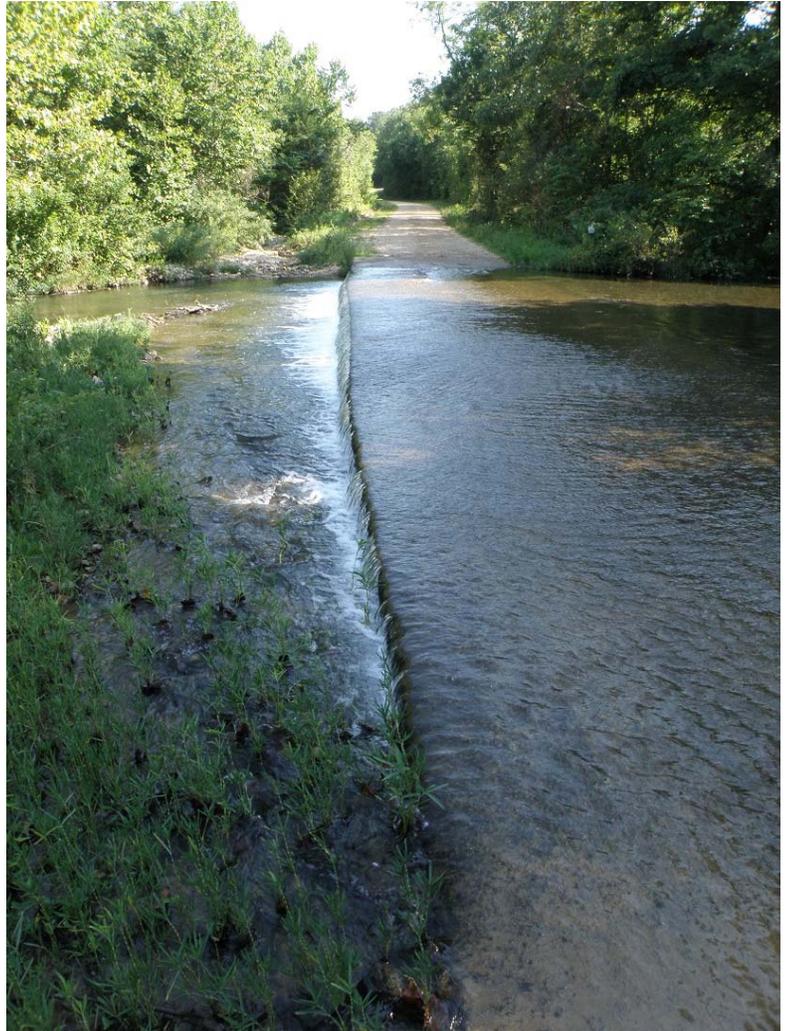


BUICK BIOTREATMENT PILOT TEST FINAL RESULTS REPORT

August 13, 2013



PREPARED FOR:

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1. EXECUTIVE SUMMARY

On September 13, 2012, Doe Run sought approval from the Missouri Department of Natural Resources (MDNR) Water Protection Program to pursue a pilot biotreatment project at its Buick Mine. On September 27, 2012 Doe Run received conditional approval from MDNR to conduct the pilot test. In October of 2012, Resource & Environmental Management Consultants, Inc. (REMC) initiated the biotreatment pilot test at the Buick Mine. Appendix A shows the location of the pilot test and presents plan and cross-section views of the system components.

The Buick pilot biotreatment system was effective at reducing metals concentrations to below proposed final Missouri State Operating Permit effluent limits for metals. The pilot biotreatment system did not pass a chronic Whole Effluent Toxicity test. Pilot test results indicate that effective management of influent TSS is required to prevent clogging of the organic substrate by fine particulates.

2. INTRODUCTION

Resource Environmental Management Consultants, Inc. (REMC) has prepared this report for the Doe Run Resources Corporation (Doe Run) to document the final results of the biotreatment pilot test conducted at the Buick Mine (Buick). Construction of the Buick pilot biotreatment system was completed in late October 2012 and laboratory data collection began in late November 2012. The biotreatment pilot test concluded in June 2013.

2.1 PILOT TEST DESIGN AND CONSTRUCTION

The Buick Mine pilot biotreatment cell (biocell) consisted of an approximately 190 cubic yard excavation into native gravelly clay soils. The in-ground excavation was surrounded by a 1-foot tall and 2-foot wide compacted soil berm. The purpose of the berm was solely to prevent stormwater run-on into the biocell. The water level in the biocell was set to the surrounding ground level and thus the berm was not a structural component of the biocell and did not serve to contain the biocell's water volume. The biocell was lined with a 60 mil HDPE liner. Water exited the biocell through a 4-inch perforated HDPE pipe. Just prior to exiting the biocell, the perforated pipe was connected to a solid 4-inch HDPE pipe that was fused to the HDPE liner to ensure a water-tight seal around the pipe.

The interior width of the biocell at the inside toe of the berm was 40 feet square. The sides of the biocell sloped at 2:1 H:V towards the bottom of the biocell. The width of the bottom of the biocell was 14 feet square and the total depth of the biocell below ground surface was 6.5 feet. The biocell contained a 6-inch layer of drainage gravel in the bottom, which was overlain by a 1-foot layer of sand and a 4.5-foot layer of organic substrate. Additionally, the bottom of the biocell was sloped 1% towards the effluent pipe to promote drainage. The organic substrate consisted of approximately 60% wood chips, 30% sawdust and 10% chopped hay (by volume).

Water was supplied to the biocell via a 1 HP electric pump. The pump drew water from the Buick meander system below the Buick tailings dam. Water was pumped to the biocell through a 2-inch PVC pipe. An electronic flow meter was installed on the influent pipe to allow accurate flow measurement. Just prior to entering the biocell, the 2-inch piping transitioned to 4-inch piping. Inflow to the top of the biocell occurred via eight 4-inch perforated HDPE laterals spaced evenly across the top of the biocell. The perforated HDPE laterals were bedded into the upper six inches of the organic substrate. Water flowed through the biocell in a downflow configuration with the water level set by a P-trap in the effluent piping outside the biocell.

After exiting the biocell, effluent water flowed to a 5-foot wide and 100-foot long open rock channel (rock filter) to oxidize residual sulfide and increase dissolved oxygen. The rock filter channel was lined with 60 mil HDPE and 3-inch minus limestone rock. Effluent from the rock filter reentered the Buick meander system approximately ten feet downstream of the pump intake.

The biocell's design flow rate was ten gallons per minute (gpm). Photos of the system components are presented below. Complete as-built construction drawings are presented in Appendix A.



Biotreatment cell.



Effluent discharging into rock filter.



Intake pump and effluent pipe in Buick meander.

2.2 PILOT TEST ANALYTICAL PARAMETERS

Operational samples were collected weekly from the influent water, direct effluent from the biocell and effluent from the rock filter. Laboratory analysis was conducted by the Doe Run SEMO Central Laboratory in Viburnum, Missouri, with the exception of 5-day biological oxygen demand (BOD5) and ammonia. Analysis for BOD5 and ammonia was conducted by Pace Analytical Services in Lenexa, Kansas.

Field Parameters

- pH;
- Temperature;
- Conductivity;
- Oxidation-reduction potential (ORP);
- Dissolved oxygen;
- Flow; and
- Weather and general conditions.

Laboratory Parameters - Metals

- Arsenic (total and dissolved);
- Cadmium (total and dissolved);
- Copper (total and dissolved);
- Lead (total and dissolved);
- Nickel (total and dissolved); and
- Zinc (total and dissolved);

Laboratory Parameters – Other

- Alkalinity;
- Ammonia;
- Biological Oxygen Demand, 5-day (BOD5);
- Chloride;
- Nitrate;
- Sulfate;
- Sulfide;
- Total organic carbon (TOC);
- Total suspended solids (TSS); and
- Chronic Whole Effluent Toxicity (WET).

2.3 PILOT TEST REVIEW

- **Winter Start-Up** – Start-up of the biocell occurred during winter. Initial sulfide production was low at the design flow rate of 10 gpm and resulted in erratic copper and lead data (Figures 3-9 and 3-10, respectively). This condition was managed by reducing the influent flow rate by half for approximately two weeks, then slowly increasing the flow rate in a step-wise fashion back to 10 gpm. The results in Sections 3.2 and 3.3 demonstrate that this remedy was highly successful in boosting sulfide production and eliminating erratic lead and copper results.
- **Influent TSS** – Rainfall events and maintenance work in the area surrounding the Buick tailings impoundment seep basin upstream of the pilot system pump intake resulted in a relatively significant spikes in influent TSS from mid-February through early May (Figure 3-21).

The relatively high TSS influent clogged pore space in the organic substrate and impacted throughput in the biocell (Figure 3-6). Earlier than expected routine maintenance was required to restore the substrate's hydraulic conductivity. The maintenance activities included mechanical agitation of the substrate and backflushing of the biocell. Several maintenance events were required to restore full throughput in the biocell.

- **Excess Sulfide** – Due to the TSS/throughput issues discussed above and rising water temperatures, the biocell produced excessive levels of residual sulfide from mid-April through the end of May. Sulfide concentrations of 5-10 mg/L in biocell effluent are considered ideal, but during this period residual sulfide peaked at 38 mg/L (Figure 3-19). Sulfide concentrations in rock filter effluent increased from the November-March mean of 0.46 mg/L to as high as 17.3 mg/L on May 17. Once normal biocell throughput was restored residual sulfide concentrations declined to normal levels (Figure 3-19).

3. PILOT TEST DATA

The following sections present graphs and discussion of pilot test results. Data collection began on November 29, 2012 and concluded on June 11, 2013. Appendix B contains the complete dataset from the test. Legend labels in the graphs represent the following three monitoring points:

- Buick Inf – Influent to the biocell from the Buick meander;
- Buick Eff – Effluent from the biocell; and
- Buick RF Eff – Effluent from the rock filter.

3.1 FIELD DATA

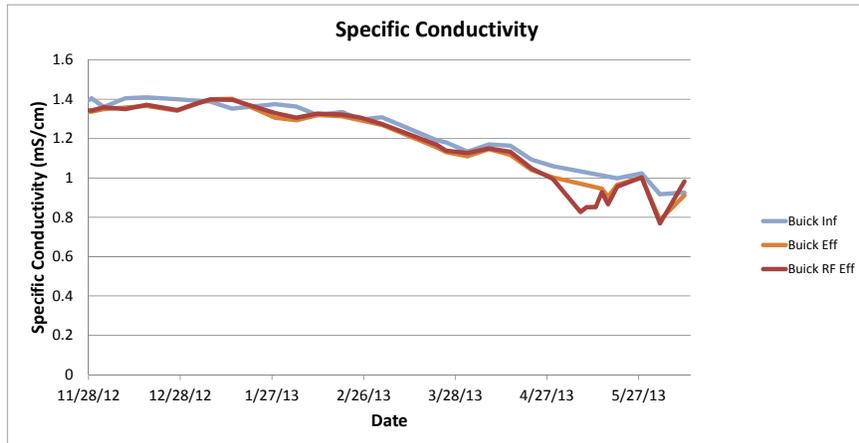


Figure 3-1: Specific Conductivity Data

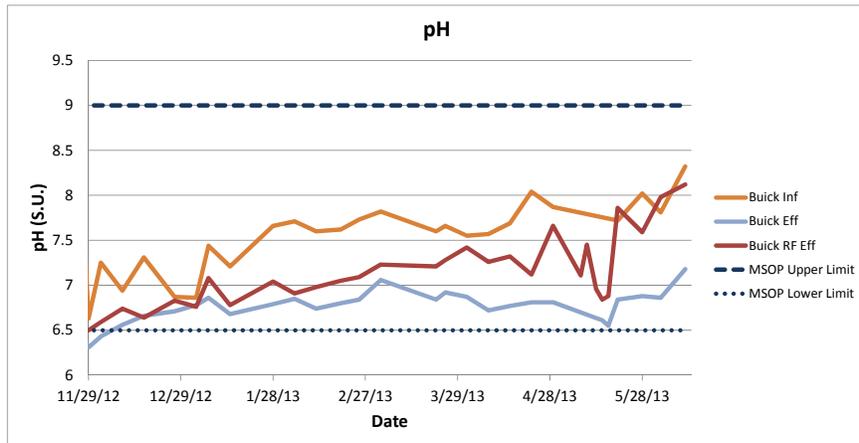


Figure 3-2: pH Data

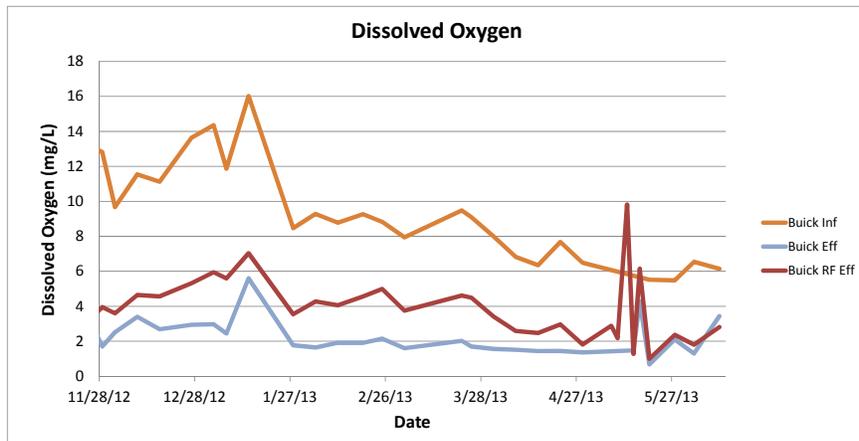


Figure 3-3: Dissolved Oxygen Data

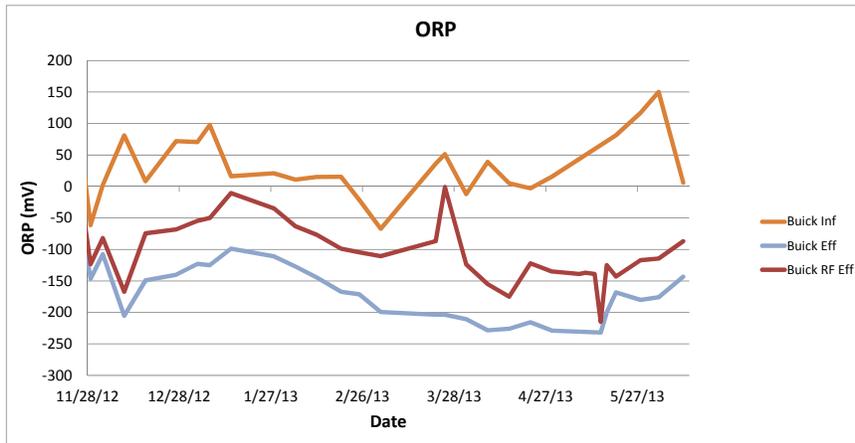


Figure 3-4: ORP Data

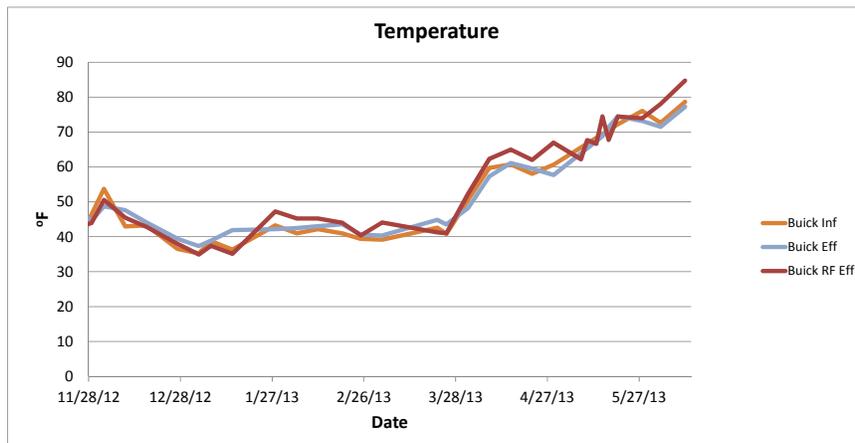


Figure 3-5: Temperature Data

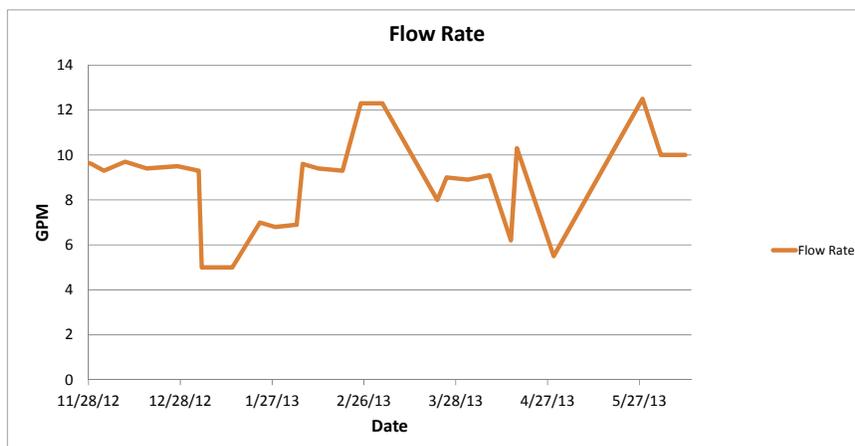


Figure 3-6: Flow Data

3.2 LABORATORY DATA – METALS

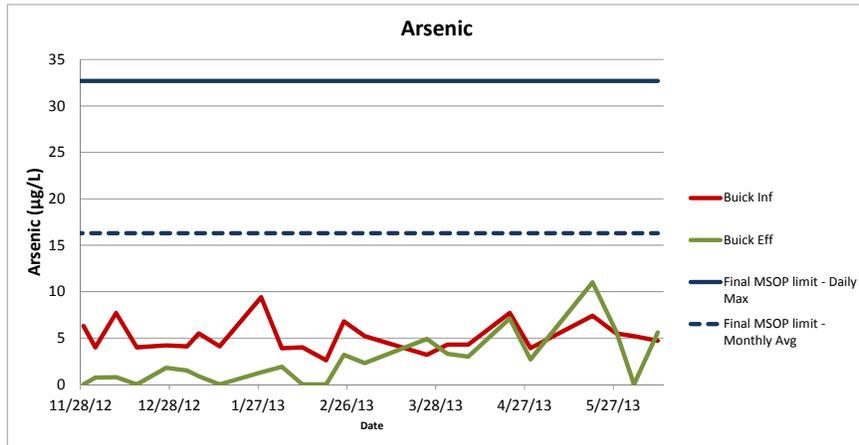


Figure 3-7: Arsenic Data

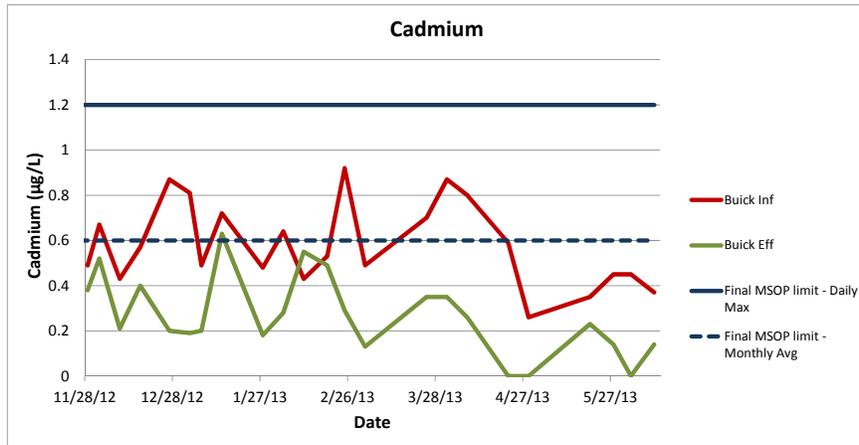


Figure 3-8: Cadmium Data

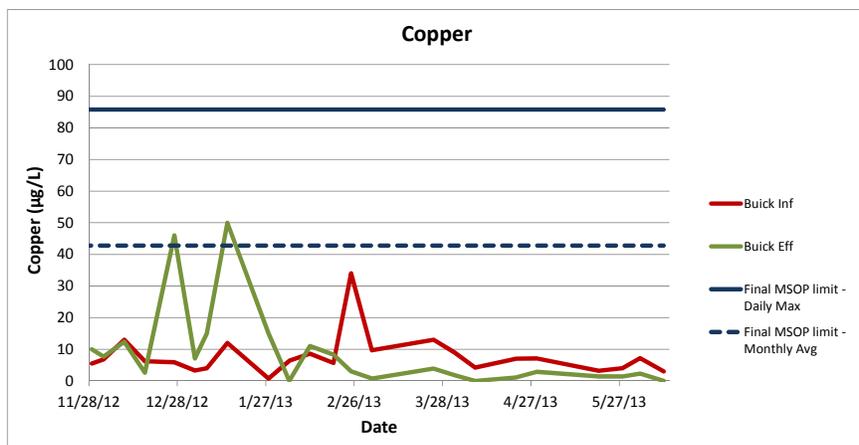


Figure 3-9: Copper Data

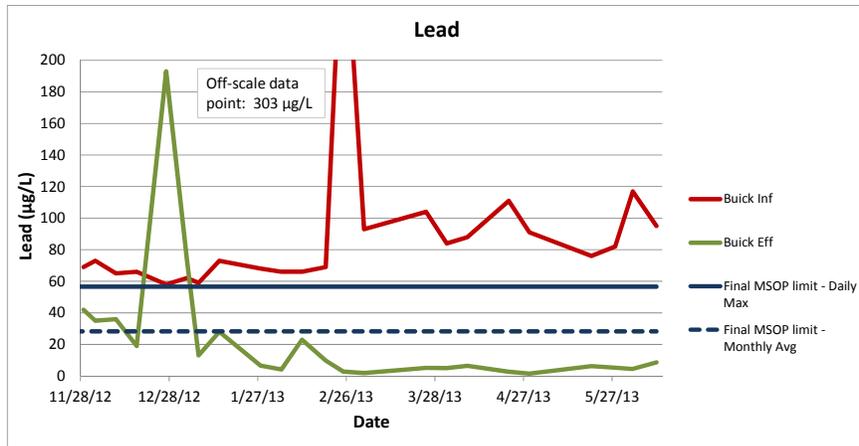


Figure 3-10: Lead Data

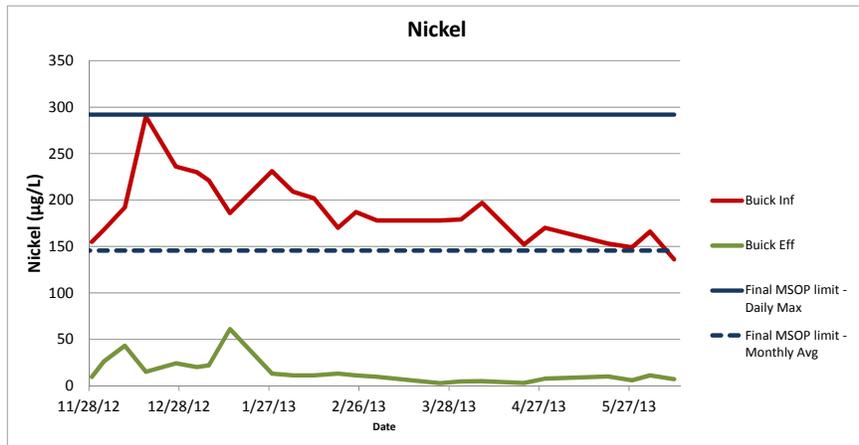


Figure 3-11: Nickel Data

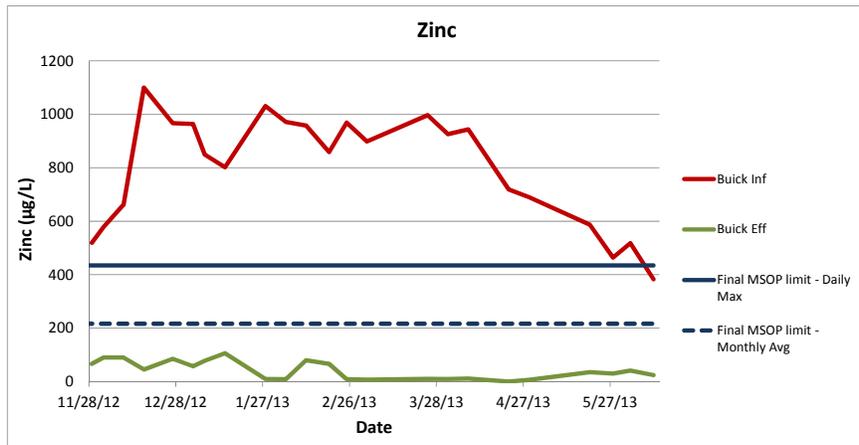


Figure 3-12: Zinc Data

3.3 LABORATORY DATA – OTHER

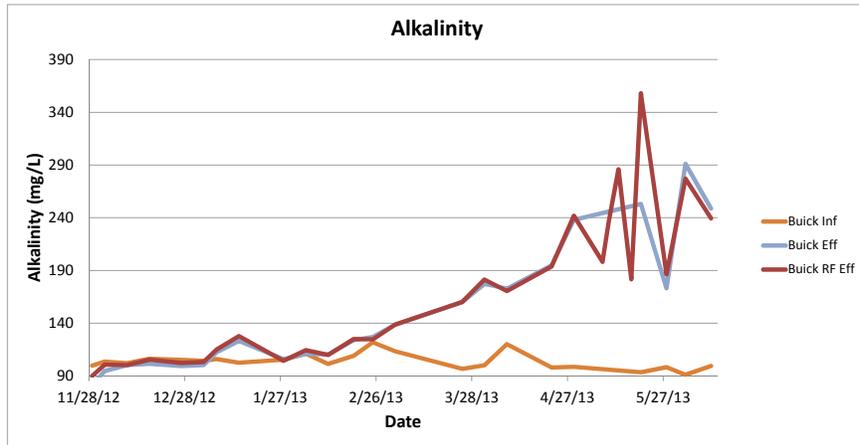


Figure 3-13: Alkalinity Data

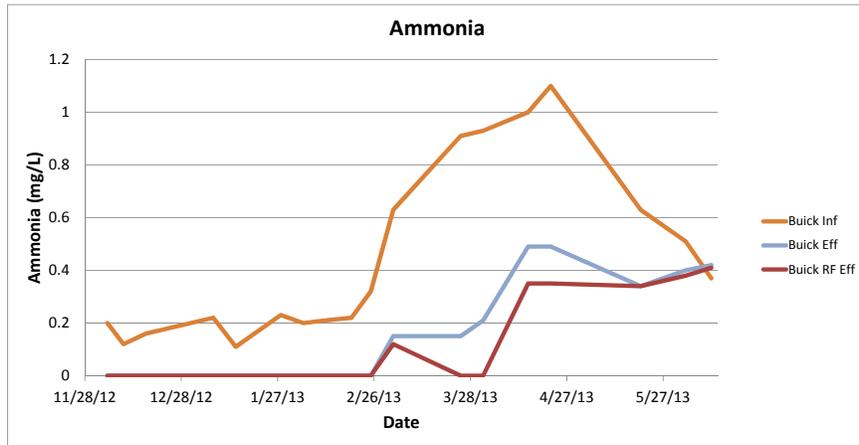


Figure 3-14: Ammonia Data

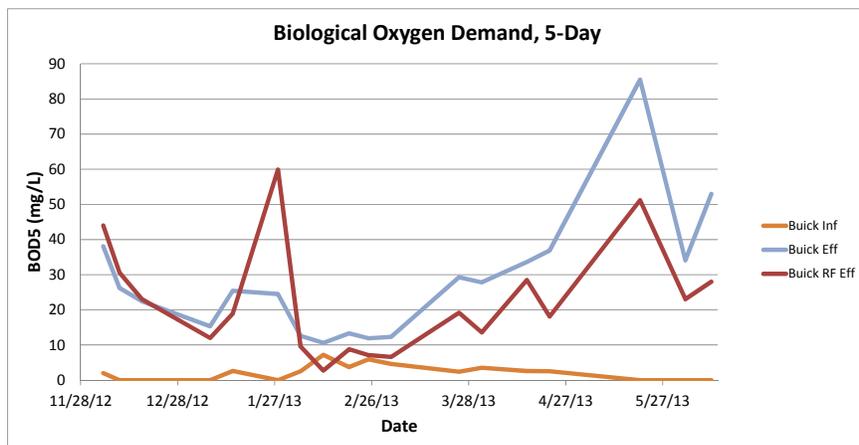


Figure 3-15: BOD5 Data

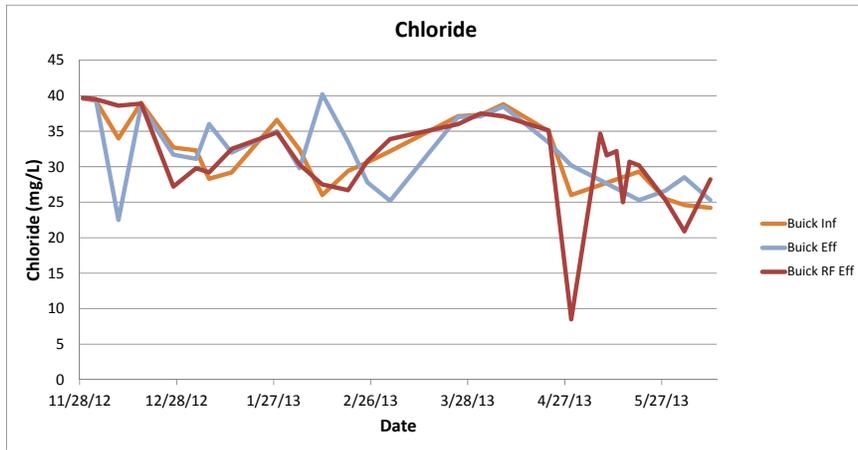


Figure 3-16: Chloride Data

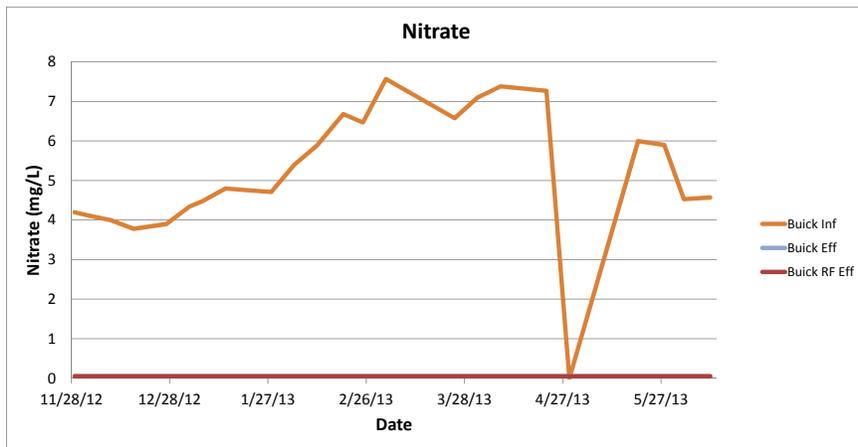


Figure 3-17: Nitrate Data

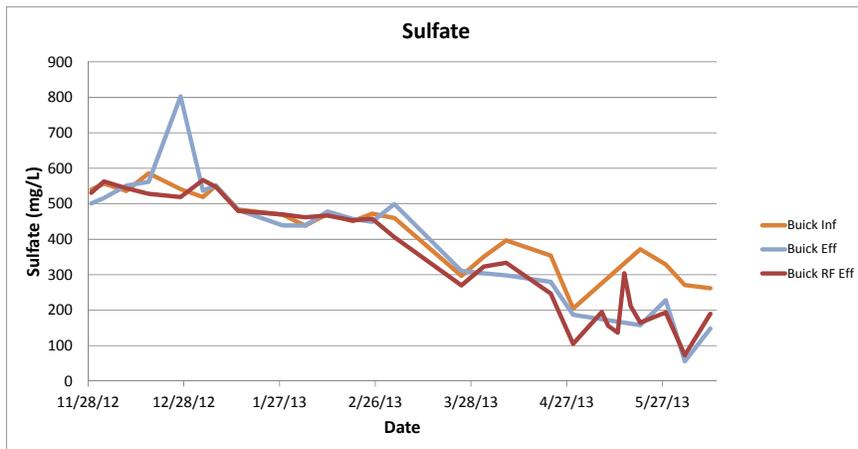


Figure 3-18: Sulfate Data

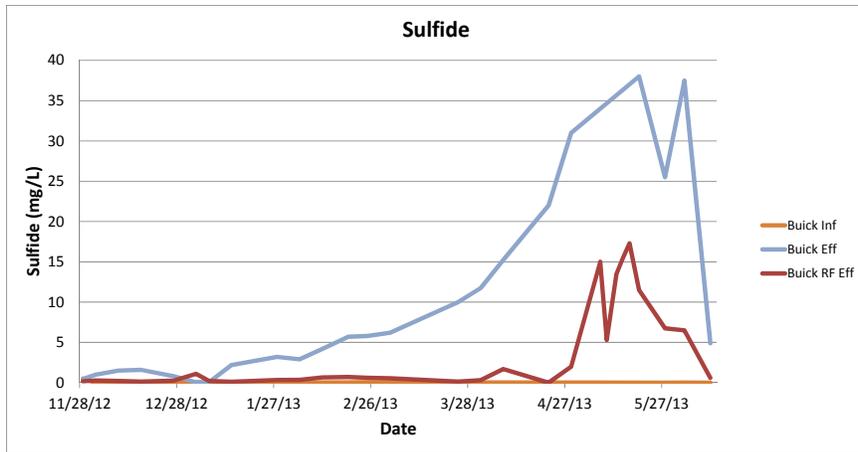


Figure 3-19: Sulfide Data

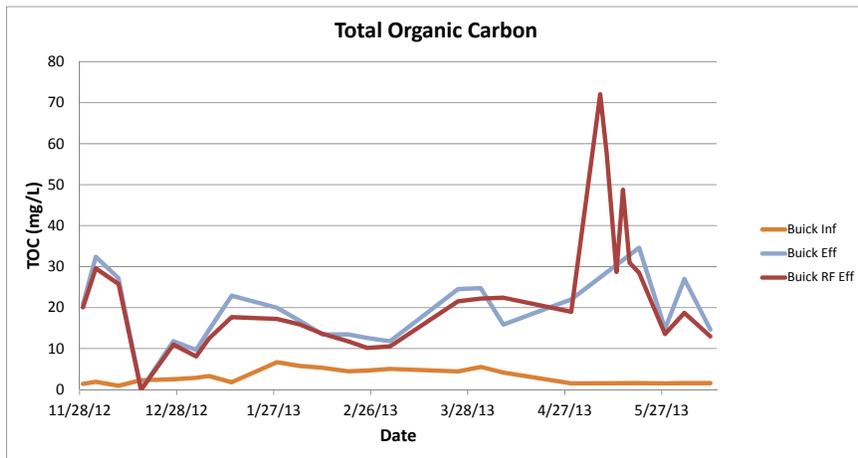


Figure 3-20: TOC Data

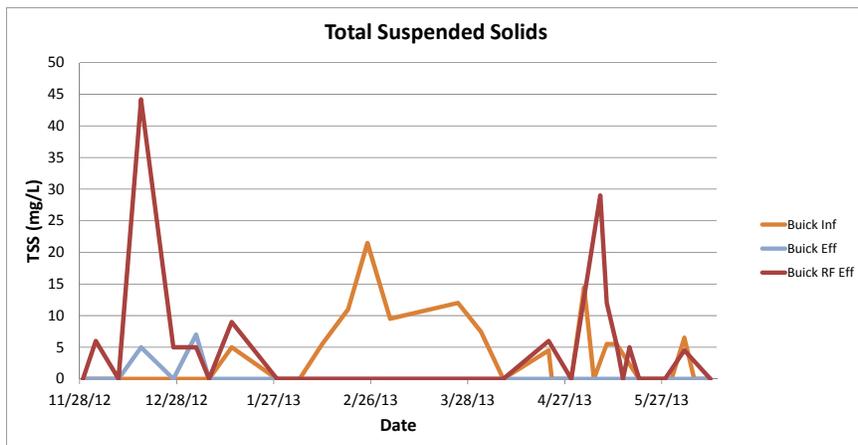


Figure 3-21: TSS Data

3.4 RESULTS DISCUSSION

Field Parameters

- pH – Biocell effluent pH was slightly acidic, with a mean of 6.80 S.U. which is within MSOP effluent limits for pH. The pH of rock filter effluent was consistently higher than biocell effluent and generally remained >7.0 S.U. after mid-February 2013. The pH increase in the rock filter is likely a combined result of:
 - Dissolution of limestone rock used in the rock filter by the mildly acidic biocell effluent; and
 - The conversion of bisulfide ions to gaseous hydrogen sulfide as residual sulfide is oxidized within the rock filter. This process consumes H^+ ions in the following reaction: $HS^- + H^+ \rightarrow H_2S$
- Temperature – Temperature data shows that influent water temperatures were very cold during winter operation. Mean influent water temperature from November 29, 2012 to March 25, 2013 was 41° F, and the minimum recorded influent water temperature was 35° F.
- Conductivity – No significant differences have been observed between influent and effluent conductivity.
- ORP – Mean ORP for the biocell influent and effluent was 29 mV and -156 mV, respectively. The mean effluent ORP is within the ideal range. Mean ORP for rock filter effluent was -96 mV. The magnitude of ORP increase between the biocell effluent and rock filter effluent sampling locations was consistent throughout the test period.
- Dissolved oxygen – Mean dissolved oxygen for the biocell influent and effluent was 9.55 mg/L and 2.18 mg/L, respectively. The mean effluent dissolved oxygen is within the ideal range. Mean dissolved oxygen for rock filter effluent was 3.85 mg/L. The magnitude of dissolved oxygen increase between the biocell effluent and rock filter effluent sampling locations was consistent through late March 2013 and then decreased through the end of test period. This is likely the result of increased water temperatures.
- Flow – Flow data shows the initial flow reduction and step-wise increases described in Section 2.3. Later flow reductions are the result of the TSS issues described in Section 2.3. Also note in Figure 3-6 that prior to the TSS challenges the biocell was operating at 23% above the design flow rate of 10 gpm and removing >90% of copper, lead, nickel and zinc.

Laboratory Parameters - Metals

- Arsenic – Influent arsenic concentration were below final MSOP effluent limits throughout the test. Mean removal efficiency for arsenic was 58.1%.

- Cadmium – One biocell effluent sample slightly exceeded the monthly average final MSOP effluent limit for cadmium. All other results were below final effluent limits. After April 22, 2013, influent cadmium concentrations did not exceed the monthly average final MSOP effluent limit. Mean removal efficiency for cadmium was 58.5%.
- Copper – Influent copper concentration were below final MSOP effluent limits throughout the test. Two biocell samples slightly exceeded the monthly average final MSOP effluent limit for copper, but these results are considered anomalies from the start-up phase (see Section 2.3). Mean removal efficiency for copper was 79.6%.
- Lead – All lead results after January 7, 2013 were below final MSOP effluent limits. Exceedances of final effluent limits for lead prior to January 7 are considered anomalies from the start-up phase (see Section 2.3). Mean removal efficiency for lead was 89.6%.
- Nickel – All nickel results were well below final MSOP effluent limits throughout the test. Mean removal efficiency for nickel was 92.1%.
- Zinc – All zinc results were well below final MSOP effluent limits throughout the test. Mean removal efficiency for zinc was 94.5%.

Laboratory Parameters – Other

- Alkalinity – Alkalinity production increased significantly over the course of the test. This is an excellent indicator of bacterial activity and sulfate reduction within the biocell.
- Ammonia – Influent ammonia concentrations were relatively constant from November 2012 to February 2013, increased by 400% between late February and late April 2013, then dropped by 66% between late April and June 11, 2013. The cause of this variation is unknown. The biocell significantly reduced ammonia concentrations throughout most of the test, but ammonia removal stopped between April 22 and June 11. The cause of the loss of ammonia removal is unknown.
- BOD5 – BOD5 concentrations were generally inversely correlated with flow rate. The TSS/throughput issues discussed in Section 2.3 resulted in significant spikes in BOD5. It is expected that BOD5 levels would have declined with extended operation at the design flow rate.
- Chloride – No significant differences were observed between influent and effluent chloride concentrations.
- Nitrate – The biocell completely removed nitrate from influent water throughout the test. Influent nitrate concentrations ranged from 3.78-7.57 mg/L with a mean of 5.44 mg/L. All biocell effluent nitrate data has been below laboratory detection limits of 0.1 mg/L.

- Sulfate – Excluding one anomalous data point from December 27, 2012, mean sulfate concentrations in the biocell effluent were 48 mg/L lower than influent concentrations. This indicates strong bacterial activity and sulfate reduction.
- Sulfide – Sulfide data indicates that the biocell substrate was easily able to produce sufficient sulfide for high rates of metals removal even during winter operation. Excessive levels of residual sulfide occurred when throughput through the biocell was below the design flow rate.
- TOC – TOC concentrations were generally inversely correlated with flow rate. The TSS/throughput issues discussed in Section 2.3 resulted in significant spikes in TOC. It is expected that TOC levels would have declined with extended operation at the design flow rate.
- TSS – Influent TSS spikes seen in Figure 3-21 are discussed in Section 2.3. Spikes in TSS effluent seen in biocell and rock filter effluent were all the direct result of substrate maintenance required to correct throughput problems resulting from influent TSS spikes.
- Chronic WET – Two chronic WET tests were conducted with effluent from the rock filter. Effluent for the first test was sampled between May 6 and May 10, and effluent for the second test was sampled between May 13 and May 17. Only the *Pimephales promelas* portion of the first test had acceptable results (Toxic Units <1.6 TUc). Chronic WET test results are summarized below in Table 3-1.

Test Dates	Toxic Units (P. promelas)	Toxic Units (C. dubia)	Toxic Unit Limit
May 6-May 10	<1 TUc	2.92 TUc	1.6 TUc
May 13-May 17	5.15 TUc	2.78 TUc	1.6 TUc

Table 3-1: Chronic WET Test Results

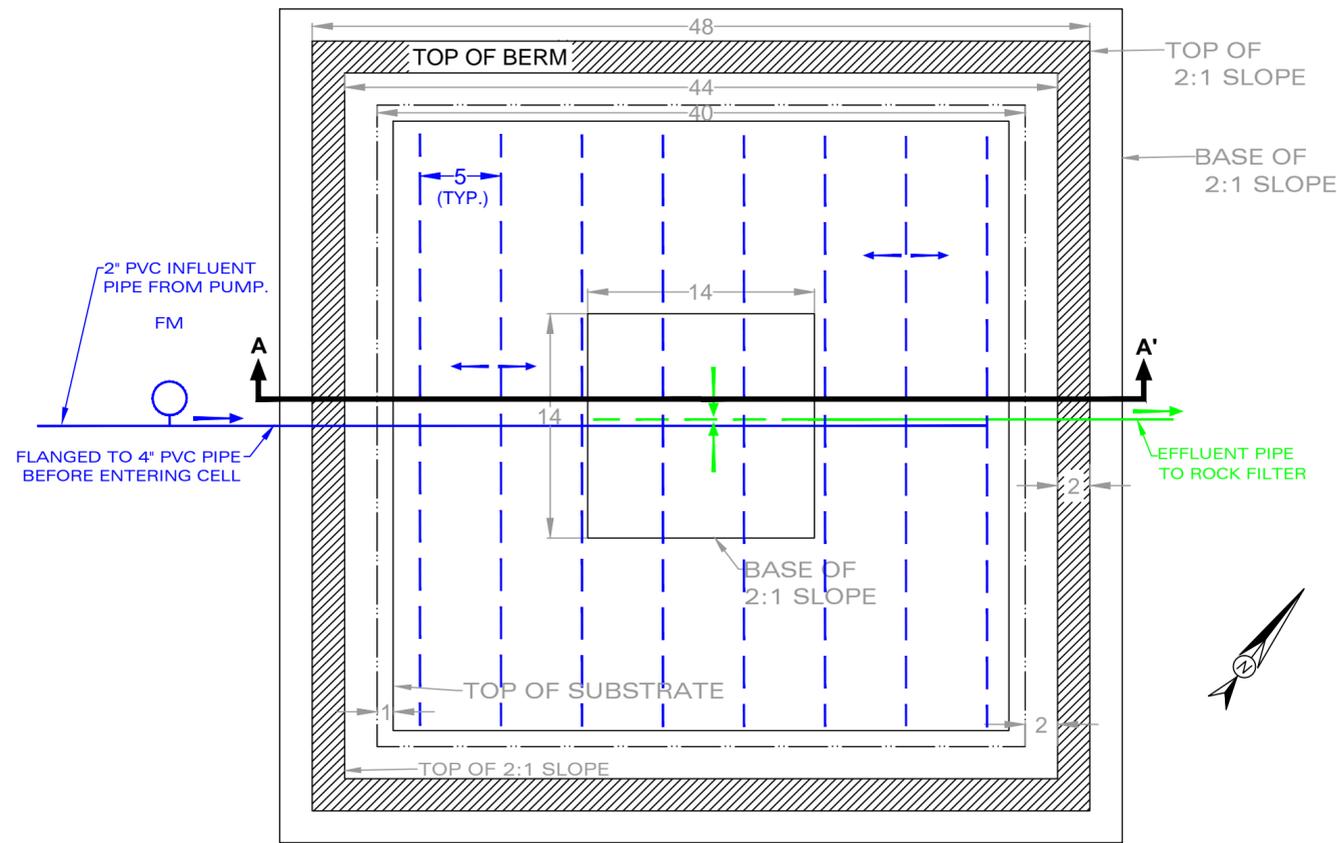
4. CONCLUSIONS

The Buick pilot biotreatment system was effective at reducing metals concentrations to below proposed final MSOP effluent limits. The pilot biotreatment system did not pass a chronic WET test. Pilot test results indicate that effective management of influent TSS is required to prevent clogging of the organic substrate by fine particulates. WET testing results may also provide Doe Run with challenges when managing a biotreatment system. These challenges may not lend to a full-scale biotreatment plant at the Buick Mine/Mill facility.

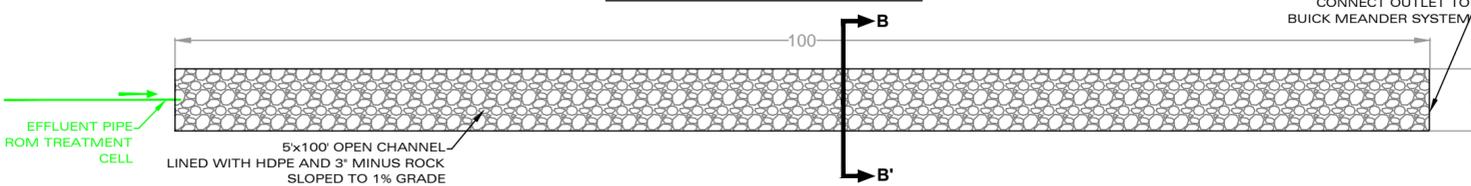
APPENDIX A

Location Map and As-Built Drawings

TREATMENT CELL PLAN VIEW



ROCK FILTER PLAN VIEW



LEGEND

- INFLUENT PIPING SOLID
- INFLUENT PIPING PERFORATED
- INFLUENT FLOW DIRECTION
- EFFLUENT PIPING SOLID
- EFFLUENT PIPING PERFORATED
- EFFLUENT FLOW DIRECTION
- WATER SURFACE ELEVATION
- FLOW METER

NOTES:
 1. ALL DIMENSIONS FEET UNLESS OTHERWISE NOTED.
 2. EFFLUENT SYSTEM IS PORTRAYED OFF-CENTER TO SHOW DETAILS.

UPON REVIEW OF AND MODIFICATION TO THIS DESIGN PACKAGE (SHEETS 1, 2 AND 3), I HEREBY CERTIFY TO THE FOLLOWING:

- ALTHOUGH NO MDNR DESIGN STANDARDS EXIST FOR SUCH A SYSTEM, THIS DESIGN IS CONSISTENT WITH MDNR'S WATER PROTECTION PROGRAM DESIGN GUIDES FOR SMALL SEWAGE WORKS (10 CSR 20-8.020) FOR THE KEY COMPONENTS OF POND SEAL AND POND DIKE STABILITY;
- THE DESIGN PACKAGE MEETS THE STANDARD OF GOOD ENGINEERING PRACTICE FOR SYSTEMS OF THIS SIZE, SCOPE AND DURATION, AND;
- AS VERBALLY REPORTED TO ME BY RESOURCE MANAGEMENT CONSULTANTS' FIELD PERSONNEL WHO PERSONALLY INSPECTED THE SYSTEM DURING CONSTRUCTION, THE BIOTREATMENT SYSTEM WAS INSTALLED IN GENERAL ACCORDANCE WITH THE DESIGN PACKAGE, WITHIN NORMAL CONSTRUCTION TOLERANCES, AND NO SIGNIFICANT DEVIATIONS WERE NOTED.

PAUL ANDREW HAGERTY
 MISSOURI PROFESSIONAL ENGINEER (PE-2003004648)

NOTES/DETAILS

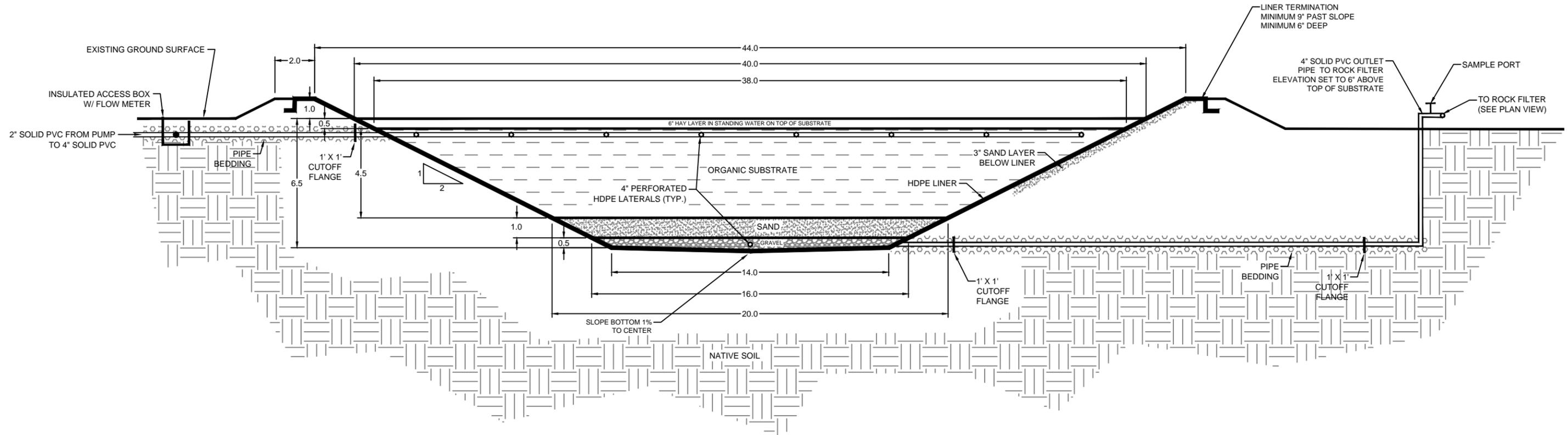
- 1) **DESCRIPTION** - THIS DESIGN APPLIES TO THE INSTALLATION OF A SMALL SCALE PILOT BIOTREATMENT SYSTEM AT THE BUICK MINE. THE MAIN COMPONENTS OF THE PILOT SYSTEM INCLUDE A LINED BIOTREATMENT CELL (52' X 52' X 7.5'), A LINED ROCK FILTER (100' X 5' X 1') AND ASSOCIATED PIPING, VALVES AND FITTINGS. THE PURPOSE OF THE PROJECT IS TO EVALUATE THE EFFICACY OF BIOTREATMENT, ESPECIALLY DURING THE WINTER MONTHS. IT IS ANTICIPATED THAT THE PILOT PROJECT WILL OPERATE FOR APPROXIMATELY 8 MONTHS FOLLOWING CONSTRUCTION AND START-UP. NO MDNR DESIGN STANDARDS EXIST FOR SUCH A SYSTEM; HOWEVER, THIS DESIGN WAS PREPARED GENERALLY CONSISTENT WITH MDNR'S WATER PROTECTION PROGRAM DESIGN GUIDES FOR SMALL SEWAGE WORKS (10 CSR 20-8.020) FOR THE KEY COMPONENTS OF POND SEAL AND POND DIKE STABILITY. THE ANTICIPATED FLOW RATE OF THE PILOT SYSTEM IS APPROXIMATELY 10 GALLONS PER MINUTE.
- 2) **LOCATION** - LOCATION WAS SELECTED DUE TO TOPOGRAPHY, DEPTH OF SOIL ABOVE BEDROCK FOR EXCAVATION AND AVAILABILITY OF ACCESS. LOCATION RELEVANT TO 25 YEAR FLOOD LIMIT IS UNKNOWN. LOCATION IS GREATER THAN 300 FEET FROM ANY WELL OR WATER SUPPLY STRUCTURE. DEPTH TO GROUNDWATER IS UNKNOWN BUT IS GREATER THAN 6.5 FEET. GROUNDWATER WAS NOT ENCOUNTERED DURING CONSTRUCTION EXCAVATION TO 6.5 FEET BELOW GROUND SURFACE.
- 3) **POND DIKE CONSTRUCTION** - POND DIKES WILL BE CONSTRUCTED LARGELY VIA EXCAVATION BELOW EXISTING GRADE, AS OPPOSED TO A CUT/FILL BALANCE. AS SUCH, DIKE HEIGHTS WILL BE MINIMAL AND THE DIKE SYSTEM WILL FUNCTION PRIMARILY AS A HYDROLOGIC BARRIER TO STORMWATER RUNOFF INTO THE BIOCELL. THE FOLLOWING VARIANCES TO 10 CSR 20-8.020 APPLY TO THE DIKE CONSTRUCTION AND ARE APPROPRIATE CONSIDERING THE SMALL PROJECT SIZE AND SHORT OPERATIONAL TIMEFRAME:
 - SOIL COMPACTION - DUE TO THE LIMITED DIKE HEIGHT AND SMALL PROJECT SIZE, THE QUANTIFIABLE STANDARD (90% OF STANDARD PROCTOR) IS NOT NECESSARY. FIELD COMPACTION WILL BE ACHIEVED VIA TRACKING WITH MECHANICAL CONSTRUCTION EQUIPMENT TO A VISUALLY STABLE CONDITION.
 - DIKE WIDTH - TOP DIKE WIDTH WILL BE TWO FEET (2') AS OPPOSED TO THE DESIGN GUIDE STANDARD OF FOUR FEET (4'). FOUR FEET IS NOT NECESSARY AS THE BERM TOPS WILL NOT BE USED FOR VEHICULAR ACCESS OR TRAFFIC.
 - DIKE HEIGHT - TOP OF DIKE HEIGHT WILL BE ONE FOOT (1') ABOVE THE POND CELL AS OPPOSED TO THE DESIGN GUIDE STANDARD OF TWO FEET (2'). EXCESS FREEBOARD IS NOT NECESSARY AS THE BIOTREATMENT CELL WILL NOT BE ACCEPTING STORMWATER RUNON, AND WATER HEIGHT IN THE CELL WILL BE CONTROLLED VIA THE OUTLET PIPING.
 - DIKE SLOPES - INNER AND OUTER DIKE SLOPES WILL BE 2:1 (H:V) AS OPPOSED TO THE DESIGN GUIDE STANDARD OF 3:1. GIVEN THE SMALL DIKE HEIGHT AND NEED TO MAXIMIZE THE CELL CONTENTS WITHIN A SMALL FOOTPRINT, THE 2:1 SLOPE WILL BE NECESSARY AND WILL NOT CAUSE ADVERSE STABILITY OR SAFETY CONDITIONS.
- 4) **POND SEAL** - THE BIOTREATMENT CELL AND THE ROCK FILTER WILL BE LINED WITH A MINIMUM 20 MIL HIGH-DENSITY POLYETHYLENE (HDPE) GEOMEMBRANE. THIS GEOMEMBRANE WILL ENSURE POND SEAL TIGHTNESS AND WILL MEET THE PERMEABILITY, TIGHTNESS, DURABILITY AND INTEGRITY REQUIREMENTS OF THE DESIGN GUIDES. THE GEOMEMBRANE WILL BE INSTALLED IN ACCORDANCE WITH MANUFACTURERS RECOMMENDATIONS. ALL PIPE PENETRATIONS WILL BE COMPLETED USING A PRE-MADE OR FIELD-FABRICATED PIPE BOOT WITH WATERTIGHT SEAL. DUE TO THE LIMITED SIZE AND DURATION OF THE PROJECT, POST-PLACEMENT QUALITY ASSURANCE (QA) SEAM AND TIGHTNESS TESTING WILL NOT BE CONDUCTED ON THE PLACED GEOMEMBRANE.
- 5) **MATERIALS OF CONSTRUCTION** - AGGREGATE AND ORGANIC SUBSTRATE MATERIALS FOR THIS PROJECT ARE LISTED IN THE MATERIALS SCHEDULE TABLE ON THIS SHEET.
- 6) **EROSION AND SEDIMENT CONTROL** - FOLLOWING CONSTRUCTION, OUTER DIKE TO BE RESEEDDED WITH SEED MIX APPROVED BY MDNR AND/OR LOCAL CONSERVATION FOR WINTER PLANTING SEASON.

MATERIALS SCHEDULE TABLE

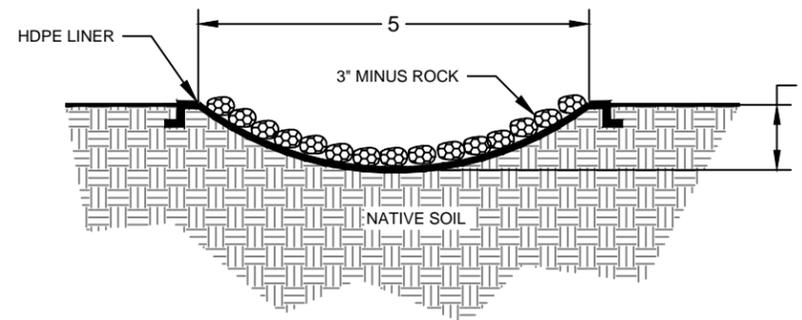
MATERIAL	SPECIFICATIONS	APPROX. QUANTITY
GRAVEL (BIOCELL PLENUM AND PIPE BEDDING)	PEA GRAVEL (OR SIMILAR)	4 CY PLUS BEDDING QTY
SAND (LINER BED AND FILTRATION LAYER)	MAX GRAIN SIZE 3/8". FILTRATION GRADATION	12 CY PLUS LINER BEDDING QTY
ROCK FILTER AGGREGATE	3" MINUS, NO FINES	10 CY
ORGANIC SUBSTRATE	SAWDUST (60%), WOOD CHIPS (30%), CHOPPED HAY (10%) (% BY VOLUME)	87 CY, 43 CY, 15 CY
PIPE	4" EXCEPT AS NOTED AT PUMP INFLUENT. PRESSURE OR DWV, PER ANTICIPATED CONDITION. WATERTIGHT JOINTS AT ALL LOCATIONS OUTSIDE OF HDPE LINER LIMITS	TBD BY CONTRACTOR

 Hagerty Environmental, LLC 202 Winding Lane - Kennett Square, PA 19348 (610) 444-5008 (484) 771-9600 (fax) www.hagertyenvironmental.com	THE DOE RUN COMPANY BUICK MINE PILOT BIOTREATMENT CELL AS-BUILT SCHEMATIC PLAN VIEW BOSS, REYNOLDS COUNTY, MISSOURI
	MO Engineering Certificate of Authority E-2012010543 Paul A. Hagerty - P.E. MO# PE-2003004648
OCTOBER 11, 2012 FIGURE 2 SCALE: VARIES	OCTOBER 2012 buick pilot cell design.dwg

TREATMENT CELL CROSS SECTION (A-A')



ROCK FILTER CROSS SECTION (B-B')



NOTE: ALL DIMENSIONS FEET UNLESS OTHERWISE NOTED

 <p>Environmental Consulting Engineering Construction Management</p> <p>Hagerty Environmental, LLC</p> <p>202 Winding Lane - Kennett Square, PA 19348 (610) 444-5008 (484) 771-9600 (fax) www.hagertyenvironmental.com</p>	<p>THE DOE RUN COMPANY</p> <p>BUICK MINE PILOT BIOTREATMENT CELL AS-BUILT SCHEMATIC CROSS SECTION & DETAILS BOSS, REYNOLDS COUNTY, MISSOURI</p>	
	<p>OCTOBER 11, 2012</p>	<p>FIGURE 3</p>
<p>MO Engineering Certificate of Authority E-2012010543</p> <p>Paul A. Hagerty - P.E. MO# PE-2003004648</p>	<p>RESOURCE MANAGEMENT CONSULTANTS</p>  <p>8138 SOUTH STATE ST. SUITE 2A MIDVALE, UT 84047 801-255-2626</p>	<p>OCTOBER 2012</p> <p>buick pilot cell design.dwg</p>

APPENDIX B

Complete Pilot Test Data Results

SAMPLE ID	DATE	TEMP (F)	SP COND (µS/cm)	DO (mg/L)	pH	ORP	FLOW RATE (GPM)	Pb (T) (µg/L)	Pb (D) (µg/L)	Zn (T) (µg/L)	Zn (D) (µg/L)	Cu (T) (µg/L)	Cu (D) (µg/L)	Cd (T) (µg/L)	Cd (D) (µg/L)	Ni (T) (µg/L)	Ni (D) (µg/L)	As (T) (µg/L)	As (D) (µg/L)	S ²⁻ (mg/L)	SO ₄ (mg/L)	TOC (mg/L)	Alkalinity (mg/L)	Cl ⁻ (mg/L)	Nitrate (mg/L)	Chlorides + Sulfates (mg/L)	BOD (mg/L)	Ammonia (mg/L)	TSS (mg/L)		
Buick Inf	11/8/2012	50.52	1.361	10.91	7.41	20.1	10.2	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A												
Buick Inf	11/12/2012	50.11	1.319	10.72	6.97	20.7	6.5	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A												
Buick Inf	11/20/2012	50.31	1.36	11.15	7.25	33	4.5	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A												
Buick Inf	11/27/2012	42.89	1.385	13.01	8.03	18.7	9.7	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A												
Buick Inf	11/29/2012	46.47	1.405	12.81	6.63	-61.5	9.6	69	56	519	504	5.5	4.4	0.49	0.5	155	148	6.3	1.9	ND	540	1.4	99.7	39.6	4.2	579.6	#N/A	#N/A	ND		
Buick Inf	12/3/2012	53.72	1.361	9.67	7.25	2.2	9.3	73	56	578	565	6.8	5.4	0.67	0.59	168	164	4	3.9	0.01	557	1.88	103.6	39.3	4.12	596.3	#N/A	#N/A	ND		
Buick Inf	12/5/2012	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	2	0.2	#N/A	
Buick Inf	12/10/2012	42.98	1.405	11.54	6.94	81.1	9.7	65	48	662	655	13	ND	0.43	0.13	192	191	7.7	7.1	0.01	536	0.94	102	34	4	570	ND	0.12	ND		
Buick Inf	12/17/2012	43.26	1.41	11.12	7.31	8.4	9.4	66	46	1100	1088	6.2	ND	0.57	0.39	290	288	4	3.1	ND	586	2.3	106.2	39	3.78	625	ND	0.16	ND		
Buick Inf	12/27/2012	36.52	1.4	13.63	6.87	72	9.5	58	47	967	966	5.9	2.2	0.87	0.8	236	234	4.2	3.8	0.02	541	2.5	105.2	32.7	3.9	573.7	#N/A	#N/A	ND		
Buick Inf	1/3/2013	35.24	1.392	14.36	6.86	70.7	9.3	62	45	964	963	3.3	1.4	0.81	0.75	230	228	4.1	4	0.02	519	2.9	104.2	32.3	4.34	551.3	#N/A	#N/A	ND		
Buick Inf	1/4/2013	#N/A	#N/A	#N/A	#N/A	#N/A	5	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Inf	1/7/2013	38.76	1.388	11.87	7.44	97.6	5	59	44	850	849	4	ND	0.49	0.35	221	219	5.5	4	0.04	551	3.31	106	28.3	4.48	579.3	ND	0.22	ND		
Buick Inf	1/14/2013	36.33	1.352	16.02	7.21	16.4	5	73	44	802	795	12	13	0.72	0.73	186	185	4.1	8.8	0.02	484	1.8	102.5	29.2	4.8	513.2	2.6	0.11	5		
Buick Inf	1/23/2013	#N/A	#N/A	#N/A	#N/A	#N/A	7	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Inf	1/28/2013	43.25	1.375	8.47	7.66	20.9	6.8	68	42	1031	1012	0.74	6.2	0.48	0.34	231	229	9.4	2.4	0.01	469	6.69	105.3	36.6	4.71	505.6	ND	0.23	ND		
Buick Inf	2/4/2013	41.02	1.363	9.28	7.71	11	6.9	66	44	972	960	6.4	2.1	0.64	0.63	209	207	3.9	4.2	0.02	438	5.77	111.1	32.4	5.4	470.4	2.5	0.2	ND		
Buick Inf	2/6/2013	#N/A	#N/A	#N/A	#N/A	#N/A	9.6	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Inf	2/11/2013	42.16	1.319	8.78	7.6	15.2	9.4	66	45	958	943	8.6	5.4	0.43	0.38	202	199	4	3.6	0.03	471	5.32	101.3	26	5.89	497	7.2	0.21	5.5		
Buick Inf	2/19/2013	40.93	1.335	9.26	7.62	15.4	9.3	69	47	859	831	5.7	3.1	0.53	0.64	170	165	2.6	2.8	0.03	451	4.48	109.1	29.4	6.68	480.4	3.7	0.22	11		
Buick Inf	2/25/2013	39.41	1.296	8.83	7.73	-21.4	12.3	303	50	969	852	34	3.4	0.92	0.55	187	177	6.8	3.8	0.08	472	4.63	121.9	30.6	6.47	502.6	5.9	0.32	21.5		
Buick Inf	3/4/2013	39.14	1.308	7.94	7.82	-67	12.3	93	60	899	886	9.7	4.6	0.49	0.34	178	177	5.2	4	0.04	460	5.05	113.3	32.2	7.57	492.2	4.6	0.63	9.5		
Buick Inf	3/22/2013	42.56	1.191	9.48	7.6	36.3	8	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Inf	3/25/2013	40.86	1.181	9.09	7.66	51.5	9	104	49	997	944	13	4.1	0.7	0.66	178	171	3.2	3.3	0.04	297	4.43	96.7	37.1	6.58	334.1	2.4	0.91	12		
Buick Inf	4/1/2013	50.48	1.134	8	7.55	-12	8.9	84	57	926	906	9	4	0.87	0.83	179	171	4.3	3.3	0.02	351	5.54	100.1	37.3	7.1	388.3	3.5	0.93	7.5		
Buick Inf	4/8/2013	59.73	1.17	6.82	7.57	39	9.1	88	70	944	938	4.2	ND	0.8	0.65	197	198	4.3	3.6	0.03	397	4.15	120.1	38.8	7.38	435.8	#N/A	#N/A	ND		
Buick Inf	4/15/2013	60.71	1.164	6.35	7.69	5.2	6.2	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	2.6	1	#N/A													
Buick Inf	4/17/2013	#N/A	#N/A	#N/A	#N/A	#N/A	10.3	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A												
Buick Inf	4/22/2013	58.06	1.093	7.68	8.04	-3	#N/A	111	72	719	654	7	2.5	0.59	0.47	152	147	7.7	6.9	0.02	354	#N/A	98	35	7.27	389	2.5	1.1	4.5		
Buick Inf	4/23/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	4/24/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	4/26/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	4/29/2013	60.62	1.06	6.49	7.87	15.7	5.5	91	64	690	673	7.1	1.9	0.26	0.13	170	168	3.9	3.2	0.02	205	1.5	98.5	26	ND	231	#N/A	#N/A	ND		
Buick Inf	5/3/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	14.5
Buick Inf	5/6/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	5/10/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	5.5
Buick Inf	5/13/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	5.5
Buick Inf	5/20/2013	72.23	0.998	5.51	7.72	81.4	#N/A	76	54	587	568	3.2	1.9	0.35	0.26	153	149	7.4	7.4	0.01	372	1.6	93.4	29.3	6	401.3	ND	0.63	ND		
Buick Inf	5/21/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	5/28/2013	76.03	1.023	5.48	8.02	117	12.5	82	48	464	435	4	1.5	0.45	0.23	149	146	5.5	5.9	0.01	329	1.52	98.2	25.5	5.9	354.5	#N/A	#N/A	ND		
Buick Inf	5/29/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	5/30/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	6/3/2013	72.63	0.917	6.54	7.81	150.1	10	117	69	518	471	7.2	2.3	0.45	0.44	166	157	5.2	11	0.02	271	1.6	91	24.6	4.53	295.6	ND	0.51	6.5		
Buick Inf	6/6/2013	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	ND
Buick Inf	6/11/2013	78.68	0.926	6.14	8.32	6	10	95	61	383	351	3	1.3	0.37	0.29	136	128	4.7	5.2	0.01	262	1.58	99.5	24.2	4.57	286.2	ND	0.37	ND		
Buick Eff	11/8/2012	48.77	1.325	1.86	6.25	-29.6	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A
Buick Eff	11/12/2012	54	1.339	1.61	6.28	-82.7	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A
Buick Eff	11/20/2012	48.76	1.351	2.16	6.03	-49.5	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A
Buick Eff	11/27/2012	45																													

Buick Eff	1/3/2013	37.33	1.379	2.97	6.78	-123	#N/A	74	ND	57	21	7.1	0.89	0.19	ND	20	19	1.5	2.8	0.05	537	9.7	100.2	31.1	ND	568.1	#N/A	#N/A	7	
Buick Eff	1/7/2013	38.87	1.4	2.45	6.86	-125	#N/A	13	ND	78	12	15	ND	0.2	ND	22	23	0.88	4.3	0.07	552	14.6	112.4	36	ND	588	15.3	ND	ND	
Buick Eff	1/14/2013	41.89	1.402	5.6	6.68	-98.8	#N/A	28	5.2	106	6.7	50	10	0.63	0.51	61	15	ND	0.81	2.2	483	22.9	123.1	32	ND	515	25.4	ND	ND	
Buick Eff	1/28/2013	42.19	1.306	1.77	6.79	-110.8	#N/A	6.5	3.9	9.5	7.7	15	6	0.18	ND	13	12	1.3	ND	3.2	439	19.98	105.8	35	ND	474	24.5	ND	ND	
Buick Eff	2/4/2013	42.49	1.293	1.64	6.85	-127	#N/A	4.1	2.7	9	8.8	ND	ND	0.28	0.28	11	11	1.9	2.6	2.9	439	16.82	111.1	29.8	ND	468.8	12.6	ND	ND	
Buick Eff	2/11/2013	43	1.32	1.91	6.74	-144.3	#N/A	23	2.9	80	6.8	11	0.32	0.55	0.17	11	11	ND	2	4.2	478	13.45	110.3	40.2	ND	518.2	10.6	ND	ND	
Buick Eff	2/19/2013	43.56	1.313	1.91	6.8	-167.1	#N/A	9.8	3.6	66	6.5	8.3	ND	0.49	0.26	13	11	ND	1.3	5.7	457	13.45	123.9	33.5	ND	490.5	13.3	ND	ND	
Buick Eff	2/25/2013	40.59	1.293	2.15	6.84	-171.2	#N/A	2.7	1.9	8.8	7.9	3	2.8	0.29	0.31	11	11	3.2	3.4	5.8	449	12.58	126.9	27.8	ND	476.8	11.9	ND	ND	
Buick Eff	3/4/2013	40.34	1.27	1.6	7.06	-199.3	#N/A	1.8	3.3	6.8	6.2	0.77	0.23	0.13	0.2	9.5	8.9	2.3	0.6	6.2	499	11.81	138.6	25.2	ND	524.2	12.3	0.15	ND	
Buick Eff	3/22/2013	44.83	1.155	2.02	6.84	-203.7	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Eff	3/25/2013	43.53	1.132	1.7	6.92	-203.6	#N/A	5.1	3	10	3	3.9	1.7	0.35	0.23	2.8	1.9	4.9	3.5	10	311	24.5	160.1	37	ND	348	29.3	0.15	ND	
Buick Eff	4/1/2013	48.22	1.11	1.56	6.87	-210.8	#N/A	5	2.9	9.6	4.7	1.8	1.8	0.35	0.37	4.6	4.1	3.3	3.4	11.75	304	24.76	177.2	37.1	ND	341.1	27.8	0.21	ND	
Buick Eff	4/8/2013	57.3	1.147	1.51	6.72	-228.4	#N/A	6.4	4.5	11	4.6	ND	ND	0.26	0.26	4.8	4.7	3	2.8	15.25	298	15.87	172.7	38.5	ND	336.5	#N/A	#N/A	ND	
Buick Eff	4/15/2013	61.14	1.117	1.44	6.77	-226.1	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Eff	4/22/2013	59.56	1.041	1.44	6.81	-215.6	#N/A	2.6	1.5	ND	ND	1.1	ND	ND	ND	2.9	2	7.1	4.7	22	280	#N/A	194.7	33.4	ND	313.4	36.9	0.49	ND	
Buick Eff	4/29/2013	57.69	1.003	1.36	6.81	-229	#N/A	1.5	ND	6.4	1.1	2.9	4	ND	ND	7.6	6.7	2.7	3	31	187	22	238	30.2	ND	217.2	#N/A	#N/A	ND	
Buick Eff	5/15/2013	68.75	0.946	1.47	6.61	-232	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Eff	5/17/2013	71.38	0.904	4.27	6.55	-200	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick Eff	5/20/2013	74.49	0.965	0.68	6.84	-168	#N/A	6.2	2	35	9.3	1.4	1.7	0.23	0.18	9.9	8.2	11	10	38	158	34.6	253	25.3	ND	183.3	85.5	0.34	ND	
Buick Eff	5/28/2013	73.17	1.002	2.1	6.88	-180.1	#N/A	5.2	1.5	30	9	1.4	ND	0.14	ND	5.7	4.8	5.7	5	25.5	228	14.81	173.1	26.6	ND	254.6	#N/A	#N/A	ND	
Buick Eff	6/3/2013	71.49	0.786	1.3	6.86	-176	#N/A	4.4	2.8	41	17	2.3	ND	ND	ND	11	8.3	ND	2.4	37.5	56	27	291	28.5	ND	84.5	34.1	0.4	ND	
Buick Eff	6/11/2013	77.26	0.911	3.44	7.18	-143.3	#N/A	8.6	3.6	24	9.1	ND	ND	0.14	0.25	6.9	6.1	5.6	6.3	4.9	148	14.67	248.8	25.3	ND	173.3	53	0.42	ND	
Buick RF Eff	11/8/2012	49.12	1.336	2.86	6.38	-2.4	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick RF Eff	11/12/2012	49.69	1.359	2.91	6.62	-45.2	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick RF Eff	11/20/2012	49.75	1.393	3.14	6.51	-39.1	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick RF Eff	11/27/2012	43.32	1.345	3.59	6.54	-54.4	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick RF Eff	11/29/2012	43.98	1.343	3.95	6.5	-123.4	#N/A	164	2.7	43	6.7	7.4	2.7	0.24	0.22	6.7	5.9	ND	1.8	0.18	531	20.1	89.8	39.7	ND	570.7	#N/A	#N/A	ND	
Buick RF Eff	12/3/2012	50.52	1.359	3.6	6.59	-82	#N/A	59	3.8	53	6.7	7.1	2	0.36	0.31	7.4	5	1.8	0.84	0.28	563	29.6	100.7	39.5	ND	602.5	#N/A	#N/A	6	
Buick RF Eff	12/5/2012	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	44	ND	#N/A
Buick RF Eff	12/10/2012	45.46	1.35	4.64	6.74	-167.1	#N/A	47	2.4	50	4.8	ND	ND	0.18	10	7.9	ND	0.85	0.21	544	25.8	100.2	38.6	ND	582.6	30.6	ND	ND		
Buick RF Eff	12/17/2012	42.83	1.372	4.56	6.64	-74.4	#N/A	71	ND	34	5	4.8	ND	0.45	0.3	13	12	1.2	1.7	0.15	528	ND	105.3	38.9	ND	566.9	23	ND	44.2	
Buick RF Eff	12/27/2012	37.99	1.345	5.31	6.83	-68.2	#N/A	320	ND	67	12	125	1.8	0.59	ND	21	17	1.5	2.1	0.25	519	11	102.2	27.2	ND	546.2	#N/A	#N/A	5	
Buick RF Eff	1/3/2013	34.92	1.382	5.95	6.76	-54.6	#N/A	154	ND	41	19	5.7	1.8	ND	ND	16	16	1.6	2.1	1.1	567	8.1	103.2	29.8	ND	596.8	#N/A	#N/A	5	
Buick RF Eff	1/7/2013	37.43	1.399	5.59	7.08	-50	#N/A	12	ND	68	12	2	1.6	0.18	ND	19	21	0.79	3.8	0.22	548	12.5	114.9	29.2	ND	577.2	12	ND	ND	
Buick RF Eff	1/14/2013	35.11	1.397	7.03	6.78	-10.6	#N/A	12	2	79	8.4	23	ND	0.64	0.21	12	11	ND	ND	0.13	480	17.7	127.7	32.5	ND	512.5	18.9	ND	9	
Buick RF Eff	1/28/2013	47.22	1.33	3.54	7.04	-34.7	#N/A	5.1	2.1	8.3	7.7	ND	ND	0.14	11	11	1.8	ND	0.33	470	17.23	104.3	34.8	ND	504.8	59.9	ND	ND		
Buick RF Eff	2/4/2013	45.24	1.306	4.27	6.91	-63	#N/A	3.5	3	4.6	7.6	1.2	ND	0.28	0.17	9.5	9.1	2.4	5.3	0.35	462	15.93	114.4	30.3	ND	492.3	9.6	ND	ND	
Buick RF Eff	2/11/2013	45.23	1.327	4.06	6.98	-76.7	#N/A	20	3.3	70	7.8	10	2	0.75	0.38	10	10	0.97	1.1	0.64	467	13.64	109.8	27.5	ND	494.5	2.7	ND	ND	
Buick RF Eff	2/19/2013	43.97	1.322	4.56	7.05	-98.9	#N/A	17	3.6	56	9.1	6.3	0.42	2.4	0.23	8.2	10	16	1.2	0.7	453	11.79	124.9	26.7	ND	479.7	8.8	ND	ND	
Buick RF Eff	2/25/2013	40.39	1.307	4.99	7.09	-104.7	#N/A	3	2.7	6.4	8.3	3.1	0.6	0.27	0.21	9.4	9.2	3.3	1.6	0.62	458	10.16	124.6	30.8	ND	488.8	7.1	ND	ND	
Buick RF Eff	3/4/2013	44.11	1.274	3.75	7.23	-110.6	#N/A	3	3.1	6.9	6.7	1.3	1.4	0.12	ND	8.3	7.8	2.9	2.1	0.54	406	10.55	138.6	33.9	ND	439.9	6.6	0.12	ND	
Buick RF Eff	3/22/2013	41.26	1.168	4.61	7.21	-86.7	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A													
Buick RF Eff	3/25/2013	41.01	1.139	4.49	7.28	-0.7	#N/A	5.2	3.6	7.9	3.3	4.1	0.67	0.2	0.2	6.5	6.4	4.4	4.1	0.15	270	21.56	160.1	36	ND	306	19.2	ND	ND	
Buick RF Eff	4/1/2013	52.23	1.127	3.41	7.42	-123.9	#N/A	5.2	4	5.6	4.2	1.5	ND	0.36	0.37	4.5	4.5	5.9	5.9	0.32	323	22.2	181.4	37.5	ND	360.5	13.6	ND	ND	
Buick RF Eff	4/8/2013	62.33	1.15	2.58	7.26	-155.2	#N/A	7.1	3.9	9.6	7.7	ND	0.15	0.22	0.19	4.6	4.4	4.9	4.5	1.7	334	22.41	170.5	37.1	ND	371.1	#N/A	#N/A	ND	
Buick RF Eff	4/15/2013	65	1.133	2.47	7.32	-175	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	#N/A	28.5	0.35	#N/A													
Buick RF Eff	4/22/2013	62.01	1.048	2.96	7.12	-122	#N/A	6.5	3.9	9.5	6.7	ND	0.58	0.34	0.29	4.4	3.6	8.3	8.2	ND	247	#N/A	193.6	35.1	ND	282.1	18.1	0.35	6	
Buick RF Eff	4/29/2013	66.96	0.994	1.81	7.66	-135	#N/A	2.7	ND	5.8	2.1	4	3.6	ND	ND	8.8	7.6	4.1	4.7	2	105	19	241.9	8.5	ND	113.5	#N/A	#N/A	ND	
Buick RF Eff	5/8/2013	62.19	0.827	2.88	7.11	-139	#N/A	14	3.6	86	25	1.8	ND	0.32	0.31	43	37	5.3	5.8	15	195	72.08	198.1	34.65	ND	229.65	#N/A	#N/A	29	
Buick RF Eff	5/10/2013	67.68	0.851	2.17	7.45	-137	#N/A	15	3.3	77																				



REFERENCE #60144112-002

Pace Analytical Services, Inc.
9608 Loiret Blvd.
Lenexa, KS 66219
Phone: 913.599.5665
Fax: 913.599.1759

May 16, 2013

Amy Sanders
The Doe Run Company
P.O. Box 500
Viburnum, MO 65566

Re: Lab Project Number: 60144112-002
Client Project ID: Wet Test

Dear:

Enclosed are the analytical results for sample(s) received by the laboratory. The results relate only to the samples included in this report. Results reported herein conform to the most current NELAC standards, where applicable, unless otherwise narrated in the body of the report.

If you have any question concerning this report, please feel free to contact me.

Sincerely,

Tim Harrell
Tim.Harrell@pacelabs.com
Technical Director

Kansas/ NELAP Certification Number E-10116
Utah Certification Number 9135995665
Texas Certification Number T104704407-08-TX
Oklahoma Certification Number 9205/9935
Louisiana Certification Number 03055
Arkansas Certification Number 05-008-0

Enclosures

REPORT OF LABORATORY ANALYSIS

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REFERENCE #60144112-002

Pace Analytical Services, Inc.
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**CHRONIC TOXICITY TEST FOR
DOE RUN COMPANY (Buick Biocell)**

PERMIT #

PERFORMED ON:

Pimephales promelas

and

Ceriodaphnia dubia

PREPARED FOR:

The Doe Run Company (Buick Biocell)
Attn: Amy Sanders
P.O. Box 500
Viburnum, MO 65566
1-573-689-4535

PREPARED BY:
Pace Analytical Services, Inc.
808 West McKay
Frontenac, KS 66763
1-620-235-0003

May 16, 2013

REPORT OF LABORATORY ANALYSIS

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SUMMARY

A Chronic Whole Effluent Toxicity Test using the 7-day chronic fathead minnows (*Pimephales promelas*), static renewal larval survival and growth test, and three brood 7-day chronic Cladoceran (*Ceriodaphnia dubia*), static renewal survival and reproduction test, was conducted on effluent discharge water collected at DOE RUN COMPANY (Buick Biocell) effluent discharge from May 6, 2013 to May 10, 2013. All the test methods followed are as listed in EPA 821-R-02-013, "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms."

Statistically significant ($p < 0.05$) mortality is determined by Dunnet's procedure using average percent survival of each test concentration versus the average survival of the controls. If significant mortality occurs, median lethal concentrations (LC50) are calculated using effluent concentrations and their corresponding percent mortality data. The LC50's and the 95% confidence intervals are calculated where appropriate by the Spearman-Kärber method. Statistical analysis is accomplished by following steps in EPA 821-R-02-013, November 2002 and by use of Toxstat version 3.4.

In minnow section of testing, it was observed that the effluent had no significant effect on the survival of the larvae at the 100% concentration. No significant mortality was observed in the other effluent concentrations after the 7-day exposure period. The No Observed Effect Concentration (NOEC) was determined to be 100% for survival. The LC50 was estimated to be >100% effluent. No significant reduction in growth was observed in the 100% effluent concentration. The Toxic Units is <1. The IC25 is >100. The NOEC for growth in effluent was determined to be 100%.

In Cladoceran section of testing, it was observed that the effluent had no significant effect on the survival of the organisms in the 100% effluent concentration. No significant mortality was observed in the other effluent concentrations after the 7-day exposure period. The No Observed Effect Concentration (NOEC) was determined to be 100% for survival. The LC50 was estimated to be >100% effluent. Significant reduction in reproduction was observed in the 50, and 100% effluent concentrations. The Toxic Units is 2.92. The IC25 is 34.28. The NOEC for reproduction in effluent was determined to be 25%.

The chronic toxicity exhibited by the fathead minnows and the *Ceriodaphnia* treated by the effluent sampled from May 6 to May 10 from DOE RUN COMPANY (Buick Biocell) effluent discharge, is not acceptable as described in EPA 821-R-02-013.

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INTRODUCTION

Pace Analytical was contracted to perform this chronic toxicity test on effluent from DOE RUN COMPANY (Buick Biocell) effluent discharge. Chronic toxicity was measured using the Pimephales promelas at larval for survival and growth test and the Ceriodaphnia dubia survival and reproduction test described in EPA 821-R-02-013, "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms." The raw data of the study is stored at Pace Analytical Services, INC. 808 West McKay, Frontenac, KS 66763.

TEST MATERIAL

DOE RUN COMPANY (Buick Biocell) personnel collected sampling of the effluent. A sample of the effluent was delivered to Pace by commercial carrier on 5-7-13. Subsequent samples followed by delivery on 5-9-13 and on 5-11-13. All samples were stored at $\leq 6^{\circ}$ Celsius. Moderately Hard Synthetic Water was used as a control and also to make the required dilutions in the test as described in EPA 821-R-02-013.

TEST METHODS

Pace used EPA test method 1000.0 for conducting the Fathead Minnow, Pimephales promelas, Larval Survival and Growth Test. EPA test method 1002.0 was used for conducting the Cladoceran, Ceriodaphnia dubia, Survival and Reproduction Test. The tests were conducted to estimate the LC50, NOEC, and LOEC for survival, growth, and reproduction of these test species.

The Pimephales and Ceriodaphnia tests were initiated on 5-7-13 and carried out until 5-14-13. The Pimephales tests were conducted in 500 ml plastic jars with 250 ml of test solution. Ten larvae were placed in each of at least 4 replicates to make a total of 40 larvae per sample concentration. The Ceriodaphnia tests were carried out in 35ml vials containing 25 ml of test solution. One Neonate was placed in each of 10 replicates to make a total of 10 neonates per sample concentration.

TEST ORGANISMS

The organisms used in these tests were cultured at Pace under controlled temperature and photoperiod conditions and/or were purchased from an external supplier. Pace maintains records of all culture techniques used in producing organisms.

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RESULTS

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TABLE 1

Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

Date Sampled	No. 1:	5-6-13	8:53
	No. 2:	5-8-13	8:48
	No. 3:	5-10-13	11:27
Test Initiated: 13:30	Date:	5-7-13	

Dilution Water used: Moderately Hard Synthetic Water

FATHEAD MINNOW LARVAE GROWTH AND SURVIVAL
(Pimephales promelas)

DATA TABLE FOR GROWTH OF FATHEAD MINNOWS

Effluent Concentration (%)	Average Dry Weight in Milligrams in Replicate Chambers				Mean Dry Weight (mg)	CV% *
	A	B	C	D		
Control 0%	0.460	0.491	0.531	0.525	0.502	4.19
Dilution 1 6.25%	0.549	0.552	0.574	0.596	0.568	2.59
Dilution 2 12.5%	0.605	0.583	0.588	0.594	0.592	1.10
Dilution 3 25%	0.523	0.632	0.578	0.544	0.569	5.65
Dilution 4 50%	0.565	0.429	0.512	0.516	0.506	7.15
Dilution 5 100%	0.456	0.496	0.604	0.450	0.502	9.11

* Coefficient of Variation = Standard Deviation X 100 / Mean

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Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

FATHEAD MINNOW SURVIVAL

Conc. %	Percent Survival in Replicate Chambers				Mean Percent Survival			CV %
	A	B	C	D	24hr	48hr	7 day	
Control 0%	90	100	100	100	100	100	97.5	5.94
Dilution 1 6.25%	100	100	100	100	100	100	100	0.00
Dilution 2 12.5%	100	100	100	100	100	100	100	0.00
Dilution 3 25%	100	100	100	90	100	100	97.5	5.94
Dilution 4 50%	100	90	100	100	100	100	97.5	5.94
Dilution 5 100%	90	90	100	90	100	100	92.5	6.32

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Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

CERIODAPHNIA SURVIVAL AND REPRODUCTION

DATA TABLE FOR CERIODAPHNIA YOUNG PRODUCTION

Replicate	Control 0%	Dilution 1 6.25%	Dilution 2 12.5%	Dilution 3 25%	Dilution 4 50%	Dilution 5 100%
1	21	19	23	28	7	0
2	16	17	29	26	0	0
3	25	23	23	28	5	12
4	21	30	26	26	17	0
5	26	25	26	28	9	5
6	25	23	22	22	8	3
7	22	19	29	27	12	4
8	20	19	25	17	16	7
9	23	17	27	22	3	0
10	21	29	25	24	0	2
Mean	22.0	22.1	25.5	24.8	7.7	3.3
SD	2.944	4.725	2.415	3.584	6.001	3.917
CV %	13.38	21.38	9.47	14.45	77.93	118.70

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Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

CERIODAPHNIA MEAN PERCENT SURVIVAL

Percent Effluent (%)						
Time Elapsed	Control 0%	Dilution 1 6.25%	Dilution 2 12.5%	Dilution 3 25%	Dilution 4 50%	Dilution 5 100%
24 hrs	100	100	100	100	100	100
48 hrs	100	100	100	100	100	100
7-day	100	100	100	100	100	100
SD	0.000	0.000	0.000	0.000	0.000	0.000
CV %	0.00	0.00	0.00	0.00	0.00	0.00

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TABLE 2
SUMMARY OF TEST CONDITIONS FOR THE FATHEAD MINNOW
(*Pimephales promelas*) LARVAL SURVIVAL AND GROWTH TEST

1. Test type	Static renewal
2. Temperature	25 degrees Celsius
3. Light quality	Ambient laboratory light
4. Light intensity	Ambient laboratory levels
5. Photoperiod	16 hr light, 8 hr dark
6. Test chamber size	500 ml
7. Test solution volume	250 ml
8. Renewal of test concentrations	Daily
9. Age of test organism	< 24 hours
10. No. larvae/chamber	10
11. No. replicates/concentration	4
12. No. larvae/concentration	40
13. Feeding regime	Feed 0.1 ml newly hatched brine shrimp nauplii three times daily. Larvae are not fed 12 hours prior to termination of test.
14. Cleaning	Siphon daily, immediately before test solution renewal
15. Aeration	None

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TABLE 2 (CONT.)

16. Dilution Water	Moderately Hard Synthetic Water prepared with MILLI-Q deionized water and reagent grade chemicals
17. Effluent concentrations	0%, 6.25%, 12.5%, 25%, 50%, 100%
18. Test duration	7 days
19. Endpoints	Survival and growth
20. Test acceptability	80% or greater survival in the controls, Average dry weight in controls >0.25 mg, Coefficient of variation in the control must not exceed 40%.

TABLE 2 (CONT.)

**SUMMARY OF TEST CONDITIONS FOR THE CLADOCERAN
(Ceriodaphnia dubia) SURVIVAL AND REPRODUCTION TEST**

1. Test type	Static renewal
2. Temperature	25 degrees Celsius
3. Light quality	Ambient laboratory light
4. Light intensity	Ambient laboratory levels
5. Photoperiod	16 hr light, 8 hr dark
6. Test chamber size	30 ml
7. Test solution volume	25 ml

REPORT OF LABORATORY ANALYSIS

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TABLE 2 (CONT.)

8. Renewal of test concentrations	Daily
9. Age of test organism	< 24 hours
10. No. larvae/chamber	1
11. No. replicates/concentration	10
12. No. larvae/concentration	10
13. Feeding regime	Feed 0.1 ml YCT three times daily. Larvae are not fed 12 hours prior to termination of test.
14. Cleaning	Siphon daily, immediately before test solution renewal
15. Aeration	None
16. Dilution Water	Moderately Hard Synthetic Water prepared with MILLI-Q deionized water and reagent grade chemicals
17. Effluent concentrations	0%, 6.25%, 12.5%, 25%, 50%, 100%
18. Test duration	7 days - 10 days
19. Endpoints	Survival and Reproduction
20. Test acceptability	80% or greater survival in the controls, Average reproduction rate of 15 young / adult. Coefficient of variation in the control must not exceed 40%.

REPORT OF LABORATORY ANALYSIS

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TABLE 2 (SECTION 2)

**BIOMONITORING CHRONIC TOXICITY REPORT
FATHEAD MINNOW (Pimephales promelas)
CHEMICAL PARAMETERS CHART**

Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

ANALYSTS: Pace Analytical Services, Inc.
Timothy Harrell
Mike Bollin

SAMPLE NO. 1 COLLECTED: DATE: 5-6-13

SAMPLE NO. 2 COLLECTED: DATE: 5-8-13

SAMPLE NO. 3 COLLECTED: DATE: 5-10-13

**TABLE 2 (SECTION 2)
INITIAL WATER QUALITY
EFFLUENT CONCENTRATION**

	Control	100%
PH	7.41	8.04
D.O.	8.20	5.80
Temp	25	25
Alk	62	468
Hard	92	526
Cond	340	910
Chlorine	<0.1	<0.1

- * D.O. is reported as mg/L
- Alkalinity is reported as mg/L CaCO₃
- Hardness is reported as mg/L CaCO₃
- Conductance is reported as umhos
- Chlorine is reported as mg/L

REPORT OF LABORATORY ANALYSIS

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TEST WATER QUALITY

24-Hour Water Quality Measurements

Effluent Concentration (%)	PH	D.O. (mg/l)	Temperature (C)
0% Control	7.57	7.00	25
6.25% Effluent	7.69	6.50	25
12.5% Effluent	7.84	5.90	25
25% Effluent	7.99	5.40	25
50% Effluent	8.17	4.50	25
100% Effluent	8.24	4.10	25

48-Hour Water Quality Measurements

Effluent Concentration (%)	PH	D.O. (mg/l)	Temperature (C)
0% Control	7.65	7.30	25
6.25% Effluent	7.79	6.80	25
12.5% Effluent	7.94	6.40	25
25% Effluent	8.03	5.40	25
50% Effluent	8.14	4.80	25
100% Effluent	8.21	3.50	25

REPORT OF LABORATORY ANALYSIS

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FINAL WATER QUALITY

EFFLUENT CONCENTRATION

	Control	100%
pH	7.62	8.09
D.O.	7.00	4.00
Temp	25	25
Alk	64	446
Hard	98	504
Cond	380	1297

- * D.O. is reported as mg/L
- Alkalinity is reported as mg/L CaCO₃
- Hardness is reported as mg/L CaCO₃
- Conductance is reported as umhos

REPORT OF LABORATORY ANALYSIS

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TEST VALIDITY

The Pimephales promelas control survival rate was 97.5. The mean dry weight (growth) of the Pimephales promelas was determined at 0.502 g/organism in the controls. The percent coefficient of variation (%CV) values for the fathead minnow control for survival and growth were 5.94 and 4.19. The Ceriodaphnia dubia survival rates were 100 in the control. The Ceriodaphnia in the control produced an average of 22.0 young over the seven-day exposure period. Percent CV values for Ceriodaphnia dubia control survival and reproduction was 0.00 and 13.38. Control data met or exceeded all criteria set out by EPA 821-R-02-013 for test acceptance.

CONCLUSIONS

The No Observed Effect Concentration (NOEC) for Pimephales promelas was 100% for survival and 100% for growth. The No Observed Effect Concentration (NOEC) for Ceriodaphnia dubia was 100% for Survival and 25% for Reproduction. The tests were ran using a synthetic control against effluent concentrations of 6.25%, 12.5%, 25%, 50%, and 100%. The effluent sampled on 5-6-13, 5-8-13, and 5-10-13 exhibited non-acceptable chronic toxicity in Pimephales promelas and in Ceriodaphnia dubia during the exposure period as described in EPA 821-R-02-013.

REPORT OF LABORATORY ANALYSIS

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APPENDIX C

REFERENCE TOXICANTS

The absence of significant control mortality during this test indicated the health of the organisms and indicated that any significant mortality in the test concentrations was not due to contaminants or variations in testing conditions.

Reference toxicity testing is routinely performed by staff members in our biomonitoring - bioassay laboratory.

Concentration of Toxicant	Pimephales promelas			
	Avg. # of Live Organisms/replicate			
	0 hrs	24 hrs	48 hrs	7 days
10 g/l	40	9	3	0
8 g/l	40	39	30	5
6 g/l	40	40	37	23
4 g/l	40	40	40	39
2 g/l	40	40	40	40

IC25 (4.99 g/l Sodium Chloride)

Survival NOEC: 4.0 g/l

Concentration of Toxicant	Ceriodaphnia Dubia			
	Avg. # of Live Organisms/replicate			
	0 hrs	24 hrs	48 hrs	7 days
2.5 g/l	10	4	0	0
2.0 g/l	10	10	8	1
1.5 g/l	10	10	10	10
1.0 g/l	10	10	10	10
0.5 g/l	10	10	10	10

IC25 (1.19 g/l Sodium Chloride)

Survival NOEC: 1.5 g/l

Submitted By: 
Timothy Harrell, Technical Director

REPORT OF LABORATORY ANALYSIS

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60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

Chi-square test for normality: actual and expected frequencies

INTERVAL	<-1.5	-1.5 to <-0.5	-0.5 to 0.5	>0.5 to 1.5	>1.5
EXPECTED	1.608	5.808	9.168	5.808	1.608
OBSERVED	0	3	20	1	0

Calculated Chi-Square goodness of fit test statistic = 21.3518
Table Chi-Square value (alpha = 0.01) = 13.277

Data FAIL normality test. Try another transformation.

Warning - The first three homogeneity tests are sensitive to non-normal data and should not be performed.

60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

Shapiro - Wilk's test for normality

D = 0.080

W = 0.854

Critical W (P = 0.05) (n = 24) = 0.916

Critical W (P = 0.01) (n = 24) = 0.884

Data FAIL normality test. Try another transformation.

Warning - The first three homogeneity tests are sensitive to non-normal data and should not be performed.

60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	4	1.249	1.412	1.371
2	6.25%	4	1.412	1.412	1.412
3	12.5%	4	1.412	1.412	1.412
4	25%	4	1.249	1.412	1.371
5	50%	4	1.249	1.412	1.371
6	100%	4	1.249	1.412	1.290

60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	0.007	0.081	0.041	5.94
2	6.25%	0.000	0.000	0.000	0.00
3	12.5%	0.000	0.000	0.000	0.00
4	25%	0.007	0.081	0.041	5.94
5	50%	0.007	0.081	0.041	5.94
6	100%	0.007	0.081	0.041	6.32

60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	0.040	0.008	1.800
Within (Error)	18	0.080	0.004	
Total	23	0.120		

Critical F value = 2.77 (0.05,5,18)
Since $F < \text{Critical } F$ FAIL TO REJECT H_0 : All equal

60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
 File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 1 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	1.371	0.975		
2	6.25%	1.412	1.000	-0.866	
3	12.5%	1.412	1.000	-0.866	
4	25%	1.371	0.975	0.000	
5	50%	1.371	0.975	0.000	
6	100%	1.290	0.925	1.732	

Dunnett table value = 2.41 (1 Tailed Value, P=0.05, df=18,5)

60144112 Doe Run Buick Biocell FATHEAD SURVIVAL
 File: 6144116A Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	4			
2	6.25%	4	0.055	5.7	-0.025
3	12.5%	4	0.055	5.7	-0.025
4	25%	4	0.055	5.7	0.000
5	50%	4	0.055	5.7	0.000
6	100%	4	0.055	5.7	0.050

60144112 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

Shapiro - Wilk's test for normality

D = 0.037

W = 0.968

Critical W (P = 0.05) (n = 24) = 0.916

Critical W (P = 0.01) (n = 24) = 0.884

Data PASS normality test at P=0.01 level. Continue analysis.

60144112 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

Bartlett's test for homogeneity of variance
Calculated B1 statistic = 9.79

Table Chi-square value = 15.09 (alpha = 0.01, df = 5)
Table Chi-square value = 11.07 (alpha = 0.05, df = 5)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

60144112 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	4	0.745	0.816	0.787
2	6.25%	4	0.834	0.882	0.853
3	12.5%	4	0.869	0.891	0.878
4	25%	4	0.808	0.919	0.855
5	50%	4	0.714	0.851	0.791
6	100%	4	0.735	0.890	0.787

60144112 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	0.001	0.033	0.016	4.19
2	6.25%	0.000	0.022	0.011	2.59
3	12.5%	0.000	0.010	0.005	1.10
4	25%	0.002	0.048	0.024	5.65
5	50%	0.003	0.057	0.028	7.15
6	100%	0.005	0.072	0.036	9.11

60144112 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	0.034	0.007	3.347
Within (Error)	18	0.037	0.002	
Total	23	0.071		

Critical F value = 2.77 (0.05,5,18)
Since $F > \text{Critical } F$ REJECT H_0 : All equal

60144112 Doe Run Buick Biocell FATHEAD GROWTH
 File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 1 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	0.787	0.502		
2	6.25%	0.853	0.568	-2.066	
3	12.5%	0.878	0.592	-2.847	
4	25%	0.855	0.569	-2.120	
5	50%	0.791	0.506	-0.117	
6	100%	0.787	0.502	0.003	

Dunnett table value = 2.41 (1 Tailed Value, P=0.05, df=18,5)

60144112 Doe Run Buick Biocell FATHEAD GROWTH
 File: 6144116B Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	4			
2	6.25%	4	0.077	15.3	-0.066
3	12.5%	4	0.077	15.3	-0.091
4	25%	4	0.077	15.3	-0.068
5	50%	4	0.077	15.3	-0.004
6	100%	4	0.077	15.3	0.000

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
6.25%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
12.5%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
25%	10	0	10

TOTAL 20 0 20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
50%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
100%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

SUMMARY OF FISHER'S EXACT TESTS

NUMBER	NUMBER	SIG
--------	--------	-----

GROUP	IDENTIFICATION	EXPOSED	DEAD	(P=.05)
	CONTROL	10	0	
1	6.25%	10	0	
2	12.5%	10	0	
3	25%	10	0	
4	50%	10	0	
5	100%	10	0	

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA SURVI
File: 6144116D Transform: NO TRANSFORM

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	10	1.000	1.000	1.000
2	6.25%	10	1.000	1.000	1.000
3	12.5%	10	1.000	1.000	1.000
4	25%	10	1.000	1.000	1.000
5	50%	10	1.000	1.000	1.000
6	100%	10	1.000	1.000	1.000

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA SURVI
File: 6144116D Transform: NO TRANSFORM

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	0.000	0.000	0.000	0.00
2	6.25%	0.000	0.000	0.000	0.00
3	12.5%	0.000	0.000	0.000	0.00
4	25%	0.000	0.000	0.000	0.00
5	50%	0.000	0.000	0.000	0.00
6	100%	0.000	0.000	0.000	0.00

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144116E Transform: NO TRANSFORMATION

Chi-square test for normality: actual and expected frequencies

INTERVAL	<-1.5	-1.5 to <-0.5	-0.5 to 0.5	>0.5 to 1.5	>1.5
EXPECTED	4.020	14.520	22.920	14.520	4.020
OBSERVED	2	18	22	15	3

Calculated Chi-Square goodness of fit test statistic = 2.1607
Table Chi-Square value (alpha = 0.01) = 13.277

Data PASS normality test. Continue analysis.

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144116E Transform: NO TRANSFORMATION

Bartlett's test for homogeneity of variance
Calculated B1 statistic = 9.02

Table Chi-square value = 15.09 (alpha = 0.01, df = 5)
Table Chi-square value = 11.07 (alpha = 0.05, df = 5)

Data PASS B1 homogeneity test at 0.01 level. Continue analysis.

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144116E Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	10	16.000	26.000	22.000
2	6.25%	10	17.000	30.000	22.100
3	12.5%	10	22.000	29.000	25.500
4	25%	10	17.000	28.000	24.800
5	50%	10	0.000	17.000	7.700
6	100%	10	0.000	12.000	3.300

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144116E Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	8.667	2.944	0.931	13.38
2	6.25%	22.322	4.725	1.494	21.38
3	12.5%	5.833	2.415	0.764	9.47
4	25%	12.844	3.584	1.133	14.45
5	50%	36.011	6.001	1.898	77.93
6	100%	15.344	3.917	1.239	118.70

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144116E Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	4563.533	912.707	54.208
Within (Error)	54	909.200	16.837	
Total	59	5472.733		

Critical F value = 2.45 (0.05,5,40)
Since $F > \text{Critical } F$ REJECT H_0 : All equal

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
 File: 6144116E Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 1 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	22.000	22.000		
2	6.25%	22.100	22.100	-0.054	
3	12.5%	25.500	25.500	-1.907	
4	25%	24.800	24.800	-1.526	
5	50%	7.700	7.700	7.793	*
6	100%	3.300	3.300	10.190	*

Dunnett table value = 2.31 (1 Tailed Value, P=0.05, df=40,5)

60144112 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
 File: 6144116E Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	10			
2	6.25%	10	4.239	19.3	-0.100
3	12.5%	10	4.239	19.3	-3.500
4	25%	10	4.239	19.3	-2.800
5	50%	10	4.239	19.3	14.300
6	100%	10	4.239	19.3	18.700

Conc. ID	1	2	3	4	5	6
Conc. Tested	0	6.25	12.5	25	50	100
Response 1	.460	.549	.605	.523	.565	.456
Response 2	.491	.552	.583	.632	.429	.496
Response 3	.531	.574	.588	.578	.512	.604
Response 4	.525	.596	.594	.544	.516	.450

*** Inhibition Concentration Percentage Estimate ***

Toxicant/Effluent: Doe Run Buick Biocell

Test Start Date: 5/7/13 Test Ending Date: 5/14/13

Test Species: Fathead

Test Duration: 7 Day

DATA FILE:

Conc. ID	Number Replicates	Concentration	Response Means	Std. Dev.	Pooled Response Means
1	4	0.000	0.502	0.033	0.558
2	4	6.250	0.568	0.022	0.558
3	4	12.500	0.592	0.009	0.558
4	4	25.000	0.569	0.048	0.558
5	4	50.000	0.506	0.056	0.506
6	4	100.000	0.502	0.071	0.502

*** No Linear Interpolation Estimate can be calculated from the input data since none of the (possibly pooled) group response means were less than 75% of the control response mean.

Conc. ID	1	2	3	4	5	6
Conc. Tested	0	6.25	12.5	25	50	100
Response 1	21	19	23	28	7	0
Response 2	16	17	29	26	0	0
Response 3	25	23	23	28	5	12
Response 4	21	30	26	26	17	0
Response 5	26	25	26	28	9	5
Response 6	25	23	22	22	8	3
Response 7	22	19	29	27	12	4
Response 8	20	19	25	17	16	7
Response 9	23	17	27	22	3	0
Response 10	21	29	25	24	0	2

*** Inhibition Concentration Percentage Estimate ***

Toxicant/Effluent: Doe Run Buick Biocell
 Test Start Date: 5/7/13 Test Ending Date: 5/14/13
 Test Species: Dubia
 Test Duration: 7 Day
 DATA FILE:

Conc. ID	Number Replicates	Concentration	Response Means	Std. Dev.	Pooled Response Means
1	10	0.000	22.000	2.944	23.600
2	10	6.250	22.100	4.725	23.600
3	10	12.500	25.500	2.415	23.600
4	10	25.000	24.800	3.584	23.600
5	10	50.000	7.700	6.001	7.700
6	10	100.000	3.300	3.917	3.300

The Linear Interpolation Estimate: 34.2767 Entered P Value: 25

Number of Resamplings: 80
 The Bootstrap Estimates Mean: 34.2440 Standard Deviation: 0.9988
 Original Confidence Limits: Lower: 32.5475 Upper: 36.5081
 Resampling time in Seconds: 0.00 Random Seed: 896318990

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Page: 1 of 1

Section A Required Client Information:
 Company: The De De Building
 Address: 370 Forest Road 2032
 City: Rock Hill
 State: SC
 Zip: 29730
 Phone: 803-485-4881
 Fax: 803-244-2178
 Requested Due Date/TAT: _____

Section B Required Project Information:
 Report To: AMY SARKIS
 Copy To: _____
 Purchase Order No.: 516746
 Project Name: BUICK (LUE) 13
 Project Number: _____

Section C Invoice Information:
 Attention: AMY SARKIS
 Company Name: The De De Building
 Address: 370 Forest Road 2032
 City: Rock Hill
 State: SC
 Zip: 29730
 Reference: Sample Church
 Site Location: MO
 State: _____

REGULATORY AGENCY
 NPDES GROUND WATER DRINKING WATER
 UST RCRA OTHER _____

ITEM #	Section D Required Client Information	Matrix Codes MATERIAL CODE	Matrix Code (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED		SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives						Analysis Test	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
					COMPOSITE START	COMPOSITE END/GRAB			H ₂ SO ₄	HNO ₃	HCl	NaOH	Na ₂ S ₂ O ₃	Methanol				
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		

ADDITIONAL COMMENTS: _____

RELINQUISHED BY / AFFILIATION: Amber Nigam DATE: 05/06/13 TIME: 11:00

ACCEPTED BY / AFFILIATION: AMY SARKIS DATE: 05/06/13 TIME: 09:05

Temp in °C: _____ Received on Ice (Y/N): _____ Custody Sealed Cooler (Y/N): _____ Samples Intact (Y/N): _____

SAMPLER NAME AND SIGNATURE: Amber Nigam DATE Signed: 05/06/13

PRINT Name of SAMPLER: _____ SIGNATURE of SAMPLER: _____

Section A Required Client Information:		Section B Required Project Information:		Section C Invoice Information:	
Company: <u>De Run Company</u>	Report To: <u>Andy Sandus</u>	Attention: <u>Anna Santos</u>	Company Name: <u>De Run Company</u>	Address: <u>10 Park St, Auburn, MA 01501</u>	REGULATORY AGENCY <input checked="" type="checkbox"/> NPDES <input type="checkbox"/> GROUND WATER <input type="checkbox"/> DRINKING WATER <input type="checkbox"/> UST <input type="checkbox"/> RCRA <input type="checkbox"/> OTHER
Address: <u>De Run SLD</u>	Copy To:	Purchase Order No.: <u>516886</u>	Address: <u>10 Park St, Auburn, MA 01501</u>	Reference: <u>Tomie Cramer</u>	Site Location STATE: <u>MA</u>
Email: <u>De Run@de-run.com</u>		Project Name: <u>BUILD WET'S</u>	Reference: <u>Tomie Cramer</u>	Pace Profile #:	
Phone: <u>573 668 4551</u> Fax: <u>573 244 2477</u>		Project Number:			
Requested Due Date/TAT: <u>5/22/13</u>					

ITEM #	Section D Required Client Information	Matrix Codes MATRIX / CODE	Matrix Code (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED		SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives	Analysis Test ↓	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
					COMPOSITE START	COMPOSITE END/GRAB							
		Drinking Water Water Waste Water Product Soil/Solid Oil Air Wipe Tissue Other	DW WT WW P SL OL WP AR TS OT		DATE	TIME	DATE	TIME	H ₂ SO ₄ HNO ₃ HCl NaOH Na ₂ S ₂ O ₃ Methanol Other	↓ Analysis Test ↓			
1	BDOO3												
2	BUILD Biocell EFF												
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													

ADDITIONAL COMMENTS		RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS	
		<u>Amber Upper</u>	<u>05/13/13</u>	<u>1:30</u>	<u>Amber Upper</u>	<u>05/16/13</u>	<u>09:00</u>	Temp in °C	Received on Ice (Y/N)
								Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER: Amber Upper
SIGNATURE of SAMPLER: Amber Upper
DATE Signed (MM/DD/YY): 05/16/13

F-ALL-Q-020rev.07, 15-May-2007
Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.



REFERENCE #60144624

Pace Analytical Services, Inc.
9608 Loiret Blvd.
Lenexa, KS 66219
Phone: 913.599.5665
Fax: 913.599.1759

May 22, 2013

Amy Sanders
The Doe Run Company
P.O. Box 500
Viburnum, MO 65566

Re: Lab Project Number: 60144624
Client Project ID: Wet Test

Dear:

Enclosed are the analytical results for sample(s) received by the laboratory. The results relate only to the samples included in this report. Results reported herein conform to the most current NELAC standards, where applicable, unless otherwise narrated in the body of the report.

If you have any question concerning this report, please feel free to contact me.

Sincerely,

Tim Harrell
Tim.Harrell@pacelabs.com
Technical Director

Kansas/ NELAP Certification Number E-10116
Utah Certification Number 9135995665
Texas Certification Number T104704407-08-TX
Oklahoma Certification Number 9205/9935
Louisiana Certification Number 03055
Arkansas Certification Number 05-008-0

Enclosures

REPORT OF LABORATORY ANALYSIS

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REFERENCE #60144624

Pace Analytical Services, Inc.

9608 Loiret Blvd.

Lenexa, KS 66219

Phone: 913.599.5665

Fax: 913.599.1759

**CHRONIC TOXICITY TEST FOR
DOE RUN COMPANY (Buick Biocell)**

PERMIT #

PERFORMED ON:

Pimephales promelas

and

Ceriodaphnia dubia

PREPARED FOR:

The Doe Run Company (Buick Biocell)

Attn: Amy Sanders

P.O. Box 500

Viburnum, MO 65566

1-573-689-4535

PREPARED BY:

Pace Analytical Services, Inc.

808 West McKay

Frontenac, KS 66763

1-620-235-0003

May 22, 2013

REPORT OF LABORATORY ANALYSIS

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REPORT OF LABORATORY ANALYSIS

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SUMMARY

A Chronic Whole Effluent Toxicity Test using the 7-day chronic fathead minnows (*Pimephales promelas*), static renewal larval survival and growth test, and three brood 7-day chronic Cladoceran (*Ceriodaphnia dubia*), static renewal survival and reproduction test, was conducted on effluent discharge water collected at DOE RUN COMPANY (Buick Biocell) effluent discharge from May 13, 2013 to May 17, 2013. All the test methods followed are as listed in EPA 821-R-02-013, "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms."

Statistically significant ($p < 0.05$) mortality is determined by Dunnet's procedure using average percent survival of each test concentration versus the average survival of the controls. If significant mortality occurs, median lethal concentrations (LC50) are calculated using effluent concentrations and their corresponding percent mortality data. The LC50's and the 95% confidence intervals are calculated where appropriate by the Spearman-Kärber method. Statistical analysis is accomplished by following steps in EPA 821-R-02-013, November 2002 and by use of Toxstat version 3.4.

In minnow section of testing, it was observed that the effluent had significant effect on the survival of the larvae at the 25, 50, and 100% concentration. No significant mortality was observed in the other effluent concentrations after the 7-day exposure period. The No Observed Effect Concentration (NOEC) was determined to be 12.5% for survival. The LC50 was estimated to be 28.2% effluent. Significant reduction in growth was observed in the 25, 50, and 100% effluent concentration. The Toxic Units is 5.15. The IC25 is 19.4. The NOEC for growth in effluent was determined to be 12.5%.

In Cladoceran section of testing, it was observed that the effluent had significant effect on the survival of the organisms in the 100% effluent concentration. No significant mortality was observed in the other effluent concentrations after the 7-day exposure period. The No Observed Effect Concentration (NOEC) was determined to be 50% for survival. The LC50 was estimated to be 70.71% effluent. Significant reduction in reproduction was observed in the 50, and 100% effluent concentrations. The Toxic Units is 2.78. The IC25 is 36.0. The NOEC for reproduction in effluent was determined to be 25%.

The chronic toxicity exhibited by the fathead minnows and the *Ceriodaphnia* treated by the effluent sampled from May 13 to May 17 from DOE RUN COMPANY (Buick Biocell) effluent discharge, is not acceptable as described in EPA 821-R-02-013.

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INTRODUCTION

Pace Analytical was contracted to perform this chronic toxicity test on effluent from DOE RUN COMPANY (Buick Biocell) effluent discharge. Chronic toxicity was measured using the Pimephales promelas at larval for survival and growth test and the Ceriodaphnia dubia survival and reproduction test described in EPA 821-R-02-013, "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms." The raw data of the study is stored at Pace Analytical Services, INC. 808 West McKay, Frontenac, KS 66763.

TEST MATERIAL

DOE RUN COMPANY (Buick Biocell) personnel collected sampling of the effluent. A sample of the effluent was delivered to Pace by commercial carrier on 5-14-13. Subsequent samples followed by delivery on 5-16-13 and on 5-20-13. The last sample was not received until Monday. The second sample was used through the weekend. All samples were stored at $\leq 6^{\circ}$ Celsius. Moderately Hard Synthetic Water was used as a control and also to make the required dilutions in the test as described in EPA 821-R-02-013.

TEST METHODS

Pace used EPA test method 1000.0 for conducting the Fathead Minnow, Pimephales promelas, Larval Survival and Growth Test. EPA test method 1002.0 was used for conducting the Cladoceran, Ceriodaphnia dubia, Survival and Reproduction Test. The tests were conducted to estimate the LC50, NOEC, and LOEC for survival, growth, and reproduction of these test species.

The Pimephales and Ceriodaphnia tests were initiated on 5-14-13 and carried out until 5-21-13. The Pimephales tests were conducted in 500 ml plastic jars with 250 ml of test solution. Ten larvae were placed in each of at least 4 replicates to make a total of 40 larvae per sample concentration. The Ceriodaphnia tests were carried out in 35ml vials containing 25 ml of test solution. One Neonate was placed in each of 10 replicates to make a total of 10 neonates per sample concentration.

TEST ORGANISMS

The organisms used in these tests were cultured at Pace under controlled temperature and photoperiod conditions and/or were purchased from an external supplier. Pace maintains records of all culture techniques used in producing organisms.

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REFERENCE #60144624

Pace Analytical Services, Inc.
9608 Loiret Blvd.
Lenexa, KS 66219
Phone: 913.599.5665
Fax: 913.599.1759

RESULTS

REPORT OF LABORATORY ANALYSIS

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TABLE 1

Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

Date Sampled	No. 1: 5-13-13	10:41
	No. 2: 5-15-13	13:50
	No. 3: 5-17-13	7:21
Test Initiated: 14:45	Date: 5-14-13	

Dilution Water used: Moderately Hard Synthetic Water

FATHEAD MINNOW LARVAE GROWTH AND SURVIVAL
(Pimephales promelas)

DATA TABLE FOR GROWTH OF FATHEAD MINNOWS

Effluent Concentration (%)	Average Dry Weight in Milligrams in Replicate Chambers				Mean Dry Weight (mg)	CV% *
	A	B	C	D		
Control 0%	0.443	0.432	0.562	0.464	0.476	7.95
Dilution 1 6.25%	0.449	0.402	0.486	0.449	0.447	4.74
Dilution 2 12.5%	0.470	0.545	0.466	0.563	0.511	6.32
Dilution 3 25%	0.201	0.296	0.304	0.245	0.262	10.36
Dilution 4 50%	0.0	0.0	0.0	0.0	0.00	0.00
Dilution 5 100%	0.0	0.0	0.0	0.0	0.00	0.00

* Coefficient of Variation = Standard Deviation X 100 / Mean

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Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

FATHEAD MINNOW SURVIVAL

Conc. %	Percent Survival in Replicate Chambers				Mean Percent Survival			CV %
	A	B	C	D	24hr	48hr	7 day	
Control 0%	100	90	100	100	100	100	97.5	5.94
Dilution 1 6.25%	100	100	100	100	100	100	100	0.00
Dilution 2 12.5%	100	100	90	100	100	100	100	0.00
Dilution 3 25%	60	70	70	60	100	100	65	6.46
Dilution 4 50%	0	0	0	0	100	100	0.00	0.00
Dilution 5 100%	0	0	0	0	82.5	80	0.00	0.00

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Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

CERIODAPHNIA SURVIVAL AND REPRODUCTION

DATA TABLE FOR CERIODAPHNIA YOUNG PRODUCTION

Replicate	Control 0%	Dilution 1 6.25%	Dilution 2 12.5%	Dilution 3 25%	Dilution 4 50%	Dilution 5 100%
1	19	22	25	16	4	0
2	19	23	29	25	16	0
3	20	24	18	16	14	0
4	15	23	23	22	14	0
5	18	18	28	14	12	0
6	19	20	25	13	15	0
7	22	16	17	20	9	0
8	23	22	22	22	9	0
9	17	15	15	23	12	0
10	17	23	25	14	13	0
Mean	18.9	20.6	22.7	18.5	11.8	0.0
SD	2.378	3.204	4.692	4.378	3.584	0.00
CV %	12.58	15.55	20.67	23.66	30.37	N/A

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Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

CERIODAPHNIA MEAN PERCENT SURVIVAL

Percent Effluent (%)						
Time Elapsed	Control 0%	Dilution 1 6.25%	Dilution 2 12.5%	Dilution 3 25%	Dilution 4 50%	Dilution 5 100%
24 hrs	100	100	100	100	100	0
48 hrs	100	100	100	100	100	0
7-day	100	100	100	100	100	0
SD	0.000	0.000	0.000	0.000	0.000	0.00
CV %	0.00	0.00	0.00	0.00	0.00	N/A

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TABLE 2
SUMMARY OF TEST CONDITIONS FOR THE FATHEAD MINNOW
(*Pimephales promelas*) LARVAL SURVIVAL AND GROWTH TEST

1. Test type	Static renewal
2. Temperature	25 degrees Celsius
3. Light quality	Ambient laboratory light
4. Light intensity	Ambient laboratory levels
5. Photoperiod	16 hr light, 8 hr dark
6. Test chamber size	500 ml
7. Test solution volume	250 ml
8. Renewal of test concentrations	Daily
9. Age of test organism	< 24 hours
10. No. larvae/chamber	10
11. No. replicates/concentration	4
12. No. larvae/concentration	40
13. Feeding regime	Feed 0.1 ml newly hatched brine shrimp nauplii three times daily. Larvae are not fed 12 hours prior to termination of test.
14. Cleaning	Siphon daily, immediately before test solution renewal
15. Aeration	None

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TABLE 2 (CONT.)

16. Dilution Water	Moderately Hard Synthetic Water prepared with MILLI-Q deionized water and reagent grade chemicals
17. Effluent concentrations	0%, 6.25%, 12.5%, 25%, 50%, 100%
18. Test duration	7 days
19. Endpoints	Survival and growth
20. Test acceptability	80% or greater survival in the controls, Average dry weight in controls >0.25 mg, Coefficient of variation in the control must not exceed 40%.

TABLE 2 (CONT.)

**SUMMARY OF TEST CONDITIONS FOR THE CLADOCERAN
(*Ceriodaphnia dubia*) SURVIVAL AND REPRODUCTION TEST**

1. Test type	Static renewal
2. Temperature	25 degrees Celsius
3. Light quality	Ambient laboratory light
4. Light intensity	Ambient laboratory levels
5. Photoperiod	16 hr light, 8 hr dark
6. Test chamber size	30 ml
7. Test solution volume	25 ml

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TABLE 2 (CONT.)

8. Renewal of test concentrations	Daily
9. Age of test organism	< 24 hours
10. No. larvae/chamber	1
11. No. replicates/concentration	10
12. No. larvae/concentration	10
13. Feeding regime	Feed 0.1 ml YCT three times daily. Larvae are not fed 12 hours prior to termination of test.
14. Cleaning	Siphon daily, immediately before test solution renewal
15. Aeration	None
16. Dilution Water	Moderately Hard Synthetic Water prepared with MILLI-Q deionized water and reagent grade chemicals
17. Effluent concentrations	0%, 6.25%, 12.5%, 25%, 50%, 100%
18. Test duration	7 days - 10 days
19. Endpoints	Survival and Reproduction
20. Test acceptability	80% or greater survival in the controls, Average reproduction rate of 15 young / adult. Coefficient of variation in the control must not exceed 40%.

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TABLE 2 (SECTION 2)

**BIOMONITORING CHRONIC TOXICITY REPORT
FATHEAD MINNOW (Pimephales promelas)
CHEMICAL PARAMETERS CHART**

Permittee: DOE RUN COMPANY (Buick Biocell) Effluent discharge.

ANALYSTS: Pace Analytical Services, Inc.
Timothy Harrell
Mike Bollin

SAMPLE NO. 1 COLLECTED: DATE: 5-13-13

SAMPLE NO. 2 COLLECTED: DATE: 5-15-13

SAMPLE NO. 3 COLLECTED: DATE: 5-17-13

**TABLE 2 (SECTION 2)
INITIAL WATER QUALITY
EFFLUENT CONCENTRATION**

	Control	100%
PH	7.56	7.30
D.O.	8.40	6.30
Temp	25	25
Alk	58	350
Hard	92	550
Cond	342	1244
Chlorine	<0.1	<0.1

- * D.O. is reported as mg/L
- Alkalinity is reported as mg/L CaCO₃
- Hardness is reported as mg/L CaCO₃
- Conductance is reported as umhos
- Chlorine is reported as mg/L

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TEST WATER QUALITY

24-Hour Water Quality Measurements

Effluent Concentration (%)	PH	D.O. (mg/l)	Temperature (C)
0% Control	7.41	6.90	25
6.25% Effluent	7.49	6.60	25
12.5% Effluent	7.56	6.40	25
25% Effluent	7.63	6.20	25
50% Effluent	7.67	5.90	25
100% Effluent	8.33	5.30	25

48-Hour Water Quality Measurements

Effluent Concentration (%)	PH	D.O. (mg/l)	Temperature (C)
0% Control	7.54	7.40	25
6.25% Effluent	7.59	7.00	25
12.5% Effluent	7.68	6.10	25
25% Effluent	7.81	5.70	25
50% Effluent	7.92	5.20	25
100% Effluent	8.34	4.30	25

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FINAL WATER QUALITY

EFFLUENT CONCENTRATION

	Control	100%
pH	7.49	8.32
D.O.	6.90	3.80
Temp	25	25
Alk	64	260
Hard	98	600
Cond	434	1290

- * D.O. is reported as mg/L
- Alkalinity is reported as mg/L CaCO₃
- Hardness is reported as mg/L CaCO₃
- Conductance is reported as umhos

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TEST VALIDITY

The Pimephales promelas control survival rate was 97.5. The mean dry weight (growth) of the Pimephales promelas was determined at 0.476 g/organism in the controls. The percent coefficient of variation (%CV) values for the fathead minnow control for survival and growth were 5.94 and 7.95. The Ceriodaphnia dubia survival rates were 100 in the control. The Ceriodaphnia in the control produced an average of 18.9 young over the seven-day exposure period. Percent CV values for Ceriodaphnia dubia control survival and reproduction was 0.00 and 12.58. Control data met or exceeded all criteria set out by EPA 821-R-02-013 for test acceptance.

CONCLUSIONS

The No Observed Effect Concentration (NOEC) for Pimephales promelas was 12.5% for survival and 12.5% for growth. The No Observed Effect Concentration (NOEC) for Ceriodaphnia dubia was 50% for Survival and 25% for Reproduction. The tests were ran using a synthetic control against effluent concentrations of 6.25%, 12.5%, 25%, 50%, and 100%. The effluent sampled on 5-13-13, 5-15-13, and 5-17-13 exhibited non-acceptable chronic toxicity in Pimephales promelas and in Ceriodaphnia dubia during the exposure period as described in EPA 821-R-02-013.

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APPENDIX C

REFERENCE TOXICANTS

The absence of significant control mortality during this test indicated the health of the organisms and indicated that any significant mortality in the test concentrations was not due to contaminants or variations in testing conditions.

Reference toxicity testing is routinely performed by staff members in our biomonitoring - bioassay laboratory.

Concentration of Toxicant	Avg. # of Live Organisms/replicate			
	0 hrs	24 hrs	48 hrs	7 days
10 g/l	40	9	2	0
8 g/l	40	38	29	5
6 g/l	40	40	38	24
4 g/l	40	40	40	39
2 g/l	40	40	40	39

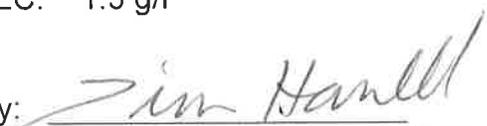
IC25 (5.25 g/l Sodium Chloride)

Survival NOEC: 4.0 g/l

Concentration of Toxicant	Avg. # of Live Organisms/replicate			
	0 hrs	24 hrs	48 hrs	7 days
2.5 g/l	10	5	0	0
2.0 g/l	10	10	8	2
1.5 g/l	10	10	10	10
1.0 g/l	10	10	10	10
0.5 g/l	10	10	10	10

IC25 (1.24 g/l Sodium Chloride)

Survival NOEC: 1.5 g/l

Submitted By: 
Timothy Harrell, Technical Director

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60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

Chi-square test for normality: actual and expected frequencies

INTERVAL	<-1.5	-1.5 to <-0.5	-0.5 to 0.5	>0.5 to 1.5	>1.5
EXPECTED	1.608	5.808	9.168	5.808	1.608
OBSERVED	0	4	18	2	0

Calculated Chi-Square goodness of fit test statistic = 14.7838

Table Chi-Square value (alpha = 0.01) = 13.277

Data FAIL normality test. Try another transformation.

Warning - The first three homogeneity tests are sensitive to non-normal data and should not be performed.

60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

Shapiro - Wilk's test for normality

D = 0.051

W = 0.780

Critical W (P = 0.05) (n = 24) = 0.916

Critical W (P = 0.01) (n = 24) = 0.884

Data FAIL normality test. Try another transformation.

Warning - The first three homogeneity tests are sensitive to non-normal data and should not be performed.

60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	4	1.249	1.412	1.371
2	6.25%	4	1.412	1.412	1.412
3	12.5%	4	1.249	1.412	1.371
4	25%	4	0.886	0.991	0.939
5	50%	4	0.159	0.159	0.159
6	100%	4	0.159	0.159	0.159

60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	0.007	0.081	0.041	5.94
2	6.25%	0.000	0.000	0.000	0.00
3	12.5%	0.007	0.081	0.041	5.94
4	25%	0.004	0.061	0.030	6.46
5	50%	0.000	0.000	0.000	0.00
6	100%	0.000	0.000	0.000	0.00

60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	7.227	1.445	511.292
Within (Error)	18	0.051	0.003	
Total	23	7.277		

Critical F value = 2.77 (0.05,5,18)
Since $F > \text{Critical } F$ REJECT H_0 : All equal

60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
 File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 1 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	1.371	0.975		
2	6.25%	1.412	1.000	-1.084	
3	12.5%	1.371	0.975	0.000	
4	25%	0.939	0.650	11.508	*
5	50%	0.159	0.000	32.251	*
6	100%	0.159	0.000	32.251	*

Dunnett table value = 2.41 (1 Tailed Value, P=0.05, df=18,5)

60144624 Doe Run Buick Biocell FATHEAD SURVIVAL
 File: 6144624A Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST - TABLE 2 OF 2 Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	4			
2	6.25%	4	0.043	4.4	-0.025
3	12.5%	4	0.043	4.4	0.000
4	25%	4	0.043	4.4	0.325
5	50%	4	0.043	4.4	0.975
6	100%	4	0.043	4.4	0.975

60144624 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144624B Transform: ARC SINE(SQUARE ROOT(Y))

Chi-square test for normality: actual and expected frequencies

INTERVAL	<-1.5	-1.5 to <-0.5	-0.5 to 0.5	>0.5 to 1.5	>1.5
EXPECTED	1.608	5.808	9.168	5.808	1.608
OBSERVED	0	6	12	6	0

Calculated Chi-Square goodness of fit test statistic = 4.1035
Table Chi-Square value (alpha = 0.01) = 13.277

Data PASS normality test. Continue analysis.

60144624 Doe Run Buick Biocell FATHEAD GROWTH
File: 6144624B Transform: ARC SINE(SQUARE ROOT(Y))

Hartley's test for homogeneity of variance
Bartlett's test for homogeneity of variance

These two tests can not be performed because at least one group has zero variance.

Data FAIL to meet homogeneity of variance assumption.
Additional transformations are useless.

60144624 Doe Run Buick Biocell FATHEAD GROWTH

File: 6144624B

Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	4	0.717	0.850	0.761
2	6.25%	4	0.687	0.771	0.732
3	12.5%	4	0.751	0.849	0.796
4	25%	4	0.465	0.584	0.535
5	50%	4	0.159	0.159	0.159
6	100%	4	0.159	0.159	0.159

60144624 Doe Run Buick Biocell FATHEAD GROWTH

File: 6144624B

Transform: ARC SINE(SQUARE ROOT(Y))

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	0.004	0.060	0.030	7.95
2	6.25%	0.001	0.035	0.017	4.74
3	12.5%	0.003	0.050	0.025	6.32
4	25%	0.003	0.055	0.028	10.36
5	50%	0.000	0.000	0.000	0.00
6	100%	0.000	0.000	0.000	0.00

60144624 Doe Run Buick Biocell FATHEAD GROWTH

File: 6144624B

Transform: ARC SINE(SQUARE ROOT(Y))

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	1.762	0.352	201.985
Within (Error)	18	0.031	0.002	
Total	23	1.793		

Critical F value = 2.77 (0.05,5,18)

Since $F > \text{Critical } F$ REJECT H_0 : All equal

60144624 Doe Run Buick Biocell FATHEAD GROWTH

File: 6144624B

Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST

TABLE 1 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	0.761	0.476		
2	6.25%	0.732	0.447	0.996	
3	12.5%	0.796	0.511	-1.197	
4	25%	0.535	0.262	7.639	*
5	50%	0.159	0.000	20.394	*
6	100%	0.159	0.000	20.394	*

Dunnett table value = 2.41 (1 Tailed Value, P=0.05, df=18,5)

60144624 Doe Run Buick Biocell FATHEAD GROWTH

File: 6144624B

Transform: ARC SINE(SQUARE ROOT(Y))

DUNNETT'S TEST

TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	4			
2	6.25%	4	0.071	14.8	0.029
3	12.5%	4	0.071	14.8	-0.035
4	25%	4	0.071	14.8	0.214
5	50%	4	0.071	14.8	0.476
6	100%	4	0.071	14.8	0.476

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
6.25%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
12.5%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
25%	10	0	10

TOTAL 20 0 20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
50%	10	0	10
TOTAL	20	0	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 10.
 Since b is greater than 6 there is no significant difference
 between CONTROL and TREATMENT at the 0.05 level.

FISHER'S EXACT TEST

IDENTIFICATION	NUMBER OF		
	ALIVE	DEAD	TOTAL ANIMALS
CONTROL	10	0	10
100%	0	10	10
TOTAL	10	10	20

CRITICAL FISHER'S VALUE (10,10,10) (p=0.05) IS 6. b VALUE IS 0.
 Since b is less than or equal to 6 there is a significant difference
 between CONTROL and TREATMENT at the 0.05 level.

SUMMARY OF FISHER'S EXACT TESTS

NUMBER	NUMBER	SIG
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GROUP	IDENTIFICATION	EXPOSED	DEAD	(P=.05)
	CONTROL	10	0	
1	6.25%	10	0	
2	12.5%	10	0	
3	25%	10	0	
4	50%	10	0	
5	100%	10	10	*

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA SURVI
File: 6144624D Transform: NO TRANSFORM

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	10	1.000	1.000	1.000
2	6.25%	10	1.000	1.000	1.000
3	12.5%	10	1.000	1.000	1.000
4	25%	10	1.000	1.000	1.000
5	50%	10	1.000	1.000	1.000
6	100%	10	0.000	0.000	0.000

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA SURVI
File: 6144624D Transform: NO TRANSFORM

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	0.000	0.000	0.000	0.00
2	6.25%	0.000	0.000	0.000	0.00
3	12.5%	0.000	0.000	0.000	0.00
4	25%	0.000	0.000	0.000	0.00
5	50%	0.000	0.000	0.000	0.00
6	100%	0.000	0.000	0.000	N/A

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144624E Transform: NO TRANSFORMATION

Chi-square test for normality: actual and expected frequencies

INTERVAL	<-1.5	-1.5 to <-0.5	-0.5 to 0.5	>0.5 to 1.5	>1.5
EXPECTED	4.020	14.520	22.920	14.520	4.020
OBSERVED	4	13	27	15	1

Calculated Chi-Square goodness of fit test statistic = 3.1701

Table Chi-Square value (alpha = 0.01) = 13.277

Data PASS normality test. Continue analysis.

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144624E Transform: NO TRANSFORMATION

Hartley's test for homogeneity of variance
Bartlett's test for homogeneity of variance

These two tests can not be performed because at least one group has
zero variance.

Data FAIL to meet homogeneity of variance assumption.
Additional transformations are useless.

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144624E Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 1 of 2

GRP	IDENTIFICATION	N	MIN	MAX	MEAN
1	CONTROL	10	15.000	23.000	18.900
2	6.25%	10	15.000	24.000	20.600
3	12.5%	10	15.000	29.000	22.700
4	25%	10	13.000	25.000	18.500
5	50%	10	4.000	16.000	11.800
6	100%	10	0.000	0.000	0.000

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144624E Transform: NO TRANSFORMATION

SUMMARY STATISTICS ON TRANSFORMED DATA TABLE 2 of 2

GRP	IDENTIFICATION	VARIANCE	SD	SEM	C.V. %
1	CONTROL	5.656	2.378	0.752	12.58
2	6.25%	10.267	3.204	1.013	15.55
3	12.5%	22.011	4.692	1.484	20.67
4	25%	19.167	4.378	1.384	23.66
5	50%	12.844	3.584	1.133	30.37
6	100%	0.000	0.000	0.000	N/A

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO
File: 6144624E Transform: NO TRANSFORMATION

ANOVA TABLE

SOURCE	DF	SS	MS	F
Between	5	3523.083	704.617	60.444
Within (Error)	54	629.500	11.657	
Total	59	4152.583		

Critical F value = 2.45 (0.05,5,40)
Since $F > \text{Critical } F$ REJECT H_0 : All equal

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO

File: 6144624E Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 1 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	TRANSFORMED MEAN	MEAN CALCULATED IN ORIGINAL UNITS	T STAT	SIG
1	CONTROL	18.900	18.900		
2	6.25%	20.600	20.600	-1.113	
3	12.5%	22.700	22.700	-2.489	
4	25%	18.500	18.500	0.262	
5	50%	11.800	11.800	4.650	*
6	100%	0.000	0.000	12.378	*

Dunnett table value = 2.31 (1 Tailed Value, P=0.05, df=40,5)

60144624 Doe Run Buick Biocell CERIODAPHNIA DUBIA REPRO

File: 6144624E Transform: NO TRANSFORMATION

DUNNETT'S TEST - TABLE 2 OF 2

Ho:Control<Treatment

GROUP	IDENTIFICATION	NUM OF REPS	Minimum Sig Diff (IN ORIG. UNITS)	% of CONTROL	DIFFERENCE FROM CONTROL
1	CONTROL	10			
2	6.25%	10	3.527	18.7	-1.700
3	12.5%	10	3.527	18.7	-3.800
4	25%	10	3.527	18.7	0.400
5	50%	10	3.527	18.7	7.100
6	100%	10	3.527	18.7	18.900

Conc. ID	1	2	3	4	5	6
Conc. Tested	0	6.25	12.5	25	50	100
Response 1	.443	.449	.470	.201	.0	.0
Response 2	.432	.402	.545	.296	.0	.0
Response 3	.564	.486	.466	.304	.0	.0
Response 4	.464	.449	.563	.245	.0	.0

*** Inhibition Concentration Percentage Estimate ***

Toxicant/Effluent: Doe Run Buick

Test Start Date: 5/14/13 Test Ending Date: 5/21/13

Test Species: Fathead

Test Duration: 7 Day

DATA FILE:

Conc. ID	Number Replicates	Concentration	Response Means	Std. Dev.	Pooled Response Means
1	4	0.000	0.476	0.060	0.478
2	4	6.250	0.447	0.034	0.478
3	4	12.500	0.511	0.050	0.478
4	4	25.000	0.262	0.048	0.262
5	4	50.000	0.000	0.000	0.000
6	4	100.000	0.000	0.000	0.000

The Linear Interpolation Estimate: 19.4039 Entered P Value: 25

Number of Resamplings: 80

The Bootstrap Estimates Mean: 19.1368 Standard Deviation: 1.0086

Original Confidence Limits: Lower: 16.8333 Upper: 20.8076

Expanded Confidence Limits: Lower: 15.2909 Upper: 21.6498

Resampling time in Seconds: 0.00 Random_Seed: -705360744

Conc. ID	1	2	3	4	5	6
Conc. Tested	0	6.25	12.5	25	50	100
Response 1	19	22	25	16	4	0
Response 2	19	23	29	25	16	0
Response 3	20	24	18	16	14	0
Response 4	15	23	23	22	14	0
Response 5	18	18	28	14	12	0
Response 6	19	20	25	13	15	0
Response 7	22	16	17	20	9	0
Response 8	23	22	22	22	9	0
Response 9	17	15	15	23	12	0
Response 10	17	23	25	14	13	0

*** Inhibition Concentration Percentage Estimate ***

Toxicant/Effluent: Doe Run

Test Start Date: 5/14/13 Test Ending Date: 5/20/13

Test Species: Dubia

Test Duration: 7 Day

DATA FILE:

Conc. ID	Number Replicates	Concentration	Response Means	Std. Dev.	Pooled Response Means
1	10	0.000	18.900	2.378	20.733
2	10	6.250	20.600	3.204	20.733
3	10	12.500	22.700	4.692	20.733
4	10	25.000	18.500	4.378	18.500
5	10	50.000	11.800	3.584	11.800
6	10	100.000	0.000	0.000	0.000

The Linear Interpolation Estimate: 36.0075 Entered P Value: 25

Number of Resamplings: 80

The Bootstrap Estimates Mean: 35.4216 Standard Deviation: 3.9296

Original Confidence Limits: Lower: 27.9095 Upper: 40.9722

Resampling time in Seconds: 0.00 Random Seed: 366601112

SPECIES TYPE	TEST CHEMICAL	SOLVENT	DATE
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Fathead	Doe Run Biocell		5/13
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CONC.	NUMBER EXPOSED	NUMBER DEAD	PERCENT DEAD	BINOMIAL PROB. (PERCENT)
100	40	40	100	LESS THAN 0.001
50	40	40	100	LESS THAN 0.001
25	40	14	35	4.034519
12.5	40	1	2.5	LESS THAN 0.001
6.25	40	0	0	LESS THAN 0.001

THE BINOMIAL TEST SHOWS THAT 12.5 AND 50 CAN BE USED AS STATISTICALLY SOUND CONSERVATIVE 95 PERCENT CONFIDENCE LIMITS BECAUSE THE ACTUAL CONFIDENCE LEVEL ASSOCIATED WITH THESE LIMITS IS GREATER THAN 95 PERCENT

AN APPROXIMATE LC50 OF 28.19766 IS OBTAINED BY NONLINEAR INTERPOLATION BETWEEN 25 AND 50

-----RESULTS CALCULATED USING THE MOVING AVERAGE METHOD-----

SPAN	G	LC50	95 PERCENT CONFIDENCE LIMITS	
4	2.372137E-02	25.4603	22.07848	29.38616
3	.02795	25.50003	21.89724	29.14278
2	2.795024E-02	26.26675	23.74996	29.13294

AN LC50 CALCULATED USING THE MOVING AVERAGE METHOD MAY NOT BE A VERY GOOD ESTIMATE IF THE SPAN IS MUCH LESS THAN THE NUMBER OF CONCENTRATIONS.

-----RESULTS CALCULATED USING THE PROBIT METHOD-----

ITERATIONS	G	H	CHI-SQUARE	PROBABILITY
6	.110553	1	3.517004	.3185632

SLOPE = 7.485043
 95 PERCENT CONFIDENCE LIMITS = 4.996302 AND 9.973783

LC50 = 26.72895
 95 PERCENT CONFIDENCE LIMITS = 23.89366 AND 29.94441

COMPARE RESULTS WITH ORIGINAL DATA TO SEE IF THEY ARE REASONABLE.

SPECIES TYPE	TEST CHEMICAL	SOLVENT	DATE
-----------------	------------------	---------	------

Dubia	Doe Run Biocell		5/13
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CONC.	NUMBER EXPOSED	NUMBER DEAD	PERCENT DEAD	BINOMIAL PROB. (PERCENT)
100	10	10	100	9.765626E-02
50	10	0	0	9.765626E-02
25	10	0	0	9.765626E-02
12.5	10	0	0	9.765626E-02
6.25	10	0	0	9.765626E-02

THE BINOMIAL TEST SHOWS THAT 50 AND 100 CAN BE USED AS STATISTICALLY SOUND CONSERVATIVE 95 PERCENT CONFIDENCE LIMITS BECAUSE THE ACTUAL CONFIDENCE LEVEL ASSOCIATED WITH THESE LIMITS IS GREATER THAN 95 PERCENT

AN APPROXIMATE LC50 OF 70.71066 IS OBTAINED BY NONLINEAR INTERPOLATION BETWEEN 50 AND 100

WHEN THERE ARE LESS THAN TWO CONCENTRATIONS AT WHICH THE PERCENT DEAD IS BETWEEN 0 AND 100, NEITHER THE MOVING AVERAGE METHOD NOR THE PROBIT METHOD CAN GIVE ANY STATISTICALLY SOUND RESULTS!

COMPARE RESULTS WITH ORIGINAL DATA TO SEE IF THEY ARE REASONABLE.

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information: Company: <u>Dee Run Company</u> Address: <u>25 RDX 500</u> City: <u>Vancouver, Missouri</u> Email: <u>OSardus@deerun.com</u> Report To: <u>Copy To: Amy Sanders</u> Project Name: <u>BUCK Pilot Test</u> Project Number: <u>11011054</u> Requested Due Date (AT): <u>05/13/13</u>		Section B Required Project Information: Report To: <u>Amy Sanders</u> Copy To: <u>Amy Sanders</u> Purchase Order No.: <u>BUCK Pilot Test</u> Project Name: <u>BUCK Pilot Test</u> Project Number: <u>11011054</u>		Section C Invoice Information: Attention: <u>Amy Sanders</u> Company Name: <u>Dee Run Company</u> Address: <u>25 RDX 500 Vancouver</u> Phone: <u>636-250-1000</u> Reference: <u>Same Check</u> Pace Project Manager: <u>Same Check</u> Pace Profile #: <u>11011054</u>	
Regulatory Agency: <input checked="" type="checkbox"/> NPDES <input type="checkbox"/> GROUND WATER <input type="checkbox"/> DRINKING WATER <input type="checkbox"/> UST <input type="checkbox"/> RCRA <input type="checkbox"/> OTHER		Site Location: STATE: <u>MO</u>		Requested Analysis Filtered (Y/N)	

ITEM #	Section D Required Client Information	Matrix Codes MATRIX / CODE	Matrix Code (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED		SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives	Analysis Test	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)
					DATE	TIME						
1	BUCK Biocell BEE	DW WT WW P SL OL WP AR TS OT	WW G	GRAB	05/13/13	11:00	11	Unpreserved		Chronic WET		N
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												

ADDITIONAL COMMENTS: <u>Relinquished by Affiliation</u>		RELINQUISHED BY / AFFILIATION: <u>[Signature]</u>		DATE: <u>05/13/13</u>		TIME: <u>1130</u>		ACCEPTED BY / AFFILIATION: <u>[Signature]</u>		DATE: <u>05/13/13</u>		TIME: <u>1100</u>		SAMPLE CONDITIONS: <u>30 Y Y Y</u>					
SAMPLER NAME AND SIGNATURE: <u>[Signature]</u>				PRINT Name of SAMPLER: <u>Amber Nipper</u>				DATE Signed (MM/DD/YY): <u>05/13/13</u>				Temp in °C		Received on Ice (Y/N)		Custody Sealed Cooler (Y/N)		Samples Intact (Y/N)	

Important Note: By signing this form, you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.
F-ALL-Q-020rev.07, 15-May-2007

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:		Section B Required Project Information:		Section C Invoice Information:	
Company Name: <u>De Run Company</u>	Report To: <u>Andy Sanders</u>	Attention: <u>Andy Sanders</u>	Company Name: <u>De Run Company</u>	Address: <u>1100...</u>	REGULATORY AGENCY
Address: <u>1100...</u>	Copy To: <u>Andy Sanders</u>	Invoice Number: <u>1635934</u>	Address: <u>1100...</u>	City: <u>...</u>	STATE: <u>...</u>
Email To: <u>...</u>	Purchase Order No.: <u>...</u>	Price Quote Reference: <u>...</u>	Price Profile #: <u>...</u>	Site Location	NPDES <input type="checkbox"/> GROUND WATER <input type="checkbox"/> DRINKING WATER <input type="checkbox"/> UST <input type="checkbox"/> RORA <input type="checkbox"/> OTHER <input type="checkbox"/>
Project Name: <u>Buck P-104</u>	Project Number: <u>...</u>	Pace Project Manager: <u>Samie Gruber</u>	Requested Analysis Filtered (Y/N) <input checked="" type="checkbox"/>	Temp in °C	Received on Ice (Y/N)
Requested Due Date/TAT: <u>5-26-13</u>				Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

ITEM #	Section D Required Client Information	Matrix Codes MATRIX / CODE	Matrix / CODE	Matrix Codes DW WT WW P SL OL WP AR TS OT	Matrix Code (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED		SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives	Analysis Test ↓	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
							COMPOSITE START	COMPOSITE END/GRAB						
1	<u>Buck Brook Eff</u>	<u>WW</u>	<u>G</u>	<u>WW</u>	<u>WW</u>	<u>G</u>	<u>05/15/13</u>	<u>15:07</u>	<u>11</u>	<u>Unpreserved</u>	<u>Chronic Wet</u>	<u>N</u>	<u>1046</u>	
2														
3														
4														
5														
6														
7														
8														
9														
10														
11														
12														

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS
	<u>Samie Gruber</u>	<u>05/15/13</u>	<u>14:15</u>	<u>Andy Sanders</u>	<u>05/16/13</u>	<u>10:00</u>	<u>Y</u>
							<u>Y</u>
							<u>Y</u>

ORIGINAL

SAMPLER NAME AND SIGNATURE
PRINT Name of SAMPLER: Samie Gruber
SIGNATURE of SAMPLER: [Signature]

DATE Signed (MM/DD/YYYY): 05/15/13

Temp in °C
Received on Ice (Y/N)
Custody Sealed Cooler (Y/N)
Samples Intact (Y/N)

