EcoCAT
Ecological Compliance Assessment Tool

Applicant:	Ethan Pressly	*IDNR Project Number:*	1412602
Contact:	Ethan Pressly	*Date:*	06/23/2014
Address:	3150 Roland Ave. Springfield, IL 69705	*Alternate Number:*	1213505

Project: Sanitary District of Decatur - Site Specific Rules
Address: 501 Dipper Lane, Decatur

Description: The Sanitary District of Decatur is seeking a site specific rules for its discharges of nickel into the Sangamon River.

Natural Resource Review Results

This project was submitted for information only. It is not a consultation under Part 1075.

The Illinois Natural Heritage Database contains no record of State-listed threatened or endangered species, Illinois Natural Area Inventory sites, dedicated Illinois Nature Preserves, or registered Land and Water Reserves in the vicinity of the project location.

Location

The applicant is responsible for the accuracy of the location submitted for the project.

County: Macon

Township, Range, Section:
16N, 2E, 17
16N, 2E, 20

IL Department of Natural Resources Contact
Impact Assessment Section
217-785-5500
Division of Ecosystems & Environment

Disclaimer

The Illinois Natural Heritage Database cannot provide a conclusive statement on the presence, absence, or condition of natural resources in Illinois. This review reflects the information existing in the Database at the time of this inquiry, and should not be regarded as a final statement on the site being considered, nor should it be a substitute for detailed site surveys or field surveys required for environmental assessments. If additional protected resources are encountered during the project's implementation, compliance with applicable statutes and regulations is required.

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1. The IDNR EcoCAT website was developed so that units of local government, state agencies and the public could request information or begin natural resource consultations on-line for the Illinois Endangered Species Protection Act, Illinois Natural Areas Preservation Act, and Illinois Interagency Wetland Policy Act. EcoCAT uses databases, Geographic Information System mapping, and a set of programmed decision rules to determine if proposed actions are in the vicinity of protected natural resources. By indicating your agreement to the Terms of Use for this application, you warrant that you will not use this web site for any other purpose.

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3. IDNR reserves the right to enhance, modify, alter, or suspend the website at any time without notice, or to terminate or restrict access.

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Exhibit 30

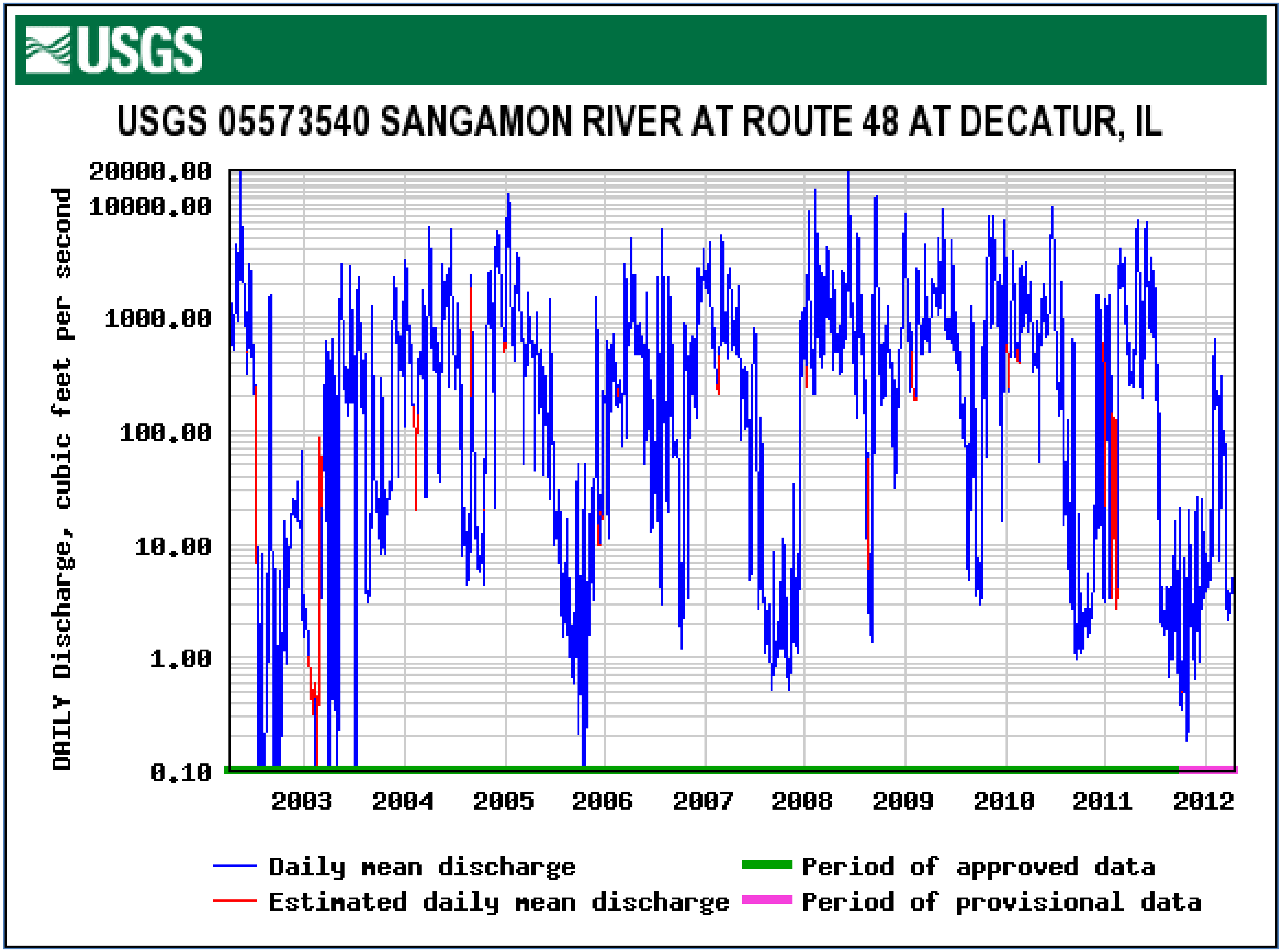

USGS
USGS 05573540 SANGAMON RIVER AT ROUTE 48 AT DECATUR, IL
DAILY Discharge, cubic feet per second
20000.00
10000.00
1000.00
100.00
10.00
1.00
0.10
2003
2004
2005
2006
2007
2008
2009
2010
2011
2012
Daily mean discharge
Estimated daily mean discharge
Period of approved data
Period of provisional data

Exhibit 31

Table 3: Summary of Technologies Reviewed by ADM Under Variance Granted by Board

[redacted]	***Nickel Capture Method***	***Concentration of Active Dosage***	***Nickel reduction***	***Pilot Status***	***Nitratox / Respirometer Testing[1]***	***Technically Feasible[2]***	***Economically Reasonable[3]***
A - Nickel Proprietary Precipitation Process							
[redacted]	Activated Clay	1%-3%	40%-60% (0.2 mg/L influent)	Not Piloted	Not tested.	No	N/A
[redacted]	Acidic Clay	4%-8% wt/wt	40% (0.09 mg/L influent)	Not Piloted	Not tested.	No	N/A
[redacted]	Chitosan Based	5% wt/wt	90% (0.2 mg/L influent)	Not Piloted	Not tested.	No	N/A
[redacted]	Proprietary	2% wt/wt	82% (0.1 mg/L influent)	Not Piloted	Not tested.	No	N/A
[redacted]	Proprietary	200 mg/L	64% (0.12 mg/L influent)	Not Piloted	Not tested.	No	N/A
[redacted]	Not disclosed	Not disclosed	40-60% (0.2 mg/L influent)	Not Piloted	Not tested.	No	N/A
B - Nickel Chemical Precipitation Process Using Carbamates or Organic Sulfides							
[redacted]	Polymeric Dimethyl Dithiocarbamate	100 mg/L with 50mg/L of CaCl2	30% (0.15 mg/L influent)	Piloted. Total Nickel reduction to 0.06 mg/L.	Passed	No	N/A
[redacted]	Polymeric Dimethyl Dithiocarbamate	20-50 mg/L	60% (0.15 mg/L influent)	Piloted. Total Nickel reduction to 0.054 mg/L.	Passed	No	N/A

[1] ADM has been working with Riverbend Laboratories in St. Charles, Missouri, to perform respirometer and nitratox testing on various chemistries using MLSS from the District. Such testing is necessary to determine whether the treated effluent is compatible with the District's treatment processes.

[2] For purposes of this Petition, "Technically Feasible" means ADM's confirmation that the specific technology evaluated will consistently meet: (a) the nickel limit in the District's current NPDES permit; and/or (b) the proposed nickel limit that would apply to ADM based upon the District's current NPDES permit.

[3] For purposes of this Petition, "Economically Reasonable" means that the capital and operating costs associated with implementing a specific technology are objectively reasonable. Where ADM determined that a specific technology was not "Technically Feasible," it did not conduct a comprehensive review of whether that technology was also "Economically Reasonable" as indicated by "N/A."

[redacted]	*Nickel Capture Method*	*Concentration of Active Dosage*	*Nickel reduction*	*Pilot Status*	*Nitratox / Respirometer Testing*	*Technically Feasible*	*Economically Reasonable*
[redacted]	Polymeric Dimethyl Dithiocarbamate	100 mg/L	41% (0.15 mg/L influent)	Piloted. Total Nickel reduction to 0.032 mg/L	Passed	Yes[4]	No
[redacted]	Dimethyl Dithiocarbamate	50 mg/L + pH 6.0	76% (0.15 mg/L influent)	Piloted. Nickel reduction seen to 0.040 mg/L	Passed	No	N/A
[redacted]	Polymeric Dimethyl Dithiocarbamate	300 mg/L + pH swing	30% (0.15 mg/L influent)	Not Piloted	Not tested.	No	N/A
[redacted]	Polymeric Dimethyl Dithiocarbamate	50 mg/L	48% (0.10 mg/L influent)	Piloted. Nickel reduction seen to 0.020 mg/L	Passed	Yes[5]	No
[redacted]	Polymeric Dimethyl Dithiocarbamate	200 mg/L	52% (0.15 mg/L influent)	Piloted. Nickel reduction seen to 0.039 mg/L	Passed	No	N/A
[redacted]	Polymeric Dimethyl Dithiocarbamate	100 mg/L	40% (0.15 mg/L influent)	Not Piloted.	Not tested.	No	N/A
[redacted]	Dimethyl Dithiocarbamate	100 mg/L	60% (0.15 mg/L influent)	Piloted. Nickel reduction seen to 0.024 mg/L	Passed	No	N/A
C - Reuse of Ion Exchange Resin							
[redacted]	Sulfonic	0.1-0.5% wt/wt	Complete removal of Ionic Nickel from the Sorbitol plant waste	Installed at Sorbitol Plant	Not required.	Yes	Yes

[4] Testing on 100 gallon pilot reactor showed total nickel reduction to below 0.037 mg/L. However, reductions were not consistently seen with variation in influent nickel levels.
[5] Testing on 100 gallon pilot reactor showed total nickel reduction to below 0.037 mg/L. However, reductions were not consistently seen with variation in influent nickel levels.

[redacted]	*Nickel Capture Method*	*Concentration of Active Dosage*	*Nickel reduction*	*Pilot Status*	*Nitratox / Respirometer Testing*	*Technically Feasible*	*Economically Reasonable*
D -Nickel and Zinc – Soybean Process Stream Alternative.							
[redacted]	Evaporation and sale of Soy Molasses	N/A	Complete	In planning stages	Not required	Yes	Yes
E - Nickel and Zinc – BioProducts Process Stream Alternative.							
[redacted]	Identified as not a significant source of Nickel	N/A	N/A	Not Piloted	Not required	No	N/A
F - Nickel and Zinc – WWTP Sludge Removal System.							
[redacted]	Centrifuges	N/A	Complete	Not Piloted	Not required	Not determined	No
G - Nickel and Zinc – Reverse Osmosis							
[redacted]	Phosphate precipitation + Reverse Osmosis	80% recovery of feed	95%+ reduction	Not Piloted	Not tested	No	N/A
	Low pressure Reverse Osmosis	30% recovery of feed	80% + reduction	Not Piloted	Not tested	No	N/A
	Sand Filter	Not disclosed	20% reduction	Not Piloted	Not tested	No	N/A

[redacted]	*Nickel Capture Method*	*Concentration of Active Dosage*	*Nickel reduction*	*Pilot Status*	*Nitratox / Respirometer Testing*	*Technically Feasible*	*Economically Reasonable*
H - Nickel and Zinc – Sludge							
[redacted]	High voltage Pulsating Electric field	N/A	N/A	Not effective	Not tested	No	N/A
I - Nickel and Zinc – Sludge Purchase							
[redacted]	Sale to fish food company	Not viable	N/A	No customers	Not tested	No	N/A
J - Electro-Chemical Decomposition and Capacitive Deionization							
[redacted]	Carbon Aerogels	Not tested	Not tested	Not effective	Not tested	No	N/A
[redacted]	Electrochemical	Not disclosed	Higher Nickel due to leaching from electrode plates	Not Piloted	Not tested	No	N/A
K - Other Approaches							
[redacted]	Ferric Chloride	100 mg/L	40%	Not Piloted	Not tested	No	N/A
[redacted]	Protein	not tested	Not tested	Not Piloted	Not tested	No	N/A
[redacted]	Hydrogen Peroxide and Ozone	5% wt/wt + pH adjustment	20% (0.15 mg/L influent)	Not Piloted	Not tested	No	N/A

[redacted]	***Nickel Capture Method***	***Concentration of Active Dosage***	***Nickel reduction***	***Pilot Status***	***Nitratox / Respirometer Testing***	***Technically Feasible***	***Economically Reasonable***
[redacted]	Protein based	Not disclosed	Not tested	Not Piloted	Not tested	No	N/A
[redacted]	pH >11.0	1-2% wt/wt	Complete	Being piloted at Polyols Plant for waste stream	Not tested	Yes[6]	Yes
L- Non-functional Resins							
[redacted]	Styrene Divinyl Benzene	2-5% wt/wt	20% (0.15 mg/L influent)	Not Piloted	Not tested	No	N/A
[redacted]	Styrene Divinyl Benzene	4% wt/wt	60% (0.15 mg/L influent)	Not Piloted	Not tested	No	N/A
[redacted]	Immobilized Ion Exchange Beads	5% wt/wt	Not significant	Not Piloted	Not tested	No	N/A

[6] Suitable for <~50,000 GPD, non-grain based wastewater with non-chelated, salt-form nickel such as Polyols Plant IX regen waste.

Exhibit 32

Table 4: Technical Challenges on Scale Up for Nickel Remediation Chemistries

[redacted]	***Vendor not cooperative with samples***	***Assessed and determined not effective***	***Not commercially available***	***High Dosages required***	***Results not scalable beyond bench scale***	***Low recoveries and brine disposal concerns***	***Technically Feasible (y/n)***	***Comments***
[redacted]	X		X				No	
[redacted]		X		X			No	Would require 5 million pounds of additive per day
[redacted]			X	X			No	
[redacted]	X			X			No	
[redacted]				X			No	Requires a pH to <2 then to pH 5.5 then to pH 10
[redacted]	X						No	
[redacted]					X		No	Plant pilot trial did not achieve required Nickel reduction.
[redacted]		X			X		No	Plant pilot trial did not achieve required Nickel reduction.
[redacted]					X		No	Plant pilot trial did not achieve required Nickel reduction.
[redacted]			X				No	
[redacted]							No	
[redacted]				X			No	
[redacted]				X			No	Decolorization resin needs 3,000 cubic feet of resin at $300/cubic foot. Resin, beds and regeneration equipment estimated at $8 - 10 million and uses Ethanol to regenerate resin.
[redacted]		X		X			No	

[redacted]	***Vendor not cooperative with samples***	***Assessed and determined not effective***	***Not commercially available***	***High Dosages required***	***Results not scalable beyond bench scale***	***Low recoveries and brine disposal concerns***	***Technically Feasible (y/n)***	***Comments***
[redacted]							Yes*	Installed at Sorbitol plant
[redacted]						X	No	
[redacted]						X	No	
[redacted]						X	No	
[redacted]			X				No	
[redacted]		X	X				No	
[redacted]		X					No	Requires over 30,000 pounds of ferric salts per day
[redacted]			X				No	
[redacted]		X					No	Raise the pH 10 and add ozone and hydrogen peroxide. Large amounts of chemicals required.
[redacted]			X				No	
[redacted]							Yes	Suitable for <~50,000 GPD, non-grain based wastewater with non-chelated, salt-form nickel such as Polyols Plant IX regen waste

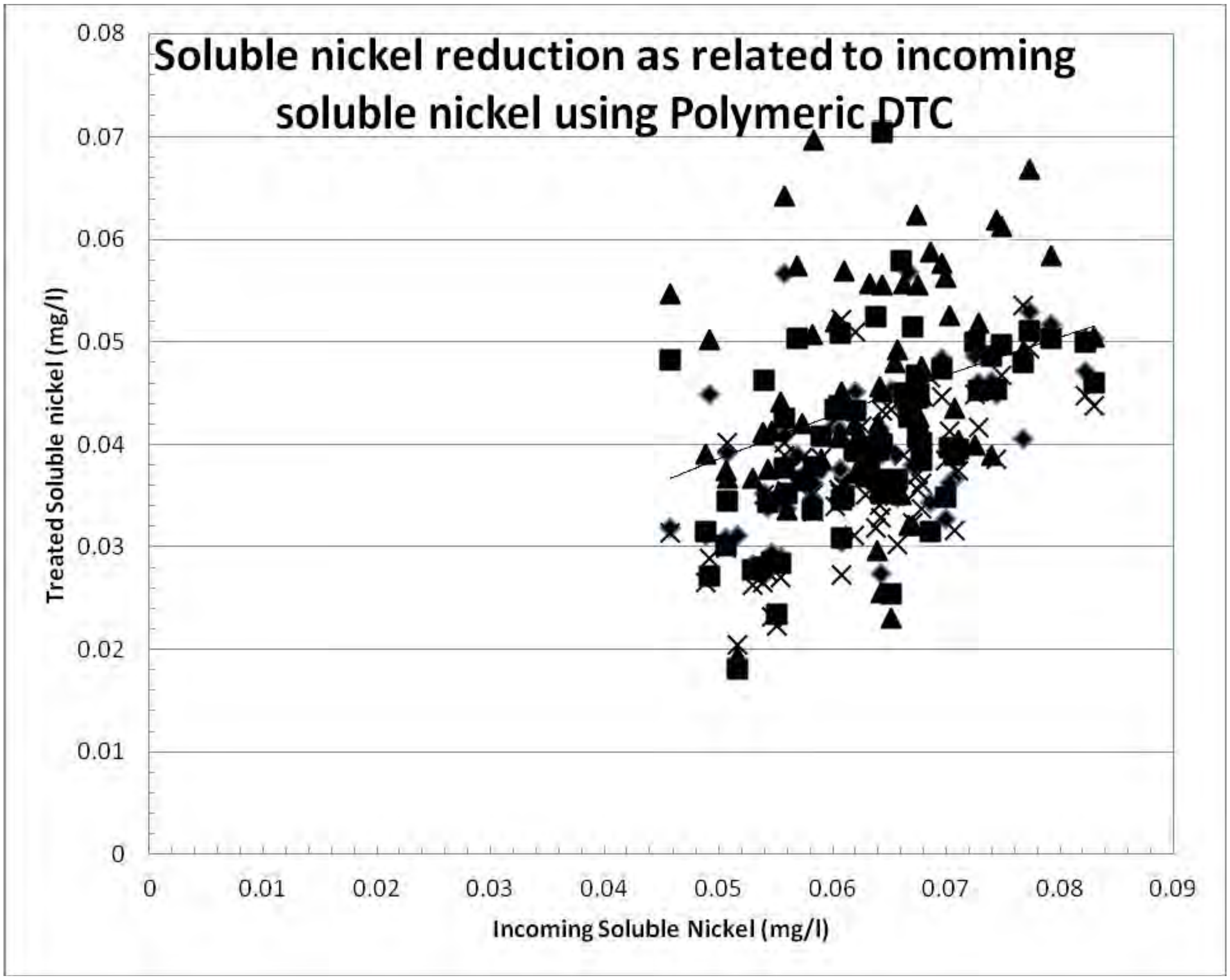
* The amount of used ion exchange resin is limited and it is most effective on non-chelated nickel. Therefore, it is being used to capture nickel from the sorbitol process.

Exhibit 33

Table 5: Capital and Operating Costs for Nickel Removal at ADM Decatur Complex			
	Initial Capital Cost	**Annual Operating & Chemical Costs**	**Status**
Active Projects			
1) Soybean Process Stream Alternative	$2.7 million	$400,000	Planned
2) Used IX resin system at Sorbitol Plant	$450,000	$200,000	Installed
3) High pH precipitation at Polyols Plant	$750,000	$600,000	Planned
Further Technical Analysis/Cost Prohibitive			
1) Polymeric DTC addition and nickel removal using different unit operations			Being piloted
a) Settling Clarifier and Sand Filter	$25.58 million	$7.2 million	Being piloted
b) Sand Float Filter	$23.14 million	$7.2 million	Being piloted
c) Sand Filter + precipitation	$24.48 million	$7.2 million	Being piloted
d) DE Filtration + Precipitation	$14.97 million	$7.2 million	Being piloted
e) DE Filtration	$ 7.05 million	$7.2 million	Being piloted
f) Sand Filter	$13.57 million	$7.2 million	Being piloted

Exhibit 34

Figure 3



SANITARY DISTRICT OF DECATUR, ILLINOIS

501 DIPPER LANE * DECATUR * ILLINOIS * 62522

INDUSTRIAL WASTEWATER DISCHARGE PERMIT APPLICATION

(Type or Print)

Business Name of Industry Applying for Discharge Permit:

ARCHER DANIELS MIDLAND COMPANY - PERMIT NUMBER 200 RENWAL

Mailing Address: PO BOX 1470

Decatur, IL 62525

Property Address: 4666 Faries Parkway

Decatur, IL 62526

Name of Property Owner: Archer Daniels Midland

Points of Discharge to the Sewerage System: Points A, B, C and D as listed in Permit Number 200

Type of Industry: Corn and Soybean Processing and Refining - "Agri-Processing"

S.I.C. or NAICS Number(s): Various. See attachment #1

Number of Employees: (part-time) ______ (full-time) See attachment #2

Hours of Operation (include shift times): Continuous 24 hours/day, 7 days/week

Products: Various products derived primarily from corn and soybeans. See attachment #3

(include a complete description - use additional sheets as needed)

Source(s) of Water and Average Volume From Each Source: See attachment #4A

City Water Account Numbers: See attachment #9

Wastewater Producing Operations (in order of significance): See attachment #4B

Water Consumption; Average Daily (gal/day): Attach #4A Maximum Daily (gal/day): Attach #4A

Wastewater Discharge; Average Daily (gal/day): Attach #4A Maximum Daily (gal/day): Attach #4A

Production &/or Cleanup: 7 days/week 24 hours/day

Mark days on which there will be a discharge: Mon [X] Tue [X] Wed [X] Thu [X] Fri [X] Sat [X] Sun [X]

List Categorical Processes: 40 CFR 406 Subpt A Corn Wet Milling; 40 CFR 439.20 Subpt B, Pharm Mfg Extraction

(Those processes defined in 40 CFR, Parts 403 - 471 as categorical)

SANITARY DISTRICT OF DECATUR, ILLINOIS

501 DIPPER LANE * DECATUR * ILLINOIS * 62522

List the Types and Descriptions of Major Processes: See attachment #3

Attachment #5 contains information concerning current environmental permits and SPCC plans

Is the First Major Process: (check one)

[x] Continuous/intermittent Discharge [ ] Batch Discharge [ ] Dry (no discharge)

Attach a current laboratory analysis report that accurately details the constituents of the industrial wastewater discharges from your entire facility, and a list of all current environmental permits issued for air, land, or water. Also include a supplemental information form for each individual process (including the one listed on this form).

Describe Method(s) of Wastewater Pretreatment at Your Facility:

See attachments #6 and #6A (block flow diagrams) and attachment #8 (area plot plans)

Hours of Pretreatment Operation: 24 hrs / 7 days per week

Name of IEPA Certified Operator: See attachment #7

Company Representative Responsible for the Industrial Wastewater Discharges From This Industry:

Name: Brad Crookshank

Title: Waste Water Treatment Supervisor

Telephone Number: 217-451-4534 E-Mail Address: brad.crookshank@adm.com

Signature for Application (Company Administrative Official):

Name: Randall R. Kampfe Position: VP Corn Production

(⇑ Type or Print Name ⇑) (⇑ Type or Print Position ⇑)

Signature: Randall R Kampfe

Date of Application: October 17, 2011

Application: [✓] Approved SDD Permit Number: 200

Effective Date: 12/21/2011

Expiration Date: 12/20/2016

[ ] Denied Reasons

By: Mark Cherry

District Director, or Authorized Agent of the Sanitary District of Decatur.

SANITARY DISTRICT OF DECATUR, ILLINOIS

501 DIPPER LANE * DECATUR * ILLINOIS * 62522

IN CONSIDERATION OF THE GRANTING OF THIS PERMIT, THE APPLICANT AGREES:

1. To furnish any additional information on industrial wastewater discharges as required by the Sanitary District of Decatur.
2. To operate and maintain any required industrial wastewater treatment devices in a satisfactory and approved manner.
3. To cooperate at all times with the Sanitary District of Decatur's personnel, or their representatives, in the inspection, sampling and study of industrial wastewater facilities and discharges.
4. To notify the Sanitary District of Decatur in the event of any accident, negligence or other occurrence that causes the discharge to the sewer of any materials whose nature and quantity might constitute a hazard to the District's personnel, wastewater treatment facilities or the environment.
5. To submit, as required by the Sanitary District of Decatur, accurate data on industrial wastewater discharge flows and wastewater constituents.
6. To apply for a revised District's industrial wastewater discharge permit if any change in industrial processes or operations creates a significant change in industrial wastewater quality or quantity.
7. To provide immediate access to authorized personnel of the Sanitary District of Decatur to any facility directly or indirectly connected to the District's sewerage system under emergency conditions and at all other reasonable times.
8. To accept and abide by all provisions of Ordinances of the Sanitary District of Decatur.
9. To submit additional pages as required to furnish any and all information if adequate room is not provided on the approved form.

SANITARY DISTRICT OF DECATUR, IL, INC.
Industrial Waste Division

INDUSTRIAL DISCHARGE PERMIT

PERMIT NUMBER: 200

Date issued:	December 21, 2011
Expiration Date:	December 20, 2016
Industrial User (IU):	Archer Daniels Midland Company (ADM)
Administrative Official:	Randall R. Kampfe
Title:	Vice President, Corn Production
Telephone Number:	(217) 424-5200
Designated Contact Person:	Brad Crookshank
Title of Contact Person:	Waste Water Treatment Supervisor
Telephone Number:	(217) 451-4534
E-Mail Address:	brad.crookshank@adm.com
Discharge Location(s):	"A" - Corn Sweeteners Pumping Station (ADM East, CSI) "B" - Faries Park Pumping Station (Faries) "C" - West Plant Discharge (Division Street) "D" - North Pump Station (To the Damon Avenue Pump Station)
Site Address:	4666 Faries Parkway Decatur, Illinois 62526
Mailing Address:	Box 1470 Decatur, IL 62525
SIC Code(s):	2038, 2046, 2048, 2075, 2079, 2099, 2833, 2869, 4213, & 4911
Industry Type:	Corn and Soybean Processing and Refining - "Agri-Processing"
Categorical Citation:	40 CFR 406, Subpart A, Corn Wet Milling Subcategory (with no categorical pretreatment standards) 40 CFR 439.20, Subpart B, Pharmaceutical Manufacturing, Extraction Subcategory

The above named **Industrial User (IU)** is required to comply with the conditions stated on the permit application form and all conditions, special and standard, as presented in this approved discharge permit. Satisfactory evidence of compliance with these conditions shall be supplied to the **Sanitary District of Decatur (SDD)** where requested. Satisfactory evidence shall consist of a minimum of written notification signed by an Authorized Representative of the IU and the submission of additional drawings and data when expressly requested.

A. SPECIFIC WASTEWATER DISCHARGE LIMITS AND SAMPLE FREQUENCY

1. Sanitary District of Decatur Monitoring Requirements and IU Discharge Limits.

This part contains the IU's discharge limits. The IU shall maintain compliance with these limits. These are the parameters that will normally be monitored by the SDD at the designated discharge points as described in Section C of this permit. The total discharge flow limit and the mass limits for BOD5, TSS, nickel, zinc, and NH_3-N apply to the combination of all plant discharges. The pH limit and all concentration limits apply to each individual discharge point. Grab sample limits apply to each individual grab sample and to entries on pH monitoring charts. Under "Monitoring Frequency," "2X/Year" means we will collect a set of samples approximately every six months (semiannually) and "4X/Year" means we will collect samples quarterly. A "24-hr Comp" is a daily composite sample collected over a 24-hour period.

Parameter	Daily Maximum	Monthly Average	Monitoring Frequency	Monitoring Method	Monitoring Locations
Discharge Flow **Total (MGD)** **Points A, B, & C (MGD)** **Point B (MGD)** **Point C (MGD)**	 22.184 11.00 1.00 1.00	 N/A N/A N/A N/A	 Continuous Continuous Continuous Continuous	 Record and Totalize At Each Point	Points: A,B,C, & D A, B, & C B C
Biochemical Oxygen Demand (BOD_5) (lb.)	54,022	N/A	Daily****	24-hour Composite	Points: A, C, & D
Total Suspended Solids (TSS) (lb.)	64,755	N/A	Daily	24-hour Composite	Points: A, C, & D
pH (minimum to maximum range in units)*	6.00 to 11.00*	N/A	4X/Year (minimum)	Grab	Points: A, C, & D
Ammonia Nitrogen (NH3-N) (lb.)	5,504	N/A	Weekly (minimum)	24-hour Composite	Points: A, C, & D
Fats, Oils & Grease – Total (FOG-T) (mg/l)	100	N/A	4X/Year** (minimum)	Grab	Points: A, C, & D
Nickel (Ni.) (Dissolved) (mg/l)	0.17	N/A	Random (at least 24X/ Year***)	24-hour Composite	Points: A & D
Zinc (Zn)(Total) (mg/l)	1.7	N/A	Random (at least 24X/ Year***)	24-hour Composite	Points: A & D
Acetone (mg/l)	20.7	8.2	2X/Year	Grab	A & D
n-Amyl acetate (mg/l)	20.7	8.2	N/A	Grab	A & D
Ethyl acetate (mg/l)	20.7	8.2	2X/Year	Grab	A & D
Isopropyl acetate (mg/l)	20.7	8.2	N/A	Grab	A & D
Methylene Chloride (mg/l)	3.0	0.7	N/A	Grab	A & D

* pH excursions between 5.0 and 6.0 or between 11.0 and 12.0 for less than a total of forty five (45) minutes in any calendar day shall not be considered reportable violations of this permit.

** Additional FOG-T monitoring may be done if the SDD sees a significant increase in FOG in the system.

*** Until July 1, 2014 we shall monitor the nickel concentration in the effluent approximately two times per discharge point each month unless we see a violation of the nickel limit shown here or other just cause for increased sampling exists in which case nickel monitoring frequency would exceed 24 times per year.

**** The BOD limit in this permit was reduced by 1482 pounds in August 2010 and that amount was added to the permit of the ADM Railcar Repair facility.

2. Industrial User Monitoring Requirements.

These are the IU's self-monitoring requirements. All monitoring results must comply with the limits stated in part 1 above.

Parameter	(units)	Monitoring Frequency	Monitoring Method	Monitoring Locations
Discharge Flow	**(GPD)**	2X/Year	Record and Totalize	A, B, C & D
BOD_5 and TSS	**(mg/l)**	2X/Year	24 Hour Composite	A, C & D
pH	**(units)**	Continuous	Record	A, C & D
FOG-T	**(mg/l)**	2X/Year	Grab	A, C & D
NH3-N	**(mg/l)**	2X/Year	24 Hour Composite	A, C & D
Ni. & Zn.	**(mg/l)**	2X/Year	24 Hour Composite	A & D
Acetone	**(mg/l)**	2X/Year	Grab	A & D
n-Amyl acetate	**(mg/l)**	Annually (d)	Grab	A & D
Ethyl acetate	**(mg/l)**	2X/Year	Grab	A & D
Isopropyl acetate	**(mg/l)**	Annually (d)	Grab	A & D
Methylene chloride	**(mg/l)**	Annually (d)	Grab	A & D

a. The SDD may waive all or part of the self-monitoring requirements.

b. Additional sampling may be done by the SDD if any discharge limit is exceeded and the permittee will be responsible for the costs of the additional monitoring.

c. When the IU's self-monitoring indicates an excursion from their permit limits, the IU shall notify the SDD immediately and shall resample for the parameter that exceeded their limit, analyze the sample, and report the results to the SDD within 30 days of the original sampling.

d. SIU results of acetone and ethyl acetate analyses shall be submitted semiannually, whereas results for the n-amyl-acetate, isopropyl acetate, and methylene chloride analyses only need to be reported if they are detected above the method detection limit. All results must be kept on file and made available to representatives of the SDD, IEPA, and USEPA upon request. If any of these three parameters shows up above detection limits, the SIU monitoring and reporting frequency for the pollutant that was detected will immediately be increased to semiannually (two times per year at equal intervals), and the SDD will also monitor for that parameter semiannually.

B. DEFINITION OF PENALTY CONDITIONS

1. When any violation of a specific or general prohibition occurs, a penalty of $1,000.00 per day per violation will result as a condition of this permit by authority of the SDD Board of Trustees. Each analytical parameter or reporting requirement related to this permit is considered distinct and penalties may be assessed individually with a maximum daily penalty equaling $1,000.00 multiplied by the distinct number of violations per day.

2. A violation of an Order of the SDD board of trustees shall be deemed a distinct violation and each day the violation persists shall be considered a new and distinct violation subject to a board assigned penalty of 1,000.00.

3. In addition to the penalties provided herein, the SDD may recover reasonable attorney's fees, court costs, court reporter fees, and other expenses of litigation by appropriate suit at law against the person found to have violated Ordinance 94-01, as amended, or the orders, rules, regulations and conditions of this permit.

C. MONITORING REQUIREMENTS

1. Description of Outfalls/Sample Points

a. Point "A" will be the existing Corn Sweeteners Pumping Station sample point directly south of the sweetener plant in the East Plant complex – this is the point known as "CSI".

b. Point "B" will be the existing Faries Park Pumping Station sample point.

c. Point "C" will be the existing West Plant sample station, which is centrally located in the West Plant complex.

d. Point "D" will be the effluent from the north Corn Sweeteners pumping station, which is located at the north side of the East Complex. This is the "Damon" discharge point.

Note: The effluent from ADM's truck wash facility on North Brush College Road is piped over to the East Plant for pretreatment and discharge to the SDD.

2. General Monitoring Requirements

a. The wastewater discharged by this facility shall be monitored at Points "A", "B", "C" and "D" as detailed in Section A this permit. For the purpose of surcharge and compliance, the concentration of pollutants at Point "B" shall be assumed identical to the concentration of pollutants at Point "A". Samples shall be collected and submitted for point C only if process flow or contaminated rainwater is discharged or if the total flow for the day exceeds 25,000 gallons (or both). All samples shall be collected so as to be representative of the IU's daily wastewater discharges, and all results are to be submitted to the SDD.

b. The IU is responsible for cleaning and maintaining the sample point to prevent any accumulation of oil, grease, sediment, or sludge; failure to do so does not invalidate the sampling and/or analytical test results. Results of analyses of samples taken at the designated location according to approved sampling procedures shall be accepted as binding.

c. All samples obtained either by the IU or the SDD for the purpose of verifying compliance with Permit or Ordinance conditions shall be collected, preserved, and analyzed in accordance with the procedures approved under 40 CFR part 136 as amended.

3. Monitoring Equipment

a. The IU will utilize and maintain:

1) automatic flow proportioned sampling devices at each discharge point (A, C, & D) that comply with the sampling and preservation criteria set in 40 CFR Part 136 as amended,

2) flow measuring and recording devices that accurately measure the volume of all wastewater discharges and are capable of sending signals to automatically collect flow proportioned samples at each discharge point, and

3) recording pH meters to continuously measure and record pH at each discharge point. These pH meters shall be standardized at least once per day with a standard log kept for all meter calibrations and standardizations.

4. Sample collection and analysis

a. At least one-half gallon of the daily composite sample from discharge points A, C, & D will be turned over to SDD personnel each morning. All samples should be ready for pick up by the SDD at the West Plant guardhouse no later than 7:00 a.m. each day. All samples shall be maintained at 40° F. Weekend samples will normally be collected by the SDD on Monday.

b. The IU will reimburse the SDD for all sample collection and analytical services according to the SDD's schedule of fees and services or according to actual costs for samples sent out.

D. REPORTING REQUIREMENTS

1. All reports required by this permit or by the SDD Pretreatment Ordinance shall be signed by the IU's administrative official or the authorized representative. An authorized representative must be authorized in writing as per Ordinance 94-01, as amended.

2. The IU shall give a thirty (30) day advance written notice to the SDD, and simultaneously shall apply to the SDD for a new permit, prior to introducing any new wastewater constituents to the sewer system or to making any substantial change beyond normal seasonal variations in the existing operations or facilities that would affect the volume or character of the wastewater being introduced to the sewerage system.

3. The IU shall submit a report to the SDD a minimum of ninety (90) days prior to any substantial change in sludge disposal practices.

4. The IU shall notify the SDD immediately (*within one-half hour after first noticing the discharge*) in the event of an accidental or slug discharge to the sewerage system as outlined in Section 300.135, III of Ordinance No. 94-01, as amended. Within five (5) days following an incident, the IU shall submit to the SDD a detailed written report describing the cause of the discharge and the measures to be taken by the IU to prevent similar future occurrences.

5. Any upset or excursion experienced by the IU of its pretreatment that places it in a temporary state of non-compliance with wastewater discharge limitations shall be reported to the SDD as soon as possible (not to exceed 24 hours from first awareness of the upset or excursion). A detailed report shall be submitted to the SDD within five (5) days of the upset or excursion. The report shall include the following:

a. A description of the cause of noncompliance;

b. The duration of non-compliance, including exact dates and times or, if not already corrected, the anticipated time the noncompliance is expected to cease; and

c. Steps being taken and/or planned to reduce, eliminate, and prevent recurrence of the noncompliance.

Failure to make a proper report of an upset or a slug load shall be deemed a distinct violation of this permit.

6. The IU shall submit an Annual Report to the SDD by March 1 each year on the report form provided by the SDD. The report is to include details of changes that have been made during the previous calendar year that could cause pass through or interference at the SDD treatment facility and/or in the collection system, or could be detrimental to the SDD's Land Application of Sludge program.

7. It is the responsibility of the IU to immediately (*within one-half hour after first noticing the discharge*) report to the SDD any materials discharged that may pass-through or interfere with the POTW.

8. The IU will deliver flow data as required by the SDD along with the daily samples.

9. The IU will provide the calibration records from the pH and flow-metering equipment as requested by the SDD.

10. The IU shall report the results of all approved self-monitoring at least twice per year. Self-monitoring shall be done at approximately the same time each year. These self-monitoring reports shall be due on or before June 1 and December 1 of each year of this permit.

E. SPECIAL CONDITIONS

1. Volumetric Assignments

The total volumetric discharge limit of 22.184 MGD shall be divided as follows:

a. The volumetric peak rate of discharge to the District's East Side Booster Pump Station through ADM's private force main (the combination of Points A, B, and C) shall be such that the total of the three permitted discharges does not exceed 11,000,000 gallons per day. [7,639 gallons per minute (gpm)]

b. The volumetric peak rate of discharge to the Fairies Park Pump Station (point B) shall not exceed 1,000,000 gallons per day. (695 gpm)

c. The volumetric peak rate of discharge to the District's 18-inch intercepting sewer located at 34th and Division Streets (ADM's West Plant discharge, Point C) shall not exceed 1,000,000 gallons per day. (695 gpm)

2. Minimum Volumetric Discharge

ADM will discharge wastewater to the SDD at such a rate to ensure a minimum monthly average discharge of 5.6 MGD. Discharge volumes of less than 5.6 MGD will not be considered a violation of this permit but will result in the calculation of monthly use charges based on 5.6 MGD.

3. Pretreatment System Operation

The IU shall have its pretreatment system under the control and direction of an Illinois Environmental Protection Agency certified operator at all times as per Ordinance 94-01, Section 300.125 Pretreatment.

4. Truck Wash Log Sheets

The IU shall keep a constant running log of every tank and truck washed at the truck wash facility. A separate entry shall be made for each tank or truck washed. The record shall include:

a. the date and time of washing,
b. the truck identification (ID) number, and the truck company,
c. the last product hauled and the source,
d. the amount of heel remaining,
e. the driver's name or signature, and
f. the truck wash attendant's name.

These wash logs shall be made available to representatives of the SDD at all reasonable times.

5. **Truck Wash Restriction**

This permit authorizes the truck wash facility to discharge wastewater only from the washing of trucks and/or trailers that have last hauled food products, as defined in 40 CFR part 442. At no time shall the IU wash any tank that contained any material that is considered a hazardous waste. Any infraction of this condition may be grounds for permit revocation.

6. **Daily Submittal of pH and Flow Charts**

The IU shall submit daily charts from the pH and flow recorders at discharge points A and D to the SDD each day along with the daily samples.

7. **New Nickel and Zinc Limits**

The following limits will take effect as of July 1, 2014 and will replace the existing dissolved nickel and total zinc limits.

Parameter	Daily Maximum	Monthly Average	Monitoring Frequency	Sample Type	Sample Locations
Nickel (total) (lb.)	14.746	3.588	Weekly*	24-hour Composite	A & D
Zinc (total) (lb.)	155.96	44.035	Weekly*	24-hour Composite	A & D

* As of July 1, 2014, the SDD will monitor the nickel and zinc concentrations in the effluent at least once per week per discharge point unless we see evidence of nickel or zinc exceeding the permitted limit or other just cause for increased sampling exists in which case nickel and/or zinc monitoring frequency would increase. As of July 1, 2014, the IU will also have to self-monitor for these parameters semiannually.

8. **Interim Reports**

The IU shall submit reports to the SDD by December 1, 2011, and June 1 and December 1, 2012 detailing their progress concerning reducing their effluent concentrations of nickel and zinc from current levels to levels that will not exceed those shown in Section E, paragraph 7, of this permit.

9. **Dissolved Metals**

The IU shall not introduce any significant sources of dissolved nickel or zinc (such as would be used in cooling towers), nor shall it change any process such that it would significantly change the ratio of total nickel or zinc to dissolved nickel or zinc in its final discharge to the sewer system without prior written notification to and approval from the SDD.

10. Technology and Economic Feasibility Review

a. Until July 31, 2012, the IU shall continue to investigate the potential for other technologies and developments in technologies already evaluated as per Section 1, h, ii, on page 31 of the Illinois Pollution Control Board's Order as issued on January 7, 2010.

F. STANDARD CONDITIONS

1. Permit Duration and Re-application

This wastewater discharge permit is being issued for a period of five (5) years. The IU shall file an application for renewal of their permit at least ninety (90) days prior to the expiration date.

2. Limitation on Permit Transfer

This permit is issued for the specific process activity outlined in the permit application and shall not be assigned, transferred, or sold to a new owner or new IU in the same or different premises or to a new or changed operation in the same or different premises without the approval of the SDD.

3. Modifications or Revisions of this Permit

a. The terms and conditions of this permit may be subject to modifications by the SDD during the term of this permit as limitations or requirements in Part 300 of Ordinance 94-01, as amended are modified or other just cause exists.

b. The terms and conditions of this permit may be modified as a result of the EPA promulgating new Categorical Discharge Standards or other applicable standards or regulations.

c. The terms and conditions may be modified in the event that the type, quality, or volume of the IU's wastewater is expected to change substantially from when this permit was granted.

d. The SDD shall inform the IU of any proposed changes in its permit at least thirty (30) days prior to the effective date of change.

e. If it becomes necessary for the SDD to modify the IU's permit, a reasonable time schedule shall be given to achieve compliance.

4. Semiannual Self-monitoring and Additional Monitoring

a. The IU shall monitor its wastewater discharges twice per calendar year at six-month intervals. This self-monitoring shall be performed and the results shall be reported to the SDD according to the schedule and on the proper form as provided by the SDD.

b. Periodically, the SDD will monitor the IU's wastewater discharges for pollutants other than those limited by this permit. The IU will reimburse the SDD for all costs incurred in this additional monitoring.

5. Reporting and Inquiries

All reports and inquiries should be sent to:

Attn: Pretreatment Coordinator
Sanitary District of Decatur
501 Dipper Lane
Decatur, IL 62522

6. Confidentiality

Matters of confidentiality shall be handled in accordance with Section 400.140 of Ordinance 94-01, as amended.

7. **Monitoring Records**

Records for monitoring information shall include:

- **a.** the sample dates, exact sample locations, sampling methods, types of samples, time of sampling, and the name of the person or persons taking the sample(s);
- **b.** the dates analyses were performed;
- **c.** the name of the laboratory that performed the analyses;
- **d.** the analytical techniques/methods used; and,
- **e.** the results of such analyses expressed in units as given in Section A, part 1, of this permit.

8. **Records Retention**

- **a.** The IU shall retain and preserve for no less than three (3) years any records, books, documents, or reports relating to monitoring, sampling, and/or analyses made by or on behalf of the IU in connection with its discharge.
- **b.** All records that pertain to matters that are the subject of special orders or any other enforcement or litigation activities brought by the SDD shall be retained and preserved by the IU until all enforcement activities have concluded and all periods of limitation with respect to any and all appeals have expired.

9. **Signatory Requirements**

All applications, reports, or information submitted to the SDD as required by this permit shall be signed by the authorized representative of the IU in the following positions:

- **a.** Corporation - principal executive officer of at least the level of a vice president.
- **b.** Partnership or Sole Proprietorship - general partner or proprietor.
- **c.** Duly authorized representatives of corporation, partnership, or sole proprietorship, if such representative is responsible for the overall operation of the facility from which the indirect discharge originates.

10. **Inspection**

The SDD shall conduct at least two complete facility inspections per year, one scheduled, and one drop-in.

11. **Access**

The IU will allow authorized representatives of the SDD, the United States Environmental Protection Agency (USEPA), or the Illinois Environmental Protection Agency (IEPA) immediate access at all reasonable times to the sampling points, areas of the plant where a discharge to the sewers may occur, and areas of the plant where records of concern to the SDD are kept. SDD personnel will present proper identification when requested by IU representatives.

12. **Authorized Personnel**

SDD personnel who are approved by the SDD Director, USEPA personnel, and IEPA personnel are authorized to carry out inspections and/or monitoring activities.

13. Falsification

Any person knowingly making any false statements on any reports or any other documents required by this permit, or who falsifies, tampers with, or knowingly renders inaccurate any monitoring device or method required to be maintained under this permit shall be subject to the penalties and costs provided for in this permit or in Section 600.100 of Ordinance 94-01, as amended; and shall in addition be guilty of a misdemeanor and, upon conviction, be punished by a fine of not more than one thousand dollars ($1,000.00) for each incident.

14. Payment: User Charges and Surcharges

a. The IU shall pay for normal costs of wastewater treatment through the user charge/surcharge system, as approved by the United States Environmental Protection Agency, or other relief authorized by the SDD's Ordinance or applicable laws.

b. The IU shall pay to the SDD, all costs incurred by the SDD in sampling and analyzing the IU's wastewater discharges.

c. Administrative penalties are not a normal cost of wastewater treatment.

15. Injunctive Relief, Attorney Fees, and Other Enforcement Procedures

The SDD may institute a civil action for an injunction to restrain violations of the terms of this permit in accordance with the procedures set forth in Section 500.130 of Ordinance 94-01, as amended, and pursue such other enforcement procedures as may be provided by the SDD Ordinance including collection of penalties or costs as per section 600.100 or revocation of the Permit for repeated or flagrant violations as per section 500.115 of Ordinance 94-01. If the SDD institutes a civil action to enforce the terms of this permit, the IU shall be responsible for all costs incurred by the SDD to enforce the terms and conditions of the permit, including but not limited to its attorney fees.

16. Severability

The conditions of this permit are severable, and if any provision(s) of this permit, or application of any provision of this permit is held invalid, the remaining provisions of this Permit shall continue in full force and effect.

17. General Prohibitions

Issuance of this permit is not a substitute for, nor does it relieve the IU from the authority of any applicable local, state, or federal industrial pretreatment regulation.

Exhibit 36

Response to U.S. EPA Toxicity Testing Comments
Sanitary District of Decatur, Illinois

Comment 1. On p. 1 of "Water Effect Ratio Testing to Support a Site-Specific Water Quality Standard Request for the Sanitary District of Decatur, Illinois," the Sanitary District of Decatur (SDD) states:

> During the summer of 2013, Region 5 requested additional information demonstrating that the BLM is consistent with certain aspects of nickel aquatic toxicity studies reported in the scientific literature. These discussions also led to a recommendation from IEPA and Region 5 that the District perform aquatic toxicity testing on its effluent discharge using the water effect ratio ("WER") procedure. The District is therefore proposing to perform WER testing to serve as additional confirmation for the predicted BLM-based WER and for the proposed site-specific water quality standard.

EPA would like to clarify that the Agency raised the *option* of pursuing a WER-based site-specific nickel criterion as an alternative to a BLM-based approach. EPA has not endorsed one method over another, but has raised concerns about the degree to which the current iteration of the nickel BLM accounts for toxicological data reported in the scientific literature. EPA raised the option of conducting a WER study to derive a site-specific nickel criterion in the context of uncertainty around the technical defensibility of the nickel BLM.

With respect to the proposed toxicity testing work, per discussions with SDD, the permittee has elected to pursue a site-specific nickel criterion using a BLM-based approach. SDD has also chosen to pursue toxicity testing on *Ceriodaphhnia dubia* and *Pimephales promelas* (though not "WER tests," per EPA's current WER guidance documents). SDD expressed that these tests are intended to determine the degree to which the nickel BLM accurately predicts toxic responses in *C. dubia* and *P. promelas* when exposed to nickel in site water. EPA will not be able to approve a site-specific criterion for nickel unless one of the following occurs:

1) SDD:
a. addresses, in a satisfactory manner, EPA's comments on the nickel BLM, generally, and EPA's comments on SDD's application of the nickel BLM to the SDD and the Sangamon River, specifically, as discussed on the December 5, 2013 conference call between EPA, IEPA, and SDD, and;

b. the application of a satisfactory nickel BLM indicates that Illinois's statewide nickel criterion can be raised to reflect site-specific water quality conditions at the SDD.

2) SDD conducts a full WER study, as described in EPA guidance and provided to SDD on November 4, 2013, and this study indicates that Illinois's statewide nickel criterion can be raised to reflect site-specific conditions at the SDD.

Response: The wording of the document has been revised to incorporate U.S. EPA's clarifications. SDD recognizes the information needs for U.S. EPA to consider approval of a site-specific water quality standard. SDD is seeking acceptance of the testing procedure for the limited purpose noted in previous discussions and in U.S. EPA's comment.

Comment 2. On p. 2 of "Water Effect Ratio Testing to Support a Site-Specific Water Quality Standard Request for the Sanitary District of Decatur, Illinois," SDD states:

> A report prepared by Robert Santore of HDR I HydroQual describes the application of the nickel BLM to the Sangamon River downstream of the District's discharge. This report, "Estimate of the BLM Adjustment to the Nickel Criterion for the Sanitary District of Decatur, Illinois," dated April 16, 2013 is included as Attachment A. The report provides an overview of the BLM and summarizes site sampling data, and proposes a WER of 2.62. The report also includes a recommended site specific water quality standard of 38.2 ug/L, based on the IEPA-assigned critical hardness value of 359 mg/L.
>
> As noted above, one round of WER testing is planned to serve as additional confirmation for the predicted BLM-based WER. This testing is proposed to be consistent with U.S. EPA guidance and will include chemical analysis of all BLM parameters for additional confirmation of the model prediction.

EPA notes that the April 16, 2013 report and the WER value proposed therein have not been revised since EPA's August 27, 2013 comments on both the nickel BLM used to conduct the modeling and the model's application to SDD. As noted in Comment 1, EPA will not be able to approve a site-specific nickel criterion for SDD until SDD successfully addresses EPA's comments on the nickel BLM and its application to SDD, or SDD conducts a full WER test, as outlined in EPA guidance documents.

Response: The April 16, 2013 report has been updated to incorporate corrected calcium and magnesium values in Table 3. SDD recognizes that additional information will need to be provided to address U.S. EPA's comments regarding the BLM.

Comment 3. The selection of organisms for toxicity testing may impact the magnitude of the resulting WER.

According to EPA's Water Quality Standards Handbook Appendix L, the primary toxicity test used to determine the WER should have an endpoint in lab dilution water that is close to, but not lower than, the CMC to which the WER is applied (http://water.epa.gov/scitech/swguidance/standards/upload/2002_06_11_standards_handbook_handbookappxL.pdf, p.45; see also Appendix D, p. 122 at the same link). If the LC50 for *C. dubia* in laboratory water is below the CMC, then the resulting WER value may be inflated. Given the apparent sensitivity of *C. dubia* to nickel (and conflicting information on exactly how sensitive *C. dubia* are), using this organism as a test species may produce a WER that overestimates the degree to which the site-specific criterion can be raised without impacting the level of protection provided by the chronic aquatic life water quality criterion.

If the proposed toxicity testing is primarily aimed at confirming the WER that is derived from the BLM, then it is important to ensure that organisms used in toxicity testing do not compromise the resulting WER due to elevated sensitivity to nickel. Choosing a test species that has been shown to be less sensitive to nickel than *C. dubia* would be one way of ensuring that the resulting WER is not skewed by test organism sensitivity.

Response: The WER would be adjusting the Illinois standard, not the national ambient water quality criteria. The Illinois CMC at a hardness of 50 mg/L is 45.9 µg/L. The toxicity of nickel to *C. dubia* at this hardness is 81 µg/L (Keithly et al., 2004). Based on the WER guidance, therefore, *C. dubia* appears to be an appropriate organism choice for the WER test.

The only other acute options would be *D. magna*, *D. pulex*.

Comment 4. Section 3.2 of the *C. dubia* study plan (p. 87 of the pdf shared by SDD) stipulates that testing be done in "very hard reconstituted laboratory water to achieve a nominal hardness, alkalinity, and pH of approximately 315 mg/L as $CaCO_3$, 225 mg/L as $CaCO_3$, and 8.0, respectively." Section 3.2 of the *P. promelas* study plan (p. 94 of the pdf shared by SDD) outlines testing to take place in "hard reconstituted laboratory water to achieve a nominal hardness, alkalinity, and pH of approximately 180 mg/L as $CaCO_3$, 120 mg/L as $CaCO_3$, and 8.0, respectively." In section IV. b. of "Water Effect Ratio Testing to Support a Site-Specific Water Quality Standard Request for the Sanitary District of Decatur, Illinois" (p. 3 of the pdf shared by SDD), it appears that the intent is to use very hard reconstituted water in all toxicity testing. Is there an error in the *P. promleas* study plan, or has a change been made to the plans as outlined by SDD?

Response: The reference to hard reconstituted water in the *P. promelas* protocol is an error. The protocol has been revised to state that testing with *P. promelas* will occur in very hard reconstituted water.

Comment 5. Given that the criterion to which a site-specific adjustment is proposed is a chronic criterion, would it make sense to conduct toxicity testing to calculate a cccWER? Given that the BLM-based WER will be derived using a model that is based solely upon acute toxicological data, would chronic toxicity testing provide a check to ensure that any chronic effects not captured in acute data sets used to develop the BLM are captured and considered?

Response: The chronic criterion for nickel is based on an acute species sensitivity distribution and an acute to chronic ratio. Deriving a site-specific chronic standard from an acute WER therefore would be no less defensible than the existing standard.

Comment 6. The Oregon State University study plans do not specify whether total or dissolved WERs will be calculated. Because calculation of both WERs is recommended in EPA's WER guidance, measurement of both total and dissolved metal at the beginning and end of tests (as well as prior to renewal, in the *P. promelas* test) is also recommended (Water Quality Standards Handbook Appendix L, p. 9). How do the methods proposed for use in calculating total and dissolved nickel compare to the methods used to derive nickel concentration in the toxicity database upon which the criterion was derived?

Response: The protocols have been revised to sample both total and dissolved nickel as recommended. Both a total and dissolved WER can be calculated based upon the analytical measurements throughout the tests.

Comment 7. How exactly is the WER to be calculated? (See Water Quality Standards Handbook Appendix L and EPA's 1997 guidance document entitled "Use of the WER Procedure with Hardness Equations" (http://water.epa.gov/scitech/swguidance/standards/handbook/upload/2003_08_06_standards_modif-intwer. pdf) for acceptable methodologies.) How are differences in site water and laboratory water composition (*i.e.* differences in physiochemical variables like hardness and ion levels) going to be accounted for in the calculation of the WER? Will the toxicity values obtained in laboratory water be adjusted to the same hardness and/or other water composition factors seen in the site water prior to determination of the WER, per EPA's guidance document entitled "Use of the WER Procedure with Hardness Equations" or Water Quality Standards Handbook Appendix L, pp. 39-43?

Response: From pg. 40 of the EPA WER guidance document: the experimentally determined WER will usually be a ratio of endpoints determined at two different hardnesses and will thus include contributions from a variety of differences between the two waters, including hardness.

The WER will be calculated as discussed in the guidance document. The use of the US EPA very hard water as a reference water is already a reasonably close match to the site hardness. The reference water LC50 could be further adjusted to match the site water hardness using the hardness slope for the Ni standard, but we anticipate that any such adjustment would be small, given the already close match in hardness anticipated in the reference and site water samples.

Comment 8. In sections 4.1 of both the *C. dubia* and *P. promelas* study plans, a dilution scheme of 0.5 is proposed. Could you please explain why the dilution factor of 0.5 was proposed (EPA recommends between 0.65 and 0.99 (Water Quality Standards Handbook Appendix L, p. 53))?

Response: The dilution scheme has been changed to the recommended factor of 0.7 in both study plans.

Comment 9. Will water be prepared and aged as recommended by EPA guidance (Water Quality Standards Handbook Appendix L, p. 54)?

Response: The test protocols have been revised to state that: The site effluent will be spiked with Ni and serial dilution will take place. The waters will then be allowed to equilibrate for 2-4 hours.

The test protocols will be revised to state that the laboratory dilution water will be prepared by serial dilution and allowed to equilibrate for 1-3 hours.

Comment 10. On p. 3 of "Water Effect Ratio Testing to Support a Site-Specific Water Quality Standard Request for the Sanitary District of Decatur, Illinois," SDD states that "[i]n addition to monitoring chemical parameters relevant to the toxicity testing, chemical analysis of BLM input parameters will be conducted on both the effluent sample and laboratory reconstituted water." To the extent that the level of any physiochemical variable relevant to the operation of the BLM is expected to change throughout the toxicity testing procedures, measurement at the beginning and end of test periods will help to ascertain the degree to which levels of these variables change and should be conducted.

Response: The only water quality parameter that is likely to change during the test is pH. We will monitor pH (and hardness) at the beginning and end of the test but we propose to measure all other parameters once at test initiation (except as noted above regarding total and dissolved nickel).

Comment 11. Section 5.0 of each of the Oregon State University study plans states "[s]tatistical analysis (hypothesis testing) of the test data will be conducted using a computer program. A statistical test (as determined by the USEPA Decision Tree [USEPA, 2002]) will be used to test for significant differences in the survival among test treatments and controls." To clarify, will the statistical methods used be consistent with EPA guidance (Water Quality Standards Handbook Appendix L, pp. 58-59 recommends probit or regression analysis)?

Response: The statistical methods used will be consistent with the most current EPA methods for determination of acute effects. The EPA Water Quality Standards Handbook Appendix L, pp. 58-59 references the older EPA acute testing version (1993). The newest EPA acute version (2002) dictates the flowchart for determination of the LC50 for multi-effluent concentration acute toxicity tests and this flowchart coincides with the statistical methodology described in the WER guidance (1994).

Comment 12. Are Oregon State University researchers confident that the acclimation procedures described in section 2 of the study plan for *P. promelas* will facilitate a successful test (*e.g.*, acceptable control mortality, etc.)? If so, please provide a brief explanation. Is the acclimation for *P. promelas* and age of organisms to be tested consistent with EPA methods (Water Quality Standards Handbook Appendix L , p. 47) and/or the toxicity data to which the new data will be compared (data used to develop BLM, data used to derive Illinois's criterion)?

Response: The Water Quality Standards Handbook Appendix L, p. 47 references EPA (1993 a, b, c) and/or by ASTM (1993 a, b, c, d, e). The most recent version of EPA guidance (2002) states that the age of organisms should be 1-14 days; less than or equal to 24-h range in age (required). A random selection of organisms (which have been acclimated to hard/very hard water) will be acclimated to the site water for as long as possible prior to the test without compromising the time constraints of first use of the site water.

To allow acclimation to the very hard water conditions, the protocol has been amended to use fish approximately 7-14 days old.

Toxicity Testing to Support a Site-Specific Water Quality Standard Request for the Sanitary District of Decatur, Illinois

I. INTRODUCTION

For approximately five years, the Sanitary District of Decatur ("District") has been developing information to pursue a site-specific water quality standard for nickel. The nickel standard is proposed to be applied to the portion of the Sangamon River influenced by the discharge from the District's main treatment plant in Macon County, Illinois. The District has anticipated that the technical basis for the proposed standard will be provided by the Biotic Ligand Model ("BLM") for nickel that has been developed by HDR | HydroQual.

During the time period that the District has been developing information, regular communications have occurred between the District, the Illinois Environmental Protection Agency ("IEPA"), and the Region 5 office of the U.S. Environmental Protection Agency ("Region 5"). During the summer of 2013, Region 5 requested additional information demonstrating that the BLM is consistent with certain aspects of nickel aquatic toxicity studies reported in the scientific literature. As part of these discussions, Region 5 raised the option of pursuing a Water Effect Ratio (WER)-based site-specific nickel criterion as an alternative to a BLM-based approach. The District is therefore proposing to perform toxicity testing following applicable portions of the federal WER guidance to serve as additional confirmation for the predicted BLM-based WER and for the proposed site-specific water quality standard.

II. STUDY PURPOSE AND APPROACH

The District's effluent discharge contains higher concentrations of nickel than typical domestic wastewater treatment plant discharges. These concentrations are also higher than the District's NPDES permit limit, which is based on the generally-applicable Illinois water quality standard. The permit limit is not currently in effect because of a variance granted to the District by the Illinois Pollution Control Board.

The flow in the Sangamon River is highly variable but because the District's discharge is located approximately three miles downstream of the dam impounding Lake Decatur, the river flow is near zero when no water is being released from the dam. The District's NPDES permit limits are therefore based on a critical 7Q10 low flow of zero.

The nickel in the District's effluent originates primarily in the pretreated discharge from one large industrial user. This industrial user has implemented both source reduction practices and wastewater treatment technology to decrease the amount of nickel discharged from its facility into the District's collection system.

Annual water quality studies have been conducted for more than a decade by personnel from the Biology Department of Eastern Illinois University, under contract to the District. These studies do not identify any negative impact on water quality in the Sangamon River due to nickel concentrations in the District's discharge. In light of the lack of any identified adverse impact from nickel in its discharge, the District is proposing a site-specific water quality standard based on the BLM.

A report prepared by Robert Santore of HDR I HydroQual describes the application of the nickel BLM to the Sangamon River downstream of the District's discharge. This report, "Estimate of the BLM Adjustment to the Nickel Criterion for the Sanitary District of Decatur, Illinois," dated April 16, 2013 is included as Attachment A. The report provides an overview of the BLM and summarizes site sampling data, and proposes a WER of 2.62. The report also includes a recommended site-specific water quality standard of 38.2 ug/L, based on the IEPA-assigned critical hardness value of 359 mg/L.

As noted above, one round of WER testing is planned to serve as additional confirmation for the predicted BLM-based WER. This testing is proposed to be consistent with U.S. EPA guidance and will include chemical analysis of all BLM parameters for additional confirmation of the model prediction.

III. BACKGROUND SITE INFORMATION

Information describing the District's wastewater treatment facility and the Sangamon River in the vicinity of the facility discharge is contained in the District's variance petition submitted to the Illinois Pollution Control Board on June 15, 2009. The petition is included as Attachment B. The petition also contains information on the nickel limit in the District's NPDES permit and nickel concentrations in the plant discharge.

Because the Sangamon River 7Q10 low flow at the discharge location is zero, the toxicity testing will be conducted using a sample of the District's effluent discharge that is undiluted by upstream flow. This condition is represented by very dry weather conditions during the late fall and winter months of 2013-2014, and river flow measured during this time at the USGS gauging station upstream of the discharge point has been 2 cfs or less except for brief periods. During these low flow conditions, the District's discharge flow is usually in the range of 19-24 mgd. To the extent reasonably possible, sample collection for the toxicity testing will be scheduled on a day that the effluent flow is within this range.

IV. SAMPLING AND TOXICITY TESTING PROCEDURE

It is the intent of the sampling and testing procedure to be consistent with U.S. EPA guidance contained in "Interim Guidance on Determination and Use of Water-Effect Ratios for Metals" (EPA 823-B-94-001). Many of the considerations in the guidance for steps that should be undertaken prior to beginning a WER study have already been done in other contexts. Information from the single toxicity testing round is not intended to be utilized as the sole basis for a WER, so the portions of the guidance dealing with scheduling of multiple sampling events, options for determining a WER, conditions for determining and using a WER, and implementing the results of a WER are inapplicable or will be addressed outside of the toxicity testing process.

The Oregon State University Aquatic Toxicology Laboratory ("OSU") has been engaged to perform the toxicity testing. OSU has provided two procedures documents for the toxicity testing, data analysis, and reporting entitled *"Water-Effect Ratio (WER) Testing of Acute Nickel Toxicity in Site Effluent Water and Laboratory Water to the Cladoceran, Ceriodaphnia dubia, under Static Test Conditions"* and *"Water-Effect Ratio (WER) Testing of Acute Nickel Toxicity in Site Effluent Water and Laboratory Water to the Fathead minnow, Pimephales promelas, under Static-Renewal Test Conditions"*; these documents are included as Attachment C.

a. Sampling Procedures

Sampling will be planned when the discharge flow is reflective of dry weather conditions, as noted above, and will be conducted when the plant operation is stable with respect to flow and pollutant loading. Flow will be measured by the District's in-place flow monitoring equipment. Sampling will consist of 24-hour time-based composite samples of the effluent collected at the plant discharge point, described in the District's NPDES permit as Outfall 001. An automatic composite sampler will be utilized, with the temperature maintained at 4 degrees C. All sample tubing will be replaced with new tubing prior to initiation of the toxicity test sampling in accordance with "clean" sampling techniques. Additional composite samplers are available if needed to collect the sample volume required by the laboratory. Sample aliquots for analyses requiring chemical preservation will be obtained from the composite sample container at the end of the compositing period.

The 24-hour sampling period will be established to end at around 6 a.m. The sample volume will correspond to that required by OSU. Samples will be placed into properly cleaned and prepared sample containers provided by OSU and shipped via priority overnight package delivery to arrive at the laboratory in time to begin testing within 36 hours of the end of the composite sampling period.

b. Toxicity Testing

The toxicity testing organisms will be *Ceriodaphnia dubia* and *Pimephales promelas*, cultured as described in Section 4.2 of the OSU procedure. The testing will utilize reconstituted "very hard" water prepared according to U.S. EPA guidance, to correspond to the high hardness usually present in the District's effluent. Sections 3 and 4 of the OSU procedure describe the toxicity testing protocol.

c. Chemical Analysis

In addition to monitoring chemical parameters relevant to the toxicity testing, chemical analysis of BLM input parameters will be conducted on both the effluent sample and laboratory reconstituted water. The chemical monitoring is also described in Section 4.5 of the OSU procedure.

V. REPORTING AND DATA ANALYSIS

The OSU laboratory will prepare a toxicity testing report as described in Section 6 of the procedure document. As noted in the procedure, the report will include all relevant information regarding the testing procedure and results.

Following review of the test results, a determination of the WER based on the toxicity testing will be made by HDR | HydroQual. As previously discussed with Region 5 and IEPA, this WER determination will serve as additional information for the overall determination of a BLM-predicted WER applicable to the District's discharge to the Sangamon River. All laboratory reports will be provided to IEPA and to Region 5 for their review.

Attachments

Attachment A – HDR | HydroQual report prepared by Robert Santore, "Estimate of the BLM Adjustment to the Nickel Criterion for the Sanitary District of Decatur, Illinois" (January 16, 2014)

Attachment B – Petition for Variance, filed by the Sanitary District of Decatur with the Illinois Pollution Control Board June 15, 2009

Attachment C – Oregon State University testing procedures, "Water-Effect Ratio (WER) Testing of Acute Nickel Toxicity in Site Effluent Water and Laboratory Water to the Cladoceran, *Ceriodaphnia dubia*, under Static Test Conditions" and "Water-Effect Ratio (WER) Testing of Acute Nickel Toxicity in Site Effluent Water and Laboratory Water to the Fathead Minnow, *Pimephales promelas*, under Static-Renewal Test Conditions" (April 2014)

Attachment A

Prepared for Proposed Site Specific Rule for Sanitary District of Decatur
From 35 Ill. Adm. Code Section 302.208(e)

ESTIMATE OF THE BLM ADJUSTMENT TO THE NICKEL CRITERION FOR THE SANITARY DISTRICT OF DECATUR, ILLINOIS

Prepared by
Robert Santore

April 16, 2013

HDR | HydroQual

I. INTRODUCTION

This report was prepared in support of the Sanitary District of Decatur's ("District") Petition to the Illinois Pollution Control Board ("Board") seeking a Site Specific Rule to establish an alternative water quality standard ("WQS") for Nickel from the point of its discharge into the Sangamon River from its Main Sewage Treatment Plant ("Main Plant") to the point of the confluence of the Sangamon River with the South Fork of the Sangamon River near Riverton, Illinois. The purpose of this report is to present the calculations, comparisons, and findings acquired from using the federally approved Biotic Ligand Model ("BLM") to adjust the Nickel WQS such that it considers local conditions found in that segment of the Sangamon River.

Adjustment of the WQS for metals in consideration of the local chemical conditions has frequently been shown to be appropriate at sites across the United States, since WQSs are based on water quality criteria ("WQC") that are defined using a traditional methodology that does not consider many of the factors that are known to affect metal toxicity to aquatic organisms. For example, the WQC for several metals (including Silver ("Ag"), Cadmium ("Cd"), Chromium (III) ("Cr(III)"), Lead ("Pb"), Nickel ("Ni"), and Zinc ("Zn"), as well as Copper ("Cu") prior to development of the BLM) are dependent on the hardness of the local water. The term "hardness" refers to the mineral content of the water and is primarily associated with the combined concentration of Calcium ("Ca") and Magnesium ("Mg"). Hardness is one of several key water quality constituents that have been shown to affect metal bioavailability and toxicity. The United States Environmental Protection Agency's ("US EPA") approach for deriving metals WQC as hardness-dependent relationships has considered how variation in toxic response may differ in areas that naturally have either very hard or very soft water.

However, factors other than hardness have been shown to affect metal bioavailability, and in particular variation in pH, alkalinity, and the presence of natural organic matter ("NOM") have all been shown to be as important, or even more important, than hardness in determining metal toxicity (Erickson, et al., 1996). These factors may increase or decrease the toxicity of metals. The dependence of metal toxicity on local chemical factors is referred to as the "bioavailability" of the metal to aquatic organisms. Since these bioavailability factors are not considered by WQC approaches that only consider hardness, the WQC may be more or less protective than needed for a specific receiving water. This issue has long been recognized by USEPA and, in response, US EPA has developed procedures for derivation of site specific adjustments to WQC (Carlson, et al. 1984; US EPA, 1992, 1994a). In particular, the Water Effect Ratio ("WER") approach is intended to account for local bioavailability factors that can affect metal toxicity (US EPA, 1994b). The site specific adjustment to a WQC provided by a WER is intended to correct for deficiencies in the WQC derivation process and to reduce the degree to which a WQC is over-protective or under-protective for a given location.

II. BACKGROUND ON NICKEL BLM

Although the WER has been in use for decades, it requires toxicity testing with multiple aquatic organisms in multiple samples. Costs and time required to accommodate WER testing can be significant. As an alternative, the BLM is a computational approach that can simulate the effects of water chemistry on metal toxicity, and on the physiological response of aquatic organisms to metals (Di Toro, et al, 2001; Santore, et al, 2001). The BLM provides information that is similar to the WER, but does so with much less cost and time required. The BLM is a mechanistic approach, not an empirical approach like the hardness equation, and it considers effects from numerous chemical factors such as pH, the presence of NOM, alkalinity, and major ions (including cations that contribute to hardness). The BLM considers how these factors affect either metal chemistry or organism physiology to determine metal bioavailability (Figure 1).

The BLM has been adopted by US EPA as a replacement for the hardness equation in the most recently updated metals criteria (US EPA, 2007). The use of the BLM provides similar benefits as the WER, and for criteria based on the BLM, the use of the WER is no longer required. For metals (such as Nickel) where US EPA has not adopted a BLM-based procedure for replacement of the hardness equation, the BLM can be used in a manner similar to the WER to modify the hardness equation based WQC. Use of the BLM to derive a site specific WQC provides the same level of protection as intended by US EPA guidelines (Stephan, et al, 1985). To the extent that a BLM derived site specific WQC is different from the national ambient WQC, those differences reflect how local factors which are not considered by the hardness-equation may change metal bioavailability and toxicity.

The BLM can be used to determine modifications to chemistry of receiving water using a procedure that is analogous to the WER. The WER compares the toxicity of Nickel or other toxicant in receiving water to that in reference water. The reference water is intended to represent the conditions comparable to those used to develop the toxicity database in which the acute and chronic WQC were developed. The WER is then simply the ratio of the measured toxic endpoint in the receiving water to that in the reference water. If multiple receiving water and reference water samples are used to generate the WER, the WER is determined for each pair of samples, and then an overall WER is usually determined as the geometric mean. The reference water chemistry must meet WER guidelines (US EPA, 1994b), and US EPA has provided synthetic recipes suitable for generating reference water samples with various hardness concentrations. These recipes can be incorporated into the BLM application to predict toxicity endpoints for suitable reference water that can be used in a WER-type analysis.

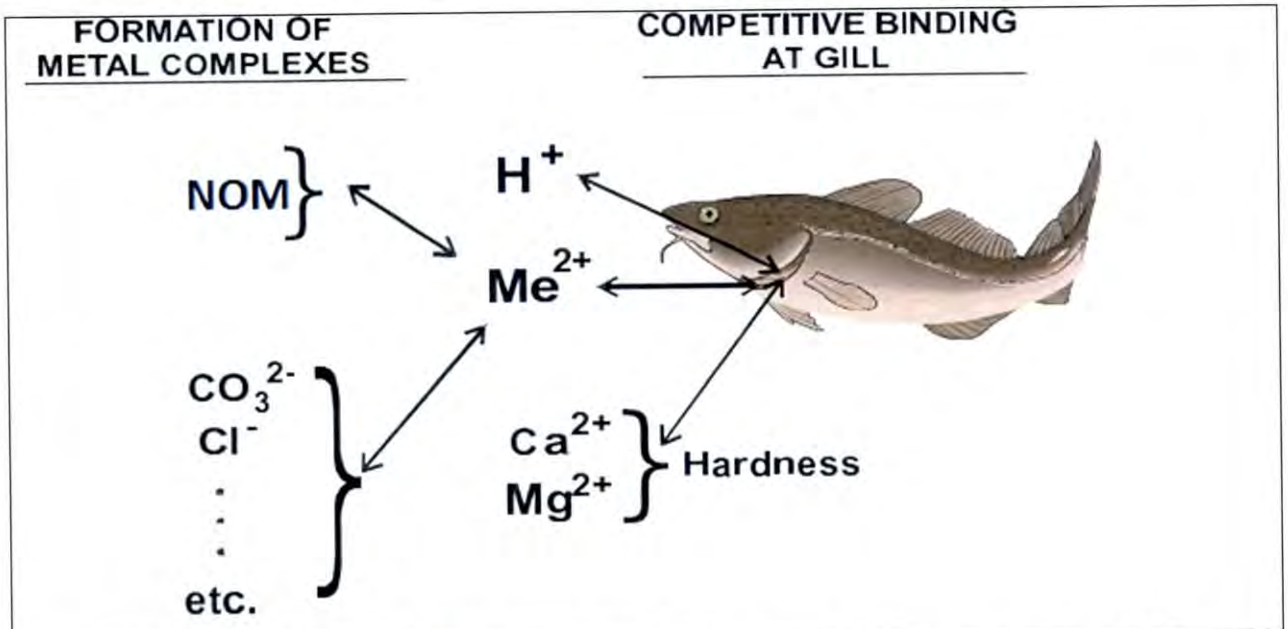


Figure 1. Conceptual model of the chemical and physiological processes represented in the BLM. Water chemistry, including inorganic complexes and binding by NOM, can affect the chemical speciation and reactivity of a metal (i.e., Me^{2+}). The accumulation of metal on biological surfaces, such as gill membranes, is related to the chemical reactivity of the metal as well as other factors such as pH and competitive binding of cations. The BLM is a general framework that has been applied to acute and chronic responses of numerous metals including Aluminum ("Al"), Ag, Cd, Cobalt ("Co"), Cu, Ni, Pb, and Zn.

III. BLM RESULTS WITH MEASURED WATER QUALITY

A. Overall Calibration Results to Fish and Invertebrates

The BLM is a generalized mechanistic approach that has been applied to a number of different metals including Nickel. Development efforts for Nickel focused on explaining available toxicity data for sensitive aquatic invertebrates and fish in a project sponsored by the Water Environment Research Foundation ("WERF") (WERF, 2003). The project for WERF included a detailed review of the chemical speciation of Nickel in freshwaters, analysis of Nickel accumulation in aquatic organisms, and a summary of important bioavailability factors, including pH, alkalinity, hardness, and the presence of NOM. The performance of the Nickel BLM was quite good, with excellent agreement between predicted and measured toxicity over a range of several orders of magnitude (Figure 2). Nearly all of the predicted toxicity values are within a factor of two of measured values.

Agreement with a factor of two of a given measured toxicity value has been shown to be about the degree to which replicate measurements agree with a mean value. Replicate toxicity tests used to determine replicate LC50 values for the same organism in the same water frequently does not produce exactly the same result. For example, replicate copper toxicity measurements, expressed as the median lethal concentration to 50% of the population (LC50), made to the same species of fish in water samples from Lake Superior tend to fall in ±2x envelope around a central mean (Figure 3; data are from Erickson et al., 1996). If replicate measurements agree with a central mean value no better than ±2x, then comparison of predicted toxicity values with measured values with a factor of ±2x would be the best that could be expected. Hence, predicted values such as those shown in Figure 2 are often shown within a ±2x envelope around the line of perfect agreement, and predicted values that fall within this envelope show excellent agreement with measured values.

The strength of the predictive ability of the BLM lies in the mechanistic and generalized nature of the model. Although the model simulates a complex set of chemical reactions and biological accumulation processes, these processes are characterized as generalized reactions based on thermodynamics. The model can therefore predict accumulation in aquatic organisms without recalibration of any of the model parameters that describe chemical speciation, or organism accumulation. Application of the same model and same model parameters are used to predict effects to diverse aquatic organisms including fish and invertebrates. The consistency of this approach is evidence of the mechanistic and generally applicable nature of this analysis. The only parameter that varies from one organism to another is the concentration of accumulated metal associated with toxicity (Santore, et al, 2001). The resulting model is capable of simulating Nickel toxicity to a range of organisms in a wide range of chemical conditions (Figure 2).

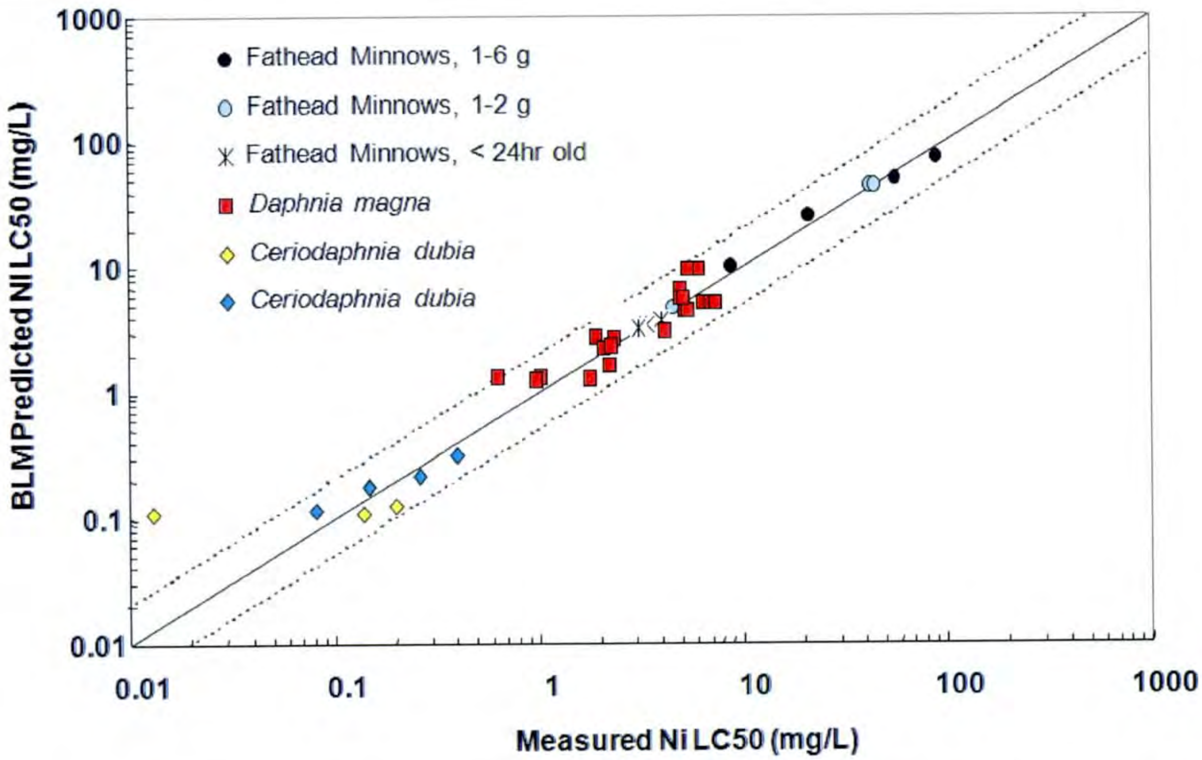


Figure 2. Comparison of the calibrated Nickel BLM to sensitive freshwater aquatic invertebrates and fish. Measured toxicity, as the lethal concentration to 50% of the test organisms, is shown on the horizontal axis. Predicted toxicity is shown on the vertical axis. The diagonal solid black line shows perfect agreement between measured and predicted values, and the dashed black lines show a region of ± factor of 2x from perfect agreement. The ± factor of 2x is intended to show agreement between measured and predicted values that comparable to the expected agreement between replicate measurements.

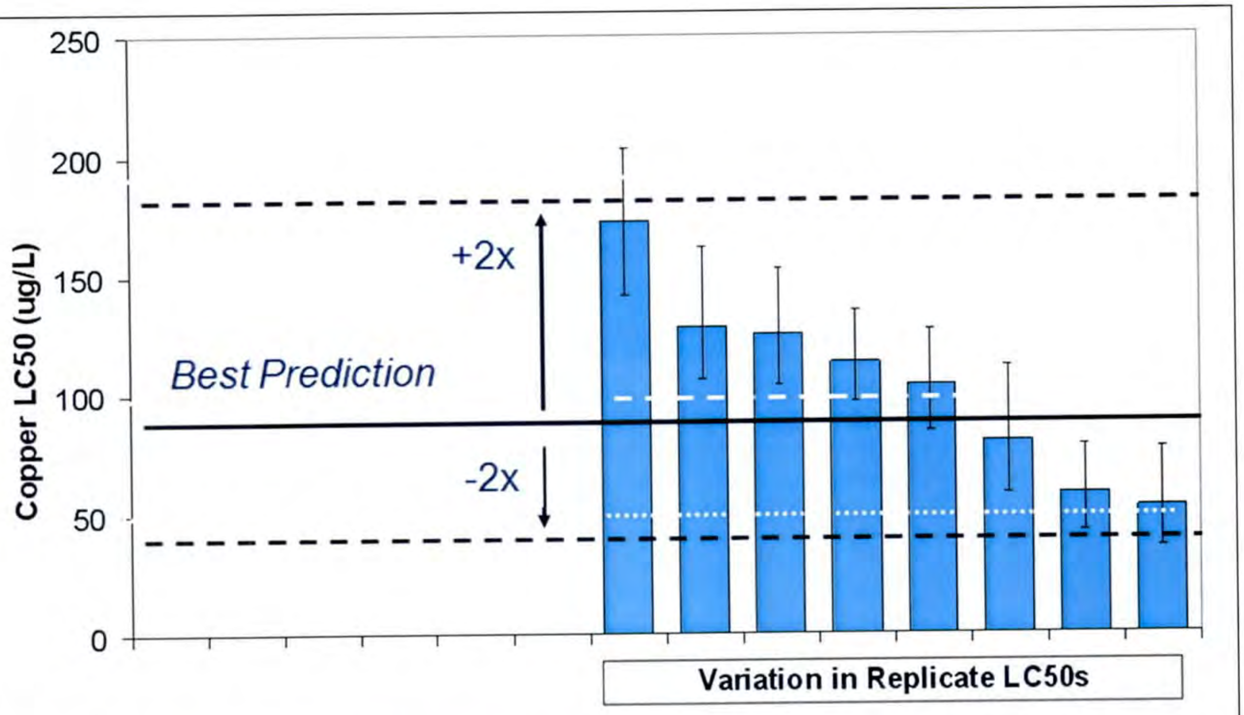


Figure 3. Variation in replicate measurements of LC50 of copper to fathead minnow in Lake Superior water tends to fall in an envelope of plus or minus 2 times the geometric mean value (date from Erickson et al., 1996). The dark solid line labeled "Best Prediction" is shown at the geometric mean of the measured values. The dashed lines correspond to an envelope showing plus or minus a factor of two. Since all of these measured values are from water samples with the same chemistry, the BLM would predict the same LC50 in every case.

IV. CALCULATED WER WITH PREDICTED TOXICITY TO *DAPHNIA MAGNA*

As discussed in Section II of this report, the BLM for Nickel can be used to calculate a site specific WQC by using the model to calculate a WER for the receiving water downstream of the Main Plant. Samples were collected at two locations downstream of the Main Plant discharge, and chemical analyses for BLM input parameters were measured on these samples. Similar analyses were made on samples taken from the Main Plant effluent, although these were not used in the WER analysis. Measured chemical parameters used as input parameters to the Nickel BLM are shown in Table 1.

The BLM for Nickel was run with these input data to determine Nickel toxicity to *D. magna*, which is a sensitive invertebrate recommended for use in WER testing for Nickel (USEPA, 1994b, Appendix I). For calculation of WER values, the predicted toxicity in these site waters was compared with toxicity in a reference water sample. According to the WER guidance document, suitable reference water must have a hardness concentration close to, but not in excess of, the measured hardness in the site water (US EPA, 1994b). The US EPA's recipe for "very hard" water with a hardness of 317 mg/L as Calcium Carbonate ("CaCO3"), compared with hardness in the site water of 347, would be a suitable choice for use as a reference water for WER testing at the site. Calculated LC50 values for site and reference water are shown in Table 2.

Table 1. Input chemistry used for BLM analyses. For site waters, Sangamon River samples collected at the Rock Springs Trail bridge approximately one-half mile downstream (RD at Rock Springs) and at the South Lincoln Memorial Parkway bridge approximately six miles downstream (RD at Lincoln) were used to characterize the chemistry of the receiving 1water downstream of the plant. The presence of NOM was characterized by the dissolved organic carbon ("DOC") concentration. For calculation of WER, the US EPA's "very hard" water recipe was used as a reference sample. Variation of an assumed DOC in the reference water sample from 0.5 to 2.0 mg C/L was included in the BLM analysis.

Sample Description		Temp	pH	DOC	Ca	Mg	Na	K	SO4	Cl	Alk
		°C		mg C/L		----------------------		mg / L	----------------------		
RD at Rock Springs	8/26/2010	23	8.00	12	56	53	396	86	298	446	365
RD at Rock Springs	9/9/2010	21	8.09	10	64	48	286	53	214	304	341
RD at Lincoln	8/26/2010	25	8.00	10	58	46	296	60	225	450	321
RD at Lincoln	9/9/2010	21	8.10	7.9	65	43	192	35	146	202	315
Final Effluent	8/26/2010	30	8.09	13	56	62	504	112	374	558	400
Final Effluent	9/9/2010	28	7.90	14	62	62	474	91	328	477	399
US EPA Very Hard	DOC=0.5	20	8.20	0.5	47	48	105	8	304	8	229
US EPA Very Hard	DOC=1.0	20	8.20	1	47	48	105	8	304	8	229
US EPA Very Hard	DOC=2.0	20	8.20	2	47	48	105	8	304	8	229

Table 2. Predicted toxicity to *D. magna* by the Nickel BLM in site and reference water samples used in WER analysis. For calculation of WER values, the average LC50 determined in site water was divided by the average LC50 in the reference water. The US EPA's "very hard" recipe for synthetic water was chosen as the reference water due to the good correspondence between the hardness in this recipe and at the site.

Sample Description		Ni LC50 mg/L	Average Ni LC50 mg/L	Average WER
RD at Rock Springs	8/26/2010	32.38	28.89	2.92
RD at Rock Springs	9/9/2010	25.61		
RD at Lincoln	8/26/2010	25.55	22.84	2.31
RD at Lincoln	9/9/2010	20.13		
Final Effluent	8/26/2010	44.52	43.78	4.42
Final Effluent	9/9/2010	43.04		
US EPA Very Hard	DOC=0.5	9.82	9.90	
US EPA Very Hard	DOC=1.0	9.88		
US EPA Very Hard	DOC=2.0	10.00		

Site water was characterized by performing two separate sampling events at both Rock Springs B and Lincoln Homestead. The BLM calculated LC50 values to *D. magna* in site-waters downstream of the Main Plant ranged from 22.84 mg/L to 28.89 mg/L (Table 2). For comparison, the calculated LC50 for reference water based on the US EPA's "very hard" water recipe was 9.9 mg/L. The WER values for each sampling location, calculated by dividing site water LC50 by the reference water LC50, correspond to 2.31 and 2.92 for Rock Springs B and Lincoln Homestead. Since these values are similar, an overall WER for the site can be determined by averaging to obtain an overall WER for the site of 2.62.

Predicted toxicity in the Final Effluent and the resulting WER value is also shown for comparison in Table 2, but these values were not averaged into the overall WER for the site. The predicted average LC50 in effluent samples was 43.78 mg/L, which is considerably higher than in downstream receiving water samples. The chemistry for the effluent shown in Table 1 indicates that effluent samples had higher concentrations of cations, such as Ca, Mg, and Sodium ("Na"), as well as a higher concentration of NOM (measured as DOC). All of these factors would tend to further mitigate against nickel toxicity to aquatic organisms, which is why the predicted LC50 in effluent samples is higher. As a result, Nickel toxicity would be lower in any areas that are poorly mixed downstream of the discharge, and the resulting WER would be protective for these areas as well.

V. SENSITIVITY TO VARIATION IN WATER CHEMISTRY

Since relatively few samples were used in the BLM analysis summarized in Tables 1 and 2, an additional analysis was conducted to see what effect natural variation in downstream water chemistry would have on the predicted toxicity. Additional monitoring data were used to characterize variation in measured chemistry corresponding to BLM input parameters. Monitoring data describing the variability in downstream chemistry was collected by the Sanitary District of Decatur, and combined with monitoring data for the Sangamon River collected by *Eastern Illinois University. Samples collected for these monitoring studies were obtained at a number of different stations downstream of the plant, including Lincoln, Rock Springs, and Wyckles Bridge, as well as unnamed stations 100 yards and 600 yards downstream. Variability in measured chemistry in the pooled data from these sampling stations includes both spatial and temporal variation. From these available data, the* 10^{th}*,* 25^{th}*,* 75^{th}*, and* 90^{th} *percentiles were estimated for key water quality parameters that are known to affect nickel bioavailability, including pH, DOC, Ca, Mg, Na, and Alkalinity (Table 3). A set of base case conditions was established as the median value for all parameters. Variation in K, SO4, and Cl was not considered since these parameters are not important in determining the bioavailability of nickel.*

Table 3. Variation in water quality parameters that affect nickel bioavailability was characterized as the 10^{th}, 25^{th}, 75^{th}, and 90^{th} percentile estimated from a dataset of pooled measurements are stations downstream of the Decatur Plant. The values for the base case were based on median values from the same dataset.

Test	Temp. C	pH SU	DOC mg/L	Ca mg/L	Mg mg/L	Na mg/L	K mg/L	SO4 mg/L	Cl mg/L	Alk mg/L
base	17.78	8.14	9.99	70.00	84.25	244.00	47.4	185.5	326.0	279.00
10th		7.96	3.7	48.0	29.9	202.4				151.2
25th		8.03	6.4	57.6	38.0	218.0				223.0
75th		8.29	14.8	138.9	122.4	270.0				321.0
90th		8.47	28.2	159.0	140.1	285.6				451.2

These data correspond to pre-existing monitoring studies and were not specifically collected for BLM analyses. Consequently, not all BLM parameters were measured in every sample. For the purposes of conducting a sensitivity analyses, these data are suitable for showing the expected downstream variation in individual parameters. Available data are plotted in Figure 4 for river samples and Figure 5 for effluent samples.

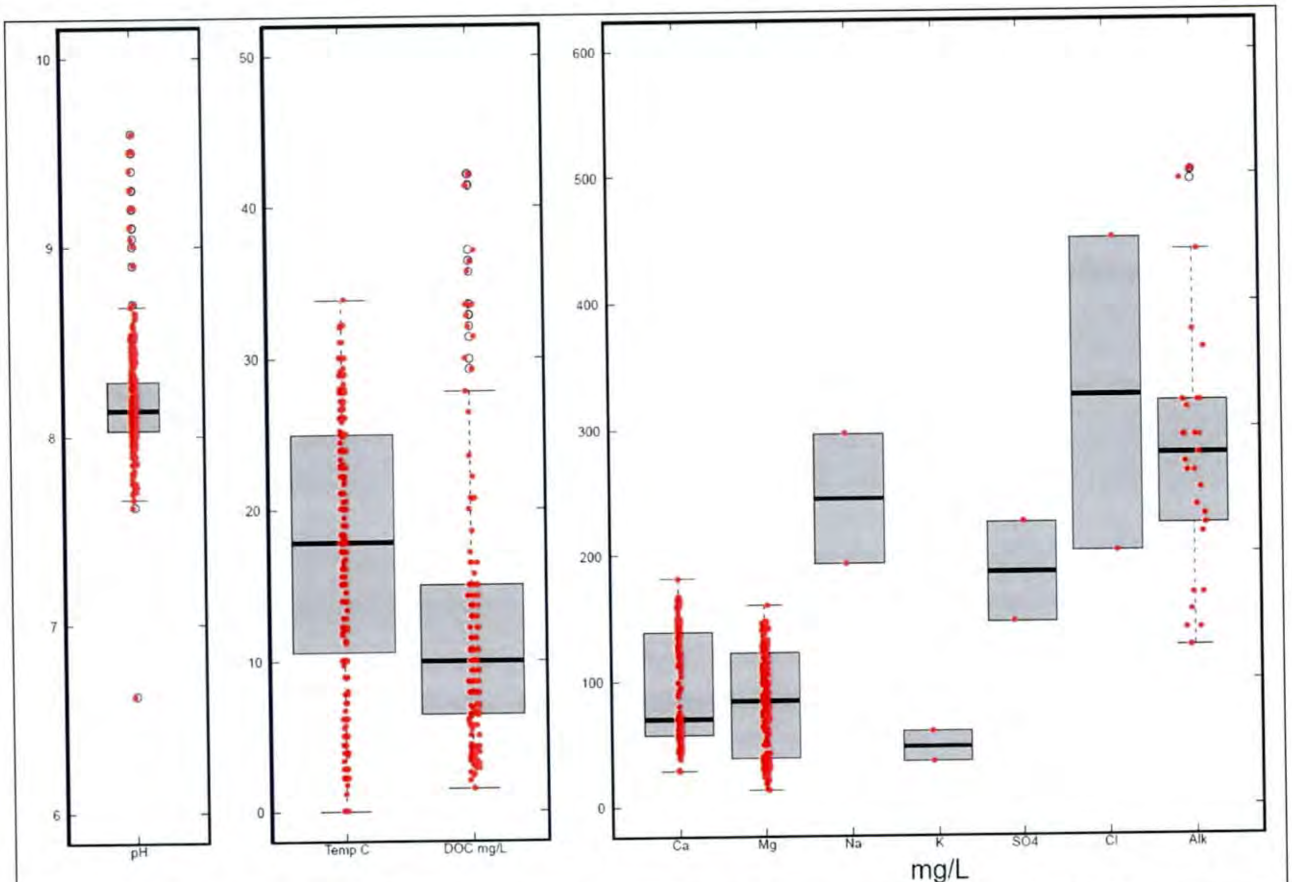


Figure 4. Box and whisker plots showing distributions of measured values for BLM input parameters in river samples. Average values are shown by a black line in the middle of each box and represent mean (pH, Temp, DOC) or geometric mean (Ca, Mg, Na, Potassium ("K"), Sulfate ("SO4"), Chlorine ("Cl"), Alkalinity ("Alk")) depending on whether parameters are expected to be normally or log-normally distributed. For each box, the lower edge of the box represents the 25th percentile, the upper edge of the box represents the 75th percentile, and whiskers extend to minimum and maximum values exclusive of extreme values. Individual observations are shown as small red circles.

The distribution of values for each parameter are shown as box and whisker diagrams constructed so that the lower edge of the box represents the 25th percentile, the upper edge of the box represents the 75th percentile, and whiskers extend to minimum and maximum values exclusive of extreme values. Median values are shown as the solid black horizontal line in the middle of each box. Individual observations are shown as small red circles. For river samples, there was a large amount of data characterizing pH, alkalinity, DOC, and hardness cations (Ca and Mg), which are the bioavailability factors that are the most important for determining nickel toxicity (Figure 4). There were relatively few samples characterizing K, and SO4, but these parameters have little to no effect on nickel toxicity and do not need to be considered in the uncertainty analysis. There were also relatively few observations for Na, but the estimated variation in Na concentrations is similar to that seen for Ca and Mg and is therefore, likely to be a reasonable characterization of variation in downstream chemistry. For effluent samples there were many more measurements of anion concentrations (Figure 5), and in comparison with river samples the effluents tended to have lower pH values and higher DOC and ion concentrations. The variation in pH, DOC, and ion concentrations show in these two datasets are consistent with the values seen in detailed sample analyses reported in Table 1.

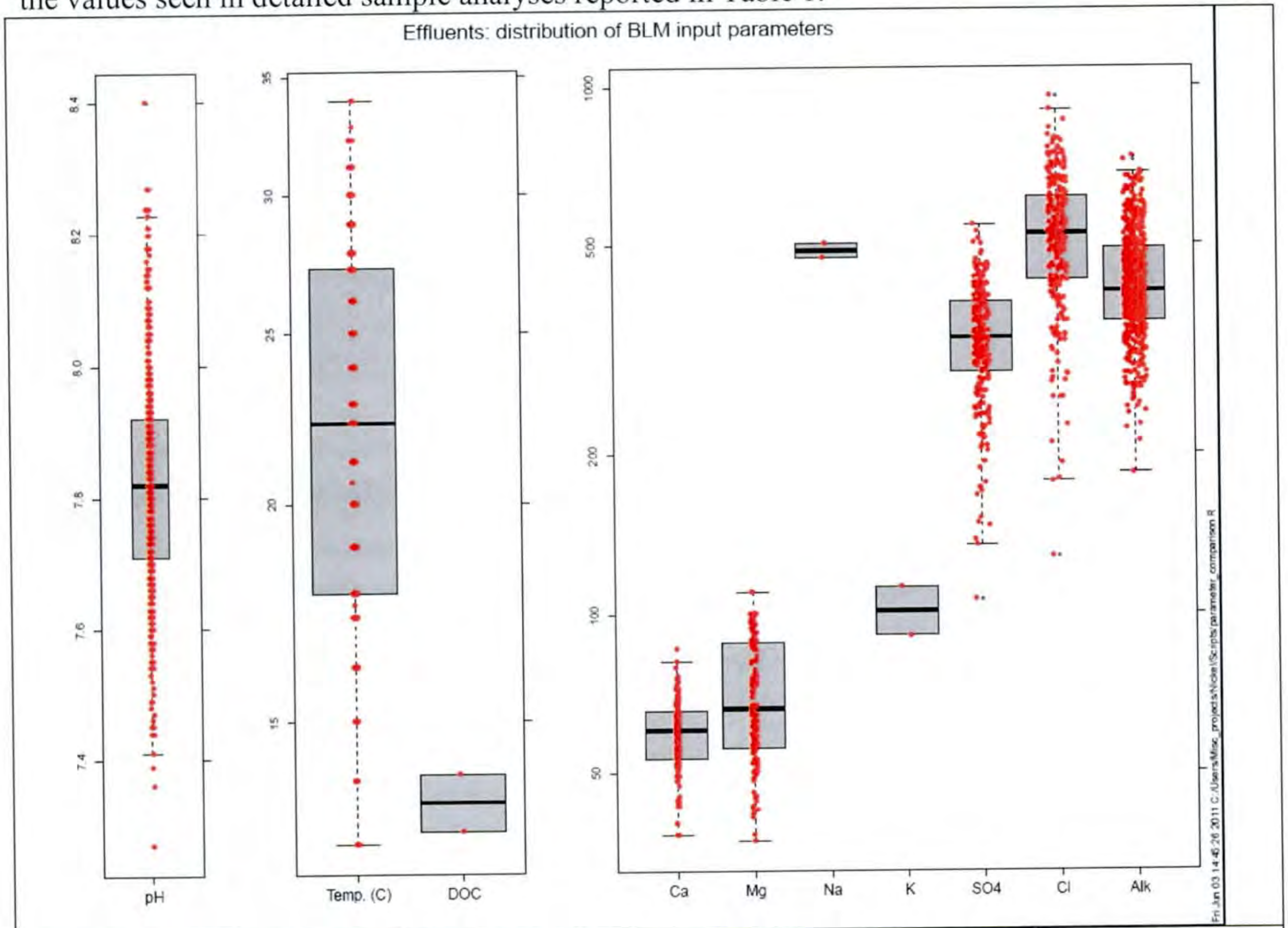


Figure 5. Box and whisker plots showing distributions of measured values for BLM input parameters in effluent samples. Average values are shown by a black line in the middle of each box and represent mean (pH, Temp, DOC) or geometric mean (Ca, Mg, Na, K, SO4, Cl, Alk) depending on whether parameters are expected to be normally or log-normally distributed. For each box, the lower edge of the box represents the 25th percentile, the upper edge of the box represents the 75th percentile, and whiskers extend to minimum and maximum values exclusive of extreme values. Individual observations are shown as small red circles.

Variability in BLM input parameters was used in a sensitivity analysis to determine the degree to which predicted toxicity may be expected to change over time. The model was first run for a base case that used median values for all parameters shown in Figure 4 and Table 3.

For each BLM parameter, two additional runs were then performed by substituting either

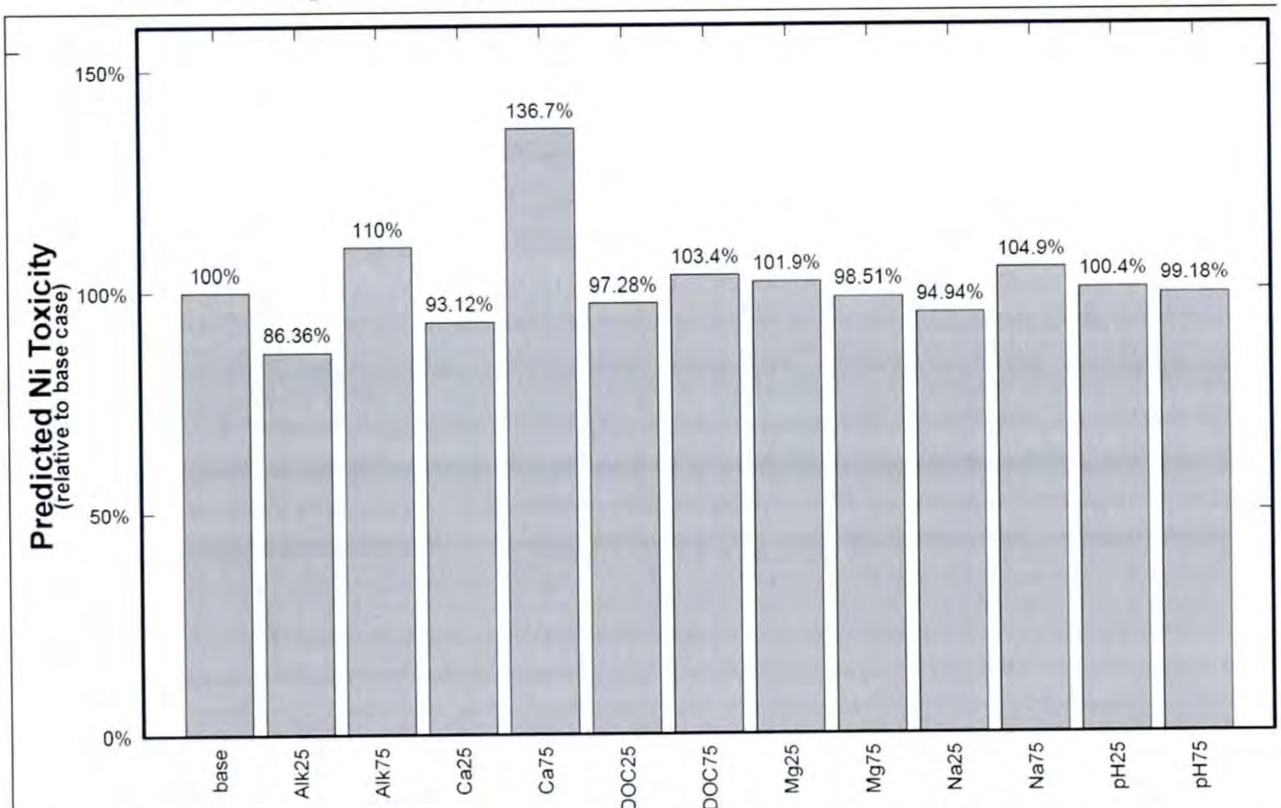


Figure 6. Sensitivity analysis of varying input parameters to the BLM on predicted Nickel toxicity in river samples. For the base case, average values for all parameters shown in Figure 4 were used. A series of additional simulations were then run to see the effect of variation in individual parameter values on the base case. For each additional simulation, the base case was modified with either the 25th or the 75th percentile value of an input variable, while all other parameters were held at the values used for the base case. For example, the result labeled "Alk25" uses the 25th percentile for alkalinity (shown in Figure 4), and the result "Alk75" uses the 75th percentile for alkalinity. Sensitivity results for other parameters are labeled with a similar labeling scheme.

the 25% or 75% value from the box and whisker plots in Figure 4 for the average value, while keeping all other parameters constant, at their respective average. The resulting sensitivity analyses are shown in Figure 6 for river samples considering variation at the 25th and 75th percentile, and Figure 7 considering variation at the 10th and 90th percentiles.

Variation in input values at the 25th and 75th percentiles for river water samples had relatively little effect on the predicted Nickel toxicity, with the largest effects resulting from changes in alkalinity and calcium concentrations. A similar pattern was observed when variation at the 10th and 90th percentiles were considered (Figure 7). Even at these extreme values, the expected variation in predicted nickel toxicity ranges from about 70 to 150 percent of the base case value. Guidance for derivation of site-specific adjustments to water quality criteria based on the WER procedure allow simple geometric means of individual WER values when the range in values is within a factor of 5. Since the effects of the variation in river water chemistry on nickel toxicity will be well within that those limits, this uncertainty analysis supports the conclusion that average conditions from a relatively small number of samples should provide an acceptable characterization for deriving a site-specific nickel criterion. As a result of these sensitivity analyses, the calculated WER for the site is not expected to significantly change as a result of variability in water quality within ranges comparable to these existing monitoring datasets.

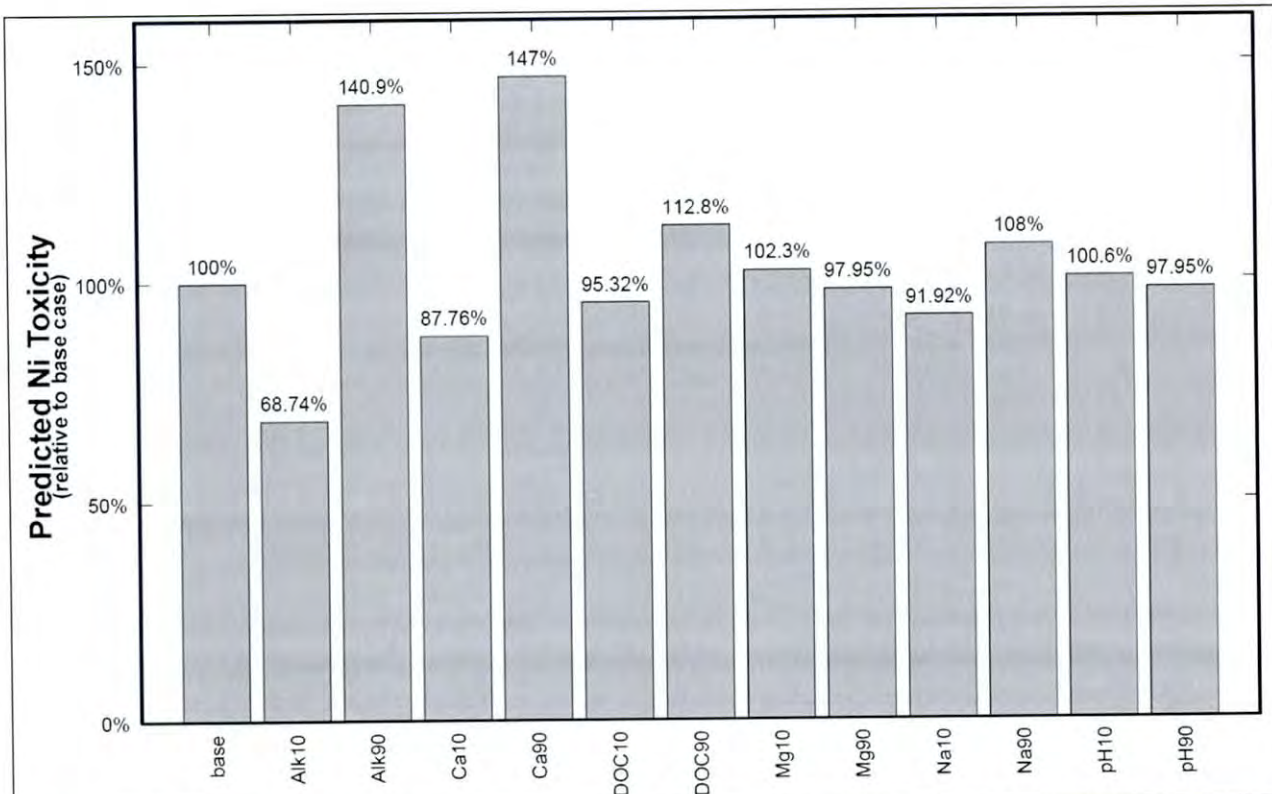


Figure 7. Sensitivity analysis of varying input parameters to the BLM on predicted Nickel toxicity in river samples. For the base case, average values for all parameters shown in Figure 4 were used. A series of additional simulations were then run to see the effect of variation in individual parameter values on the base case. For each additional simulation, the base case was modified with either the 10^{th} or the 90^{th} percentile value of an input variable, while all other parameters were held at the values used for the base case. For example, the result labeled "Alk10" uses the 10^{th} percentile for alkalinity (shown in Figure 4), and the result "Alk90" uses the 90^{th} percentile for alkalinity. Sensitivity results for other parameters are labeled with a similar labeling scheme.

For effluent samples (Figure 8), variation in alkalinity had the largest effect on predicted nickel toxicity. However, the resulting variation in predicted LC50 values was small, corresponding to a little more than 10% change relative to the base case. Variation in effluent characteristics is only presented for comparison to that seen for river water, since it is only the downstream river water that will be used to estimate the site-specific nickel adjustment.

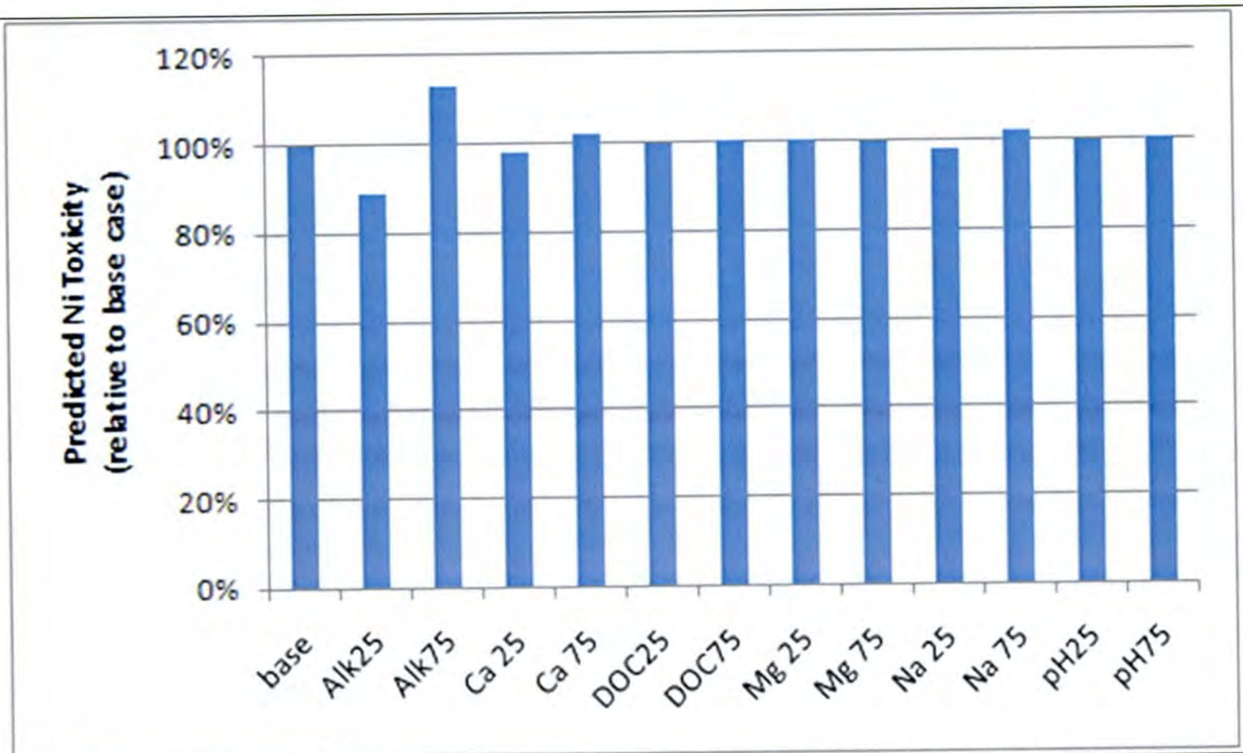


Figure 8. Sensitivity analysis of varying input parameters to the BLM on predicted Nickel toxicity in effluent samples. For the base case, average values for all parameters shown in Figure 5 were used. A series of additional simulations were then run to see the effect of variation in individual parameter values on the base case. For each additional simulation, the base case was modified with either the 25th or the 75th percentile value of an input variable, while all other parameters were held at the values used for the base case. For example, the result labeled "Alk25" uses the 25th percentile for alkalinity (shown in Figure 5), and the result "Alk75" uses the 75th percentile for alkalinity. Sensitivity results for other parameters are labeled with a similar labeling scheme.

VI. PREDICTED ESTIMATE OF WQC

With the WER calculated in Section IV, site specific acute and chronic WQC can be calculated for the site. The site specific criteria are calculated as the state standards times the WER. For the receiving water downstream of the site, the average WER is 2.6, resulting in a site specific acute WQC of 614.1 μg/L and a site specific chronic WQC of 37.2 μg/L (Table 4).

Table 4. Summary of values for corresponding acute[a] and chronic[b] standards, WER, and resulting site specific standards in receiving water samples downstream of the plant. The Illinois acute and chronic standards for Nickel are based on hardness dependent equations. The average for samples collected in this study are based on the average measured hardness in samples collected for the BLM analysis. Also shown are the site-specific values based on a hardness of 359, which was assigned by the State of Illinois for this site.

Sample Date	Sample Location	Hardness	Nickel Acute[a] Standard	Nickel Chronic[b] Standard	Water Effect Ratio	Site Specific Acute Standard	Site Specific Chronic Standard
		mg/L as $CaCO_3$	μg/L	μg/L		μg/L	μg/L
8/26	RD at Rock Springs	357	241.7	14.7	2.6	628.5	38.1
9/9	RD at Rock Springs	360	243.5	14.8		633.0	38.4
8/26	RD at Lincoln	332	227.3	13.8	2.6	591.1	35.8
9/9	RD at Lincoln	341	232.5	14.1		604.6	36.6
	Average (this study)	**347.5**	**236.2**	**14.3**	**2.6**	**614.1**	**37.2**
	Site specific values using Illinois EPA-assigned critical hardness	**359**	**242.9**	**14.7**	**2.6**	**631.5**	**38.2**

Notes:

[a]: Nickel Acute Standard = exp[A+B*ln(H)] * 0.998 (where A=0.5173; B=0.846)

[b]: Nickel Chronic Standard = exp[A+B*ln(H)] * 0.997 (where A= -2.286; B=0.846)

VII. CONCLUSIONS

Water quality factors such as pH, alkalinity, ion content, and the presence of natural organic matter have been shown to affect metal toxicity. However, the WCQ for many metals consider only hardness, making them potentially over-protective or under-protective for many site waters. The BLM is a mechanistic framework suitable for a number of metals, including Nickel, which allows for the consideration of many additional water quality factors. The BLM has been adopted by US EPA in the most recently updated metals criteria (US EPA, 2007). For metals that do not yet have an approved WQC approach, the BLM can be used to calculate a WER adjustment to derive site specific acute and chronic criteria. Application of the Nickel BLM to calculate Nickel toxicity in samples taken from the Sangamon River downstream of the District's Main Plant compared to a reference water results in a calculated average WER of 2.6. This WER results in a site specific acute criterion of 614.1 μg/L and a site specific chronic criterion of 37.2 μg/L at a hardness equal to 347.5 mg/L. Utilizing the Illinois EPA-assigned hardness of 359 mg/L, the WER results in a corresponding acute criterion of 631.5 μg/L and a site specific chronic criterion of 38.2 μg/L.

VIII. REFERENCES

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Attachment B

OSU Aquatic Toxicology Laboratory
33972 Texas St. SW
Albany, Oregon 97321 USA
Office 541-737-2565
Lab 541-926-1254

Oregon State UNIVERSITY OSU

Title:	Water-Effect Ratio (WER) Testing of Acute Nickel Toxicity in Site Effluent Water and Laboratory Water to the Cladoceran, *Ceriodaphnia dubia*, under Static Test Conditions
Testing Facility:	Oregon State University Aquatic Toxicology Laboratory (OSU AquaTox) 33972 Texas Street SW Albany, OR 97321 USA
Study Sponsor:	Sanitary District of Decatur 501 S. Dipper Lane Decatur, Illinois 62522
Sponsor's Study Officer:	Timothy R. Kluge, Technical Director
Principal Investigator:	William Stubblefield, Ph.D.
Study Director:	Allison Cardwell

1.0 INTRODUCTION

1.1 Objective

To utilize the water-effect ratio (WER) procedure to determine the acute effects of nickel on the freshwater cladoceran, *Ceriodaphnia dubia*, in site effluent water and laboratory reconstituted water, under static test conditions.

1.2 Experimental Approach

C. dubia will be continuously exposed to differing concentrations of nickel in both site effluent water and laboratory reconstituted water during acute aqueous exposures.

1.3 Test Substance

The test substance will be in the form of nickel chloride hexahydrate ($NiCl_2$ x $6H_2O$; CAS # 7791-20-0).

2.0 BASIS AND TEST SYSTEM

2.1 Basis

This protocol is designed to comply with USEPA testing guidance (USEPA 2002) and WER procedures (USEPA 1994).

2.2 Test Species

1. Species: Cladoceran/Water Flea (*Ceriodaphnia dubia*).
2. Number: A total of 20 organisms will be tested for each treatment and control (four replicates per treatment).
3. *C. dubia* will start as less than 24 hr old neonates.
4. Source: *C. dubia* are cultured at Oregon State University's Aquatic Toxicology Lab (OSU AquaTox, Albany, OR).
5. Culture/Holding Water: For acclimation of organisms to the expected hardness of the site effluent water, *C. dubia* adults are maintained in a mass culture in very hard reconstituted water (nominal hardness, alkalinity, and pH of approximately 305 mg/L as $CaCO_3$, 225 mg/L as $CaCO_3$, and 8.5, respectively). In order to track reproduction of test organisms, at least two weeks prior to testing, organisms will be maintained individually in 30 mL plastic containers in an environmental chamber.
6. Feeding: No feeding will occur during the conduct of the test. Prior to the initiation of the test, < 24 hr old neonates will be grouped together and fed a suspension of Yeast/Trout Chow/Cereal leaves mixture (YTC) and algae suspension (*Pseudokirchneriella subcapitata*, 1:1) for at least 2 hours prior to the test. Organisms (which have been acclimated to very hard reconstituted water) for the site effluent testing will be acclimated to the site water for as long as possible prior to the test without compromising the time constraints of first use of the site water.
7. Procedure for identification: *C. dubia* have been verified to species by the original organism supplier.

2.3 Test Diet

The organisms will not be fed during the toxicity test.

3.0 EXPOSURE SYSTEM

3.1 Route of Administration

Method: Appropriate volumes of nickel stock will be mixed with both the site effluent water and the laboratory reconstituted water to achieve nominal concentrations. Following the spiking of nickel to the site effluent, the waters will be serially diluted and allowed to equilibrate for 2-4 hours prior to use. Following spiking of nickel to the laboratory reconstituted water, the waters will be serially diluted and allowed to equilibrate for 1-3 hours.

Equipment: The laboratory reconstituted water will be prepared with reagent grade salts (see section 1.3) and will be weighed/apportioned using an electronic micro-balance and micro-pipettes.

Frequency: This is a static test. No water renewal will occur during the conduct of the test.

3.2 Dilution Water

Dilution water for the laboratory water test will be a very hard reconstituted laboratory water made from deionized water amended with the appropriate reagent grade salts ($CaSO_4 \cdot 2H_2O$, $MgSO_4$, KCl, and $NaHCO_3$) to achieve a nominal hardness, alkalinity, and pH of approximately 315 mg/L as $CaCO_3$, 225 mg/L as $CaCO_3$, and 8.0, respectively. Reconstituted water will be prepared as detailed in standard USEPA methods (USEPA 2002) with a Ca to Mg (Ca:Mg) ratio of 0.7.

The site effluent water will not be diluted.

3.3 Test Temperature

Test temperature will be 25 $\pm$ 2 °C. Testing will be conducted in a temperature-controlled environmental chamber.

3.4 Test Chamber

Test containers will be 30-mL plastic Soufflé cups containing 25-mL of test solution. Containers will be covered with Plexiglas to prevent contamination.

3.5 Photoperiod

Lighting for the entire test duration will be a photoperiod of 16-hours light and 8-hours dark, provided by cool-white or daylight illumination.

3.6 Dissolved Oxygen Concentrations

Dissolved oxygen concentrations will be maintained at $\geq$ 60 percent of saturation.

4.0 TEST DESIGN

4.1 Test Concentrations/Dosages

For each test (site effluent and laboratory water), five test treatments and a control will be tested using a 0.7 dilution scheme. The nominal test concentrations will be estimated based upon expected acute toxicity of nickel based upon the hardness and pH of the waters and historical data. Nominal test concentrations will be described in the raw data packet. A concurrent moderately hard reconstituted control water (USEPA 2002; without nickel) will also be tested.

4.2 Number of Test Organisms

For each test (site effluent and laboratory water), a total of 20 organisms will be tested in four replicates for each treatment and control. A concurrent moderately hard reconstituted water control (without nickel) will be tested in the same conditions as the site effluent and laboratory water. Five *C. dubia* will be randomly partitioned into each test vessel at the start of the test.

4.3 Bias Control

To control bias, test chambers will be numbered according to a 4 X 6 randomization sheet and placed in the environmental chamber.

4.4 Test Initiation

Following preparation of each concentration, solutions will be allocated to each replicate. Organisms will then be randomly allocated into each replicate until 5 organisms are in each chamber.

4.5 Chemical and Physical Monitoring

At a minimum, the following measurements will be made according to the methods laid out in OSU AquaTox SOPs:

1. Hardness, alkalinity, dissolved oxygen, temperature, conductivity, total ammonia, total residual chlorine, and pH will be measured in the site effluent water and the laboratory reconstituted water at test initiation. Hardness and pH of the control, one middle concentration and the highest concentration, will also be measured at the 48 hour renewal time point (of both new renewal waters and old waters) and at test termination.

2. A sample of the site effluent water and the laboratory reconstituted water will be collected for characterization of calcium, magnesium, sodium, potassium, chloride, sulfate, and dissolved organic carbon (DOC) and measured at an outside commercial laboratory.

3. Dissolved oxygen, temperature, conductivity, and pH will be measured daily in each treatment.

4.6 Biological Monitoring

Observations of live and dead organisms will be recorded daily. Dead organisms will be removed immediately following observation.

4.7 Analytical Chemistry

Samples for nickel analysis will be collected from each treatment according to the following schedule: On Day 0 (initiation), samples for total recoverable (unfiltered and acidified with concentrated nitric acid to a pH < 2) and dissolved (filtered through 0.45 µm-porosity filter prior to acidification) will be collected separately into a 15 ml polypropylene conical tube from each treatment. Samples for analysis of total and dissolved nickel will also be collected from old test waters (from a composite of the four replicates for each treatment) at test termination. Filters (0.45 µm-porosity) used for dissolved metal collections will be flushed with 5 ml of sample prior to sample collection. Total recoverable and dissolved nickel samples will be analyzed via Inductively Coupled Plasma Optical Emission Spectrometry or Mass Spectrometry (ICP-OES/MS).

4.8 Test Duration

The test duration will be 48 hours ± 1 hour.

4.9 Quality Criteria

- The test will not be considered valid if control mortality exceeds 10%.
- The dissolved oxygen concentration must be > 60 percent saturation.
- There must be evidence that the temperature, dissolved oxygen, and concentration of the test substance being tested have been satisfactorily maintained, based on time-weighted averages, over the test period.

5.0 DATA ANALYSIS

Statistical analysis (hypothesis testing) of the test data will be conducted using a computer program. A statistical test (as determined by the USEPA Decision Tree in acute toxicity test guidance [USEPA, 2002]) will be used to test for significant differences in the survival among test treatments and controls. EPA methodology (2002) dictates the flowchart for determination of the LC50 for multi-effluent concentration acute toxicity tests and this flowchart coincides with the statistical methodology described in the WER guidance (1994).The no observable effect concentration (NOEC) and lowest observable effect concentration (LOEC) will be calculated on the basis of survival ($p < 0.05$). In addition, a median lethal concentration (LC50) will be calculated along with the determination of outliers and the need for data transformation (i.e., arc sine, square root, logarithmic, etc.).

The experimentally determined WER will typically be a ratio of endpoints determined at two different hardnesses and will thus include contributions from a variety of differences between the two waters, including hardness. The WER will be calculated as discussed in the guidance document (USEPA 1994). The use of the USEPA very hard reconstituted laboratory water as a reference water is a reasonably close match to the site effluent hardness. The reference water

LC50 may be further adjusted to match the site water hardness using the hardness slope for the Ni standard.

6.0 TEST REPORT

The report will be a typed document describing the results of the test and will be signed by the Principal Investigator and Study Director. The report will include, but not be limited to, the following:

- Name and address of the test facility;
- Dates of test initiation, completion, and/or termination;
- Objectives of the study as stated in the test protocol, including any changes from the protocol;
- Statistical methods used in data analysis;
- Identification of the test substances (by name, CAS number, or code number) and description of substance purity, strength, composition, stability, solubility, and/or other appropriate characteristics documented by the Study Sponsor (location of documentation shall be specified);
- A description of the methods used during testing;
- A description of the test system used including, where applicable, algal density or biomass, source of supply, species, strain, sub-strain, age, and procedure for identification;
- A description of the exposure concentrations, dosing regimen, route of administration, and duration of exposure;
- A description of all circumstances that may have affected the quality and/or integrity of the data;
- The name of the Principal Investigator and Study Director and the names of other scientists, professionals, or supervisory personnel (e.g. task manager, senior biomonitoring technician) involved in the study;
- A description of the methods of data analysis; a summary and analysis of the data, and a statement of the conclusions drawn from the analysis;
- Signature and date of the Study Director and/or other professionals involved in the study as required by the testing facility or Sponsor;
- The location(s) where all specimens, raw data, and final report are to be stored;
- A statement of Quality Assurance

7.0 RECORD RETENTION

All records will be maintained and archived in the OSU AquaTox archives in accordance with OSU AquaTox SOP 5403.

8.0 PROTOCOL AMENDMENTS AND DEVIATIONS

All changes (i.e., amendments, deviations, and final report revisions) of the approved protocol, plus the reasons for the changes, must be documented in writing. The changes will be signed and dated by the Study Director and maintained with the protocol.

OSU Aquatic Toxicology Laboratory
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Oregon State UNIVERSITY OSU

Title:	Water-Effect Ratio (WER) Testing of Acute Nickel Toxicity in Site Effluent Water and Laboratory Water to the Fathead Minnow, *Pimephales promelas*, under Static-Renewal Test Conditions
Testing Facility:	Oregon State University Aquatic Toxicology Laboratory (OSU AquaTox) 33972 Texas Street SW Albany, OR 97321 USA
Study Sponsor:	Sanitary District of Decatur 501 S. Dipper Lane Decatur, Illinois 62522
Sponsor's Study Officer:	Timothy R. Kluge, Technical Director
Principal Investigator:	William Stubblefield, Ph.D.
Study Director:	Allison Cardwell

1.0 INTRODUCTION

1.1 Objective

To utilize the water-effect ratio (WER) procedure to determine the acute effects of nickel on the fathead minnow, *Pimephales promelas*, in site effluent water and laboratory reconstituted water, under static-renewal test conditions.

1.2 Experimental Approach

P. promelas will be continuously exposed to differing concentrations of nickel in both site effluent water and laboratory reconstituted water during acute aqueous exposures.

1.3 Test Substance

The test substance will be in the form of nickel chloride hexahydrate ($NiCl_2$ x $6H_2O$; CAS # 7791-20-0).

2.0 BASIS AND TEST SYSTEM

2.1 Basis

This protocol is designed to comply with USEPA testing guidance (USEPA 2002) and WER procedures (USEPA 1994).

2.2 Test Species

1. Species: Fathead Minnow (*Pimephales promelas*).
2. Number: Each test will consist of a total of 40 organisms per treatment, with each treatment containing four replicates. Ten larval fish will be partitioned into each vessel at the start of the test.
3. Age: Larval fish, *P. promelas* (~7-14 days old), at start of test.
4. Source: *P. promelas* will be obtained from in-house cultures located at OSU AquaTox.
5. Holding Conditions: *P. promelas* adults are maintained in brood tanks using a continuous flow-through system with natural well water that is saturated with dissolved oxygen and has a nominal hardness, alkalinity, and pH of approximately 100 mg/L as $CaCO_3$, 100 mg/L as $CaCO_3$, and 7.8, respectively.
6. Larval fish will be held in holding tanks with feeding and water renewal prior to use in the toxicity test. Fish will be acclimated to the very high hardness conditions by daily water renewals, increasing hardness by approximately 50 mg/L as $CaCO_3$, daily. Fish will be approximately 7-14 days old at test initiation. Organisms (which have been acclimated to very hard reconstituted water) for the site effluent testing will be acclimated to the site water for as long as possible prior to the test without compromising the time constraints of first use of the site water.
7. Feeding: At the 48-hour time point in testing, 0.2 mL brine shrimp nauplii will be fed to each test chamber. Feeding will be allowed for a minimum of 2 hours, prior to solution renewal.

8. Procedure for identification: *P. promelas* have been verified to species by the original organism supplier.

2.3 Test Diet

Brine shrimp cysts (Brine Shrimp Direct, Ogden UT, USA) are hatched in the laboratory and are typically < 30 hours old (*Artemia* nauplii) when fed to the test organisms. A sample of newly-hatched *Artemia* nauplii are chemically analyzed on an annual basis for total metals, organochlorine pesticides, and PCBs as per OSU AquaTox's Standard Operation Procedures (SOPs).

3.0 EXPOSURE SYSTEM

3.1 Route of Administration

Method: Appropriate volumes of nickel stock will be mixed with both the site effluent water and the laboratory reconstituted water to achieve nominal concentrations. Following the spiking of nickel to the site effluent, the waters will be serially diluted and allowed to equilibrate for 2-4 hours prior to use. Following spiking of nickel to the laboratory reconstituted water, the waters will be serially diluted and allowed to equilibrate for 1-3 hours.

Equipment: The laboratory reconstituted water will be prepared with reagent grade salts (see section 1.3) and will be weighed/apportioned using an electronic micro-balance and micro-pipettes.

Frequency: An 80% renewal of control and treatment solutions will occur at 48 hours by siphoning out 80% of the old water and waste and pouring freshly prepared test solutions (following the aging periods stated in the method above), as appropriate, back into the chambers.

3.2 Dilution Water

Dilution water for the laboratory water test will be a very hard reconstituted laboratory water made from deionized water amended with the appropriate reagent grade salts ($CaSO_4 \cdot 2H_2O$, $MgSO_4$, KCl, and $NaHCO_3$) to achieve a nominal hardness, alkalinity, and pH of approximately 315 mg/L as $CaCO_3$, 225 mg/L as $CaCO_3$, and 8.0, respectively. Reconstituted water will be prepared as detailed in standard USEPA methods (USEPA 2002) with a Ca to Mg (Ca:Mg) ratio of 0.7.

The site effluent water will not be diluted.

3.3 Test Temperature

Test temperature will be 25 $\pm$ 2 °C. Testing will be conducted in a temperature-controlled environmental chamber.

3.4 Test Chamber

Test containers will be 250-mL glass beakers containing 200-mL of test solution. Containers will be covered with Plexiglas to prevent contamination.

3.5 Photoperiod

Lighting for the entire test duration will be a photoperiod of 16-hours light and 8-hours dark, provided by cool-white or daylight illumination.

3.6 Dissolved Oxygen Concentrations

Dissolved oxygen concentrations will be maintained at ≥ 60 percent of saturation.

4.0 TEST DESIGN

4.1 Test Concentrations/Dosages

For each test (site effluent and laboratory water), five test treatments and a control will be tested using a 0.7 dilution scheme. The nominal test concentrations will be estimated based upon expected acute toxicity of nickel based upon the hardness and pH of the waters and historical data. Nominal test concentrations will be described in the raw data packet. A concurrent moderately hard reconstituted control water (USEPA 2002; without nickel) will also be tested.

4.2 Number of Test Organisms

For each test (site effluent and laboratory water), a total of 40 organisms will be tested in four replicates for each treatment and control. A concurrent moderately hard reconstituted water control (without nickel) will be tested in the same conditions as the site effluent and laboratory water. Ten larval fish will be randomly partitioned into each test vessel at the start of the test.

4.3 Bias Control

To control bias, test chambers will be numbered according to a 4 X 7 randomization sheet and placed in the environmental chamber.

4.4 Test Initiation

Following preparation of each concentration, solutions will be allocated to each replicate. Organisms will then be randomly allocated into each replicate until 10 organisms are in each chamber.

4.5 Chemical and Physical Monitoring

At a minimum, the following measurements will be made according to the methods laid out in OSU AquaTox SOPs:

1. Hardness, alkalinity, dissolved oxygen, temperature, conductivity, total ammonia, total residual chlorine, and pH will be measured in the site effluent water and the

laboratory reconstituted water at test initiation. Hardness and pH of the control, one middle concentration and the highest concentration, will also be measured at the 48 hour renewal time point (of both new renewal waters and old waters) and at test termination.

2. A sample of the site effluent water and the laboratory reconstituted water will be collected for characterization of calcium, magnesium, sodium, potassium, chloride, sulfate, and dissolved organic carbon (DOC) and measured at an outside commercial laboratory.

3. Dissolved oxygen, temperature, conductivity, and pH will be measured daily in each treatment.

4.6 Biological Monitoring

Observations of live and dead organisms will be recorded daily. Dead organisms will be removed immediately following observation.

4.7 Analytical Chemistry

Samples for nickel analysis will be collected from each treatment according to the following schedule: On Day 0 (initiation), samples for total recoverable (unfiltered and acidified with concentrated nitric acid to a pH < 2) and dissolved (filtered through 0.45 µm-porosity filter prior to acidification) will be collected separately into a 15 ml polypropylene conical tube from each treatment. Samples for analysis of total and dissolved nickel will also be collected on Day 2 from old test waters (from a composite of the four replicates for each treatment) and freshly prepared renewal waters. Total and dissolved metals samples will also be taken at test termination. Filters (0.45 µm-porosity) used for dissolved metal collections will be flushed with 5 ml of sample prior to sample collection. Total recoverable and dissolved nickel samples will be analyzed via Inductively Coupled Plasma Optical Emission Spectrometry or Mass Spectrometry (ICP-OES/MS).

4.8 Test Duration

The test duration will be 96 hours ± 1 hour.

4.9 Quality Criteria

- The test will not be considered valid if control mortality exceeds 10%.
- The dissolved oxygen concentration must be > 60 percent saturation.
- There must be evidence that the temperature, dissolved oxygen, and concentration of the test substance being tested have been satisfactorily maintained, based on time-weighted averages, over the test period.

5.0 DATA ANALYSIS

Statistical analysis (hypothesis testing) of the test data will be conducted using a computer program. A statistical test (as determined by the USEPA Decision Tree in acute toxicity test guidance [USEPA, 2002]) will be used to test for significant differences in the survival among test treatments and controls. EPA methodology (2002) dictates the flowchart for determination of the LC50 for multi-effluent concentration acute toxicity tests and this flowchart coincides with the statistical methodology described in the WER guidance (1994).The no observable effect concentration (NOEC) and lowest observable effect concentration (LOEC) will be calculated on the basis of survival ($p < 0.05$). In addition, a median lethal concentration (LC50) will be calculated along with the determination of outliers and the need for data transformation (i.e., arc sine, square root, logarithmic, etc.).

The experimentally determined WER will typically be a ratio of endpoints determined at two different hardnesses and will thus include contributions from a variety of differences between the two waters, including hardness. The WER will be calculated as discussed in the guidance document (USEPA 1994). The use of the USEPA very hard reconstituted laboratory water as a reference water is a reasonably close match to the site effluent hardness. The reference water LC50 may be further adjusted to match the site water hardness using the hardness slope for the Ni standard.

6.0 TEST REPORT

The report will be a typed document describing the results of the test and will be signed by the Principal Investigator and Study Director. The report will include, but not be limited to, the following:

- Name and address of the test facility;
- Dates of test initiation, completion, and/or termination;
- Objectives of the study as stated in the test protocol, including any changes from the protocol;
- Statistical methods used in data analysis;
- Identification of the test substances (by name, CAS number, or code number) and description of substance purity, strength, composition, stability, solubility, and/or other appropriate characteristics documented by the Study Sponsor (location of documentation shall be specified);
- A description of the methods used during testing;
- A description of the test system used including, where applicable, algal density or biomass, source of supply, species, strain, sub-strain, age, and procedure for identification;
- A description of the exposure concentrations, dosing regimen, route of administration, and duration of exposure;
- A description of all circumstances that may have affected the quality and/or integrity of the data;
- The name of the Principal Investigator and Study Director and the names of other scientists, professionals, or supervisory personnel (e.g. task manager, senior biomonitoring technician) involved in the study;
- A description of the methods of data analysis; a summary and analysis of the data, and a statement of the conclusions drawn from the analysis;
- Signature and date of the Study Director and/or other professionals involved in the study as required by the testing facility or Sponsor;
- The location(s) where all specimens, raw data, and final report are to be stored;
- A statement of Quality Assurance

7.0 RECORD RETENTION

All records will be maintained and archived in the OSU AquaTox archives in accordance with OSU AquaTox SOP 5403.

8.0 PROTOCOL AMENDMENTS AND DEVIATIONS

All changes (i.e., amendments, deviations, and final report revisions) of the approved protocol, plus the reasons for the changes, must be documented in writing. The changes will be signed and dated by the Study Director and maintained with the protocol.

9.0 LITERATURE CITED

USEPA. 1994. Interim Guidance on Determination and Use of Water-Effect Ratios for Metals. Office of Water. Washington, D.C. EPA-823-B-94-001.

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

10.0 SPONSOR AND STUDY DIRECTOR APPROVAL

Sponsor :

Print Name: ______________________
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Date: 18 APRIL 2014

Study Director:

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Date: 18 April 2014

Comment 1. Thank you for clarifying.

Comment 2. It does not appear as though the calcium or magnesium values included in Table 3 of the "Estimate of the BLM Adjustment to the Nickel Criterion for the Sanitary District of Decatur, Illinois" document contained within the "WER study plan with attachments rev 4_23_14.docx" file provided on April 23, 2014 have changed since the April 16, 2013 version of the "Estimate" report. If there is a newer version of the report that includes these revisions, please feel free to forward this to EPA.

In addition, EPA looks forward to receiving the information necessary to verify the performance of the nickel BLM.

Comment 3. EPA understands that the proposal is to modify IL's criterion and not the national recommended criterion.

EPA acknowledges that IL's acute nickel criterion to protect aquatic life at a hardness of 50 mg/L is 45.9 µg/L and that Keithly *et al.*'s study identified a nickel LC50 value of 81 µg/L at 50 mg/L of hardness. However, during development of its nickel criterion, IL identified a *Ceriodaphnia* genus mean acute value (GMAV) (normalized to a hardness of 50 mg/L) of 30.16 µg/L. This GMAV was derived using data generated by Schubauer-Berigan *et al*., who identified *C. dubia* LC50 values of 32.6 µg/L (at pH = 7.1) and 3.03 µg/L (at pH = 8.6) (after normalizing to a hardness of 50 mg/L). Therefore, based on other data in the scientific literature, there appears to be uncertainty around the concentration of nickel necessary to generate toxic effects in *C. dubia* at a hardness of 50 mg/L.

If the Sanitary District of Decatur (SDD) wishes to pursue the use of *C. dubia* as a test organism, it may be possible to perform initial range-finding tests in very hard water and site water to determine how the LC50 values compare to IL's hardness-adjusted CMC. If these tests demonstrate that *C. dubia*'s LC50 value is less than or equal to the CMC, then the use of an alternative test organism (such as *Daphnia* sp.) would be consistent with EPA's guidance.

Comment 4. Thank you for clarifying and updating the test protocol.

Comment 5. IL's chronic nickel criterion was developed using toxicological data, not an acute-chronic ratio.

Given that SDD proposes to modify a chronic criterion, a chronic WER seems as though it would be most directly applicable.

Comment 6. Thank you for addressing this comment.

Comment 7. Can you please provide additional information on site water hardness (vs. proposed test water)? While EPA agrees that the waters are similar, additional analysis of the difference in hardness values and any effects on WER calculations should be explored. In particular, EPA recommends considering EPA's guidance document entitled "Use of the WER Procedure with Hardness Equations" for any further adjustments to hardness.

Comment 8. Thank you for addressing this comment.

Comment 9. Thank you for addressing this comment.

Comment 10. Calcium precipitation has been reported in testing using EPA hard water. Therefore, EPA recommends that hardness (as well as pH) are monitored at the beginning and end of testing. It appears that SDD's sampling protocol will address EPA's major concern, although researchers should note when and if precipitation occurs. If precipitation is common and significant, it may be advantageous to conduct sampling of ions at the end of the test in a subset of test waters.

In addition, EPA notes that static test conditions are susceptible to low dissolved oxygen (DO) conditions and that SDD proposes to monitor DO (as well as temperature, conductivity, and pH) daily over the course of the procedure. How will these measurements be carried out? Will probes be used to measure DO and other physiochemical variables in experimental chambers? If so, what steps will be taken to prevent the introduction of probes from inadvertently influencing experimental results (*e.g.*, via material transfer from one test chamber to another)? Or, will chemistry controls (*i.e.*, treatment waters in chambers without organisms) be used to measure physiochemical variables, including DO?

Comment 11. It is unclear which document "the newest EPA acute version (2002)" is referencing. Will SDD please provide the full citation and/or a link to the document?

Comment 12. It is not clear whether the age of fathead minnows used in the proposed test will be similar to the ages of the organisms used to derive IL's criteria and/or the nickel BLM. What steps will be taken to prevent age-related differences in nickel sensitivity from influencing the WER?