# **EXHIBIT G**

Interim Report December 19, 2012

# Sanitary District of Decatur

501 DIPPER LANE • DECATUR, ILLINOIS 62522 • 217/422-6931 • FAX: 217/423-8171

December 19, 2012

Illinois Environmental Protection Agency Bureau of Water Compliance Assurance Section, MC #19 1021 North Grand Avenue East P.O. Box 19276 Springfield, Illinois 62794-9276

Re: NPDES Permit IL0028321

IPCB Order PCB 09-125

Interim Report

Dear Sir or Madam:

Enclosed is the Interim Report regarding compliance with nickel and zinc limits required by Special Condition 18 of the Sanitary District of Decatur's NPDES Permit and the Pollution Control Board Order in PCB 09-125.

Please contact me at 422-6931 ext. 214 or at <u>timk@sdd.dst.il.us</u> if you have any questions regarding this report.

Sincerely,

Timothy R. Kluge, P.E.

**Technical Director** 

cc: Rick Pinneo, IEPA (via email)

Bob Mosher, IEPA (via email)

SDD File

# Sanitary District of Decatur Nickel and Zinc Limits December 2012 Interim Report

The modified NPDES permit for the Sanitary District of Decatur that became effective July 1, 2009 contains limits for nickel and zinc and a one-year compliance schedule extension for meeting the limits. Special Condition 17 requires the District to achieve compliance with final nickel and zinc effluent limitations by July 1, 2010. This Special Condition also notes that the permit may be modified to include revised compliance dates in Pollution Control Board orders, and that prior to such permit modification, the revised dates in the appropriate orders shall govern the Permittee's compliance.

On January 7, 2010 the Illinois Pollution Control Board granted a variance to the District allowing additional time to comply with final permit limits (PCB 09-125). The final compliance date contained in the Board Order is July 1, 2014. The District's NPDES Permit has not yet been modified or reissued to incorporate the variance. The Board Order also requires that an interim report be submitted to Illinois EPA by January 1, 2013. This report is submitted to meet both the permit and variance requirements.

### **Plant Influent and Effluent Sampling**

Concentration, mg/L

0

Ongoing influent sampling for nickel and zinc continues at a frequency of twice monthly, and effluent sampling is done five days per week according to NPDES monitoring requirements. A summary of influent and effluent values during the past twelve months is shown below.

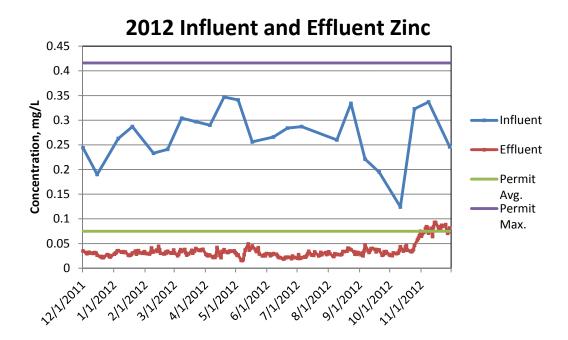
2012 Influent and Effluent Nickel

5/1/2012

A11/2012

212/2012/2012





Data shows that the plant effluent is not able to consistently meet the current nickel permit limit. Effluent zinc concentrations remain at or below the permit limit.

### **Receiving Stream Sampling**

Upstream and downstream sampling continues at a twice monthly frequency to provide a more complete picture of nickel and zinc in the Sangamon River. One upstream and four downstream sampling sites are being monitored. A summary of 2011-2012 river monitoring data is attached. Downstream nickel results remain high during times of low upstream river flow; low flows have prevailed during most of the past two years. With one exception, upstream and downstream zinc results during 2011 and 2012 have been below the Illinois water quality standard.

### **Pretreatment Ordinance Limits**

The District's pretreatment ordinance was amended in October 2009 as noted in previous interim reports.

### **Stream Flow-Based Compliance Options**

The District continues investigation of flow-based permit limits, to take advantage of upstream flow for mixing when it is available. A USGS flow gauging station is located about two miles upstream of the District's discharge point, and provides near-real time flow information. A proposal for flow-based limits will be a part of relief requested from the Pollution Control Board.

## **Water Quality Standard Investigations**

The District is in the final stages of preparing a petition for a site-specific nickel standard, which we expect to file with the Pollution Control Board in the very near future. Comments and questions received from U.S. EPA on December 7, 2012 are being evaluated.

We are also following the Pollution Control Board rulemaking currently underway to correct an error found in the existing zinc water quality standard. If the Board adopts the corrected standard, utilizing the corrected number to determine our permit limit should provide further assurance of compliance.

### **Industrial Source Sampling and Investigations**

Sampling at Archer Daniels Midland Company for metals continues at a frequency of twice monthly and other industries discharging metals are sampled quarterly. Sample results obtained from ADM within the past two years are attached.

The District's operating permit issued to ADM was modified on November 18, 2009 and again on June 17, 2010 to reflect the new limits and provide a compliance schedule for meeting the limits. Final local limits will be determined following Board action on the District's site-specific WQS request.

Both ADM and Tate & Lyle formerly utilized zinc as part of their cooling tower treatment programs, and both have eliminated or greatly reduced zinc in their towers. At this time, both industries are meeting the zinc pretreatment limit. ADM is continuing to investigate the possible impact of the zinc limit on their planned wasting of solids from their pretreatment system to the District's collection system.

The discharge from ADM is by far the most significant industrial source of nickel. ADM has been very active in seeking treatment technology for nickel removal, involving plant management and research department personnel in addition to environmental compliance and legal staff. District staff met with ADM personnel several times during the past year, most recently on November 30, 2012. The District's pretreatment permit requires semi-annual reports of ADM's investigations, and the most recent report is attached. Completed and anticipated modifications made by ADM are listed on page 4 of the report.

### **Additional Pretreatment Limit Investigations**

Pretreatment ordinance limits adopted in 2009 were adopted as total (rather than soluble) limits based on review of soluble/insoluble data. Refinement of pretreatment limits is an ongoing process and will depend on final permit limits as well as treatment technologies that might be employed by industrial users.

### **Compliance Plan**

In summary, the District's compliance plan includes the following:

- 1. Continue to work with ADM to implement nickel discharge reductions and removal technologies. ADM's November 30, 2012 Interim Report describes the completed and planned reductions.
- 2. Complete and file a petition for a site-specific water quality standard for nickel, based on bioavailability. We have been working with Illinois EPA to address questions and comments through the summer and fall of 2012. Currently we are evaluating comments and questions received from U.S. EPA on December 7, 2012.
- 3. The Board petition will contain a request for variable permit limits based on the amount of flow available in the Sangamon River.

Nickel and Zinc River Data 2011-2012

|          |          |           | Divers  | Divers  |           | Divers   |          |          |           | Divers  | Divers   | I         | D:       |          |          |                      |
|----------|----------|-----------|---------|---------|-----------|----------|----------|----------|-----------|---------|----------|-----------|----------|----------|----------|----------------------|
|          |          |           | River   | River   |           | River    |          |          |           | River   | River    |           | River    |          |          |                      |
|          | Plant    | River     | 100 yds | 600 yds |           | Rock     | River    | Plant    | River     | 100 yds | 600 yds  |           | Rock     | River    | Plant    | River                |
|          | Final    | Up-       | Down    | Down-   | Steven's  | Springs  | Wyckle's | Final    | Up-       | Down    | Down-    | Steven's  | Springs  | Wyckle's | Final    | Up-                  |
|          | Effluent | stream    | stream  | stream  | Creek     | Bridge   | Road     | Effluent | stream    | stream  | stream   | Creek     | Bridge   | Road     | Effluent | stream               |
| Sample   | Nickel   | Nickel    | Nickel  | Nickel  | Nickel    | Nickel   | Nickel   | Zinc     | Zinc      | Zinc    | Zinc     | Zinc      | Zinc     | Zinc     | Flow     | Flow                 |
| Date     | mg/L     | mg/L      | mg/L    | mg/L    | mg/L      | mg/L     | mg/L     | mg/L     | mg/L      | mg/L    | mg/L     | mg/L      | mg/L     | mg/L     | mgd      | ft <sup>3</sup> /sec |
| 01/13/11 | 0.0181   | <0.00131  | 0.00519 | 0.00495 | <0.00131  | 0.00426  | 0.00504  | 0.0503   | <0.00660  |         | 0.0152   | <0.00660  | 0.0133   | 0.0149   | 29.48    | 121                  |
| 01/27/11 | 0.0218   | <0.00131  | 0.0144  | 0.0138  | <0.00131  | 0.0113   | 0.0102   | 0.0773   | <0.00660  |         | 0.0481   | <0.00660  | 0.0394   | 0.0350   | 30.71    | 3.9                  |
| 02/10/11 | 0.0214   | <0.00131  | 0.0141  | 0.0128  | <0.00131  | 0.0112   | 0.00971  | 0.0701   | <0.00660  |         | 0.0413   | 0.00761   | 0.0364   | 0.0313   | 27.94    | 5.4                  |
| 02/24/11 | 0.0132   | 0.00160   | 0.00242 | 0.00252 | 0.00150   | 0.00214  | 0.00205  | 0.0406   | 0.00841   | 0.0106  | 0.0108   | 0.0138    | 0.0114   | 0.00992  | 44.38    | 1970                 |
| 3/10/11  | 0.0123   | 0.00169   | 0.00194 | 0.00198 | 0.00153   | 0.00184  | 0.00208  | 0.0321   | 0.00972   | 0.00978 | 0.00992  | 0.0103    | 0.00974  | 0.0100   | 47.51    | 2900                 |
| 3/24/11  | 0.0132   | <0.00131  | 0.00133 | 0.00133 | <0.00131  | <0.00131 | <0.00131 | 0.0161   | <0.00660  |         | <0.00660 | <0.00660  | <0.00660 | <0.00660 | 33.28    | 667                  |
| 4/7/11   | 0.0163   | <0.00131  | 0.00343 | 0.00252 | <0.00131  | 0.00241  | 0.00237  | 0.0246   | <0.00660  | 0.00884 | 0.00689  | <0.00660  | 0.00732  | 0.00691  | 30.62    | 326                  |
| 4/21/11  | 0.0118   | <0.00131  | 0.00236 | 0.00195 | 0.00254   | 0.00157  | 0.00188  | 0.0215   | 0.00729   | 0.00878 | 0.00822  | 0.0170    | 0.00939  | 0.00934  | 52.22    | 2540                 |
| 5/5/11   | 0.0147   | 0.00177   | 0.00279 | 0.00238 | 0.00137   | 0.00218  | 0.00223  | 0.0295   | <0.00660  |         | 0.00862  | <0.00660  | 0.00760  | 0.00898  | 41.88    | 1670                 |
| 5/19/11  | 0.0125   | <0.00131  | 0.00211 | 0.00186 | <0.00131  | 0.00153  | 0.00150  | 0.0213   | <0.00660  |         | <0.00660 | <0.00660  | <0.00660 | 0.00777  | 32.29    | 1290                 |
| 6/9/11   | 0.0187   | <0.00131  | 0.00143 | 0.00194 | 0.00183   | 0.00162  | 0.00177  | 0.0434   | < 0.00660 |         | 0.00672  | <0.00660  | <0.00660 | 0.0124   | 29.12    | 1540                 |
| 6/23/11  | 0.0154   | 0.00210   | 0.00335 | 0.00307 | 0.00154   | 0.00280  | 0.00329  | 0.0203   | 0.0131    | 0.0134  | 0.0138   | 0.0112    | 0.0129   | 0.0155   | 36.23    | 800                  |
| 7/14/11  | 0.0170   | <0.00131  | 0.0118  | 0.0116  | < 0.00131 | 0.00886  | 0.00890  | 0.0242   | 0.00519   | 0.0162  | 0.0171   | <0.00660  | 0.0136   | 0.0130   | 27.12    | 200                  |
| 7/28/11  | 0.0188   | <0.00131  | 0.0187  | 0.0168  | < 0.00131 | 0.0158   | 0.0159   | 0.0255   | <0.00660  |         | 0.0219   | <0.00660  | 0.0205   | 0.0207   | 27.85    | 2.1                  |
| 8/11/11  | 0.0218   | 0.00143   | 0.0255  | 0.0212  | < 0.00131 | 0.0204   | 0.0199   | 0.0294   | <0.00660  | 0.0576  | 0.0292   | <0.00660  | 0.0266   | 0.0271   | 24.82    | 1.6                  |
| 8/25/11  | 0.0193   | <0.00131  | 0.0187  | 0.0190  | < 0.00131 | 0.0183   | 0.0189   | 0.0161   | <0.00660  |         | 0.0158   | <0.00660  | 0.0142   | 0.0137   | 24.19    | 1.1                  |
| 9/8/11   | 0.0233   | 0.00142   | 0.0208  | 0.0222  | < 0.00131 | 0.0207   | 0.0196   | 0.0341   | <0.00660  |         | 0.0303   | <0.00660  | 0.0279   | 0.0254   | 27.07    | 0.15                 |
| 9/14/11  | 0.0237   | 0.00132   | 0.0231  | 0.0235  | <0.00131  | 0.0228   | 0.0231   | 0.0460   | <0.00660  |         | 0.0438   | <0.00660  | 0.0413   | 0.0385   | 28.62    | 1.9                  |
| 10/6/11  | 0.0276   | 0.00140   | 0.0263  | 0.0265  | < 0.00131 | 0.0255   | 0.0259   | 0.0329   | <0.00660  |         | 0.0314   | < 0.00660 | 0.0296   | 0.0288   | 23.96    | 0.75                 |
| 10/20/11 | 0.0211   | <0.00131  | 0.0189  | 0.0195  | < 0.00131 | 0.0159   | 0.0181   | 0.0260   | 0.0107    | 0.0235  | 0.0238   | <0.00660  | 0.0193   | 0.0199   | 23.28    | 2.8                  |
| 11/3/11  | 0.0250   | 0.00197   | 0.0277  | 0.0304  | 0.00175   | 0.0260   | 0.0275   | 0.0322   | 0.0115    | 0.0314  | 0.0354   | < 0.00660 | 0.0281   | 0.0271   | 42.99    | 18                   |
| 11/17/11 | 0.0307   | <0.00131  | 0.0281  | 0.0283  | 0.00178   | 0.0273   | 0.0277   | 0.0368   | <0.00660  | 0.0285  | 0.0304   | < 0.00660 | 0.0275   | 0.0247   | 25.80    | 1.1                  |
| 12/1/11  | 0.0221   | < 0.00131 | 0.0177  | 0.0173  | <0.00131  | 0.0149   | 0.0149   | 0.0349   | 0.00728   | 0.0245  | 0.0230   | 0.00824   | 0.0207   | 0.0190   | 27.64    | 2.1                  |
| 1/5/12   | 0.0207   | <0.00131  | 0.0193  | 0.0206  | <0.00131  | 0.0170   | 0.0174   | 0.0355   | <0.00660  | 0.0328  | 0.0346   | <0.00660  | 0.0298   | 0.0278   | 27.19    | 4.1                  |
| 1/19/12  | 0.0245   | 0.00146   | 0.0164  | 0.0166  | 0.00135   | 0.0126   | 0.0127   | 0.0307   | 0.0265    | 0.0229  | 0.0240   | 0.00838   | 0.0203   | 0.0184   | 26.24    | 8.9                  |
| 2/9/12   | 0.0241   | <0.00131  | 0.00567 | 0.00496 | <0.00131  | 0.00480  | 0.00421  | 0.0329   | <0.00660  | 0.00944 | 0.00838  | < 0.00660 | 0.00788  | 0.00782  | 29.94    | 228                  |
| 2/23/12  | 0.0227   | <0.00131  | 0.0135  | 0.0147  | <0.00131  | 0.0118   | 0.0115   | 0.0343   | <0.00660  | 0.0213  | 0.0256   | < 0.00660 | 0.0182   | 0.0172   | 28.01    | 50                   |
| 3/8/12   | 0.0245   | <0.00131  | 0.0111  | 0.0111  | <0.00131  | 0.00964  | 0.00941  | 0.0338   | <0.00660  | 0.0167  | 0.0161   | < 0.00660 | 0.0149   | 0.0150   | 27.78    | 79                   |
| 3/22/12  | 0.0277   | <0.00131  | 0.0241  | 0.0211  | <0.00131  | 0.0180   | 0.0185   | 0.0399   | <0.00660  | 0.0501  | 0.0387   | < 0.00660 | 0.0245   | 0.0227   | 26.74    | 2.5                  |
| 4/5/12   | 0.0313   | <0.00131  | 0.0226  | 0.0226  | <0.00131  | 0.0205   | 0.0207   | 0.0260   | <0.00660  |         | 0.0227   | <0.00660  | 0.0185   | 0.0172   | 26.05    | 4.6                  |
| 4/19/12  | 0.0334   | <0.00131  | 0.0246  | 0.0238  | 0.00149   | 0.0187   | 0.0199   | 0.0375   | <0.00660  | 0.0331  | 0.0308   | <0.00660  | 0.0240   | 0.0216   | 26.08    | 4.2                  |
| 5/3/12   | 0.0262   | 0.00158   | 0.0120  | 0.0105  | <0.00131  | 0.00755  | 0.00770  | 0.0270   | 0.00690   | 0.0231  | 0.0194   | <0.00660  | 0.0148   | 0.0142   | 26.95    | 8.7                  |
| 5/17/12  | 0.0317   | 0.00156   | 0.00859 | 0.00888 | 0.00141   | 0.00775  | 0.00806  | 0.0450   | <0.00660  | 0.0160  | 0.0171   | <0.00660  | 0.0139   | 0.0148   | 25.37    | 97                   |
| 6/7/12   | 0.0319   | 0.00259   | 0.0182  | 0.0173  | 0.00402   | 0.0160   | 0.0169   | 0.0296   | 0.0106    | 0.0180  | 0.0181   | <0.00660  | 0.0163   | 0.0184   | 22.57    | 6.6                  |
| 6/21/12  | 0.0296   | 0.00136   | 0.0222  | 0.0218  | 0.00146   | 0.0215   | 0.0214   | 0.0225   | <0.00660  |         | 0.0165   | <0.00660  | 0.0164   | 0.0139   | 23.81    | 0.06                 |
| 7/5/12   | 0.0303   | 0.00164   | 0.0247  | 0.0240  | 0.00217   |          | 0.0232   |          | <0.00660  |         |          | <0.00660  |          | 0.0144   | 23.57    | 0.40                 |
| 7/19/12  | 0.0307   | 0.00195   | 0.0242  | 0.0236  | 0.00142   |          | 0.0235   | 0.0289   | <0.00660  |         | 0.0252   | <0.00660  |          | 0.0230   | 23.18    | 0.10                 |
| 8/9/12   | 0.0356   | 0.00147   | 0.0250  | 0.0252  | 0.00160   | 0.0256   | 0.0248   | 0.0283   | <0.00660  |         | 0.0227   | <0.00660  | 0.0232   | 0.0205   | 18.56    | 0.26                 |
| 8/23/12  | 0.0382   | 0.00185   | 0.0305  | 0.0305  | 0.00198   | 0.0302   | 0.0298   | 0.0374   | 0.00907   |         | 0.0324   | <0.00660  | 0.0314   | 0.0298   | 19.55    | 0.33                 |
| 9/6/12   | 0.0278   | 0.00206   | 0.0206  | 0.0212  | 0.00252   | 0.0169   | 0.0180   | 0.0471   | 0.0108    | 0.0253  | 0.0280   | 0.0100    | 0.0245   | 0.0229   | 20.73    | 1.3                  |
| 9/20/12  | 0.0289   | 0.00193   | 0.0228  | 0.0234  | 0.00160   | 0.0221   | 0.0226   | 0.0370   | 0.00772   |         | 0.0304   | <0.00660  | 0.0284   | 0.0280   | 18.57    | 0.27                 |
| 10/11/12 | 0.0280   | 0.00161   | 0.0192  | 0.0195  | 0.00150   | 0.0186   | 0.0180   | 0.0434   | <0.00660  |         | 0.0303   | <0.00660  | 0.0281   | 0.0260   | 18.38    | 0.27                 |
| 10/25/12 | 0.0330   | 0.00152   | 0.0212  | 0.0216  | 0.00136   | 0.0184   | 0.0182   | 0.0462   | 0.00772   | 0.0312  | 0.0310   | <0.00660  | 0.0276   | 0.0232   | 28.23    | 2.90                 |
| 11/8/12  | 0.0409   | 0.00156   | 0.0345  | 0.0345  | 0.00141   | 0.0316   | 0.0324   | 0.0711   | <0.00660  |         | 0.0778   | <0.00660  | 0.0707   | 0.0717   | 22.74    | 0.50                 |
| 11/29/12 | 0.0388   | 0.00168   | 0.0298  | 0.0307  | 0.00137   | 0.0287   | 0.0290   | 0.0815   | 0.00746   | 0.0649  | 0.0669   | 0.00783   | 0.0625   | 0.0603   | 22.74    | 0.41                 |

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|------------|-------------|---------------|-------------|-------------|
|            | ADIVI NIC   | Kei aliu Zili | t nesuits   |             |
|            | ADM Point A | ADM Point A   | ADM Point D | ADM Point D |
| Sample     | Nickel, Tot | Zinc, Tot     | Nickel, Tot | Zinc, Tot   |
| Date       | mg/L        | mg/L          | mg/L        | mg/L        |
| 1/5/2011   | 0.0629      | 0.53          | 0.0669      | 0.204       |
| 1/10/2011  | 0.0577      | 0.495         | 0.0666      | 0.188       |
| 2/7/2011   | 0.0836      | 0.756         | 0.0892      | 0.329       |
| 2/14/2011  | 0.0589      | 0.472         | 0.0598      | 0.18        |
| 3/7/2011   | 0.0773      | 0.447         | 0.0627      | 0.128       |
| 3/14/2011  | 0.086       | 0.51          | 0.1         | 0.449       |
| 4/4/2011   | 0.07        | 0.428         | 0.0841      | 0.387       |
| 4/20/2011  | 0.0687      | 0.33          | 0.0861      | 0.347       |
| 5/2/2011   | 0.0712      | 0.304         | 0.0809      | 0.302       |
| 5/9/2011   | 0.06        | 0.301         | 0.0712      | 0.3         |
| 6/6/2011   | 0.0648      | 0.285         | 0.0786      | 0.276       |
| 6/13/2011  | 0.0692      | 0.293         | 0.0809      | 0.314       |
| 7/11/2011  | 0.0542      | 0.226         | 0.0625      | 0.209       |
| 8/1/2011   | 0.0491      | 0.165         | 0.0621      | 0.172       |
| 8/8/2011   | 0.0567      | 0.215         | 0.074       | 0.242       |
| 9/1/2011   | 0.0662      | 0.285         | 0.0842      | 0.327       |
| 9/7/2011   | 0.0684      | 0.311         | 0.0884      | 0.344       |
| 10/3/2011  | 0.094       | 0.518         | 0.114       | 0.515       |
| 10/10/2011 | 0.0643      | 0.191         | 0.073       | 0.189       |
| 11/7/2011  | 0.0912      | 0.377         | 0.116       | 0.529       |
| 11/22/2011 | 0.221       | 1.28          | 0.136       | 0.623       |
| 12/1/2011  | 0.0917      | 0.416         | 0.11        | 0.492       |
| 12/5/2011  | 0.094       | 0.423         | 0.117       | 0.508       |
| 1/5/2012   | 0.0921      | 0.451         | 0.111       | 0.531       |
| 1/9/2012   | 0.0868      | 0.424         | 0.109       | 0.491       |
| 2/6/2012   | 0.121       | 0.441         | 0.134       | 0.488       |
| 2/13/2012  | 0.127       | 0.49          | 0.159       | 0.601       |
| 3/5/2012   | 0.128       | 0.431         | 0.15        | 0.493       |
| 3/12/2012  | 0.12        | 0.406         | 0.141       | 0.482       |
| 4/12/2012  | 0.169       | 0.621         | 0.191       | 0.705       |
| 4/19/2012  | 0.148       | 0.516         | 0.176       | 0.674       |
| 5/1/2012   | 0.0797      | 0.251         | 0.152       | 0.564       |
| 5/7/2012   | 0.137       | 0.494         | 0.141       | 0.448       |
| 6/4/2012   | 0.133       | 0.412         | 0.147       | 0.468       |
| 6/11/2012  | 0.12        | 0.366         | 0.144       | 0.452       |
| 7/2/2012   | 0.129       | 0.375         | 0.158       | 0.462       |
| 7/9/2012   | 0.109       | 0.322         | 0.132       | 0.402       |
| 8/1/2012   | 0.127       | 0.426         | 0.17        | 0.574       |
| 8/6/2012   | 0.097       | 0.193         | 0.12        | 0.242       |
| 9/6/2012   | 0.105       | 0.289         | 0.117       | 0.271       |
| 9/10/2012  | 0.479       | 0.531         | 0.165       | 0.559       |
| 10/1/2012  | 0.15        | 0.46          | 0.168       | 0.54        |
| 10/8/2012  | 0.129       | 0.421         | 0.152       | 0.444       |
| 11/1/2012  | 0.16        | 0.487         | 0.184       | 0.568       |
| 11/12/2012 | 0.158       | 0.444         | 0.197       | 0.525       |

To: Illinois Environmental Protection Agency

**Decatur Sanitary District** 

From: ADM Decatur WWTP

CC: ADM Corn Processing, ADM Oilseeds Processing, ADM JRRRC

Date: November 30, 2012

Re: Status Report Compliance Strategy for 2012 for Decatur Sanitary District and ADM

Decatur WWTP for waste treatment. (Covers updates post June 2012- date)



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ADM Research and Decatur complex have been actively pursuing technologies to remove Nickel (Ni) from its effluent stream released to the SDD treatment plant. As part of our Industrial Users permit we are enclosing our updated technical report on our efforts to mitigate nickel in the Decatur complex effluent.

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

#### - ADM SDD Industrial Users Permit 200.

ADM met with the SDD and IEPA in June 2012 and provided them with an overview detailing the progress and ADM's compliance efforts. In addition ADM has provided the district and its attorneys with input in finalizing a draft petition for site specific rulemaking being discussed with IEPA. Enclosed is a report on the progress ADM has made since the last update issued on June 2012.

### 1 Background and Update (post June 2012)

Nickel and Zinc are present in effluent leaving the ADM Decatur Complex Waste Water plant. Of the two metals, nickel is more difficult to remove from the effluent. ADM has conducted 5 plant material balances to understand the sources of Nickel in its internal streams. ADM's Decatur Complex consists of multiple, separate processing plants, which send wastewater to the on-site wastewater treatment plant ("WWTP") operated by Corn Plant personnel. These processing plants consist of the Corn Wet Mill, BioProducts Plant, Cogeneration Plant, East Soybean Processing Plant, West Soybean Processing Plant, Vitamin E Plant, Corn Germ Processing Plant, Glycols Plant and the Polyols Plant. Each of these unique plants produces multiple products, using both batch and continuous processes, and creates water streams which generally are reused multiple times prior to being discharged to the WWTP. The WWTP treats approximately 11 MGD through a newer anaerobic treatment system followed by aerobic treatment prior to discharge to the District.

The incoming soybeans contain approximately 4.1 parts per million ("ppm") nickels, while incoming corn contains approximately 0.53 ppm nickel. Given that ADM's Decatur Complex processes approximately 600,000 bushels of corn and 200,000 bushels of soybeans per day, our incoming Nickel load is about 49.2 lbs from the soybeans and 19.1 lbs from the corn. A small portion of the incoming nickel is discharged in the effluent.

In the ADM Decatur Facility, effluent water originates in the corn and soybeans being processed at the facility. During the processing, the metals are released and enter the processing water, some of which eventually ends up at the wastewater treatment plant.

ADM has monitored soluble Nickel at the Damon and Front stations continuously (see <u>Figures 1-3</u>) and made a number of modifications in its operations:

- 1) Our nickel in effluent discharge has been fairly stable and trending slightly down.
- Spent catalyst from the West Soybean Processing Plant is collected and sent to a landfill.
   Spilled catalyst is collected and disposed of as solid rather than washed into a sump.
- 3) Particulate catalyst from the Corn Plant Sorbitol production is captured by filters and physically recovered for recycling or disposal. ADM has installed an ion exchange resin system at the Sorbitol Plant to capture soluble nickel from wastewater. The system is undergoing startup and troubleshooting and results from testing are reported later in the current report.
- 4) The East Soybean Processing Plant is finalizing its design of a system that will remove the soy molasses stream (containing approximately 2.4 lb/day, approximately 35% of the soluble nickel from the Decatur Complex) from going to the WWTP. This stream is high in digestible, fermentable sugars but will need to be concentrated for stability. The East Soybean Processing Plant has prepared a cost estimate for this process change. Once the system design is complete and the cost estimate approved, ADM anticipates spending several million dollars to install it. Our anticipated start date for this project is late 2013 pending environmental and regulatory approvals.
- 5) The Polyols Plant accounts for approximately 11% of the soluble nickel from the Decatur Complex. The Polyols Plant has determined this nickel can be precipitated by pH adjustment. ADM is now determining how to implement this change on its process stream. As described later in this report, an internal request for funding ("AFE") has been submitted and we expect startup in later 2013 pending environmental and regulatory approvals.
- 6) We have also collected soluble nickel data for the past 8+ yrs. and it shows that our soluble nickel number remains largely unchanged with the only exception being the

change in total nickel due to startup of the anaerobic digesters post August 2008 (this data was shared in June 2012 update).

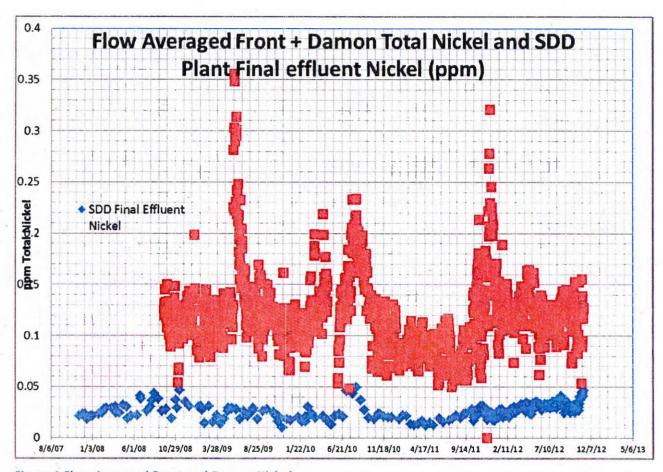


Figure 1 Flow Averaged Front and Damon Nickel

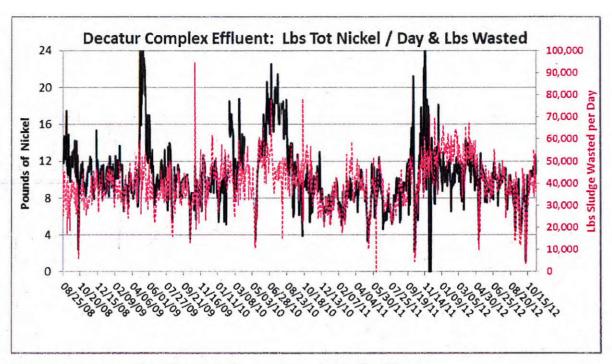


Figure 2 Decatur complex effluent-sludge wasting and nickel in effluent

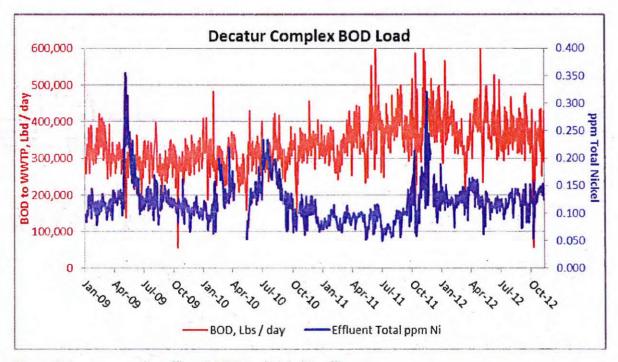


Figure 3 Decatur complex effluent- BOD and nickel in effluent

As reported in the 2010 (summer) - 2012 (summer) updates, ADM has, thus far, investigated 46 technologies that had the potential to control nickel at the Decatur Complex WWTP. (This was in addition to the work ADM has undertaken to reduce nickel within the individual wastewater streams.) As indicated in Table 1, these technologies can be segregated into six broad categories:

- Nickel Proprietary Precipitation Process;
- 2. Nickel Chemical Precipitation;
- 3. Ion Exchange Resin;
- 4. Filtration;
- pH Modification
- 6. Noncommercial, Experimental Technologies.

Additional details about some of the technologies identified in <u>Table 1</u> are presented in <u>Table 2</u>, including a general list of reasons why certain of those technologies are not technically feasible and are not currently being pursued.

ADM has finished piloting the various technologies listed in our summer 2012 report. <u>Table 3</u>, summarizes the capital, operating and chemical costs for the approaches it is scaling and either installing or continuing to trial and a summary of their best results.

Thus, of all of the technologies investigated by ADM to date, the only viable option that has not already been fully planned, installed or employed by ADM is the nickel capture process based upon high pH precipitation at the Polyols Plant. Because such technology has been determined to be both technically feasible and economically reasonable for the specific application, ADM will install that system at the Polyols Plant after necessary pilot testing is complete. However, that reduction, even when combined with the other reductions achieved by ADM, will still not reduce nickel to the levels sought by the District under its current permit. Even if ADM could overcome the technical obstacles it faces regarding the use of polymeric dimethyl dithiocarbamate to reduce nickel from the final wastewater effluent, testing indicates that residual soluble nickel concentrations close to 0.050 mg/L will remain irrespective of contact time and incoming nickel levels. ADM's investment from 2009 to December 2011 to identify and implement viable solutions to meet the nickel standard has been approximately \$1.02 million in employee costs and \$0.45 million in equipment rental and pilot trial costs. In addition, ADM has spent \$0.45 million to install a resin capture system at the Decatur Sorbitol plant. It is also preparing to spend an additional \$2.5 million to install a system to allow removal of the soy molasses stream and roughly \$0.75 million to install a high pH precipitation and filtration process at

the Polyols Plant. ADM has also significantly improved housekeeping in the West Plant to minimize nickel catalyst from entering the wastewater system. Finally, ADM has finished pilot trials on its ability to scale up a potentially viable chemical technology for installation at the Decatur Complex WWTP based on polymeric dimethyl dithiocarbamate to reduce nickel from its effluent. At this point, all reasonably identifiable options have been explored and all technically feasible and economically reasonable solutions are being pursued.

|          |                  | Table 1 S  | ummary of Technolog | ies Reviewed by ADM      |        |     |      |
|----------|------------------|------------|---------------------|--------------------------|--------|-----|------|
|          |                  |            |                     |                          |        | Те  | Eco  |
|          |                  |            |                     |                          |        | ch  | no   |
|          |                  |            |                     |                          |        | nic | mic  |
|          |                  |            |                     |                          |        | all | ally |
|          |                  |            |                     |                          | Nitrat | У   | Rea  |
|          |                  |            |                     |                          | ox/Res | Fe  | son  |
|          |                  |            |                     |                          | piram  | asi | abl  |
|          |                  |            |                     |                          | eter   | ble | e    |
|          |                  |            | Nickel Reduction    |                          | Testin | (y/ | (y/  |
|          | Chemistry        | Dosage     | (%)                 | Current Status           | g*     | n)  | n)   |
| Category | 1 - Nickel Propr | ietary Pre | cipitation Process  |                          |        |     |      |
|          |                  |            |                     |                          |        |     |      |
|          |                  | 1%-3%      |                     |                          |        |     |      |
|          |                  | by         |                     |                          | Not    |     |      |
|          | Activated        | weight     | 40%-60% (from       | Not Active, High dosages | tested |     |      |
|          | Clay             | of clay    | 200ppb influent)    | unscalable.              | ,      | No  | No   |
|          |                  |            |                     |                          | Not    |     |      |
|          |                  | 4%-8%      | 40% (from 90ppb     |                          | tested |     |      |
|          | Acidic Clay      | w/w        | influent)           | Stopped. High dosage.    |        | No  | No   |
|          |                  |            |                     |                          |        |     |      |
|          |                  |            |                     | Abandoned. High          | Not    |     |      |
|          | Chitosan         | 5%         | 90% from 200 ppb    | dosage, Concerns with    | tested |     |      |
|          | Based            | w/w        | influent            | Chitosan Availability    |        | No  | No   |

|          |                      | }           | 1               | 1                          | 1               |    | 1 1 |
|----------|----------------------|-------------|-----------------|----------------------------|-----------------|----|-----|
|          |                      |             |                 |                            |                 |    |     |
|          |                      |             |                 |                            | Not             |    |     |
|          |                      | 2%          | 82% (from       | Abandoned. Company         | tested          |    |     |
|          | Proprietary          | w/w         | 100ppb)         | went out of business       |                 | No | No  |
|          |                      |             |                 | Shelved. Strong pH         |                 |    |     |
|          |                      |             | }               | swing (acidification to    | Not             |    |     |
|          |                      | 200         | 64% (from       |                            | tested          |    |     |
|          | Proprietary          | ppm         | 120ppb)         | and neutralization)        |                 | No | No  |
|          |                      | Not         | ,               |                            | Not             |    |     |
|          | Not                  | disclos     | 40-60% (from    | Shelved. Company not       | tested          |    |     |
|          | disclosed            | ed          | 200ppb)         | sharing samples.           |                 | No | No  |
| Category | 2 - Nickel Chem      | ical Precip | <u> </u>        | g Carbamates or Organic Su | lfid <b>e</b> s |    |     |
|          |                      | 100рр       |                 |                            |                 |    |     |
|          | Polymeric            | m with      |                 |                            |                 |    |     |
|          | Dimethyl             | 50ppm       |                 |                            | Ę               |    |     |
|          | Dithiocarba          | of          |                 | Piloted. Total Nickel      | Passe           |    |     |
|          | mate                 | CaCl2       | 30% from 150ppb | reduction to 60ppb.        | d               | No | No  |
|          | Polymeric            |             |                 |                            |                 |    |     |
|          | Dimethyl             |             |                 |                            |                 |    |     |
|          | Dithiocarba          | 20-         |                 | Piloted. Total Nickel      | Passe           | Ye |     |
|          | mate                 | 50ppm       | 60% from 150ppb | reduction to 54 ppb.       | d               | s  | No  |
|          | Polymeric            |             |                 |                            |                 |    |     |
|          | Dimethyl             |             |                 |                            |                 |    |     |
|          | Dithiocar <b>b</b> a | 100рр       |                 | Piloted. Total Nickel      | Passe           | Уе |     |
|          | mate                 | m           | 41% from 150ppb | reduction to 32ppb         | d               | S  | No  |
| **       | Dimethyl             | 50ppm       |                 |                            |                 |    |     |
|          | Dithiocarba          | + pH        |                 | Piloted. Nickel reduction  | Passe           | Ye |     |
|          | mate                 | 6.0         | 76% from 150ppb | seen to 40ppb              | d               | s  | No  |
|          | Polymeri <b>c</b>    | 300pp       |                 | Not active. Modified       | Not             |    |     |
|          | Dimethyl             | m + pH      | 30%             | chemistry from Nalco       | tested          | No | No  |

|          | Dithiocarba      | swing      |                  | being tested              |        |    |     |
|----------|------------------|------------|------------------|---------------------------|--------|----|-----|
| 1        | mate             |            |                  |                           |        |    | İ   |
|          | Polymeric        | <u></u>    |                  |                           |        |    |     |
|          | Dimethyl         |            |                  |                           |        |    |     |
|          | Dithiocarba      |            |                  | Piloted. Nickel reduction | Passe  | Ye |     |
|          | mate             | 50ppm      | 48% from 100ppb  | seen to 20ppb             | d      | s  | No  |
|          | Polymeric        |            |                  |                           |        |    |     |
|          | Dimethyl         |            | ;                |                           |        |    |     |
|          | Dithiocarba      | 200рр      |                  | Piloted. Nickel reduction | Passe  |    |     |
|          | mate             | m          | 52% from 150ppb  | seen to 39 ppb            | d      | No | No  |
|          | Polymeric        |            |                  | - 1.00                    |        |    |     |
| ı        | Dimethyl         |            |                  | Not piloted. GE has not   | Not    |    |     |
|          | Dithiocarba      | 100рр      |                  | scaled up commercial      | tested |    |     |
|          | mate             | m          | 40% from 150ppb  | manufacturing.            |        | No | No  |
|          | Dimethyl         |            |                  |                           |        |    |     |
|          | Dithiocarba      | 100pp      |                  | Piloted. Nickel reduction | Passe  |    |     |
|          | mate             | m          | 60% from 150ppb  | seen to 24 ppb            | d      | No | No  |
| Category | 3 - Non Functio  | nal Resins |                  |                           |        |    |     |
|          | Styrene          |            |                  |                           |        |    |     |
|          | Divinyl          | 2-5%       |                  | Not scaled. High          | Not    |    |     |
|          | Benzene          | w/w        | 20%              | regeneration costs        | tested | No | No  |
|          |                  |            |                  | Not scaled. Very high     |        |    |     |
|          | Styrene          |            |                  | resin use. Caustic        |        |    |     |
|          | Divinyl          | 4%         |                  | /ethanol based            | Not    |    |     |
|          | Benzene          | w/w        | 60%              | regeneration              | tested | No | No  |
|          | Immobilized      |            |                  |                           |        |    |     |
|          | Ion              |            |                  |                           | l<br>  |    |     |
|          | Exchange         |            |                  |                           | Not    |    |     |
|          | Beads            | 5%         | Not significant  | Shelved                   | tested | No | No  |
| Category | 4 - Reuse of lor | Exchange   | Resin            |                           |        |    | -   |
|          | Sulfonic         | 0.1-       | Complete removal | Installed at 5orbitol     | Not    | Ye | Yes |

|            |                 | 0.5%    | of Ionic Nickel   | plant                   | requir | 5  | ]  |
|------------|-----------------|---------|-------------------|-------------------------|--------|----|----|
|            |                 |         | from the Sorbitol |                         | ed     |    |    |
|            |                 |         | plant waste       |                         |        |    |    |
| l          |                 |         |                   |                         |        |    |    |
| Category ! | 5 - Filtration  |         |                   |                         |        |    |    |
|            | Phosphate       | 80%     |                   | _                       |        |    |    |
|            | precipitation   | recover |                   |                         | Not    |    |    |
|            | + Reverse       | y of    |                   | Shelved. Brine disposal | requir |    |    |
|            | Osmosis         | feed    | 95%+ reduction    | issues. High capex      | ed     | No | No |
|            | Low             | 30%     |                   |                         |        |    |    |
|            | pressure        | recover |                   |                         | Not    |    |    |
|            | Reverse         | y of    |                   | Shelved. Brine disposal | requir |    |    |
|            | Osmosis         | feed    | 80% + reduction   | issues. High capex      | ed     | No | No |
|            |                 |         |                   |                         |        |    |    |
|            |                 | Not     |                   |                         | Not    |    |    |
|            |                 | disclos |                   |                         | requir |    |    |
|            | Sand Filter     | ed      | 20% reduction     | Insufficient efficacy   | ed     | No | No |
| Category   | 6 - Other Appro | aches   |                   |                         |        | -  |    |
|            |                 |         |                   | Company went out of     |        |    |    |
|            | Carbon          | Not     |                   | business. CD also binds | Not    |    |    |
|            | Aerogels        | tested  | Not tested        | other ions              | tested | No | No |
|            |                 | Not     | Higher Nickel due |                         |        |    |    |
|            | Electrochem     | disclos | to leaching from  |                         | Not    |    |    |
|            | ical            | ed      | electrode plates  | Shelved after 4 trials. | tested | No | No |
|            |                 |         |                   | ,                       |        |    |    |
|            | Ferric          | 100pp   |                   | Unscalable due to       | Not    |    |    |
|            | Chloride        | m       | 40%               | chloride limits         | tested | No | No |
|            |                 |         |                   |                         |        |    |    |
|            |                 | not     |                   |                         | Not    |    |    |
|            | Protein         | tested  | Not tested        | Lab scale only          | tested | No | No |

|   |             | 5%      |                    |                          |        |    |     |
|---|-------------|---------|--------------------|--------------------------|--------|----|-----|
|   |             | w/w +   |                    |                          |        |    |     |
| 1 | Hydrogen    | pН      |                    |                          |        |    |     |
|   | Peroxide    | adjust  |                    | Significant chemical     | Not    |    |     |
|   | and Ozone   | ment    | 20% from 150ppb    | usage                    | tested | No | No  |
|   |             | Not     |                    |                          |        |    |     |
|   | Protein     | disclos |                    | Other ions compete with  | Not    |    |     |
|   | based       | ed      | Not tested         | nickel. Not scalable.    | tested | No | No  |
|   |             |         |                    |                          |        |    |     |
|   |             |         |                    |                          |        |    |     |
|   |             |         |                    |                          |        |    | ]   |
|   |             | 1-3%    |                    | Very high chemical       | Not    |    |     |
|   | pH Swing    | w/w     | 30% from 150ppb    | usage.                   | tested | No | No  |
|   |             |         | Complete for ionic |                          |        |    |     |
|   |             | 1-2%    | regeneration       | Being piloted at Polyols | Not    | Ye |     |
|   | pH >11.0    | w/w     | waste              | plant for waste stream   | tested | s  | Yes |
|   |             |         |                    | Bench scale trials       |        |    |     |
|   |             |         | Reduced soluble    | complete. Unable to      |        |    |     |
|   | Proprietary |         | nickel to below    | scale up due to startup  | Not    |    |     |
|   | Adsorbent   | 5%      | 0.037ppb           | nature of technology     | tested | No | No  |

| Technology / Provider | Vendor not cooperative with samples | Assessed and determined not effective | Not commercially available | High Dosages required | Results not scalable beyond bench scale | cow recoveries and<br>nine disposal<br>concerns | Technicolly Feasible (y/n) | Comments                         |
|-----------------------|-------------------------------------|---------------------------------------|----------------------------|-----------------------|---|---|----------------------------|----------------------------------|
|                       | Х                                   |                                       | Х                          |                       |   |   | No                         |                                  |
|                       |                                     | _                                     |                            |                       |   |   |                            | Would require 5 million pounds o |
|                       |                                     | x                                     |                            | х                     |   |   | No                         | additive per day                 |

|      | ŀ |   | 1 | , <del>-</del> | 1 | _   |  |
|------|---|---|---|----------------|---|-----|--|
|      |   | x | X |                |   | No  |  |
|      |   | _ |   |                |   |     |  |
| X    |   |   | X |                |   | No  |  |
| <br> |   |   | _ |                |   | 110 | Description and the state of th |
|      |   |   |   |                |   |     | Requires a pH to <2 then to pH 5.5 then  |
|      |   |   | X |                |   | No  | to pH 10   |
| Х    |   |   |   |                |   | No  |  |
|      |   |   |   |                |   |     | Plant pilot trial did not achieve required   |
|      |   |   |   | Х              |   | No  | Nickel reduction.  |
|      |   |   |   |                |   |     | Plant pilot trial did not achieve required   |
|      | x |   |   | x              |   | No  | Nickel reduction.  |
|      |   |   |   |                |   |     | Plant pilot trial did not achieve required   |
|      |   |   |   | x              |   | No  | Nickel reduction.  |
|      |   | Х |   |                |   | No  |  |
|      |   |   |   |                |   | No  |  |
|      |   |   | Х |                |   | No  |  |
|      |   |   |   |                |   | 1   | 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   |
|      |   |   | х |                |   | No  | Ethanol to regenerate resin.   |
|      | Х |   | х |                |   | No  |  |
|      |   |   |   | }              |   | Yes |  |
|      |   |   |   |                |   | *   | Installed at Sorbitol plant  |
|      |   |   |   |                | Х | No  |  |
|      |   |   |   |                |   |     |  |
|      |   |   |   |                | х | No  |  |
|      |   |   |   |                |   |     |  |
|      |   |   |   |                | Х | No  |  |

|     |   | İ | Х |   | No     |   |
|-----|---|---|---|---|--------|---|
|     |   | Х | Х |   | No     |   |
|     |   |   |   |   |        | Requires over 30,000 pounds of ferric                                 |
|     |   | х |   |   | No     | salts per day   |
|     |   |   | Х |   | <br>No |   |
|     |   |   |   |   |        | Raise the pH 10 and add ozone and hydrogen peroxide. Large amounts of |
|     |   | х |   |   | No     | chemicals required.   |
| NAC |   |   | Х |   | No     |   |
|     |   |   |   |   |        | Suitable for <~50,000 GPD, non-grain                                  |
|     |   |   |   |   |        | based wastewater with non-chelated,                                   |
| 100 |   |   |   |   |        | salt-form nickel such as Polyols Plant IX                             |
|     |   |   |   |   | Yes    | regen waste   |
|     |   |   |   |   |        | We are discussing a pilot trial with the                              |
|     |   |   |   |   |        | vendor but they don't have capabilities                               |
|     | х |   | x | x |        | to manufacture quantities required.                                   |

<sup>\*</sup> 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.

|          |                        |                                       |        | Initial | Capital         | Annual    | Operating                               | & | Status                             |
|----------|------------------------|---------------------------------------|--------|---------|-----------------|-----------|---|---|------------------------------------|
|          |                        |                                       |        | Cost    |                 | Chemical  | Costs                                   |   |                                    |
| Act      | tive Projects          |                                       |        |         |                 |           |   |   |                                    |
|          |                        |                                       |        |         | 4               |           | 100000000000000000000000000000000000000 |   | Manager and Company of the Company |
| 1)       | Soybean                | Process                               | Stream |         |                 |           |   |   | Planned                            |
| 1)       | Soybean<br>Alternative | Process                               | Stream | \$2.7 m | illi <b>o</b> n | \$400,000 |   |   | Planned                            |
| 1)<br>2) | ,                      | · · · · · · · · · · · · · · · · · · · |        | \$2.7 m | illi <b>o</b> n | \$400,000 |   |   | Planned<br>Installed               |

| 3) | High pH precipitation at Polyols<br>Plant | \$750,000      | \$600,000                     | Planned         |
|----|---|----------------|-------------------------------|-----------------|
|    | Further Technical Analysis/Cost           |                |                               |                 |
|    | Prohibitive                               | 1,511,421      |                               |                 |
| 1) | Polymeric DTC addition and nickel         |                |                               | Pilot trials    |
|    | removal using different unit              |                |                               | finished.       |
|    | operations                                | Not available  | Not available                 |                 |
|    |   |                |                               | Effluent        |
|    |   |                |                               | Irreversibly    |
|    |   |                |                               | fouled          |
|    |   |                |                               | Membrane.       |
|    |   |                |                               | Filtrate rate   |
|    | a) Single-Pass Membrane                   |                |                               | decreased       |
|    | Filter                                    | \$ 25million   | \$ 3 million                  | more than 90%   |
|    |   |                | \$ 2.3 million                | Average nickel  |
|    |   |                |                               | reduction       |
|    |   |                |                               | insufficient to |
|    | b) Dissalved Air                          |                |                               | meet proposed   |
|    | Floatation and Sand Filter                | \$ 15 million  |                               | limits          |
|    |   |                | \$ 2 million                  | Average nickel  |
|    |   |                |                               | reduction is    |
|    |   |                |                               | insufficient to |
|    | c) :                                      |                |                               | meet proposed   |
|    | Dissolved Air Floatation Filter           | \$ 25million   |                               | limits          |
|    |   |                | \$ 1.5 million + \$30 million | Average nickel  |
|    |   |                | in disposal costs + 7000      | reduction is    |
|    |   |                | tons per day of clay          | insufficient to |
|    | d) Rotory                                 |                | disposal.                     | meet proposed   |
|    | Vacuum Filter                             | \$ 1.8 million |                               | limits          |
|    | e) Enhanced                               |                | Not available                 | Average nickel  |
|    | Air Flootation Filter                     | Not available  |                               | reduction is    |

|                         |               |               | insufficient to meet proposed limits              |
|-------------------------|---------------|---------------|---|
|                         |               | \$2.1 million | Average Total                                     |
|                         |               |               | reduction to                                      |
| f) Lamella Gravity      |               |               | insufficient                                      |
| Settler and Sand Filter | \$6.2 million |               | proposed limits                                   |
|                         |               | Not available | Technology is not scalable.  Vendor has only made |
| g) Addition to          |               |               | multi gram  |
| Anaerobic Influent      | Not available |               | quantities.                                       |

### 2 ADM Pilot trials update

As noted above ADM has completed its pilot plant trials for chemical sequestration of nickel and key cost numbers which are summarized in <u>Table 3</u>. Detailed technical results of the pilot trials are summarized below.

# 2.1 Precipitation of Nickel

Waste Water entering the Decatur WWTP is separated into two treated, process streams: High Salt and Low Salt. Each stream is separately treated with Anaerobic and Aerobic systems. The Low Salt stream is recycled internally for water recovery and use. The High Salt stream is the primary source of nickel to SDD (Sanitary District of Decatur).

The High Salt stream must be treated to remove Nickel. The pending permit limit of 0.037 ppm is based on total Nickel. If all the precipitated nickel is removed; then, the remaining soluble nickel becomes the total nickel. To reduce and eliminate nickel from the WWTP (Waste Water Treatment

Plant) stream – soluble nickel must be converted to an insoluble state; then, the challenge becomes the separation of the small amount of precipitated nickel from the large volume liquid flow.

Precipitating nickel from the High Salt stream was achieved using Nalco TX. The Nalco TX chemical and High Salt stream were combined and the reaction was time dependent – as depicted in Figure 4, the nickel solubility decreases with time. From conditions reported in Figure 4, the Nalco TX reduces the soluble nickel below 0.037 ppm; however, 100% of the precipitated nickel must be removed to achieve the limit. Longer hold times may benefit overall nickel reduction since the soluble nickel level decreases and separation of precipitated nickel efficiency will be less than 100%. A 6 MGD process will require a 250,000 gallon tank to achieve a one hour hold time.

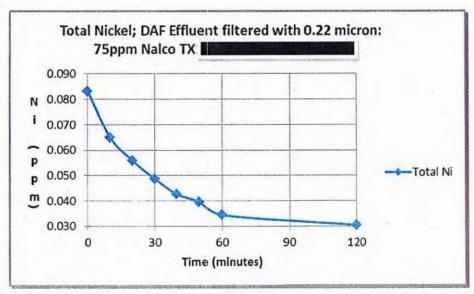


Figure 4 Maximum Nickel Reduction vs. time. After addition of metal precipitant. Starting nickel 0.083 ppm.

#### HIGH SALT INFLUENT PRECIPITATION ANAEROBIC DIGESTERS

Nickel precipitation was tested with the influent to the Anaerobic Digesters (Figure 5). Nalco TX was added to the stream, mixed in on the suction side of a centrifugal pump and sampled. Anaerobic digesters currently run a 7 day residence time. <u>Table 4</u> shows the results of the experiments which demonstrate decreased soluble nickel after addition of Nalco TX. Precipitated nickel would still require removal to achieve desired nickel concentration; but, all tests reduced soluble Nickel to below 0.037

ppm. This nickel is believed to become insoluble and precipitate from the solution and settle in digesters.

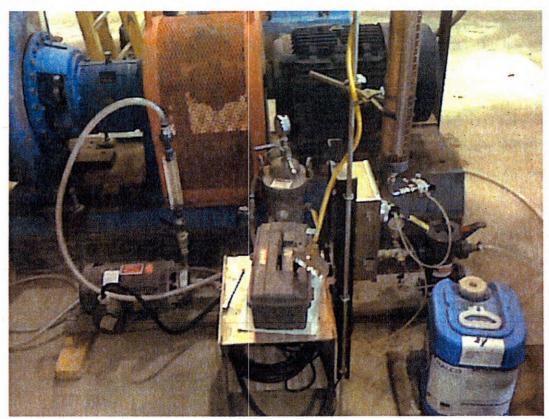


Figure 5 Nickel Precipitation skid (foreground). Nickel Precipitation of Anaerobic Digester Influent.

Table 4. NalcoTX addition at Anaerobic Digester. Reduction of Soluble Nickel

|        |          | Feed    | Product | Soluble   |
|--------|----------|---------|---------|-----------|
|        | Nalco    | Soluble | Soluble | Ni        |
| Sample | Addition | Ni      | Ni s    | Reduction |
| ID     |          | (mg/kg) | (mg/kg) | (%)       |
| 1      | 81       | 0.152   | 0.028   | 92        |
| 2      | 121      | 0.152   | 0.030   | 80        |
| 3      | 138      | 0.186   | 0.011   | 94        |
| 4      | 202      | 0.280   | 0.010   | 96        |

| 5 | 274 | 0.152 | 0.026 | 83 |
|---|-----|-------|-------|----|
|   |     |       |       |    |

HIGH SALT EFFLUENT STREAM; AFTER AEROBIC TREATMENT; AFTER DAF (DISSOLVED AIR FLOATATION).

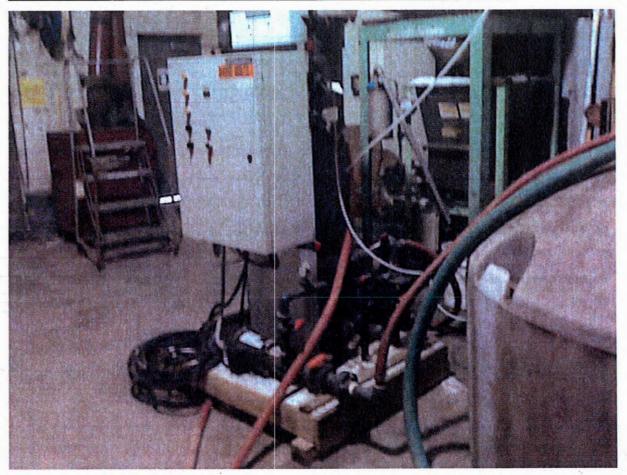


Figure 6 Kroff Engineering/Shelton Associates - Nickel removal with Single-Pass Microfiltration

Our tests at Kroff's, Pittsburgh, PA lab included trials of their metal precipitant process, which consists of filtration through progressively decreasing pore sized membranes, to measure the range of particle sizes that would need to be filtered. Kroff chemistry reduces soluble Nickel; but a problem remains in the separation method that removes the precipitated nickel from water- using a single pass membrane has not worked in trials.. There are some differences between the analytical results of Kroff and ADM – however both followed a similar trend of results. Kroff was only able to reduce nickel in a controlled laboratory environment.

Table 5. Comparison of Kroff & ADM Analytic results for identical samples

|   | ADM    | Kroff  |              | Added     |          | Filter   |
|---|--------|--------|--------------|-----------|----------|----------|
|   | Tot Ni | Tot Ni |              | Kroff DTC |          | Size     |
|   | (ppm)  | (ppm)  | Contents     | ppm       | Filtered | (micron) |
| 1 | 0.024  | 0.003  | DAF Effluent | 20        | Yes      | 0.2      |
| 2 | 0.043  | 0.025  | DAF Effluent | 0         | Yes      | 0.2      |
| 3 | 0.066  | 0.038  | DAF Effluent | 0         | No       |          |
| 4 | 0.018  | N.D.   | DAF Influent | 20        | Yes      | 0.2      |

A Kroff, single-pass microfiltration skid was tested using effluent from the dissolved air flotation process (DAF) in the Decatur WWTP, DAF Effluent. A high concentration of phosphorous in the DAF effluent fouls the filter and the filtration rate rapidly decreases from twenty to less than two GPM very rapidly. Back-pulses (which are designed filter cleaning cycles) have little or no effect on this unknown substance. Kroff Engineering had tried for months to identify this contaminant without success. To provide space for screening alternate nickel filtration equipment, testing Kroff filter was terminated and equipment returned.

GE Power & Water - Nickel Removal with EAF (Enhanced Air Floatation)

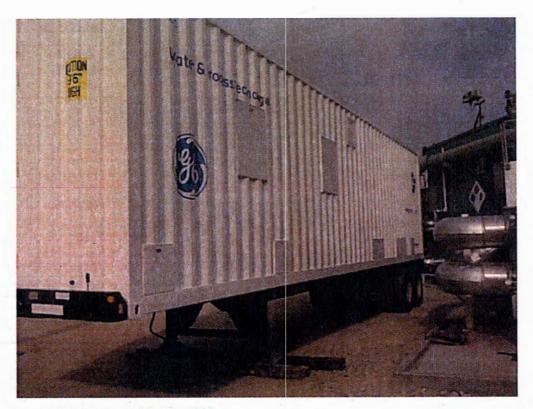


Figure 7 GE EAF Portable Test Skid

We rented and tested the GE EAF unit. It was self-contained unit, built inside a shipping container and loaded onto a flatbed trailer. ADM DAF Effluent was pumped continuously, at 25 GPM, treated with Nalco TX and pumped to a holding tank for a thirty minute residence time. This treated stream was pumped to the GE EAF equipment.

Variables tested with the GE EAF unit:

- 1. Circulation flow rate
- 2. Air flow rate as well as air pressure
- 3. Metal precipitant dosage as well as metal precipitant brand.
- 4. Coagulant and Flocculent dosages

The GE EAF system was ineffective for separating the precipitated nickel. In some tests, the soluble Ni decreased below the 0.037 ppm threshold; but, the precipitated nickel would not separate using EAF.

### Table 6. EAF treated DAF Effluent.

| Feed  | EAF F | Product |       | Coagulant | Flocculent |          |
|-------|-------|---------|-------|-----------|------------|----------|
| Total | Total | Soluble | Metal |           |            | Total Ni |

|       | Ni    | Ni    | Ni    | Metal  | Precip |      |       |      |       | Reduced |
|-------|-------|-------|-------|--------|--------|------|-------|------|-------|---------|
| ID    | (ppm) | (ppm) | (ppm) | Precip | (ppm)  | Туре | (ppm) | Туре | (ppm) | (%)     |
| 16Apr | 0.090 | 0.095 | 0.039 | TX     | 500    |      |       |      | 5     | -6      |
| 17Apr | 0.104 | 0.100 | 0.056 | TX     | 75     |      |       |      | 5     | 4       |
| 18Apr | 0.101 | 0.094 | 0.044 | TX     | 75     |      |       |      | 5     | 7       |
| 19Apr | 0.094 | 0.089 | 0.033 | TX     | 500    |      |       |      | 5     | 5       |
| 24Apr | 0.099 | 0.098 | 0.032 | TX     | 75     |      |       |      | 5     | 1       |
| 27Apr | 0.076 | 0.077 | 0.034 | TX     | 75     |      |       |      | 5     | -1      |
| 4May  | 0.077 | 0.065 | 0.027 | Namet  | 75     |      |       |      | 10    | 16      |

Alum: Aluminum Sulfate

Parkson (Tested in Pompano Beach, FL) – Nickel removal with LGS (Lamella Gravity Settler) and Dynasand (counter-current) sand filters.

Initial testing from Parkson Water Research Facility barely achieved 15% Ni removal. Parkson did not screen material for almost one month; so, a second sample was shipped for testing. This was tested within two days. Numerous tests were completed; of those, six tests had total nickel that was reduced below the limit. During the second session, we had six tests with product (total Ni) at, or below 0.037 ppm. After successful results, pilot equipment was sent to Decatur for further testing.

Table 7. Total Nickel and test conditions reducing Nickel concentrations below 0.038 mg/kg. Samples from 2<sup>nd</sup> Parkson Water Research Facility Test. January 2012.

|        |          | Prod  |       |   |   |     |        | Total     |
|--------|----------|-------|-------|---|---|-----|--------|-----------|
|        | Feed     | Total | Nalco |   |   |     | Sand   | Ni        |
| Sample | Total Ni | Ni    | ppm   |   |   |     | Filter | Reduction |
| ID     | ppm      | ppm   |       |   |   |     | Used   | (%)       |
| 3      | 0.155    | 0.034 | 50    | 2 | 2 | 0   | N      | 78        |
| 4      | 0.155    | 0.037 | 50    | 0 | 0 | 100 | N      | 76        |
| 5      | 0.155    | 0.037 | 50    | 0 | 2 | 100 | N      | 76        |

| 6 | 0.155 | 0.036 | 50 | 2 | 0 | 100 | N | 77 |
|---|-------|-------|----|---|---|-----|---|----|
| 8 | 0.155 | 0.035 | 50 | 0 | 2 | 100 | N | 77 |
| 9 | 0.155 | 0.037 | 50 | 2 | 0 | 100 | N | 76 |

Equipment was sent to Decatur on two trucks – due to variety of reasons, the equipment was not ready to test for almost three months. Set-up required extensive use of contractors, electricians, and numerous replaced and/or new parts. The trials were finally completed towards the end of September 2012.



Figure 8 Parkson D2 Sand Filters (Foreground on trailer)

The flow treatment scheme required a 50 GPM flow rate; addition of Nalco TX; 30 minute hold time; Lamella; Primary Sand Filter (1.35-1.45mm) and then Secondary Sand Filter (0.85-0.95mm).

The Lamella system was ineffective at separating nickel- which was a small percentage of solids. About 40-100 ppb nickel was present in about 300 ppm of biological solids. Looking at <u>Table 8</u> below,

reducing air flow rate through sand filters increased nickel removal. As air flow is reduced, it is postulated that precipitated nickel accumulated and formed a self-filtering media. Only one test resulted in a total nickel reduction to below 0.037ppm.

Table 8. Parkson Results from Decatur testing of Lamella Gravity Settler and D2 Sand Filters.

|                 |          | Lamella | Sand Filter | •     |   |     |     | Air to  | Total      |
|-----------------|----------|---------|-------------|-------|---|-----|-----|---------|------------|
|                 | Feed     | Total   | Total       | Nalco |   | _   |     | Sand    | Ni         |
| Sample          | Total Ni | Ni      | Ni          | ppm   |   |     |     | Filters | Reduction  |
| ID.             | ppm      | ppm     | ppm         |       |   |     |     | SCFH    | (%)        |
| Parkson3July-1  | 0.090    | 0.084   | 0.062       | 100   | 0 | 0   | 100 | 60      | 31         |
| Parkson10July-1 | 0.080    | 0.071   | 0.057       | 50    | 0 | 1   | 100 | 75      | 29         |
| Parkson11July-1 | 0.101    | 0.138   | 0.060       | 100   | 1 | 1   | 100 | 75      | 41         |
| Parkson12July-1 | 0.087    | 0.065   | 0.050       | 100   | 2 | 2   | 100 | 70      | 43         |
| Parkson13July-1 | 0.083    | 0.069   | 0.050       | 100   | 1 | 0   | 100 | 70      | 40         |
| Prkson17July-1  | 0.086    | 0.056   | 0.039       | 200   | 0 | 1   | 200 | 60      | 55         |
| Parkson18July-1 | 0.081    | 0.056   | 0.040       | 200   | 0 | 1   | 200 | 35      | 51         |
| Parkson19July-1 | 0.089    | 0.071   | 0.037       | 200   | 0 | 1   | 200 | 30      | 58         |
| Parkson24July-1 | 0.095    | 0.074   | 0.044       | 100   | 0 | 1   | 100 | 45      | 54         |
| Parkson24July-2 | 0.095    | 0.074   | 0.043       | 100   | 0 | 1.5 | 100 | 40      | <b>5</b> 5 |
| Parkson25July-1 | 0.081    | 0.068   | 0.041       | 100   | 0 | 1   | 100 | 30      | 49         |
| Parkson26July-1 | 0.094    | 0.072   | 0.042       | 100   | 0 | 1.5 | 100 | 45      | <b>5</b> 5 |
| Parkson26July-2 | 0.094    | 0.072   | 0.046       | 100   | 0 | 1.5 | 100 | 40      | 54         |

Lamella gravity settlers use gravity for the settling of the solids. However, significant problems with the Lamella were found. For example, floating solids accumulated on top of lamella during testing at Decatur WWTP. Parkson representatives traveled to Decatur and tested DAF Influent. Conclusions from testing were that Parkson does not recommend Lamella for the current ADM process. This is because the influent to current ADM DAF system is generating tiny gas bubbles that cause solids to float and the Lamella system was unsuccessful in precipitating or removing the floated solids.

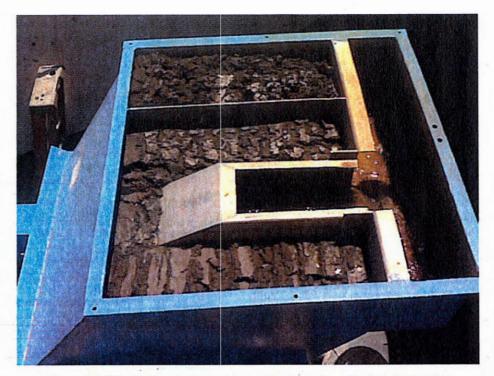


Figure 9 Solids Accumulation on top of Parkson Lamella Gravity Settler

Ecolab/Krofta Chemical Company, Inc. – Nickel removal using Dissolved Air Floatation (DAF) and Sandfloat Pilot.

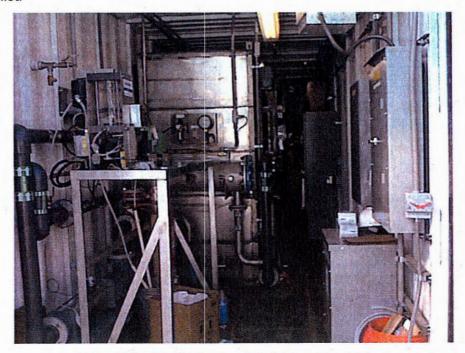


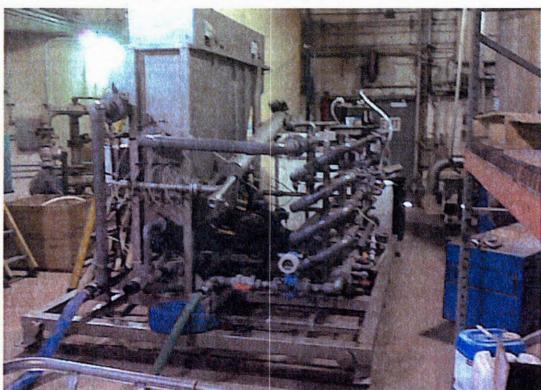
Figure 10 Ecolab/Krofta Test Skid DAF and Sand Filters

This unit contained DAF (Dissolved Air Floatation) and a sand filter. DAF removed majority of precipitated nickel. Sand Filter accounted for less than 5% of total Nickel removed. In <u>Table 9</u>, no operating conditions equaled or reduced nickel below 0.037 ppm.

Table 9. Ecolab/Krofta Nickel reduction with DAF and Sand Filter

|           | Naico    | Feed  | DAF   | _       | Coag  | Floc  | Total     |
|-----------|----------|-------|-------|---------|-------|-------|-----------|
|           | Addition | Total | Total | Sludge  | Used  | Used  | Ni        |
| Sample    | ppm      | Ni    | Ni    | Recycle | /     | /     | Reduction |
| ID        |          | (ppm) | (ppm) | used    | (ppm) | (ppm) | (%)       |
| Ecolab-1  | 28       | 0.089 | 0.080 | No      |       |       | 10        |
| Ecolab-2  | 28       | 0.067 | 0.068 | No      |       |       | (-)2      |
| Ecolab-3  | 28       | 0.077 | 0.079 | No      |       |       | (-)3      |
| Ecolab-4  | 28       | 0.077 | 0.052 | No      |       |       | 33        |
| Ecolab-5  | 55       | 0.078 | 0.055 | No      |       | . –   | 30        |
| Ecolab-6  | 29       | 0.076 | 0.050 | No      |       |       | 34        |
| Ecolab-7  | 61       | 0.075 | 0.052 | No      |       |       | 31        |
| Ecolab-10 | 48       | 0.070 | 0.051 | No      |       |       | 27        |
| Ecolab-13 | 48       | 0.071 | 0.050 | Yes     | ,     |       | 30        |
| Ecolab-15 | 48       | 0.073 | 0.055 | No      |       |       | 25        |
| Ecolab-18 | 50       | 0.077 | 0.045 | No      |       |       | 42        |
| Ecolab-19 | 50       | 0.081 | 0.066 | No      |       |       | 19        |
| Ecolab-20 | 50       | 0.081 | 0.060 | No      |       |       | 26        |
| Ecolab-21 | 50       | 0.081 | 0.079 | No      |       |       | 3         |
| Ecolab-22 | 50       | 0.081 | 0.065 | No      |       |       | 20        |





FRC Systems International, LLC - Nickel removal using Dissolved Air Floatation

Figure 11 FRC Systems International DAF Skid

This pilot was a DAF (Dissolved Air Floatation) system only. No test, using this equipment, equaled or reduced total nickel in the discharge to below 0.037 ppm. A series of experiments were performed, with FRC equipment utilizing recycled WAS (Waste Activated Sludge) achieved lower Ni levels, than similar test conditions without WAS. It was hypothesized that the WAS, may provide additional surface area, that enhances separation of precipitated Nickel. Looking at <u>Table 10</u>, Comparing FRC-12 & 13, with FRC-14 & 15; samples using less coagulant (FRC-14 & 15) had greater total Nickel reduction – the only difference being use of recycled sludge. Comparing FRC-1 & 2; with FRC-3, 4 & 5; samples FRC-3, 4 & 5 removed more Nickel using less coagulant.

Table 10. FRC DAF, Feed Rate ~25 GPM

| Nalco    | Feed  | Product |      |         | Coag | Floc | Total     |
|----------|-------|---------|------|---------|------|------|-----------|
| Addition | Total | Total   | Hold | Sludge  | Used | Used | Ni        |
| ppm      | Ni    | Ni      | Time | Recycle | 1    | 1    | Reduction |

|        |    | (ppm) | (ppm) | (min) | Used | (ppm) | (ppm) | (%) |
|--------|----|-------|-------|-------|------|-------|-------|-----|
| FRC-1  | 20 | 0.079 | 0.075 | 30    | No   |       |       | 5   |
| FRC-2  | 59 | 0.078 | 0.073 | 30    | No   |       |       | 6   |
| FRC-3  | 50 | 0.072 | 0.051 | 30    | Yes  |       |       | 29  |
| FRC-4  | 50 | 0.072 | 0.053 | 30    | Yes  |       |       | 26  |
| FRC-5  | 50 | 0.072 | 0.057 | 30    | Yes  |       |       | 21  |
| FRC-12 | 50 | 0.074 | 0.059 | 0     | No   |       |       | 20  |
| FRC-13 | 50 | 0.074 | 0.065 | 0     | No   |       |       | 12  |
| FRC-14 | 50 | 0.074 | 0.048 | 0     | Yes  |       |       | 35  |
| FRC-15 | 50 | 0.074 | 0.042 | 0     | Yes  |       |       | 43  |

Alar Corp. – Nickel removal using diatomaceous earth and RVF (Rotary Vacuum Filter)

This series of screening experiments were at the Alar Corp Facility. A bench-scale filter and Diatomaceous Earth (DE), equivalent to FW-20, was used for simulating RVF technology. As can be seen in <u>Table 11</u>, whatever soluble Ni was precipitated – it was removed. This was the only equipment that has achieved filtrate Nickel below 0.037 ppm.

Table 11. Alar Corp Rotary Vacuum Filtration. Various Nalco chemistries with 30 minute residence time and Medium grade DE.

|      | Feed  | Product | Product | Nalco    | Coag  | Floc  |            | Total     |
|------|-------|---------|---------|----------|-------|-------|------------|-----------|
| Test | Total | Total   | Soluble | TX       | Used  | Used  | Filtration | Ni        |
|      | Ni    | Ni      | Ni      | Addition | /     | /     | Rate       | Reduction |
|      | (ppm) | (ppm)   | (ppm)   | ppm      | (ppm) | (ppm) | Gal/ft²/Hr | (%)       |
| 1    | 0.070 | 0.026   | 0.026   | 50       |       |       | 22         | 63        |
| 2    | 0.070 | 0.027   | 0.026   | 50       |       |       | 27         | 61        |
| 3    | 0.070 | 0.028   | 0.027   | 50       |       |       | 28         | 60        |
| 4    | 0.070 | 0.028   | 0.027   | 50       |       |       | 23         | 60        |
| 5    | 0.070 | 0.028   | 0.028   | 25       |       |       | 26         | 60        |

| 6  | 0.070 | 0.028 | 0.027 | 25 | <b>EXKONY</b>  | MARIE | 24 | 60 |
|----|-------|-------|-------|----|----------------|-------|----|----|
| 7  | 0.070 | 0.028 | 0.027 | 25 | REMAIN         |       | 27 | 60 |
| 8  | 0.070 | 0.029 | 0.027 | 25 | O. T. A. A. I. |       | 42 | 59 |
| 9  | 0.070 | 0.031 | 0.028 | 75 | MANAGE         |       | 39 | 56 |
| 10 | 0.070 | 0.035 | 0.034 | 75 | Markey         |       | 26 | 50 |

Previous testing at Decatur WWTP incorrectly assumed the thirty minute residence time, after metal precipitant addition, was unnecessary. The majority of tests (see <u>Table 12</u>, next page) did not remove precipitated nickel. The primary reason for omitting the thirty minute hold is that the bottom four rows of <u>Table 12</u> incorporate the 30 minute residence time. Soluble and Total Nickel numbers are similar, indicating that almost all the precipitated nickel has been filtered. Medium grade DE (FW-20) also appears to be necessary for removal of precipitated nickel. Coarse grade DE had higher filtration rates, but Nickel separation was lower.



Figure 12 Alar Corp Rotary Vacuum Pilot Skid

Table 12. ALAR RVF. Screening: metal precipitant, Coagulant, with and without 30 minute hold after adding Metal precipitant. Tests with the 30 minute residence time, after Nalco TX addition, are in bold font.

|        | Feed  | Produc   | Produc  | Me     | tal   | Coagulant    |    |     |     |           | Total    |
|--------|-------|----------|---------|--------|-------|--------------|----|-----|-----|-----------|----------|
| Sample | Total | t        | t       | Precip | itant |              |    |     |     |           | Ni       |
| ID     | Ni    | Total Ni | Soluble |        |       |              |    |     | D   | Filtrate  | Reductio |
|        | (ppm  | (ppm)    | Ni      | Bran   | рр    | Bra <b>n</b> | рр | d   | E   | Rate      | n        |
|        | )     |          | (ppm)   | d      | m     | d            | m  |     |     | Gal/Hr/Ft | (%)      |
|        |       |          | :       |        |       | }            | :  |     |     | 2         |          |
| 05Mar  | 0.081 | 0.068    | 0.066   |        |       |              |    | Eff | С   | 39        | 16       |
| -1     |       |          |         |        |       |              |    |     |     |           |          |
| 05Mar  | 0.083 | 0.044    | 0.033   |        |       |              |    | Eff | С   | 31        | 47       |
| -2     |       |          |         |        |       |              |    |     |     |           |          |
| 06Mar  | 0.085 | 0.046    | 0.044   |        |       |              |    | Eff | С   | 37        | 46       |
| -1     | ;     |          |         |        |       |              |    |     |     |           |          |
| 07Mar  | 0.079 | 0.051    | 0.050   |        |       |              |    | Eff | С   | 40        | 35       |
| -1     |       |          |         |        |       |              |    |     | · ' |           |          |
| 08Mar  | 0.079 | 0.050    | 0.050   |        |       |              |    | Eff | С   | 37        | 37       |
| -1     | !     |          | :       |        |       |              |    |     |     |           |          |
| 09Mar  | 0.079 | 0.050    | 0.028   |        |       |              |    | Eff | С   | 32        | 37       |
| -1     |       |          |         |        |       |              |    |     |     |           | ]        |
| 12Mar  | 0.073 | 0.060    | 0.025   |        |       |              |    | Eff | С   | 42        | 18       |
| -1     |       |          |         |        |       |              |    |     |     |           |          |
| 16Mar  | 0.083 | 0.073    | 0.020   |        |       |              |    | Eff | С   | 42        | 12       |
| -1     |       |          |         |        |       |              |    |     |     |           |          |
| 19Mar  | 0.088 | 0.072    | 0.049   |        |       |              |    | Eff | С   | 42        | 18       |
| -1     |       |          |         |        |       |              |    |     |     |           |          |
| 19Mar  | 0.086 | 0.070    | 0.060   |        |       |              |    | Eff | М   | 42        | 19       |
| -2     |       |          |         |        |       |              |    |     |     |           |          |

| 20Mar<br>-1 | 0.085 | 0.074 | 0.037 |  |  | Eff | С | 39 | 13 |
|-------------|-------|-------|-------|--|--|-----|---|----|----|
| 20Mar<br>-2 | 0.086 | 0.073 | 0.035 |  |  | Eff | М | 33 | 15 |
| 21Mar<br>-1 | 0.085 | 0.073 | 0.035 |  |  | Eff | С | 50 | 14 |
| 21Mar<br>-2 | 0.082 | 0.073 | 0.033 |  |  | Eff | M | 33 | 11 |
| 22Mar<br>-2 | 0.083 | 0.069 | 0.033 |  |  | Eff | С | 39 | 17 |
| 03Apr-      | 0.095 | 0.055 | 0.045 |  |  | Eff | С | 46 | 42 |
| 10Apr-      | 0.099 | 0.044 | 0.042 |  |  | Eff | М | 46 | 56 |
| 11Apr-<br>1 | 0.094 | 0.042 | 0.039 |  |  | Eff | M | 33 | 55 |
| 11Apr-<br>2 | 1.16  | 0.045 | 0.042 |  |  | In  | М | 3  | 96 |

Average DAF Effluent Feed: 0.084 ppm total NI; 0.081 ppm Soluble Ni

Average DAF Influent Feed: 0.828 ppm total Ni; 0.098 ppm Soluble Ni



Sanitary District of Decatur (SDD) collected influent and effluent samples, three times per week, beginning in January 2012. Assuming the Sanitary District of Decatur, Waste Water Treatment Plant is removing precipitated Nickel during routine handling.

Table 13: Average Results from January 2012 SDD samples.

| Soluble Ni | Total Ni |
|------------|----------|
| (ppm)      | (ppm)    |

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| SDD Influent | 0.025 | 0.078 |
|--------------|-------|-------|
| SDD Effluent | 0.023 | 0.024 |

#### 2.2 Membrane Filtration

Salt Creek Tech: Hollow Fiber Membrane Filtration.

Salt Creek Tech equipment was in Decatur on a bench-scale; the equipment consisted of a hollow-fiber membrane filter. Tests with the hollow-fiber membrane achieved Nickel levels below permit limit, if Aluminum Sulfate (Alum) and Nalco TX were added to the feed, before processing. Screened solids had nickel removed, however no flux data was shared by the vendor on the bench top trials.

Table 14. DTC 75ppm Nalco; 300ppm Alum; 30 minute residence time; Samples processed with Hollow Fiber filter

|                       |          | Total Ni |               | Soluble Ni |
|-----------------------|----------|----------|---------------|------------|
|                       | Total Ni | Reduced  | Soluble Ni    | Reduced    |
| Sample Name           | (ppm)    | (%)      | (ppm)         | (%)        |
| Untreated Feed        | 0.080    |          | 0.07 <b>7</b> |            |
| Alum/DTC treated feed | 0.054    | 33       | 0.025         | 65         |
| Alum/DTC treated perm | 0.017    | 79       | 0.015         | 78         |
| Alum treated Feed     | 0.076    | 5        | 0.067         | 13         |
| Alum Treated Perm     | 0.055    | 31       | 0.055         | 28         |
| Raw Material Feed     | 0.071    | 11       | 0.069         | 10         |
| Raw Material Perm     | 0.042    | 48       | 0.039         | 48         |

ADM: Membrane Filtration

ADM trialed a hollow fiber membrane module to evaluate the approach from Salt Creek Technology. Starting material for ADM test was identical to Alum/DTC treated DAF Effluent used with the lowest nickel results from Salt Creek Tech. ADM started the test and allowed the experiment to run for ~5 hours. Sampled and measured permeate results are in <u>Table 15</u> below. Flux after one hour: 17.5 L/Hr/m² (0.43 gal/Hr/ft²)

Table 15. 75ppm Nalco TX300ppm Alum; 30 minute residence time; Test Filter: Pall USP-143 Hollow Fiber Module.

| Sample ID | Flux    | Flux       | Total Ni | Soluble Ni | Total Ni      |
|-----------|---------|------------|----------|------------|---------------|
|           | L/Hr/m² | Gal/Hr/ft² | (mg/kg)  | (mg/kg)    | Reduction (%) |
| Feed      | N.A.    | N.A.       | 0.102    | 0.082      |               |
| 10 min    | N.A.    | N.A.       | 0.020    | 0.021      | 80            |
| 60 min    | 17.5    | 0.43       | N.A.     | N.A.       |               |
| 140 min   | 11.8    | 0.29       | 0.021    | 0.020      | 79            |
| 290 min   | 8.5     | 0.21       | 0.020    | 0.020      | 80            |

## 2.3 Proprietary adsorbent

As part of continuing effort to screen Nickel removal technologies – ADM contacted

support, impregnated with ligands. Very preliminary results reported in <u>Table 16</u>. This test, completed at Tusaar, used ADM DAF Effluent, pumping 0.4 mL/min through 4.0 g of Tusaar media. The sample listed on the bottom row of <u>Table 16</u>, was obtained after ~4 liters DAF Effluent had passed through the media – which would be approaching 450 bed volumes of treated liquid. <u>All</u> samples processed with this media have been below 0.037 ppm total Nickel. ADM and Tusaar are discussing next steps for pilot planning.

Table 16. Selected Results of DAF Effluent treated at 0.4 mL/min through 4.0 grams of Media

|   |          |          | Total       |          |
|---|----------|----------|-------------|----------|
|   | Feed     | Product  | Bed Volumes | Total Ni |
| : | Total Ni | Total Ni | Processed   | Reduced  |
|   | (ppm)    | (ppm)    | (estimated) | (%)      |
|   | 0.096    | 0.002    | 67          | 98       |
|   | 0.096    | 0.006    | 124         | 94       |
|   | 0.096    | 0.010    | 180         | 90       |
|   | 0.096    | 0.012    | 236         | 87       |
|   | 0.096    | 0.014    | 292         | 85       |

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| 0.096 | 0.014 | 348 | 85 |
|-------|-------|-----|----|
| 0.096 | 0.015 | 449 | 84 |

## 2.4 Other approaches

#### **GEM (Gas Energy Mixing)**

In April 2012, ADM traveled to Clear Water Technology for an equipment demonstration. This technology utilizes a series of six, LSGM (Liquid Solid Gas Mixers) "heads" allowing different points of injection and mixing. Mixing streams (chemical precipitant, coagulant, flocculent) are added tangentially, to a vertical pipe, very much like a hydroclone. The two-phase system operates at 100-120 psi, with a variable amount of pipe filled with air. A thin liquid film circulates around the inside of pipe. Mixing energy is controlled by adjusting the port size, the number of ports/injection points and the levels inside pipe. Mixing energy can be adjusted:

- 1. to prevent overmixing
- 2. Uncoil long molecular chains, exposing more/all charge sites to particles.

Through the six LSGM heads, air is dissolved into waste water, prior to formation of floc – the dissolved air essentially becomes part of the floc structure once it is formed. As stream depressurizes and enters the floatation tank, air bubbles expand and push out water from the floc – making a very buoyant, drier sludge. Formation of air bubble and floc nucleation occur simultaneously. Examples at the Clean Water Technology showroom were still buoyant years after test were completed. The estimated test costs from March 2012 were \$38,367

Additionally we have plotted a summary of all soluble nickel reduction achieved as a function of the incoming nickel concentration in DAF effluent. As is evident we are not seeing a consistent reduction to below 0.037 ppm.

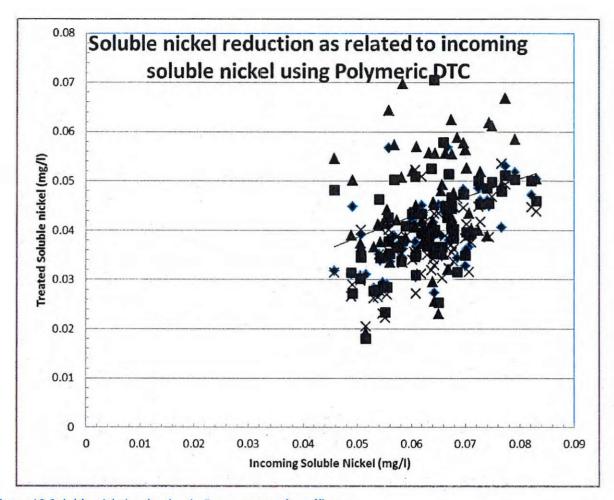


Figure 13 Soluble nickel reduction in Decatur complex effluent

#### 2.5 Toxicity trials

**River Bend Labs** 

Anaerobic Tox test for Nalco TX at: 0.01, 0.1, 1.0 10.0, & 100.0 ppm

Starting material for this test was Decatur High Salt Anaerobic Influent. This was tested at River Bend Labs (St Charles, MO). Summarizing River Bend results: Increasing the concentration of TX had no effect on Anaerobic Digesters. Gas production varied across every concentration; but, in general, all tests followed the same trend and were not statistically different.

#### Respirometery Test for:

- 1. mixed liquor city influent (control)
- 2. ADM DAF effluent
- 3. ADM DAF effluent, treated with ALAR RVF and mixed liquor (city)

River Bend Results: All tests behaved identical to control. The material seems to not be toxic to Heterophic bacteria at the city of Decatur.

#### Nitratox Test for:

- 1. Control DI water
- 2. City of Decatur Influent
- 3. 40% City of Decatur Influent; 60% ADM DAF Effluent
- 4. 40% City of Decatur Influent; 60% ADM DAF Effluent treated through the ALAR RVF.

River Bend Results: Nitrification inhibition occurred on all samples but the DI water control. This points to something inhibiting on the city influent to nitrification.

#### 3 Corn Plant used IX system

As previously disclosed, ADM has been working to install a used ion exchange resin bed system to capture Nickel leaching from the sorbitol process catalyst. This system ran manually for 6 weeks in late 2011. Thus far, about 5 lbs of Nickel have been removed from the treated stream and no Nickel has been detected in the effluent. This is shown in <u>Figure 14</u>. We are using 105 cu ft of resin and expect a Nickel binding capacity of about 3.4 lbs per cubic ft.

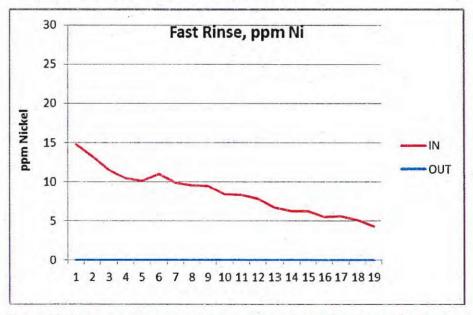


Figure 14 Used ion exchange resin treating material leaving the sorbitol process

The IX units went into continual operation in early 2012 and performance degraded by spring of 2012. In addition, a leak was discovered in a Mott sintered filter. Check filters were brought on line prior to IX in case this happens again.

No progress had been made by summer of 2012 in pin-pointing the cause of the performance problems. In July, we began employing continuous Isco samplers. <u>Figure 15</u> illustrates the erratic performance.

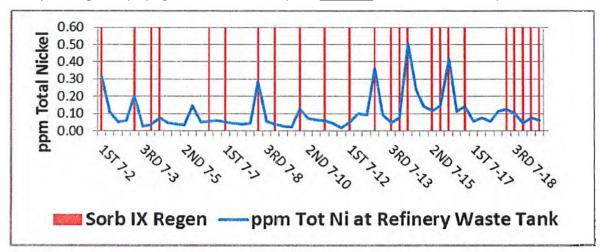


Figure 15 Nickel losses to WWTP during Sorbitol IX regenerations

An Isco sampler was moved to the Nickel bed inlet, but the source of the problem still could not be identified. The vessels weren't set up to sample the common outlet, so piping modifications were made in mid-November 2012. This will allow two Isco samplers to be used directly at the inlet and outlet of the beds. A new round of sampling began the week of 11/26/12.

#### 4 Review Ceased for Technologies

Since the summer 2012 update, we have completed all planned pilot trials. At present, we continue to monitor our effluent discharges and update the SDD. No additional pilot trials are planned at this time.

#### 5 Polyols IX waste stream treatment

We have identified our Polyols ix waste stream (between 16-22% of total Nickel load) as a significant contributor of inorganic Nickel due to corrosion of our distillation columns. Initial work using high pH precipitation has shown almost a complete removal of soluble Nickel.

Initial work suggests a pH modification would eliminate all soluble Nickel from the IX regen streams with chemical costs about \$300 per day. We have performed additional testing with 3M using cartridge filters for sizing of plant equipment. Capital expenditures for the plant system are estimated at \$450,000. Figure 16 is the layout of the proposed nickel removal system at Polyols. We expect the system to be installed in 2013 pending regulatory and environmental approvals.

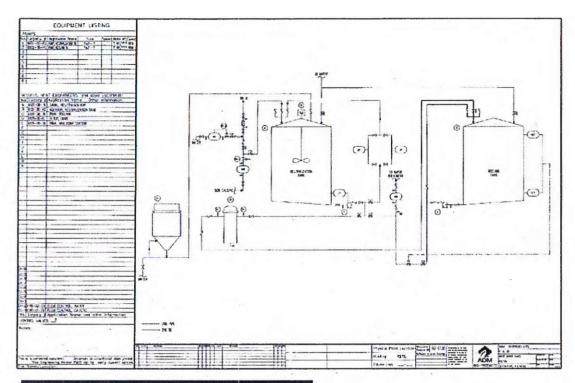


Figure 16

#### 6 Respirometer and Nitratox Testing

Results from Respirometer and Nitratox testing of Decatur Sanitary Districts MLSS using Nickel reduction chemistries piloted at ADM was shared with the district in May 2012. No additional tests were conducted as all the chemistries being piloted successfully passed the district's requirements on residual ammonia and respirometer performance.

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# EXHIBIT H

Interim Report June 27, 2013 Electronic Filing - Received, Clerk's Office: 02/21/2014

## Sanitary District of Decatur 501 DIPPER LANE • DECATUR, ILLINOIS 62522 • 217/422-6931 • FAX: 217/423-8171

June 27, 2013

Illinois Environmental Protection Agency Bureau of Water Compliance Assurance Section, MC #19 1021 North Grand Avenue East P.O. Box 19276 Springfield, Illinois 62794-9276

Re: NPDES Permit IL0028321

IPCB Order PCB 09-125

Interim Report

Dear Sir or Madam:

Enclosed is the Interim Report regarding compliance with nickel and zinc limits required by Special Condition 18 of the Sanitary District of Decatur's NPDES Permit and the Pollution Control Board Order in PCB 09-125.

Please contact me at 422-6931 ext. 214 or at timk@sddcleanwater.org if you have any questions regarding this report.

Sincerely,

Timothy R. Kluge, P.E.

Technical Director

Rick Pinneo, IEPA (via email) cc:

Bob Mosher, IEPA (via email)

SDD File

## Sanitary District of Decatur Nickel and Zinc Limits June 2013 Interim Report

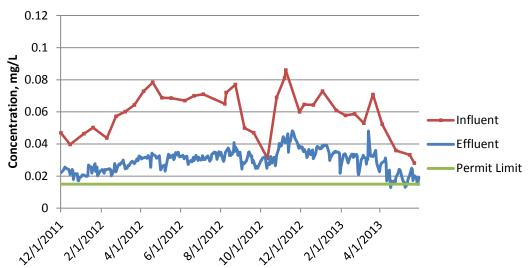
The modified NPDES permit for the Sanitary District of Decatur that became effective July 1, 2009 requires the District to achieve compliance with final nickel and zinc effluent limitations by July 1, 2010. Special Condition 17 also notes that the permit may be modified to include revised compliance dates in Pollution Control Board orders, and that prior to such permit modification, the revised dates in the appropriate orders shall govern the Permittee's compliance.

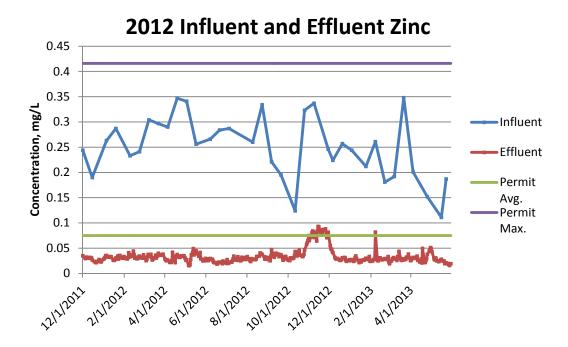
On January 7, 2010 the Illinois Pollution Control Board granted a variance to the District allowing additional time to comply with final permit limits (PCB 09-125). The compliance date contained in the Board Order is July 1, 2014. The District's NPDES Permit has not yet been modified or reissued to incorporate the variance. The Board Order also requires that an interim report be submitted to Illinois EPA by July 1, 2013. This report is submitted to meet both the permit and variance requirements.

#### **Plant Influent and Effluent Sampling**

Ongoing influent sampling for nickel and zinc continues at a frequency of twice monthly, and effluent sampling is done five days per week according to NPDES monitoring requirements. A summary of influent and effluent values during the past eighteen months is shown below.







Data shows that the plant effluent is not able to consistently meet the current nickel permit limit. Effluent zinc concentrations remain at or below the permit limit although a brief spike did occur in November 2012. Higher than normal concentrations of both nickel and zinc in the fall of 2012 are believed to be related to extreme drought conditions in central Illinois.

#### **Receiving Stream Sampling**

Upstream and downstream sampling continues at a twice monthly frequency to provide a more complete picture of nickel and zinc in the Sangamon River. One upstream and four downstream sampling sites are being monitored. A summary of 2011-2013 river monitoring data is attached. Downstream nickel results remain high during times of low upstream river flows, which prevailed from mid-2011 through 2012. The current nickel water quality standard has been met thus far in 2013, reflecting higher river flows. With one exception, upstream and downstream zinc results during the past two years have been below the Illinois chronic water quality standard.

#### **Pretreatment Ordinance Limits**

The District's pretreatment ordinance was amended in October 2009 as noted in previous interim reports.

#### **Stream Flow-Based Compliance Options**

The District continues investigation of flow-based permit limits, to take advantage of upstream flow for mixing when it is available. A USGS flow gauging station is located

about two miles upstream of the District's discharge point, and provides near-real time flow information. A proposal for flow-based limits will be a part of relief requested from the Pollution Control Board.

#### **Water Quality Standard Investigations**

The District is in the final stages of preparing a petition for a site-specific nickel standard, which we expect to file with the Pollution Control Board in the very near future. We are waiting for a determination from U.S. EPA regarding resolution of their most recent set of comments and questions.

Also, we anticipate that future permits will contain zinc limits based on the revised chronic water quality standard adopted by the Illinois Pollution Control Board in R11-18. Utilizing the corrected number to determine our permit limit should provide further assurance of compliance.

#### **Industrial Source Sampling and Investigations**

Sampling at Archer Daniels Midland Company for metals was increased early in 2013 from twice monthly to twice weekly, and other industries discharging metals are sampled quarterly. Sample results obtained from ADM within the past two years are attached.

The District's operating permit issued to ADM was modified on November 18, 2009 and again on June 17, 2010 to reflect the new limits and provide a compliance schedule for meeting the limits. Final local limits will be determined following Board action on the District's site-specific WQS request.

Both ADM and Tate & Lyle formerly utilized zinc as part of their cooling tower treatment programs, and both have greatly reduced zinc in their towers. At this time, both industries are meeting the zinc pretreatment limit. ADM is continuing to investigate the possible impact of the zinc limit on their planned wasting of solids from their pretreatment system to the District's collection system.

The discharge from ADM is by far the most significant industrial source of nickel. ADM has been very active in seeking treatment technology for nickel removal, involving plant management and research department personnel in addition to environmental compliance and legal staff. District staff continue regular contacts with ADM personnel. The District's pretreatment permit requires semi-annual reports of ADM's investigations, and the most recent report is attached. Completed and anticipated modifications made by ADM are listed on page 4 of the report.

#### **Additional Pretreatment Limit Investigations**

Pretreatment ordinance limits adopted in 2009 were adopted as total (rather than soluble) limits based on review of soluble/insoluble data. Refinement of pretreatment limits is an

ongoing process and will depend on final permit limits as well as treatment technologies that might be employed by industrial users.

### **Compliance Plan**

In summary, the District's compliance plan includes the following:

- 1. Continue to work with ADM to implement nickel discharge reductions and removal technologies. ADM's May 30, 2013 Interim Report describes the completed and planned reductions.
- 2. Complete and file a petition for a site-specific water quality standard for nickel, based on bioavailability. We have been working with Illinois EPA to address questions and comments through the summer and fall of 2012. Currently we are awaiting U.S. EPA's response to supplemental information provided in response to questions from Region 5 personnel.
- 3. The Board petition will contain a request for variable permit limits based on the amount of flow available in the Sangamon River.

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Nickel and Zinc River Data 2011-2013

|          |          | Ī        | Б.      | D:       | T .       | D:      |          |          |           |          | D:       | T T       |          |          |          |                      |
|----------|----------|----------|---------|----------|-----------|---------|----------|----------|-----------|----------|----------|-----------|----------|----------|----------|----------------------|
|          |          |          | River   | River    |           | River   |          |          |           | River    | River    |           | River    |          |          |                      |
|          | Plant    | River    | 100 yds | 600 yds  |           | Rock    | River    | Plant    | River     | 100 yds  | 600 yds  |           | Rock     | River    | Plant    | River                |
|          | Final    | Up-      | Down    | Down-    | Steven's  | Springs | Wyckle's | Final    | Up-       | Down     | Down-    | Steven's  | Springs  | Wyckle's | Final    | Up-                  |
|          | Effluent | stream   | stream  | stream   | Creek     | Bridge  | Road     | Effluent | stream    | stream   | stream   | Creek     | Bridge   | Road     | Effluent | stream               |
| Sample   | Nickel   | Nickel   | Nickel  | Nickel   | Nickel    | Nickel  | Nickel   | Zinc     | Zinc      | Zinc     | Zinc     | Zinc      | Zinc     | Zinc     | Flow     | Flow                 |
| Date     | mg/L     | mg/L     | mg/L    | mg/L     | mg/L      | mg/L    | mg/L     | mg/L     | mg/L      | mg/L     | mg/L     | mg/L      | mg/L     | mg/L     | mgd      | ft <sup>3</sup> /sec |
| 7/14/11  | 0.0170   | <0.00131 | 0.0118  | 0.0116   | <0.00131  | 0.00886 | 0.00890  | 0.0242   | 0.00519   | 0.0162   | 0.0171   | <0.00660  | 0.0136   | 0.0130   | 27.12    | 200                  |
| 7/28/11  | 0.0188   | <0.00131 | 0.0187  | 0.0168   | <0.00131  | 0.0158  | 0.0159   | 0.0255   | <0.00660  | 0.0279   | 0.0219   | <0.00660  | 0.0205   | 0.0207   | 27.85    | 2.1                  |
| 8/11/11  | 0.0218   | 0.00143  | 0.0255  | 0.0212   | < 0.00131 | 0.0204  | 0.0199   | 0.0294   | <0.00660  | 0.0576   | 0.0292   | < 0.00660 | 0.0266   | 0.0271   | 24.82    | 1.6                  |
| 8/25/11  | 0.0193   | <0.00131 | 0.0187  | 0.0190   | < 0.00131 | 0.0183  | 0.0189   | 0.0161   | <0.00660  | 0.0153   | 0.0158   | <0.00660  | 0.0142   | 0.0137   | 24.19    | 1.1                  |
| 9/8/11   | 0.0233   | 0.00142  | 0.0208  | 0.0222   | < 0.00131 | 0.0207  | 0.0196   | 0.0341   | < 0.00660 | 0.0294   | 0.0303   | < 0.00660 | 0.0279   | 0.0254   | 27.07    | 0.15                 |
| 9/14/11  | 0.0237   | 0.00132  | 0.0231  | 0.0235   | < 0.00131 | 0.0228  | 0.0231   | 0.0460   | < 0.00660 | 0.0425   | 0.0438   | <0.00660  | 0.0413   | 0.0385   | 28.62    | 1.9                  |
| 10/6/11  | 0.0276   | 0.00140  | 0.0263  | 0.0265   | < 0.00131 | 0.0255  | 0.0259   | 0.0329   | < 0.00660 | 0.0318   | 0.0314   | < 0.00660 | 0.0296   | 0.0288   | 23.96    | 0.75                 |
| 10/20/11 | 0.0211   | <0.00131 | 0.0189  | 0.0195   | < 0.00131 | 0.0159  | 0.0181   | 0.0260   | 0.0107    | 0.0235   | 0.0238   | < 0.00660 | 0.0193   | 0.0199   | 23.28    | 2.8                  |
| 11/3/11  | 0.0250   | 0.00197  | 0.0277  | 0.0304   | 0.00175   | 0.0260  | 0.0275   | 0.0322   | 0.0115    | 0.0314   | 0.0354   | < 0.00660 | 0.0281   | 0.0271   | 42.99    | 18                   |
| 11/17/11 | 0.0307   | <0.00131 | 0.0281  | 0.0283   | 0.00178   | 0.0273  | 0.0277   | 0.0368   | <0.00660  | 0.0285   | 0.0304   | <0.00660  | 0.0275   | 0.0247   | 25.80    | 1.1                  |
| 12/1/11  | 0.0221   | <0.00131 | 0.0177  | 0.0173   | <0.00131  | 0.0149  | 0.0149   | 0.0349   | 0.00728   | 0.0245   | 0.0230   | 0.00824   | 0.0207   | 0.0190   | 27.64    | 2.1                  |
| 1/5/12   | 0.0207   | <0.00131 | 0.0193  | 0.0206   | <0.00131  | 0.0170  | 0.0174   | 0.0355   | <0.00660  | 0.0328   | 0.0346   | <0.00660  | 0.0298   | 0.0278   | 27.19    | 4.1                  |
| 1/19/12  | 0.0245   | 0.00146  | 0.0164  | 0.0166   | 0.00135   | 0.0126  | 0.0127   | 0.0307   | 0.0265    | 0.0229   | 0.0240   | 0.00838   | 0.0203   | 0.0184   | 26.24    | 8.9                  |
| 2/9/12   | 0.0241   | <0.00131 | 0.00567 | 0.00496  | < 0.00131 | 0.00480 | 0.00421  | 0.0329   | <0.00660  | 0.00944  | 0.00838  | < 0.00660 | 0.00788  | 0.00782  | 29.94    | 228                  |
| 2/23/12  | 0.0227   | <0.00131 | 0.0135  | 0.0147   | < 0.00131 | 0.0118  | 0.0115   | 0.0343   | <0.00660  | 0.0213   | 0.0256   | <0.00660  | 0.0182   | 0.0172   | 28.01    | 50                   |
| 3/8/12   | 0.0245   | <0.00131 | 0.0111  | 0.0111   | < 0.00131 | 0.00964 | 0.00941  | 0.0338   | <0.00660  | 0.0167   | 0.0161   | <0.00660  | 0.0149   | 0.0150   | 27.78    | 79                   |
| 3/22/12  | 0.0277   | <0.00131 | 0.0241  | 0.0211   | <0.00131  | 0.0180  | 0.0185   | 0.0399   | <0.00660  | 0.0501   | 0.0387   | <0.00660  | 0.0245   | 0.0227   | 26.74    | 2.5                  |
| 4/5/12   | 0.0313   | <0.00131 | 0.0226  | 0.0226   | <0.00131  | 0.0205  | 0.0207   | 0.0260   | <0.00660  | 0.0214   | 0.0227   | <0.00660  | 0.0185   | 0.0172   | 26.05    | 4.6                  |
| 4/19/12  | 0.0334   | <0.00131 | 0.0246  | 0.0238   | 0.00149   | 0.0187  | 0.0199   | 0.0375   | <0.00660  | 0.0331   | 0.0308   | <0.00660  | 0.0240   | 0.0216   | 26.08    | 4.2                  |
| 5/3/12   | 0.0262   | 0.00158  | 0.0120  | 0.0105   | <0.00131  | 0.00755 | 0.00770  | 0.0270   | 0.00690   | 0.0231   | 0.0194   | <0.00660  | 0.0148   | 0.0142   | 26.95    | 8.7                  |
| 5/17/12  | 0.0317   | 0.00156  | 0.00859 | 0.00888  | 0.00141   | 0.00775 | 0.00806  | 0.0450   | <0.00660  | 0.0160   | 0.0171   | <0.00660  | 0.0139   | 0.0148   | 25.37    | 97                   |
| 6/7/12   | 0.0319   | 0.00259  | 0.0182  | 0.0173   | 0.00402   | 0.0160  | 0.0169   | 0.0296   | 0.0106    | 0.0180   | 0.0181   | < 0.00660 | 0.0163   | 0.0184   | 22.57    | 6.6                  |
| 6/21/12  | 0.0296   | 0.00136  | 0.0222  | 0.0218   | 0.00146   | 0.0215  | 0.0214   | 0.0225   | < 0.00660 | 0.0173   | 0.0165   | < 0.00660 | 0.0164   | 0.0139   | 23.81    | 0.06                 |
| 7/5/12   | 0.0303   | 0.00164  | 0.0247  | 0.0240   | 0.00217   | 0.0230  | 0.0232   | 0.0214   | <0.00660  | 0.0202   | 0.0165   | <0.00660  | 0.0139   | 0.0144   | 23.57    | 0.40                 |
| 7/19/12  | 0.0307   | 0.00195  | 0.0242  | 0.0236   | 0.00142   | 0.0234  | 0.0235   | 0.0289   | <0.00660  | 0.0250   | 0.0252   | <0.00660  | 0.0243   | 0.0230   | 23.18    | 0.10                 |
| 8/9/12   | 0.0356   | 0.00147  | 0.0250  | 0.0252   | 0.00160   | 0.0256  | 0.0248   | 0.0283   | < 0.00660 | 0.0221   | 0.0227   | < 0.00660 | 0.0232   | 0.0205   | 18.56    | 0.26                 |
| 8/23/12  | 0.0382   | 0.00185  | 0.0305  | 0.0305   | 0.00198   | 0.0302  | 0.0298   | 0.0374   | 0.00907   | 0.0330   | 0.0324   | < 0.00660 | 0.0314   | 0.0298   | 19.55    | 0.33                 |
| 9/6/12   | 0.0278   | 0.00206  | 0.0206  | 0.0212   | 0.00252   | 0.0169  | 0.0180   | 0.0471   | 0.0108    | 0.0253   | 0.0280   | 0.0100    | 0.0245   | 0.0229   | 20.73    | 1.3                  |
| 9/20/12  | 0.0289   | 0.00193  | 0.0228  | 0.0234   | 0.00160   | 0.0221  | 0.0226   | 0.0370   | 0.00772   | 0.0298   | 0.0304   | < 0.00660 | 0.0284   | 0.0280   | 18.57    | 0.27                 |
| 10/11/12 | 0.0280   | 0.00161  | 0.0192  | 0.0195   | 0.00150   | 0.0186  | 0.0180   | 0.0434   | <0.00660  | 0.0315   | 0.0303   | <0.00660  | 0.0281   | 0.0260   | 18.38    | 0.27                 |
| 10/25/12 | 0.0330   | 0.00152  | 0.0212  | 0.0216   | 0.00136   | 0.0184  | 0.0182   | 0.0462   | 0.00772   | 0.0312   | 0.0310   | <0.00660  | 0.0276   | 0.0232   | 28.23    | 2.90                 |
| 11/8/12  | 0.0409   | 0.00156  | 0.0345  | 0.0345   | 0.00141   | 0.0316  | 0.0324   | 0.0711   | <0.00660  | 0.0797   | 0.0778   | <0.00660  | 0.0707   | 0.0717   | 22.74    | 0.50                 |
| 11/29/12 | 0.0388   | 0.00168  | 0.0298  | 0.0307   | 0.00137   | 0.0287  | 0.0290   | 0.0815   | 0.00746   | 0.0649   | 0.0669   | 0.00783   | 0.0625   | 0.0603   | 22.74    | 0.41                 |
| 12/6/12  | 0.0367   | 0.00201  | 0.0292  | 0.0290   | <0.00131  | 0.0259  | 0.0249   | 0.0413   | 0.0110    | 0.0380   | 0.0374   | <0.00660  | 0.0324   | 0.0327   | 23.12    | 1.10                 |
| 12/20/12 | 0.0308   | 0.00174  | 0.0224  | 0.0247   | <0.00131  | 0.0132  | 0.0253   | 0.0311   | 0.0137    | 0.0199   | 0.0270   | 0.00722   | 0.0184   | 0.0206   | 33.13    | 21                   |
| 1/3/13   | 0.0380   | <0.00240 | 0.00569 | 0.00531  | <0.00240  |         | 0.00639  | 0.0274   | <0.00660  | <0.00660 | <0.00660 | <0.00660  | <0.00660 | 0.00717  | 23.43    | 372                  |
| 1/24/13  | 0.0348   | <0.00240 | 0.00948 | 0.00829  | <0.00240  |         | 0.00764  | 0.0301   | <0.00660  | 0.0121   | 0.0123   | <0.00660  |          | 0.00819  | 22.26    | 140                  |
| 2/7/13   | 0.0336   | <0.00240 | 0.00408 | 0.00363  | <0.00240  |         | 0.00309  | 0.0818   | <0.00660  | <0.00660 |          |           |          |          | 32.11    | 456                  |
| 2/21/13  | 0.0323   | <0.00240 | 0.00459 | 0.00328  | <0.00240  |         | 0.00332  | 0.0294   | <0.00660  | <0.00660 |          |           |          | <0.00660 | 25.77    | 351                  |
| 3/7/13   | 0.0318   | <0.00240 | 0.00372 | 0.00262  | <0.00240  |         |          | 0.0296   | <0.00660  | <0.00660 |          | <0.00660  |          | <0.00660 | 29.62    | 756                  |
| 3/21/13  | 0.0321   | <0.00240 | 0.00332 | 0.00294  | <0.00240  |         | 0.00267  | 0.0266   | <0.00660  | 0.00723  | <0.00660 | <0.00660  |          | <0.00660 | 24.92    | 375                  |
| 4/4/13   | 0.0285   | <0.00240 | 0.00321 | 0.00249  | <0.00240  |         | <0.00240 | 0.0317   | <0.00660  | 0.00836  | <0.00660 | <0.00660  | 0.00725  | 0.00698  | 29.55    | 659                  |
| 4/25/13  | 0.0196   | 0.00563  | 0.00563 | 0.00567  | 0.00332   | 0.00504 | 0.00562  | 0.0379   | 0.0288    | 0.0253   | 0.0269   | 0.0260    | 0.0225   | 0.0257   | 39.54    | 4410                 |
| 5/16/13  | 0.0208   | <0.00240 | 0.00290 | <0.00240 | <0.00240  |         | 0.00298  | 0.0281   | <0.00660  | 0.00758  | 0.00673  | 0.0069    | <0.00660 | <0.00660 | 29.25    | 895                  |
| 5/23/13  | 0.0203   | <0.00240 | 0.00267 | 0.00255  | <0.00240  |         |          | 0.0210   | <0.00660  | 0.00710  | 0.00802  | <0.00660  | 0.00665  | 0.00830  | 29.36    | 781                  |
| 6/6/13   | 0.0201   | <0.00240 | 0.00243 | 0.00255  | <0.00240  |         | 0.00259  | 0.0213   | <0.00660  | 0.0101   | 0.00850  | 0.00819   | 0.00720  | 0.0103   | 33.49    | 2440                 |
|          |          |          |         |          |           |         |          |          |           |          |          |           |          |          |          |                      |

|                        | ADM Nic        | kel and Zin | c Results      |                |
|------------------------|----------------|-------------|----------------|----------------|
|                        | ADM Point A    | ADM Point A | ADM Point D    | ADM Point D    |
| Sample                 | Nickel, Tot    | Zinc, Tot   | Nickel, Tot    | Zinc, Tot      |
| Date                   | mg/L           | mg/L        | mg/L           | mg/L           |
| 7/11/2011              | 0.0542         | 0.226       | 0.0625         | 0.209          |
| 8/1/2011               | 0.0491         | 0.165       | 0.0621         | 0.172          |
| 8/8/2011               | 0.0567         | 0.215       | 0.074          | 0.242          |
| 9/1/2011               | 0.0662         | 0.285       | 0.0842         | 0.327          |
| 9/7/2011               | 0.0684         | 0.311       | 0.0884         | 0.344          |
| 10/3/2011              | 0.094          | 0.518       | 0.114          | 0.515          |
| 10/10/2011             | 0.0643         | 0.191       | 0.073          | 0.189          |
| 11/7/2011              | 0.0912         | 0.377       | 0.116          | 0.529          |
| 11/22/2011             | 0.221          | 1.28        | 0.136          | 0.623          |
| 12/1/2011              | 0.0917         | 0.416       | 0.11           | 0.492          |
| 1/5/2011               | 0.094          | 0.423       | 0.117<br>0.111 | 0.508          |
| 1/9/2012               | 0.0868         | 0.424       | 0.109          | 0.491          |
| 2/6/2012               | 0.121          | 0.441       | 0.134          | 0.488          |
| 2/13/2012              | 0.127          | 0.49        | 0.159          | 0.601          |
| 3/5/2012               | 0.128          | 0.431       | 0.15           | 0.493          |
| 3/12/2012              | 0.12           | 0.406       | 0.141          | 0.482          |
| 4/12/2012              | 0.169          | 0.621       | 0.191          | 0.705          |
| 4/19/2012              | 0.148          | 0.516       | 0.176          | 0.674          |
| 5/1/2012               | 0.0797         | 0.251       | 0.152          | 0.564          |
| 5/7/2012               | 0.137          | 0.494       | 0.141          | 0.448          |
| 6/4/2012               | 0.133          | 0.412       | 0.147          | 0.468          |
| 6/11/2012              | 0.12           | 0.366       | 0.144          | 0.452          |
| 7/2/2012               | 0.129          | 0.375       | 0.158          | 0.462          |
| 7/9/2012<br>8/1/2012   | 0.109<br>0.127 | 0.322       | 0.132          | 0.402          |
| 8/6/2012               | 0.097          | 0.193       | 0.17           | 0.242          |
| 9/6/2012               | 0.105          | 0.289       | 0.117          | 0.271          |
| 9/10/2012              | 0.479          | 0.531       | 0.165          | 0.559          |
| 10/1/2012              | 0.15           | 0.46        | 0.168          | 0.54           |
| 10/8/2012              | 0.129          | 0.421       | 0.152          | 0.444          |
| 11/1/2012              | 0.16           | 0.487       | 0.184          | 0.568          |
| 11/12/2012             | 0.158          | 0.444       | 0.197          | 0.525          |
| 12/3/2012              | 0.127          | 0.387       | 0.157          | 0.45           |
| 12/10/2012             | 0.106          | 0.218       | 0.123          | 0.25           |
| 1/7/2013               | 0.14           | 0.374       | 0.181          | 0.448          |
| 1/14/2013              | 0.103          | 0.229       | 0.121          | 0.263          |
| 2/4/2013               | 0.13           | 0.313       | 0.142          | 0.329          |
| 2/11/2013<br>3/2/2013  | 0.116<br>0.139 | 0.265       | 0.147          | 0.308<br>0.235 |
| 3/4/2013               | 0.139          | 0.314       | 0.112          | 0.269          |
| 3/9/2013               | 0.122          | 0.283       | 0.129          | 0.289          |
| 3/11/2013              | 0.13           | 0.317       | 0.138          | 0.321          |
| 3/16/2013              | 0.134          | 0.355       | 0.156          | 0.431          |
| 3/20/2013              | 0.171          | 0.676       | 0.2            | 0.78           |
| 3/23/2013              | 0.158          | 0.578       | 0.191          | 0.686          |
| 3/27/2013              | 0.123          | 0.334       | 0.122          | 0.332          |
| 3/30/2013              | 0.122          | 0.356       | 0.127          | 0.371          |
| 4/3/2013               | 0.129          | 0.369       | 0.144          | 0.419          |
| 4/6/2013               | 0.118          | 0.266       | 0.102          | 0.16           |
| 4/8/2013               | 0.0832         | 0.151       | 0.0979         | 0.149          |
| 4/13/2013              | 0.107          | 0.279       | 0.118          | 0.303          |
| 4/15/2013<br>4/20/2013 | 0.09           | 0.246       | 0.116          | 0.3            |
| 4/24/2013              | 0.101          | 0.307       | 0.0829         | 0.273          |
| 4/27/2013              | 0.117          | 0.342       | 0.116          | 0.272          |
| 5/1/2013               | 0.0809         | 0.162       | 0.0945         | 0.157          |
| 5/4/2013               | 0.107          | 0.411       | 0.123          | 0.45           |
| 5/6/2013               | 0.0947         | 0.266       | 0.103          | 0.281          |
| 5/11/2013              | 0.0744         | 0.0981      | 0.0741         | 0.0749         |
| 5/15/2013              | 0.0867         | 0.204       | 0.108          | 0.226          |
| 5/18/2013              | 0.0871         | 0.0921      | 0.0932         | 0.0848         |
| 5/22/2013              | 0.103          | 0.283       | 0.109          | 0.28           |
| 5/25/2013              | 0.127          | 0.439       | 0.155          | 0.513          |
| 5/29/2013              | 0.145          | 0.574       | 0.181          | 0.691          |
| 6/1/2013               | 0.0913         | 0.111       | 0.0702         | 0.0883         |
| 6/3/2013               | 0.0884         | 0.1         | 0.0919         | 0.111          |

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To: Illinois Environmental Protection Agency

**Decatur Sanitary District** 

From: ADM Decatur WWTP

CC: ADM Corn Processing, ADM Oilseeds Processing, ADM JRRRC

Date: May 30, 2013

Re: Status Report Compliance Strategy for 2013 for Decatur Sanitary District and ADM

Decatur WWTP for waste treatment. (Covers updates post December 2012- date)



Electronic Filing - Received, Clerk's Office : 02/21/2014 - \* \* \* PCB 2014-111 \* \* \*

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Nickel and Zinc are present in effluent leaving the ADM Decatur Complex Waste Water plant. Of the two metals, nickel is more difficult to remove from the effluent. ADM Research and the ADM Decatur complex have been actively pursuing technologies to remove Nickel (Ni) from its effluent stream released to the SDD treatment plant. As part of our Industrial Users permit we are enclosing our updated technical report on our efforts to mitigate nickel in the Decatur complex effluent.

The IU shall submit reports to the SDD by December 1, 2011, and June 1 and December 1, 2012detailing 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

#### - ADM SDD Industrial Users Permit 200.

ADM met with the SDD and IEPA in December 2012 and provided them with an overview detailing the progress and ADM's compliance efforts. In addition ADM has provided the district and its attorneys with input in finalizing a draft petition for site specific rulemaking being discussed with IEPA. Enclosed is a report on the progress ADM has made since the last update issued on December 2012.

#### 1 Background and Update (post December 2012)

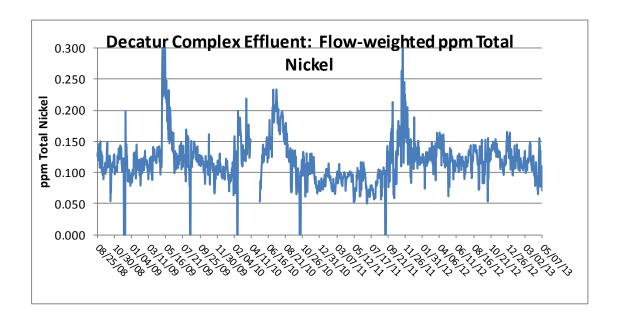
ADM has conducted 5 plant material balances to understand the sources of Nickel in its internal streams. ADM's Decatur Complex consists of multiple, separate processing plants, which send wastewater to the on-site wastewater treatment plant ("WWTP"). These processing plants consist of the Corn Wet Mill, BioProducts Plant, Cogeneration Plant, East Soybean Processing Plant, West Soybean Processing Plant, Vitamin E Plant, Corn Germ Processing Plant, Glycols Plant and the Polyols Plant. Each of these unique plants produces multiple products, using both batch and continuous processes, and creates water streams which generally are reused multiple times prior to being discharged to the WWTP. The WWTP treats approximately 11 MGD through a newer anaerobic treatment system followed by aerobic treatment prior to discharge to the District.

The incoming soybeans contain approximately 4.1 parts per million ("ppm") nickels, while incoming corn contains approximately 0.53 ppm nickel. Given that ADM's Decatur Complex processes approximately 600,000 bushels of corn and 200,000 bushels of soybeans per day, our incoming Nickel load is about 49.2 lbs from the soybeans and 19.1 lbs from the corn. A small portion of the incoming nickel is discharged in the effluent.

In the ADM Decatur Facility, effluent water originates in the corn and soybeans being processed at the facility. During the processing, the metals are released and enter the processing water, some of which eventually ends up at the wastewater treatment plant.

ADM has monitored soluble Nickel at the Damon and Front stations continuously (see <u>Figures 1-3</u>) and made a number of modifications in its operations:

- Our nickel in effluent discharge has been fairly stable and trending slightly down. We are seeing flow averaged total nickel to below 70ppb down from an historical average of 150 ppb.
- 2) We have noticed a small increase in our soluble nickel towards the end of 2012 (Figures 4-5) but this has not been directly attributable to any specific changes in our operations.
- Spent catalyst from the West Soybean Processing Plant is collected and sent to a landfill.
   Spilled catalyst is collected and disposed of as solid rather than washed into a sump.
- 4) Particulate catalyst from the Corn Plant Sorbitol production is captured by filters and physically recovered for recycling or disposal. ADM has installed an ion exchange resin system at the Sorbitol Plant to capture soluble nickel from wastewater. The system is undergoing startup and troubleshooting and results from testing are reported later in the current report.
- 5) The East Soybean Processing Plant is finalizing its design of a system that will remove the soy molasses stream (containing approximately 2.4 lb/day, approximately 35% of the soluble nickel from the Decatur Complex) from going to the WWTP. This stream is high in digestible, fermentable sugars but will need to be concentrated for stability. The East Soybean Processing Plant has prepared a cost estimate for this process change. Once the system design is complete and the cost estimate approved, ADM anticipates spending several million dollars to install it. Our anticipated start date for this project is June 2013 pending environmental and regulatory approvals.
- 6) The Polyols Plant accounts for approximately 11% of the soluble nickel from the Decatur Complex. The Polyols Plant has determined this nickel can be precipitated by pH adjustment. ADM is now determining how to implement this change on its process stream. As described later in this report, an internal request for funding ("AFE") has been submitted and we expect startup in July 2013 pending environmental and



**Figure 1 Flow Averaged Front and Damon Nickel** 

regulatory approvals.

7) We have also collected soluble nickel data for the past 8+ yrs. and it shows that our soluble nickel number remains largely unchanged with the only exception being the change in total nickel due to startup of the anaerobic digesters post August 2008 (this data was shared in June 2012 update).

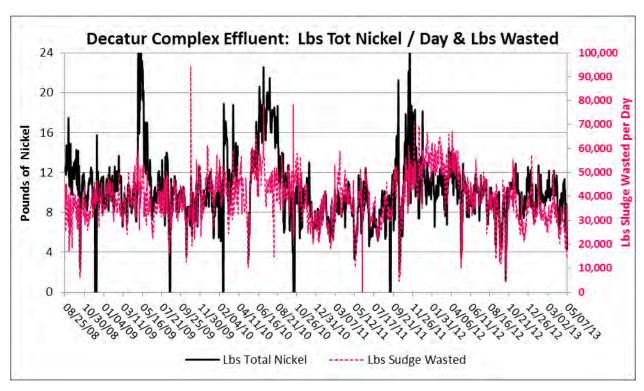


Figure 2 Decatur complex effluent- sludge wasting and nickel in effluent

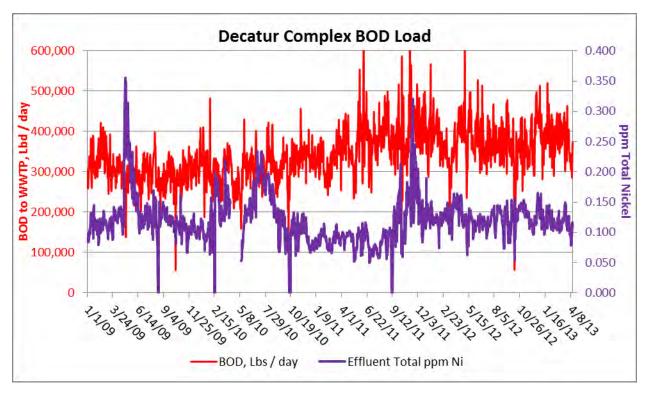


Figure 3 Decatur complex effluent- BOD and nickel in effluent

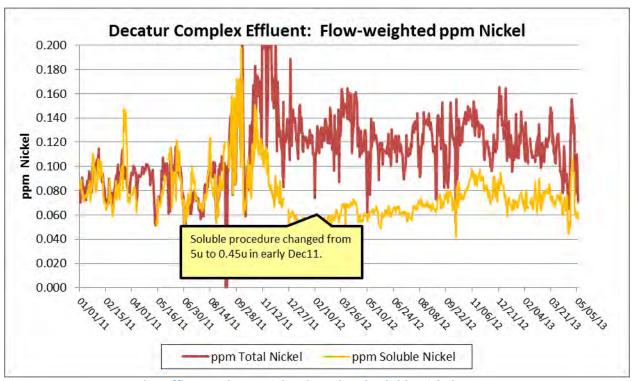


Figure 4 Decatur Complex effluent- Flow weighted total and soluble nickel

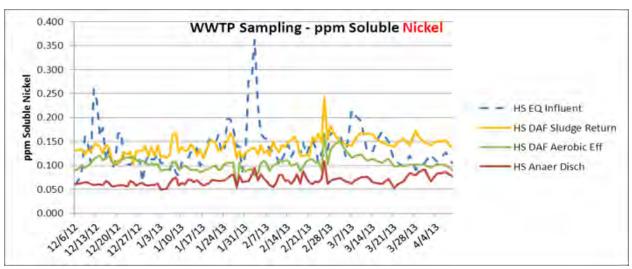


Figure 5 Soluble nickel through internal WWTP flows

As reported in the 2010 (summer) - 2012 (summer) updates, ADM has, thus far, investigated 46 technologies that had the potential to control nickel at the Decatur Complex WWTP. (This was in addition to the work ADM has undertaken to reduce nickel within the individual wastewater streams.) As indicated in <u>Table 1</u>, these technologies can be segregated into six broad categories:

- Nickel Proprietary Precipitation Process;
- 2. Nickel Chemical Precipitation;
- Ion Exchange Resin;
- 4. Filtration;
- 5. pH Modification
- 6. Noncommercial, Experimental Technologies.

Additional details about some of the technologies identified in <u>Table 1</u> are presented in <u>Table 2</u>, including a general list of reasons why certain of those technologies are not technically feasible and are not currently being pursued.

ADM has finished piloting the various technologies listed in our summer 2012 report. <u>Table 3</u>, summarizes the capital, operating and chemical costs for the approaches it is scaling and either installing or continuing to trial and a summary of their best results.

Thus, of all of the technologies investigated by ADM to date, the only viable option that has not already been fully planned, installed or employed by ADM is the nickel capture process based upon high pH precipitation at the Polyols Plant. Because such technology has been determined to be both technically feasible and economically reasonable for the specific application, ADM will install that system at the Polyols Plant after necessary pilot testing is complete. However, that reduction, even when combined with the other reductions achieved by ADM, will still not reduce nickel to the levels sought by the District under its current permit. Even if ADM could overcome the technical obstacles it faces regarding the use of polymeric dimethyl dithiocarbamate to reduce nickel from the final wastewater effluent, testing indicates that residual soluble nickel concentrations close to 0.050 mg/L will remain irrespective of contact time and incoming nickel levels. ADM's investment from 2009 to December 2011 to identify and implement viable solutions to meet the nickel standard has been approximately \$1.02 million in employee costs and \$0.45 million in equipment rental and pilot trial costs. In addition, ADM has spent \$0.45 million to install a resin capture system at the Decatur Sorbitol plant. It is also preparing to spend an additional \$2.5 million to install a system to allow removal of the soy molasses stream and roughly \$0.75 million to install a high pH precipitation and filtration process at the Polyols Plant. ADM has also significantly improved housekeeping in the West Plant to minimize nickel catalyst from entering the wastewater system. Finally, ADM has finished pilot trials on its ability to scale up a potentially viable chemical technology for installation at the Decatur Complex WWTP based on polymeric dimethyl dithiocarbamate to reduce nickel from its effluent. At this point, all reasonably Electronic Filing - Received, Clerk's Office : 02/21/2014 - \* \* \* PCB 2014-111 \* \* \*

identifiable options have been explored and all technically feasible and economically reasonable solutions are being pursued.

|   |             | Table 1 S | ummary of Technolog | ies Reviewed by ADM      |            |     |      |  |  |  |  |  |
|---|-------------|-----------|---------------------|--------------------------|------------|-----|------|--|--|--|--|--|
|   |             |           |                     |                          |            | Те  | Есо  |  |  |  |  |  |
|   |             |           |                     |                          |            | ch  | no   |  |  |  |  |  |
|   |             |           |                     |                          |            | nic | mic  |  |  |  |  |  |
|   |             |           |                     |                          |            | all | ally |  |  |  |  |  |
|   |             |           |                     |                          | Nitrat     | у   | Rea  |  |  |  |  |  |
|   |             |           |                     |                          | ox/Res     | Fe  | son  |  |  |  |  |  |
|   |             |           |                     |                          | pirom      | asi | abl  |  |  |  |  |  |
|   |             |           |                     |                          | eter       | ble | e    |  |  |  |  |  |
|   |             |           | Nickel Reduction    |                          | Testin     | (y/ | (y/  |  |  |  |  |  |
|   | Chemistry   | Dosage    | (%)                 | Current Status           | <i>g</i> * | n)  | n)   |  |  |  |  |  |
| Category 1 - Nickel Proprietary Precipitation Process |             |           |                     |                          |            |     |      |  |  |  |  |  |
|   |             |           |                     |                          |            |     |      |  |  |  |  |  |
|   |             | 1%-3%     |                     |                          |            |     |      |  |  |  |  |  |
|   |             | by        |                     |                          | Not        |     |      |  |  |  |  |  |
|   | Activated   | weight    | 40%-60% (from       | Not Active, High dosages | tested     |     |      |  |  |  |  |  |
|   | Clay        | of clay   | 200ppb influent)    | unscalable.              |            | No  | No   |  |  |  |  |  |
|   |             |           |                     |                          | Not        |     |      |  |  |  |  |  |
|   |             | 4%-8%     | 40% (from 90ppb     |                          | tested     |     |      |  |  |  |  |  |
|   | Acidic Clay | w/w       | influent)           | Stopped. High dosage.    |            | No  | No   |  |  |  |  |  |
|   |             |           |                     |                          |            |     |      |  |  |  |  |  |
|   |             |           |                     | Abandoned. High          | Not        |     |      |  |  |  |  |  |
|   | Chitosan    | 5%        | 90% from 200 ppb    | dosage, Concerns with    | tested     |     |      |  |  |  |  |  |
|   | Based       | w/w       | influent            | Chitosan Availability    | •          | No  | No   |  |  |  |  |  |
|   |             |           |                     |                          |            |     |      |  |  |  |  |  |
|   |             |           |                     |                          | Not        |     |      |  |  |  |  |  |
|   |             | 2%        | 82% (from           | Abandoned. Company       | tested     |     |      |  |  |  |  |  |
|   | Proprietary | w/w       | 100ppb)             | went out of business     | •          | No  | No   |  |  |  |  |  |

|          |               |         |                 | Shelved. Strong pH          |             |     |     |
|----------|---------------|---------|-----------------|-----------------------------|-------------|-----|-----|
|          |               |         |                 | swing (acidification to     | Not         |     |     |
|          |               | 200     | 64% (from       | pH 2, alkalination to 10    | tested      |     |     |
|          | Metclear      | ppm     | 120ppb)         | and neutralization)         |             | No  | No  |
|          |               | Not     | - 1-1 7         | ,                           | Not         |     |     |
|          | Not           | disclos | 40-60% (from    | Shelved. Company not        | tested      |     |     |
|          | disclosed     | ed      | 200ppb)         | sharing samples.            |             | No  | No  |
| Category |               |         |                 | g Carbamates or Organic Sul | ·<br>lfides | 110 |     |
| category | 2 Wicker Chem | 100pp   |                 | Gearbarnates or Organic Sur |             |     | 1   |
|          | Polymeric     | m with  |                 |                             |             |     |     |
|          | Dimethyl      | 50ppm   |                 |                             |             |     |     |
|          | Dithiocarba   | of      |                 | Piloted. Total Nickel       | Passe       |     |     |
|          | mate          | CaCl2   | 30% from 150ppb | reduction to 60ppb.         | d           | No  | No  |
|          | Polymeric     | Caciz   | 3070 ПОШ 130рро | reduction to copps.         | <u>u</u>    | 140 | 110 |
|          | Dimethyl      |         |                 |                             |             |     |     |
|          | Dithiocarba   | 20-     |                 | Piloted. Total Nickel       | Passe       | Ye  |     |
|          | mate          | 50ppm   | 60% from 150ppb | reduction to 54 ppb.        | d           | S   | No  |
|          |               | Зоррін  | 00% ПОШ 130рры  | reduction to 54 ppb.        | u           | 3   | NO  |
|          | Polymeric     |         |                 |                             |             |     |     |
|          | Dimethyl      | 100     |                 | Dilated Tatal Nieled        | D           | V   |     |
|          | Dithiocarba   | 100рр   | 440/ 5 450      | Piloted. Total Nickel       | Passe       | Ye  |     |
|          | mate          | m       | 41% from 150ppb | reduction to 32ppb          | d           | S   | No  |
|          | Dimethyl      | 50ppm   |                 |                             |             | .,  |     |
|          | Dithiocarba   | + pH    |                 | Piloted. Nickel reduction   | Passe       | Ye  |     |
|          | mate          | 6.0     | 76% from 150ppb | seen to 40ppb               | d           | S   | No  |
|          | Polymeric     |         |                 |                             |             |     |     |
|          | Dimethyl      | 300pp   |                 | Not active. Modified        | Not         |     |     |
|          | Dithiocarba   | m + pH  |                 | chemistry from Nalco        | tested      |     |     |
| 1        | mate          | swing   | 30%             | being tested                | •           | No  | No  |
|          | Polymeric     |         |                 | Piloted. Nickel reduction   | Passe       | Ye  |     |
|          | Dimethyl      | 50ppm   | 48% from 100ppb | seen to 20ppb               | d           | S   | No  |

|          | Dithiocarba                        |          |                   |                           |        |          |          |  |  |  |  |  |  |
|----------|------------------------------------|----------|-------------------|---------------------------|--------|----------|----------|--|--|--|--|--|--|
|          | mate                               |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Polymeric                          |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Dimethyl                           |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Dithiocarba                        | 200рр    |                   | Piloted. Nickel reduction | Passe  |          |          |  |  |  |  |  |  |
|          | mate                               | m        | 52% from 150ppb   | seen to 39 ppb            | d      | No       | No       |  |  |  |  |  |  |
|          | Polymeric                          |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Dimethyl                           |          |                   | Not piloted. GE has not   | Not    |          |          |  |  |  |  |  |  |
|          | Dithiocarba                        | 100рр    |                   | scaled up commercial      | tested |          |          |  |  |  |  |  |  |
|          | mate                               | m        | 40% from 150ppb   | manufacturing.            |        | No       | No       |  |  |  |  |  |  |
|          | Dimethyl                           |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Dithiocarba                        | 100рр    |                   | Piloted. Nickel reduction | Passe  |          |          |  |  |  |  |  |  |
|          | mate                               | m        | 60% from 150ppb   | seen to 24 ppb            | d      | No       | No       |  |  |  |  |  |  |
| Category | Category 3 - Non Functional Resins |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Styrene                            |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Divinyl                            | 2-5%     |                   | Not scaled. High          | Not    |          |          |  |  |  |  |  |  |
|          | Benzene                            | w/w      | 20%               | regeneration costs        | tested | No       | No       |  |  |  |  |  |  |
|          |                                    |          |                   | Not scaled. Very high     |        |          |          |  |  |  |  |  |  |
|          | Styrene                            |          |                   | resin use. Caustic        |        |          |          |  |  |  |  |  |  |
|          | Divinyl                            | 4%       |                   | /ethanol based            | Not    |          |          |  |  |  |  |  |  |
|          | Benzene                            | w/w      | 60%               | regeneration              | tested | No       | No       |  |  |  |  |  |  |
|          | Immobilized                        |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Ion                                |          |                   |                           |        |          |          |  |  |  |  |  |  |
|          | Exchange                           |          |                   |                           | Not    |          |          |  |  |  |  |  |  |
|          | Beads                              | 5%       | Not significant   | Shelved                   | tested | No       | No       |  |  |  |  |  |  |
| Category | 4 - Reuse of Ion                   | Exchange | Resin             |                           | I      | <u>I</u> | <u> </u> |  |  |  |  |  |  |
|          |                                    |          | Complete removal  |                           |        |          |          |  |  |  |  |  |  |
|          |                                    |          | of Ionic Nickel   |                           | Not    |          |          |  |  |  |  |  |  |
|          |                                    | 0.1-     | from the Sorbitol | Installed at Sorbitol     | requir | Ye       |          |  |  |  |  |  |  |
|          | Sulfonic                           | 0.5%     | plant waste       | plant                     | ed     | S        | Yes      |  |  |  |  |  |  |
|          |                                    |          |                   |                           |        |          |          |  |  |  |  |  |  |
| L        | I                                  | l .      | I                 |                           | l      | l        |          |  |  |  |  |  |  |

|      | Phosphate       | 80%        |                   |                         |        |    |   |
|------|-----------------|------------|-------------------|-------------------------|--------|----|---|
|      | precipitation   | recover    |                   |                         | Not    |    |   |
|      | + Reverse       | y of       |                   | Shelved. Brine disposal | requir |    |   |
|      | Osmosis         | feed       | 95%+ reduction    | issues. High capex      | ed     | No | N |
|      | Low             | 30%        |                   | 0 1                     |        |    |   |
|      | pressure        | recover    |                   |                         | Not    |    |   |
| T    | Reverse         | y of       |                   | Shelved. Brine disposal | requir |    |   |
|      | Osmosis         | feed       | 80% + reduction   | issues. High capex      | ed     | No | ١ |
|      |                 |            |                   |                         |        |    |   |
|      |                 | Not        |                   |                         | Not    |    |   |
|      |                 | disclos    |                   |                         | requir |    |   |
|      | Sand Filter     | ed         | 20% reduction     | Insufficient efficacy   | ed     | No | ١ |
| gory | 6 - Other Appro | aches      | I                 |                         |        | ı  | 1 |
|      |                 |            |                   | Company went out of     |        |    |   |
|      | Carbon          | Not        |                   | business. CD also binds | Not    |    |   |
|      | Aerogels        | tested     | Not tested        | other ions              | tested | No | ١ |
|      |                 | Not        | Higher Nickel due |                         |        |    |   |
|      | Electrochem     | disclos    | to leaching from  |                         | Not    |    |   |
|      | ical            | ed         | electrode plates  | Shelved after 4 trials. | tested | No | ١ |
|      | Ferric          | 100nn      |                   | Unscalable due to       | Not    |    |   |
|      | Chloride        | 100pp<br>m | 40%               | chloride limits         | tested | No | N |
|      | Cinoriae        |            | .070              |                         | teoted |    |   |
|      |                 | not        |                   |                         | Not    |    |   |
|      | Protein         | tested     | Not tested        | Lab scale only          | tested | No | ١ |
|      |                 | 5%         |                   |                         |        |    |   |
|      |                 | w/w +      |                   |                         |        |    |   |
|      | Hydrogen        | рН         |                   |                         |        |    |   |
|      | Peroxide        | adjust     |                   | Significant chemical    | Not    |    |   |
|      | and Ozone       | ment       | 20% from 150ppb   | usage                   | tested | No | N |

|             | Not     |                    |                          |        |    |     |
|-------------|---------|--------------------|--------------------------|--------|----|-----|
| Protein     | disclos |                    | Other ions compete with  | Not    |    |     |
| based       | ed      | Not tested         | nickel. Not scalable.    | tested | No | No  |
|             |         |                    |                          |        |    |     |
|             |         |                    |                          |        |    |     |
|             |         |                    |                          |        |    |     |
|             | 1-3%    |                    | Very high chemical       | Not    |    |     |
| pH Swing    | w/w     | 30% from 150ppb    | usage.                   | tested | No | No  |
|             |         | Complete for ionic |                          |        |    |     |
|             | 1-2%    | regeneration       | Being piloted at Polyols | Not    | Ye |     |
| pH >11.0    | w/w     | waste              | plant for waste stream   | tested | S  | Yes |
|             |         |                    | Bench scale trials       |        |    |     |
|             |         | Reduced soluble    | complete. Unable to      |        |    |     |
| Proprietary |         | nickel to below    | scale up due to startup  | Not    |    |     |
| Adsorbent   | 5%      | 0.037ppb           | nature of technology     | tested | No | No  |

| Table 2: Technical Ch | naller                              | iges (                                | on Sco                     | ale U                 | o for                                   | Nickel Re  | media                      | ntion Chemistries                       |
|-----------------------|-------------------------------------|---------------------------------------|----------------------------|-----------------------|---|--|----------------------------|---|
| Technology / Provider | 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<br>brine disposal<br>concerns | Technically Feasible (y/n) | Comments                                |
|                       | Х                                   |                                       | Х                          |                       |   |  | No                         |   |
|                       |                                     |                                       |                            |                       |   |  |                            | Would require 5 million pounds of       |
|                       |                                     | Х                                     |                            | Х                     |   |  | No                         | additive per day                        |
|                       |                                     |                                       |                            |                       |   |  |                            |   |
|                       |                                     |                                       | Х                          | х                     |   |  | No                         |   |
|                       |                                     |                                       |                            |                       |   |  |                            |   |
|                       | Х                                   |                                       |                            | х                     |   |  | No                         |   |
|                       |                                     |                                       |                            | Х                     |   |  | No                         | Requires a pH to <2 then to pH 5.5 then |

|          |          |          |          |          |          |          | to pH 10                                   |
|----------|----------|----------|----------|----------|----------|----------|--|
|          |          |          |          |          |          | 1        | το μιτ το                                  |
| Х        |          |          |          |          |          | No       |  |
|          |          |          |          |          |          |          | Plant pilot trial did not achieve required |
|          |          |          |          | Х        |          | No       | Nickel reduction.                          |
|          |          |          |          |          |          |          | Plant pilot trial did not achieve required |
|          | Х        |          |          | Х        |          | No       | Nickel reduction.                          |
|          |          |          |          |          |          |          | Plant pilot trial did not achieve required |
|          |          |          |          | Х        |          | No       | Nickel reduction.                          |
|          |          | Х        |          |          |          | No       |  |
|          |          |          |          |          |          | No       |  |
|          |          |          | Х        |          |          | 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     |
|          |          |          | Х        |          |          | No       | Ethanol to regenerate resin.               |
|          | Х        |          | Х        |          |          | No       |  |
|          |          |          |          |          |          | Yes      |  |
|          |          |          |          |          |          | *        | Installed at Sorbitol plant                |
|          |          |          |          |          | Х        | No       |  |
|          |          |          |          |          |          |          |  |
|          |          |          |          |          | Х        | No       |  |
|          |          |          |          |          |          |          |  |
|          |          |          |          |          | Х        | No       |  |
|          |          |          |          |          |          |          |  |
|          |          |          |          |          |          |          |  |
|          |          | х        |          |          |          | No       |  |
|          | Х        | Х        |          |          |          | No       |  |
|          |          |          |          |          |          |          | Requires over 30,000 pounds of ferric      |
|          | х        |          |          |          |          | No       | salts per day                              |
|          |          | Х        |          |          |          | No       |  |
| <u> </u> | <u> </u> | <u> </u> | <u> </u> | <u> </u> | <u> </u> | <u> </u> | <u>l</u>                                   |

|   |   |   |   |     | Raise the pH 10 and add ozone and         |
|---|---|---|---|-----|---|
|   |   |   |   |     | hydrogen peroxide. Large amounts of       |
|   | Х |   |   | No  | chemicals required.                       |
|   |   | Х |   | No  |   |
|   |   |   |   |     | Suitable for <~50,000 GPD, non-grain      |
|   |   |   |   |     | based wastewater with non-chelated,       |
|   |   |   |   |     | salt-form nickel such as Polyols Plant IX |
|   |   |   |   | Yes | regen waste                               |
|   |   |   |   |     | We are discussing a pilot trial with the  |
|   |   |   |   |     | vendor but they don't have capabilities   |
| х |   | x | х |     | to manufacture quantities required.       |

<sup>\*</sup> 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.

|                                     | Initial Capital<br>Cost | Annual Operating & Chemical Costs     | Status        |
|-------------------------------------|-------------------------|---------------------------------------|---------------|
| Active Projects                     |                         |                                       |               |
|                                     |                         |                                       | Currently     |
|                                     |                         |                                       | under         |
|                                     |                         |                                       | installation. |
|                                     |                         |                                       | Schedule for  |
| 1) Soybean Process Stream           |                         |                                       | June 2013     |
| Alternative                         | \$2.7 million           | \$400,000                             | startup       |
| 2) Used IX resin system at Sorbitol |                         |                                       | Installed     |
| Plant                               | \$450,000               | \$200,000                             |               |
|                                     |                         |                                       | Scheduled for |
| 3) High pH precipitation at Polyols |                         |                                       | July 2013     |
| Plant                               | \$750,000               | \$600,000                             | startup       |
| Further Technical Analysis/Cost     |                         | + + + + + + + + + + + + + + + + + + + |               |

|    | Prohibitive                       |                |                               |                 |
|----|-----------------------------------|----------------|-------------------------------|-----------------|
| 1) | Polymeric DTC addition and nickel |                |                               | Pilot trials    |
|    | removal using different unit      |                |                               | finished.       |
|    | operations                        | Not available  | Not available                 |                 |
|    |                                   |                |                               | Effluent        |
|    |                                   |                |                               | Irreversibly    |
|    |                                   |                |                               | fouled          |
|    |                                   |                |                               | Membrane.       |
|    |                                   |                |                               | Filtrate rate   |
|    | a) Single-Pass Membrane           |                |                               | decreased       |
|    | Filter                            | \$ 25million   | \$ 3 million                  | more than 90%   |
|    |                                   |                | \$ 2.3 million                | Average nickel  |
|    |                                   |                |                               | reduction       |
|    |                                   |                |                               | insufficient to |
|    | b) Dissolved Air                  |                |                               | meet proposed   |
|    | Floatation and Sand Filter        | \$ 15 million  |                               | limits          |
|    |                                   |                | \$ 2 million                  | Average nickel  |
|    |                                   |                |                               | reduction is    |
|    |                                   |                |                               | insufficient to |
|    | c) :                              |                |                               | meet proposed   |
|    | Dissolved Air Floatation Filter   | \$ 25million   |                               | limits          |
|    |                                   |                | \$ 1.5 million + \$30 million | Average nickel  |
|    |                                   |                | in disposal costs + 7000      | reduction is    |
|    |                                   |                | tons per day of clay          | insufficient to |
|    | d) Rotary                         |                | disposal.                     | meet proposed   |
|    | Vacuum Filter                     | \$ 1.8 million |                               | limits          |
|    |                                   |                | Not available                 | Average nickel  |
|    |                                   |                |                               | reduction is    |
|    | e) Enhanced                       |                |                               | insufficient to |
|    | Air Floatation Filter             | Not available  |                               | meet proposed   |

|                         |               |               | limits        |
|-------------------------|---------------|---------------|---------------|
|                         |               | \$2.1 million | Average Total |
|                         |               |               | Nickel        |
|                         |               |               | reduction to  |
|                         |               |               | 0.06 ppm and  |
|                         |               |               | insufficient  |
| f) Lamella Gravity      |               |               | proposed      |
| Settler and Sand Filter | \$6.2 million |               | limits        |
|                         |               | Not available | Technology is |
|                         |               |               | not scalable. |
|                         |               |               | Vendor has    |
|                         |               |               | only made     |
| g) Addition to          |               |               | multi gram    |
| Anaerobic Influent      | Not available |               | quantities.   |

#### 2 ADM Pilot trials update

As noted above ADM has completed its pilot plant trials for chemical sequestration of nickel and key cost numbers which are summarized in <u>Table 3</u>. Update on additional testing performed since December 2012 is summarized below.

## 2.1 Precipitation of Nickel

No additional testing was performed on precipitation based approaches for nickel removal.

#### 2.2 Proprietary adsorbent

As part of continuing effort to screen Nickel removal technologies – ADM contacted ., a start-up company, based in Lafayette, CO. Themedia is proprietary - a granular carbon based support, impregnated with ligands. Very preliminary results reported in <u>Table 16</u>. This test, completed at Tusaar, used ADM DAF Effluent, pumping 0.4 mL/min through 4.0 g of Tusaar media. The sample listed on the bottom row of <u>Table 16</u>, was obtained after ~4 liters DAF Effluent had passed through the media

 which would be approaching 450 bed volumes of treated liquid. <u>All</u> samples processed with this media have been below 0.037 ppm total Nickel. ADM and Tusaar are discussing next steps for pilot planning.

| Table 16. Selected Results of DAF Effluent treated at 0.4 mL/min through 4.0 grams of Medi | able 16. Selected Results of DAF Effluent treated at 0.4 mL/min through 4.0 grams of | Media. |
|--|--|--------|
|--|--|--------|

|          |          | Total       |          |
|----------|----------|-------------|----------|
| Feed     | Product  | Bed Volumes | Total Ni |
| Total Ni | Total Ni | Processed   | Reduced  |
| (ppm)    | (ppm)    | (estimated) | (%)      |
| 0.096    | 0.002    | 67          | 98       |
| 0.096    | 0.006    | 124         | 94       |
| 0.096    | 0.010    | 180         | 90       |
| 0.096    | 0.012    | 236         | 87       |
| 0.096    | 0.014    | 292         | 85       |
| 0.096    | 0.014    | 348         | 85       |
| 0.096    | 0.015    | 449         | 84       |

proposed an optimization process, at their Colorado facility (~\$70,000; 4-month study). Two parallel processes are being assembled: first will process "as is" DAF Effluent and the second will test 10 micron filtered DAF Effluent. Testing will begin in Summer 2013.

<u>Siemens Industry, Inc.</u> - (Advanced Reactive Media System) Proprietary treatment process – an iron containing media, acts as an electron generator to chemically reduce soluble metal cations and oxyanions to insoluble forms. The treated contaminants are removed by surface adsorption and chemical incorporation into iron oxidation products. Downstream solids separation would be separate process. This technology is still under development. Siemens offered to conduct a Static Test evaluation of DAF Effluent, based on results – further discussion will be evaluated.

## 2.3 Other approaches

We are not pursuing other new approaches at this time.

#### 2.4 Toxicity trials

No additional trials were done as we have not looked at any new chemistries beyond what we reported in December 2012.

#### 3 Corn Plant used IX system

As previously disclosed, ADM has been working to install a used ion exchange resin bed system to capture Nickel leaching from the sorbitol process catalyst. This system ran manually for 6 weeks in late 2011. During this trial, about 5 lbs of Nickel had been removed from the treated stream and no Nickel has been detected in the effluent. We are using 105 cu ft of resin and expect a Nickel binding capacity of about 3.4 lbs per cubic ft.

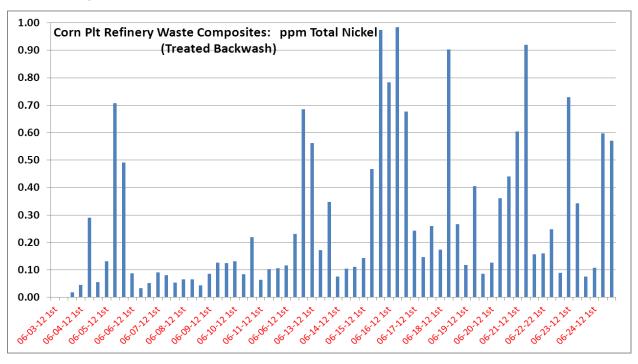


Figure 6 Used ion exchange resin treating material leaving the sorbitol process

The IX units went into continual operation in early 2012 and performance degraded by spring of 2012. In addition, a leak was discovered in a Mott sintered filter. Check filters were brought on line prior to IX in case this happens again.

An Isco sampler was moved to the Nickel bed inlet, but the source of the problem still could not be identified. The vessels weren't set up to sample the common outlet, so piping modifications were made in mid-November 2012. This allowed direct inlet and outlet of the beds to be sampled and compared against the overall Corn Plant Refinery waste.

Figure 6 shows a significant number of elevated nickel concentrations leaving the Corn Plant Refinery. One of the IX regeneration steps being treated (Backwash step) was deemed to be causing performance issues due to entrained air. Since this step is least likely to contain nickel, it was programmed to by-pass the nickel beds.

Figure 7 shows improved performance in the Refinery Waste from Dec12 thru mid-Jan13 with Backwash by-passing the nickel beds.

With some nickel spikes still showing up that were not coming from the nickel beds, it was thought that IX valves might be intermittently leaking by. A programming change for a valve at the main waste line eliminated the effects of such leaks. Figure 8 indicates even more improvement in the overall Corn Plant Refinery Waste.

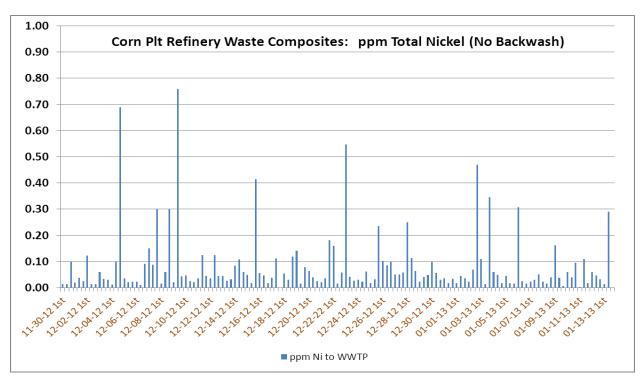


Figure 7

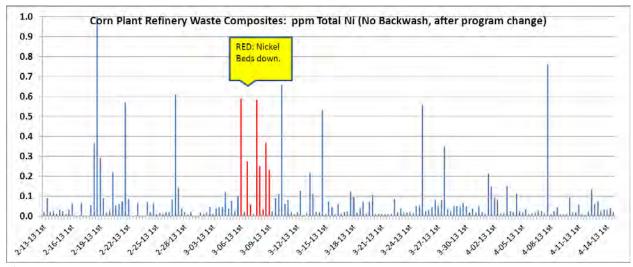


Figure 8

#### 4 Review Ceased for Technologies

Since the summer 2012 update, we have completed all planned pilot trials. At present, we continue to monitor our effluent discharges and update the SDD. We are planning a pilot trial with active media and will be conducting a material balance once the East Plant evaporator and Polyol IX waste stream treatment is online.

#### 5 East Plant Soy Molasses Stream Removal

As indicated previously the soy molasses stream from Decatur East Plant is about 35% of the soluble nickel load in the effluent. As part of our petition to the SDD we have indicated our intention to evaporate and eliminate this stream from our WWTP. We are currently proceeding through the installation of the equipment. Figure below is the evaporator being unloaded earlier this week. We anticipate startup in June 2013.



Figure 9 Evaporator installation for East Plant Soy Molasses Removal

#### 6 Polyols IX waste stream treatment

We have identified our Polyols IX waste stream (between 16-22% of total Nickel load) as a significant contributor of inorganic Nickel. Initial work using high pH precipitation has shown almost complete removal of soluble Nickel. A commercial system is being installed to remove nickel.

We envision this system to be online July 2013.

#### 7 Respirometer and Nitratox Testing

Results from Respirometer and Nitratox testing of Decatur Sanitary Districts MLSS using Nickel reduction chemistries piloted at ADM was shared with the district in May 2012. No additional tests were conducted as all the chemistries being piloted successfully passed the district's requirements on residual ammonia and respirometer performance.

Electronic Filing - Received, Clerk's Office : 02/21/2014 - \* \* \* PCB 2014-111 \* \* \*

# **EXHIBIT I**

Interim Report December 20, 2013 Electronic Filing - Received, Clerk's Office: 02/21/2014 - \* \* \* PCB 2014-111 \* \* \*

### Sanitary District of Decatur

501 DIPPER LANE • DECATUR, ILLINOIS 62522 • 217/422-6931 • FAX: 217/423-8171

December 20, 2013

Illinois Environmental Protection Agency Bureau of Water Compliance Assurance Section, MC #19 1021 North Grand Avenue East P.O. Box 19276 Springfield, Illinois 62794-9276

Re:

NPDES Permit IL0028321 IPCB Order PCB 09-125

Interim Report

Dear Sir or Madam:

Enclosed is the Interim Report regarding compliance with nickel and zinc limits required by Special Condition 18 of the Sanitary District of Decatur's NPDES Permit and the Pollution Control Board Order in PCB 09-125.

Please contact me at 422-6931 ext. 214 or at timk@sddcleanwater.org if you have any questions regarding this report.

Sincerely,

Timothy R. Kluge, P.E.

Technical Director

cc:

Rick Pinneo, IEPA (via email)

Bob Mosher, IEPA (via email)

SDD File

#### Sanitary District of Decatur Nickel and Zinc Limits December 2013 Interim Report

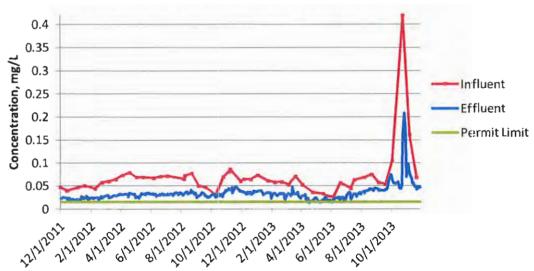
The modified NPDES permit for the Sanitary District of Decatur that became effective July 1, 2009 requires the District to achieve compliance with final nickel and zinc effluent limitations by July 1, 2010. Special Condition 17 also notes that the permit may be modified to include revised compliance dates in Pollution Control Board orders, and that prior to such permit modification, the revised dates in the appropriate orders shall govern the Permittee's compliance.

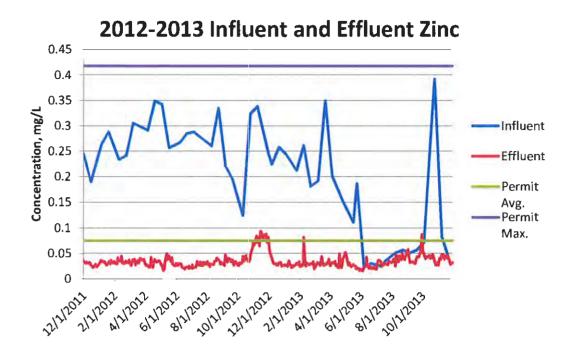
On January 7, 2010 the Illinois Pollution Control Board granted a variance to the District allowing additional time to comply with final permit limits (PCB 09-125). The compliance date contained in the Board Order is July 1, 2014. The District's NPDES Permit has not yet been modified or reissued to incorporate the variance. The Board Order also requires that an interim report be submitted to Illinois EPA by January 1, 2014. This report is submitted to meet both the permit and variance requirements.

#### Plant Influent and Effluent Sampling

Ongoing influent sampling for nickel and zinc continues at a frequency of twice monthly, and effluent sampling is done five days per week according to NPDES monitoring requirements. A summary of influent and effluent values during the past eighteen months is shown below.







The charts show a substantial spike in influent nickel and zinc occurring in October and November, 2013. Effluent nickel also spiked during this time period although effluent zinc concentrations remained at or below the permit limit. The influent concentration increase is attributed to a process change at ADM's pretreatment facility that was implemented to reduce long-term discharges of nickel. This change involved the segregation of a soy solubles waste stream from the wastewater pretreatment system and redirection of that waste into a new evaporation process and ultimately to an animal feed product. The soy solubles waste stream was a significant source of nickel discharges and was eliminated as part of ADM's effort to reduce their total nickel discharge. The process change, however, also substantially reduced the organic loading on the pretreatment system which in turn led to a pH change in the treatment facility. The pH decrease resulted in resolubilization of metals from sludge in the system and the higher discharge concentrations. Both nickel and zinc were trending toward reduced levels by mid-December.

The District's treatment facility maintained excellent removal of zinc and the final zinc permit limit was achieved. However, nickel is received primarily in the soluble form and is not removed as well as zinc in the District's facility, as zinc is received primarily in the insoluble form.

#### **Receiving Stream Sampling**

Upstream and downstream sampling continues at a twice monthly frequency to provide a more complete picture of nickel and zinc in the Sangamon River. One upstream and four downstream sampling sites are being monitored. A summary of 2012-2013 river monitoring data is attached. Downstream nickel results remain high during times of low

upstream river flows, which prevailed in the summer and fall months of both 2012 and 2013. With one exception, upstream and downstream zinc results during the past two years have been below the Illinois chronic water quality standard.

#### **Pretreatment Ordinance Limits**

The District's pretreatment ordinance was amended in October 2009 as noted in previous interim reports.

#### **Stream Flow-Based Compliance Options**

The District continues investigation of flow-based permit limits, to take advantage of upstream flow for mixing when it is available. A USGS flow gauging station is located about two miles upstream of the District's discharge point, and provides near-real time flow information. A proposal for flow-based limits will be a part of relief requested from the Pollution Control Board.

#### Water Quality Standard Investigations

The District is in the final stages of preparing a petition for a site-specific nickel standard, which we expect to file with the Pollution Control Board in the very near future. During the summer and fall of 2013, numerous discussions were held with Illinois EPA and U.S. EPA to try to resolve questions regarding the District's draft proposal. We have completed a draft plan for Water Effect Ratio testing as has been discussed to provide additional confirmation for the Biotic Ligand Model, and expect to perform the testing very early in 2014.

Also, we anticipate that future permits will contain zinc limits based on the revised chronic water quality standard adopted by the Illinois Pollution Control Board in R11-18. Utilizing the corrected number to determine our permit limit should provide further assurance of compliance.

#### **Industrial Source Sampling and Investigations**

Sampling for metals at Archer Daniels Midland Company continues at a twice monthly frequency, and other industries discharging metals are sampled quarterly. Sample results obtained from ADM within the past two years are attached.

The District's operating permit issued to ADM was modified on November 18, 2009 and again on June 17, 2010 to reflect the new limits and provide a compliance schedule for meeting the limits. Final local limits will be determined following Board action on the District's site-specific WQS request.

Both ADM and Tate & Lyle formerly utilized zinc as part of their cooling tower treatment programs, and both have greatly reduced zinc in their towers. At this time, both industries are meeting the zinc pretreatment limit. ADM is continuing to investigate

the possible impact of the zinc limit on their planned wasting of solids from their pretreatment system to the District's collection system.

The discharge from ADM is by far the most significant industrial source of nickel. ADM has been very active in seeking treatment technology for nickel removal, involving plant management and research department personnel in addition to environmental compliance and legal staff. District staff continue regular contacts with ADM personnel.

ADM has implemented two significant nickel reduction treatment processes at its facility and a third is scheduled for startup in early 2014. The two completed projects include an ion exchange system implemented during April 2013 in the Sorbitol area to reduce nickel catalyst loading, and the soy solubles waste stream evaporation project noted above which began operation in October 2013. A precipitation and filtration treatment system for ADM's Polyol manufacturing process is scheduled to begin operation within the next few months.

#### **Additional Pretreatment Limit Investigations**

Pretreatment ordinance limits adopted in 2009 were adopted as total (rather than soluble) limits based on review of soluble/insoluble data. Refinement of pretreatment limits is an ongoing process and will depend on final permit limits as well as treatment technologies that might be employed by industrial users.

#### Compliance Plan

In summary, the District's compliance plan includes the following:

- 1. Continue to work with ADM as they implement remaining nickel discharge reductions and removal technologies.
- 2. Complete and file a petition for a site-specific water quality standard for nickel, based on bioavailability. We have been working with Illinois EPA to address questions and comments through the summer and fall of 2013. We anticipate completing effluent toxicity testing very early in 2014 as noted above. Because of the extended length of time involved in discussions with U.S. EPA, a request to extend the existing variance is being considered.
- 3. The Board petition for site-specific relief will contain a request for variable permit limits based on the amount of flow available in the Sangamon River.

## Electronic Filing - Reseived: Clack's Affice: 02/21/2014 - \* \* \* PCB 2014-111 \* \* \*

Nickel and Zinc River Data 2012-2013

|          |          | _         |         |          |           |          |          |          |          |          |          |          |          |          |          | <del></del>          |
|----------|----------|-----------|---------|----------|-----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------------------|
|          |          |           | River   | River    |           | River    |          |          |          | River    | River    |          | River    |          |          | ĺ                    |
|          | Plant    | River     | 100 yds | 600 yds  |           | Rock     | River    | Plant    | River    | 100 yds  | 600 yds  |          | Rock     | River    | Plant    | River                |
| 1        | Final    | Up-       | Down    | Down-    | Steven's  | Springs  | Wyckle's | Final    | Up-      | Down     | Down-    | Steven's | Springs  | Wyckle's | Final    | Up-                  |
|          | Effluent | stream    | stream  | stream   | Creek     | Bridge   | Road     | Effluent | stream   | stream   | stream   | Creek    | Bridge   | Road     | Effluent | stream               |
| Sample   | Nickel   | Nickel    | Nickel  | Nickel   | Nickel    | Nickel   | Nickel   | Zinc     | Flow     | Flow                 |
| Date     | mg/L     | mg/L      | mg/L    | mg/L     | mg/L      | mg/L     | mg/L     | mg/L     | mg/L     | mg/L     | mg/L     | mg/L     | mg/L     | mg/L     | mgd      | ft <sup>3</sup> /sec |
| 1/5/12   | 0.0207   | <0.00131  | 0.0193  | 0.0206   | <0.00131  | 0.0170   | 0.0174   | 0.0355   | <0.00660 | 0.0328   | 0.0346   | <0.00660 | 0.0298   | 0.0278   | 27.19    | 4.1                  |
| 1/19/12  | 0.0245   | 0.00146   | 0.0164  | 0.0166   | 0.00135   | 0.0126   | 0,0127   | 0.0307   | 0.0265   | 0.0229   | 0.0240   | 0.00838  | 0.0203   | 0.0278   | 26.24    | 8.9                  |
| 2/9/12   | 0.0241   | <0.00131  | 0.00567 | 0.00496  | < 0.00131 | 0.00480  | 0.00421  | 0.0329   | <0.00660 | 0.00944  | 0.00838  | <0.00660 | 0.00788  | 0.00782  | 29.94    | 228                  |
| 2/23/12  | 0.0227   | <0.00131  | 0.0135  | 0.0147   | < 0.00131 | 0.0118   | 0.0115   | 0.0343   | <0.00660 | 0.0213   | 0.0256   | <0.00660 | 0.0182   | 0.0172   | 28.01    | 50                   |
| 3/8/12   | 0.0245   | <0.00131  | 0.0111  | 0.0111   | < 0.00131 | 0.00964  | 0.00941  | 0.0338   | <0.00660 | 0.0167   | 0.0161   | <0.00660 | 0.0149   | 0.0150   | 27.78    | 79                   |
| 3/22/12  | 0.0277   | < 0.00131 | 0.0241  | 0.0211   | < 0.00131 | 0.0180   | 0.0185   | 0.0399   | <0.00660 | 0.0501   | 0.0387   | <0.00660 | 0.0245   | 0.0227   | 26.74    | 2.5                  |
| 4/5/12   | 0.0313   | < 0.00131 | 0.0226  | 0.0226   | <0.00131  | 0.0205   | 0.0207   | 0.0260   | <0.00660 | 0.0214   | 0.0227   | <0.00660 | 0.0185   | 0.0172   | 26.05    | 4.6                  |
| 4/19/12  | 0.0334   | <0.00131  | 0.0246  | 0.0238   | 0.00149   | 0.0187   | 0.0199   | 0.0375   | <0.00660 | 0.0331   | 0.0308   | <0.00660 | 0.0240   | 0.0216   | 26.08    | 4.2                  |
| 5/3/12   | 0.0262   | 0.00158   | 0.0120  | 0.0105   | <0.00131  | 0.00755  | 0.00770  | 0.0270   | 0.00690  | 0.0231   | 0.0194   | <0.00660 | 0.0148   | 0.0142   | 26.95    | 8.7                  |
| 5/17/12  | 0.0317   | 0.00156   | 0.00859 | 0.00888  | 0.00141   | 0.00775  | 0.00806  | 0.0450   | <0.00660 | 0.0160   | 0.0171   | <0.00660 | 0.0139   | 0.0148   | 25.37    | 97                   |
| 6/7/12   | 0.0319   | 0.00259   | 0.0182  | 0.0173   | 0.00402   | 0.0160   | 0.0169   | 0.0296   | 0.0106   | 0.0180   | 0.0181   | <0.00660 | 0.0163   | 0.0184   | 22.57    | 6.6                  |
| 6/21/12  | 0.0296   | 0.00136   | 0.0222  | 0.0218   | 0.00146   | 0.0215   | 0.0214   | 0.0225   | <0.00660 | 0.0173   | 0.0165   | <0.00660 | 0.0164   | 0.0139   | 23.81    | 0.06                 |
| 7/5/12   | 0.0303   | 0.00164   | 0.0247  | 0.0240   | 0.00217   | 0.0230   | 0.0232   | 0.0214   | <0.00660 | 0.0202   | 0.0165   | <0.00660 | 0.0139   | 0.0144   | 23.57    | 0.40                 |
| 7/19/12  | 0.0307   | 0.00195   | 0.0242  | 0.0236   | 0.00142   | 0.0234   | 0.0235   | 0.0289   | <0.00660 | 0.0250   | 0.0252   | <0.00660 | 0.0243   | 0.0230   | 23,18    | 0.10                 |
| 8/9/12   | 0.0356   | 0.00147   | 0.0250  | 0.0252   | 0.00160   | 0.0256   | 0.0248   | 0.0283   | <0.00660 | 0.0221   | 0.0227   | <0.00660 | 0.0232   | 0.0205   | 18.56    | 0.26                 |
| 8/23/12  | 0.0382   | 0.00185   | 0.0305  | 0.0305   | 0.00198   | 0.0302   | 0,0298   | 0.0374   | 0.00907  | 0.0330   | 0.0324   | <0.00660 | 0.0314   | 0.0298   | 19.55    | 0.33                 |
| 9/6/12   | 0.0278   | 0.00206   | 0.0206  | 0.0212   | 0.00252   | 0.0169   | 0.0180   | 0.0471   | 0.0108   | 0.0253   | 0.0280   | 0.0100   | 0.0245   | 0.0229   | 20.73    | 1.3                  |
| 9/20/12  | 0.0289   | 0.00193   | 0.0228  | 0.0234   | 0.00160   | 0.0221   | 0.0226   | 0.0370   | 0.00772  | 0.0298   | 0.0304   | <0.00660 | 0.0284   | 0.0280   | 18.57    | 0.27                 |
| 10/11/12 | 0.0280   | 0.00161   | 0.0192  | 0.0195   | 0.00150   | 0.0186   | 0.0180   | 0.0434   | <0.00660 | 0.0315   | 0.0303   | <0.00660 | 0.0281   | 0.0260   | 18.38    | 0.27                 |
| 10/25/12 | 0.0330   | 0.00152   | 0.0212  | 0.0216   | 0.00136   | 0.0184   | 0.0182   | 0.0462   | 0.00772  | 0.0312   | 0.0310   | <0.00660 | 0.0276   | 0.0232   | 28.23    | 2.90                 |
| 11/8/12  | 0.0409   | 0.00156   | 0.0345  | 0.0345   | 0.00141   | 0.0316   | 0.0324   | 0.0711   | <0.00660 | 0.0797   | 0.0778   | <0.00660 | 0.0707   | 0.0717   | 22.74    | 0.50                 |
| 11/29/12 | 0.0388   | 0.00168   | 0.0298  | 0.0307   | 0.00137   | 0.0287   | 0.0290   | 0.0815   | 0.00746  | 0.0649   | 0.0669   | 0.00783  | 0.0625   | 0.0603   | 22.74    | 0.41                 |
| 12/6/12  | 0.0367   | 0.00201   | 0.0292  | 0.0290   | <0.00131  | 0.0259   | 0.0249   | 0.0413   | 0.0110   | 0.0380   | 0.0374   | <0.00660 | 0.0324   | 0.0327   | 23.12    | 1.10                 |
| 12/20/12 | 0.0308   | 0.00174   | 0.0224  | 0.0247   | <0.00131  | 0.0132   | 0.0253   | 0.0311   | 0.0137   | 0.0199   | 0.0270   | 0.00722  | 0.0184   | 0.0206   | 33,13    | 21                   |
| 1/3/13   | 0.0380   | <0.00240  | 0.00569 | 0.00531  | < 0.00240 | 0.00536  | 0.00639  | 0.0274   | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | 0.00717  | 23.43    | 372                  |
| 1/24/13  | 0.0348   | <0.00240  | 0.00948 | 0.00829  | <0.00240  | 0.00775  | 0.00764  | 0.0301   | <0.00660 | 0.0121   | 0.0123   | <0.00660 | 0.00864  | 0.00819  | 22.26    | 140                  |
| 2/7/13   | 0.0336   | <0.00240  | 0.00408 | 0.00363  | <0.00240  | 0.00400  | 0.00309  | 0.0818   | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | 32.11    | 456                  |
| 2/21/13  | 0.0323   | <0.00240  | 0.00459 | 0.00328  | <0.00240  | 0.00355  | 0.00332  | 0.0294   | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | 25,77    | 351                  |
| 3/7/13   | 0.0318   | <0.00240  | 0.00372 | 0.00262  | < 0.00240 | <0.00240 | <0.00240 | 0.0296   | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | <0.00660 | 29.62    | 756                  |
| 3/21/13  | 0.0321   | <0.00240  | 0.00332 | 0.00294  | <0.00240  | 0.00291  | 0.00267  | 0.0266   | <0.00660 | 0.00723  | <0.00660 | <0.00660 | <0.00660 | <0.00660 | 24.92    | 375                  |
| 4/4/13   | 0.0285   | <0.00240  | 0.00321 | 0.00249  | <0.00240  | <0.00240 | <0.00240 | 0.0317   | <0.00660 | 0.00836  | <0.00660 | <0.00660 | 0.00725  | 0.00698  | 29.55    | 659                  |
| 4/25/13  | 0.0196   | 0.00563   | 0.00563 | 0.00567  | 0.00332   | 0.00504  | 0.00562  | 0.0379   | 0.0288   | 0.0253   | 0.0269   | 0.0260   | 0.0225   | 0.0257   | 39.54    | 4410                 |
| 5/16/13  | 0.0208   | <0.00240  | 0.00290 | <0.00240 | <0.00240  | <0.00240 | 0.00298  | 0.0281   | <0.00660 | 0.00758  | 0.00673  | 0.0069   | <0.00660 | <0.00660 | 29.25    | 895                  |
| 5/23/13  | 0.0203   | <0.00240  | 0.00267 | 0.00255  | <0.00240  | <0.00240 | <0.00240 | 0.0210   | <0.00660 | 0.00710  | 0.00802  | <0.00660 | 0.00665  | 0.00830  | 29.36    | 781                  |
| 6/6/13   | 0.0201   | <0.00240  | 0.00243 | 0.00255  | < 0.00240 |          | 0.00259  | 0.0213   | <0.00660 | 0.0101   | 0.00850  | 0.00819  | 0.00720  | 0.0103   | 33.49    | 2440                 |
| 6/20/13  | 0.0229   | <0.00240  | 0.00253 | 0.00258  | <0.00240  |          | <0.00240 | 0,0220   | <0.00660 | <0.00660 | <0.00660 | <0,00660 | <0.00660 |          | 29.25    | 589                  |
| 7/11/13  | 0.0213   | 0.0154    | 0.00336 | 0.00242  | 0.00645   | 0.00302  |          | 0.0246   | 0.00833  |          | 0.00800  | 0.0326   | 0.0116   | 0.0174   | 43.56    | 1200                 |
| 7/18/13  | 0.0319   | <0.00240  | 0.00897 | 0.00781  | <0.00240  |          | 0.00672  | 0.0279   | <0.00660 | 0.0117   | 0.00921  | <0.00660 |          | 0.0105   | 29.53    | 98                   |
| 8/8/13   | 0.0390   | <0.00240  | 0.0284  | 0.0270   | <0.00240  |          | 0.0252   | 0.0467   | 0.00830  | 0.0306   | 0.0276   | <0.00660 |          | 0.0253   | 23.32    | 2.5                  |
| 8/22/13  | 0.0431   | <0.00240  | 0.0338  | 0.3420   | <0.00240  |          | 0.0322   | 0.0442   | 0.0109   | 0.0361   | 0.0360   | <0.00660 | 0.0327   | 0.0298   | 21.47    | 2.4                  |
| 9/5/13   | 0.0390   | <0.00240  | 0.0331  | 0.0345   | <0.00240  |          | 0.0341   | 0.0317   | 0.00891  | 0.0295   | 0.0302   | <0.00660 | 0.0284   | 0.0304   | 19.03    | 1.7                  |
| 9/19/13  | 0.0410   | <0.00240  | 0.0316  | 0.0316   | <0.00240  |          | 0.0312   | 0.0432   | <0.00660 | 0.0346   | 0.0343   | <0.00660 | 0.0321   | 0.0288   | 19.51    | 1,6                  |
| 10/3/13  | 0.0599   | <0.00240  | 0.0540  | 0.0545   | <0.00240  |          | 0.0544   | 0.0484   | 0.00759  | 0.0485   | 0.0490   | <0.00660 | 0.0459   | 0.0433   | 35.62    | 12                   |
| 10/24/13 | 0.161    | <0.00240  | 0.116   | 0.109    | <0.00240  |          | 0.0637   | 0.0333   | <0.00660 | 0.0282   | 0.0280   | <0.00660 | 0.0248   | 0.0266   | 20.38    | 0.28                 |
| 11/7/13  | 0.0742   | <0.00240  | 0.0549  | 0.0549   | <0.00240  |          | 0.0386   | 0.0477   | 0.0088   | 0.0363   | 0.0355   | <0.00660 | 0.0263   | 0.0232   | 21.99    | 0.76                 |
| 11/21/13 | 0.0426   | <0.00240  | 0.0365  | 0.0369   | <0,00240  | 0.0345   | 0.0358   | 0.0386   | <0.00660 | 0.0368   | 0.0370   | <0.00660 | 0.0338   | 0.0326   | 28.21    | 1.3                  |

|                         | ADIV   | Nickel and Zinc | , addita    |               |
|-------------------------|--|-----------------|-------------|---------------|
| Female                  | ADM Point A  | ADM Point A     | ADM Point D | ADM Point     |
| Sample                  | Nickel, Tot  | Zinc, Tot       | Nickel, Tot | Zinc, Tot     |
| Date                    | mg/L<br>0.0921   | mg/L            | mg/L        | mg/L<br>0.531 |
| 1/5/2012                | 0.0921   | 0.451           | 0.111       | 0.531         |
| 2/6/2012                | 0.121  | 0.424           | 0.109       | 0.488         |
| 2/13/2012               | 0.127  | 0.49            | 0.159       | 0.601         |
| 3/5/2012                | 0.128  | 0.431           | 0.15        | 0.493         |
| 3/12/2012               | 0.12   | 0.406           | 0.141       | 0.482         |
| 4/12/2012               | 0.169  | 0.621           | 0.191       | 0.705         |
| 4/19/2012               | 0.148  | 0.516           | 0.176       | 0.674         |
| 5/1/2012                | 0.0797   | 0.251           | 0.152       | 0.564         |
| 5/7/2012                | 0.137  | 0.494           | 0.141       | 0.448         |
| 6/4/2012                | 0.133  | 0.412           | 0.147       | 0.468         |
| 6/11/2012               | 0.12   | 0.366           | 0.144       | 0,452         |
| 7/2/2012                | 0.129  | 0.375           | 0.158       | 0.462         |
| 7/9/2012                | 0.109  | 0.322           | 0.132       | 0.402         |
| 8/1/2012                | 0.127  | 0.426           | 0.17        | 0.574         |
| 8/6/2012                | 0.097  | 0.193           | 0.12        | 0.242         |
| 9/6/2012                | 0.105  | 0.289           | 0.117       | 0.271         |
| 9/10/2012               | 0.479  | 0.531           | 0.165       | 0.559         |
| 10/1/2012               | 0.15   | 0.46            | 0.168       | 0.54          |
| 10/8/2012               | 0.129  | 0.421           | 0.152       | 0.444         |
| 11/1/2012               | 0.16   | 0.487           | 0.184       | 0.568         |
| 1/12/2012               | 0.158  | 0.444           | 0.197       | 0.525         |
| 12/3/2012               | 0.127  | 0.387           | 0.157       | 0.45          |
| 12/10/2012              | 0.106  | 0.218           | 0.123       | 0.25          |
| 1/7/2013                | 0.14   | 0.374           | 0.181       | 0.448         |
| 1/14/2013               | 0.103  | 0.229           | 0.121       | 0.263         |
| 2/4/2013                | 0.13   | 0.313           |             | 0.329         |
|                         | 0.116  | 0.285           | 0.147       | 0.308         |
| 3/2/2013                | 0.139  | 0.314           | 0.112       | 0.269         |
| 3/9/2013                | 0.122  | 0.283           | 0.103       | 0.289         |
| 3/11/2013               | 0.122  | 0.283           | 0.129       | 0.321         |
| 3/16/2013               | 0.134  | 0.355           | 0.156       | 0.431         |
| 3/20/2013               | 0.171  | 0.676           | 0.2         | 0.78          |
| 3/23/2013               | 0.171  | 0.578           | 0.191       | 0.686         |
| 3/27/2013               | 0.123  | 0.334           | 0.122       | 0.332         |
| 3/30/2013               | 0.122  | 0.356           | 0.127       | 0.371         |
| 4/3/2013                | 0.129  | 0.369           | 0.144       | 0.419         |
| 4/6/2013                | 0.118  | 0.266           | 0.102       | 0.16          |
| 4/8/2013                | 0.0832   | 0.151           | 0.0979      | 0.149         |
| 4/13/2013               | 0.107  | 0.279           | 0.118       | 0.303         |
| 4/15/2013               | 0.09   | 0.246           | 0.116       | 0.3           |
| 4/20/2013               | 0.101  | 0.307           | 0.0829      | 0.273         |
| 4/24/2013               | 0.116  | 0.343           | 0.0942      | 0.272         |
| 4/27/2013               | 0.117  | 0.342           | 0.116       | 0.31          |
| 5/1/2013                | 0.0809   | 0.162           | 0.0945      | 0.157         |
| 5/4/2013                | 0.107  | 0.411           | 0.123       | 0.45          |
| 5/6/2013                | 0.0947   | 0.266           | 0.103       | 0.281         |
| 5/11/2013               | 0.0744   | 0.0981          | 0.0741      | 0.0749        |
| 5/15/2013               | 0.0867   | 0.204           | 0.108       | 0.226         |
| 5/18/2013               | 0.0871   | 0.0921          | 0.0932      | 0.0848        |
| 5/22/2013               | 0.103  | 0.283           | 0.109       | 0.28          |
| 5/25/2013               | 0.127  | 0.439           | 0.155       | 0.513         |
| 5/29/2013               | 0.145  | 0.574           | 0.181       | 0.691         |
| 6/1/2013                | 0.0913   | 0.111           | 0.0702      | 0.0883        |
| 6/3/2013                | 0.0884   | 0.1             | 0.0919      | 0.111         |
| 6/10/2013               | 0.0962   | 0.205           | 0.108       | 0.228         |
| 7/1/2013                | 0.152  | 0.648           | 0.182       | 0.722         |
| 7/8/2013                | 0.141  | 0.512           | 0.157       | 0.553         |
| 8/1/2013                | 0.135  | 0.457           | 0.161       | 0.532         |
| 8/5/2013                | 0.133  | 0.451           | 0.163       | 0.544         |
| 9/4/2013                | 0.151  | 0.438           | 0.200       | 0.661         |
| 9/9/2013                | 0.114  | 0.456           | 0.184       | 0.670         |
| 10/1/2013               | 0.195  | 0.453           | 0.331       | 0.702         |
| 10/7/2013               | 0.154  | 0.340           | 0.217       | 0.453         |
| 10/25/2013              | 0.802  |                 | 1.09        |               |
| 10/26/2013              | 0.596  |                 | 0.905       |               |
| 10/27/2013              | 0.501  |                 | 0.729       |               |
| 10/28/2013              | 0.406  |                 | 0.557       |               |
| 10/29/2013              | 0.358  |                 | 0.560       | -             |
| A 144 - 14              | 0.335  |                 | 0.853       |               |
| 10/30/2013              |  |                 | 0.767       |               |
| 11/4/2013               | 0.486  | 0.403           | 0.767       | 0.522         |
| 11/4/2013<br>11/11/2013 | 0.152  | 0.114           | 0.191       | 0.113         |
| 11/4/2013               | The state of the s |                 | 1000        |               |