

BEFORE THE ILLINOIS POLLUTION CONTROL BOARD

SANITARY DISTRICT OF)	
DECATUR,)	
)	
Petitioner,)	
)	
v.)	PCB 14- 111
)	(Variance – Water)
ILLINOIS ENVIRONMENTAL)	
PROTECTION AGENCY,)	
)	
Respondent.)	

NOTICE OF FILING

TO: Mr. John T. Therriault	Carol Webb, Esq.
Assistant Clerk of the Board	Hearing Officer
Illinois Pollution Control Board	Illinois Pollution Control Board
100 W. Randolph Street	1021 North Grand Avenue East
Suite 11-500	Post Office Box 19274
Chicago, Illinois 60601	Springfield, Illinois 62794-9274
(VIA ELECTRONIC MAIL)	

PLEASE TAKE NOTICE that I have today filed with the Office of the Clerk of the Illinois Pollution Control Board the **PETITIONER'S RESPONSES TO THE ILLINOIS POLLUTION CONTROL BOARD'S QUESTIONS PURSUANT TO THE MARCH 18, 2014 HEARING OFFICER ORDER** copies of which are herewith served upon you.

Respectfully submitted,

SANITARY DISTRICT OF DECATUR,

Dated: April 21, 2014

By: /s/Katherine D. Hodge
Katherine D. Hodge

Katherine D. Hodge
Ethan S. Pressly
HODGE DWYER & DRIVER
3150 Roland Avenue
Post Office Box 5776
Springfield, Illinois 62705-5776
(217) 523-4900

CERTIFICATE OF SERVICE

I, Katherine D. Hodge, the undersigned, hereby certify that I have served the attached PETITIONER'S RESPONSES TO THE ILLINOIS POLLUTION CONTROL BOARD'S QUESTIONS PURSUANT TO THE MARCH 18, 2014 HEARING

OFFICER ORDER, upon:

Mr. John T. Therriault
Assistant Clerk of the Board
Illinois Pollution Control Board
100 West Randolph Street, Suite 11-500
Chicago, Illinois 60601

via electronic mail on April 21, 2014; and upon:

Sara Terranova, Esq.
Division of Legal Counsel
Illinois Environmental Protection Agency
1021 North Grand Avenue East
Post Office Box 19276
Springfield, Illinois 62794-9276

Carol Webb, Esq.
Hearing Officer
Illinois Pollution Control Board
1021 North Grand Avenue East
Post Office Box 19274
Springfield, Illinois 62794-9274

Division Chief of Environmental Enforcement
Office of the Attorney General
69 West Washington Street
Chicago, Illinois 60602

Office of Legal Services
IL Department of Natural Resources
One Natural Resources Way
Springfield, IL 62702-1271

depositing said documents in the United States Mail, postage prepaid, in Springfield, Illinois, on April 21, 2014.

/s/Katherine D. Hodge
Katherine D. Hodge

BEFORE THE ILLINOIS POLLUTION CONTROL BOARD

SANITARY DISTRICT OF)	
DECATUR,)	
)	
Petitioner,)	
)	
v.)	PCB 14- 111
)	(Variance – Water)
ILLINOIS ENVIRONMENTAL)	
PROTECTION AGENCY,)	
)	
Respondent.)	

PETITIONER’S RESPONSES TO THE ILLINOIS POLLUTION CONTROL BOARD’S QUESTIONS PURSUANT TO THE MARCH 18, 2014 HEARING OFFICE ORDER

NOW COMES Petitioner, SANITARY DISTRICT OF DECATUR (“District”), by and through its attorneys, HODGE DWYER & DRIVER, and pursuant to the March 18, 2014 Hearing Office Order, hereby responds to the Illinois Pollution Control Board’s (“Board”) Questions, the District states as follows:

1. **PCB 09-125 Condition 1(e)**
(a) The District states that the requested variance extension would “allow it more time to continue its investigation and implementation of adequate solutions regarding its *nickel* discharges. Pet. at 2 and 7, emphasis in original. Please specifically identify which of the items listed under Condition 1(e) for which the District Plans to continue its investigation and implementation during the extension period.

RESPONSE:

During the extension period, the District plans to continue its investigation and implementation in the development of a site-specific water quality standard proposal. Specifically, the District is in need of additional time to respond to questions related to the District’s proposed use of the Biotic Ligand Model (BLM) to support a site-specific standard petition.

Prior to and throughout the term of the current variance, the District has worked closely with Illinois Environmental Protection Agency ("Illinois EPA"), and through the Illinois EPA's assistance, with the United States Environmental Protection Agency ("U.S. EPA") Region 5, toward the preparation of a proposal for a site-specific standard. During the summer and fall of 2013, the District participated in a number of telephone conference calls with personnel from Illinois EPA, U.S. EPA Region 5, U.S. EPA's Duluth Research Laboratory, and U.S. EPA Headquarters. Several of the U.S. EPA personnel involved in these calls had not previously been involved in conversations with the District. During this period, U.S. EPA raised a number of additional questions regarding the technical basis of the BLM and information on a number of nickel toxicity studies reported in the scientific literature. These questions were summarized in a memorandum that was provided to the District on August 26, 2013. The questions were further discussed and clarified in subsequent conversations, most recently on December 5, 2013. The District's consultant has obtained the additional data that U.S. EPA requested be reviewed and evaluated, and is preparing responses to the questions.

Also during these telephone conversations, U.S. EPA suggested the option of performing aquatic toxicity testing to develop a proposed Water Effect Ratio (WER) to either supplement or substitute for a proposed standard based on the BLM. The District prepared and submitted a proposed WER testing plan to Illinois EPA and U.S. EPA Region 5 on January 30, 2014 and received review comments from U.S. EPA Region 5 on March 7. The District's consultant is also preparing responses to the testing plan review comments.

While the District is working diligently to provide responses, it is unlikely that the responses and any follow-up discussions or requests for information will be completed prior to expiration of the current variance on July 1, 2014. Resolution of these questions is needed to ensure that a site-specific water quality standard proposal presented to the Board for consideration would be approvable by Illinois EPA and by U.S. EPA.

1. **PCB 09-125 Condition 1(e)**

(b) The District states that a new round of chronic toxicity testing will be performed in the second half of 2011 because of inconsistencies in the chronic whole effluent toxicity testing results from 2007. Exh. D at 4. Please provide information regarding updated toxicity information.

RESPONSE:

The District stated its intention to conduct additional chronic toxicity in a semiannual report submitted to Illinois EPA in June 2011. Subsequently, the District completed additional acute toxicity testing as part of application requirements for NPDES permit renewal. A summary of the results of this acute toxicity testing is attached as Exhibit A. Because the acute testing did not indicate toxicity concerns, and chronic testing was not required either for permit renewal or for a site-specific standard request based on the BLM, the District has decided to defer additional chronic testing.

2. **PCB 09-125 Condition 1(f)**

(a) This condition requires industrial monitoring for nickel and zinc at least twice monthly at ADM and Tate & Lyle and at least semi-annually at other industrial users. While Tate & Lyle is complying with its zinc and nickel pretreatment limits and ADM is meeting its zinc limits, there is no mention of zinc and nickel monitoring at other industrial users. Exh. B at 2. Please clarify whether any other industrial users were required to monitor for zinc and nickel. If so, address whether the results of the industrial monitoring have identified any other significant sources of nickel that could be targeted for nickel reduction efforts.

RESPONSE:

The District determined that eight industrial users in addition to ADM and Tate & Lyle have the potential to discharge nickel and zinc. Discharges from these industrial users have been monitored since 2009 and none have been found to be significant sources of nickel or zinc due to low concentrations, low flow rates, or both. A summary of monitoring results from these industries is attached hereto as Exhibit B.

2. **PCB 09-125 Condition 1(f)**
(b) The District's Interim Reports starting with June 29, 2011 Interim Report (Exh. D) do not include monitoring results for nickel and zinc at Tate & Lyle. Please indicate if the District still requires industrial monitoring for nickel and zinc through its pretreatment ordinance for Tate & Lyle to comply with the variance condition.

RESPONSE:

The District continues to monitor the discharge from Tate & Lyle twice monthly for nickel and zinc, in compliance with the variance condition. The nickel and zinc contributions from Tate & Lyle were determined by the District to be not significant relative to the contributions from ADM, and monitoring results were therefore not included in semiannual reports. A summary of recent nickel and zinc monitoring results from Tate & Lyle is attached hereto as Exhibit C.

3. **PCB 09-125 Condition 1(g)**
This condition requires ongoing verification monitoring to confirm that cooling tower treatment programs are achieving the necessary zinc reductions. The petition indicates that both ADM and Tate & Lyle are meeting the zinc pretreatment limit. Exh. I at 3. Please indicate whether the District requires ongoing verification monitoring for cooling tower treatment program at Tate & Lyle beyond the monitoring information provided in the July 1, 2010 Interim Report. If so, please address whether ongoing monitoring show compliance with the pretreatment limits.

RESPONSE:

As noted in the response to Question 2(a) and 2(b) above, the discharge from Tate & Lyle is monitored twice monthly for zinc. Monitoring shows the discharge to be in compliance with the industry's pretreatment permit limits.

4. **PCB 09-125 Condition 1(h)(i)**

This condition requires the District to require ADM to complete technical and economic feasibility reviews of control technologies listed in Condition 1(h)(i)(A)-(J) by December 31, 2010. While ADM's December 22, 2010 review addresses the items in Condition 1(h)(i)(A)-(I), the review does not specifically address Condition 1(h)(i)(J), which refers to "Electro-Chemical Decomposition and Capacitive Deionization". (Exh. J). Please clarify whether the evaluation described in ADM's December 12, 2011 review (Exh. E) on page 8 was meant to cover "Electro-Chemical Decomposition and Capacitive Deionization" under Condition 1(h)(i)(J).

RESPONSE:

The Electro-Chemical Decomposition and Capacitive Deionization treatment technologies were investigated by ADM and their evaluation was included in ADM's June 2010 semi-annual report. A copy of the report is attached hereto as Exhibit D. The conclusions from the report were as follows:

Electro-Coagulation (EC): ADM Corn worked w/two different EC manufacturers: GlobalSep and Kaselco. GlobalSep actually showed a nickel increase due to their electrode construction. Kaselco had more expertise and tried pH reduction followed by CO2 removal prior to EC. The EC treatment then caused a pH increase. Little removal was shown. (p. 28)

Exhibit D at 28.

Captive Deionization (CDT)

At the suggestion of the Decatur Sanitary District, ADM had discussions with Dr. Michael Karpuk, President of TDA Research in Golden, CO (karpuk@tda.com) to understand the potential and applications of the CDT technology. CDT works using electrodes from carbon aerogels which, when placed under an electric charge,

bind charged ions and remove them from the contaminated water source. TDA indicated that they have licensed the technology to two separate companies (CDT systems in the United States and an unnamed licensee in Japan). However, CDT Systems is no longer in existence in the United States. During discussions with their CEO, John Davies, (972) 974-3667 (jddvrd@gmail.com) ADM learned that the technology was never scaled up beyond bench scale and that the company has been placed under receivership. There are three main challenges with captive deionization:

1. Lack of pilot scale or commercial scale supplier. To date, no companies have manufactured the electrodes.
2. Lack of selectivity. CDT will pick up all charged species in the water stream not just nickel and zinc. Consequently, when applied to ADM's high salt waste water stream, it will remove the bulk of the salts (about 3,000 ppm TDS) and this would entail evaporate a wastewater stream generating over 100 lbs of salt waste per day.
3. Electrode adsorption. The CDT electrodes are essentially activated carbons with charge groups on them. ADM believes that the presence of BOD/COD and color components in the wastewater stream will compete with the charged species for binding on the electrode and negate any benefit of using them.

CONCLUSIONS: This technology seems ill-suited for application in a complicated matrix such as wastewater treatment. (p. 26).

Exhibit D at 26.

5. **PCB 09-125 Condition 1(i)(ii)**
This condition requires the District, in part, to determine how much of the insoluble nickel and zinc entering the District's Main Plant is removed in the sludge. Please address the District's determination of how much of the insoluble nickel and zinc entering the District's Main Plant is removed in the sludge.

RESPONSE

The District completed the determination of insoluble nickel and zinc removal in 2009. The study indicated that less than one-half of the nickel and the majority of the

zinc entering the District's treatment facility is in the insoluble form. This insoluble metal is nearly completely removed by the treatment process. From this information, the District concluded that the pretreatment limit for nickel needs to regulate the total nickel concentration rather than the dissolved fraction to ensure that a total nickel effluent limit is met. This question has become less critical for zinc but pretreatment permit limits expressed as total zinc concentration are being retained for consistency and to simplify laboratory analysis for compliance determinations. A summary of laboratory results from the study is attached hereto as Exhibit E.

WHEREFORE, Petitioner, SANITARY DISTRICT OF DECATUR, submits the above Responses to the Illinois Pollution Control Board's Questions, pursuant to the March 18, 2014 Hearing Officer Order.

Respectfully Submitted,

SANITARY DISTRICT OF DECATUR,

Dated: April 21, 2014

By: /s/Katherine D. Hodge
Katherine D. Hodge

Katherine D. Hodge
Ethan S. Pressly
HODGE DWYER & DRIVER
3150 Roland Avenue
Post Office Box 5776
Springfield, Illinois 62705-5776
(217) 523-4900

Exhibit A



ACUTE TOXICITY TEST REPORT

Ceriodaphnia dubia

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-31458

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME:	Sanitary District of Decatur	NPDES PERMIT#:	IL0028321
SAMPLING DATE:	12/8/2010	DILUTION WATER:	MHSF Lab Control #112410LC
ORGANISM:	<i>Ceriodaphnia dubia</i>	LOCATION:	SDD FE
ORIGIN:	TestAmerica - In house cultures	TEST TYPE:	48 Hour ACUTE
AGE and DOB:	<24 hrs. old	SAMPLE TYPE:	Unchlorinated
TEST START:	12/9/2010 14:20	SAMPLE METHOD:	Composite
TEST END:	12/11/2010 13:05		

STATISTICAL ENDPOINT: LC50, TUa

TEST RESULTS

	LC ₅₀	TUa	A-NOEC
Concentration with statistical difference (LC50, TUa) TUa = 100%/LC50 (%)	>100%	<0.2	100.00%
Acute-No Observed Effect Concentration (A-NOEC)			

STATISTICAL METHOD: Linear Interpolation

95% confidence limits:	Lower:	Upper:
	N/A	N/A

SURVIVAL DATA SUMMARY

SURVIVAL (%)

Date	24 hr.	Lab Control	6.25%	12.50%	25%	50%	100%
12/10/2010	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
12/11/2010	48 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%

GENERAL CHEMISTRY - INIT. EFFLUENT SAMPLE

DISSOLVED OXYGEN:	9.5 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	2660 µS/cm	DISSOLVED OXYGEN	
pH:	7.8	AFTER AERATION:	N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-term Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).

YES (y/n)
 GRB

Analyst Initials

Primary Data Review
GRB 12/20/10
(Initial/Date)

Secondary Data Review
GRB 12-21-10
(Initial/Date)

GRB: Gary Benoit
EN: Ellen Nestaba
PS: Pat Sullivan
AMS: Alysa Stewart
RWE: Rich Emerich



ACUTE TOXICITY TEST REPORT

Pimephales promelas

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-31458

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME:	Sanitary District of Decatur	NPDES PERMIT#:	IL0028321
SAMPLING DATE:	12/8/2010	DILUTION WATER:	MHSF Lab Control #112410LC
ORGANISM:	<i>Pimephales promelas</i>	LOCATION:	SDD FE
ORIGIN:	Aquatic Bio Systems (Colorado)	TEST TYPE:	96 Hour ACUTE
AGE and DOB:	2 days old	SAMPLE TYPE:	Unchlorinated
TEST START:	12/9/2010 14:35	SAMPLE METHOD:	Composite
TEST END:	12/13/2010 14:05		

STATISTICAL ENDPOINT: LC₅₀, TUa

TEST RESULTS

	LC ₅₀	TUa	A:NOEC
Concentration with statistical difference (LC ₅₀ , TUa) TUa = 100%/LC50 (%)	>100%	<0.2	100%
Acute-No Observed Effect Concentration (A-NOEC)			

STATISTICAL METHOD: Linear Interpolation

95% confidence limits	Lower	Upper
	N/A	N/A

SURVIVAL DATA

SURVIVAL (%)

DATE		Lab Control	6/25%	12/50%	25%	50%	100%
12/10/2010	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
12/11/2010	48 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
12/12/2010	72 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
12/13/2010	96 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	95.0%

GENERAL CHEMISTRY SUMMARY- INIT EFFLUENT SAMPLE

DISSOLVED OXYGEN:	9.5 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	2660 µS/cm	DISSOLVED OXYGEN AFTER AERATION:	N/A mg/L
pH:	7.8		

All methods and guidelines used were consistent with the protocol from Short-term Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s). YES (y/n) GRB

Analyst Initials

Primary Data Review
GRB 12/20/10
(Initial/Date)

Secondary Data Review
GRB 12-21-10
(Initial/Date)

- GRB: Gary Benoit
- EN: Ellen Nasiatka
- PS: Pat Sullivan
- AMS: Alyse Stewart
- RWE: Rich Emerich



TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

ACUTE TOXICITY TEST REPORT

Ceriodaphnia dubia

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-32642

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME:	Sanitary District of Decatur	NPDES PERMIT#:	IL0028321
SAMPLING DATE:	3/9/2011	DILUTION WATER:	MHSF Lab Control # 030911LC
ORGANISM:	<i>Ceriodaphnia dubia</i>	LOCATION:	SDD FE
ORIGIN:	TestAmerica - In house cultures	TEST TYPE:	48 Hour ACUTE
AGE and DOB:	<24 hrs. old	SAMPLE TYPE:	Unchlorinated
TEST START:	3/10/2011 12:15	SAMPLE METHOD:	Composite
TEST END:	3/12/2011 11:20		

STATISTICAL ENDPOINT: LC50, TUa

TEST RESULTS

	LC50	TUa	A-NOEC
Concentration with statistical difference (LC50, TUa) TUa = 100%/LC50 (%)	>100%	<0.2	100.00%
Acute-No Observed Effect Concentration (A-NOEC)			

STATISTICAL METHOD: Linear Interpolation

95% confidence limits:

N/A	N/A
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SURVIVAL DATA SUMMARY

SURVIVAL (%)

DATE	TIME	Survival	25%	12.50%	6.25%	3.125%	1.5625%	0.78125%
3/11/2011	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
3/12/2011	48 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%

GENERAL CHEMISTRY - INIT. EFFLUENT SAMPLE

DISSOLVED OXYGEN:	9.8 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	2170 µS/cm	DISSOLVED OXYGEN	
pH:	7.9	AFTER AERATION:	N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-terms Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).

YES (y/n)
 GRB
Analyst Initials

Primary Data Review
GRB 3/18/11
(Initial/Date)

Secondary Data Review
GRB 3-18-11
(Initial/Date)

GRB: Gary Benoit
EN: Ellen Nastatka
PS: Pat Sullivan
AMS: Alyse Stewart
RVE: Rich Emerich



TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

ACUTE TOXICITY TEST REPORT

Pimephales promelas

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-32642

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME:	Sanitary District of Decatur	NPDES PERMIT#:	IL0028321
SAMPLING DATE:	3/9/2011	DILUTION WATER:	MHSF Lab Control # 030911LC
ORGANISM:	<i>Pimephales promelas</i>	LOCATION:	SDD FE
ORIGIN:	Aquatic Bio Systems (Colorado)	TEST TYPE:	96 Hour ACUTE
AGE and DOB:	<48 hours old	SAMPLE TYPE:	Unchlorinated
TEST START:	3/10/2011 12:40	SAMPLE METHOD:	Composite
TEST END:	3/14/2011 12:20		

STATISTICAL ENDPOINT: LC₅₀, TUa

TEST RESULTS

	LC ₅₀	TUa	A-NOEC
Concentration with statistical difference (LC ₅₀ , TUa) TUa = 100%/LC50 (%)	>100%	<0.2	100%
Acute-No Observed Effect Concentration (A-NOEC)			

STATISTICAL METHOD: Maximum Likelihood-Weibull

95% Confidence Interval	Upper	Lower
	N/A	N/A

SURVIVAL DATA

SURVIVAL (%)

DATE	Lab Control	6.25%	12.5%	25%	50%	100%
3/11/2011 24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
3/12/2011 48 hr.	100.0%	100.0%	100.0%	100.0%	95.0%	100.0%
3/13/2011 72 hr.	100.0%	100.0%	100.0%	100.0%	95.0%	100.0%
3/14/2011 96 hr.	100.0%	100.0%	100.0%	100.0%	95.0%	92.5%

GENERAL CHEMISTRY SUMMARY- INIT EFFLUENT SAMPLE

DISSOLVED OXYGEN:	9.8 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	2170 µS/cm	DISSOLVED OXYGEN	
pH:	7.9	AFTER AERATION:	N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-terms Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).

YES (y/n)
GRB
Analyst Initials

Primary Data Review
GRB 3/18/11
(Initial/Date)

Secondary Data Review
GRB 3/21/11
(Initial/Date)

GRB: Gary Benoit
EN: Ellen Nasatka
PS: Pat Sullivan
AMS: Alyse Stewart
RWE: Rich Emerich

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TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

ACUTE TOXICITY TEST REPORT

Ceriodaphnia dubia

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-36316

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME: Sanitary District of Decatur
SAMPLING DATE: 9/14/2011
ORGANISM: *Ceriodaphnia dubia*
ORIGIN: TestAmerica - In house cultures
AGE and DOB: <24 hrs. old
TEST START: 9/15/2011 12:16
TEST END: 9/17/2011 13:10

NPDES PERMIT#: IL0028321
DILUTION WATER: MHSF Lab Control # 090811LC
LOCATION: SDD FE
TEST TYPE: 48 Hour ACUTE
SAMPLE TYPE: Unchlorinated
SAMPLE METHOD: Composite

STATISTICAL ENDPOINT: LC50, TUa

TEST RESULTS

	LC50	TUa	A-NOEC
Concentration with statistical difference (LC50, TUa) TUa = 100%/LC50 (%)	>100%	<0.2	100.00%
Acute-No Observed Effect Concentration (A-NOEC)			

STATISTICAL METHOD: Linear Interpolation

95% Confidence Limits	N/A	N/A
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SURVIVAL DATA SUMMARY

SURVIVAL (%)

Date	Time	Control	Survival	50%	25%	50%	100%
9/16/2011	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
9/17/2011	48 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%

GENERAL CHEMISTRY - INIT. EFFLUENT SAMPLE

DISSOLVED OXYGEN: 8.4 mg/L
CONDUCTIVITY: 3490 µS/cm
pH: 8.0

RESIDUAL CHLORINE: <0.02 mg/L
DISSOLVED OXYGEN AFTER AERATION: N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-term Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).

YES (y/n)
 GRB
Analyst initials

Primary Data Review
GRB 9/28/11
(Initial/Date)

Secondary Data Review
CPR 9/27/11
(Initial/Date)

GRB: Gary Bando
EN: Ellen Nestlaka
RWE: Rich Emerich
AMS: Alyse Stewart

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TestAmerica <small>THE LEADER IN ENVIRONMENTAL TESTING</small>	ACUTE TOXICITY TEST REPORT <i>Pimephales promelas</i>
TestAmerica, Inc. 53 Southampton Road Westfield, MA 01085	Aquatic Toxicology - Biology Department Job#: 360-36316

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME: Sanitary District of Decatur SAMPLING DATE: 9/14/2011 ORGANISM: <i>Pimephales promelas</i> ORIGIN: Aquatic Bio Systems (Colorado) AGE and DOB: 4 days old TEST START: 9/15/2011 12:29 TEST END: 9/19/2011 12:14	NPDES PERMIT#: IL0028321 DILUTION WATER: MHSF Lab Control # 090811LC LOCATION: SDD FE TEST TYPE: 96 Hour ACUTE SAMPLE TYPE: Unchlorinated SAMPLE METHOD: Composite STATISTICAL ENDPOINT: LC ₅₀ , TUa
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TEST RESULTS

Concentration with statistical difference (LC ₅₀ , TUa) TUa = 100%/LC50 (%) Acute-No Observed Effect Concentration (A-NOEC)	>100%	0.3	50%
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STATISTICAL METHOD: Maximum Likelihood-Probit

Lower N/A	Upper N/A
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SURVIVAL DATA **SURVIVAL (%)**

DATE	TIME	CONCENTRATION	6.25%	12.50%	25%	50%	100%
9/16/2011	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
9/17/2011	48 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	95.0%
9/18/2011	72 hr.	100.0%	97.5%	100.0%	95.0%	95.0%	90.0%
9/19/2011	96 hr.	100.0%	92.5%	95.0%	92.5%	90.0%	85.0%

GENERAL CHEMISTRY SUMMARY- INIT EFFLUENT SAMPLE

DISSOLVED OXYGEN:	8.4 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	3490 µS/cm	DISSOLVED OXYGEN	
pH:	8.0	AFTER AERATION:	N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-terms Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).
 YES (y/n)
 GRB
 Analyst Initials

Primary Data Review GRB 9/26/11 (Initial/Date)	Secondary Data Review <u>CFL 9/27/11</u> (Initial/Date)	GRB: Gary Benoit EN: Ellen Naalaka RWE: Rich Emerich AMS: Alyse Stewart
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TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

ACUTE TOXICITY TEST REPORT

Ceriodaphnia dubia

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-34326

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME:	Sanitary District of Decatur	NPDES PERMIT#:	IL0028321
SAMPLING DATE:	6/8/2011	DILUTION WATER:	MHSF Lab Control # 060211LC
ORGANISM:	<i>Ceriodaphnia dubia</i>	LOCATION:	SDD FE
ORIGIN:	TestAmerica - In house cultures	TEST TYPE:	48 Hour ACUTE
AGE and DOB:	<24 hrs. old	SAMPLE TYPE:	Unchlorinated
TEST START:	6/9/2011 15:20	SAMPLE METHOD:	Composite
TEST END:	6/11/2011 14:49		

STATISTICAL ENDPOINT: LC50, TUa

TEST RESULTS

	LC50	TUa	NOEC
Concentration with statistical difference (LC50, TUa) TUa = 100%/LC50 (%)	>100%	0.4	100.00%
Acute-No Observed Effect Concentration (A-NOEC)			

STATISTICAL METHOD: Linear Interpolation

SURVIVAL DATA SUMMARY

SURVIVAL (%)

Date	Time	Lab Control	100%	75%	50%	25%	0%
6/10/2011	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	85.0%
6/11/2011	48 hr.	100.0%	100.0%	100.0%	100.0%	100.0%	80.0%

GENERAL CHEMISTRY - INIT. EFFLUENT SAMPLE

DISSOLVED OXYGEN:	9.2 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	2840 µS/cm	DISSOLVED OXYGEN	
pH:	8.1	AFTER AERATION:	N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-terms Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).

YES (y/n)
 GRB

Analyst Initials

Primary Data Review
GRB 6/22/11
(Initial/Date)

Secondary Data Review
GRB 6/22/2011
(Initial/Date)

GRB: Gary Benoit
EN: Ellen Nestaka
RWE: Rich Emerich
AMS: Alyse Stewart

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ACUTE TOXICITY TEST REPORT

Pimephales promelas

TestAmerica, Inc.
53 Southampton Road
Westfield, MA 01085

Aquatic Toxicology - Biology Department

Job#: 360-34326

SAMPLE AND TEST IDENTIFICATION

CLIENT NAME:	Sanitary District of Decatur	NPDES PERMIT#:	IL0028321
SAMPLING DATE:	6/8/2011	DILUTION WATER:	MHSF Lab Control # 080211LC
ORGANISM:	<i>Pimephales promelas</i>	LOCATION:	SDD FE
ORIGIN:	Aquatic Bio Systems (Colorado)	TEST TYPE:	96 Hour ACUTE
AGE and DOB:	<48 hours old	SAMPLE TYPE:	Unchlorinated
TEST START:	6/9/2011 15:40	SAMPLE METHOD:	Composite
TEST END:	6/11/2011 14:57		

STATISTICAL ENDPOINT: LC₅₀, TUa

TEST RESULTS

Concentration with statistical difference (LC ₅₀ , TUa) TUa = 100%/LC50 (%)	LC ₅₀	TUa	RANGE
Acute-No Observed Effect Concentration (A-NOEC)	>100%	0.2	50%

STATISTICAL METHOD: Maximum Likelihood-Probit	95% Confidence Limit Lower	Upper
	N/A	N/A

SURVIVAL DATA

SURVIVAL (%)

DATE	TIME	100%	75%	50%	25%	100%
6/10/2011	24 hr.	100.0%	100.0%	100.0%	100.0%	100.0%
6/11/2011	48 hr.	100.0%	97.5%	100.0%	100.0%	100.0%
6/12/2011	72 hr.	100.0%	97.5%	100.0%	100.0%	100.0%
6/13/2011	96 hr.	100.0%	92.5%	97.5%	95.0%	97.5%

GENERAL CHEMISTRY SUMMARY- INIT EFFLUENT SAMPLE

DISSOLVED OXYGEN:	9.2 mg/L	RESIDUAL CHLORINE:	<0.02 mg/L
CONDUCTIVITY:	2840 µS/cm	DISSOLVED OXYGEN	
pH:	8.1	AFTER AERATION:	N/A mg/L

All methods and guidelines used were consistent with the protocol from Short-terms Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, October 2002, EPA-821-R-02-012.

All acceptable method criteria were met: 90% or greater survival in the control(s).

YES (y/n)

GRB

Analyst Initials

Primary Data Review

GRB 6/22/11

(Initial/Date)

Secondary Data Review

GRB 6-22-2011

(Initial/Date)

GRB: Gary Benoit

EN: Ellen Nasalaka

RWE: Rich Emerich

AMS: Alyse Stewart

Exhibit B

Minor Industries - Nickel and Zinc Results			
Industrial Discharge Point	Dates of Sampling	Nickel Range, mg/L	Zinc Range, mg/L
Caterpillar Point A	4/2009 - 1/2014	0.002 - 0.015	0.068 - 1.62
Caterpillar Point B	4/2009 - 1/2014	0.004 - 0.024	0.039 - 2.12
Caterpillar Point D	4/2009 - 8/2010	0.001 - 0.004	0.060 - 0.092
Decatur Plating	7/2009 - 1/2014	0.004 - 0.040	0.116 - 6.16
Dec. Mem. Hosp. Point A	4/2009 - 2/2011	0.003 - 0.003	0.108 - 0.316
Dec. Mem. Hosp. Point D	4/2009 - 2/2014	0.002 - 0.015	0.038 - 0.283
Dec. Mem. Hosp. Point E	4/2009 - 2/2014	0.001 - 0.005	0.043 - 0.726
ICPC	7/2009 - 1/2014	0.002 - 0.094	0.102 - 1.48
Mason Mfg.	5/2009 - 7/2013	0.001 - 0.065	0.009 - 0.494
Mueller Point 1A	3/2014	0.005	0.268
Mueller Point 4D	5/2009 - 8/2013	0.001 - 0.042	0.017 - 4.73
Mueller Point 4E	5/2013 - 8/2013	0.002 - 0.003	0.031 - 0.394
PPG	1/2011 - 7/2012	0.002 - 0.007	0.044 - 1.92
St. Mary's Hosp. Point B	5/2009 - 2/2014	0.001 - 0.007	0.017 - 0.931
St. Mary's Hosp. Point C	5/2009 - 2/2014	0.002 - 0.004	0.036 - 0.128

Exhibit C

TLIA Metals - Discharge Point A						
	Total	Total			Total	Total
Sample	Nickel, Tot	Zinc		Sample	Nickel, Tot	Zinc
Date	mg/L	mg/L		Date	mg/L	mg/L
1/14/2013	0.00607	0.0386		9/4/2013	0.0133	0.0687
2/4/2013	0.00572	0.0687		9/7/2013	0.0135	0.051
2/11/2013	0.00695	0.0554		9/9/2013	0.0134	0.0702
3/2/2013	0.0052	0.0419		9/14/2013	0.0104	0.0407
3/4/2013	0.00582	0.0508		9/18/2013	0.0126	0.0458
3/9/2013	0.00372	0.031		9/21/2013	0.011	0.0514
3/11/2013	0.00413	0.0377		9/25/2013	0.0122	0.0537
3/16/2013	0.00551	0.035		9/28/2013	0.0105	0.0516
3/20/2013	0.00671	0.0416		10/1/2013	0.0104	0.0457
3/23/2013	0.00524	0.0404		10/7/2013	0.0101	0.0596
3/27/2013	0.00434	0.0384		10/27/2013	0.0228	0.0278
3/30/2013	0.00591	0.0481		10/28/2013	0.00884	0.0281
4/3/2013	0.00332	0.0183		11/4/2013	0.0236	0.163
4/6/2013	0.00477	0.0405		11/11/2013	0.0085	0.0808
4/8/2013	0.00485	0.0447		12/2/2013	0.0106	0.0533
4/13/2013	0.00399	0.0412		12/9/2013	0.00739	0.0485
4/15/2013	0.00594	0.0767		1/6/2014	0.00659	0.0917
4/20/2013	0.00525	0.049		1/20/2014	0.00489	0.0809
4/24/2013	0.0041	0.0514		2/3/2014	0.00942	0.148
4/27/2013	0.0072	0.0693		2/10/2014	0.00672	0.103
5/1/2013	0.00557	0.0303		3/3/2014	0.0041	0.0538
5/4/2013	0.00633	0.0514		3/10/2014	0.00645	0.0732
5/6/2013	0.00565	0.0618				
5/11/2013	0.00663	0.0422				
5/15/2013	0.0061	0.061				
5/18/2013	0.00565	0.0299				
5/22/2013	0.00537	0.0533				
5/25/2013	0.0133	0.198				
5/29/2013	0.00581	0.0389				
6/1/2013	0.00716	0.131				
6/3/2013	0.00723	0.0848				
6/8/2013	0.00485	0.0452				
6/10/2013	0.00385	0.025				
6/15/2013	0.00681	0.0662				
6/19/2013	0.00928	0.105				
6/22/2013	0.00641	0.0472				
6/26/2013	0.00811	0.139				
6/29/2013	0.0104	0.0974				
7/1/2013	0.0098	0.0693				
7/6/2013	0.00791	0.053				
7/8/2013	0.0109	0.117				
7/13/2013	0.00731	0.0539				
7/17/2013	0.00678	0.0493				
7/20/2013	0.00855	0.0605				
7/24/2013	0.0107	0.057				
7/27/2013	0.00885	0.0458				
8/1/2013	0.00802	0.0508				
8/3/2013	0.00721	0.0518				
8/5/2013	0.00661	0.0418				
8/10/2013	0.00592	0.0459				
8/14/2013	0.00887	0.068				
8/17/2013	0.00813	0.0587				
8/21/2013	0.00785	0.0458				
8/24/2013	0.00921	0.0857				
8/28/2013	0.0126	0.059				
8/31/2013	0.0126	0.0962				

Exhibit D



To: Decatur Sanitary District
Illinois Environmental Protection Agency

From: Archer Daniels Midland

Date: June 30, 2010

Re: Status Report Compliance Strategy for 2009-2010 for Decatur Sanitary District and
ADM Decatur WWTP for waste treatment.

Summary

ADM Research has been evaluating treatment options for the reduction of nickel and zinc in ADM's wastewater streams. Over 24 companies with methods or processes have been investigated to reduce these metals. To date there has been some success with five approaches that merit additional evaluation:

1. Di- Methyl Di Thio Carbamate product from Hychem and Hydrite. In both cases Ni was reduced to below 0.037 ppm.
2. Ultra-filtering and the RO of the waste water to a ND nickel
3. Binding the nickel complex with a polymer and microfiltration which reduced it to 0.038 ppm
4. Using a decolorizing resin which requires a very high dosage of adsorbent (>10g/100g)
5. Using a chitosan based adsorbent which reduced nickel to 0.012 ppb but also required very high dosages (>10g/100g).

ADM Research is continuing to work to identify additional protocols and chemicals to reduce nickel and zinc in the Decatur Plant effluent.

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ADM has been actively pursuing technologies to remove nickel and zinc from its effluent discharge. This document reports the progress ADM has made in the past year.

BACKGROUND: Nickel and zinc are present in effluent leaving the ADM Decatur Complex Waste Water plant. New limits are proposed which will reduce the discharge limits to 0.037 ppm for nickel and 0.35 ppm for zinc¹. Of the two metals, nickel is more difficult to remove from the effluent. Typical concentrations and quantities of the various waste water treatment plant influents are shown in TABLE 1.

TABLE 1 - HIGH SALT SIDE of WWTP ONLY															
	EAST PLANT			BIO PRODUCTS			THREONINE, GLYCOL & OTHER			CORN PLANT			High Salt Effluent (not including Sludge Wasting)		
	Suspended	Soluble	TOTAL	Suspended	Soluble	TOTAL	Suspended	Soluble	TOTAL	Suspended	Soluble	TOTAL	Suspended	Soluble	TOTAL
ppm Nickel	0.02	0.17	0.19	0.002	0.03	0.032	0.05	0.03	0.08	0	0.12	0.12	0.01	0.11	0.12
ppm Zinc	0.2	0.6	0.8	0.1	0.2	0.3	1	0.1	1.1	0	0.4	0.4	0.02	0.04	0.06
ppm Suspended Solids	1,800			1,300			5,600			0			225		
ppm Dissolved Salts	2,450			5,000			N/A			6,700			4,000		
Flow Rate (MGD)	2.2			1.4			0.6			2.0			6.2		
Lbs / day Nickel (suspended)	0.36			0.023			0.18			0			0.52		
Lbs / day NI (soluble)	3.1			0.35			0.11			2.0			5.66		
Lbs / day total Zinc	14.6			3.5			5.5			6.6			3.1		

As stated above, the nickel is more difficult to remove from the effluent stream than zinc, the majority of which precipitates as zinc sulfide in the anaerobic process in the wastewater treatment plant. Circumstantial evidence indicates that the nickel is complexed with another compound making it unavailable for many conventional removal technologies. The complexing compound is believed to be primarily a phosphorous containing material because for some nickel removal technologies, most of the phosphorous compound needed to be removed before the nickel could be removed. However, subsequent testing with an adsorbent that removed 99+% P did not achieve the nickel limit. Thus, it is likely that nickel is complexed with more than one type of compound.

The majority of nickel and zinc in the ADM effluent originates in the corn and soybeans being processed in the plant. During the processing, the metals are released and enter the processing water which eventually ends up at the wastewater treatment plant.

¹ Decatur Sanitary District ADM Permit 2009

To identify methods to reduce the nickel and zinc concentration in the ADM wastewater treatment plant effluent, 24 technologies/companies were investigated. Because ~85% of the incoming zinc forms an insoluble metal sulfide in the waste treatment's anaerobic fermentation vessels, zinc is a primary issue in solid waste leaving the treatment plant (see Section 1.6).

Soluble nickel containing compounds, which are the focus of the current report, originate mainly in the East Plant (1.0 kg/day) and Corn Plant refinery (0.71 kg/day). West Plant soluble nickel, although relatively low, presents an unusual problem in that it is cycled up ~4 times in the Corn Plant cooling towers. This results in nickel concentration issues in the non-High Salt waste. The main hurdles with soluble nickel removal are its already low concentration and its being tightly bound as in a complex. The major process flows with metal concentrations are shown in TABLE 1. A diagram of ADM's Decatur facilities wastewater treatment plant is shown in FIGURE 1.

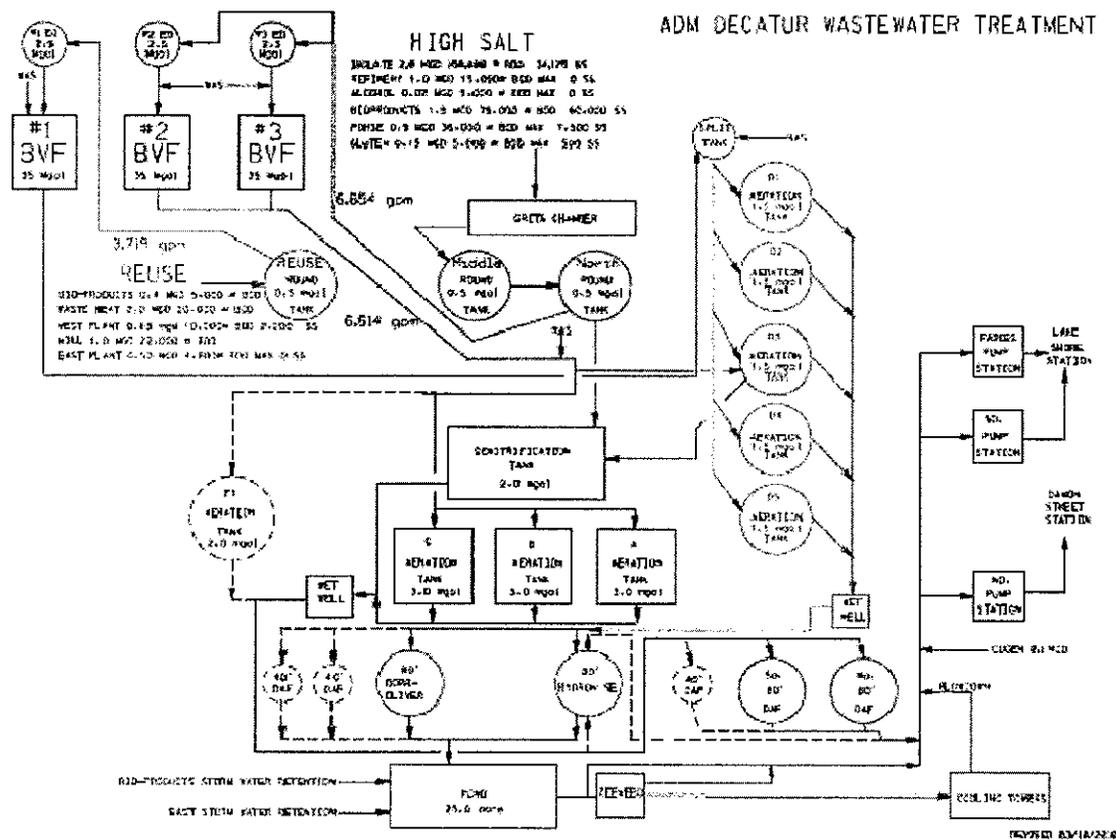


FIGURE 1

While removal of suspended nickel (insoluble) is relatively straightforward, it is a significant challenge to remove soluble nickel from ADM's Decatur wastewater discharge, which constitutes the bulk of the nickel discharge.

One of the key challenges is that several of the techniques reported herein work best at an alkaline pH. Such an approach would require the facility to add a substantial amount of caustic to the DAF (Dissolved Air Flotation) effluent. The FIGURE 2 illustrates daily addition of caustic to our DAF effluent as a function of pH.

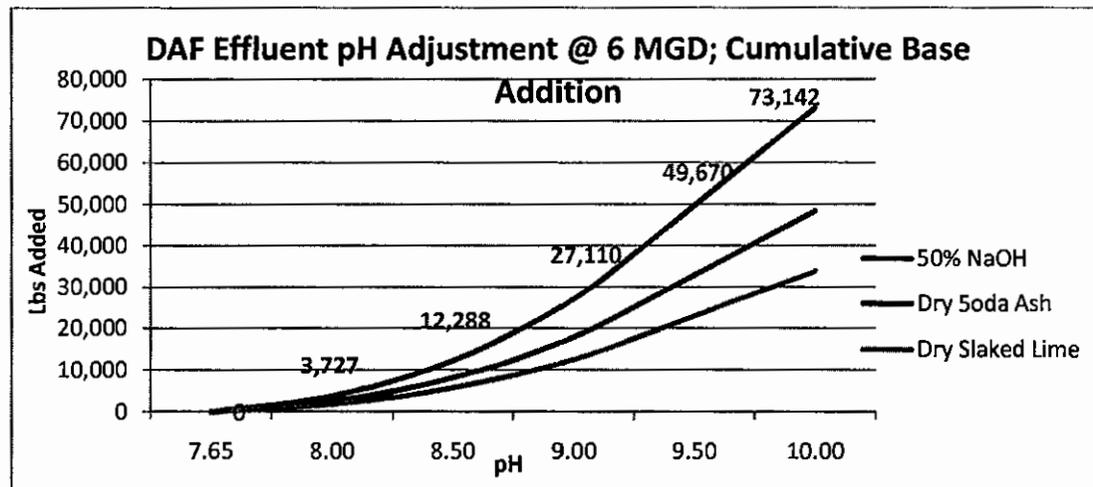


FIGURE 2

The discussion below summarizes the various technologies/companies that have been investigated. Some of the technologies have been tried using ADM process discharge samples, and in a number of cases, chemical usage and treatment costs have been estimated. It is believed that the results reported below are accurate as of the sample and analysis date. However, it must be recognized that there is significant flow variability in the processes at the ADM facility depending on the processing conditions. Consequently, any results reported below are specific to those dates of analysis and should be construed as a broad generalization of the operating conditions and treatment methods. Also, note that a significant portion of the analyses reported below were performed at outside laboratories and/or companies. As such, they were outside the control of ADM and will need to be reproduced internally to verify accuracy.

Finally, pertaining to any adsorbent-related technology (Eagle-Picher NXT-2, Dow Optipore, Carbon, Clays, etc), while these materials are capable of removing organically-chelated nickel they do so by also adsorbing the bulk of the other soluble organic matter. This causes very high usage rates and makes these processes extremely uneconomical.

1 Deliverables:

1.1 Nickel- Proprietary Precipitation Process:

1.1.1 EcoVu:

Description: An Ottawa based startup company has performed some pioneering work on removing metals from the St. Lawrence River. Several ADM samples have been run with the EcoVu process and have seen between 60-70% reduction in soluble Ni as shown in [FIGURE 3](#) and listed in [TABLE 2](#). The technology captures most of metal ions listed in [FIGURE 4](#).

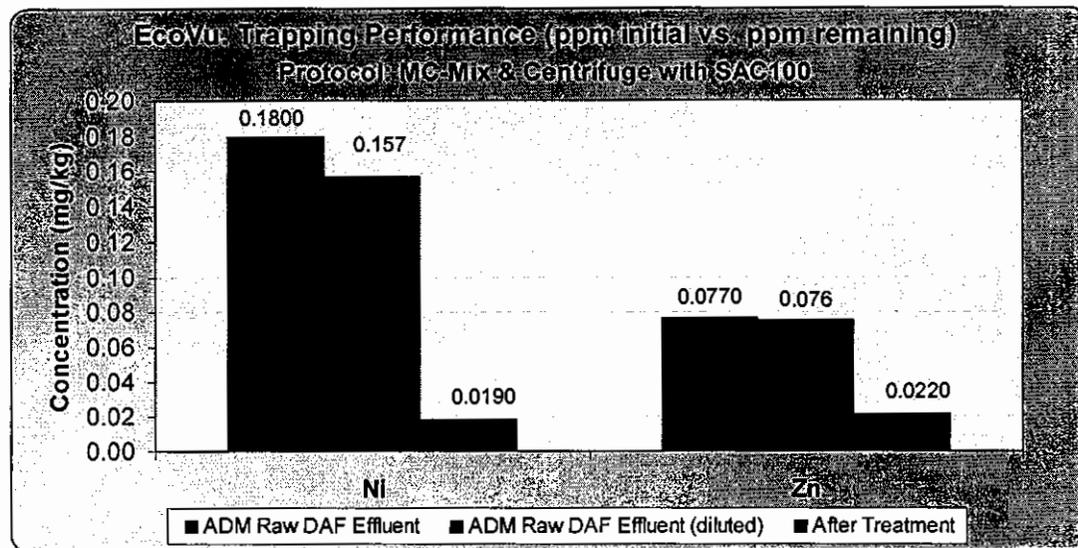


FIGURE 3

TABLE 2

Protocol	EcoTrap™	[Ni] _{initial} , mg/kg	[Ni] _{after EcoVu treatment} , mg/kg	Trapping Performance, %
Mix&Settle	SAC100	0.110	0.052	53
Mix&Settle	S45100	0.100	0.052	48
Mix&Filter	SAC100/S45010	0.095	0.065	32
Mix,Centrifuge&Filter	SAC100/S45010	0.110	0.010	91
Mix&Centrifuge	SAC100/S45010	0.157	0.019	88
Pre-filtered DAF + Mix&Filter	SAC100/S45010	0.155	0.050	68
Pre-filtered DAF + Mix,Centrifuge&Filter	SAC100/S45010	0.155	0.040	74

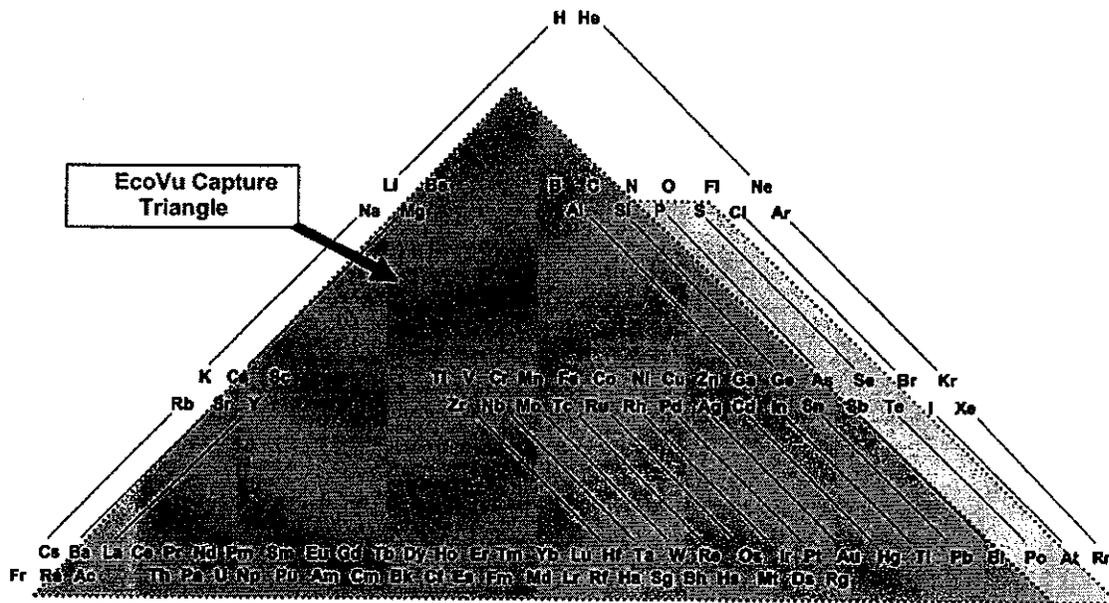


FIGURE 4: The Zmaczynski Periodic Triangle.

1.1.1.1 Status

Results have shown significant variability and require large contact times for the material to adequately sequester the nickel. In addition, a process of mixing, followed by centrifugation and filtration, is needed to recover the adsorbent material.

1.1.1.2 Technical Feasibility

The EcoVu product could be added either in a precoat filter or in the DAF which will allow for adequate contact time for nickel sequestration. This is shown in FIGURE 5.

FIGURE 5

1.1.1.3 Capital and Operation Costs

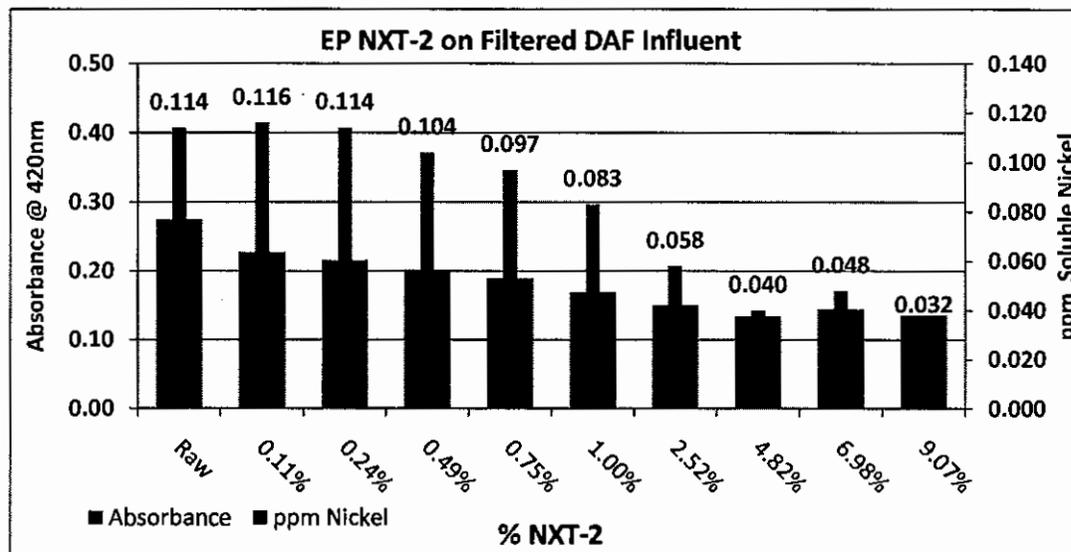
Based on initial discussions with the company for a 6,000,000 gallon per day high salt waste stream, about 1000 kg of Ecotap is required at \$2500 / ton.

1.1.1.4 Reliability

Ecovu has been unwilling to share samples of their material for onsite trials, and it was difficult to develop a working arrangement for Ecovu's process at the facility. However, trials at Ecovu's facility, effluent from the ADM plant has shown a reduction in both nickel and phosphate. The latest proposal from Ecovu seeks an investment of over \$500,000 into the company to scale up manufacturing to perform a pilot trial at ADM Decatur.

1.1.2 EP Minerals NXT-2

EP Minerals manufactures acidic clay that has been tried with ADM wastewaters with limited success in nickel removal. NXT-2 is a high surface media that is marketed by EP minerals for arsenic removal from wastewater to below 5 ppb. NXT-2 is widely used in municipal water treatment and approved for use by the EPA for such applications. Results are shown in FIGURE 6.



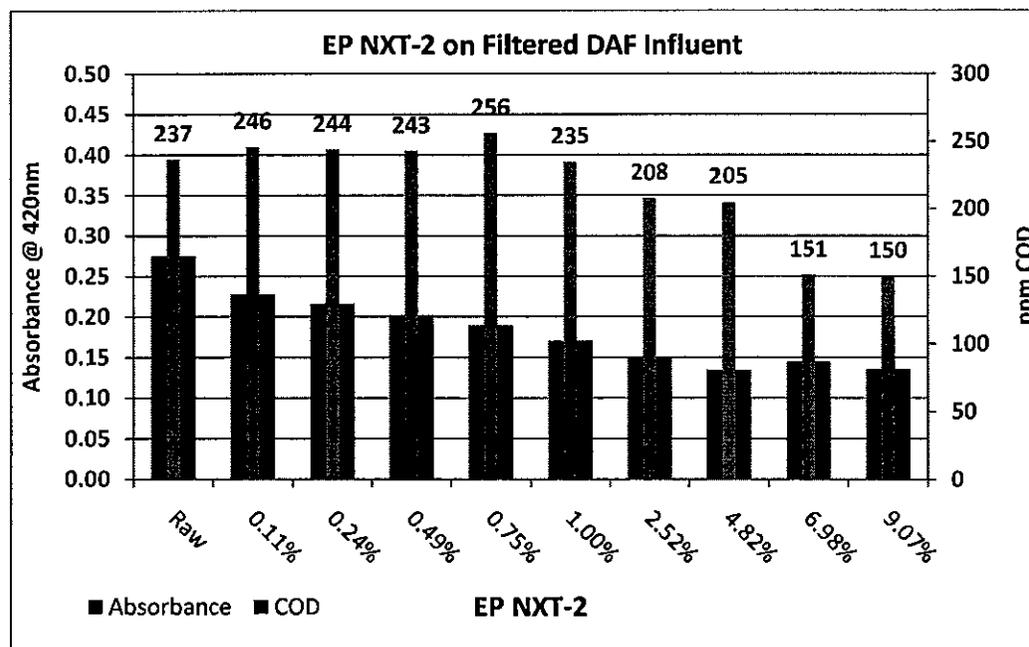


FIGURE 6

Additionally, ADM is testing a liquid slurry version (NXT-CF) of the adsorbent that is now being marketed.

1.1.2.1 Technical Feasibility

Current dosages for NXT-2 on ADM wastewater effluent are very high. Additional dosage trials are being developed to optimize the loading. Regeneration of NXT-2 is based on a pH 14 rinse to produce a concentration stream of nickel waste.

1.1.2.2 Capital and Operation Costs

NXT-2 is currently listed at 360\$/cuft. At a 1% dosage it would require about 5,000,000 lbs per day usage for the ADM WWTP plant. This does not appear to be a feasible application unless a packed column design is developed with ability to regenerate the adsorbent.

1.1.2.3 Reliability

EP Minerals is a large supplier of clay to ADM's oil refineries. No issues are expected with sourcing or supply of the material.

1.1.3 Crystal Clear Technologies

Crystal Clear Technologies (CCT) of Oregon has developed nanocoating technology that when bonded to a high surface area substrate (primary based on chitosan), can transform the substrate into a high capacity heavy metal adsorption media. CCT has focused on the heavy metals in the EPA primary drinking water standard as well as metal removal to meet EPA discharge standards. It has developed several functionalized medias that can adsorb heavy metals in the presence of high TDS complex matrices. This is not a precipitation technology. Being able to adsorb the heavy metals and not the salt or other ions comprising the TDS load, is a major advantage for treating wastewater.

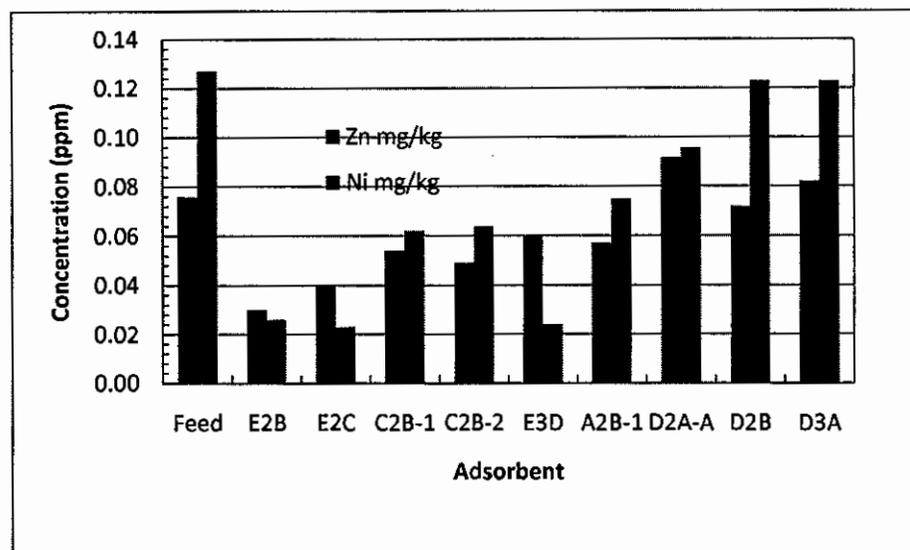


FIGURE 7

Several samples of ADM effluent have been sent to their facility for testing and analysis, the results of which are shown in FIGURE 7. ADM is in the process of setting up a research program with CCT.

1.1.3.1 Technical Feasibility

Current dosages for CCT's E3D, which is the best performing adsorbent on ADM's effluent, are very high. ADM is currently working with CCT to set up additional dosage trials to optimize the loading.

1.1.3.2 Capital and Operation Costs

Based on the prior work where CCT's material removed ~27 gm of arsenic on a Kg of material w/ 8 successive layers of ligands at \$41 / Kg, that equates to approximately 100 Kg per day to treat the total nickel leaving the ADM facility of 6 lbs of Ni, at a cost of \$4100 / day

1.1.3.3 Reliability

CCT is a startup company with no manufacturing facilities. ADM is working with them to develop a program for production of pilot quantities of the material.

1.1.4 Siemens WT

Siemens has been working with their proprietary metal removal chemistry in a packed bed column. Siemens indicated a pH reduction to 6.5 was required, but while running the DAF effluent through a SCU media filter, nickel was removed by about 82.1% to about 20 ppb as shown in FIGURE 8 and TABLE 3.

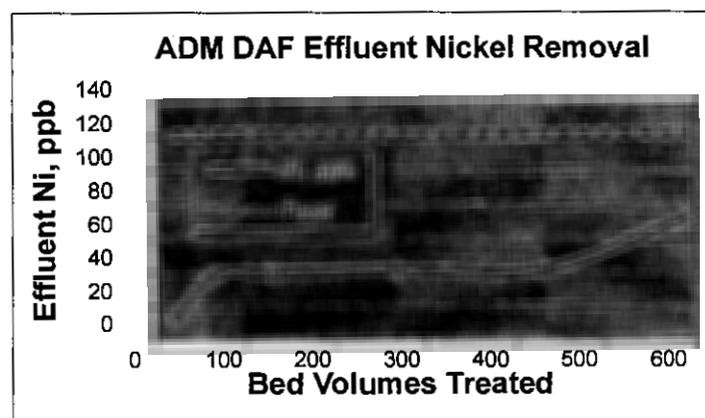


FIGURE 8

TEST NO.	TEST CODE	COMMERCIALY AVAILABLE?	NICKEL, ppb	% NICKEL REMOVE D	< 37 ppb LIMIT ?	COMMENTS
CHEMICAL REDUCTION TESTS						
1	CHEM-C	NA	130	NA	NA	Sample as received filtered thru 934AH glass fiber filter
2	CHEM1-1	YES	98.9	-23.9	NO	Treatment for 1 hr with reducing agent 1
3	CHEM1-D	NA	160	23.1	NO	Duplicate of sample 2 but contaminated
4	CHEM2	YES	89.0	-31.5	NO	Treatment for 1 hr with reducing agent 2
5	CHEM3	YES	106	-18.5	NO	Treatment for 1 hr with reducing agent 3
ISOTHERM TESTS						
6	CON	NA	112	NA	NA	Sample as received, no treatment of any kind
7	CAP	YES	62.8	-43.9	NO	
8	SCU	YES	56.5	-49.6	NO	
9	SCZ	YES	76.5	-31.7	NO	
10	CHW	YES	84.8	-24.3	NO	
11	ACC	YES	63.4	-43.4	NO	
12	CAM	YES	123	9.8	NO	500 ml ADM sample contacted with 10 g of various ion exchange and adsorptive media for 96 hours, filtered thru Whatman #1 paper
MODIFIED COLUMN TESTS						
13	ACV-D	YES	74.5	NA	NA	
14	ACV-C	YES	48.6	NA	NA	
15	AC1-D	NO	95.7	28.5	NO	
16	AC1-C	NO	57.7	18.7	NO	
17	AC2-D	NO	79.4	6.6	NO	
18	AC2-C	NO	49.3	1.4	NO	
19	AC3-D	NO	66.1	-11.3	NO	
20	AC3-C	NO	42.7	-12.1	NO	
21	AC4-D	NO	82.8	11.1	NO	
22	AC4-C	NO	59.3	22.0	NO	
23	SCU-CTPH	YES	20.0	-82.1	YES	pH adjusted to 6.5; RSSCT thru 20BV using SCU media
24	SCU-CT	YES	68.0	-39.3	NO	RSSCT thru 20 BV, no pH adjust, using SCU media

TABLE 3

1.1.4.1 Technical Feasibility

It is possible to adjust the pH, but current usage of adsorbent is high. ADM is investigating reducing the dosage for nickel removal.

1.1.4.2 Capital and Operation Costs.

SCU-CTPH costs about \$500/cu ft at 45 lbs per cubic foot. Current dosages are very high. Breakthrough testing was performed and nickel broke through at 40ppb after about 400 bed volumes. Additional testing is planned to test a UF treated DAF effluent with an adsorbent column for additional capacity trials.

1.1.4.3 Reliability

The material is approved for use, but there have not yet been a sufficient number of trials to answer all regulatory questions.

1.1.5 General Electric Water

General Electric Water (GE) has evaluated nickel removal with their proprietary metal precipitant, Metclear. Metclear is the polymeric DTC chemistry GE has been using on electroplating wastes. After several trials, this process was abandoned. A 64% reduction was seen with DAF effluent to about 40 ppb. However this was achieved with strong acidification (<2 pH), two-step alkalization using lime (to pH 5.5) and then $Mg(OH)_2$ (to ~10), two-step MetClear addition to a total of 200 ppm MR2405, with 75 ppm Flocculent PolyFloc CE1163. Half the MetClear was added in Step 1, and half in Step 2. Such wide fluctuations in pH from below 2 followed by alkalization to over 10 will result in large volumes of chemical usage and does not warrant further exploration of this approach.

1.1.5.1 Technical Feasibility

Adjusting the pH to below 2 followed by alkalization to over 10 will result in such large volumes of chemical usage that further exploration of this approach is unwarranted.

1.1.5.2 Capital and Operation Costs

Heavy chemical usage.

1.1.5.3 Reliability

GE is using this chemical for inorganic metals removal. However it is not suitable for application in the Decatur ADM Facility.

1.2 Nickel- Chemical Precipitation Process Using Carbamates or Organic Sulfides-**1.2.1 Nalco- Nalmet:**

Nalco is marketing an EPA approved chemical sold under the trade name NALMET which essentially is a bonded dimethyl dithio carbamate on a polymeric (PAA) backbone. Structure of Nalmet is shown in [FIGURE 9](#).

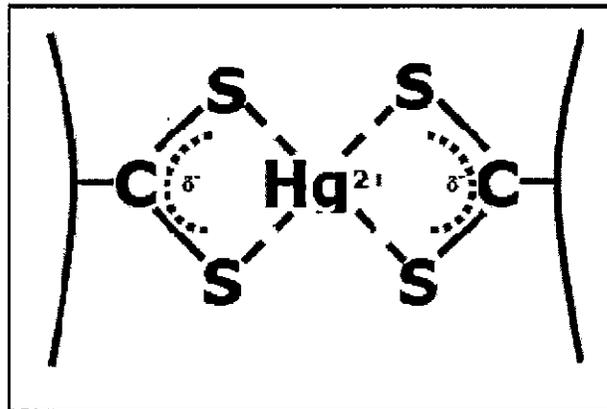


Figure 2. Metal Complex Schematic of Small Molecule and Polymeric Dithiocarbamates

FIGURE 9

Nalmet is marketed by Nalco for treatment of metal mining wastes and mercury remediation. Nalmet was laboratory tested on the six major streams that make up the feed streams to the Decatur complex discharge. The results on the DAF effluent are shown in [TABLE 4](#).

TABLE 4		
Sample Name	Nickel	Zinc
ppm Nalmet	mg/kg	mg/kg
Feed	0.104	0.02
50	0.084	0.02
100	0.083	0.02
200	0.074	0.03
300	0.067	0.02

1.2.1.1 Technical Feasibility

A multi-day sampling trial was conducted in the laboratory on DAF effluent using NALMET treatment. The results are shown in [TABLE 4](#). The average reduction in soluble nickel was about 30%. This strongly indicates NALMET is not capturing all the complexed soluble nickel (see [TABLE 1](#) for details). After a relatively quick reduction to 50 ppm, there was only a slight additional reduction in nickel with additional usage.

1.2.1.2 Capital and Operation Costs

Nalco estimated the cost for NALMET as \$1.40 per lb delivered in the ADM Facility.

1.2.1.3 Reliability

It is unlikely that treatment with Nalco's Nalmet will reduce the Nickel and Zinc concentrations to the proposed discharge limits. No additional work should be performed with Nalmet.

1.2.2 Nalco - Nalmet + Microfiltration

Nalco is running tests for ADM in which Nalmet is used to complex with the nickel followed by removal with an open pore membrane. Initial results have been promising, and ADM is pursuing additional experiments to optimize the protocol. Nickel concentrations were reduced from 98 ppb to 38 ppb using combined chemical treatment and filtration in jar testing as shown in TABLE 5. This treatment has not yet been optimized.

TABLE 5

Nalmet ppm	Nickel ppb of unfiltered and filtered samples						
	Super- natant without filtration	Pore size for filtration of supernatant					
		0.1 um	0.22 um	0.45 um	0.8 um	1.2 um	8 um
50	72	50	60	60	54	54	58
100	60	ND	ND	46	ND	ND	ND
150	52	48	46	42	46	46	50
200	46	ND	ND	40	ND	ND	ND
250	48	38	40	46	44	44	46
400	40	ND	ND	40	ND	ND	ND

1.2.2.1 Technical Feasibility

Initial experiments with a multitude of membrane pore sizes show promise in nickel reduction.

1.2.2.2 Capital and Operation Costs

Nalco estimated the cost for NALMET as \$1.40 per lb delivered. However, the work on using Nalmet in combination with a membrane is very preliminary at this stage.

1.2.2.3 Reliability

ADM needs additional data to verify reproducibility and reliability of the procedure.

1.2.3 Chemtreat:

Chemtreat has used their proprietary Carbamates and Organic Sulfides based water chemistry to treat the DAF effluent. Chemtreat has shown reductions in nickel to 37 ppm using a combination of calcium chloride (CaCl₂) with P8007L as shown in FIGURE 10 and in TABLE 6. For the trials performed by Chemtreat, the best nickel reduction used 100 ppm P8007L mixed with about 200 ppm calcium chloride. Chemtreat believes that the chelating agent binding the nickel can be freed by addition of calcium chloride which can subsequently be reacted with the binding agent.

On-site confirmation testing has not reduced the nickel concentration to the levels Chemtreat reported in their lab. Also, additional calcium ions were shown to have no benefit. Finally, ADM has concerns regarding the size of the floc and how it is removed from solution. ADM is following up on that aspect of the testing.

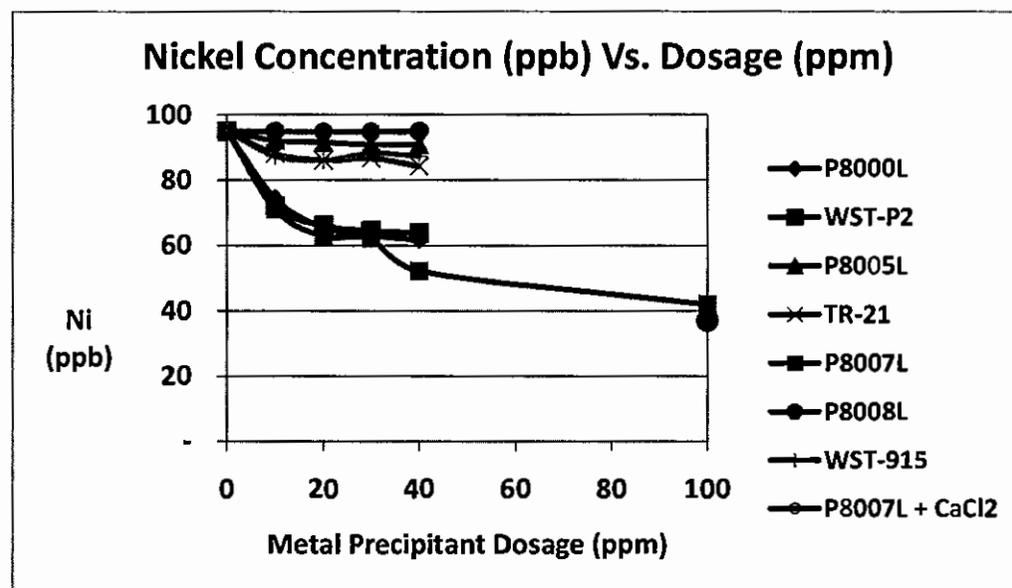


FIGURE 10

ADM has tried to reproduce Chemtreat's work independently but has had only limited success.

TABLE 6

Sample Name	Nickel mg/kg	Zinc mg/kg	
Raw DAF	0.126	0.084	
8007L-12ppm	0.101	0.099	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction
8007L-11ppm +CaCl2	0.091	0.090	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + CaCl2
8007L-11ppm +CaAcet	0.095	0.106	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + Ca Acetate

8007L-29ppm	0.090	0.089	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction
8007L-29ppm +CaCl2	0.088	0.086	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + CaCl2
8007L-32ppm +CaAcet	0.088	0.084	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + Ca Acetate
8007L-70ppm	0.081	0.061	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction
8007L-70ppm +CaCl2	0.079	0.048	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + CaCl2
8007L-72ppm +CaAcet	0.079	0.041	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + Ca Acetate
8007L-104ppm	0.077	0.049	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction
8007L-100ppm +CaCl2	0.079	0.047	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + CaCl2
8007L-98ppm +CaAcet	0.076	0.052	Chemtreat 8007-L, 2 hours and then acid kill to stop reaction + Ca Acetate

1.2.3.1 Technical Feasibility

Current treatment protocol does not require pH modification. However, the precipitated nickel is recovered through a very tight filter (0.45microns). ADM is working to set up a trial to determine the optimum dosage of Chemtreat's precipitant and a suitable recovery mechanism.

1.2.3.2 Capital and Operation Costs

Chemtreat estimates the cost for P8007L at about \$2.70/lb. Based on these costs, and work done to date, ADM estimates a chemical cost of about \$12,150/ day for the DAF effluent.

1.2.3.3 Reliability

ADM has reproduced some, but not all, of Chemtreat's work internally and expects to conduct a pilot trial with their material.

1.2.4 Hychem Chemical Company

Hychem is a water treatment company which supplies polymer to ADM. Of the many chemicals they provided to ADM for testing, two metal precipitants have worked well. The preferred chemical (DP4) is a blend of several materials which contains <30% dimethyl di-thiocarbamate(DMDTC).

1.2.4.1 Status

A number of successful bench tests have been performed. However, attempts to remove residual DMDTC have been only partially successful. Discussions are underway as to whether further work with DP4 is warranted.

1.2.4.2 Technical Feasibility

DP4 is a liquid product and is easy to apply and use. It produces a very small amount of flocculated material.

1.2.4.3 Capital and Operating Costs

Of the numerous chemicals ADM has investigated, DP4 is by far the least expensive and can operate at the lowest dose. DP4 is currently listed at \$ 0.79/lb.

1.2.4.4 Reliability

The effectiveness of the chemical is predictable and does not appear to be affected by normal changes in the effluent from ADM's wastewater treatment plant. The toxicity of DMDTC and the DTC is of particular concern as is its affect on the treatment process used by the Decatur Sanitary District. Field trials would need to be conducted to identify the lowest dose vs. reaction time for the material. From these field trials, one can determine the residual DTC that would be expected from the process. Proper controls could be implemented to reduce the possibility of a DMDTC overdose. For example, ferrous sulfate will tie up one-half to two-thirds of the residual DTC. However, usage rates and sludge production using this scenario would be very high.

Studies have not been done to determine if the reaction is quenched or to determine actual reaction times for the DP4 product and the ADM effluent. Typical concentration of soluble nickel when using DP4 is 0.038 – 0.045ppm.

1.2.5 GE Betz DTC

ADM has tested DTC from GE Betz using the Decatur plant DAF effluent as the substrate. However, even at dosages up to 100 ppm, there was only about a 30% reduction in soluble nickel as shown in [FIGURE 10](#). Based these results, no additional work using DTC from GE Betz is planned.

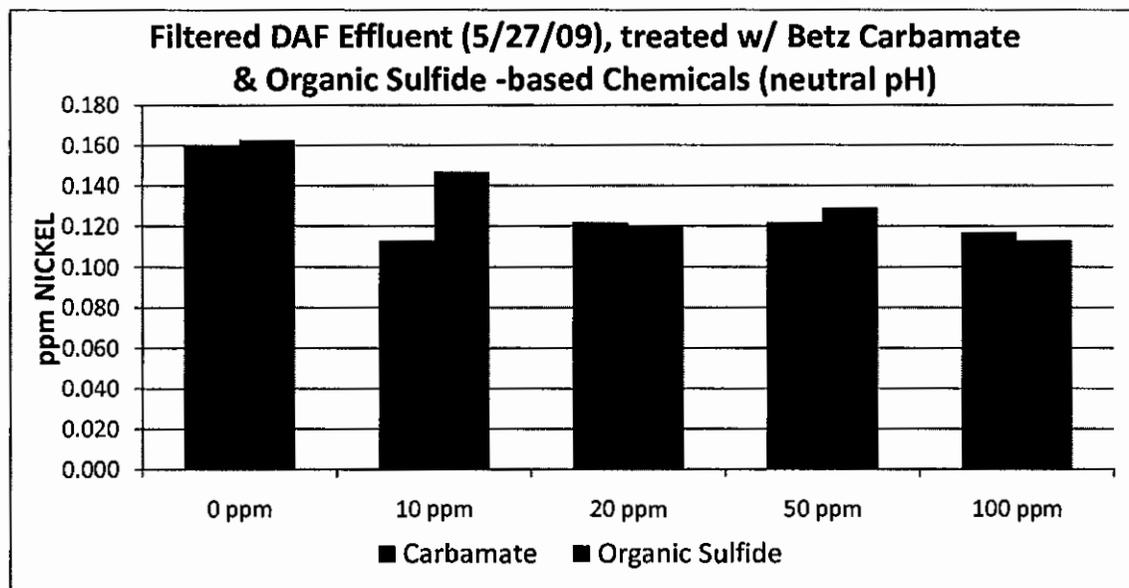


FIGURE 10

1.2.5.1 Technical Feasibility

Current treatment protocol using GE Betz's DTC does not require pH modification. However, there is insufficient reduction in soluble nickel in the effluent samples tested. ADM does not believe this approach is feasible.

1.2.5.2 Capital and Operation Costs

Since the GE Betz technology is not effective at reducing the soluble nickel to the proposed limits in the wastewater effluent, cost data was not obtained.

1.2.5.3 Reliability

ADM facilities have used GE extensively for treatment of wastewater.

1.2.6 Hydrite

ADM has tested Hydrite Chemicals' DTC product on the DAF effluent and DAF influent streams from the Decatur wastewater facility. One Hydrite product (1740) showed reduction in soluble nickel at a 20 ppm dosage as shown in TABLE 7 and TABLE 8.

	Nickel	Zinc
	mg/kg	mg/kg
DAF effluent as is	0.098	0.05
1740 10 ppm	0.043	0.02
1740 20 ppm	0.040	0.02
1750 10 ppm	0.097	0.03
1750 20 ppm	0.099	0.02
1753 10 ppm	0.103	0.03
1753 20 ppm	0.100	0.03
1754 10 ppm	0.104	0.02
1754 20 ppm	0.099	0.02

	Nickel	Zinc
	mg/kg	mg/kg
DAF influent as is	0.100	0.03
1740 35 ppm	0.074	0.17
1740 70 ppm	0.040	0.03
1750 35 ppm	0.108	0.07
1750 70 ppm	0.099	0.02
1753 35 ppm	0.110	0.07
1753 70 ppm	0.104	0.03

1754 35 ppm	0.110	0.06
1754 70 ppm	0.102	0.03

1.2.6.1 Technical Feasibility

The product was able to reduce soluble Nickel in the effluent from the ADM Decatur facility. However, Hydrite's permission is required to perform a pilot scale test.

1.2.6.2 Capital and Operation Costs

Operational cost information will be collected.

1.2.6.3 Reliability

ADM is planning to perform additional work to determine the suitability of this application.

1.3 Nickel- Ion Exchange Resin

1.3.1 Dowex Optipore SD-2

Another methodology that has been investigated is the use of a decolorizing resin from Dow and its ability to adsorb nickel and zinc. Optipore SD 2 has been tested on both the DAF effluent and influent with positive results. However, a very large dosage is required to achieve the desired reduction. The results can be seen in FIGURE 11, FIGURE 12, and FIGURE 13. Based on preliminary discussions with the Dow Engineering team and with a 3 Bed Volume / hr cycle time, this corresponds to a two column design with about 1500 gallons of resin in each column.

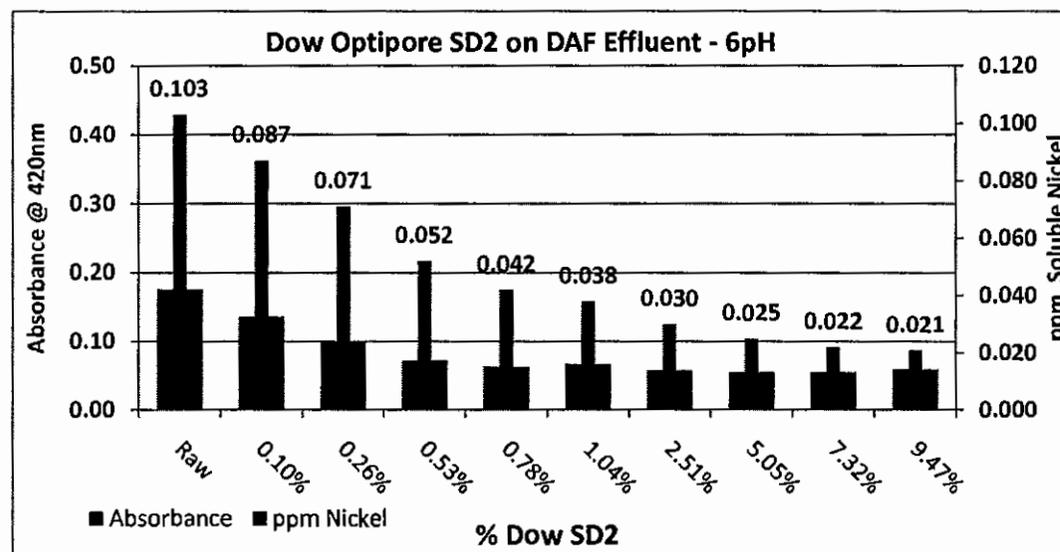


FIGURE 11

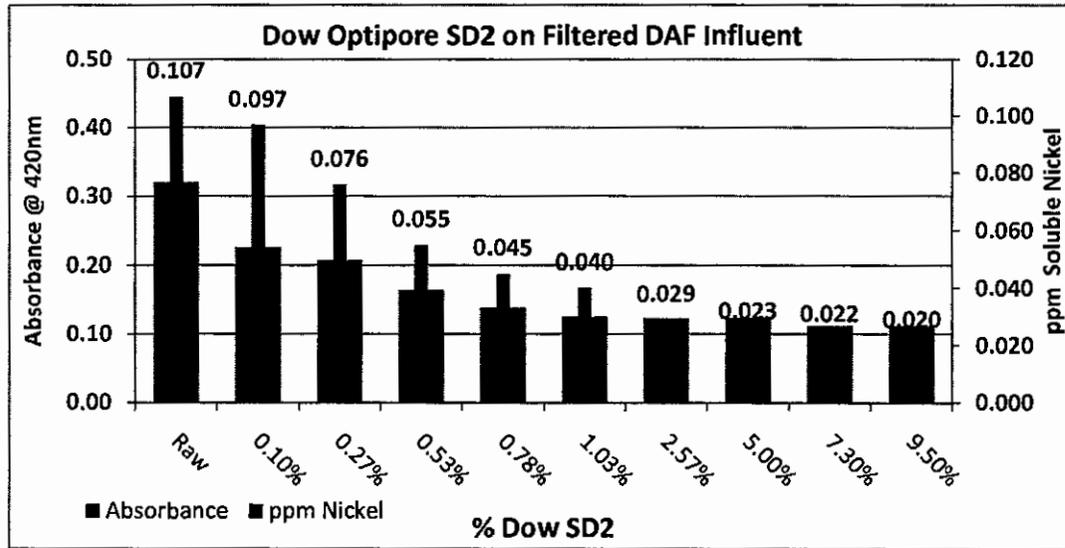


FIGURE 12

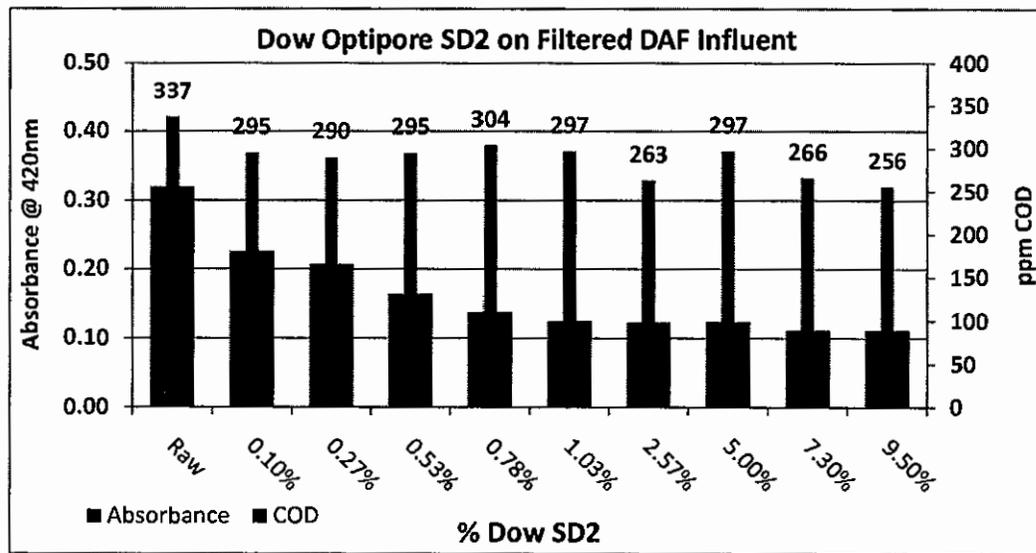


FIGURE 13

1.3.1.1 Technical Feasibility

Based on studies to date, dosages for Optipore SD-2 on ADM's wastewater are very high. Dow is recommending a hot caustic and/or ethanol regeneration of the resin, but that would be very difficult to operate.

1.3.1.2 Capital and Operation Costs

SD-2 is currently listed at \$300.00/cu ft. Based on the dosages determined in the investigation, the cost for resin, two large packed beds and regeneration equipment was preliminarily estimated at between \$8,000,000 to \$10,000,000.

1.3.1.3 Reliability

As with most resin processes, the resin will lose adsorbent capacity as it is used. Cycle testing will have to be conducted to determine its optimum life.

1.3.2 Vivenano- IX Nanoparticles.

A startup company based in Toronto has developed negatively charged 3-5 nm core with 2-5 nm polymer shell. The core can be used to immobilize ions inside polymer and can produce metal, metal oxide, mixed metal oxides, doped systems, etc. This concept is represented in [FIGURE 14](#).

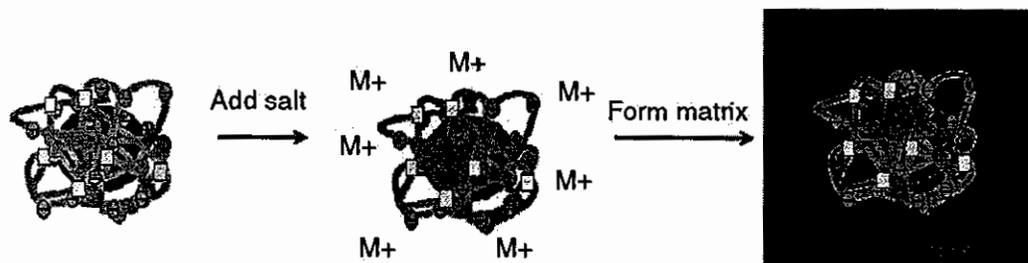


FIGURE 14

1.3.2.1 Status

ADM has tested Vivenano's product on simulated waste streams using inorganic metal salts and a complete removal of nickel was seen. However, when tested with actual DAF effluent from the Decatur Plant, no nickel adsorption was detected.

1.3.2.2 Technical Feasibility

Ion Exchange Nanoparticles do not work for reducing soluble nickel from the Decatur Facility effluent.

1.3.2.3 Capital and Operation Costs

This was not calculated since the technology does not achieve the target nickel concentration in the effluent.

1.3.2.4 Reliability

Laboratory tests with inorganic metal spiked water samples found that Vivenano's IX Nanoparticle can reduce the nickel concentration significantly. Further experiments using ADM's actual effluent did not result in a similar reduction.

Vivenano has agreed to ship a new slurry material to ADM's Decatur facility for testing. Barring dramatically better results, this technology will be abandoned.

It is highly unlikely that any adsorbent process will be economically feasible. This is due to the fact that as much as 100+ ppm of BOD (organic material) must be adsorbed to remove the 0.10 ppm of chelated nickel.

1.4 Nickel and Zinc- Soybean Process Stream Alternative.

ADM continues to evaluate this stream for alternative treatments and uses.

1.5 Nickel and Zinc- BioProducts Process Stream Alternative

Initially, ADM believed there was a high nickel concentration in this internal Bioproducts stream. However, the levels subsequently measured have not been elevated. (This is not the stream identified in TABLE 1.) Preventing this stream from entering the ADM Decatur wastewater treatment facility would reduce influent nickel only slightly, perhaps only 3% to 5%.

1.6 Nickel and Zinc- WWTP Sludge Removal System

ADM has investigated the removal of wastewater treatment plant sludge and believes a process whereby the material is centrifuged followed by sludge drying could possibly be feasible. The dried sludge would be disposed of either by incineration or landfilling, depending on environmental permitting. Preliminary testing has been performed and the data is being reviewed.

1.7 Nickel and Zinc- Reverse Osmosis

A plant trial is being developed for Ultrafiltration/Reverse Osmosis (UF/RO) and UF/Nanofiltration (UF/NF) treatment of high salt wastewaters. While it has been successful in lab trials, ADM has significant concerns regarding the handling of the concentrated stream from the membrane process. One possibility is to evaporate the rejected stream, crystallize it and then dry the salts. However, depending on the size of the stream to be evaporated, the cost to remove the water maybe prohibitive.

Trials were run with two nano filtration membranes (DL supplied by GE and SG) and one low pressure RO membrane (AK, supplied by GE). 100% nickel removal was seen in all trials. However, permeate recovery was low (30%) due a limitation of the equipment. ADM has solicited and received quotations from GE, Siemens, Nalco and Separation Engineering for a 100-200 gpm pilot scale trial to be run at the Decatur wastewater treatment facility. ADM is currently attempting to identify the optimum membrane combination for running the pilot trials.

1.8 Nickel and Zinc- Sludge (WWTP organism cell wall rupture).

This is a pulsating electric field that ruptures the cell walls of the bacteria. This is based on the concept that the filaments that previously caused operational difficulty were aerobic and being constantly seeded from the aeration system. The process tested actually adds BOD to the anaerobic wastewater treatment plant reactors and creates more biogas.

1.9 Nickel and Zinc- Sludge Purchase :

ADM has provided samples of the sludge to a fish food company as a possible protein source on a new product. At present other sources are being evaluated, and sludge from the Decatur wastewater treatment plant is not slated to be used.

2 Other Approaches

2.1 Procorp

Procorp has a hardness removal system that has been tried on ADM facility's cooling tower water. It requires pH adjustment to over 8.5 and in best case just a 20% reduction in soluble nickel was seen as illustrated in TABLE 9.

Experiment #	Date	Time	Elapsed Time(h)	pH	[Nickel] due Dilution	% red in [Nickel]	Comments
1	3/16/2010	9:45	0.42	8.63	48.417	5.0%	Recirculation Mode
2	3/17/2010	10:35	0.42	8.74	49.296	20.9%	One Pass Treatment
3	3/18/2010	9:50	0.50	8.59	49.151	2.3%	One Pass Treatment w Ca supplement

CONCLUSIONS: Further work is needed to prove the technology for this application and to develop the economics.

2.2 KML / SPS

KML has reported numerous successful tests at an ADM Soy facility but this is without confirmation work by ADM. Testing on the DAF effluent started in the summer of 2009. Through a long series of tests and two extended on-site trials, KML has not been able to show consistent removal of soluble nickel as demonstrated in TABLE 10. Additionally, KML has been unwilling to allow ADM to perform its own independent testing with their materials or to observe KML while it performed the nickel reduction process. Finally, KML believes that sludge must be present for their chemistry to function, which causes a number of operational problems. Since ADM has not been able to verify or observe KML's process, it has ceased working with them.

Week -1	Al	P	S	Zinc	Nickel	Fe	Mg	Ca	Chloride	COD
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	ppm
Raw DAF Influent Total:	0.63	465.2	844	0.44	0.767	1.13	675	333	8,617	762
DAF Effluent (primary) Total:	7.17	250.3	1,230	0.27	0.729	1.35	658	305	8,949	970
Metal Treatment Total:	30.55	57.5	1,427	0.25	0.375	0.48	499	175	9,978	1,038
% In/Decrease:	-4726%	88%	-69%	42%	51%	58%	26%	48%	-16%	-36%

Week -2	Al	P	S	Zinc	Nickel	Fe	Mg	Ca	Chloride	COD
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	ppm
Raw DAF Influent Total:	1.04	555.8	1,236	0.86	1.24	1.82	811	395	11,572	3,074
DAF Effluent (primary) Total:	10.09	189	1,597	0.61	1.119	1.69	845	520	13,459	4,966
Metal Treatment Total:	14.71	5.4	1,832	0.68	0.517	0.58	769	389	16,990	5,296
% In/Decrease:	-1320%	99%	-48%	21%	58%	68%	5%	2%	-47%	-72%

2.3 Alysok Chemicals

This company has experience in metal finishing and electronics facilities. Their bench work at the ADM Decatur site proved ineffective at nickel removal. However, they have a relationship with the main manufacturer of metal precipitants in the U.S. Alysok is working out details whereby the chemical manufacturer would perform research for ADM with the goal of finding a lower toxicity precipitant that yields low nickel at reasonable doses.

2.4 Captive Deionization (CDT)

At the suggestion of the Decatur Sanitary District, ADM had discussions with Dr. Michael Karpuk, President of TDA Research in Golden, CO (karpuk@tda.com) to understand the potential and applications of the CDT technology. CDT works using electrodes from carbon aerogels which, when placed under an electric charge, bind charged ions and remove them from the contaminated water source. TDA indicated that they have licensed the technology to two separate companies (CDT systems in the United States and an unnamed licensee in Japan). However, CDT Systems is no longer in existence in the United States. During discussions with their CEO, John Davies, (972) 974-3667 (jddvrd@gmail.com) ADM learned that the technology was never scaled up beyond bench scale and that the company has been placed under receivership. There are three main challenges with captive deionization:

1. Lack of pilot scale or commercial scale supplier. To date, no companies have manufactured the electrodes.
2. Lack of selectivity. CDT will pick up all charged species in the water stream not just nickel and zinc. Consequently, when applied to ADM's high salt waste water stream, it will remove the bulk of the salts (about 3,000 ppm TDS) and this would entail evaporate a wastewater stream generating over 100 lbs of salt waste per day.
3. Electrode adsorption. The CDT electrodes are essentially activated carbons with charge groups on them. ADM believes that the presence of BOD/COD and color components in the wastewater stream will compete with the charged species for binding on the electrode and negate any benefit of using them.

CONCLUSIONS: This technology seems ill-suited for application in a complicated matrix such as wastewater treatment.

2.5 Ferric Salt Precipitation

Ferric salts have long been recognized as an effective scavenger of heavy metals. In literature, extensive treatment has been given to the application and underlying removal mechanism. It has also been recognized that metals, primarily ferric, hydroxide and oxide coatings in the soil and sediments, play an important role in the transport, biotransformation and ultimate fate of trace constituents in natural systems. At neutral to alkaline pH, ferric salts precipitate as amorphous hydrated oxide or oxy-hydroxide, which has relatively stable and reproducible surface properties. Upon aging, the precipitate transforms gradually into a crystalline iron oxide (goethite) form. However, its absorptive properties remain quite similar. The ability of the ferric hydroxide precipitate to absorb ions with heavy metals is characterized in single and multi-adsorbate systems. Heavy metals could be absorbed both as cations (Cr^{+3} , Pb^+ , Cu^{+2} , Zinc^{+2} , Ni^{+2} , Cd^{+2}) in neutral to high pH, and as anions (SeO_4^{-2} , CrO_4^{-2} , $\text{VO}_3(\text{OH})^{-2}$, AsO_4^{-3}) in neutral to mildly acidic pH.

ADM has had discussions with two separate companies, Entex Inc. (Richard Pehrson dick.pehrson@entexinc.com) and Joe Zuback (jzuback@globalwateradvisors.com) on the use of Ferric salts for nickel/zinc removal. Literature reports demonstrate high nickel/zinc reductions using ferric chloride. In such an application the iron is bound by the sludge/waste solids while the chloride goes out with the waste water stream. Due to ADM's proposed discharge limit of 660 ppm chloride, using ferric chloride to reduce the nickel and zinc is not an option. However, it has been mentioned that ferric sulfate would be equally effective.

Unfortunately, ADM has received two separate opinions on its efficacy. Enetex suggested a near neutral pH would be adequate for such removal, while Joe Zuback felt a higher pH was desired. A solubility chart for Nickel precipitation is shown in FIGURE 15 and it appears that the entire waste stream needs to have a pH>10.0 for complete nickel removal.

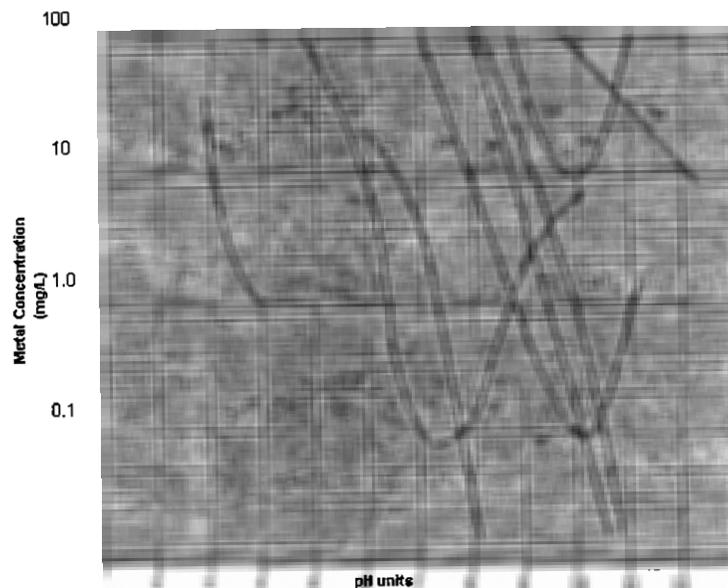


FIGURE 9

An additional problem when using this technology to remove the nickel and zinc is that using a 3,000 mg/l ferric salt dose generates a large volume of sludge, which is reportedly between 10 to 15% of the wastewater volume. With a discharge limit on sludge disposal this is a potentially significant problem.

Using ferric salts to reduce the nickel and zinc in waste water, while technically feasible, is very impractical. Additionally, this will greatly increase the amount of solids leaving the plant. Further, bench testing has shown typical inorganic coagulants to be ineffective on organic nickel using a 2-pass treatment @ 1000ppm each pass as shown in TABLE 11.

TABLE 11	
Sample Name	Nickel
	mg/kg
2208 4/20 DAF inf raw	0.127
2208 DAF Inf 1X Alum6	0.102
2208 DAF Inf 2X Alum6	0.076
2208 DAF Inf 1X Ferrous Sulfate @ 6pH	0.099
2208 DAF Inf 2X Ferrous Sulfate @ 6pH	0.074
2208 DAF Inf 1X Sodium Aluminate @ 6pH	0.096
2208 DAF Inf 2X Sodium Aluminate @ 6pH	0.084

2.6 Metallothionein (MT)

Metallothionein is a family of cysteine-rich, low molecular weight (MW ranging from 3500 to 14000 Da) proteins. MTs have the capacity to bind both physiological (such as zinc, copper, selenium) and xenobiotic (such as cadmium, mercury, silver, arsenic) heavy metals through the thiol group of its cysteine residues, which represents nearly the 30% of its amino acidic residues. Roger Acey, professor at California State University, Long Beach (racey@csulb.edu) is working on metallothionein based removal of nickel and zinc and has seen good results. However, the technology is still at the bench scale. They have one issued patent (US Patent 6,750,056, 6/15/04): Metal Binding Proteins & Associated Methods. ADM has requested a sample request but has not heard back on any assays.

CONCLUSION: Promising technology; however, a significant amount of laboratory work needs to be completed to fully evaluate it.

2.7 Other contacts / Approaches.

In addition to the routes explored above, ADM Research has had preliminary discussions with several other companies

- 2.7.1 **Hard Hat Inc.-** Suggested they might be able to modify the Decatur Plant's anaerobic treatment to increase nickel sequestration. However, Hard Hat has not made any progress
- 2.7.2 **Veolia ES-** Veolia believed they had a better version of DTC (see item 1.2 above). Since there are a number of companies willing to supply this chemical, ADM did not pursue this.
- 2.7.3 **Bioactive Peptides-** Working with a professor at Iowa State University to develop phage peptide to capture nickel ion specifically. Preliminary bench scale results appear promising; however, further development is required.
- 2.7.4 **Nickel / Zinc Gluconate Manufacturers:** ADM Research contacted manufacturers of nickel/zinc gluconate to inquire how they managed waste water treatment. Unfortunately, the only suppliers found were in India (2) and China (1). The company in India did not seem to be worried about discharge standards, so no progress was made.
- 2.7.5 **Electro-Coagulation (EC):** ADM Corn worked w/two different EC manufacturers: GlobalSep and Kaselco. GlobalSep actually showed a nickel increase due to their electrode construction. Kaselco had more expertise and tried pH reduction followed by CO₂ removal prior to EC. The EC treatment then caused a pH increase. Little removal was shown.
- 2.7.6 **Advanced Oxidation Process (AOP):** This process uses combinations of oxidizers (*i.e.*: Hydrogen peroxide and ozone) to effect the breakdown of soluble organic compounds. To get the full benefit of the peroxide, a pH of 10 is necessary. Testing at the manufacturer's site did show nickel coming out of solution as the oxidation took place. However, the amount of base and oxidizers needed to treat 6,000,000 gallons per day is cost prohibitive.
- 2.7.7 **Fermentation of Soy Solubles:** ADM Research is also investigating fermentation of soy soluble stream for ethanol production to prevent the stream from entering the waste treatment facility. Preliminary results indicate that sugars in the soluble stream can be fermented with commercially available yeasts and enzymes.
- 2.7.8 **Ion Exchange Resins, Chelating:** A number of conversations took place between Dow & ADM concerning chelating resins. The following list, when taken as a whole, explains why testing was not pursued:

- High BOD influent streams present resin fouling and pre-filtering issues.
- Chelating resins have a narrow pH operating range (in the 4s) requiring a major pre-ion exchange decrease and post-ion exchange increase.
- The hydrogen ion and sodium ion form resin would remove calcium & magnesium, which uses up ion exchange capacity and lowers hardness.
- The calcium ion form resin has a very narrow pH operating range (+/-0.15 to +/-0.25) and will release nickel if operated out of that range.
- Hydrochloric acid cannot be used as a regenerant (effluent chloride levels) and sulfuric acid is problematic due to calcium sulphate precipitation in the bed.
- Resin fouling is also expected when treating effluent, resulting in caustic use for clean-ups.
- An acid, nickel-containing, regeneration stream would have to be further treated by a concentration process and then some type of disposal.
- Capital expenditure would be very high.

Nickel and Zinc Soluble and Total Study

Soluble Metal Removal

Dissolved Nickel						
	Primary	% Removal	Final	% Removal	Total	
	Influent	Effluent	by	Effluent	by	%
Sample Date	mg/L	mg/L	Primary	mg/L	Post Primary	Removal
9/28/2009	0.0392	0.0295	24.7	0.0298	-1.0	24.0
9/29/2009	0.0367	0.0302	17.7	A		
9/30/2009	0.0360	0.0293	18.6	0.0289	1.4	19.7
10/1/2009	0.0276	0.0238	13.8	0.0287	-20.6	-4.0
Dissolved Zinc						
	Primary	% Removal	Final	% Removal	Total	
	Influent	Effluent	by	Effluent	by	%
Sample Date	mg/L	mg/L	Primary	mg/L	Post Primary	Removal
9/28/2009	0.0283	0.0263	7.1	0.0397	-51.0	-40.3
9/29/2009	0.0228	0.0385	-68.9	A		
9/30/2009	0.0469	0.0285	39.2	0.0408	-43.2	13.0
10/1/2009	0.0310	0.0272	12.3	0.0348	-27.9	-12.3

A indicates analysis problem;
suspected unwashed filter

Nickel and Zinc Soluble and Total Study

Soluble Metal Removal

Exhibit E

							Dissolved Nickel	
		Primary	% Removal	Final	% Removal	Total		
	Influent	Effluent	by	Effluent	by	%		
Sample Date	mg/L	mg/L	Primary	mg/L	Post Primary	Removal		
9/28/2009	0.0392	0.0295	24.7	0.0298	-1.0	24.0		
9/29/2009	0.0367	0.0302	17.7	A			A indicates analysis problem;	
9/30/2009	0.0360	0.0293	18.6	0.0289	1.4	19.7	suspected unwashed filter	
10/1/2009	0.0276	0.0238	13.8	0.0287	-20.6	-4.0		
							Dissolved Zinc	
		Primary	% Removal	Final	% Removal	Total		
	Influent	Effluent	by	Effluent	by	%		
Sample Date	mg/L	mg/L	Primary	mg/L	Post Primary	Removal		
9/28/2009	0.0283	0.0263	7.1	0.0397	-51.0	-40.3		
9/29/2009	0.0228	0.0385	-68.9	A				
9/30/2009	0.0469	0.0285	39.2	0.0408	-43.2	13.0		
10/1/2009	0.0310	0.0272	12.3	0.0348	-27.9	-12.3		

Nickel and Zinc Soluble and Total Study

Total Metal Removal

			Total Nickel			
	Influent	Primary Effluent	% Removal by Primary	Final Effluent	% Removal by Post Primary	Total Removal %
Sample Date	mg/L	mg/L	Primary	mg/L	Post Primary	Removal
9/28/2009	0.0564	0.0322	42.9	0.0277	14.0	50.9
9/29/2009	0.0619	0.0359	42.0	0.0282	21.4	54.4
9/30/2009	0.0594	0.0351	40.9	0.0294	16.2	50.5
10/1/2009	0.0478	0.0286	40.2	0.0285	0.3	40.4
			Total Zinc			
	Influent	Primary Effluent	% Removal by Primary	Final Effluent	% Removal by Post Primary	Total Removal %
Sample Date	mg/L	mg/L	Primary	mg/L	Post Primary	Removal
9/28/2009	0.242	0.0749	69.0	0.0428	42.9	82.3
9/29/2009	0.260	0.0915	64.8	0.0415	54.6	84.0
9/30/2009	0.262	0.0823	68.6	0.0421	48.8	83.9
10/1/2009	0.269	0.0909	66.2	0.0385	57.6	85.7