	48-h Acute static non-renewal	48-h Acute static non-renewal
Chemical Test	Lead and Zinc (individually)	Lead and Zinc (individually)
Temperature	25°C	25°C
Light Quality	Ambient Laboratory	Ambient Laboratory
Light Intensity	50-100 ft-c	50-100 ft-c
Photoperiod	16h light, 8h dark	16h light, 8h dark
Test Chamber Type	30 mL minimum	30 mL minimum
Test Solution Volume	15 mL minimum	15 mL minimum
Solution Renewal	None <sup>1</sup>	None <sup>1</sup>
Age of Test Organisms	<24-h	<24-h
No. Organisms/Chamber	5	5
No. of Replicate Chambers	4	4
Feeding Regime	None	None
Aeration	None	None
Dilution Water	Moderately Hard Reconstituted	100% Effluent <sup>3</sup>
Dilution Ratio	0.65	0.65
Lead – Number or treatments <sup>2</sup> (concentrations)	6 treatments (200, 130, 84.5, 54.9, 35.7, 23.2 µg/L)	To be determined by range finding test.
Lead – Highest Concentration <sup>2</sup>	200 µg/L	To be determined by range finding test.
Zinc - Number of treatments <sup>2</sup> (metal concentrations)	6 treatments (250, 162.5, 105.6, 68.7, 44.6, 29.0 µg/L)	To be determined by range finding test.
Zinc - Highest Concentration <sup>2</sup>	250 µg/L	To be determined by range finding test.

<sup>1</sup> The concentration of lead or zinc will be measured in the test solution before test initiation and at the conclusion of the testing.

<sup>2</sup> Treatment number and concentration may vary based on initial testing results.

<sup>3</sup> One test will be completed for both lead and zinc with a site water mix where percent effluent is less than 100%, proportional to upstream flow.

An LC50 for each test (site water mix (SWM) and lab water (LW)) will be calculated. The LC50 of the SWM will be normalized to the hardness of the lab water. Following the Oklahoma specific WER guidance, the ratio of LW-LC50/SWM-LC50 is the WER for that test (EPA guidance is SWM-LC50/LW-LC50).

Table 4. Summary of test conditions for lead and zinc fathead minnow WER toxicity testing.

Parameter	Test condition (Lab Water)	Test Condition (Site Water Mix)
Test Type	48-h Acute static non-renewal	48-h Acute static non-renewal
Chemical Test	Lead and Zinc (individually)	Lead and Zinc (individually)
Temperature	25°C	25°C
Light Quality	Ambient Laboratory	Ambient Laboratory
Light Intensity	50-100 ft-c	50-100 ft-c
Photoperiod	16h light, 8h dark	16h light, 8h dark
Test Chamber Type	250 mL minimum	250 mL minimum
Test Solution Volume	200 mL minimum	200 mL minimum
Solution Renewal	None <sup>1</sup>	None <sup>1</sup>
Age of Test Organisms	1-14 days	1-14 days
No. Organisms/Chamber	10	10
No. of Replicate Chambers	2	2
Feeding Regime	None during testing	None during testing
Aeration	None	None
Dilution Water	Moderately Hard Reconstituted	100% Effluent
Dilution Ratio	0.65	0.65
Lead – Number of treatments <sup>2</sup> (concentrations)	6 treatments <sup>3</sup> (To be determined by range finding test)	To be determined by range finding test.
Lead – Highest Concentration <sup>2</sup>	To be determined by range finding test.	To be determined by range finding test.
Zinc - Number of treatments <sup>2</sup> (metal concentrations)	6 treatments <sup>3</sup> (1600, 1040, 676, 439, 286, 186 µg/L)	To be determined by range finding test.
Zinc - Highest Concentration <sup>2</sup>	1600 µg/L	To be determined by range finding test.

- <sup>1</sup> The concentration of lead or zinc will be measured in the test solution before test initiation and at the conclusion of the testing.
- <sup>2</sup> The final WER (fWER) for each metal will be calculated as a geometric mean from a minimum of three individual WERs, with at least one individual WER from the secondary test species.
- <sup>3</sup> Treatment concentration could be adjusted following initial testing.
- <sup>4</sup> After preparation, spiked samples using effluent will be allowed to stand for a minimum of 2 hours and spiked lab water samples will stand a minimum of 1 hour prior to use.

## 4.2 Copper

The “Streamlined Water-Effect Procedure for Discharges of Copper” (EPA 2001) will be followed for development of a water-effect ratio specifically for copper. A minimum of two WER toxicity tests sets (consisting of a toxicity test using a SWM of 100% effluent; and a LW toxicity test) will be conducted using *Ceriodaphnia dubia*. A summary of copper WER test conditions for *Ceriodaphina* is presented in Table 5.

Lab water used for both copper and zinc tests will be reconstituted moderately hard water. The hardness of the lab water will not exceed the hardness of the site water unless the site water is below 50 mg/L.

Table 5. Summary of test conditions for copper WER toxicity testing.

Parameter	Test condition (Lab Water)	Test Condition (Site Water Mix)
Test Type	48-h Acute static non-renewal	48-h Acute static non-renewal
Chemical Test	Copper	Copper
Temperature	25°C	25°C
Light Quality	Ambient Laboratory	Ambient Laboratory
Light Intensity	50-100 ft-c	50-100 ft-c
Photoperiod	16h light, 8h dark	16h light, 8h dark
Test Chamber Type	30 mL minimum	30 mL minimum
Test Solution Volume	15 mL minimum	15 mL minimum
Solution Renewal	None <sup>1</sup>	None <sup>1</sup>
Age of Test Organisms	<24-h	<24-h
No. Organisms/Chamber	5	5
No. of Replicate Chambers	4	4
Feeding Regime	None	None
Aeration	None	None
Dilution Water (Test Water)	Moderately Hard Reconstituted	100% Effluent
Dilution Ratio	0.65	0.65
Number of treatments <sup>2</sup> (metal concentrations)	6 treatments (15, 9.8, 6.3, 4.1, 2.7, 1.7 µg/L)	To be determined by range finding test.
Highest Copper Concentration	15 µg/L	To be determined by range finding test.

<sup>1</sup> Copper concentration will be measured in the test solution before test initiation and at the conclusion of the testing.

<sup>2</sup> Treatment number and concentration may vary based on results of first WER testing.

Individual WERs will be calculated as the lesser of the site water LC50 divided by the laboratory water LC50, or the site water LC50 divided by the species mean acute value (SMAV). The SMAV will be adjusted for site hardness. The fWER will be calculated as the geometric mean from the results of the individual WERs.

### 4.3 Copper, Lead, and Zinc Additive/Synergistic Test

Following calculation of the individual WERs for copper, lead, and zinc a minimum of one additional toxicity test set will be completed to evaluate the potential for toxicity additivity or synergism by subjecting test organisms to a SWM containing all three metals. The additional toxicity test will contain a mixture of metals at concentrations proposed as the

post WER site specific criteria. A *Ceriodaphina dubia* 48-hour acute static non-renewal toxicity test will be used for the additional test. The site water mix will be the same as for all the individual WER tests. Except for the number of treatments, the summary conditions found in Tables 3 and 5 apply to the Additive/Synergistic test.

#### **4.4 Dissolved Translator**

A dissolved translator will be developed according to procedures outlined in "Guidance Document for the Development of Site-Specific Water Quality Criteria for Metals" (OWRB 2003). This translator will be used in conjunction with the WER to develop site-specific total recoverable criterion as opposed to the default statewide dissolved criterion. A minimum of ten samples from Outfall 001 will be collected and analyzed for dissolved and total copper, lead, and zinc. For this portion of the study, grab samples will be collected using clean sampling techniques.

## **5.0 DATA HANDLING AND INTERPRETATION**

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The laboratory's toxicity test results, analytical results, chain of custody forms and laboratory sheets will be reviewed for QA/QC and the data will be recorded in a spreadsheet format. Calculations will then be performed to develop a final water effect ratio (fWER), a dissolved translator (f), and a final criterion translator (T). The OWRB guidance for calculation of a WER is expressed as the inverse of the EPA guidance. Calculations for the WER will be performed according to the OWRB guidance to agree with the dissolved translator and final criterion translator portions of the OWRB guidance.

An LC50 will be calculated for each SWM test and each lab water test using the metal concentrations measured in the test dilutions. Each SWM LC50 will be normalized to the hardness of the lab water test using the hardness equation for each respective metal. The equation used for copper, lead, and zinc hardness normalization is provided below:

Copper - Normalized LC50 = SWM LC50 \* (Lab water Hdns/ SWM Hdns)<sup>0.9422</sup>

Lead – Normalized LC50 = SWM LC50 \* (Lab water Hdns/ SWM Hdns)<sup>1.273</sup>

Zinc - Normalized LC50 = SWM LC50 \* (Lab water Hdns/ SWM Hdns)<sup>0.8473</sup>

(4 significant digits should be maintained in calculations)

Once the LC50's have been calculated for each test the ratio of the LW LC50 / SWM LC50 is the WER ratio for that test pair. A WER will be calculated for each pair of toxicity tests completed. This should result in a total of 2 copper WER's (both for ceriodaphnids) and 4 lead and zinc WER's (three for ceriodaphnids and one for fathead minnows for each metal). The fWER for each metal is calculated as the geometric mean of the WER's determined from the testing for that metal.

The dissolved translator (f) will be calculated as the ratio of dissolved metal / total metal from 10 or more acceptable samples.

A final criterion translator (T) will be determined by calculating the product of the fWER and the dissolved translator (fWER x f = T). The statewide dissolved criterion will be divided by the criterion translator (T), which is the product of the Oklahoma-defined dissolved fWER and f, to obtain a site-specific total recoverable criterion. Subsequent to calculation of the revised water quality criterion the facilities permit limits will be revised to reflect the new in-stream criterion resulting from the WER(s).

## **6.0 QUALITY ASSURANCE/QUALITY CONTROL**

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Trained scientists will conduct or supervise the field sampling and other associated activities at the sample location. Notes will be kept in field notebooks and/or specific field data forms that record information collected during the study, unusual observations, and a log of each day's activities. All data forms, calibration logs, field notes, and other study documentation will be reviewed by the Project Manager or Senior Scientist for completeness and accuracy.

## 6.1 Sample Collection QA/QC

Duplicate samples for key constituents (e.g. metals, TSS, etc.) shall be collected on 10% of the samples during the study. Duplicate samples should vary by no more than 30% relative percent difference (RPD) or the sample results will be considered suspect. In the event an RPD exceeds 30%, the Project Manager will investigate the incident to determine the cause of the exceedance and what action, if any, is necessary.

One field blank will be collected during each sample event for analysis of total metals (copper and zinc). Field blanks will consist of a sample of ultra pure laboratory water poured into the appropriate sample container in the field to simulate all possible contaminant exposures. Sampling methodology and equipment must be the same for field blanks as for routine sampling in the study. If a field blank is found to be contaminated, (>120% of the MDL) by a chemical of concern, an analysis will be conducted to determine the potential impact of the contamination on the results of the associated batch of samples. The Project Manager will determine the appropriate course of action from the results of the analysis.

## 6.2 Analytical QA/QC

The laboratory will validate analytical data by use of blanks, laboratory controls, spikes, and spike duplicates. Laboratory blanks measure the amount of each respective analyte contributed from the analytical procedure. A laboratory blank is considered out of control for a specific analyte if the value exceeds the higher of either the minimum detection limit (MDL) or 5% of the measured concentration in the sample. A laboratory control measures the ability of the laboratory to recover an analyte from a blank matrix. The laboratory spike sample is used to evaluate the laboratory's ability to recover an analyte in the sample matrix. The QC exceedance criteria for laboratory controls and spikes is based on upper and lower control limits derived from the laboratory's method specialized limits. The laboratory spike duplicate is used to evaluate the laboratory's precision (ability to attain similar analytical results from duplicate samples). A RPD is calculated for the spike and

spike duplicate. The RPD is compared to method specialized limits to determine QC exceedance. Any significant excursion from one of the QC parameters will result in a repeat of the analysis in question following an investigation by the laboratory as to the cause of the QC excursion and a report of the corrective actions taken.

### **6.3 Toxicity Testing QA/QC**

Toxicity testing will be completed following EPA method 2002.O for the *Ceriodaphnia* and EPA method 2000.D for the fathead minnow. Specific conditions are outlined in Section 4.0 of this study plan. Test acceptance criteria will meet standard method requirements:

1. 90% survival in controls; and
2. Laboratory organisms must be in the labs normal acceptable range for reference toxicity testing.

Initial metal concentrations will be compared to the final metal concentrations in the WER tests to ensure a sufficient concentration of metal was present throughout the duration of the test. Dissolved oxygen will be monitored on a daily basis to ensure levels remain within acceptable limits during laboratory testing. Should oxygen levels drop below 3.0 mg/L, test treatments will be renewed or aerated as appropriate.

### **6.4 General QA/QC Procedures and Information**

GBM<sup>c</sup> & Associates conducts scientific studies (both field and laboratory) in support of regulatory applications in various media including water and wastewater. An integral part of any successful scientific study is the Quality Assurance Plan and/or site specific work plan. Large-scale and/or long-term studies such as water quality modeling require a rigorous quality assurance program that can be implemented consistently by all participants throughout the duration of the study period. Study teams with GBM<sup>c</sup> & Associates are provided a copy of the GBM<sup>c</sup> & Associates Quality Assurance Plan (QAP) and, if available,

a Study Work Plan to follow throughout the course of each study. GBM<sup>c</sup> & Associates' full QAP is available upon request. If a project specific plan exists for a given study, its procedures will supercede those of the GBM<sup>c</sup> & Associates QAP, unless otherwise determined. The QAP contains information regarding quality assurance and quality control activities and procedures designed to facilitate the production of scientifically defensible data with a high level of accuracy and precision. Activities governed by this plan include:

1. aquatic ecology field studies,
2. general field operations,
3. sampling/monitoring programs, and
4. data reporting activities.

The QAP is composed mostly of standard operating procedures (SOPs) designed to provide methodology pertinent to completion of tasks in a consistent and defensible manner. All SOPs are based on accepted methodologies found in documents published by groups such as US-EPA, USGS, and Water Environment Federation (WEF). These SOPs are modified to take specific requirements into account when appropriate. Generally, modifications of SOPs based on state specific requirements are minor changes to the procedures followed by GBM<sup>c</sup> personnel. The following sections provide general quality assurance/quality control guidance that supports the SOPs found in the Quality Assurance Plan for GBM<sup>c</sup> & Associates, Scientific and Field Studies.

#### **6.4.1 Key Personnel**

Ultimate authority on any project falls into the hands of a GBM<sup>c</sup> & Associates Principal who must approve all study plans and reports. Each study team is headed by a Project Manager who has oversight responsibility for the study procedures, applications, and the data generated. During field studies a designated field team leader, usually the Senior Scientist, will be in charge of the field operations. The field team leader is responsible for completion of data collection following appropriate QA/QC guidelines. Each

study team member is responsible to ensure that the appropriate procedures are followed and that safety and ethical standards are maintained to ensure the highest quality study results.

#### **6.4.2 Training**

All personnel participating in studies have been trained by experienced scientists/engineers to complete the necessary tasks or are in the process of being trained with appropriate oversight. Personnel participating in scientific studies shall be familiar with the SOPs appropriate to that particular study and the QAP. Personnel participating in scientific studies conducted pursuant to specific procedures specified by a regulatory authority (*e.g.*, a state or federal environmental agency) shall be familiar with those specific procedures.

#### **6.4.3 Field Trip Preparation**

To ensure that all field activities can be conducted completely and efficiently, field teams will complete a Field Equipment Checklist prior to loading for the trip to ensure all necessary equipment is identified. The field team will check the condition and confirm proper function of all equipment and supplies before traveling to a site. In addition, they will prepare sample containers and labels for use to the extent possible prior to departure to the study site.

#### **6.4.4 Instrument Inspections and Performance Tests**

Where appropriate, calibration and performance tests are described in the SOP of the respective application. Generally, all equipment will be utilized per the manufacturer's directions. If during the course of the field activities equipment fails to conform to known QA/QC requirements, the equipment will be repaired or replaced with similar equipment that will meet QA/QC requirements.

#### **6.4.5 Equipment Care and Maintenance**

Equipment cleaning and maintenance procedures will follow manufacturer recommendations. Each day during a field trip equipment should be inspected before use (during calibration, etc.) to ensure functionality. All equipment will be inspected and cleaned immediately following a field trip and stored in a safe place to allow its future readiness. Portable field meters should be calibrated in the lab at least once a month to monitor readiness.

#### **6.4.6 Assurance of Complete Data Collection**

Upon conclusion of all activities at a given study location, the study plan should be reviewed to ensure all necessary data was collected. The field team should review all completed data forms and sample labels for accuracy, completeness, and legibility, and make a final inspection of samples. If information is missing from the forms or labels, the team leader should fill in the missing information prior to proceeding to the next study location. Any missing and/or compromised samples should be collected immediately. A field notebook should be maintained by the field team leader (at a minimum) to document field activities, data collected, deviations from method, and general observations and information related to the study. Every person should maintain individual field logs to document activities and observations during daily activities.

#### **6.4.7 Data Handling and Analysis**

All data collected during scientific studies should be checked by the team leader for completeness and accuracy. Field data forms should be complete and initialed by the completing scientist and the reviewing scientist. Data entry to spreadsheets and databases along with spreadsheet calculations shall be checked for accuracy at a rate of 10% (minimum) of the entries and calculation cells. Copies of the checked data and spreadsheets should be initialed by the reviewer and retained in the records. All calculations should be detailed in the body of written reports, or shown on GBM<sup>c</sup> &

Associates Calculation Pages. Good notes regarding calculations should be kept and filed in the project notebook. All scientific reports shall be peer reviewed and/or reviewed by the Project Manager prior to approval by a GBM<sup>c</sup> & Associates Principal.

## **7.0 REPORTING**

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Upon completion of the study, a complete report containing methods, test results, and measurements performed during the study (including sample custody forms, toxicity test data sheets, reference toxicant control charts, analytical chemistry reports, and statistical analyses); calculation procedures for fWERs, dissolved translators, and total criterion translators for copper, lead, and zinc; recommendations for criteria modification; and QA/QC discussion will be prepared and submitted to the Oklahoma Water Resources Board for review and approval.

## **Appendix B**

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# **Summary of Analytical Data**

## **In-situ Measurements**

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Broken Bow WER  
 4040-12-050  
 In-Situ Data

Date	Grab	Flow (gpm)	Temperature (°C)	SPC (µs/cm <sup>2</sup> )	D.O. (%)	D.O. (mg/L)	pH (S.U.)
6/26/2013	#1	482	26.2	1826	95.1	7.6	7.7
6/26/2013	#2	413	26.6	4740	94.1	7.45	7.8
6/26/2013	#3	488	27.7	6184	92.5	7.19	7.8
6/26/2013	#4	412	27.7	5909	91.5	7.08	7.8
10/1/2013	#1	298	25.1	321	85.4	7.05	7.6
10/1/2013	#2	350	25.2	172	84.3	6.93	7.5
10/1/2013	#3	400	25.4	323	83.4	6.85	7.6
10/1/2013	#4	450	25.7	324	81	6.58	7.6
11/19/2013	#1	550	18.4	120	95.1	8.93	7.4
11/19/2013	#2	530	18.1	102	102	9.67	7.5
11/19/2013	#3	505	18.4	160	98.9	9.28	7.5
11/19/2013	#4	475	18.5	103	103	9.67	7.5
3/18/2014	#1	760	13.5	277	99	10.3	7.2
3/18/2014	#2	780	14.1	209	97	9.96	7.3
3/18/2014	#3	795	14.5	132	91	9.34	7.5
3/18/2014	#4	695	15.2	162	91	9.14	7.5
3/25/2014	#1	466	14.3	343	106	10.9	7.2
3/25/2014	#2	541	14.5	411	105	10.7	7.3
3/25/2014	#3	455	14.9	227	103	10.4	7.4
3/25/2014	#4	590	15.2	251	104	10.4	7.6

## **DMR Data**

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City of Broken Bow  
4040-12-050  
DMR Data

Date	NH3-N (mg/L)	CBOD (mg/L)	TSS (mg/L)	Hg (mg/L)	Cu (µg/L)	Pb (µg/L)	Zn (µg/L)	Se (µg/L)
6/25/2013	1.06	3.7	8	< 0.2	< 10	< 3	< 10	< 5
10/1/2013	0.59	5.8	< 2	< 0.2	< 10	< 3	< 10	< 5
11/19/2013	0.44	< 2.0	< 2.0	< 0.2	< 10	< 3	< 10	< 5
3/18/2014	0.33	3.4	< 2.0	< 0.2	< 10	< 3	29	< 5
3/25/2014	0.19	2.2	2	< 0.2	< 10	< 3	34	< 5

Results from Environmental Resource Technologies, LLC.

## **Water Quality Data**

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City of Broken Bow  
 4040-12-050  
 Summary of Water Quality Results

Site Water Mix (SWM)

Date	D.O. (mg/L)	pH (S.U.)	Alkalinity (mg/L)	Hardness (mg/L)	Conductivity (umhos/cm)	TOC (mg/L)	DOC (mg/L)	TSS (mg/L)	TDS (mg/L)
6/25/2013	8.1	8	63	92	380	13	9.8	7	220
10/1/2013	8.5	8	60	79	380	10	8.7	< 4	260
11/19/2013	7.8	7.7	86	120	500	13	12	< 20	320
3/18/2014	7.6	7.4	44	80	350	12	11	< 4	240
<b>Synergistic Test</b>									
3/25/2014	7.8	7.2	45	84	390	14	12	< 4	310

Lab Water (LW)

Date	D.O. (mg/L)	pH (S.U.)	Alkalinity (mg/L)	Hardness (mg/L)	Conductivity (umhos/cm)	TOC (mg/L)	DOC (mg/L)	TSS (mg/L)	TDS (mg/L)
6/25/2013	7.7	7.9	60	88	350	< 1	< 1	< 4	160
10/1/2013	8.6	8.3	63	86	310	< 1	< 1	< 4	180
11/19/2013	7.8	8	61	81	330	< 1	< 1	< 4	190
3/18/2014	7.8	7.8	60	88	320	*Data not available.			
<b>Synergistic Test</b>									
3/25/2014	7.7	7.7	62	84	300	*Data not available.			

\*Due to lab error, lab water for the samples collected on 3/18/14 and 3/25/14 were not analyzed for TOC, DOC, TSS, and TDS. Lab water should remain similar and previous samples should be reflective of this information.

## **Rainfall Data**

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City of Broken Bow  
 4040-12-050  
 Rainfall Data for Broken Bow, OK

WER Sampling Trip #1

Broken Bow Mesonet Data Summary	
Date	Rainfall (in.)
6/11/2013	0
6/12/2013	0
6/13/2013	0
6/14/2013	0
6/15/2013	0
6/16/2013	3.14
6/17/2013	0.84
6/18/2013	0
6/19/2013	0
6/20/2013	0
6/21/2013	0
6/22/2013	0
6/23/2013	0
6/24/2013	0
6/25/2013	0

WER Sampling Trip #2

Broken Bow Mesonet Data Summary	
Date	Rainfall (in.)
9/16/2013	0
9/17/2013	0
9/18/2013	0
9/19/2013	0.39
9/20/2013	2.03
9/21/2013	0.01
9/22/2013	0
9/23/2013	0
9/24/2013	0
9/25/2013	0
9/26/2013	0
9/27/2013	0
9/28/2013	1.33
9/29/2013	0.06
9/30/2013	0
10/1/2013	0.01

WER Sampling Trip #3

Broken Bow Mesonet Data Summary	
Date	Rainfall (in.)
11/4/2013	0.22
11/5/2013	2.24
11/6/2013	1
11/7/2013	0.01
11/8/2013	0
11/9/2013	0.02
11/10/2013	0
11/11/2013	0
11/12/2013	0
11/13/2013	0
11/14/2013	0.03
11/15/2013	0.31
11/16/2013	0.01
11/17/2013	0
11/18/2013	0
11/19/2013	0

WER Sampling Trips #4-#5

Broken Bow Mesonet Data Summary	
Date	Rainfall (in.)
3/4/2014	0
3/5/2014	0
3/6/2014	0
3/7/2014	0
3/8/2014	0.07
3/9/2014	0
3/10/2014	0
3/11/2014	0
3/12/2014	0
3/13/2014	0
3/14/2014	0
3/15/2014	1.4
3/16/2014	0.01
3/17/2014	0
3/18/2014	0
3/19/2014	0
3/20/2014	0
3/21/2014	0
3/22/2014	0.3
3/23/2014	0.01
3/24/2014	0
3/25/2014	0

## **Metals Data**

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City of Broken Bow  
 4040-12-050  
 WER Study Metals Concentrations - Initial to Final

6/25/2013, Copper, Zinc, and Lead concentrations in spiking dilutions.

	Total Copper		Initial	Zinc	Dissolved Zinc		RPD
	Initial (ug/L)	Final (ug/L)			Initial (ug/L)	Final (ug/L)	
Mod Water ug/l	<1	1.09		2.42	<2	2.89	-36.4
Site Water ug/l	5.1	4.93		9.29	6.12	9.02	-38.3
Site Water - 300 ug/l	308	293		484	379	387	-2.1
Site Water - 195 ug/l	202	172		197	259	158	48.4
Site Water - 127 ug/l	133	109		142	182	114	45.9
Site Water - 82 ug/l	87.5	74.1		98.5	119	78.4	41.1
Site Water - 54 ug/l	60.2	57.5		90.7	80.5	68	16.8
Site Water - 35 ug/l	40.7	40.6		64.9	52.8	47.4	10.8
Lab Water - 30 ug/l	31	27.3		122	159	120	28.0
Lab Water - 20 ug/l	20	17.2		93.5	102	91.6	10.7
Lab Water - 13 ug/l	12.2	11.1		59.9	65.9	56.8	14.8
Lab Water - 8 ug/l	8.43	10.2		38.6	43	37.4	13.9
Lab Water - 5 ug/l	5.88	6.65		36.5	28.2	37	-27.0
Lab Water - 3 ug/l	4.15	4.39		23.2	19	23.4	-20.8

\*RPD = Relative percent difference between initial and dissolved metal.

10/1/2013, Copper, Zinc, and Lead concentrations in spiking dilutions.

	Total Copper		Initial	Zinc	Dissolved Zinc		RPD
	Initial (ug/L)	Final (ug/L)			Initial (ug/L)	Final (ug/L)	
Mod Water ug/l	<1	1.52		5.27	<2	4.24	-71.8
Site Water ug/l	3.39	3.83		24	24.8	23.4	5.8
Site Water - 300 ug/l	290	264		430	526	251	70.8
Site Water - 195 ug/l	179	166		261	324	227	35.2
Site Water - 127 ug/l	114	106		184	220	156	34.0
Site Water - 82 ug/l	74.4	66.5		132	152	113	29.4
Site Water - 54 ug/l	46.6	42		97.1	106	87.9	18.7
Site Water - 35 ug/l	32.3	29.4		69.8	79.7	66.4	18.2
Lab Water - 30 ug/l	26.7	26.7		204	209	205	1.9
Lab Water - 20 ug/l	16	16.7		129	134	131	2.3
Lab Water - 13 ug/l	10.3	10.8		94.5	86.6	85.6	1.2
Lab Water - 8 ug/l	7.36	7.07		56.9	56.7	52.9	6.9
Lab Water - 5 ug/l	5.22	4.94		34.3	35.4	34.6	2.3
Lab Water - 3 ug/l	3.72	3.41		23.4	24.2	23	5.1

\*RPD = Relative percent difference between initial and dissolved metal.

11/19/2013, Copper, Zinc, and Lead concentrations in spiking dilutions.

	Total Lead		Initial
	Initial (ug/L)	Final (ug/L)	
Mod Water ug/l	<1	<1	
Site Water ug/l	<1	2.62	
Site Water - 1000 ug/l	816	1020	
Site Water - 650 ug/l	508	572	
Site Water - 423 ug/l	319	347	
Site Water - 275 ug/l	204	209	
Site Water - 179 ug/l	130	135	
Site Water - 116 ug/l	81.7	82.1	
Lab Water - 500 ug/l	444	399	
Lab Water - 325 ug/l	297	363	
Lab Water - 211 ug/l	186	224	
Lab Water - 137 ug/l	117	139	
Lab Water - 89.3 ug/l	73.9	87.3	
Lab Water - 58 ug/l	48.3	56.8	
Lab Water - 37.7 ug/l	31.3	33	
Lab Water - 24.5 ug/l	18.3	19.2	

\*RPD = Relative percent difference between initial and dissolved metal.

11/19/2013, Lead and Zinc concentrations in spiking dilutions. Pimepha

	Total Lead		Initial
	Initial (ug/L)	Final (ug/L)	
Site Water - PP - 40000 ug/l	35100	29100	
Site Water - PP - 26000 ug/l	24600	21900	
Site Water - PP - 16900 ug/l	16100	15300	
Site Water - PP - 10985 ug/l	10600	7650	
Site Water - PP - 7140 ug/l	6840	5120	
Site Water - PP - 4641 ug/l	4460	3440	
Site Water - PP - 3017 ug/l	2960	2060	
Site Water - PP 1961 ug/l	1930	1380	
Site Water - PP - 1275 ug/l	1290	960	
Site Water - PP - 828 ug/l	856	608	
Lab Water - PP - 40000 ug/l	36900	1690	
Lab Water - PP - 26000 ug/l	26200	697	
Lab Water - PP - 16900 ug/l	16800	642	
Lab Water - PP - 10985 ug/l	11000	452	
Lab Water - PP - 7140 ug/l	7170	322	
Lab Water - PP - 4641 ug/l	4710	298	
Lab Water - PP - 3017 ug/l	3140	277	
Lab Water - PP 1961 ug/l	2080	297	
Lab Water - PP - 1275 ug/l	1340	259	
Lab Water - PP - 828 ug/l	900	206	

\*RPD = Relative percent difference between initial and dissolved metal.

03/18/2014, Zinc Concentrations in spiking dilutions. Ceriodaphnia WEI

	Total Zinc		Initial
	Initial (ug/L)	Final (ug/L)	
Mod Water ug/l	<2	4.04	
Site Water ug/l	18.8	46.5	
Site Water - 500 ug/l	552	532	
Site Water - 325 ug/l	362	330	
Site Water - 211 ug/l	238	248	
Site Water - 137 ug/l	160	162	
Site Water - 89.3 ug/l	109	125	
Site Water - 58.0 ug/l	77.8	87.3	
Lab Water - 200 ug/l	209	197	
Lab Water - 130 ug/l	133	126	
Lab Water - 84.5 ug/l	84.3	79	
Lab Water - 54.9 ug/l	54.6	49.7	
Lab Water - 35.7 ug/l	35.6	30.3	
Lab Water - 23.2 ug/l	22.8	18.7	

\*RPD = Relative percent difference between initial and dissolved metal.

3/25/2014, Copper and Zinc Concentration in spiking dilutions. Synergis

	Total Copper		Initial
	Initial (ug/L)	Final (ug/L)	
Mod Water ug/l	<1	1.24	
Site Water ug/l	5.58	5.9	
Mod Water - 30Cu + 200Zn ug/l	28.1	76.5	
Mod Water - 20Cu + 130Zn ug/l	18	15.4	
Mod Water - 13Cu + 85Zn ug/l	12	10	
Mod Water - 8Cu + 55Zn ug/l	7.98	7.45	
Mod Water - 5Cu + 36Zn ug/l	6.11	5.53	
Site Water - 385Cu + 278Zn ug/l	377	451	
Site Water - 250Cu + 181Zn ug/l	218	198	
Site Water - 163Cu + 117Zn ug/l	162	150	
Site Water - 106Cu + 76Zn ug/l	103	109	
Site Water - 69Cu + 50Zn ug/l	76.5	73.6	

\*RPD = Relative percent difference between initial and dissolved metal.

## **Dissolved Translator Analytical Summary**

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City of Broken Bow  
 4040-12-050  
 Dissolved Translator Analytical Summary

Effluent Results (001)

	Copper (mg/L)		Lead (mg/L)		Zinc (mg/L)	
	Total	Dissolved	Total	Dissolved	Total	Dissolved
2/19/2013	0.00681	0.00431	0.001	0.001	0.0278	0.0268
6/25/2013	0.00521	0.00322	0.001	0.001	0.00487	0.002
6/26/2013	0.00536	0.0031	0.001	0.001	0.00746	0.002
8/6/2013	0.0055	0.0049	0.001	0.001	0.018	0.015
9/30/2013	0.00372	0.00252	0.001	0.001	0.0278	0.025
10/1/2013	0.00368	0.0025	0.001	0.001	0.0274	0.0262
11/19/2013	0.00772	0.00256	0.001	0.001	0.0277	0.0254
3/18/2014	0.00622	0.00476	0.001	0.001	0.032	0.0287
3/24/2014	0.00555	0.00438	0.001	0.001	0.0325	0.0276
3/25/2014	0.00525	0.00407	0.001	0.001	0.0296	0.0254

\*The RL is used for results with a value of "non-detect".

QAQC - Duplicates (001-D)

	Copper (mg/L)		Lead (mg/L)		Zinc (mg/L)	
	Total	Dissolved	Total	Dissolved	Total	Dissolved
2/19/2013	0.00707	0.00484	0.001	0.001	0.0289	0.0268
6/25/2013	0.00545	0.00301	0.001	0.001	0.00952	0.002
6/26/2013	0.00526	0.00303	0.001	0.001	0.00601	0.002
8/6/2013	0.0059	0.0037	0.001	0.001	0.019	0.014
10/1/2013	0.00348	0.00239	0.001	0.001	0.0253	0.0248
11/19/2013	0.00405	0.00249	0.001	0.001	0.0235	0.0236
3/18/2014	0.0582	0.0439	0.001	0.001	0.0301	0.028
3/24/2014	0.00537	0.00424	0.001	0.001	0.0305	0.0267
RPD (%)	3.75	11.58	0.00	0.00	3.88	0.00
RPD (%)	4.50	-6.74	0.00	0.00	64.63	0.00
RPD (%)	-1.88	-2.28	0.00	0.00	-21.53	0.00
RPD (%)	7.02	-27.91	0.00	0.00	5.41	-6.90
RPD (%)	-5.59	-4.50	0.00	0.00	-7.97	-5.49
RPD (%)	-62.36	-2.77	0.00	0.00	-16.41	-7.35
RPD (%)	161.38	160.87	0.00	0.00	-6.12	-2.47
RPD (%)	-3.30	-3.25	0.00	0.00	-6.35	-3.31

QAQC - Field Blanks (FB)

	Copper (mg/L)		Lead (mg/L)		Zinc (mg/L)	
	Total	Dissolved	Total	Dissolved	Total	Dissolved
2/19/2013	< 0.001	< 0.001	< 0.001	< 0.001	< 0.002	< 0.002
6/25/2013	< 0.001	< 0.001	< 0.001	< 0.001	0.017	0.0183
9/30/2013	< 0.001	< 0.001	< 0.001	< 0.001	0.00781	0.00496
3/18/2014	< 0.001	< 0.001	< 0.001	< 0.001	0.0131	0.0136
3/25/2014	< 0.001	< 0.001	< 0.001	< 0.001	0.00298	0.00277

QAQC - Trip Blanks (TB)

	Copper (mg/L)		Lead (mg/L)		Zinc (mg/L)	
	Total	Dissolved	Total	Dissolved	Total	Dissolved
2/19/2013	< 0.001	< 0.001	< 0.001	< 0.001	< 0.002	< 0.002
6/25/2013	< 0.001	< 0.001	< 0.001	< 0.001	0.00955	0.01
9/30/2013	< 0.001	< 0.001	< 0.001	< 0.001	0.00744	0.00653

Dissolved Translator

	Copper	Lead	Zinc
	(Dissolved/Total)	(Dissolved/Total)	(Dissolved/Total)
2/19/2013	0.63	1.00	0.96
6/25/2013	0.62	1.00	0.41
6/26/2013	0.58	1.00	0.27
8/6/2013	0.89	1.00	0.83
9/30/2013	0.68	1.00	0.90
10/1/2013	0.68	1.00	0.96
11/19/2013	0.33	1.00	0.92
3/18/2014	0.77	1.00	0.90
3/24/2014	0.79	1.00	0.85
3/25/2014	0.78	1.00	0.86
Sum	6.74	10.00	7.85
Average	0.67	1.00	0.79

**Appendix C**

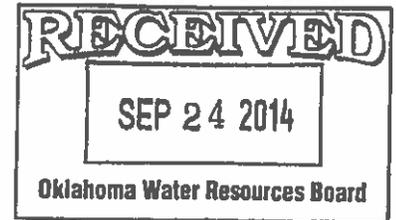
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**Laboratory Reports**



September 15, 2014

Jason Childress  
Environmental Programs Manager  
Water Quality Standards Section  
Oklahoma Water Resources Board  
3800 North Classen Boulevard  
Oklahoma City, OK 73118  
(405) 530-8800



Re: Response to EPA Region 6 Technical Comments on Final Report for:  
Broken Bow Public Works Authority- *Water Effects Ratio and Dissolved Translator Study*  
GBM<sup>c</sup> No. 4040-12-050

Dear Mr. Childress:

This letter contains our response to EPA Region 6 Technical Comments concerning the Broken Bow Public Works Authority - *Water Effects Ratio and Dissolved Translator Study* Final Report dated August 1, 2014. The bullets below correspond to comments provided by EPA.

### Comments Specific to Zinc

1. Initiation of the October 2013 *Ceriodaphia dubia* and November 2013 *Pimephales promelas* tests occurred approximately 50 hours after sample collection. While samples were delivered to the laboratory in adequate time to meet the 36 hour timeframe recommended in the 1994 Interim Guidance, the tests were not started immediately, due to workload at the laboratory. While we realize this test is outside of the stated holding time for the zinc WER, the Streamlined Method for copper provides for a 96 hour holding time as the tests are not intended for measurement of whole effluent toxicity. It is reasonable to assume this rationale would apply to zinc as well. Samples were held below 6°C until initiation of the test to preserve effluent integrity.
2. As noted in the comments, the June 25, 2013 zinc WER was noticeably lower than the other 3 WERs used in calculating the fWER. Elevated ammonia and TSS concentrations in the effluent are factors that may have contributed to the lower WER value. However, this is not certain and additional (unknown) factors may have played a role as well. It was noted throughout the study that the zinc LC50s in the lab water tests varied noticeably. Neither GBM<sup>c</sup> nor the City has additional information about the lower zinc WER.
3. While the guidance suggests the secondary species WER should be used to confirm the results of the primary species it is unclear if the data should be used in the calculation of the fWER. Due to the similar results as noted in comments provided by the EPA, we request the inclusion of these test results in the fWER.
4. The incorrect date in Table 14 has been corrected in the revised Final Report.

**Comments for Both Copper and Zinc**

5. Based upon the comments provided, it is assumed that the drop in dissolved oxygen concentration in the October 2013 WER test is not viewed as problematic to the test results or calculation of the fWER. As noted by EPA, organism response was not affected and a drop in D.O. was not observed in other test WERs.
6. The tests were conducted using an incubator and water bath which are held at 25°C and continuously monitored. The laboratory does not note temperature data in their reports unless a temperature issue develops during the test.
7. The TSS value for 11/19/13 in the Water Quality Data summary table in Appendix B of the report was a typo and has been revised as appropriate.
8. Initial laboratory reports provided different metals concentrations for one of the treatments in the synergistic tests. A revised lab report has been issued and the report revised as appropriate.
9. Calculations have been checked and revised to include four significant digits.
10. Appropriate revisions have been made to the final report to create site-specific total acute and chronic copper and zinc criteria.
11. Thank you for providing the additional considerations for future WER workplans.

Necessary edits based on the provided comments have been made to the Final Report for the Broken Bow WER Study. Revised copies of the report are included for your consideration. If further clarification or additional information is needed please do not hesitate to contact me, Greg Phillips, or Shon Simpson at (501) 847-7077.

Respectfully submitted,  
GBM<sup>c</sup> & ASSOCIATES



Jonathan Brown  
Environmental Scientist