



**BEFORE THE ILLINOIS POLLUTION CONTROL BOARD**

IN THE MATTER OF: )  
)  
PETITION OF EMERALD PERFORMANCE ) AS 13-2  
MATERIALS, LLC FOR ADJUSTED ) (Adjusted Standard – Water)  
STANDARD FROM 35 ILL. ADM. CODE )  
304.122(b) )

**MOTION TO FILE INSTANTER**

Now comes Emerald Performance Materials, LLC (“Emerald”) by its attorneys, Drinker Biddle & Reath LLP, by and through their counsel, and hereby submits a Motion to File Instanter the attached two documents into the record for use by the Illinois Pollution Control Board in consideration of the requested Adjusted Standard. In support thereof, the following statements are made:

1. Emerald filed its Petition for Adjusted Standard on August 28, 2012 with fourteen Exhibits including Exhibit 13 which was a letter from Mr. T. Houston Flippin, Brown and Caldwell, dated August 27, 2012 to the undersigned.

2. On January 12, 2013, the Illinois Environmental Protection Agency (“Agency”) filed its Recommendation to deny Emerald’s requested relief. The Agency’s Recommendation included 9 conditions that they believed should be included if the Board grants Emerald regulatory relief over the Agency’s objections.

3. On December 17, 2012 the Hearing Officer directed Emerald to provide answers to a number of Questions.

4. Emerald provided detailed responses on April 12, 2013 to these questions.

5. On August 1, 2013 the Hearing Officer directed the Parties to respond to additional questions.

6. Emerald responded on October 8, 2013 to these questions. In this response, Emerald stated that it would provide the results of additional Whole Effluent Toxicity Testing to the Board and to the Agency.

7. Attached as Appendix A is a revised letter dated July 8, 2013 prepared by Mr. Flippin to replace that which was originally submitted as Exhibit 13. A copy of this revised document has been previously provided to the Agency.

8. Attached as Appendix B is copy of the results of Whole Effluent Toxicity Testing dated November 22, 2013. A copy of this testing report has been previously provided to the Agency.

9. Emerald and the Agency have reached an agreement on the recommended conditions that should be included in any regulatory relief granted by the Board and on June 17, 2014 filed an agreed motion setting forth the agreement. Emerald and the Agency also filed a joint motion requesting that the Board rescind or modify its prior order setting forth this case for hearing and requesting that the case be decided based upon the record in the previous case and the information submitted with the petition and the responses to the two Hearing Officer Orders. These motions remain pending. Emerald requests that the Revised Brown and Caldwell report and most recent Whole Effluent Toxicity Report be included into the record for the Board's consideration.

10. The undersigned has been authorized to state that the Agency does not have any objection to this request to include these two Attachments into the Record of this proceeding.

WHEREFORE, for the reasons stated herein, Emerald respectfully requests that the grant this Motion for Leave to File Instantly and accept the two attached documents into the record of this proceeding for consideration in ruling on the requested relief.

Emerald Performance, LLC by its attorney



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**CERTIFICATE OF SERVICE**

Roy M. Harsch herein certifies that he has served a copy of the foregoing Notice of Filing and Motion for Leave to File Instantly on Friday, June 20, 2014, to each person on the attached service list.

It is hereby certified a true copy of the foregoing was hand delivered to the following on Friday, June 20, 2014:

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July 8, 2013



**Privileged and Confidential-Attorney Work Product**

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140975

Subject: Ammonia-Nitrogen Treatment Alternatives For Emerald Performance  
Materials, LLC-Henry, IL Plant

Dear Mr. Harsch:

In November 2004, the Illinois Pollution Control Board (PCB) adopted an Opinion and Order in AS 02-5 that granted Noveon an adjusted standard from the ammonia water quality standard and established a daily maximum effluent limitation of 155 milligrams per liter (mg/L) that was contingent upon several conditions. Subsequently, Emerald Performance Materials LLC (Emerald) purchased the Henry Plant from Noveon and continues to operate it pursuant to a National Pollutant Discharge Elimination System (NPDES) Permit which incorporates the conditions imposed in the PCB Order.

One of these conditions was that Emerald continue to investigate production methods and technologies that contribute less ammonia to Emerald's discharge into the Illinois River. Where practical, Emerald must substitute current methods or technologies with new ones so long as the substitution generates less ammonia in Emerald's discharge. It should be noted that most of the effluent ammonia discharged originates as influent organic nitrogen that is bio-hydrolyzed to ammonia during the treatment provided in the onsite wastewater treatment facility (WWTF). Organic nitrogen compounds serve as building blocks for Emerald products and therefore are used throughout the production processes. Consequently, this evaluation focused on both influent Total Kjeldahl Nitrogen (TKN) and ammonia loadings.

Brown and Caldwell (BC) was involved in the effort to obtain the relief in AS 02-5. The existing chemical processes at the Henry Plant and their associated waste streams were evaluated. After this evaluation, it was determined that there were no economically feasible treatment alternatives that would reliably reduce the effluent ammonia-nitrogen (NH<sub>3</sub>-N) concentrations to comply with the effluent limitations set forth by the PCB. BC previously prepared a report which was used as an exhibit in AS 02-5 and testified in support of the requested relief. The PCB accepted in large part the results of the work as the basis for the relief it granted. The purpose of this letter is to revisit this determination and see what (if any) changes have occurred since 2004.

### **Reductions in Influent and Effluent NH<sub>3</sub>-N Loads**

A comparison of the influent and effluent NH<sub>3</sub>-N loadings from 2002 and 2011 are presented in Tables 1 and 2. Minimal sampling of the influent (3 samples) indicates that the influent nitrogen loading may have increased. Furthermore, the very limited

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 Drinker, Biddle & Reath LLP  
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data indicate that the influent TKN loading may have shifted from the PC Tank to the PVC Tank discharge. This influent data for TKN and NH<sub>3</sub>-N are likely not representative as they stand in contrast to the much more extensive influent chemical oxygen demand (COD) and effluent NH<sub>3</sub>-N data available. Extensive sampling (5 or more days per week) indicates that the influent COD loads summarized in Table 1 have decreased by 38 percent and effluent NH<sub>3</sub>-N loads summarized in Table 2 have decreased by 48 percent. These decreases are principally due to lower COD and TKN loads being discharged through the PC Tank to the influent to the WWTF. This reduction has been attributed to the shutdown of X70 and Geltrol, much lower production of OBTS (2 months every 3 months versus weekly before), much lower production of C-18 (2 weeks every quarter versus monthly before), and improved recovery in the tertiary butyl amine (TBA) column. This production decrease and other production related matters are illustrated in Table 3 for the period of 2002 through 2011.

Emerald is in the process of regaining total production levels previously observed in 2004. As production increases, the effluent flow rate, NH<sub>3</sub>-N load, and effluent NH<sub>3</sub>-N concentration are expected to increase. The extent of this increase will depend on product mix, which is dictated by unpredictable market conditions. The quantity of effluent NH<sub>3</sub>-N discharged as a function of annual production has varied by 38 percent. Any observed reductions in effluent flow rate and NH<sub>3</sub>-N load between 2002 and early 2011 will not be realized as production increases. Consequently, the wasteload estimates and treatment cost estimates presented herein are only applicable for the production levels and product mix present during late 2010 and early 2011. **Emerald will need to retain the current effluent NH<sub>3</sub>-N allocation to allow the plant to comply with effluent limitations while seeking to restore production capacity.**

Table 1. Influent Wasteloads Used in Developing Treatment Alternatives

Parameter	PVC Tank	PC Tank	C-18 Tank	Holding Pond/Well No. 3	Total
<b>Flow Rate, gpm</b>					
2002 Average	401	107	6	46	560
2002 Peak	499	150	15	105	769
2011 <sup>a</sup> Average	345	72	3	118	538
2011 <sup>a</sup> Peak	400	94	3	154	652
<b>SCOD, lbs/day</b>					
2002 Average	2650	8280	1320	50	12300
2002 Peak	4330	10840	2940	50	18160
2011 Average	2514	4396	776	Not Analyzed	7685
2011 Peak	6532	7711	1258	Not Analyzed	15500
<b>Estimated BOD, lbs/day</b>					
2002 Average	795	2485	395	15	3690
2002 Peak	1300	3250	880	15	5445

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**Table 1. Influent Wasteloads Used in Developing Treatment Alternatives**

Parameter	PVC Tank	PC Tank	C-18 Tank	Holding Pond/Well No. 3	Total
2011 Average	754 <sup>a</sup>	1319 <sup>a</sup>	233 <sup>b</sup>	Not Analyzed	2305
2011 Peak	1960 <sup>a</sup>	2313 <sup>a</sup>	377 <sup>b</sup>	Not Analyzed	4650
<b>TKN, lbs/day</b>					
2002 Average	459	494	82	3	1038
2002 Peak	640	693	198	7	1538
2011 Average	1091	287	63	3	1443
2011 Peak	1296	612	74	5	1987
<b>NH<sub>3</sub>-N, lbs/day</b>					
2002 Average	295	62	27	1	385
2002 Peak	411	87	66	3	567
2011 Average	235 <sup>b</sup>	8	21 <sup>c</sup>	1 <sup>c</sup>	265
2011 Peak	469 <sup>b</sup>	8	25 <sup>c</sup>	2 <sup>c</sup>	504

<sup>a</sup> For period of March 2010 to February 2011 for flow and COD data. TKN and NH<sub>3</sub>-N data were gathered during a 3-day period of June 29 through July 1, 2011.

<sup>b</sup> Values estimated based on prior BOD/COD ratio of 0.3.

<sup>c</sup> Value estimated for C-18 based upon previous NH<sub>3</sub>-N/TKN ratio. Value estimated for PVC Tank by calculation using available PVC lift station and side stream data.

**Table 2. Effluent Wasteloads Used in Developing Treatment Alternatives**

Parameter	Effluent Value
<b>Flow Rate, gpm</b>	
2002 Average	560
2002 Peak	769
2011 <sup>a</sup> Average	538
2011 <sup>a</sup> Peak	652
<b>NH<sub>3</sub>-N, lbs/day</b>	
2002 Average	909
2002 Peak	1408
2011 Average	473
2011 Peak	940

<sup>a</sup> For period of March 2010 to February 2011.

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**Table 3. Production and Effluent Quality (2002 through 2011)**

Year	Inter-mediate	Finished	Total Product	NH <sub>3</sub> -N	Flow	NH <sub>3</sub> -N	Flow
	(mm lbs)	(mm lbs)	(mm lbs)	(mm lbs)	(MG)	(lb/lb product)	(gal/lb product)
2002	11.9	23.1	35.0	0.267	276	0.0076	7.9
2003	11.0	22.9	33.9	0.192	274	0.0057	8.1
2004	14.0	26.9	40.9	0.286	315	0.0070	7.7
2006	8.9	20.8	29.7	0.224	332	0.0075	11.2
2007	11.7	24.8	36.5	0.231	320	0.0063	8.8
2008	11.3	22.6	33.9	0.185	328	0.0055	9.7
2009	6.2	14.0	20.2	0.146	314	0.0072	15.5
2010	9.4	19.2	28.6	0.179	285	0.0063	10.0
2011	8.9	18.6	27.5	0.206	289	0.0075	10.5

## Changes in WWTF Operations

The WWTF has made the following changes since 2002.

1. Implemented carbon dioxide (CO<sub>2</sub>) addition plus 400 gallons per day (gpd) of 98 percent sulfuric acid to PC Tank versus prior use of acid only.
2. Synthetic flocculent addition only in primary treatment versus prior ferric chloride and anionic flocculent additions.
3. Synthetic flocculent and synthetic coagulant additions in secondary treatment versus prior alum and anionic flocculent additions.
4. Operation of West and North biotreaters now, versus prior operation of East and Center biotreaters also (1.3 million gallons volume versus 1.9 million gallons of prior biotreater volume).

These changes appear not to have caused any appreciable change in effluent quality based on the average effluent biochemical oxygen demand (BOD) and COD remaining at approximately 8 mg/L and 370 mg/L, respectively, from 2002 through 2011. Recent sampling indicates that the effluent NH<sub>3</sub>-N and TKN continue to remain comparable (within 10 percent of each other) indicating near complete hydrolysis of organic nitrogen.

The WWTF still operates at conditions that would promote biological nitrification (Mean Cell Residence Time greater than 30 days, mixed liquor temperatures and dissolved oxygen (DO) concentrations of 80 to 96 degrees Fahrenheit and 1.5 to 4.5 mg/L DO, respectively, effluent alkalinity of greater than 150 mg/L, and effluent orthophosphate-phosphorus concentrations of greater than 0.5 mg/L). The lack of nitrification continues to be due to bio-inhibition to nitrifying bacteria as discussed in Attachment A. This inhibition prevents nitrification of the primary clarifier effluent even after 16-fold dilution with "inhibition free water". This inhibition would also require the secondary clarifier effluent to be diluted 5-fold to promote inhibition free nitrification.

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This finding of significant nitrification inhibition is consistent with our prior evaluations. This inhibition has been largely attributed to the presence of mercaptobenzothiazole in the wastewater. This compound is the building block for the products made at the Emerald plant and has a published nitrification threshold of less than 3 mg/L<sup>1</sup>. The presence of this inhibitor and the complex nature of the Henry Plant influent wastewater render nitrification alternatives for effluent NH<sub>3</sub>-N control at the Henry Plant not reliable.

## Previously Considered Treatment Alternatives

Numerous treatment alternatives were previously considered for reduction of effluent NH<sub>3</sub>-N<sup>2</sup>. All but three of these alternatives were reconsidered. Nitrification alternatives were not reconsidered due to their prior poor economic viability and the continued presence of significant nitrification inhibition, which made these treatment alternatives of questionable reliability. The reconsidered alternatives are listed below, illustrated in Attachment B, redefined in terms of impact and costs in Attachment C, and discussed in terms of reliability in Attachment D.

- Alkaline air stripping of PC Tank contents with off-gas collection and treatment (prior Treatment Alternative No. 1 or No. 1)
- Alkaline air stripping of PVC Tank contents (No. 2)
- Alkaline air stripping of secondary clarifier effluent (No. 3)
- Struvite (NH<sub>4</sub>MgPO<sub>4</sub> · 6H<sub>2</sub>O) precipitation from combined influent (No. 4)
- Breakpoint chlorination of secondary clarifier effluent (No. 5)
- Ion exchange treatment of final effluent (No. 8)
- Ozonation of final effluent (No. 9)

Costs that had been developed in the prior document were scaled by a series of factors to produce equivalent costs for 2011. Emerald provided current costs for labor, electricity, sodium hydroxide, sulfuric acid, and phosphoric acid. Costs for magnesium hydroxide, hydrochloric acid, and chlorine gas were obtained from Brenntag, a national chemical supplier. A cost for resin was obtained from Dow Chemical. The cost of natural gas was taken from an industry average. All remaining costs were updated for inflation using Engineering News Record (ENR) Construction Indices.

After making adjustments for 2011, quantities were scaled based upon loading. All capital costs, equipment costs, and power requirements were updated using the Rule of Six-Tenths<sup>3</sup> and the loading corresponding to the alternative. Chemical and resin costs were assumed to be directly proportional to the corresponding loading. Each item was then scaled using a ratio of the loadings from 2011 and 2002. Table 4 below indicates the loading corresponding to each alternative.

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<sup>1</sup> Journal of Water Pollution Control Federation, Volume 48, 1976 by M.R. Hockenbury and C.P.L. Grady.

<sup>2</sup> Ammonia-Nitrogen Treatment Alternatives Support Exhibit developed by Brown and Caldwell on May 17, 2002 and held by Illinois Pollution Control Board.

<sup>3</sup> "Six-tenths Factor Applies to Complete Plant Costs", C.H. Chilton, Chemical Engineering, Volume 57, No. 4, page 112, 1959.

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**Table 4. Loading Scales for Treatment Alternatives**

Alternative Number	Loading Used for Scaling
1	PC Tank Flow Rate
2	PVC Tank Flow Rate
3	Effluent Flow Rate
4	Influent NH <sub>3</sub> -N
5	Effluent NH <sub>3</sub> -N
8	Effluent Flow Rate, Effluent NH <sub>3</sub> -N
9	Effluent TKN

For alternatives involving stripping (Nos. 1, 2, and 3), the loading used for scaling is flow rate because the amount of aeration and quantity of chemicals are both directly proportional to the volume of water treated. For struvite precipitation (No. 4), the loading used for scaling is influent NH<sub>3</sub>-N because NH<sub>3</sub>-N is precipitated from the influent as struvite. For breakpoint chlorination (No. 5), the loading used for scaling is effluent NH<sub>3</sub>-N because NH<sub>3</sub>-N is removed as nitrogen gas after reacting with chlorine. For ion exchange (No. 8), the loading used for scaling is based upon both effluent flow and effluent NH<sub>3</sub>-N. Ion exchange scales with flow because it is based upon the volume of water treated. However, the quantity of hydrochloric acid used for regeneration of the resin scales with effluent NH<sub>3</sub>-N. Finally, ozonation (No. 9) scales with effluent TKN because both NH<sub>3</sub>-N and organic nitrogen are oxidized by ozone.

A summary of conceptual level comparative capital costs for each of these alternatives is provided in Table 5. The total costs presented in this table are considered accurate to within ±50 percent.

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**Table 5. Capital Cost Estimates For Treatment Alternatives**

Cost Components	Upgrade Cost in Millions of Dollars for Treatment Alternative Number						
	1	2	3	4	5	8	9
Pretreatment	0.71	0.13	--	0.06	--	--	--
Primary Treatment	--	--	--	--	--	--	--
Secondary Treatment	--	--	--	--	--	--	--
Tertiary Treatment	--	--	5.7	--	0.70	0.77	6.35
Sub-total	0.71	0.13	5.7	0.06	0.70	0.77	6.35
Site work/Interface Piping	0.11	0.01	0.43	0.01	0.10	0.12	0.28
Electrical/Instrumentation	0.27	0.20	0.54	0.18	0.24	0.32	0.69
Contractor Indirects (8%)	0.06	0.01	0.45	0.00	0.06	0.06	0.51
Engin./Constr. Mgmt (18%)	0.13	0.02	1.02	0.01	0.13	0.14	1.1
Performance Bonds (1%)	0.007	0.001	0.057	0.001	0.007	0.008	0.063
Sub-total	1.3	0.37	8.2	0.26	1.2	1.4	9.0
Contingency (15%)	0.19	0.08	1.2	0.04	0.19	0.21	1.4
Total Installed Cost	1.5	0.43	9.4	0.30	1.4	1.6	10.4

A summary of conceptual level operations and maintenance costs for each of these alternatives is provided in Table 6. The total costs presented in this table are considered accurate to within  $\pm 50$  percent.

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**Table 6. Annual Operating and Maintenance Cost Estimates for Treatment Alternatives**

Cost Components	Annual O/M Costs in Thousands of Dollars for Treatment Alternative Number						
	1	2	3	4	5	8	9
Labor (\$40/hr)	32	32	60	8.0	60	60	30
Electrical (\$0.039/kWh)	33	18	136	0.2	2	6	886
Natural Gas (\$0.06/therm)	12	0.0	0.0	0.0	0.0	0.0	0.0
Chemicals (Plant Costs)	393	3,259	1,428	1,294	1,460	309	471
Resin Replacement (\$215.50/CF)	0.0	0.0	0.0	0.0	0.0	302	0.0
Off-Site Disposal <sup>a</sup>	0.0	0.0	0.0	0.0	0.0	36	0.0
Maintenance Materials <sup>b</sup>	18	2.5	142	0.8	18	19	159
Sub-total	488	3,311	1,766	1,303	1,539	733	1,545
Contingency (10%)	48.8	331	177	130	154	73	155
Total Annual	536	3,643	1,942	1,433	1,692	806	1,699

<sup>a</sup> Cost of disposing spent regenerant containing 29.7 percent by weight  $\text{NH}_4\text{Cl}$  (8 percent N) assumed to be \$0.14/gallon. Does not include costs of excess sludge disposal from Alternative No. 4.

<sup>b</sup> Based on 5 percent equipment costs.

A comparison of alternatives with respect to total annual costs and ammonia removal is provided in Table 7.

**Table 7. Comparison of Total Annual Costs and Ammonia Removal for Treatment Alternatives**

Components	Total Annual Costs in Thousands of Dollars						
	1	2	3	4	5	8	9
$\text{NH}_3\text{-N}$ Removal, lbs/day	7	212	449	88	464	464	464
$\text{NH}_3\text{-N}$ Removal, %	2	45	95	19	98	98	98
Total Annual Costs							
Capital <sup>a</sup>	177	52	1131	36	171	196	1248
O/M <sup>b</sup>	403	4176	2227	1643	1940	924	1948
Total	580	4228	3357	1678	2111	1121	3196
Total, \$/lb $\text{NH}_3\text{-N}$ removed	227	55	20	52	12	6.6	19

<sup>a</sup> Based on a 10-year period, 3.5 percent annual interest and no salvage value.

<sup>b</sup> Based on 10 year period and 3.0 percent inflation rate.

The minimum total annual cost for a 98 percent reduction in effluent  $\text{NH}_3\text{-N}$  is \$1,121,000 per year at \$6.60/lb  $\text{NH}_3\text{-N}$  removed provided under Alternative 8. If 25 percent reduction were provided under Alternative 8, the total annual cost would be \$343,000 per year at a cost of \$8.10/lb  $\text{NH}_3\text{-N}$  as described in Attachment C.

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## **New Treatment Technologies**

Since 2004, several new treatment technologies have become demonstrated, which could provide effluent NH<sub>3</sub>-N reduction at the Henry Plant. However, none of these technologies are as economically viable as the ones discussed above.

### **CASTion Ammonia Recovery Process (ARP)**

This process removes ammonia by combining stripping with ion exchange. The waste stream is first conditioned to volatilize ammonia for capture by vacuum distillation. Subsequently, the waste stream is exposed to an ion exchange resin. This process is more costly to build and operate than the separate alkaline air stripping and ion exchange alternatives considered above.

### **Ostara Pearl**

The Ostara Pearl process recovers nutrients from wastewater, including phosphorus and nitrogen containing compounds, and, subsequently, combines these nutrients with magnesium hydroxide to precipitate struvite. Ostara Pearl is simply struvite precipitation that has been considered above, but under a proprietary name.

### **Liqui-Cel Membrane**

The Liqui-Cel Membrane uses a membrane module to separate ammonia from a waste stream. The ammonia is then converted to ammonium salt. Since stripping is part of the process, the Liqui-Cel Membrane similarly requires a pH of greater than 10. As previously determined with alkaline air stripping, pH control would be required to elevate pH for stripping and lower pH for effluent discharge. Additionally, the Liqui-Cel Membrane requires a temperature of 40 to 55 degrees Celsius. The power requirements to heat the waste stream would be expensive. The overall costs and impact would not be as viable as alkaline air stripping alternatives considered above.

### **Anammox**

Anammox is a biological process that removes ammonia through anaerobic biological treatment. These systems are more subject to process upsets than aerobic biological nitrification that was discounted at the Henry Plant due to the presence of known bio-inhibitors and the complexity of site-wide wastewaters.

### **Anodic Oxidation**

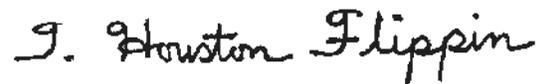
Anodic oxidation is capable of removing ammonia from waste streams by electrochemical oxidation. By applying a current to the wastewater, ammonia is removed by deposition on the anode. In order to achieve anodic oxidation at the Emerald facility, the power cost alone would be at least \$5 million annually. In addition, significant capital would be required to outfit the facility for this process. Finally, this process has only been proven to remove ammonia at the bench-scale; no full-scale facility currently exists.

Please call me at 615-250-1220 to discuss this report at your convenience.

Mr. Roy M. Harsch, Esq.  
Drinker, Biddle & Reath LLP  
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Very truly yours,

**Brown and Caldwell**



T. Houston Flippin, P.E., BCEE  
Industrial Wastewater Process Leader

MEM:ter

Attachments (4)

1. Attachment A: Nitrification Testing
2. Attachment B: Alternative Process Flow Schematics
3. Attachment C: Cost Analysis for Treatment Alternatives
4. Attachment D: Reliability Comparison

*Limitations:*

*This document was prepared solely for Emerald Performance Materials in accordance with professional standards at the time the services were performed and in accordance with the contract between Emerald Performance Materials and Brown and Caldwell on April 5, 2011. This document is governed by the specific scope of work authorized by Emerald Performance Materials; it is not intended to be relied upon by any other party except for regulatory authorities contemplated by the scope of work. We have relied on information or instructions provided by Emerald Performance Materials and other parties and, unless otherwise expressly indicated, have made no independent investigation as to the validity, completeness, or accuracy of such information.*

**Attachment A: Nitrification Testing**

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## Technical Memorandum

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Privileged and Confidential-Attorney/Client Work Product

Prepared for: Emerald Performance Materials  
Project Title: Renewal of Adjustment Standard  
Project No: 140975

### Technical Memorandum

Subject: Nitrification Testing  
Date: July 7, 2011  
To: Mike Strabley, HSE Manager  
From: T. Houston Flippin, P.E., BCEE  
Copy to: Steve McGuire, P.E.

Prepared by: Michael Mecredy  
Michael Mecredy, Project Engineer

Reviewed by: Steve McGuire  
Steve McGuire, Project Manager

Reviewed by: T. Houston Flippin  
T. Houston Flippin, P.E., BCEE, Industrial Wastewater Process Leader

#### Limitations:

*This is a draft memorandum and is not intended to be a final representation of the work done or recommendations made by Brown and Caldwell. It should not be relied upon; consult the final report.*

*This document was prepared solely for Emerald Performance Materials in accordance with professional standards at the time the services were performed and in accordance with the contract between Emerald Performance Materials and Brown and Caldwell. This document is governed by the specific scope of work authorized by Emerald Performance Materials; it is not intended to be relied upon by any other party except for regulatory authorities contemplated by the scope of work. We have relied on information or instructions provided by Emerald Performance Materials and other parties and, unless otherwise expressly indicated, have made no independent investigation as to the validity, completeness, or accuracy of such information.*

## Section 1 Introduction

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### 1.1 Background

The combined wastewater generated at the Emerald Performance Materials- Henry Plant has historically contained high concentrations of Total Kjeldahl Nitrogen (TKN) and ammonia-nitrogen ( $\text{NH}_3\text{-N}$ ), as well as a known nitrification-inhibiting compound, mercaptobenzothiazole (MBT). This known inhibitor is the compound that serves as the foundational building block of essentially all products at the Henry Plant. Several bench-scale tests have previously been performed to evaluate the viability of nitrification of both the principal wastewaters (PVC Tank and PC Tank discharges) that comprise the primary clarifier influent and the secondary clarifier effluent. On all previous occasions, nitrification has been inhibited, despite sufficient nutrients, and carefully controlled pH, alkalinity, orthophosphate-phosphorus ( $\text{PO}_4\text{-P}$ ) and dissolved oxygen (DO) concentrations.

Emerald personnel collected the following samples on May 30, 2011.

- One-gallon sample of Return Activated Sludge (RAS)
- One-gallon sample of Primary Effluent
- One-gallon sample of Secondary Effluent
- One-gallon sample of PC Tank

All samples, except RAS, were kept under refrigeration until treatability testing was performed. The RAS was mixed and aerated until testing, and deionized water was added, as necessary, to maintain volume.

### 1.2 Scope of Work

In order to determine the extent to which the wastewaters are inhibitory to nitrification, three Fed Batch Reactor (FBR) tests were performed. Table 1 below provides the general setup for the three tests.

Test	Type	Biomass	Wastewater
Test 1	FBR	Nitrifiers <sup>a</sup>	Tap Water with $\text{NH}_4\text{Cl}$
Test 2	FBR	Nitrifiers <sup>a</sup>	Secondary Effluent
Test 3	FBR	RAS <sup>a</sup> + Nitrifiers <sup>a</sup>	Primary Effluent

<sup>a</sup> Washed with Total Dissolved Solids (TDS)-adjusted tap water to remove any soluble inhibitory compounds.

The first test was a control containing pure culture nitrifiers designed to obtain an uninhibited nitrification rate. The second test investigated the extent to which the secondary effluent is inhibitory to nitrification. The third test evaluated the extent to which the primary effluent is inhibitory to nitrification.

## Section 2 Results

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### 2.1 Characterization

The samples provided by Emerald Performance Materials were characterized and the results are shown below in Table 2.

Table 2. Characterization of Samples

Parameter	Primary Clarifier Effluent	Secondary Clarifier Effluent
TCOD, mg/L	1,653	350
FCOD, mg/L	1260	Not Analyzed
TSS, mg/L	155	14
VSS, mg/L	137	8
NH <sub>3</sub> -N, mg/L	49	113
PO <sub>4</sub> -P, mg/L	0	0
Alkalinity, mg/L as CaCO <sub>3</sub>	1,000	500

A comparison of the NH<sub>3</sub>-N results indicates that significant quantity of organic nitrogen is converted to NH<sub>3</sub>-N at the Henry Plant, which has always been the case. The effluent alkalinity is sufficient to support nitrification and absent inhibition, with an effluent alkalinity much greater than 150 mg/L. The primary and secondary clarifier effluents contained inadequate PO<sub>4</sub>-P to support unhindered biological treatment on May 30, 2011. Samples were supplemented with phosphorus prior to testing. There was a higher nitrogen loading in the secondary effluent.

## 2.2 Fed Batch Reactor (FBR) Testing

During a FBR test, a wastewater is fed to a batch reactor with a fixed biomass population. This configuration allows for the fraction of wastewater in the beaker to increase over time. Thus, the nitrification rate as well as the fraction of wastewater inhibitory to the biomass can be ascertained from the results.

Alkalinity was added, as necessary, to the wastewaters as sodium bicarbonate (NaHCO<sub>3</sub>) to provide sufficient alkalinity for complete nitrification. Dipotassium phosphate (K<sub>2</sub>HPO<sub>4</sub>) was added to provide a total phosphorus concentration of 30 mg/L. The pH in all tests was maintained between 7.0 and 8.5. The DO was maintained above 2.0 mg/L.

The average nitrification rate observed for the control reactor was 0.61 mg N removed per mg volatile suspended solid (VSS) nitrifier per day (mg/mg-day). A nitrification rate in the range of 0.6 mg/mg-day to 1.0 mg/mg-day is typically observed when nitrifying bacteria are uninhibited. Figure A-1 below illustrates the control nitrification rate during the course of the test.

## Nitrification Testing

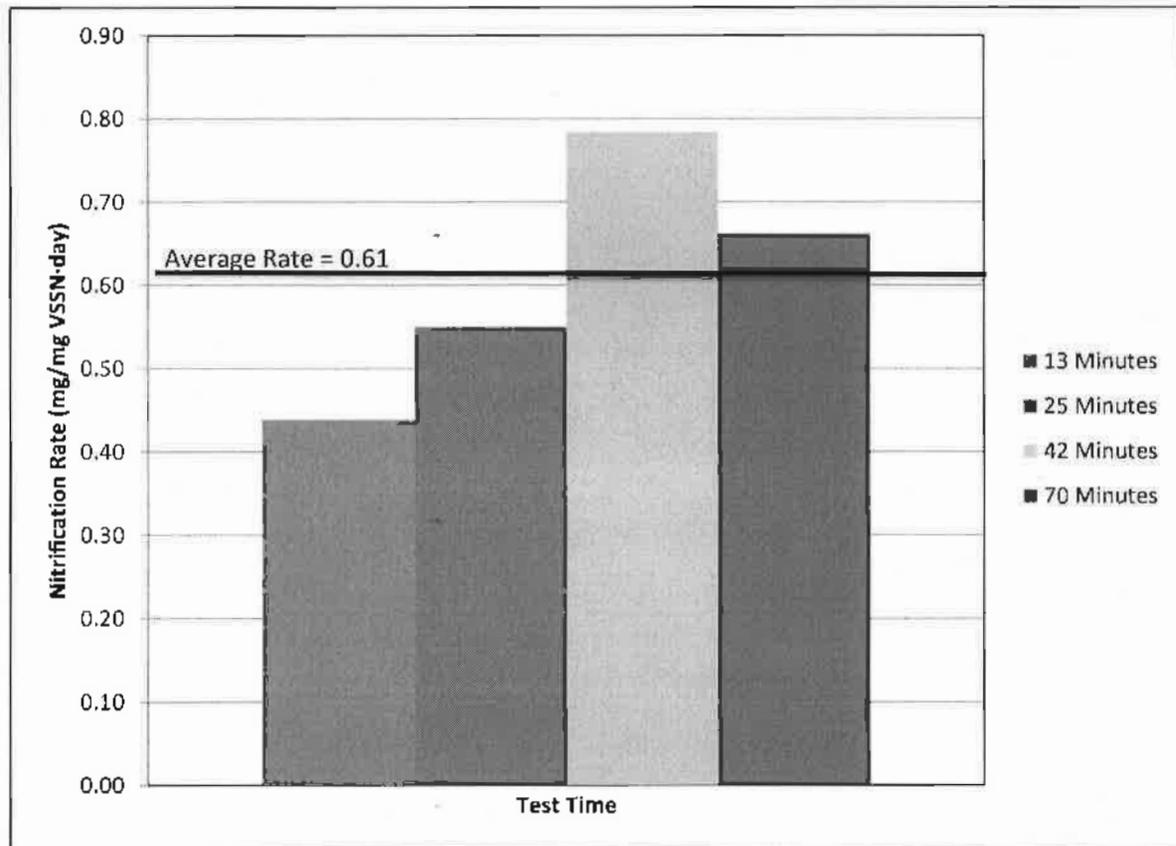


Figure A-1: Control Nitrification Rate as a Function of Time

In contrast, the nitrification rate for the secondary effluent test peaked at 0.46 mg/mg-day at 21 percent secondary clarifier effluent contribution by volume. At 0.46 mg/mg-day, the test may have already been exhibiting inhibition. The nitrification rate dropped during the remainder of the test and reached 0 mg/mg-day by the end of the test when the secondary clarifier effluent contribution reached 95 percent by volume contribution. Figure A-2 below illustrates the nitrification rates during the course of the test.

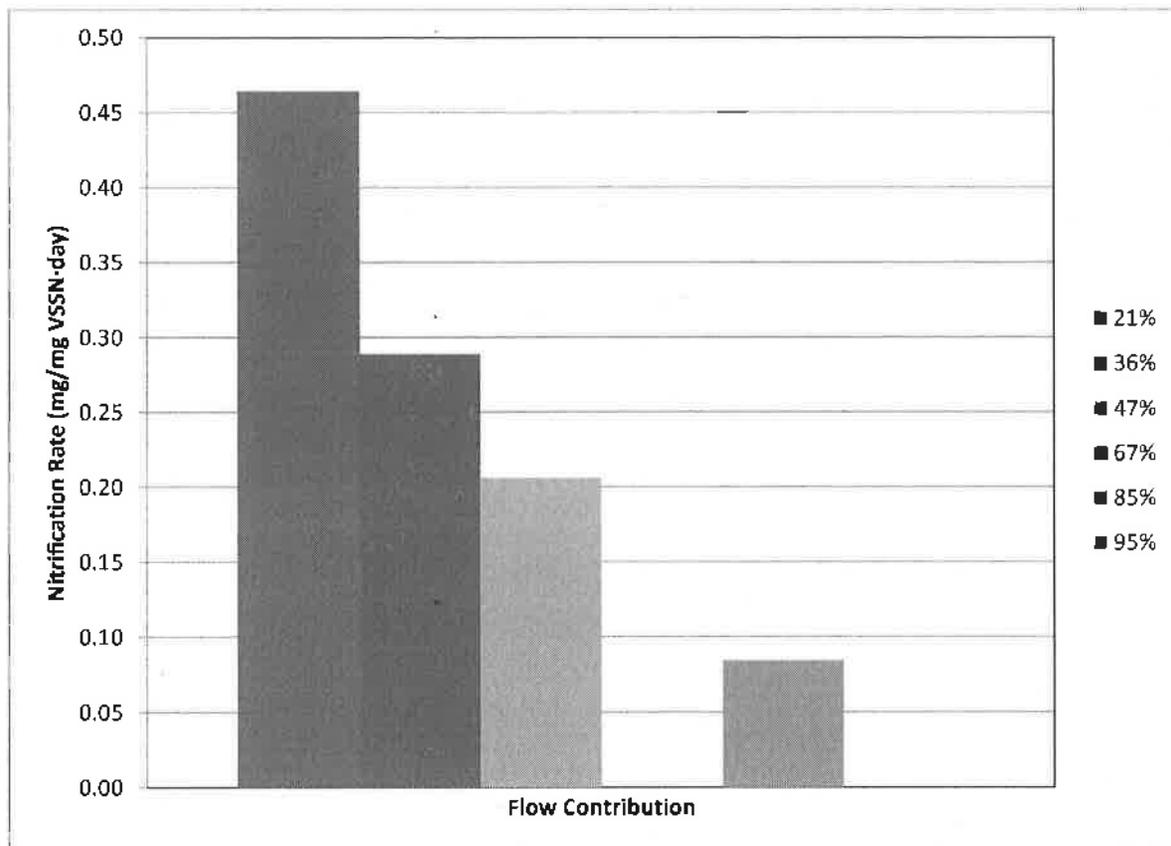


Figure A-2: Nitrification Rate as a Function of Secondary Effluent Flow Contribution

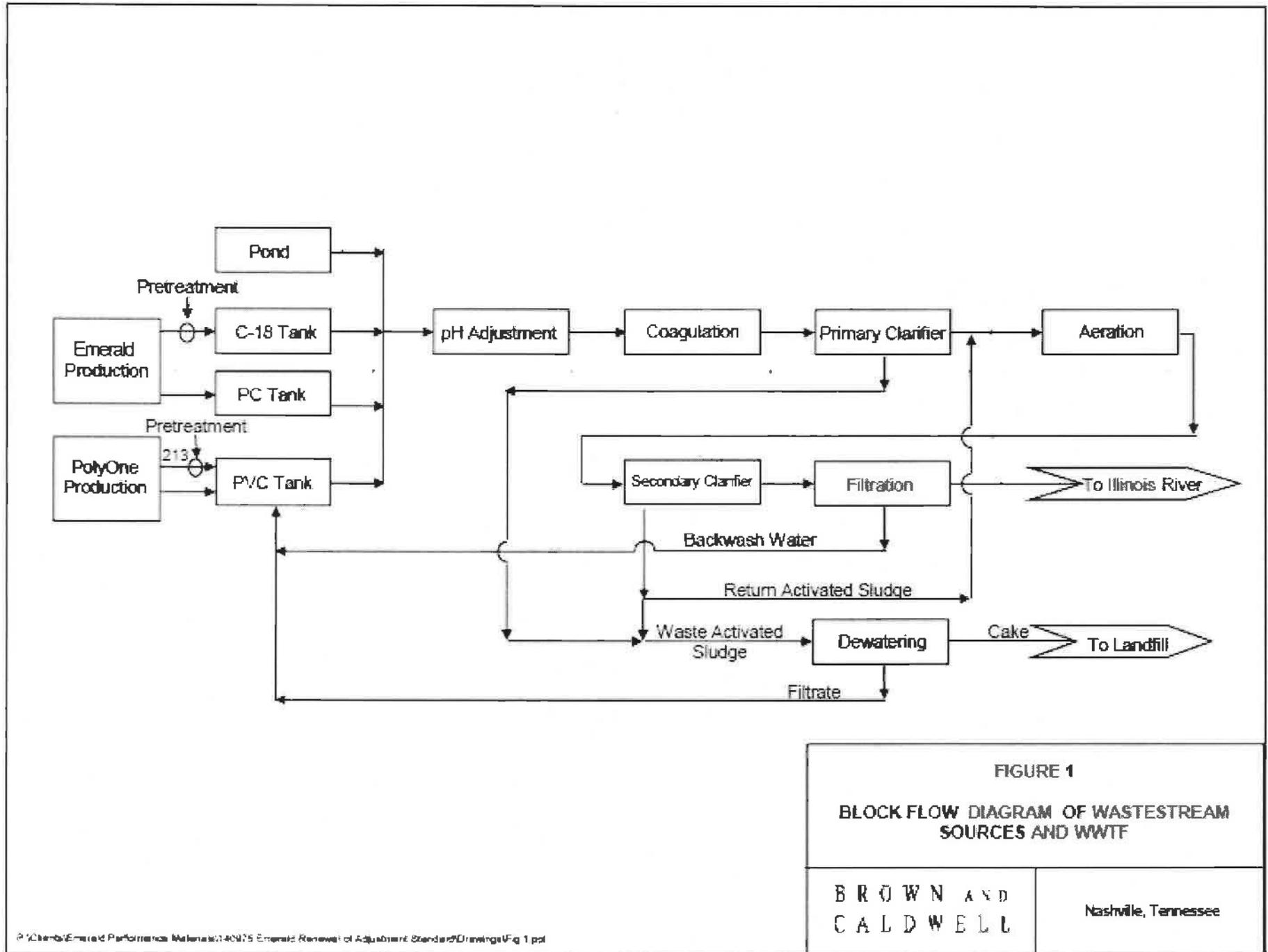
Figure A-2 demonstrates a downward trend toward a zero nitrification rate and indicates that secondary clarifier effluent is inhibitory to nitrification even at low wastewater contribution.

Demonstrating even higher levels of inhibition than the secondary effluent, the primary clarifier effluent test produced a nitrification rate of 0.006 mg/mg-day at 6 percent by volume contribution. In order to corroborate this result, 20-fold washed RAS was combined with pure culture nitrifiers, provided  $\text{NH}_3\text{-N}$  as ammonium chloride in the presence of excess alkalinity and phosphorus, and allowed to aerate overnight. Even with no primary effluent, the observed nitrification rate was 0.06 mg/mg-day. Lastly, the TKN hydrolysis to  $\text{NH}_3\text{-N}$  during the primary clarifier effluent biological treatment test was only 34 percent versus near complete hydrolysis typically being achieved. This may have been due to the test being conducted on a RAS sample that was not freshly collected. Consequently, the FBR test of the primary clarifier effluent likely exhibited stronger inhibition than would have with a freshly collected RAS sample. The primary clarifier effluent exhibited nitrification inhibition in testing at less than 15 percent by volume contribution versus less than 6 percent in this test.

### 2.3 Summary

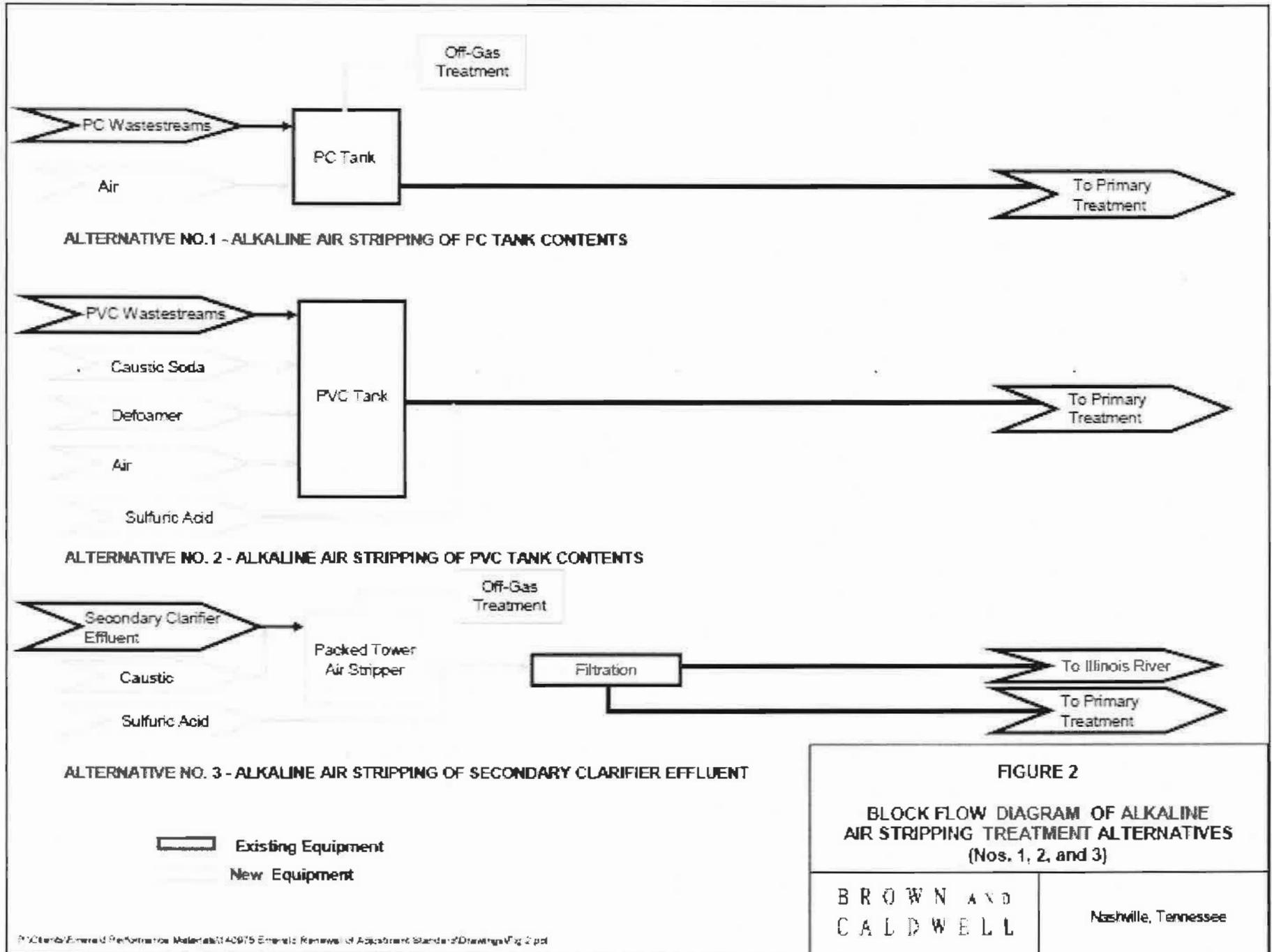
Based upon the performed FBR testing, both the primary and secondary clarifier effluents continue to be significantly inhibitory to nitrification. This finding kept Brown and Caldwell from considering nitrification as a reliable method of effluent  $\text{NH}_3\text{-N}$  control in the associated report.

**Attachment B: Alternative Process Flow Schematics**



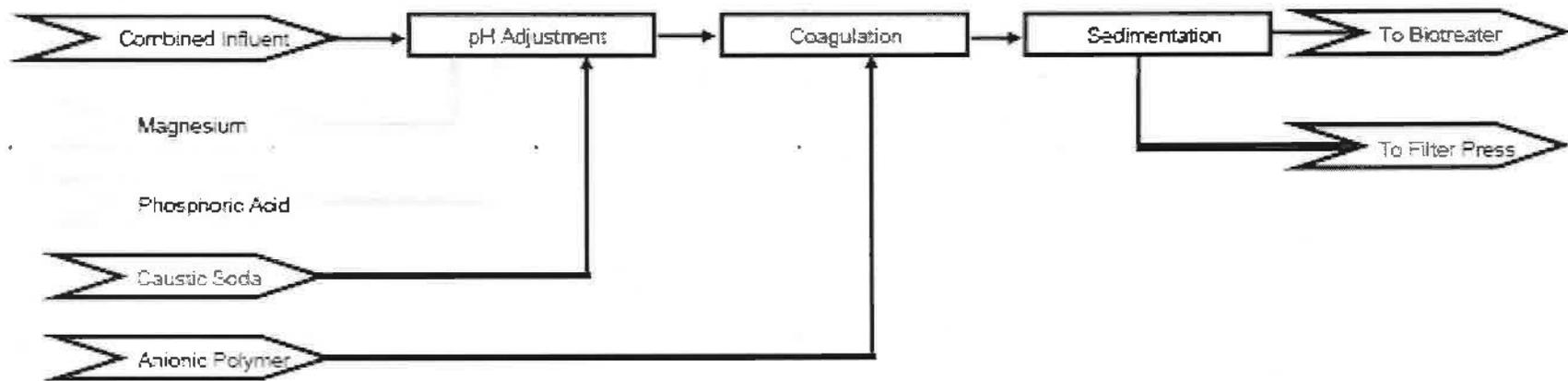
**FIGURE 1**  
**BLOCK FLOW DIAGRAM OF WASTESTREAM**  
**SOURCES AND WWTF**

<b>BROWN AND</b> <b>CALDWELL</b>	Nashville, Tennessee
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**FIGURE 2**  
**BLOCK FLOW DIAGRAM OF ALKALINE AIR STRIPPING TREATMENT ALTERNATIVES (Nos. 1, 2, and 3)**

BROWN AND CALDWELL	Nashville, Tennessee
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NOTE: Existing  $FeCl_3$  Addition would be discontinued

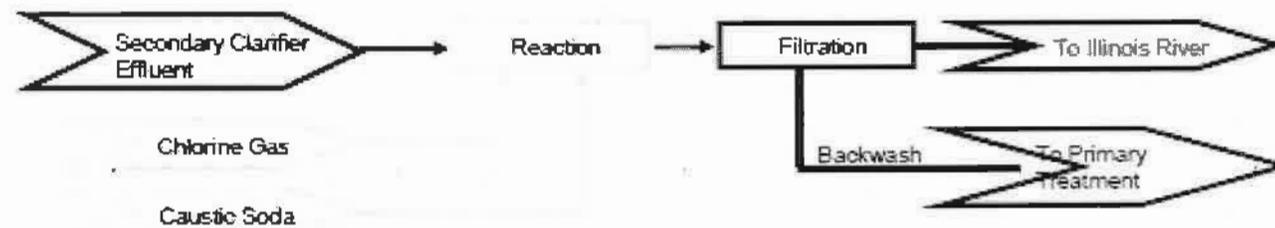
Existing Equipment  
New Equipment

FIGURE 3

BLOCK FLOW DIAGRAM OF STRUVITE  
PRECIPITATION TREATMENT ALTERNATIVE  
(No. 4)

BROWN AND  
CALDWELL

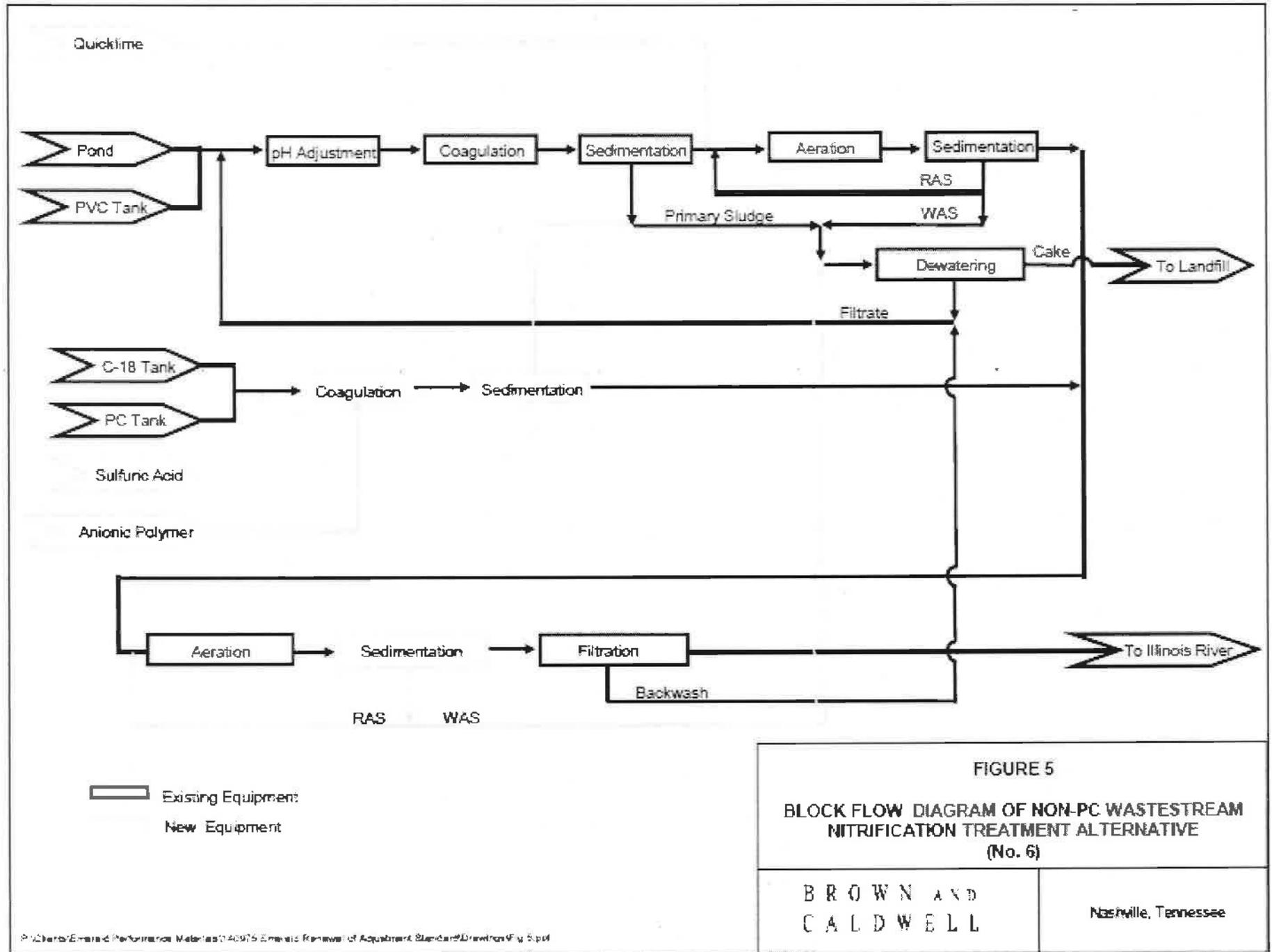
Nashville, Tennessee

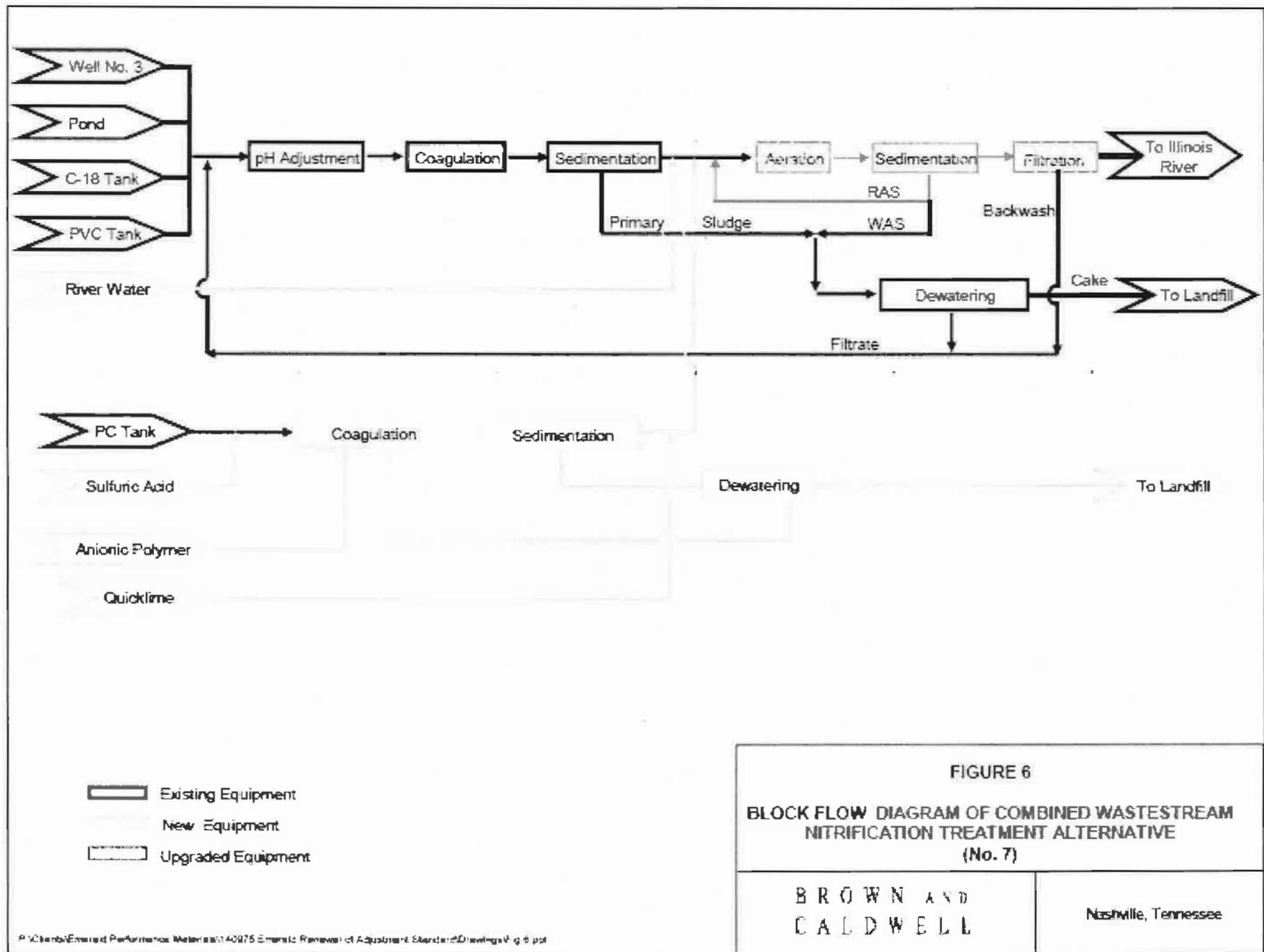


Existing Equipment  
New Equipment

**FIGURE 4**  
**BLOCK FLOW DIAGRAM OF BREAKPOINT CHLORINATION ALTERNATIVE (No. 5)**

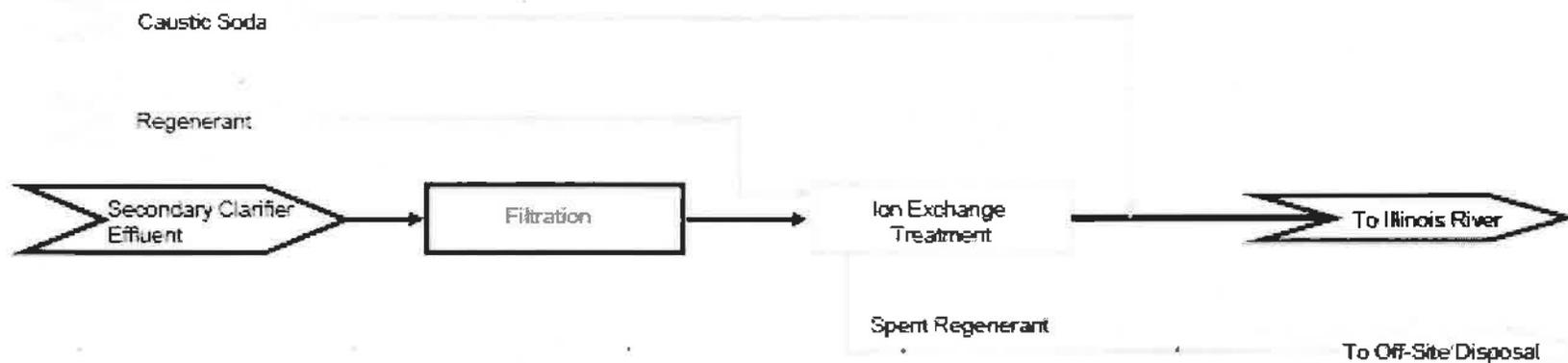
<b>BROWN AND CALDWELL</b>	Nashville, Tennessee
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**FIGURE 6**  
**BLOCK FLOW DIAGRAM OF COMBINED WASTESTREAM**  
**NITRIFICATION TREATMENT ALTERNATIVE**  
**(No. 7)**

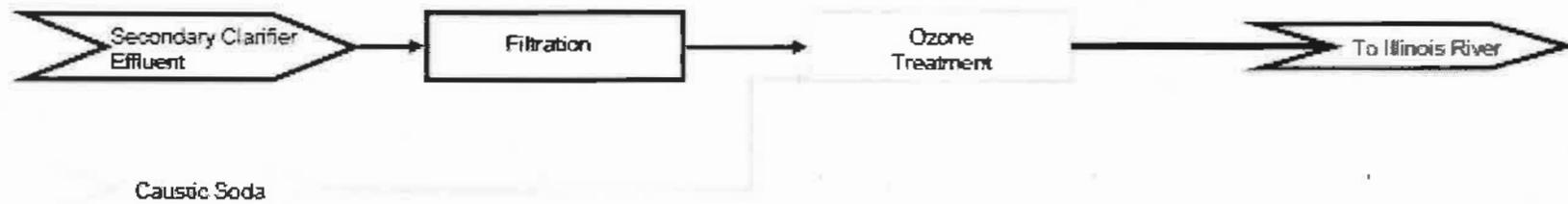
BROWN AND CALDWELL	Nashville, Tennessee
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 Existing Equipment  
 New Equipment

**FIGURE 7**  
**BLOCK FLOW DIAGRAM OF ION EXCHANGE TREATMENT ALTERNATIVE (No. 8)**

<b>BROWN AND CALDWELL</b>	Nashville, Tennessee
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Existing Equipment  
New Equipment

FIGURE 8  
BLOCK FLOW DIAGRAM OF OZONE  
TREATMENT ALTERNATIVE  
(No. 9)

BROWN AND CALDWELL	Nashville, Tennessee
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## **Attachment C: Cost Analysis for Treatment Alternatives**

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Summary of Cost Analysis for Providing Incremental Ammonia-Nitrogen Removal at the Emerald Performance Materials Facility

WWTF Component	Basis	PC Tank Stripping w/ Off-gas 2% Removal	PC Tank Stripping w/ Off-gas 1% Removal	PVC Tank Stripping w/o Off-gas 45% Removal	PVC Tank Stripping w/o Off-gas 22% Removal	Effluent Stripping w/ Off-gas	Effluent Stripping No Off-gas	Effluent Stripping No Off-gas 75% Removal	Effluent Stripping No Off-gas 50% Removal	Effluent Stripping No Off-gas 25% Removal	Struvite Precipitation 19% Removal	Struvite Precipitation 9% Removal	Effluent BP Chlorination	Effluent Ion Exchange	Effluent Ion Exchange 75% Removal	Effluent Ion Exchange 50% Removal	Effluent Ion Exchange 25% Removal	Ozonation
Alternative No.		1	1	2	2	3	3	3	3	3	4	4	5	8	8	8	8	9
Additional Operations/Maintenance Labor																		
Labor Hours		800	800	800	800	1500	1300	1300	1000	1000	200	200	1500	1500	1500	1500	1500	750
Annual Cost	\$40/hr	\$32,000	\$32,000	\$32,000	\$32,000	\$60,000	\$52,000	\$52,000	\$40,000	\$40,000	\$8,000	\$8,000	\$60,000	\$60,000	\$60,000	\$60,000	\$60,000	\$30,000
Electrical Usage																		
hp		128	128	69	69	532	493	439	293	293	1	1	7	24	18	12	6	3,475
kwh		834,694	834,694	447,826	447,826	3,476,930	3,221,743	2,870,860	1,913,907	1,913,907	5,223	5,223	44,159	159,492	119,619	79,746	39,873	22,709,729
Annual Cost	\$0.039/kwh	\$32,553	\$32,553	\$17,465	\$17,465	\$135,600	\$125,648	\$111,964	\$74,642	\$74,642	\$204	\$204	\$1,722	\$6,220	\$4,665	\$3,110	\$1,555	\$885,679
Maintenance Materials																		
Low End Equipment Cost		\$359,238	\$359,238	\$50,462	\$50,462	\$2,838,660	\$1,703,196	\$1,366,223	\$851,598	\$510,959	\$16,552	\$16,552	\$349,871	\$382,801	\$306,241	\$229,681	\$114,840	\$3,173,151
Annual Cost	5% of Equipment Costs	\$17,962	\$17,962	\$2,523	\$2,523	\$141,933	\$85,160	\$68,311	\$42,580	\$25,548	\$828	\$828	\$17,494	\$19,140	\$15,312	\$11,484	\$5,742	\$158,658
Chemical Costs																		
50% NaOH	\$500/ton	\$266,350	\$152,200	\$3,173,310	\$1,813,320	\$868,646	\$868,646	\$868,646	\$434,323	\$217,161	\$0	\$0	\$1,035,870	\$259,915	\$194,937	\$129,957	\$64,978	\$470,529
98% H2SO4	\$190/ton	\$126,502	\$72,295	\$86,133	\$49,224	\$559,512	\$475,585	\$475,585	\$279,756	\$139,878	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
75% H3PO4	\$1,168/ton	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$977,121	\$977,121	\$0	\$0	\$0	\$0	\$0	\$0
62% Mg(OH)2	\$430/ton	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$316,430	\$158,215	\$0	\$0	\$0	\$0	\$0	\$0
38% HCl	\$150/ton	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$49,012	\$36,759	\$24,506	\$12,253	\$0
Chlorine Gas	\$560/ton	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$423,581	\$0	\$0	\$0	\$0	\$0
Annual Cost		\$392,852	\$224,495	\$3,259,443	\$1,862,544	\$1,428,157	\$1,344,231	\$1,344,231	\$714,079	\$357,039	\$1,293,551	\$1,135,336	\$1,459,451	\$308,927	\$231,696	\$154,463	\$77,231	\$470,529
Annual Resin Replacement	\$215.50/CF	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$302,080	\$226,561	\$151,040	\$75,520	\$0
Annual Off-site Disposal	\$0.14/gal	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$36,445	\$27,333	\$18,222	\$9,111	\$0
Natural Gas Cost	\$0.06/therm	\$12,274	\$12,274	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0
Annual Cost		\$12,274	\$12,274	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$0	\$338,525	\$253,894	\$169,262	\$84,631	\$0
Subtotal Annual Costs		\$487,641	\$319,284	\$3,311,431	\$1,914,532	\$1,765,691	\$1,607,039	\$1,576,505	\$871,301	\$497,230	\$1,302,582	\$1,144,367	\$1,538,667	\$732,813	\$565,567	\$398,318	\$229,160	\$1,544,866
Contingency (10%)		\$48,764	\$31,928	\$331,143	\$191,453	\$176,569	\$160,704	\$157,651	\$87,130	\$49,723	\$130,258	\$114,437	\$153,867	\$73,281	\$56,557	\$39,832	\$22,916	\$154,487
Total Annual O/M Costs, \$/year		\$536,405	\$351,212	\$3,642,574	\$2,105,986	\$1,942,260	\$1,767,742	\$1,734,156	\$958,431	\$546,953	\$1,432,840	\$1,258,804	\$1,692,533	\$806,094	\$622,124	\$438,150	\$252,076	\$1,699,353
Total Annual O/M Costs, \$/year	10 years, 3 percent	\$614,928	\$402,625	\$4,175,803	\$2,414,277	\$2,226,583	\$2,026,519	\$1,988,015	\$1,098,734	\$627,020	\$1,642,591	\$1,443,078	\$1,940,300	\$924,097	\$713,195	\$502,290	\$288,976	\$1,948,117
Total Annual Capital Costs, \$/year	10 years, 3.5 percent	\$176,947	\$176,947	\$51,810	\$51,810	\$1,130,731	\$507,344	\$422,980	\$275,292	\$172,916	\$35,629	\$35,629	\$170,618	\$196,429	\$122,894	\$88,380	\$53,866	\$1,248,031
Capital Costs		\$1,471,595	\$1,471,595	\$430,881	\$430,881	\$9,403,844	\$4,219,377	\$3,517,761	\$2,289,492	\$1,438,071	\$296,315	\$296,315	\$1,418,961	\$1,633,625	\$1,022,064	\$735,022	\$447,982	\$10,379,378
Total Annual Cost, \$/year		\$791,874	\$579,572	\$4,227,613	\$2,466,086	\$3,357,314	\$2,533,862	\$2,410,996	\$1,374,025	\$799,936	\$1,678,220	\$1,478,707	\$2,110,918	\$1,120,526	\$836,090	\$590,670	\$342,842	\$3,196,148
Average NH3-N Removal, lb/day		7	7	212	106	449	449	337	225	112	88	102	464	464	348	232	116	464
Average NH3-N Removal, %		1.5	1.5	44.8	22.4	95.0	95.0	71.3	47.5	23.8	18.6	21.5	98.0	98.0	73.5	49.0	24.5	98.0
Total, \$/lb NH3-N Removed		309.93	226.84	54.63	63.74	20.47	15.45	19.60	16.76	19.51	52.25	39.79	12.48	6.62	6.59	6.98	8.11	18.89

## **Attachment D: Reliability Comparison**

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**Comparison of Projected Removals and Reliability of Effluent NH<sub>3</sub>-N Removal Processes  
for the Emerald Performance Materials Wastewater Treatment Facility**

Process	Effluent NH <sub>3</sub> -N Removal		
	(Average %)	Reliability Rating <sup>1</sup>	Comments
PC Tank Stripping with Off-gas Control	30	8	Involves adding caustic, surface aerator, oversized withdrawal fan, off-gas collection, and thermal oxidation of off-gas. Acid addition in primary system will be required to lower pH to 9.0 s.u. Off-gas collection and treatment are needed for VOC control. Performance will vary as volatile amine content varies in wastewater. Average removals of 0 to 30 percent could be achieved by varying the size of the surface aerator placed in the tank. Will increase effluent TDS.
PVC Tank Stripping without Off-gas Control	74	7	Involves adding caustic and surface aerator to PVC tank contents. Acid addition in primary system will be required to lower pH to 9.0 s.u. Simple to operate. Strong foaming potential in PVC Tank, which would reduce effectiveness. Performance will vary based on production discharges of NH <sub>3</sub> -N and volatile amines and NH <sub>3</sub> -N returned in sludge dewatering filtrate and tertiary filter backwash. Removals of 0 to 74 percent could be achieved by varying the size of the surface aerator placed in the tank. Will increase effluent TDS.
Effluent Stripping with Off-gas Control	95	7	Involves pumping sand filter effluent through two packed towers in series. Caustic is added to increase pH to 11.5 s.u. and acid is added to lower the treated effluent pH to 8 s.u. Off-gas is directed to an acid scrubber for recovery of (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> . Scrubber discharge would be disposed off-site. Complex to operate. Equipment must be housed in heated building to prevent freezing. Fouling of tower media with precipitants is anticipated. Removals of 75 to 95 percent would be achieved by treating the whole effluent through different sized columns. Removals of 25 to 75 percent would be achieved by treating only a portion of the final effluent. Will increase effluent TDS.
Effluent Stripping without Off-gas Control	95	8	Same as above but without off-gas collection and treatment. NH <sub>3</sub> -N would be discharged to atmosphere. Will increase effluent TDS.
Struvite Precipitation	34	6	Involves feeding magnesium hydroxide and phosphoric acid to existing primary treatment system. Simple to operate; however, the precipitant is prone to foul pumps and piping. Removal could be varied between 22 and 34 percent depending upon the quantity of magnesium hydroxide added. Performance will

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			vary strictly as a function of influent NH <sub>3</sub> -N load. Will increase effluent TDS.
Effluent Breakpoint Chlorination	98	9	Involves routing secondary clarifier effluent through chlorination step prior to tertiary filtration. Caustic is fed to maintain pH control. Reliable process. Creates safety concerns and may form chlorinated organics. Very complex system requiring active monitoring and safety controls. Will increase effluent TDS.
Effluent Ion Exchange	98	6	Involves pumping sand filter effluent through two resin columns in series. Caustic is added to neutralize effluent from strong acid resin treatment. Resins would be regenerated daily using acid, and spent regenerant (high cation content NH <sub>4</sub> CL solution) would be disposed off-site. Complex to operate. Equipment must be housed in heated building to prevent freezing. Fouling of media with precipitants and biomass is anticipated. Removals of 25 to 75 percent would be achieved by treating only a portion of the whole effluent. Should have little net effect on effluent TDS.
Effluent Ozonation	98	8	Involves routing secondary clarifier effluent through ozonation step prior to tertiary filtration. Caustic is fed to maintain pH control. Very complex system requiring active monitoring and safety controls. Will increase effluent TDS.

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<sup>1</sup> Reliability Rating based on a relative assessment of mechanical and process performance reliability to achieve the average percent removal (10 being highest reliability). Reliability means the ability of the treatment process to achieve the predicted effluent ammonia-nitrogen (NH<sub>3</sub>-N) concentrations on a routine basis.



November 22, 2013

Emerald Performance Materials  
 Attn: Kellie Staab  
 1550 County Road 1450 N  
 Henry, IL 61537

RE: Biomonitoring Results  
 COA Number: 3110636 & 3110864

Dear Ms. Staab:

Enclosed are your biomonitoring results for Emerald Performance Materials. A summary of the findings is presented below.

<b>Test Concentrations</b>	3.125, 6.25, 12.5, 25 and 50%	
<b>Permit Limit:</b>	Acute Toxic Unit	
<b>Sample Collection Date/Time</b>	11/11/13 07:53 & 11/13/13 07:59	
<b>Outfall #</b>	Outfall 001	
<b>Test Organism</b>	<i>Ceriodaphnia dubia</i>	<i>Pimephales promelas</i>
<b>Test Type</b>	Acute 48-Hour Definitive	Acute 96-Hour Definitive
<b>LC<sub>50</sub> Result</b>	16.49%	16.79%

If you have any comments or questions concerning the enclosed report, please do not hesitate to contact me at 859-276-3506.

Sincerely,

Lisa Martin  
 Biology Section Supervisor

Enclosures  
 Emerald01

Microbac Laboratories, Inc  
 Louisville | Lexington | Paducah | Hazard | Evansville  
 3323 Gilmore Industrial Blvd. | Louisville, KY 40213 | 502.962.6400 p | 502.962.6411 f  
[www.microbac.com](http://www.microbac.com)

Test Type: Acute Definitive

**TOXICITY TEST REPORT SHEET**  
**COA NUMBERS: 3110636 & 3110864**

- 1) Facility/Discharger: Emerald Performance Materials Report Date: 11/22/13
- 2) Address: 1550 County Road 1450 N Henry, IL 61537
- 3) Permit #: IL0001392-1
- 4) Receiving Stream: Illinois River
- 5) Facility Contact: Kellie Staab Phone #: (309) 364-9411
- 6) Testing Lab Name: Microbac Laboratories, Inc. – Lexington Division
- 7) Lab Contact: Ms. Lisa Martin Phone #: (859) 276-3506
- 8) Outfall(s) Tested: Outfall 001
- 9) Average daily flow (MGD): 599.5
- 10) Test Species:     #1 *Ceriodaphnia dubia*                             #2 *Pimephales promelas*
- 11) Species Age:     #1 < 24 hours   #2 4 days
- 12) Organism Source: #1 In-house culture                             #2 In-house culture
- 13) Acclimation Procedure:   #1 N/A   #2 N/A
- 14) Test Conditions: (Static, Static-Renewal): Static, Static-Renewal
- 15) Dilution Water Type (synthetic, receiving stream): Receiving Water
- 16) Aeration? (Before/During Test): No
- 17) Dechlorination?: No                             Original Chlorine Level: 0.06 mg/L
- 18) Reference Toxicant Test Results:

Species	Date	Time	Duration	Toxicant	Results (LC <sub>50</sub> )
<i>Ceriodaphnia dubia</i>	11/04/13	11:30	48 hours	NaCl	2041 mg/L
<i>Pimephales promelas</i>	11/05/13	09:00	48 hours	NaCl	7549 mg/L

**TABLE 1  
ACUTE TOXICITY TEST SAMPLING DATA**

Sampling Summary for Acute Toxicity Tests			
Sampling Location & Description	Sample Collection		Weather/Receiving Stream Conditions
	Beginning MM/DD/Time	Ending MM/DD/Time	
Final Effluent: Outfall: Outfall 001 Type (Grab/Composite): Composite Volume Collected: 1 gallon	N/A	11/11/13 07:53	Temp. = NR
Upstream Station: Waterbody: Illinois River Station No.: N/A Type (Grab/Composite): Grab Volume Collected: 2 gallons	N/A	11/11/13 07:26	Receiving stream conditions: Flow: NR Height: NR Turbidity: NR
Additional Stations (If needed): Waterbody: N/A Type (Grab/Composite): N/A Volume Collected: N/A	N/A	N/A	N/A
Additional Stations (If needed): Waterbody: N/A Type (Grab/Composite): N/A Volume Collected: N/A	N/A	N/A	N/A
Waterbody: N/A Type (Grab/Composite): N/A Volume Collected: N/A	N/A	N/A	N/A

**TABLE 2**  
**TOXICITY TEST CONDITIONS**

Summary of Toxicity Test Conditions <i>Ceriodaphnia dubia</i> Acute 48-Hour Definitive Test Method: EPA 821-R-02-012	
1. Test Species and Age:	<i>Ceriodaphnia dubia</i> ; < 24 hours
2. Test Type and Duration:	Static; 48-Hour
3. Test Dates:	11/12/13 – 11/14/13
4. Test Temperature (°C):	25 ± 1
5. Light Quality:	Ambient laboratory illumination (cool white)
6. Photoperiod:	16 hours light, 8 hours dark
7. Feeding Regime:	Not fed
8. Size of Test Vessel:	30 mL
9. Volume and Depth of Test Solutions:	15 mL; 2.3 cm
10. No. of Test Organisms per Test Vessel:	5
11. No. of Test Vessels per Test Solution:	4
12. Total No. of Test Organisms per Test Solution:	20
13. Test concentrations (as percent by volume effluent):	3.125, 6.25, 12.5, 25 and 100%
14. Renewal of Test Solutions:	Not renewed
15. Dilution and Primary Control Water:	Receiving stream water
16. Secondary Control Water:	Reconstituted water
17. Aeration? Before/During Test:	None required
18. Endpoints Measured:	Death and immobility
19. If secondary control water was used as a diluent due to toxicity in primary control water, indicate number of consecutive tests conducted with alternative diluent:	N/A

**TABLE 3**  
**TOXICITY TEST CONDITIONS**

Summary of Toxicity Test Conditions <i>Pimephales promelas</i> Acute 96-Hour Definitive Test Method: EPA 821-R-02-012	
1. Test Species and Age:	<i>Pimephales promelas</i> ; 4 days
2. Test Type and Duration:	Static-Renewal; 96-Hour
3. Test Dates:	11/12/13 – 11/16/13
4. Test Temperature (°C):	25 ± 1
5. Light Quality:	Ambient laboratory illumination (cool white)
6. Photoperiod:	16 hours light, 8 hours dark
7. Feeding Regime:	fed at 48 hours
8. Size of Test Vessel:	270 mL
9. Volume and Depth of Test Solutions:	200 mL; 6 cm
10. No. of Test Organisms per Test Vessel:	10
11. No. of Test Vessels per Test Solution:	2
12. Total No. of Test Organisms per Test Solution:	20
13. Test concentrations (as percent by volume effluent):	3.125, 6.25, 12.5, 25 and 100%
14. Renewal of Test Solutions:	renewed at 48 hours
15. Dilution and Primary Control Water:	Receiving stream water
16. Secondary Control Water:	Reconstituted water
17. Aeration? Before/During Test:	None required
18. Endpoints Measured:	Death and immobility
19. If secondary control water was used as diluent due to toxicity in primary control water, indicate number of consecutive tests conducted with alternative diluent:	N/A

**TABLE 4  
ACUTE TOXICITY TEST RESULTS**

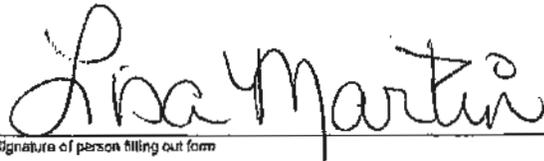
Results of a <i>Ceriodaphnia dubia</i> 48-Hour Static Acute Toxicity Test Conducted 11/12/13 – 11/14/13 Using Effluent from Outfall 001					
Test Solutions	Cumulative Percent Mortality (Cumulative Percent Affected) <sup>a</sup>		LC <sub>50</sub> Values		
	24-Hr	48-Hr	24-Hr	48-hr	
Primary Control/ Dilution Water	0 (0)	0 (0)	<25%	16.49%	
Secondary Control	0 (0)	0 (0)	LC <sub>50</sub> Confidence Limits		
3.125% Effluent	0 (0)	0 (0)	Limit	24-hr	48-hr
6.25% Effluent	5 (5)	5 (5)	LL UL	N/A N/A	14.99 18.15
12.5% Effluent	0 (0)	5 (5)			
25% Effluent	100 (100)	100 (100)	LL = Lower Limit UL = Upper Limit		
50% Effluent	100 (100)	100 (100)			
Near-Field Sample	N/A (N/A)	N/A (N/A)	Method(s) Used to Determine LC <sub>50</sub> , EC <sub>50</sub> and Confidence Limit Values: Trimmed Spearman- Karber		
<sup>a</sup> Cumulative percent affected is the total percentage of test organisms observed dead, immobile, exhibiting loss of equilibrium or other defined endpoints (specify below) <hr/>					

**TABLE 5**  
**ACUTE TOXICITY TEST RESULTS**

Results of a <i>Pimephales promelas</i> 96-Hour Static Acute Toxicity Test Conducted 11/12/13 – 11/16/13 Using Effluent from Outfall 001									
Test Solutions	Cumulative Percent Mortality (Cumulative Percent Affected) <sup>a</sup>				LC <sub>50</sub> Values				
	24-Hr	48-Hr	72-Hr	96-Hr	24-Hr	48-hr	72-Hr	96-Hr	
Primary Control/ Dilution Water	0 (0)	0 (0)	5 (5)	5 (5)	>100%	<25%	<25%	16.79%	
Secondary Control	0 (0)	0 (0)	0 (0)	0 (0)	LC <sub>50</sub> Confidence Limits (EC <sub>50</sub> Confidence Limits)				
3.125% Effluent	0 (0)	5 (5)	10 (10)	15 (15)	Limit	24-hr	48-Hr	72-Hr	96-Hr
6.25% Effluent	0 (0)	0 (0)	0 (0)	10 (10)	LL UL	N/A N/A	N/A N/A	N/A N/A	N/A N/A
12.5% Effluent	0 (0)	5 (5)	10 (10)	15 (15)					
25% Effluent	55 (55)	100 (100)	100 (100)	100 (100)	LL = Lower Limit UL = Upper Limit				
50% Effluent	100 (100)	100 (100)	100 (100)	100 (100)					
Near-Field Sample	N/A (N/A)	N/A (N/A)	N/A (N/A)	N/A (N/A)	Method(s) Used to Determine LC <sub>50</sub> , EC <sub>50</sub> and Confidence Limit Values: : Trimmed Spearman- Kärber				
<sup>a</sup> Cumulative percent affected is the total percentage of test organisms observed dead, immobile, exhibiting loss of equilibrium or other defined endpoints (specify below)									

**ADDITIONAL TOXICITY TEST INFORMATION**

- 1) Submit copies of all bench sheets and statistical calculations/printouts obtained during the test(s). Data must be presented in tabular form and must include all physical and/or chemical measurements recorded during the test (e.g. temperature, conductivity, total residual chlorine, dissolved oxygen, etc.). **See appendix.**
  
- 2) Methods/Instrumentation used in chemical analysis:  
  
Dissolved Oxygen: SM 4500 O-G Using a Extech Model 407510  
  
pH: SM 4500 H+ Using a Fisher Accumet AB15.  
  
Conductivity: SM 4500 2510 B Using a Fisher Accumet AB30.  
  
Alkalinity: Titrimetric Method SM 2320A  
  
Hardness: SM 2340B Using an ICP Analyzer  
  
Chlorine: Method SM 4500 CL-G  
  
EPA Acute Manual Edition and Date: EPA 821-R-02-012 5<sup>th</sup> Edition October 2002
  
- 3) Indicate below any other relevant information that may aid in the evaluation of this report. Include any deviations from EPA methodology that were necessary for these tests as well as any sample manipulations which were performed, such as aeration, dechlorination with sodium thiosulfate, etc. and the justification for such manipulations or deviations. Attach additional pages as needed. **None performed**



Signature of person filling out form

11-22-13

Date

Lisa Martin

Name (typed or printed)

Section Supervisor

**APPENDIX**

**CHEMICAL ANALYSIS REPORT  
ACUTE TOXICITY BENCH SHEETS  
CHAIN OF CUSTODY RECORD  
REFERENCE TOXICANT CONTROL CHART**



# Microbac Laboratories, Inc.

KENTUCKY TESTING LABORATORY DIVISION  
 3323 Gilmore Industrial Blvd. Louisville, KY 40213 502.962.6400 Fax: 502.962.6411  
 Evansville 812.464.9000 | Lexington 859.276.3506 | Paducah 270.898.3637 | Hazard 606.487.0511

Member



**Chemical, Biological, Physical, Molecular, and Toxicological Services**

**CERTIFICATE OF ANALYSIS**

**3110636**

**Emerald Performance Materials**  
**Brenda Abke**  
 1550 County Road 1450 North  
 Henry IL, 61537

**Date Reported** 11/20/2013  
**Date Due** 11/21/2013  
**Date Received** 11/12/2013  
**Customer #** E6920  
**Customer P.O.** HE-40047185-UB

**WET Testing**

Analysis	QOC	Qualifier	Result	Units	Min	Max	Method	Rpt Limit	Date	Time	Tech
<b>Sample: 01 Effluent - Composite</b>											
Sampled By Customer										Sampled	11/11/2013 @ 7:53
Alkalinity, Total as CaCO3			190	mg/L			SM 2320B	5.0	11/18/2013	18:05	JLC
Chlorine, Total Residual		H1	0.060	mg/L			SM 4500 Cl G	0.020	11/12/2013	17:07	KEM
Nitrogen, Ammonia			24	mg/L			SM 4500 NH3 G	2.5	11/19/2013	15:11	JLC
Toxicity, Acute - C. dubia			See	TU			EPA 821-R-02-012		11/12/2013	10:35	KEM
			Attached								
Toxicity, Acute - P. promelas			See	TU			EPA 821-R-02-012		11/12/2013	10:53	DZW
			Attached								
<u>Hardness Pkg. By ICP</u>											
Calcium			56	mg/L			EPA 200.7	0.50	11/14/2013	12:38	FML
Magnesium			24	mg/L			EPA 200.7	0.20	11/14/2013	12:38	EML
Hardness, Total as CaCO3			240	mg/L			SM 2340B	1.2	11/14/2013	12:38	EML
<b>Sample: 02 River Water</b>											
Sampled By Customer										Sampled	11/11/2013 @ 7:26
Alkalinity, Total as CaCO3			160	mg/L			SM 2320B	5.0	11/18/2013	18:05	JLC
Chlorine, Total Residual		H1	0.050	mg/L			SM 4500 Cl G	0.020	11/12/2013	17:02	KEM
Nitrogen, Ammonia			<0.25	mg/L			SM 4500 NH3 G	0.25	11/18/2013	19:16	JLC
<u>Hardness Pkg. By ICP</u>											
Calcium			59	mg/L			EPA 200.7	0.50	11/14/2013	13:14	EML
Magnesium			24	mg/L			EPA 200.7	0.20	11/14/2013	13:14	EML
Hardness, Total as CaCO3			250	mg/L			SM 2340B	1.2	11/14/2013	13:14	EML

**Qualifier Definitions**

H1 Sample received outside of holding time for these analytes.

The following analyses were not run at the main Louisville lab within the Microbac Kentucky Division, but at a satellite location.

Laboratory

Microbac Laboratories, Kentucky Testing Laboratory, Lexington Site

Analysis

Chlorine, Total Residual  
 Toxicity, Acute - P. promelas  
 Toxicity, Acute - C. dubia

Method

SM 4500 Cl G  
 EPA 821-R-02-012  
 EPA 821-R-02-012



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**Chemical, Biological, Physical, Molecular, and Toxicological Services**

## CERTIFICATE OF ANALYSIS

**3110636**

**Emerald Performance Materials  
Brenda Abke**

**Date Reported** 11/20/2013  
**Date Received** 11/12/2013  
**Date Sampled** 11/11/2013

**WET Testing**

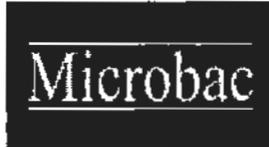
*THIS REPORT HAS BEEN REVIEWED AND APPROVED FOR RELEASE:*

LISA MARTIN, A.M.

DIVISION MANAGER, KENTUCKY DIVISION

*As regulatory limits change frequently, Microbac advises the recipient of this report to confirm such limits with the appropriate Federal, state, or local authorities before acting in reliance on the regulatory limits provided.*

*For any feedback concerning our services, please contact Michael Flournoy, the Division Manager at 502.962.6400. You may also contact Sean Hyde, Chief Operating Officer at [sean.hyde@microbac.com](mailto:sean.hyde@microbac.com) or James Nokes, President at [james.nokes@microbac.com](mailto:james.nokes@microbac.com)*



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Member



**Chemical, Biological, Physical, Molecular, and Toxicological Services**

**CERTIFICATE OF ANALYSIS**

**3110864**

**Emerald Performance Materials**  
**Brenda Abke**  
 1550 County Road 1450 North  
 Henry IL, 61537

Date Reported 11/21/2013  
 Date Due 11/25/2013  
 Date Received 11/14/2013  
 Customer # E6920  
 Customer P.O. HE-40047185-UB

**WET Testing**

Analysis	OOC	Qualifier	Result Units	Min	Max	Method	Rpt Limit	Date	Time	Tech
<b>Sample: 01 Effluent - Composite</b>										
								Sampled	11/13/2013 @	7:59
Sampled By Customer										
Alkalinity, Total as CaCO3			210 mg/L			SM 2320B	5.0	11/20/2013	17:00	ATM
Chlorine, Total Residual		HI	0.070 mg/L			SM 4500 Cl G	0.020	11/14/2013	12:33	DZW
Nitrogen, Ammonia			25 mg/L			SM 4500 NH3 G	2.5	11/20/2013	17:24	JLC
<u>Hardness Pkg. By ICP</u>										
Calcium			58 mg/L			EPA 200.7	0.50	11/18/2013	11:32	MSR
Magnesium			24 mg/L			EPA 200.7	0.20	11/18/2013	11:32	MSR
Hardness, Total as CaCO3			240 mg/L			SM 2340B	1.2	11/18/2013	11:32	MSR
<b>Sample: 02 River Water</b>										
								Sampled	11/13/2013 @	7:28
Sampled By Customer										
Alkalinity, Total as CaCO3			170 mg/L			SM 2320B	5.0	11/20/2013	17:00	ATM
Chlorine, Total Residual		HI	0.040 mg/L			SM 4500 Cl G	0.020	11/14/2013	12:38	DZW
Nitrogen, Ammonia			<0.25 mg/L			SM 4500 NH3 G	0.25	11/20/2013	13:59	JLC
<u>Hardness Pkg. By ICP</u>										
Calcium			64 mg/L			EPA 200.7	0.50	11/18/2013	11:37	MSR
Magnesium			25 mg/L			EPA 200.7	0.20	11/18/2013	11:37	MSR
Hardness, Total as CaCO3			260 mg/L			SM 2340B	1.2	11/18/2013	11:37	MSR

**Qualifier Definitions**

HI Sample received outside of holding time for these analytes.

The following analyses were not run at the main Louisville lab within the Microbac Kentucky Division, but at a satellite location.

Laboratory	Analysis	Method
Microbac Laboratories, Kentucky Testing Laboratory, Lexington Site	Chlorine, Total Residual	SM 4500 Cl G



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Member



**Chemical, Biological, Physical, Molecular, and Toxicological Services**

## CERTIFICATE OF ANALYSIS

**3110864**

**Emerald Performance Materials  
Brenda Abke**

**Date Reported** 11/21/2013  
**Date Received** 11/14/2013  
**Date Sampled** 11/13/2013

**WET Testing**

*THIS REPORT HAS BEEN REVIEWED AND APPROVED FOR RELEASE:*

LISA MARTIN, A.M.

DIVISION MANAGER, KENTUCKY DIVISION

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Microbac Laboratories, Inc. KTL Division Lexington

### Freshwater Acute Ceriodaphnia dubia Toxicity Benchsheet

48-Hour Static Non-Renewal

EPA 821-R-02-012 Method 2002.0 (Non-Potable Water) SOP Revision 130721

Discharger	Emerald Performance	Dilution Water Used	Mod. Hard Synthetic Fresh
Location	Outfall 001	Dilution Water Batch Number	E30228 + River
Sample Number	3110636-01	Source Culture	Mass 1106
Test Initiated Date/Time/Analyst	11/12/13 10:35 DZW	Organism Age	<24 hrs
Test Terminated Date/Time/Analyst	11/14/13/ 9:55 KEM	Date/time Sample Collected	11/11/2013 7:53

Sample (% Eff.)	Replicate ID	Live Organism at Hour			pH (Standard Units)			Dissolved Oxygen (mg/L)			Conductivity (umhos/cm)			Temperature (Deg. C)		
		0	24	48	0	24	48	0	24	48	0	24	48	0	24	48
control	1	5	5	5	7.4		7.4	8.3		9.2	294		314	24.8	26.0	25.4
	2	5	5	5												
	3	5	5	5												
	4	5	5	5												
River	5	5	5	5	7.5		8.1	8.3		9.5	774		730	25.8	26.0	25.4
	6	5	5	5												
	7	5	5	5												
	8	5	5	5												
3.125	9	5	5	5	7.6		8.3	8.7		9.6	1281		1180	25.8	26.0	25.6
	10	5	5	5												
	11	5	5	5												
	12	5	5	5												
6.25	13	5	5	5	7.7		8.3	8.8		9.5	1733		1591	25.8	25.8	25.6
	14	5	4	4												
	15	5	5	5												
	16	5	5	5												
12.5	17	5	5	4	7.7		8.4	8.8		9.6	2703		2501	25.8	25.8	25.4
	18	5	5	5												
	19	5	5	5												
	20	5	5	5												
25	21	5	0	0	7.5		8.4	8.6		9.6	4452		4197	25.8	25.8	25.4
	22	5	0	0												
	23	5	0	0												
	24	5	0	0												
Analyst		DZW	KEM	KEM	DZW		KEM	DZW		KEM	DZW		KEM	DZW	KEM	KEM

Sample Water TRC (mg/L)	Control H <sub>2</sub> O Alkalinity (mg/L)	Control H <sub>2</sub> O Hardness (mg/L)	Thermo-meter	pH Meter	DO Meter	Conductivity Meter	Randomization Template ID:
0.06	57	86	Laser 2	pH-3	DO-5	COND-1	

Comments:

Microbac Laboratories, Inc. KTL Division Lexington

### Freshwater Acute Ceriodaphnia dubia Toxicity Benchsheet

48-Hour Static Non-Renewal

EPA 821-R-02-012 Method 2002.0 (Non-Potable Water) SOP Revision 130721

Discharger	Emerald Performance	Dilution Water Used	Mod. Hard Synthetic Fresh
Location	Outfall 001	Dilution Water Batch Number	E30228 + River
Sample Number	3110636-01	Source Culture	Mass 1106
Test Initiated Date/Time/Analyst	11/12/13 10:35 DZW	Organism Age	<24 hrs
Test Terminated Date/Time/Analyst	11/14/13 9:55 KEM	Date/time Sample Collected	11/11/2013 7:53

Sample (% Eff.)	Replicate ID	Live Organism at Hour			pH (Standard Units)			Dissolved Oxygen (mg.L)			Conductivity (umhos/cm)			Temperature (Deg. C)		
		0	24	48	0	24	48	0	24	48	0	24	48	0	24	48
50	1	5	0	0	7.4		8.3	8.6		8.4	7875		7527	25.8	25.6	25.4
	2	5	0	0												
	3	5	0	0												
	4	5	0	0												
	5															
	6															
	7															
	8															
	9															
	10															
	11															
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	14															
	15															
	16															
	17															
	18															
	19															
	20															
	21															
	22															
	23															
	24															
Analyst		DZW	KEM	KEM	DZW		KEM	DZW		KEM	DZW		KEM	DZW	KEM	KEM

Sample Water TRC (mg/L)	Control H <sub>2</sub> O Alkalinity (mg/L)	Control H <sub>2</sub> O Hardness (mg/L)	Thermo-meter	pH Meter	DO Meter	Conductivity Meter	Randomization Template ID:
0.06	57	86	Laser 2	pH-3	DO-5	COND-1	

Comments:

Acute Daphnid Test-48 Hour Survival

Start Date: 11/12/2013 10:35 Test ID: 3110536-C Sample ID: Emerald Performance  
 End Date: Lab ID: KTL-Microbac Laboratories Sample Type: EFF2-Industrial  
 Sample Date: Protocol: EPA 821-R-02-012 Test Species: CD-Ceriodaphnia dubia  
 Comments:

Conc-%	1	2	3	4
River Control	1.0000	1.0000	1.0000	1.0000
3.125	1.0000	1.0000	1.0000	1.0000
6.25	1.0000	0.8000	1.0000	1.0000
12.5	0.8000	1.0000	1.0000	1.0000
25	0.0000	0.0000	0.0000	0.0000
50	0.0000	0.0000	0.0000	0.0000

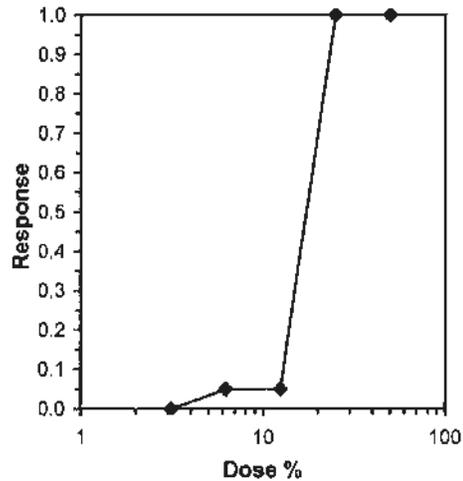
Conc-%	Transform: Arcsin Square Root							Rank Sum	1-Tailed Critical	Number Resp	Total Number
	Mean	N-Mean	Mean	Min	Max	CV%	N				
River Control	1.0000	1.0000	1.3453	1.3453	1.3453	0.000	4			0	20
3.125	1.0000	1.0000	1.3453	1.3453	1.3453	0.000	4	18.00	10.00	0	20
6.25	0.9500	0.9500	1.2857	1.1071	1.3453	9.261	4	16.00	10.00	1	20
12.5	0.9500	0.9500	1.2857	1.1071	1.3453	9.261	4	16.00	10.00	1	20
25	0.0000	0.0000	0.2255	0.2255	0.2255	0.000	4			20	20
50	0.0000	0.0000	0.2255	0.2255	0.2255	0.000	4			20	20

Auxiliary Tests	Statistic	Critical	Skew	Kurt
Shapiro-Wilk's Test indicates non-normal distribution (p <= 0.01)	0.67657	0.844	-1.807	2.82967
Equality of variance cannot be confirmed				
Hypothesis Test (1-tail, 0.05)	NOEC	LOEC	CHV	TU
Steel's Many-One Rank Test	12.5	25	17.6777	8

Trimmed Spearman-Kärber

Trim Level	EC50	95% CL	
0.0%	16.494	14.991	18.147
5.0%	17.358	16.720	18.020
10.0%	17.358	16.720	18.020
20.0%	17.358	16.720	18.020
Auto-0.0%	16.494	14.991	18.147

LC50 = 16.494  
49%



Microbac Laboratories, Inc. KTL Division Lexington

**Freshwater Acute Pimephales promelas Renewal Toxicity Benchsheet**

EPA 821-R-02-012 Method 2000.0 (Non-Potable Water) SOP Revision 130721

Discharger	Emerald Performance			Dilution Water Used	Mod. Hard Synthetic Fresh
Location	Outfall 001			Dilution Water Batch No.	E30228-29 + River
Sample Number	3110636-01 3110864-01			Source Culture	1108
Test Initiated Date/Time/Analyst		11/12/13 10:53 DZW		Organism Age	4 days
Test Terminated Date/Time/Analyst		11/16/13 10:53 DZW		Date/Time Sample Collected	11/11/2013 7:53
				Renewal Time	11/14/13 10:30 DZW

Eff. Conc. (%)	Replicate ID	Number of Live Organisms at Hour				pH (Standard Units)				Dissolved Oxygen (mg/L)				Conductivity (umhos/cm)				
		24	48	72	96	I	P	R	T	I	P	R	T	I	P	R	T	
control	1	10	10	10	10	7.4	7.3	7.7	7.8	8.3	8.0	8.1	6.8	294	301	295	319	
	2	10	10	10	10													
river	3	10	10	10	10	7.5	8.0	7.7	8.3	8.3	8.0	9.7	6.8	774	733	773	809	
	4	10	10	9	9													
3.125	5	10	10	10	9	7.6	8.2	7.8	8.3	8.7	7.8	9.7	6.8	1281	1209	1288	1366	
	6	10	9	8	8													
6.25	7	10	10	10	9	7.7	8.2	7.8	8.3	8.8	7.6	9.7	7.0	1733	1665	1720	1826	
	8	10	10	10	9													
12.5	9	10	9	9	9	7.7	8.2	7.7	8.3	8.8	7.6	9.6	6.9	2703	2638	2613	2826	
	10	10	10	9	8													
25	11	6	0	-	-	7.5	8.2	-		8.6	7.6	-		4452	4286	-		
	12	5	0	-	-													
50	13	0	0	-	-	7.4	8.2	-		8.6	7.6	-		7875	7611	-		
	14	0	0	-	-													
Analyst		KEM	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW	DZW

Sample Water TRC (mg/L)	Control Water Alkalinity (mg/L)	Control Water Hardness (mg/L)	DO Meter ID	pH Meter ID	Chlorine Meter ID	Conductivity Meter ID	Thermometer ID
0.06 \ 0.07	57	86	DO-5	PH-3	NA	COND-1	Laser 2
						Approved By	LLM

Comments:

Microbac Laboratories, Inc. KTL Division Lexington

### Renewal Toxicity Test Temperature Record

96 Hour Acute Pimephales promelas EPA 821-R-02-012 Method 2000.0 (Non-Potabl Water) SOP Rev. 130721

Discharger	Emerald Performance	Dilution Water Used	Mod. Hard Synthetic Fresh
Location	Outfall 001	Dilution Water Batch Number	E30228 + River
Sample Number	3110636-01 / 3110864-01	Source Culture	1108
Test Initated Date/Time/Analyst	11/12/13 10:53 DZW	Organism Age	4 days
Test Terminated Date/Time/Analyst	11/16/13 10:53 DZW	Date/Time Sample Collected	11/11/2013 7:53

Test Concentration	Temperature (°C)					
	0 Hours	24 Hours	48 Hours Pre	48 Hours Post	72 Hours	96 Hours
control	24.8	25.2	25.8	24.4	24.4	24.6
river	25.8	25.2	24.4	25.2	24.8	24.6
3.125	25.8	25.4	24.8	24.8	25.0	24.8
6.25	25.8	25.4	24.6	24.6	25.2	24.8
12.5	25.8	25.4	24.6	24.4	25.0	24.8
25	25.8	25.2	-	25.0	-	-
50	25.8	25.4	-	25.2	-	-
<b>Analyst</b>	DZW	KEM	DZW	DZW	DZW	DZW
<b>Thermometer ID</b>	Laser 2					

Comments:

**Acute Fish Test-96 Hour Survival**

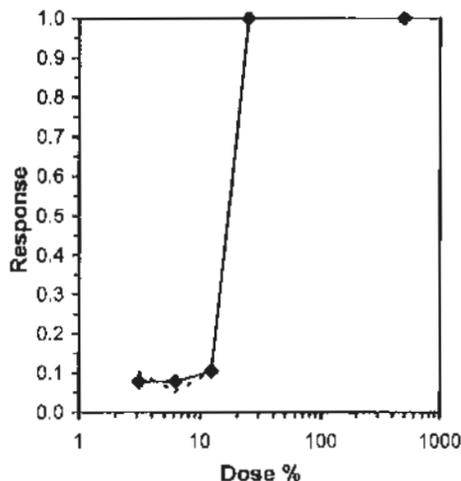
Start Date: 11/12/2013 10:53 Test ID: 3110636-F Sample ID: Emerald Performance  
 End Date: 11/16/2013 10:53 Lab ID: KTL-Microbac Laboratories Sample Type: EFF2-Industrial  
 Sample Date: Protocol: EPA 821-R-02-012 Test Species: PP-Pimephales promelas  
 Comments:

Conc-%	1	2
River Control	1.0000	0.9000
3.125	0.9000	0.8000
6.25	0.9000	0.9000
12.5	0.9000	0.8000
25	0.0000	0.0000
500	0.0000	0.0000

Conc-%	Mean	N-Mean	Transform: Arcsin Square Root					N	Number Resp	Total Number
			Mean	Min	Max	CV%	N			
River Control	0.9500	1.0000	1.3305	1.2490	1.4120	8.661	2	1	20	
3.125	0.8500	0.8947	1.1781	1.1071	1.2490	8.517	2	3	20	
6.25	0.9000	0.9474	1.2490	1.2490	1.2490	0.000	2	2	20	
12.5	0.8500	0.8947	1.1781	1.1071	1.2490	8.517	2	3	20	
25	0.0000	0.0000	0.1588	0.1588	0.1588	0.000	2	20	20	
500	0.0000	0.0000	0.1588	0.1588	0.1588	0.000	2	20	20	

Auxiliary Tests	Statistic	Critical	Skew	Kurt
Normality of the data set cannot be confirmed				
Equality of variance cannot be confirmed				

Trim Level	EC50	95% CL	
0.0%			
5.0%			
10.0%	16.964	14.510	19.833
20.0%	16.971	15.992	18.010
Auto-7.9%	16.794	14.850	18.992



LC50 = 16.79%

BIOMONITORING CHAIN OF CUSTODY



2520 Regency Road  
Lexington, Kentucky 40503-2921  
(859) 276-3506

3110636 LISA MARTIN

CA No.:  
Client: Emerald Performance  
materials

Collected by:  
Outfall/Station: 001

SAMPLE TYPE:

Grab	Collection		Temp (°C) upon Pick up / shipping				Temp (°C) upon receipt at Lab			Volume Collected	Visual Description
	Date	Time	On site	Rec. by	Client Init.	Time	Laboratory	Rec. by	Time		
<u>River water</u>											
1	<u>11/11/13</u>	<u>7:24</u>					<u>6.7</u>	<u>LM</u>	<u>0835</u>		<u>clear</u>
2	<u>11/11/13</u>	<u>7:26</u>					<u>6.7</u>	<u>LM</u>	<u>0835</u>		<u>clear</u>
3											
4											

Composite	From		To		Temp (°C) upon Pick up / shipping				Temp (°C) upon receipt at Lab:			Volume Collected	Visual Description
	Date	Time	Date	Time	On-site	Rec. by	Client Init.	Time	Laboratory	Rec. by	Time		
<u>Plant Effluent</u>													
1	<u>11/10/13</u>	<u>20:00</u>	<u>11/11/13</u>	<u>6:53</u>					<u>6.7</u>	<u>LM</u>	<u>0835</u>		<u>orangish</u>
2													

First Day Rain Event: Yes Amount (in.) No Trace Daily Flow (MGD) 599.5  
 Second Day Rain Event: Yes Amount (in.) No Trace Daily Flow (MGD)

COMMENTS: Kelly Stead pulled River water samples from Hennepin Landing.

SAMPLE RECEIVING (Fill in from top down):  
 Relinquished by: [Signature] 11/11/13 09:16  
 Signature: \_\_\_\_\_ Date/Time: \_\_\_\_\_  
 Received by: [Signature] 11/11/13 09:10  
 Signature: \_\_\_\_\_ Date/Time: \_\_\_\_\_  
UPS 11-12-13 0830 [Signature] 11-12-13 0830  
 Signature: \_\_\_\_\_ Date/Time: \_\_\_\_\_  
 Signature: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Sample Deliver: UPS ( ) FED EX ( )  
 DHL ( ) OTHER ( )

TEST TYPE: Acute Cerio (X) Chronic Cerio ( )  
 Acute Fish (X) Chronic Fish ( ) Acute Magna ( )

BIOMONITORING CHAIN OF CUSTODY



2520 Regency Road  
Lexington, Kentucky 40503-2921  
(859) 276-3506

L3

3110864 LISA MARTIN

LA No.:  
Site: Emerald Performance MHD

Collected by: Dave Westings, Vic Carroll  
Outfall/Station: 001 + Kellie Staab

SAMPLE TYPE:

Grab	Collection		Temp (°C) upon Pick up / shipping				Temp (°C) upon receipt at Lab			Volume Collected	Visual Description
	Date	Time	On site	Rec. by	Client Init.	Time	Laboratory	Rec. by	Time		
1	11/13/14	7:26a					6.5	UM	0825		gray
2	11/13/14	7:28a					6.5	UM	0825		gray
3											
4											

Composite	From		To		Temp (°C) upon Pick up / shipping				Temp (°C) upon receipt at Lab			Volume Collected	Visual Description
	Date	Time	Date	Time	On site	Rec. by	Client Init.	Time	Laboratory	Rec. by	Time		
1	11/12	20:04	11/13	7:59a					6.5	UM	0825		orange
2													

First Day Rain Event: Yes Amount (in.)       No Trace Daily Flow (MGD)     

Second Day Rain Event: Yes Amount (in.)       No Trace Daily Flow (MGD)     

**SAMPLE RECEIVING (Fill in from top down):**

Relinquished by: <u>[Signature]</u>	Date/Time: <u>11-13-13</u>	Received by: <u>Kellie Staab</u>	Date/Time: <u>11/13/13 0840</u>
Signature	Date/Time	Signature	Date/Time
<u>ups</u>	<u>11-14-13 0820</u>	<u>[Signature]</u>	<u>08:20 11-14-13</u>
Signature	Date/Time	Signature	Date/Time
Signature	Date/Time	Signature	Date/Time

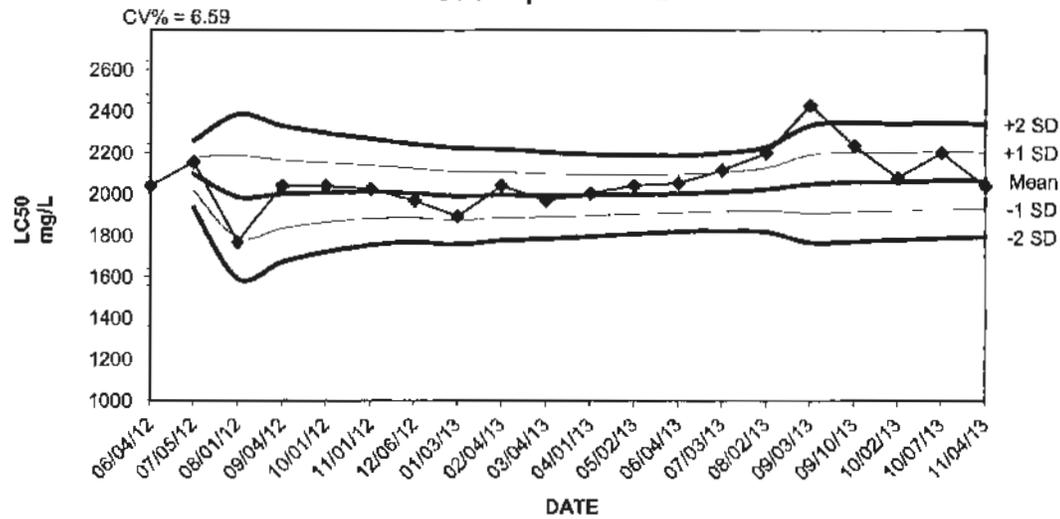
  

**COMMENTS:** Kellie Staab pulled river water samples from Hennepin landing

**Sample Deliver:** UPS ( ) FED EX ( )  
DHL ( ) OTHER ( )

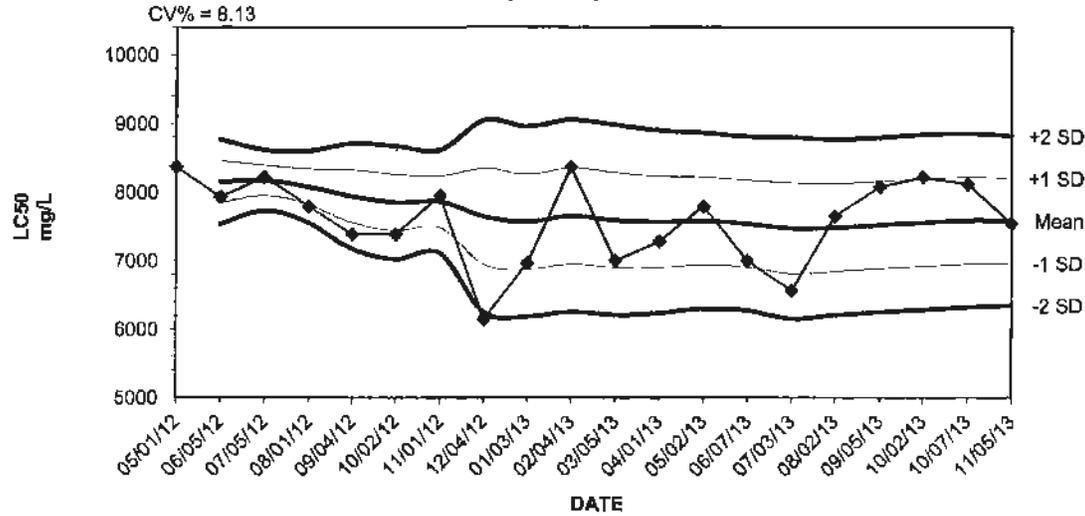
**TEST TYPE:** Acute Cerio (  ) Chronic Cerio ( )  
Acute Fish (  ) Chronic Fish ( ) Acute Magna ( )

**ACUTE REFERENCE TOXICANT CONTROL CHART**  
*Ceriodaphnia dubia*



Dates	Values	Mean	-1 SD	-2 SD	+1 SD	+2 SD
06/04/12	2040.0000					
07/05/12	2154.0000	2097.0000	2016.3898	1935.7797	2177.6102	2258.2203
08/01/12	1767.0000	1987.0000	1788.1307	1589.2614	2185.8693	2384.7386
09/04/12	2041.0000	2000.5000	1835.8944	1671.2888	2165.1056	2329.7112
10/01/12	2040.0000	2008.4000	1864.7570	1721.1141	2152.0430	2295.6859
11/01/12	2026.0000	2011.3333	1882.6544	1753.9755	2140.0123	2268.6912
12/06/12	1969.0000	2005.2857	1886.7337	1768.1818	2123.8377	2242.3897
01/03/13	1893.0000	1991.2500	1874.5333	1757.8166	2107.9667	2224.6834
02/04/13	2041.0000	1996.7778	1886.3471	1775.9163	2107.2085	2217.6392
03/04/13	1969.0000	1994.0000	1889.5150	1785.0300	2098.4850	2202.9700
04/01/13	2004.0000	1994.9091	1895.7401	1796.5711	2094.0781	2193.2471
05/02/13	2042.0000	1998.8333	1903.3072	1807.7811	2094.3595	2189.8856
06/04/13	2052.0000	2002.9231	1910.2827	1817.6423	2095.5635	2188.2039
07/03/13	2116.0000	2011.0000	1917.0033	1823.0065	2104.9967	2198.9935
08/02/13	2198.0000	2023.4667	1920.8238	1818.1810	2126.1095	2228.7523
09/03/13	2427.0000	2048.6875	1907.2286	1765.7696	2190.1464	2331.6054
09/10/13	2235.0000	2059.6471	1915.4185	1771.1900	2203.8756	2348.1041
10/02/13	2079.0000	2060.7222	1920.7257	1780.7291	2200.7188	2340.7153
10/07/13	2201.0000	2068.1053	1928.2987	1786.4921	2207.9118	2347.7184
11/04/13	2041.0000	2066.7500	1930.5374	1794.3247	2202.9628	2339.1753

**ACUTE REFERENCE TOXICANT CONTROL CHART**  
*Pimephales promelas*



Dates	Values	Mean	-1 SD	-2 SD	+1 SD	+2 SD
05/01/12	8367.0000	8148.5000	7839.4943	7530.4887	8457.5057	8766.5113
06/05/12	7930.0000	8172.0000	7949.7411	7727.4823	8394.2589	8616.5177
07/05/12	8219.0000	8076.7500	7813.6475	7550.5450	8339.8525	8602.9550
08/01/12	7791.0000	7938.4000	7554.1855	7169.9710	8322.6145	8706.8290
09/04/12	7385.0000	7846.0000	7434.5110	7023.0220	8257.4890	8668.9780
10/02/12	7384.0000	7860.2857	7482.7526	7105.2195	8237.8188	8815.3519
11/01/12	7946.0000	7646.0000	6946.3454	6246.6908	8345.6546	9045.3092
12/04/12	6146.0000	7570.8667	6878.2773	6185.8880	8263.0560	8955.4453
01/03/13	8968.0000	7650.3000	6950.6210	6250.9420	8349.9790	9049.6580
02/04/13	8367.0000	7591.1818	6899.0545	6206.9273	8283.3091	8975.4364
03/05/13	7000.0000	7565.3333	6899.3688	6233.4042	8231.2979	8897.2624
04/01/13	7281.0000	7582.6154	6941.9654	6301.3154	8223.2654	8863.9154
05/02/13	7790.0000	7541.0000	6906.0933	6271.1867	8175.9067	8810.8133
06/07/13	7000.0000	7476.0000	6814.4200	6152.8399	8137.5800	8799.1601
07/03/13	6566.0000	7487.0000	6846.3402	6205.6805	8127.6598	8768.3195
08/02/13	7652.0000	7521.4706	6885.0809	6248.6911	8157.8603	8794.2501
09/05/13	8073.0000	7560.2222	6921.3175	6282.4128	8199.1269	8838.0317
10/02/13	8219.0000	7589.5263	6955.6199	6321.7134	8223.4328	8857.3392
10/07/13	8117.0000	7587.5000	6970.4342	6353.3685	8204.5658	8821.6315
11/05/13	7549.0000					