



# ST. CLOUD WASTEWATER TREATMENT FACILITY EMERGING CONTAMINANTS NUTRIENT HARVESTING PILOT PROJECT 2014 TECHNICAL REPORT, FINAL EMMA LARSON, CITY OF ST. CLOUD, MN

#### 1. ABSTRACT

The City of St. Cloud completed five simultaneous pilot projects to better understand the dewaterability and subsequent side stream nutrient recovery opportunities from anaerobically digested biosolids. Unanticipated nutrient paths and precipitation led to supplemental analysis and further investigation. Preliminary testing showed significant concentration of soluble phosphorous in the stored digested biosolids, suggesting nutrient recovery would be required to prevent predicted Biological Nutrient Removal upsets in the liquid stream treatment train upon return to the headworks. Pilot set up included significant pumping and aeration of the product leading to CO<sub>2</sub> release, rising of pH and precipitation of phosphorous in forms such as calcium phosphate and struvite. Crystalline particles were held in the solids product of dewatering and thus reducing concentrations in filtrate; filtrate represents the potential return stream or a potential feed to a nutrient harvesting technology. Full scale implementation of dewatering scenarios and future considerations were reviewed.

#### 2. INTRODUCTION

The St. Cloud Wastewater Treatment Facility (SCWWTF) receives ten million gallons a day (MGD) of wastewater from the cities of St. Cloud, St. Joseph, Sartell, Waite Park, Sauk Rapids and St. Augusta, and beneficially land applies 12 to 13 million gallons of Class B, liquid biosolids annually. The SCWWTF has full Biological Nutrient Removal (BNR) with UV Disinfection of the liquid stream, and anaerobic digestion of solids.

In 2010-13 the facility underwent a rehabilitation, upgrade and expansion of the liquid treatment, which increased rated capacity from 13 MGD to 17.9 MGD. In order to match this capacity on the solids treatment train, long-term planning options for the biosolids program, including mechanical dewatering of the anaerobically digested biosolids are being considered. The facility has five million gallons of biosolids storage which, on average stores biosolids for 155 days. This storage will be insufficient as populations continue to trend upwards. Additionally, the SCWWIF desires to improve energy efficiency by co-digesting High Strength Waste (HSW) to increase methane production; this program will be limited by the existing storage capacity. The water removed from the solids during dewatering was hypothesized to contain significant concentrations of dissolved phosphorus and ammonia, which is typical of anaerobically digested biosolids from a BNR facility. On this assumption, supernatant must be reprocessed prior to return to the headworks as phosphorus recycling from solids to liquids within a WWIF is thought to be one of the most common causes of failure to produce low effluent phosphorus in BNR Wastewater Treatment Facilities (WWIFs)<sup>1</sup>.

Over the past 10 years, effluent phosphorous at the SCWWTF has ranged between 1.0 mg/L and the lowest recorded annual average of 0.23 in 2013, Figure 1. Historically, the facility produced effluent with much higher effluent phosphorous concentrations and in 2002 a Phosphorous Management Plan was completed for which a Governor's Award was received in 2003; the City continues to be proud of its commitment to nutrient removal. Considering the potential impact a biosolids dewatering project could have on phosphorus removal efficiencies, high levels of recycled phosphorous is considered a likely contaminant of emerging concern, and is a potential road block to Biosolids Management expansion at the facility.



## St. Cloud Wastewater Treatment Facility Effluent Phosphorus



Figure 1. Historic Effluent Phosphorous Concentrations at St. Cloud WWTF.



Figure 2. St. Cloud WWITF Overview.



#### 3. METHOD DISCUSSION

#### 3.1 PILOT PROJECT AND REQUEST FOR PROPOSALS SUBMITTAL

In January 2014, the Minnesota Pollution Control Agency (MPCA) published a Request for Proposals (RFP) under the Clean Land and Legacy Act Fund, for the Emerging Contaminants Wastewater Initiative; WWTP Pilot Projects Proposal Grant. Advanced Engineering and Environmental Services (AE<sub>2</sub>S) approached the SCWWTF with the suggestion of submitting a joint proposal. A successful proposal was submitted on February 26, 2014 and a grant contract was signed on July 11. The proposed pilot study would test an innovative struvite (magnesium ammonium phosphate) harvesting process from biosolids dewatering supernatant. This was considered a viable control method for the return of potential excess phosphorous to the head of the liquid stream of the facility.

The grant was awarded at a cost share of 61% applicant to 39% Clean Land and Legacy Act, with significant in-kind hours from both the City of St. Cloud and AE<sub>2</sub>S.

#### 3.2 DEWATERING

St Cloud WWTF currently co-thickens raw and waste solids on a gravity belt table (GBT) to approximately nine percent total solids (TS) prior to anaerobic digestion. Anaerobic digestion provides significant volatile solid destruction, and the TS is typically 3-4%. No thickening, dewatering or drying takes place after digestion. Digested biosolids are transferred from the digester complex to underground storage located to the northwest of the facility (Figure 2) using centrifugal pumps. Class B liquid biosolids are transferred between holding cells and the day tank, which ultimately feeds to two load out bays in the biosolids building using positive displacement screw pumps to load semi-trailers which deliver the 3 to 4% TS product for land application.

Mechanical dewatering of the anaerobically digested solids will most likely be required in the near future to relieve storage constraints; this was an excellent opportunity to 'preview' potential options. In recent years, various dewatering technologies have been discussed and considered by various WWTF team members and through discussions at the time of RFP submittal, screw press technology was significantly favored. Uses for screw-presses range from pharmaceutical manufacturing, food production, paper production and wastewater treatment and re-use.



Figure 3. Solids handling at St. Cloud WWTF



In order to produce sufficient volume of supernatant to fulfil the requirements of the nutrient harvesting pilot and to meet timing constraints, two dewatering pilots were required. The nutrient recovery vendors helped coordinate the screw-press manufacturers and cooperation between vendors and manufacturers enabled the sequential dewatering pilots. The two screw press manufacturers selected for this project both work on the concept of the compression of flocculated slurry using a slow, rotating screw into a perforated screen. Two 21,000 gallon holding tanks were rented, initially for storage of biosolids pre-dewatering and filtrate storage. As the project progressed, operational changes were made due to stagnation and pH changes in the stored biosolids product and both tanks were subsequently used for storage of filtrate.

#### 3.3 NUTRIENT HARVESTING

Two main vendors were considered for nutrient harvesting equipment: Ostara Pearl Nutrient Recovery and Multiform Harvest. After careful consideration, Ostara was selected for piloting due to the past performances and experience in the USA, documented pilot project success and operational and maintenance considerations.

Ostara's Pearl process is based on the concept of controlling the precipitation of struvite (magnesium ammonium phosphate), a crystal salt, in a fluidized bed reactor. Formation of struvite is a naturally occurring chemical reaction that occurs when the three components are present in a solution above a saturation point. The Ostara process is based on the addition of nutrients that are limiting (where required) to encourage struvite formation, such that the all three components are removed from solution. The struvite saturation point of a solution is strongly influenced by pH, Figure 11; sodium hydroxide and magnesium chloride dosing to the feed stream is used to optimize the precipitation.

#### 3.4 PROPOSED ANALYSIS

Requirements set forth in the grant contract with the MPCA as well as desired knowledge gained by WWTF team members determined the locations and parameters that would be sampled. Figure 4 details the location and parameters that were selected prior to the project. Sampling locations are identified on Figure 5.

LOCATION # AND NAME	ANALYSIS AND MINIMUM FREQUENCY
1. PRE-DEWATERED PRODUCT (BIOSOLIDS)	TS, TP, TKN (x1 on batch), NH3 (x1 on batch)
2. DEWATERED CAKE BIOSOLIDS	TS, TP, TKN, NH3
3. Dewatered supernatant (pre-harvest Liquid)	TSS, TP, TKN (x1 on batch), Soluble P, NH3 (x1 on batch), Alkalinity
<ol> <li>POST-HARVEST SUPERNATANT (RETURN FEED)</li> </ol>	TSS, TP, TKN (x1 on batch), Soluble P, NH3 (x1 on batch), Alkalinity
5. RECOVERED PRODUCT (PEARL)	Ostara to complete most of sampling
TS: Total Solids as percentage TP: Total Phosphorous, mg/L TKN: Total Kjeldehl Nitrogen, mg/L	NH3: Ammonia Nitrogen, mg/L Soluble P: PO4-P (soluble P)

Figure 4. Sampling Location and parameters as determined prior to project start date.

When this table was developed, an assumption was made that a 'batch' system of transferring biosolids product for dewatering would be possible using the rented holding tanks. This, however, proved to be unattainable and a much smaller batching system was developed using the semi-trailers. This will be addressed and discussed in subsequent sections.





Figure 5. Sampling locations as determined in planning stages of project.

## 4. RESULTS AND DISCUSSIONS

#### 4.1 DEWATERING PILOT #1.

Between August 25 and September 4, 2014, an Ishagaki, ISGH 040 screw press was piloted; a progressive cavity sludge pump, with polymer injection and a blending chamber, as well as a biosolids – polymer mixing chamber for flocculation. This unit experienced significant start up issues on the first days of the trial; preliminary filtrate was produced on the afternoon of August 26<sup>th</sup>.

Ishagaki reached a maximum cake total solids of 29%. Cake solids were variable dependent on specific polymer, of which there were three piloted, however the solids capture rate remained relatively constant between 95 and 98% over all polymer variations and dosing.

Ishagaki experienced significant operational issues including blown fuses and motors, polymer clogging, modification of polymer mixing chamber propellers as well as the failing of the polymer feed control panel. The use of European equipment and parts lead to significant lead times for required replacements for which alternatives had to be sourced. An alternative control panel was sourced and arrived onsite the following day. Wastewater Treatment Facility staff spent significantly more time on the set up of the Ishagaki Pilot than expected and repaired all of the electrical components as well as acquiring all the required hoses and pumps.

Initially, biosolids product was fed from a semi-trailer that had been filled with pre-tested product from the day tank through the load out bays in the biosolids building. The intention was to fill the 'first' holding tank with biosolids once dewatering was taking place, and treating this as a batch, (Figure 5, sampling location 1). Initial monitoring and sampling indicated a decrease in soluble phosphate concentration in the biosolids across phases from the digesters to the storage in the semi-trailer. Investigation by the pilot team led to the belief that premature precipitation of phosphorous was occurring. This was confirmed using significant sampling of pH, total and soluble-phosphorous at digester discharge, cell storage and semi-trailer storage, as well as analysis of the cake product and filtrate produced from the Ishagaki screw-press. Additional monitoring to enable mass balance of magnesium and ammonia confirmed the hypothesis that struvite precipitation was occurring before dewatering. The resulting amendments of this discovery are discussed in section *4.4.2. Premature Precipitation of Struvite: WASSTRIP Bonus Pilot*.

Ishagaki Pilot goals included demonstration of their equipment to produce the highest total solids cake possible, however the goals of the WWTF team also focused on the quality of the filtrate produced in order to feed the nutrient harvesting equipment. See Appendix 1 for Ishagaki Report.



#### 4.2 DEWATERING PILOT #2.

Schwing Bioset FSP600 Screw-Press arrived onsite on September 8<sup>th</sup> and produced cake and filtrate through September 12<sup>th</sup>. Schwing's pilot unit was significantly larger in size compared to Ishagaki and set up time was significantly less. The pilot consisted of a progressive cavity feed pump, a polymer system, a floc mixing tank, a screw-press dewatering unit and control panels. The trailer unit arrived, was situated and was producing cake and supernatant within 6 hours.

Schwing trialed six polymers with variable success. This pilot demonstrated flocculation over a wide range of dosing and in return also returned a larger range of cake total solids. This larger unit also allowed for greater output of cake product and filtrate enabling sufficient feed product (filtrate) to be produced for the remainder of the nutrient harvesting pilot. See Appendix 2 for Schwing Report.

#### 4.3 DEWATERING PILOT COMPARISONS AND DISCUSSION

Only one polymer was used by both screw press pilots which limited direct comparisons between the manufacturers. Figure 6 shows the basic information from each screw press across all polymers tested. A factor that was not initially considered when reviewing filtrate results was the volume of polymer blend water used and the contribution this made to the final filtrate quality due to dilution. Under Average Polymer Blend: Feed Ratio, at any given time, the polymer and its dilution water accounted for 30% / 20%, therefore skewing the overall performance, relating to filtrate quality. Filtrate quality ranged significantly across both screw press manufacturers (Figure 6). This was significant for the nutrient harvesting pilot as phosphorous concentrations in the filtrate were diluted with the polymer dilution water.

It is recognized that the screw press, and dewatering pilots in general, are primarily concerned with demonstrating the performance of their equipment with regards to producing the highest percent solids possible and optimizing polymer use. Filtrate quality is not a primary concern of the dewatering manufacturer demonstrations, however in this pilot project the St. Cloud team was monitoring filtrate quality closely to ensure a sufficient quality of feed for the nutrient harvesting equipment.

	AV. Biosolids Feed GPH	av. Polymer Dilution Rate	AV. POLYMER BLEND: FEED RATIO	MIN. POLYMER BLEND: FEED	MAX. POLYMER BLEND: FEED	AV.LBS ACTIVE POLYMER : DT CAKE	AV. FILTRATE TSS MG/L	MIN. Filtrate MG/L	MAX FILTRATE TSS MG/L
ISHAGAKI	258	0.5	30%				1,559	420	7,760
SCHWING	1,553	0.75%	20%	14%	24%	37	161	60	255
Figure 6. Comparison of screw press performance.									

#### 4.4 NUTRIENT HARVESTING

#### 4.4.1 OSTARA PEARL REACTOR PILOT #3

Ostara Peal Pilot equipment was comprised of a reactor with a fluidized bed of seed struvite, a caustic feed system, a magnesium chloride supplemental feed system, pumping systems and a control panel. Feed of screw-press filtrate was managed by WWTF pumps and hoses (see Section 6, Lessons Learnt), which fed from a discharge port of the '1<sup>st</sup>' holding tank (Figure 7). Ostara 'pearl' production technology is based on controlling the naturally occurring precipitation in a fluidized reactor that recovers struvite in the form of highly pure crystalline pellets or 'pearls'. Dewatering filtrate feed streams were supplemented with magnesium chloride to achieve applicable ratios of magnesium, ammonia and phosphate, and sodium hydroxide for pH augmentation before being fed into the Pearl reactor where minute particles or struvite "seeds" begin to form. It was proposed that up to 90% phosphorous recovery would be achieved at St. Cloud WWTF. See Appendix 3 for Ostara Pearl Report.









Figure 7. Ostara reactor set up at St. Cloud WWTF in Biosolids handling building.

#### 4.4.2 PREMATURE PRECIPITATION OF STRUVITE: WASSTRIP BONUS PILOT, PILOT #4

Crystalline particles observed in the cake product immediately upon production by Ishagaki were suspected to be immature struvite crystals; the significant reduction in soluble phosphate in the biosolids feed to the screw-press and the subsequent filtrate produced by the press clarified this early precipitation. Note: both screw-presses experienced the same apparent loss of soluble-phosphate (Figure 7, 9 & 10).

	DIGESTER	BIOSOLIDS STORAGE	BIOSOLIDS HOLDING	FILTRATE
MAGNESIUM (MG/L)	90	50	50	20
AMMONIUM (MG/L)	2500	2490	2460	2300
PHOSPHORUS (MG/L)	250	180	160	25
PH	7.6	7.8	7.9	8.1





Figure 9. Precipitation of nutrients as pH increases across SCWWTF treatment during Pilot Projects.

Figure 10. Precipitation of Calcium and Magnesium across process.



Figure 11. Donohue 2014. pH influent on Struvite precipitation potential, St. Cloud pH and nutrient concentration values plotted.

Carbon dioxide (CO<sub>2</sub>) off-gassing due to aeration of the biosolids by pumping and discharging throughout the transfer from digesters – trucks – screw-press, resulted in a rise of pH, and was considered the likely reason for premature precipitation. Struvite precipitation correlation to saturation levels for struvite decreases with increased pH (Figure 12). This was confirmed by the decrease in molecular magnesium and calcium across the processes (Figure 10). Various options for reducing this premature precipitation were considered including but not limited to, (1) more direct methods of feeding the biosolids from the digester complex to the screw-press, (2) chemically maintaining the pH of the biosolids in the semi-trailer and (3) creating a CO<sub>2</sub> blanket in the trailer to reduce the off gassing. Planning then moved to considering the artificial supplement of the filtrate with a higher soluble-phosphate side stream to feed to the nutrient harvesting equipment; potentially by maintaining an increased blanket depth on the final clarifiers with mechanical decant or upscaling of the WASSTRIP from bench scale to pilot scale. It was understood that this supplementation may not demonstrate full scale processes, however it would enable successful piloting of the nutrient harvesting equipment for the purpose of this project and potentially provide valuable full scale design information. Note that it is assumed that these



issues were mostly related to the pilot set up, and the CO2 off-gassing issue could be more effectively mitigated during a full scale design.

WASSTRIP is Ostara's version of the process of concentrating phosphorous, magnesium and potassium prior to waste activated sludge (WAS) thickening. Team discussions lead to the decision that this was the most viable and logical solution to ensure success of the nutrient harvesting pilot. An un-used 5000 gallon grit tank located at the rear of the GBT Thickening facility, as seen in Figure 3, was filled with WAS through re-fitted pipes and pumps (Figure 12). Initial plans were to ferment the grit tank of WAS for 24 to 36 hours; samples were taken and at 24 hours no significant release of phosphorous was observed in the collected samples. It was hypothesized that sludge had settled to the bottom of the grit tank and was holding the phosphorous; the tank was mixed and sampling continued and the 'test' extended.



Figure 12. Reconfiguration on basement piping enabled enclosed transfer of waste activated sludge (WAS) from final clarification tanks to the un-used grit tanks to fermentation.

Mixing at hour 27 caused an increase of soluble phosphorous of 33%, with another significant release after mixing at hour 46, 38% increase (Figure 13). A determination was made that optimum release had been achieved and this phosphorous rich WAS product was transferred to a semi-trailer as a supplemental fed to the nutrient harvesting pilot. The remaining sludge blanket at the bottom of the grit tank was rinsed and returned to the headworks of the facility, the process was repeated to continue production of this product to enable continued piloting of the Pearl reactor. Subsequent batches of WAS product were optimized by decanting and refilling at approximately hours 20 – 24 to give a greater concentration of released phosphorous. During this process the highest recorded soluble-phosphate sampled in the grit tank was 210 mg/L, which is significantly higher than the 30 mg/L that was found in the screw-press filtrate.



Figure 13: WAS phosphorous release during phase 1 WASSTRIP pilot.



Figure 14: Transfer of WAS product to a semi-trailer.



#### 4.4.3 WASSTRIP: FILTRATE RATIO FEED RATE

In order to maintain a suitable ratio of P: Mg: N to create a fertilizer product, WASSTRIP decent was fed at 3:1 with screw-press filtrate. The screw-press filtrate produced sufficient concentrations of ammonia, WASSTRIP decant produced soluble phosphorous and a supplement of magnesium chloride added the magnesium component.

#### 4.4.4 NUTRIENT HARVESTING RESULTS AND DISCUSSION

Ostara nutrient harvesting averaged a removal rate of 76%, with a maximum removal of 87%. However the relevance of these results are under discussion as to the augmentation of the feed that was required in order to achieve them.

The unexpected precipitation of struvite prior to and during dewatering opened new discussions on the potential options for the future of Biosolids Management at St. Cloud WWTF. If at full scale dewatering implementation, precipitation occurs earlier than expected it could lead to significant operation and maintenance costs as associated with the removal of struvite from digesters, pumps and pipes. The rise in pH across the process stages (Figure 9) shows that the conditions are optimal for struvite formation (Figure 12). Treatment options to remove or reduce the concentration of one or more of the required components of struvite (P, Mg or N) may reduce the likelihood of precipitation occurring, however the potential precipitation of other crystalline salts such as calcium phosphate can also cause operational and maintenance issues at WWTFs.

This earlier than expected precipitation could remove the potential influence of recycled excess phosphorous causing elevated effluent phosphorous at St. Cloud WWTF, however there is no guarantee that the experience at the pilot scale would be recreated at full scale execution. Note; a WASSTRIP or similar process would remove significant amounts of phosphorus prior to digestion, which would reduce the struvite precipitation potential during digestion and storage.

Currently, the application rates for land application of Class B biosolids are governed by nitrogen loading rates, with a supplemental phosphorus management plan requirement for sites with Bray 1 Phosphorous of greater than 200 parts per million. Application rates are governed by rules sets forth in Minnesota Rules Chapter 7041. The precipitated phosphorous being retained in the dewatered solids would not prove problematic as long as application rates continue to be based on nitrogen concentrations of biosolids product.

#### 4.5 BIOSET LIME STABILIZATION TO CLASS A PRODUCT: THE PLANNED BONUS PILOT, PILOT #6

Bioset is an alkaline stabilization of cake biosolids to produce Class A cake using quicklime and sulfamic acid. Schwing provided the pilot scale reactor which worked in line with the screw-press. Ammonia released during an exothermic reaction inactivates the pathogens within the product prior to the pH or temperature doing so, which led the Environmental Protection Agency (EPA 2011, Appendix 5) to approve a lower operational temperature of Bioset verses other lime stabilization processes to create Class A standard biosolids. At the time of publication, the Minnesota Pollution Control Agency (MPCA), the control authority over the St. Cloud Biosolids Management Program has yet to approve this process. The equipment can be operated with cake of between 14 and 30% total solids, therefore well within range of the projected dewatered biosolids at St. Cloud. Test results for Fecal Coliform on the Bioset product produced during the St. Cloud Pilot were <91 Most Probable Number (MPN) per gram. The MPCA Class A requirements include testing for indicator organisms of fecal coliform and salmonella sp.bacteria. Results for salmonella were not supplied by Schwing Bioset; fecal coliform were significantly under the 1000 MPN/g limit.



#### 5. CONCLUSIONS

Objectives derived prior to the project for the grant application focused on the two main processes that would be piloted throughout the project, with little consideration given to potential for other reactions and processes taking place, such as precipitation of struvite into small crystalline particles in the caked biosolids.

#### **Objective 1: Determine Biosolids Dewaterability**

St. Cloud Biosolids were dewatered to a considerably higher than expected total solids concentration, with a reasonable polymer dosing rate using screw-press technology. This can be hypothesized to be transferable to other dewatering technologies and at other facilities.

#### **Objective 2: Determine Feasibility of Struvite Harvesting**

St. Cloud Biosolids dewatering filtrate proved to have a significantly lower than expected solublephosphorous, inhibiting the ability for nutrient removal and potentially removing the requirement for it. The process was augmented with WASSTRIP decant as described above in order to sufficiently pilot the Ostara Pearl reactor equipment. The question that remains is at full scale implementation would dewatering filtrate have significant enough phosphorous concentrations to upset the BNR process and ultimately affect the removal efficiency of the WWTF liquid stream? It was shown that significant soluble phosphorus reduction (up to 87%) was possible with this struvite harvesting technology.

#### **Objective 3: Document Results and Transfer Data**

The Nutrient Harvesting Pilot Team has successfully completed analysis and presentation of results in required quarterly reports and this final technical report, as well as submittal to present pilot experiences to fellow wastewater professionals.

*Summary Objective:* The Request for Proposal application identified the success of the project as the successful return of nutrient reduced supernatant to the head of the WWTF such that the quality of the liquid effluent discharge to the receiving waters would not be compromised.

Using the soluble-phosphate results from Biosolids dewatering filtrate alone, even after consideration of the high volume of polymer dilution water, the concern for the quality of liquid stream effluent could be minimal, however modelling completed by Donohue for the purposes of an separate project, shows that the assumption of soluble phosphate precipitating prior to dewatering, and thus remaining with the solids in the cake product, may be unrepresentative of full scale operation. In addition to modelling, the WWTF completed supplemental sampling that follows soluble phosphorous and ammonia across treatment (Figures 16 & 17). These sampling events demonstrated that high levels of soluble phosphorous do exist after digestion, and under full scale implementation of dewatering after digestion, may, as originally hypothesized, create a filtrate from dewatering with an elevated soluble phosphorous concentration. Modelling and supplemental sampling are not sufficient to determine the fate of nutrients from liquid biosolids, dewatering and return to headworks, however this data, along with the Struvite Precipitation Potential graph (Figure 11) would suggest that nutrients are present in the applicable concentrations, forms and at a pH that would enable and even promote the precipitation of struvite. If un-controlled, this would cause significant operation and maintenance issues. Return stream phosphorous that is not consumed in controlled or uncontrolled precipitation may still result in elevated effluent total phosphorous from the WWTF thus exceeding the goal of 0.3 mg/L or even exceeding the regulated discharge limit of 1.0 mg/L.

	CURRENT OPERATIONS	DEWATERING OPERATIONS – STRUVITE PRECIPITATION NOT SIMULATED	DEWATERING OPERATIONS - STRUVITE PRECIPITATION SIMULATED
MLSS	2,870	2,874	2,874
EFFLUENT AMMONIA N [mg/L]	0.5	0.5	0.5
EFFLUENT NITRATE N [mg N/L]	9	11.5	11.5
EFFLUENT TOTAL N [mg N/L]	12	14.5	14.5
EFFLUENT SOLUBLE PO4-P [mg P/L]	0.4	1.9	1.8
EFFLUENT TOTAL P [mg P/L]	0.6	2.1	2.1

Figure 16. Modelled effluent conditions based on current conditions at St. Cloud WWTF and pilot data. (Biowin modelling completed by Donohue and Associates, 2015.)





with four separate sampling events across the facility.

The City of St. Cloud continues to work towards the completion of a Master Plan for Resource Recovery and Energy Efficiency (R2E2), with the data and analysis from this pilot project being a vital key to decisions to be made with regards to the future of the program.

#### 6. LESSONS LEARNT

Simultaneous piloting and projects leads to significant staff resources being expended and strained. More extensive planning, where possible would potentially reduce this strain on resources. Review of previously completed, similar piloting/projects may enable further establishment of sampling requirements and desired monitoring prior to project initiation.

Support from leadership and Council is vital to enable piloting to be successful and for trials to be completed.

Significant logistical obstacles arise when coordinating multiple pilots at one location, simultaneously. One such obstacle was the transfer of filtrate from the end point of the dewatering pilot into a storage location (holding tank) to enable feed to the nutrient harvesting portion of the project. Innovative thinking by WWIF team members lead to the storage tote wet-well and sump-pump set up. See Figure 18.





Figure 18. Recycled tote cut in half was used as a wet well and a sump pump and flat hose were used to transfer from end of screw-press to holding tanks.

#### 7. OPPORTUNITIES FOR FURTHER EVALUATION

- Resource Recovery and Energy Efficiency (R2E2) Master Plan has been established to evaluate the opportunities at the St. Cloud WWTF, due for completion in Spring 2015.
- Evaluation of polymer dilution and wash water dilution of filtrate and how this would affect full scale implementation filtrate.
- Other nutrient harvesting options are available to the St. Cloud WWTF such as Multiform Harvest which has installation at a similar location as Ostara, and Air Prex which removes struvite particles prior to digestion.
- Consideration of other technologies that are available for further treatment of biosolids products and subsequent side stream. Winter 2014/15, Lystek Biosolids and Organic Processing trialed at bench scale using St. Cloud Biosolids product and thickened sludge.
- Consideration of a stepped approach using various options.

#### 8. APPENDIX

- 1. Ishagaki pilot Technical report
- 2. Schwing Bioset Pilot technical Report
- 3. Ostara Pearl Pilot Technical Report
- 4. City of St. Cloud Laboratory Data

#### 9. REFERENCES

- *"Nitrogen and Phosphorous Rich Side streams: Managing the Nutrient Merry-go-Round"*; Heather M. Phillips, P.E et al. 2006.
- 2 AE2S: Advanced Environmental Engineering Solutions, Contracted Engineer; Scott Schaefer, P.E.
- 3 Donohue and Associates, Contracted Engineers; Mike Gerbitz, P.E., Leon Downing, Ph.D., P.E.,

# Appendix 1 Ishagaki Pilot Report



# Pilot Test St. Cloud, MN WWTP

Pilot Test from:	08/25/14 - 9/4/14	
Test Attendants:	Yoshiharu Watanabe Mark Eklund	Ishigaki USA Ishigaki USA
Press Model:	ISGK 040	
Project:	St. Cloud, MN WWTP	

# Pilot Unit ISGK 040

The dewatering machine is a screw press with a conical screw shaft and cylindrical perforated drum screen. The machine is subdivided into three zones, gravity drainage, thickening, and compression with pneumatic backpressure cone. The pilot unit is mounted on a skid that contains all necessary matters to run the dewatering machine.

This includes:

- Screw press Model ISGK 040
- NETZCH progressive cavity sludge pump
- Polymer blending station
- Flow meter for sludge and polymer
- Injection and mixing devices for polymer
- Control Panel

The most important parameters are:

- Desired flow rate of sludge and polymer in [GPM]
- Dry solids (DS) of sludge IN and OUT in [%]
- Polymer consumption in [lbs active poly / ton DS]
- Speed of screw press in [%]

208 Heritage Drive, Unit J Portsmouth NH 03801 (603) 433-3334



Plant Address:

City of St. Cloud, MN WWTP 525 60<sup>th</sup> Street South St. Cloud, MN 56301

Contact: Chris Plautz, Wastewater Services Supervisor chris.plautz@ci.stcloud.mn.us

Phone: 320-650-2955

# Current Waste Water Treatment Plant Operations

Daily flow (design)	Unknown
System:	Bar screen, grit removal, primary sedimentation, aeration, secondary sedimentation, gravity thickening, disinfection
Sludge:	Anaerobically Digested Sludge
Sludge age:	Unknown
Waste sludge flow:	32,000 GPD
Solid content:	4.0 – 4.5% DS
Volatile solids:	60 - 64%
Existing solids handling system:	Anaerobic digestion, sludge storage prior to land spreading
Future improvements:	New dewatering system





#### Introduction

The Ishigaki screw press was tested on St. Cloud's anaerobically digested sludge from 8/25/14 to 9/4/14. Testing was performed at different machine settings on the dry cake solids, dry cake solids capacity, polymer consumption, and filtrate quality to determine the screw press's performance. Three different polymers were used.

In addition to testing, the filtrate was saved for additional testing by Ostara, a company who has technology to recover nutrients from the liquid.

#### Methods

Before starting the trials, several Solenis (formerly Ashland Chemicals) polymers were jar tested to determine their effectiveness in coagulating the raw sludge. Two were selected for testing, K 144 L and K 111 L. In addition, a polymer supplied to the plant by SNF Polydyne, Clarifloc 6272, was also used in the testing.

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The operation of the screw press was optimized using the three different polymers and changing the parameters of sludge feed rate, polymer feed rate, and screw speed. The press was operated with the goal of maximizing cake solids dryness and solids capture efficiency, over a wide range of polymer dosing rates.

## Conditions

The solids concentration of the raw sludge varied from 4.3 to 4.5% during the course of the testing. The volatile solids fraction in the raw sludge varied from 60% to 64%.

## Results (K 144 L Polymer)

For tests #'s 3 - 9, the K 144 L polymer was used. Cake solids were good at 23% - 29%, and filtrate quality was good with 96% to 98% solids capture. Polymer consumption started out at 30 pounds of active polymer per dry ton of solids dewatered. The higher the dose rate, the higher the cake solids were. The lower the dose rate, the lower the cake solids were.



## **Results (Clarifloc 6282 Polymer)**

For tests #'s 1, 2, 10, and 11, SNF Polydyne's Clarifloc 6282 polymer was used. Cake solids were good at 23% - 29%, and filtrate quality was good with 95% - 98%. Polymer consumption started out at 43 pounds of active polymer per dry ton of solids dewatered. The higher the dose rate, the higher the cake solids were. The lower the dose rate, the lower the cake solids were.





## Results (K 111 L Polymer)

For tests #'s 12 – 19, the K 111 L polymer was used. Cake solids were good at 20% - 25%, and filtrate quality varied from 82% to 98% solids capture efficiency. Polymer consumption started out at 37 pounds of active polymer per dry ton of solids dewatered. The dewatered cake solids did not vary much over the range of polymer dosing rates.



208 Heritage Drive, Unit J Portsmouth NH 03801 (603) 433-3334



## **Results (Dry Solids Capacity)**

The dry solids capacity with the use of both the K 144 L, and Clarifloc 6282 polymer was essentially the same. The K 111 L polymer had the lowest capacity at any given speed.



## **Test Pictures**









208 Heritage Drive, Unit J Portsmouth NH 03801 (603) 433-3334





#### **General Observations/Conclusions**

It was noted that polymer consumption had a dramatic effect on the dryness of the cake solids. A limit was reached, however, as the maximum cake solids achieved was 29%. Increasing the polymer usage did not increase cake solids dryness above 29%.

The solids capture efficiency, however, remained flat at 95% to 98% over the wide range of polymer dosage.

Due to the wide range of polymer consumption verses the wide range of cake solids dryness, it will need to be determined how a full scale installation is to be operated to best benefit plant operations.

All three polymers performed well to varying degrees. But the K 144 L polymer provided the best balance between cake solids, solids capture efficiency, and polymer consumption. The Clarifloc 6282 polymer achieved the same results on cake solids and filtrate, as the K 144 L polymer, but at a higher polymer dose rate.



All three polymers will work for this application. But the K 144 L polymer delivers the best performance with the lowest polymer dose rate.

Visual observation indicates that the press should be washed once or twice per day using the automatic spray bar.

Ishigaki USA would like to thank you for the opportunity to present our screw press Model ISGK 040 at St. Cloud, MN. We appreciate all the help provided by the plant personnel at St. Cloud, MN to make this pilot possible and a successful one.

# Appendix 2 Bioset and Schwing Pilot Report







# **REPORT PRESENTED BY:**

SCHWING BIOSET, INC. 350 SMC DRIVE, SOMERSET, WI 54025 PHONE: (715) 247-3433 FAX: (715) 247-3438

# **CONTACT:**

# SCOTT KELLY, MIDWEST REGIONAL SALES MANAGER PH: 720-326-4366 E-MAIL: SKELLY@SCHWINGBIOSET.COM

# ST. CLOUD, MN WWTF 535 60<sup>TH</sup> STREET SOUTH, ST. CLOUD, MN 56301

Schwing Bioset Screw Press and Bioset Lime Stabilization Pilot September 8<sup>th</sup> – 12<sup>th</sup>, 2014

Dewatering Screw Press System



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- Tab 1: Facility & Material Summary
- Tab 2: Schwing Bioset Test Equipment
- Tab 3:Pilot Test Results
- Tab 4: Conclusion
- Tab 5: Class A Biosolids EPA Standards and Lab Report







Schwing Screw Press and Bioset Set-up at St. Cloud, MN



# PLANT AND MATERIAL SUMMARY

Plant Flow: Sludge Type: Primary/Secondary Solids Content: Existing Dewatering Equipment: 13.0 MGD
Anaerobic Waste Activated
27%/73%
3.5 - 4.0%
Belt Thickeners then land application

The St. Cloud WWTF produces an anaerobic waste activated sludge that is currently being thickened by Gravity Belt Thickeners to 3.5% - 4.0% solids then land plied to on local farmland. The purpose for the Schwing Screw Press and Bioset Class A demo is to show how Schwing Bioset's Screw Press technology performs on the sludge on the St. Cloud thickened sludge and support the selection of Schwing Bioset's screw press dewatering technology for future dewatering plans. The dewatered material was then treated with quicklime in the Schwing Bioset process to produce Class A Biosolids for future crop application consideration.

The Plant's lab collected periodic samples to test cake solids, feed solids and filtrate quality from the Screw Press.

The sections below describe the equipment used and it's configuration for the pilot at St. Cloud followed by a summary of the dewatering results seen during the pilot.



# **TEST EQUIPMENT SUMMARY**

### **SLUDGE DEWATERING SCREW PRESS**

#### **Design Conditions:**

Schwing Bioset dewatering system sized for: Design dewatering rate (225 dry lbs/hr.) at 1.0% Aerobic Feed solids @ Maximum 45 GPM

Model:	FSP600
Quantity:	One (1)
Press Length:	15 feet 8.5 inches
Press Width:	3 feet 9.9 inches
Press Height:	4 feet 11.6 inches
Design Capacity (2.5% solids):	360 dry lb./hr.
Press HP:	3 HP
Weight est.:	6,330 lbs

#### System includes:

- 1. The SBI Screw Press system is designed for continuous dewatering of flocculated slurry. The system consists of; a progressive cavity feed pump, polymer system, Screw Press dewatering unit, and controls.
- 2. The Screw Press dewatering unit compresses and dewaters flocculated slurry using a screw rotating at very slow speed in a perforated screen. Observation windows and flaps allow a direct view of the dewatering process. The simple operating principle is achieved with only a few functional component groups. Slow movement and the high quality design of the structural components guarantee a high service life.
- 3. Press dewatering system is powered using a Premium efficient, TEFC motor. Variation of the Screw speed is achieved with a VFD.
- 4. The system will continuously discharge cake from the press into the conveyor. The filtrate will discharge from a drip tray below the perforated screen into a discharge pipe.
- 5. For general housekeeping purposes the back washing cycle cleans the screens intermittently (generally less than 5 minutes/day). Dewatering operations are not suspended during washing cycle.
- 6. Air Compressor for discharge pressure cone actuator and movement of wash ring.





# FSP600 Screw Press Setup

## POLYMER FEED SYSTEM

Quantity:	One (1)
Model	Velodyne VM-5P-1200-E
Dilution water flow	2-20 gpm
Neat polymer flow	0.25 - 5 gph

# System includes:

1. Effective mixing of the water and polymer is achieved in Polymer Feed System. The water polymer blend is manually controlled using a hand valve. In permanent installations the blend control is automated. The feed of the water polymer blend into the Sludge Flocculation Tank is by VFD controlled PC pump. The control panel automatically monitors the flow rate and the pressure of the Flocculation tank.





Polymer System Screw Press Pilot

## SLUDGE FLOCCULATION TANK

Quantity:	One (1)
Tank Volume	224.5 gal (850 L)
Tank Depth	48 inches
Tank Width	30 inches
Tank Overall Height	60 inches
Tank Weight	485 lbs

#### System includes:

- 1. Effective flocculation is achieved in the Sludge Flocculation Tank. It is a closed design with observation windows for viewing of the current flocculation quality. Special designed paddle and flow breakers for effective mixing and gentle transport of the flocks. Diluted/activated polymer from the Polymer Mixing System enters sludge pipe at tee upstream of Sludge Flocculation Tank.
- 2. A pressure sensor is included in the top of the tank. The pressure sensor sends a signal back to the control panel to automate the system and prevent over pressurization of the tank.





Floc Mixing Tank Setup Schwing Bioset Pilot

## SCREW PRESS CONTROL PANEL

Quantity:	One (1)
Power Supply	480V/3Ø/60Hz

## Scope includes:

- 1. The PLC Panel is NEMA4X, with Allen Bradley PLC and Panelview HMI touchscreen controls the system. PLC includes operating program made in RSLogix5000; The PLC Panel shall be used to directly control and power Screw Press and all the feed equipment including:
  - One (1) Sludge Feed Pump
  - One (1) Dewatering Polymer Mixing System (via Polymer Mixing System Control Panel).
  - One (1) Sludge Flocculation Tank





Control Panel

# MOISTURE ANALYZER

Quantity:	One (1)
Model	Ohaus MB45



Moisture Analyzer



### SCHWING BIOSET PROCESS – Mobile Unit MB12

Design Conditions:								
Schwing Bioset Class A Lime Stabilization System sized for:								
10-40% Total dry Solids								
8,800 wt-lb/hr.								
20 GPM								
Process Material Description:	Municipal Sewage Biosolids							
Process Material Solids Content:	14% - 30% Total Dry Solids							
Design Biosolids Processing Rate:	2200 to 8800 wet-lb./hr.							
	[ 1000 to 4000 wet-kg/hour]							
Estimated Lime Feed Rate:	352 to 1408 lb./hr. [ 160 to 640 kg/hour]							
	(16% of wet sludge cake mass)							
Estimated Sulfamic Acid Feed Rate:	2 to 9 lb./hr. [1 to 4 kg/hour]							
	(0.1% of wet sludge cake mass)							
Pump Flow rate	2554 to 10,217 wet-lb./hour (5 to 20 GPM)							
(includes added chemicals):	[ 1161 to 4644 wet-kg/hour ( 18 to 74 LPM)]							



Schwing Bioset MB12 Mobile Unit

# **TEST DATA SUMMARY**

## NOTES:

The types polymers which presented the most promising results based on previous Screw Press demos were: Polydyne C-6272, C-6286, C-6288, and C-9555. These were the only polymers used during the pilot testing.



The demo was performed indoors in temperatures between 60-70 degrees F.

SCHWING BIOSET 600 Screw Press- St Cloud Anaerobic									
Date	Time	TRIAL	Flow GPM	FEED SLUDGE DS	Polymer GPH	LBS ACTIV POLY/to DS	RAPID DRY Cake %	DRY lbs CAKE/HR	
Polydyne C-6272 45%									
9/Sep	7:30	1	18.00	4.11	1.91	40.00	21.00	360.00	
	8:15	2	25.00		2.73	40.00	20.95	513.00	
	8:45	3	30.00		3.28	40.00	21.00	615.00	
	9:20	4	33.00		3.78	42.00	21.28	718.00	
	11:30	5	24.00		2.62	40.00	20.78	492.00	
	12:45	6	28.00		3.01	38.00		595.00	
Polydyne C-6286 41%									
	14:15	7	28.00		3.36	40-60	lost floc		
Polydyne C-6272 45%									
	15:00	8	28.00		3.17	40.00	21.14	595.00	
Polydyne C-6288 41%									
10/Sep	8:00	9	21.00	4.23	2.01	31.00	22.04	431.00	
	9:00	10	25.00		2.39	31.00	21.00	513.00	
	9:45	11	25.00		2.24	29.00	21.98	513.00	
	10:15	12	25.00		2.08	27.00	20.37	513.00	
	10:50	13	25.00		1.93	25.00	18.16	513.00	
Polydyne C-9555 41%									
	12:30	14	25.00		3.48	45.00	21.83	513.00	
	13:30	15	25.00		3.09	40.00	23.24	513.00	
11/Sep	11:00	16	28.00		3.46	40.00	23.45	513.00	
Polydyne C-6272 45%									
	12:15	17	28.00		3.46	40.00	24.15	513.00	

# Cake Solids Data from Screw Press:





Polydyne C-6272 Floc



Screw Press Filtrate in Collection Tank


## **Solids Capture:**

Filtrate samples were collected by the St. Cloud WWTF lab and consistently ranged between 150 and 190 mg/l. On test at the end of the demo reported 290 mg/l.



Filtrate Samples Taken From Frac Tank

**Dewatered Screw Press Cake:** 



21.98% Cake at St. Cloud



Reactor 1/4 Way Temp147 °FPump Discharge Temp140 °FReactor 1/2 Way Temp151 °FReactor Pressure15 psiReactor 3/4 Way Temp142 °FTransition Pressure0 psiHopper Level11 %	Reactor Inlet Temp	145 °F	Reactor Outlet Temp -40 °F
Reactor 1/2 Way Temp     151 °F     Reactor Pressure     15 psi       Reactor 3/4 Way Temp     142 °F     Transition Pressure     0 psi       Hopper Level     11 %	Reactor 1/4 Way Temp	147°F	Pump Discharge Temp 140 °F
Reactor 3/4 Way Temp 142 °F Transition Pressure 0 psi Hopper Level 11 %	Reactor 1/2 Way Temp	151 °F	Reactor Pressure 15 psi
	Reactor 3/4 Way Temp	142 °F	Transition Pressure 0 psi Hopper Level 11 %

Bioset Control Panel (Reactor Outlet Sensor Not Working)



Bioset Cake – 12.4 pH, 33.8% Solids



## Monday September 8<sup>th</sup>:

The Screw Press trailer showed up at the St. Cloud WWTF Monday morning. The equipment was situated inside the biosolids building, and connected to electrical and water services.

Functional testing was performed.

## **Tuesday September 9th:**

Initial jar testing was performed and it was decided that the Ashland Polydyne C-6272 polymer would be initially tested.

Sludge feed was initially set at 18 GPM (360 lbs./hr.) to get the Screw Press and auxiliary components dialed in. Over time, flow was increased to 33 GPM (718 lbs/hr.), then backed down to 28 GPM (595lbs/hr.) to prevent the filtrate feed tank from overflowing. Polymer dosages ranged between 38 to 40 lbs/DT. Depending on the dosage rate, the C-6272 produced a medium floc with a yellowish color of cloudiness. The color could be attributed to the physical make-up of the sludge. Cake solids were consistently around 21% solids throughout this polymer testing period.

Polydyne C-6286 Polymer was tried in the afternoon but is was quickly observed that floc could not be made and the testing was stopped and the polymer was change back to the C-6272.

The Bioset process ran uninterrupted throughout the day. Lime addition was between 10-12% by weight of the biosolids throughout the testing week Sulphamic Acid was added at a rate of 0.1% by weight of the biosolids throughout the testing week. No Bioset cake samples were taken.

## Wednesday, September 10<sup>th</sup>:

On Wednesday, the polymer was switched to Polydyne C-6288 at dosages between 25 and 31 lbs/DT. After initial start-up at 21 GPM, sludge feed was kept at 25 GPM (513 lbs./hr.). A medium floc with the same yellowish cloudiness as previously seen. The C-6288 resulted in similar cake solids (approx. 21%) as the C-6272, but at lower dosage rates (25-31 lbs./DT).

The polymer was switched out to the Polydyne C-9555 in the afternoon. This polymers best result was 23.45% solids at a dosage rate of 40 lbs./DT at a sludge feed of 25 GPM (513 lbs./hr.).

The Bioset process continued to operate uninterrupted throughout the day. No samples were taken.

## Thursday, September 11<sup>th</sup>:

Two more test runs were performed on Thursday at a fixed sludge feed of 28 GPM (513 lbs/hr.) to accommodate visitors that came by to watch the Screw Press and Bioset system in operation. Polydyne C-9555 and C-6272 polymers were run. The last test on the C-6272 produced 24.14% solids at a dosage rate of 40 lbs/DT.



Sludge cake samples off the Bioset discharge were taken to a local lab for Ammonia, Fecal, pH and Total Solids analysis.

The Screw Press and Bioset was shut down and disconnected and cleaned Thursday afternoon.

## CONCLUSIONS

Currently, St. Cloud WWTF thickens their waste activated Anaerobic to 3.5-4.0% sludge for land application on local farmlands. St. Cloud is looking at dewatering options to produce cake that can be ultimately save in storage and land application costs in the future. The pilot was able to demonstrate that the Schwing Bioset FSP 600 screw press is ideally suited to improve the dewatering of the 4.0% anaerobically waste activated sludge at St. Cloud WWTF. 22% cake solids was achieved at a polymer dosage rate of 29 lbs/DT and screw press loading rate of 513 lbs/hr. using the Polydyne C-6288 polymer. The C-6288 consistently produced quality performance at lower dosage rates. Tested filtrate solids ranged between 150 and 290 mg/l.

Independent lab results that tested the Bioset cake showed that the process was able to produce 33.8% solids with a pH of 12.4. Ammonia was 1.6 mg/g which is well above the 0.5mg/g requirement to meet PFRP. The reaction time, temperature, Ammonia content and pH of the Bioset end product meets Class A requirements.

The Screw Press and Bioset testing at St. Cloud provided a great indicator regarding the ease of operation and the quality of performance that can be expected from a permanent installation in the future.

If there are any questions regarding any part of this Pilot report, please do not hesitate to contact us at your earliest convenience. Based on the successful performance of the equipment the Screw Press and Bioset system can offer high performance dewatering and Class A process from an easy to maintain, low energy consuming package.



Sample	Collection	Collection	%ts	%vs	pН
Location	date	Time			
Feed	9.9.14	13:20	4.20		8.41
Feed	9.9.14	15:25	4.30		8.29
Feed	9.10.14	9:30	4.20		8.41
Feed	9.11.14	11:15	4.30	60.66	7.73
Feed	9.11.14	14:00	need to a	re-run	7.86
Cake	9.9.14	13:20	20.4		
Cake	9.9.14	15:25	16.3		
Cake	9.10.14	9:30	20.7		
Cake	9.11.14	11:15	20.8		
Cake	9.11.14	14:00	22.8		
Filtrate	9.9.14	13:20	0.16		
Filtrate	9.9.14	15:25	0.15		
Filtrate	9.10.14	9:30	0.18		
Filtrate	9.11.14	11:15	0.19		
Filtrate	9.11.14	14:00	0.29		

## St. Cloud Lab Results





UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON D.C., 20460

August 16, 2011

Office of Water

Franz Tillman Schwing Bioset Inc. 98 Mill Plain, Suite 2A Danbury, CT 06811-6101

Dear Mr. Tillman:

Re: Request for National Equivalency of Schwing Bioset's "Bioset" Process

This is in response to your "Pathogen Reduction Equivalency Application Package for Biosolids Treatment Processes" which was signed on February 22, 2011 and submitted to Mark C. Meckes, Chair of EPA's Pathogen Equivalency Committee (PEC). Supporting documents were submitted on May 13, and July 28, 2011. The PEC has reviewed the application, data, appendices and supporting documentation as well as an operation and maintenance manual which was submitted with an earlier request.

The "Bioset" process is described as follows: Dewatered municipal sludge solids between six to thirty-five percent total solids by weight are mechanically mixed with calcium oxide (quicklime) to achieve a pH of greater than or equal to twelve standard units. Sulphamic acid is added to, and mixed with the sludge/quicklime to promote an exothermic reaction which increases the temperature of the mixture to equal to or greater than 55°C (131°F). The sludge/quicklime/sulphamic acid mixture is then directed to a pressurized plug flow reactor for a minimum solids retention time of forty minutes at a minimum temperature of 55°C (131°F). Based on the information/data provided, the PEC believes that the "Bioset" process is equivalent to a process to further reduce pathogens (PFRP) when operated as described below and in accordance with the "Process Operation and Maintenance Manual."

The "Bioset" process is considered to be a PFRP equivalent process when it is operated under the following conditions:

- The "Bioset" process is to be used to treat municipal wastewater sludge with a total solids concentration between 6 and 35% by weight and with a minimum ammonium concentration in the reactor discharge of 0.5 mg NH<sup>+</sup><sub>4</sub>/g dry weight.
- Dewatered sludge solids must be mechanically mixed with calcium oxide (quicklime) to achieve a pH of equal to or greater than 12 standard units.
- Sulphamic acid must be mixed with the sludge/quicklime mixture to maintain the temperature of the mix at equal to or greater than 55°C (131°F).



• The process must be operated in a plug flow regime with a minimum operating pressure of 27 kPa (4 psi) and a minimum solids retention time of 40 minutes at a minimum temperature of 55°C (131°F).

I concur with the PEC's opinion and statement that Schwing Bioset's "Bioset" process operating under the above conditions is equivalent to a PFRP process in accordance with 40 CFR 503.32(a)(8). As always the final decision on equivalency rests with the relevant permitting authority at the state or EPA-Regional Office.

As with any wastewater treatment plant using a Class A alternative, you still need to monitor the final product to insure that fecal coliform densities are below 1,000 MPN per gram of total solids (dry weight basis), or *Salmonella* sp. bacteria are below detection limits (3 MPN per 4 grams total solids [dry weight basis]) at the time the sewage sludge is used or disposed, at the time the sewage sludge is prepared for sale or given away in a bag or other container for land application, or at the time the sewage sludge or material derived from the sewage sludge is prepared to meet the requirements in 40CFR503.10(b), 503.10(c), 503.10(e), or 503.10(f)." You will of course also need to meet the requirements for vector attraction reduction (VAR). It is our understanding that VAR will be met using option six as specified in 40CFR503.33(b)(6). That is: Addition of sufficient alkali to raise the pH to at least 12 at 25°C (77°F) and maintain a pH  $\geq$ 12 for 2 hours and a pH $\geq$ 11.5 for 22 more hours.

Best regards and I wish you success with your municipal sludge treatment process. Should you have any questions, please call Mr. Mark C. Meckes at 513-569-7348.

Sincerely,

cc:

Rick Stevens, Senior Scientist (4304T) Biosolids Coordinator Health and Ecological Criteria Division Office of Science and Technology

> PEC EPA Regional Biosolids Coordinators Mark C. Meckes, ORD

**Dewatering Screw Press System** 





# Appendix 3 Pearl Pilot Report





# Nutrient Recovery Demonstration Project at The City of St. Cloud Waste Water Treatment Facility (WWTF)

November 3, 2014

Prepared for: The City of St. Cloud, Minnesota



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# 1. Executive Summary

The City of St. Cloud is evaluating the Ostara Pearl® Nutrient Recovery Process to recover phosphorus and ammonia from the liquid stream produced by a proposed dewatering process at the Wastewater Treatment Facility (WWTF). The Pearl demonstration project operated from August 29 through September 23, 2014 and was paired with two separate pilot scale dewatering processes.

Ostara's nutrient recovery technology is more than just a solution to the issue of finite phosphorus resources. It offers an innovative and sustainable approach to managing the complex interdependencies between the environment, the global food supply and our collective stewardship of the planet's water resources. Instead of discharging phosphorus into waterways, Ostara recovers phosphorus as a high quality fertilizer and reduces phosphorus pollution.

When Waste Activated Sludge (WAS) from the Biological Nutrient Removal (BNR) process is anaerobically digested, the phosphorus that was biologically removed is released back into solution. When the digested biosolids are thickened, the soluble phosphorus is concentrated in the filtrate. This filtrate must be returned to the plant for retreatment. The recycled phosphorus becomes trapped in a vicious cycle, eventually overwhelming the BNR process.

Additionally, recycled orthophosphate can combine with magnesium and ammonia, both typically present in sufficient concentrations to form struvite, which precipitates as scale and grit in sludge treatment infrastructure, impacting reliability and increasing costs. Ostara Nutrient Recovery Technologies intercepts this recycle stream and recovers the phosphorus along with some of the nitrogen, producing a slow release fertilizer called Crystal Green®.

The Ostara pilot demonstrated the Pearl process performance, operational characteristics, and quality of manufactured product. In addition to the Pearl process, a test procedure for the WASSTRIP<sup>®</sup> process was developed and implemented from September 8 to September 23. The Waste Activated Sludge Stripping to Recover Internal Phosphorus (WASSTRIP®) process extracts phosphate from WAS prior to thickening and digestion by holding it under anaerobic conditions. By shunting nutrients around the digesters (P, Mg, K), the WASSTRIP process creates an auxiliary wastewater stream that increases the ortho-phosphorus available for the Pearl reactor and the volume of Crystal Green produced.

BNR allows the City to meet its 1 mg/l phosphorus limit without adding ferric, but anaerobically digesting waste activated sludge from the BNR process creates phosphorus management challenges. Combining biological phosphorus removal with

nutrient recovery eliminates ferric costs while recovering struvite as a slow-release fertilizer. Nutrient recovery provides a third exit for phosphorus from the plant, providing operators with improved nutrient management control.

Nutrient recovery offers triple-bottom-line benefits (Social, Environmental, Financial) to St. Cloud:

- **Phosphorus removal** the Pearl<sup>®</sup> process provides an alternative to metal salts (e.g. ferric chloride) to remove phosphorus from wastewater, thus avoiding chemical procurement, dosing and sludge management costs and impacts.
- **Ammonia removal** struvite precipitation recovers one mole of ammonia for each mole of phosphorus recovered, reducing the ammonia load.
- Environmental stewardship Crystal Green's low energy, local production reduces carbon footprint, and its slow release performance reduces nutrient runoff.
- **Financial** the revenue paid to the City for the fertilizer produced covers the system operating costs (power, labor, chemicals). Ostara commits to purchase every ton of fertilizer the City produces for a fixed price, allowing the system to operate at net zero cost in perpetuity.

The principal results of the study include:

- 1. Phosphorus recovery at 87%, with an average of 76% phosphorus recovered, and ortho-phosphate concentration average of 68 mg/l.
- 2. The pilot produced 80 pounds of struvite and released a maximum concentration of 98 mg/l of othro-phosphate (PO<sub>4</sub>-P) from WASSTRIP process.
- 3. The implementation of WASSTRIP to release phosphorus from the Waste Activated Sludge (WAS) was the driving force behind the production of fertilizer.
- 4. The process produced high-quality fertilizer conforming to applicable fertilizer quality standards.

Ostara is confident that these results demonstrate that the Pearl® process is a viable technology to recover nutrients into a high quality fertilizer, branded as Crystal Green, at the City of St. Cloud WWTF. Ostara acknowledges the diligent support received from the City, Advanced Engineering and Environmental Services, Inc. (AE2S), and Vessco during planning, setup, operation, and decommissioning of the pilot demonstration. This assistance was critical to the success of the project.

# 2. Background

The City of St. Cloud received a pilot scale demonstration of Ostara's Pearl process and testing of Ostara's Waste Activated Sludge Stripping to Recover Internal Phosphorus (WASSTRIP) process at the Wastewater Treatment Facility. The Pearl process treats post digested, dewatered sludge following anaerobic treatment. The Waste Activated Sludge Stripping to Recover Internal Phosphorus (WASSTRIP®) process extracts phosphate from waste activated sludge (WAS) prior to thickening and digestion by holding it under anaerobic conditions. The pilot goals were to demonstrate the Pearl process performance, operational characteristics, and the quality of manufactured product.

The City of St. Cloud facility is designed for biological nutrient removal utilizing the Modified Ludzack-Ettinger (MLE) process that treats an average flow of 13 million gallons per day. Currently, the SCWTTF co-thickens primary sludge and waste activated sludge (WAS) to about 8-9% prior to digestion. About 32,000 gpd (10,000 ppd) of sludge exits the digesters daily at about 3.8% solids and is pumped to a 5 MG storage area where it stays until it can be land applied. The digested sludge is projected to increase to about 56,400 gpd (18,000 ppd) by 2035 and be at the same concentration (3.8%).

Ostara's Pearl process was paired with two separate pilot scale dewatering processes. The digested biosolids were stored in a truck before entering the dewatering pilot. After dewatering of the sludge, the liquid stream was stored in two tanks operated in series before entering the Pearl reactor.

WAS from BNR processes readily releases stored phosphate under anaerobic conditions. BNR selects for phosphorus accumulating organisms (PAOs), which store cations such as potassium (K+) and magnesium (Mg2+) as a counter ion when they store phosphate (PO4 3-). In WASSTRIP, the PAOs release these cations in proportion to phosphate release. Magnesium is typically the rate limiting precipitant for struvite (MgNH3PO4.6H2O) formation in anaerobic digestion. Its removal in WASSTRIP therefore controls struvite formation in the digester and downstream. Potassium impacts digested sludge dewatering performance at plants practicing BNR by unfavorably changing the ratio of monovalent to divalent cations in digested sludge. Its removal in WASSTRIP therefore improves dewaterability conditions.

The WWTF Pearl demonstration project began on August 29 and was completed on September 23, 2014.

## 2.1 The Pearl® Process

The Pearl<sup>®</sup> process (shown diagrammatically in Figure 2) is based on the controlled precipitation of struvite within an up-flow fluidized bed reactor. Struvite is an equi-molar crystalline matrix of magnesium, ammonium and phosphate ( $NH_4MgPO_4\bullet6(H_2O)$ ), which forms when these three ions are present in a solution above its saturation point. The saturation point is governed by two factors: the concentration of these three ions and the solution pH.

Two principles are fundamental to the process – maximizing the efficient removal of phosphorus, and consistently recovering a high quality product. The resulting design incorporates features that support these objectives, such as the reactor geometry and the process control philosophy. These design features together with Ostara's extensive experience of struvite precipitation enable the Pearl process to consistently produce a high quality fertilizer product – branded Crystal Green<sup>®</sup>.



Figure 2: The Pearl® Process - a simple, proven technology that recovers phosphorus from used waters producing a premium quality, slow release fertilizer.

The rate of struvite formation in the Pearl process is carefully controlled to create a high quality product with specific physical properties (size, hardness, purity etc.). Adjusting pH controls process performance (the process is generally operated between pH 7.5 and 8.5). The Pearl® process is capable of achieving phosphate removal rates in excess of 90%, with the economic optimum performance depending on waste stream characteristics and treatment objectives.

Nutrient recovery offers triple-bottom-line benefits (Social, Environmental, Financial) to St. Cloud:

- **Phosphorus removal** the Pearl<sup>®</sup> process provides an alternative to metal salts (e.g. ferric chloride) to remove phosphorus from wastewater, thus avoiding chemical procurement, dosing and sludge management costs and impacts.
- **Ammonia removal** struvite precipitation recovers one mole of ammonia for each mole of phosphorus recovered, reducing the ammonia load.
- Environmental stewardship Crystal Green's low energy, local production reduces carbon footprint, and its slow release performance reduces nutrient runoff.
- **Financial** the revenue paid to the City for the fertilizer produced covers the system operating costs (power, labor, chemicals). Ostara commits to purchase every ton of fertilizer the City produces for a fixed price, allowing the system to operate at net zero cost in perpetuity.

Ostara markets and sells the product made by the Pearl process as a slow release fertilizer (branded Crystal Green®). Crystal Green is completely inorganic, very high in purity, and pathogen-free. Crystal Green has a number of characteristics that differentiate it relative to conventional phosphorus fertilizers and position it as a valuable product, including slow nutrient release, beneficial magnesium content, and environmentally sustainable production. Ostara has certified Crystal Green® as a fertilizer in the United States, Canada and Europe, and has made substantial investments to establish it as a recognized fertilizer brand.

## 2.2 The WASSTRIP® Process

The WASSTRIP® process solves struvite challenges throughout sludge treatment streams by extracting phosphorus and magnesium from sludge. In WASSTRIP waste activated sludge (WAS) is held under anaerobic conditions prior to thickening and digestion. Enhanced Biological Nutrient Removal (EBNR) sludge readily releases stored phosphorus and magnesium under anaerobic conditions. The hydraulic retention time of the WASSTRIP process is influenced by the phosphorus content of the WAS and the availability of volatile fatty acids (VFAs). Subsequent sludge thickening diverts released

phosphorus and magnesium into thickening liquor - away from anaerobic digestion. This controls struvite formation in the digester (where ammonia is formed) as magnesium and phosphorus are shunted around the digester. Thickening liquor is then sent to the Pearl® process (together with digested sludge dewatering liquor), where nutrients are recovered as Crystal Green®.

The diagram below shows how Pearl® and WASSTRIP® would integrate with the treatment process at City of St. Cloud WWTF:



WASSTRIP® in combination with Pearl® delivers significant benefits:

- Enhanced prevention of struvite scale throughout the sludge treatment stream.
- Improved sludge digestion, reducing sludge volumes and increasing gas production.
- Increased removal of phosphorus and ammonia in the Pearl® process.
- Reduced phosphorous content of biosolids.
- Increased Crystal Green® production, hence increased environmental benefits.

# 3. Objectives

The objectives of this study were to:

- Demonstrate the phosphorus and nitrogen removal performance of Ostara's Pearl® nutrient recovery process on The City of St. Cloud's dewatered liquid stream.
- Demonstrate Pearl's operational characteristics and process robustness.
- Demonstrate the relationship between reactor pH, sodium hydroxide dosing, and process performance.
- Demonstrate the possibilities of phosphorus release using the WASSTRIP process.
- Demonstrate the efficiency of the Crystal Green fertilizer production as a part of the Pearl Process.

## 4. Method

Ostara's pilot equipment was located in a building with close access to the feed stream(s). The area required sufficient head space for Ostara's equipment (see below) with the necessary utilities made available. The pilot equipment is designed to run automatically, and should be operated continuously (24/7). A feed flow rate of up to approximately 6500 gallons/day is currently estimated for the project.

The primary objectives of the operation phase center on establishing stable treatment performance and demonstrating product quality. Feed and/or Pearl process conditions can be changed to evaluate process performance. Ostara's operational duties for the Pearl pilot include routine sampling and analysis, chemical preparation and equipment oversight/maintenance.

Ostara assembled its Pearl® pilot plant at The City of St. Cloud WWTF on August 21<sup>st</sup>, 2014. On August 22<sup>nd</sup>, Ostara seeded the reactor with product from an existing full-scale nutrient recovery facility. On August 29<sup>th</sup>, the liquid stream coming off of the dewatering unit began feeding to the system. The photo below (Figure 3) shows Ostara's equipment setup.



Figure 3: Ostara's Pearl® 5 pilot unit at The City of St. Cloud WWTF

The filtrate feed to the Pearl unit was obtained by collecting and directing the liquid stream coming off of the dewatering unit into a 21,000 gallon frac tank. The residual solids in the filtrate settled in the tank as the liquid travelled from the receiving end of the tank to the feed outlet on the opposite side of the tank.

The City of St. Cloud used two different dewatering contractors for this pilot study. Both dewatering units were screw presses that operated in batches to produce 10 gal/min of filtrate. The dewatering vendors were Ishigaki USA Ltd. that operated from August 28<sup>th</sup> to September 5<sup>th</sup> followed by Schwing from September 8<sup>th</sup> to September 19<sup>th</sup>. The supply of filtrate allowed the Pearl reactor to operate continuously with periodic stoppages for maintenance or for Crystal Green harvest.

During the end of the second week of operation, the phosphorus concentration in the filtrate was insufficient to drive fertilizer production. A change in pH across the process from the digester to the dewatering pilot through the storage trucks in series into the Pearl reactor precipitated struvite within the process. This was a result of the configuration for the pilot scale demonstration, which will not occur at full scale. Changes to this configuration were not enough to elevate the phosphorus concentration, so we focused on releasing the phosphorus from the WAS through WASSTRIP.

Ostara developed a phosphorus release process for the WWTF that was implemented the weekend of September 6<sup>th</sup>. By September 8<sup>th</sup>, a dual feed system into the Pearl unit was assembled to feed the phosphorus-rich WASSTRIP stream, as well as the ammonia-rich filtrate stream.

The WASSTRIP feed was prepared on a pilot scale in batches. Waste Activated Sludge (WAS) was pumped into a grit chamber and allowed to ferment for up to 72 hours. The fermenting process releases phosphorus, which was mixed for a half hour every 24 hours. WASSTRIP samples were tested several times each day to monitor the phosphorus release. When a WASSTRIP batch reached an acceptable concentration, it was pumped into a tanker truck, and stored in order to simultaneously feed the Pearl unit.

Process filtrate feed, WASSTRIP feed, and effluent samples were collected daily and analyzed on site using a Hach spectrophotometer. Test includes ammonia, dissolved magnesium, dissolved calcium, ortho-phosphate, and total phosphorus concentrations. Fertilizer samples were collected by Ostara for additional analysis to determine the quality of the product.

The process was stabilized by maintaining a consistent 1:1 molar ratio of magnesium to phosphorus in the reactor. The 1:1 molar ratio was maintained by adjusting magnesium chloride dosing relative to the ortho-phosphate concentration measured on-site.

Magnesium chloride stock solution was made by adding water to three 50lb bags of magnesium chloride flakes in a dilute magnesium chloride storage/batch tank. The solution was then metered into the reactor through a dosing pump at the calculated set point.

Sodium hydroxide was dosed automatically using feedback from the reactor's pH probe to maintain a target pH set point of 8.0. A 25% sodium hydroxide solution was prepared by mixing 10 gallons of a 50% NaOH solution into 10 gallons of non-potable water. The sodium hydroxide automatically dosed using a metering pump.

Ostara's pilot scale equipment consisted of:

- Reactor tower 18" square and 18'6" tall. This is shipped in two sections, which were bolted together then lifted into position. The tower requires attachment to a structure (e.g. a wall or handrail) above its mid height for stability. A total floor to ceiling height of ~21' was needed to allow for reactor seeding, which also required a safe means of access for Ostara's technician.
- Equipment skid that occupied a footprint of approximately 8' square and houses electrical/control equipment, feed & recycle pumps, and chemical dosing equipment. The skid needs to be located within ~12' of the reactor, with easy access between each.
- Three chemical storage tanks that are approximately 42" (for MgCl2), 36" (varying uses, not always needed) and 24" (for NaOH) in diameter respectively. These require locating within ~12' of the skid.
- Clarifier tank that is approximately 30" in diameter and is located immediately adjacent to the reactor. The overflow from this tank (Ostara's treated effluent) was discharged to an appropriate drain located within ~12'.
- Product drying rack that is approximately 2' by 4'
- Interconnecting piping

# 5. Results and Analysis

The MS Excel spreadsheet included with this report ("Pearl Demonstration Project – City of St. Cloud.xls") provides the raw chemical analyses from the Hach test kit analysis and the operational data collected during the test. An analysis of the key data is presented below.

## 5.1 Pearl Pilot Results



Orthophosphate removal results are summarized in Figure 4 below:

# Figure 4: Pearl® influent and effluent ortho-phosphorus concentration and recovery rates

The pilot demonstration included two phases with distinctly different results. Before the WASSTRIP feed was setup, the influent filtrate ortho-phosphate concentrations ranged from 16 mg/L to 33 mg/L, with effluent concentration average of 16 mg/L. The percent recovery was relatively low during this phase with an average of 29% ortho-phosphate recovery. During the second phase, WASSTRIP ortho-phosphorus concentrations averaged 77 mg/L, bringing the combined influent concentrations to an average of 66 mg/L. The percent recovery reached 87% with an average of 76% phosphorus recovery. By temporarily operating at a higher pH of 8.3, the ortho-phosphate removal efficiency was increased. Table 1 below summarizes average study conditions and removal rates.

Parameter	Ostara Influent	Ostara Effluent	% Removal
PO4-P (mg/l)	66	15	76
NH3-N (mg/l)	396	446	6.5
Mg (mg/l)	45	42	
Ca (mg/l)	62	62	
Crystal Green Production	80	bs. (36.3 kg)	

## Table 1: Average demonstration study conditions and performance for Phase II

The results indicate the phosphorus and ammonia recovery rates were acceptable. The ammonia values seen in table 1 illustrate a compound error in feed flow and concentration. We had to calculate the combined feed concentration using a 3:1 blending ratio. Blend ratio is determined by the equimolar concentration for all three components in struvite. The 3:1 Average describes the flow into the reactor. It is three (3) parts WASSTRIP for one (1) part filtrate. The pilot needed 1/4 of the flow from the filtrate in order to get the correct ratio of Mg:P:N. The data spreadsheet shows this to give a snapshot of what the concentrations of the flow going into the reactor were each day. In order to correctly adjust the dosing each day, we calculated the daily chemical averages to represent the total flow that was going into the reactor.

Feed Mg:P ratio is calculated based on the daily concentrations to determine how supersaturated the solution and understand the difference of Mg required. This provides information to understanding feed line scaling and potential feed formation issues.

Unfortunately, NH3-N Hach measurements can be unreliable due to a number of issues. Stoichiometric ally, ammonia removal is equimolar since we are making struvite. A calculation in the attached spreadsheet based on P removal yields approximately 6% NH3-N removal.

The amount of fertilizer produced during this study was lower than expected for the duration of the pilot due to the low concentration of phosphorus in the filtrate. Prior to the WASSTRIP feed, the struvite reaction was incomplete. Once corrected, we completed five harvest events. After subtracting the initial seed volume, the pilot produced 80 pounds (36.3 kg) of struvite. At full scale, performance design should be approximately 2 tons per day.

## 5.1 Struvite Quality

Struvite produced from the pilot (or Crystal Green®) was sent to a third party laboratory to measure nutrient content and metals content to confirm that the product formed was substantially made up of struvite and to ensure the product would meet all fertilizer

standards for metals. Appendix A contains the full lab reports for the samples analyzed, and table 2 below summarized the most relevant results. The data confirms that the struvite produced at the St. Cloud WWTF met all regulatory requirements, and that a full-scale facility will produce fertilizer meeting the high quality commercial fertilizer standards.

	Harvest #1	Harvest #2	
Analytes	9/11/2014	9/18/2014	Units
P2O5	29.5	29.7	%
Ammonia (NH3-N)	5.72	5.43	%
Aluminum (Al)	29.2	24.7	mg/kg
Arsenic (As)	1.36	1.33	mg/kg
Barium (Ba)	0.83	0.84	mg/kg
Cadmium (Cd)	0.07	0.07	mg/kg
Calcium (Ca)	937	2720	mg/kg
Chromium (Cr)	0.04	0.4	mg/kg
Cobalt (Co)	0.1	0.12	mg/kg
Copper (Cu)	1.57	2.55	mg/kg
lron (Fe)	76.2	127	mg/kg
Lead (Pb)	0.16	0.16	mg/kg
Magnesium (Mg)	10.1	9.72	%
Manganese (Mn)	4510	13200	mg/kg
Molybdenum (Mo)	0.21	0.21	mg/kg
Nickel (Ni)	0.31	0.54	mg/kg
Potassium (K)	797	799	mg/kg
Selenium (Se)	0.8	0.8	mg/kg
Silver (Ag)	0.36	0.06	mg/kg
Sodium (Na)	103	189	mg/kg
Zinc (Zn)	1.21	1.51	mg/kg
Mercury	0.024	0.02	mg/kg

#### Table 2: Struvite Harvest Lab Results

The data confirms that the struvite formed in the pilot and that none of the metals measured are present at levels that would inhibit the marketing of the formed product as a fertilizer. The analysis also shows that there are substantial trace components (0.5 to 2% Na, K, Fe and Ca). None of these impurities are of a concern in terms of fertilizer regulation, but some may interfere with the physical characteristics and/or yield recovery of a Pearl system.

# 6. Discussion and Conclusions

The pilot scale demonstration complete on September 23, 2014 confirmed that P removal and recovery as struvite is feasible based P from the dewatering pilot with WASSTRIP P release. The issues experienced with the pilot process configuration would be eliminated at full scale.

Based on Ostara's experience with over 30 pilot tests, we consider this pilot demonstration project to have successfully fulfilled all identified objectives.

- Achieved max of 87% P recovery with an average of 76% P recovery.
- Operated continuously at target pH of 8.0 to produce 80 pounds of struvite
- Released max concentration of 98 mg/l of othro-phosphate (PO<sub>4</sub>-P) from WASSTRIP process

The results confirm stable nutrient removal performance in accordance with expectations.

The final product sent for testing passed fertilizer regulations to be branded Crystal Green. Analysis of the struvite recovered during the study confirmed that high quality fertilizer will be recovered by a full-scale Pearl process at the WWTF.

Ostara's nutrient recovery technology is a viable option for The City of St. Cloud that delivers considerable technical, financial and environmental benefits. Based on these results, we recommend conducting an economic evaluation to verify that the capital and operating costs for a full scale facility

APPENDIX A: STRUVITE PRODUCT ANALYSIS RESULTS



PES Phoslab Environmental Services, Inc.

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#### CERTIFICATE OF ANALYSIS

Client:

Ostara USA, LLC 1723 S. Kings Ave. Brandon, FL 33511

Attention: Phone Number: Fax Number: Project Name: Project Number: Project Location: Sampled By: Date Sampled: Date Received: Date Reported: Lab. Report #:

Ram Prasad (rprasad@ostara.com) 813-285-1321 (Mobile) 813-436-8748 CG QC n/a Florida Client 09/11/14 - 09/18/14 10/24/14 1 10/27/14 10:45 102414-009

#### **Project Description**

The analytical results for the samples identified in this report were submitted for analysis as outlined by the attached Chain of Custody. The results for the quality control samples were reviewed and found to meet the acceptance criteria for precision and accuracy or properly Ragged. Unless noted in this report or a case narrative, all data in this analytical report is in compliance with NELAC standards. This report may not be reproduced in part without the permission of PES.

Notes: Sample results reported at the Method Ddetection Limit (MDL)

Approved By: Megan Skeen Megan Skeen, Quality Assurance officer

igon, D

If you have any questions, the above name should be contacted at 863-682-5897 8:00 A.M. - 5:00 PM M-F

PES Report Data Qualifier COC Sample Log-In Total Pages:

9

1

COC: 102414-009

Page 1 of 9



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#### CASE NARRATIVE

Lab. Report #: Project Name:

102414-009 CG QC

I. Sample Receiving Notes Samples listed on Chain of Custody # 102414-009 were received with containers intact, and at the proper temperature for the requested analyses.

II. Analytical Data Notes The analyses were performed in accordance with Phoslab Environmental Services SOP's and industry-standard methodologies in compliance with FDEP/NELAC criteria. There were no notable problems encountered in the analytical process.

III, Quality Control Notes

There were not significant quality control anomalies associated with this work order.

COC: 102414-009

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#### CASE NARRATIVE

Lab. Report #: Project Name: 102414-009 CG QC

I. Sample Receiving Notes Samples listed on Chain of Custody # 102414-009 were received with containers intact, and at the proper temperature for the requested analyses.

II. Analytical Data Notes The analyses were performed in accordance with Phoslab Environmental Services SOP's and industry-standard methodologies in compliance with FDEP/NELAC criteria. There were no notable problems encountered in the analytical process.

III, Quality Control Notes There were not significant quality control anomalies associated with this work order.

COC: 102414-009

Page 2 of 9



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#### CERTIFICATE OF ANALYSIS EPA 6010B - Metals / EPA 7471B - Mercury

Sample ID: Sample Description/ Sample Date: Preparation Date/M Analysis Date/Time: Method: Batch No.: Initials:	Matrix: ethod:	102414-2; St. Cloud 09/11/14 10/24/14 10/27/14 EPA 6010 102714A- SN	2 Harvest # n/a EPA 305 10:10 0B \$1332	1 / Grab solid 0B		
Analytes:	Cas No.	Results	Unite	Dilution	MDL	POL
Aluminum (Al)	7429-90-5	29.2	me/ke	lx	3.85	25.0
Arsenic (As)	7440-38-2	1.36	mg/kg	lx	0.19	0.25
Barium (Ba)	7440-39-3	0.83	mg/kg	1x	0.04	0.25
Cadmium (Cd)	7440-43-9	0.07 U	mg/kg	lx	0.07	0.25
Calcium (Ca)	7440-70-2	937	mg/kg	lx.	3.80	25.0
Chromium (Cr)	7440-47-3	0.04 U	mg/kg	lx	0.04	0.25
Cobalt (Co)	7440-48-4	0.10 I	mg/kg	lx	0.06	0.25
Copper (Cu)	7440-50-8	1.57	mg/kg	lx	0.21	0.25
Iron (Fe)	7439-89-6	76.2	mg/kg	1x	0.74	2.50
Lead (Pb)	7439-92-1	0.16 U	mg/kg	1x	0.16	0.25
Magnesium (Mg)	7439-95-4	10.1	%	500x	0.033	0.125
Manganese (Mn)	7439-96-5	4,510	mg/kg	50x	5.00	12.5
Molybdenum (Mo)	7439-98-7	0.21 U	mg/kg	1x	0.21	0.25
Nickel (Ni)	7440-02-0	0.31	mg/kg	1 x	0.18	0.25
Potassium (K)	7440-09-7	797	mg/kg	1 x	25.7	125
Selenium (Se)	7782-49-2	0.80 U	mg/kg	1x	0.80	2.50
Silver (Ag)	7440-22-4	0.36	mg/kg	1x	0.06	0.25
Sodium (Na)	7440-23-5	103	mg/kg	1 x	16.6	25.0
Zinc (Zn)	7440-66-6	1.21 1	mg/kg	1x	0.73	2.50
Sample ID: Sample Description// Sample Date/Time: Preparation Date/Mi Analysis Date/Time: Method: Batch No.	Matrix: ethod:	102414-22 St. Cloud 1 09/11/14 10/24/14 10/27/14 EPA 7471 102714-Hj	2 Harvest # n/a EPA 747 14:18 B g309S	I / Grab solid I B		
Analytes: Mercury	Cas No. 7439-97-6	SN Results 0.024 I	Units mg/kg	Dilution Ix	MDL 0.005	PQL 0.025

Note: These results are reported on a natural basis.

COC: 102414-009

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#### CERTIFICATE OF ANALYSIS EPA 6010B - Metals / EPA 7471B - Mercury

Sample ID: Sample Description/ Sample Date: Preparation Date/M Analysis Date/Time: Method: Batch No.: Initials:	Matrix: ethod:	102414-2: St. Cloud 09/18/14 10/24/14 10/27/14 EPA 6010 102714A- SN	3 Harvest # 4 n/a EPA 3050 10:10 B S1332	/ Grab solid B		
Analytes:	Cas No.	Results	Units	Dilution	MDL	POL.
Aluminum (Al)	7429-90-5	24.7	mg/kg	lx	3.85	25.0
Arsenic (As)	7440-38-2	1.33	mg/kg	lx	0.19	0.25
Barium (Ba)	7440-39-3	0.84	mg/kg	lx	0.04	0.25
Cadmium (Cd)	7440-43-9	0.07 U	mg/kg	lx	0.07	0.25
Calcium (Ca)	7440-70-2	2,720	mg/kg	10x	38.0	250
Chromium (Cr)	7440-47-3	0.40	mg/kg	1x	0.04	0.25
Cobalt (Co)	7440-48-4	0.121	mg/kg	1x	0.06	0.25
Copper (Cu)	7440-50-8	2.55	mg/kg	1x	0.21	0.25
Iron (Fe)	7439-89-6	127	mg/kg	1x	0.74	2.50
Lead (Pb)	7439-92-1	0.16 U	mg/kg	1x	0.16	0.25
Magnesium (Mg)	7439-95-4	9.72	%	500x	0.033	0.125
Manganese (Mn)	7439-96-5	13,200	mg/kg	100x	10.0	25.0
Molybdenum (Mo)	7439-98-7	0.21 U	mg/kg	lx	0.21	0.25
Nickel (Ni)	7440-02-0	0.54	mg/kg	Ix	0.18	0.25
Potassium (K)	7440-09-7	799	mg/kg	Ix	25.7	125
Selenium (Se)	7782-49-2	0.80 U	mg/kg	1 x	0.80	2.50
Silver (Ag)	7440-22-4	0.06 U	mg/kg	1 x	0.06	0.25
Sodium (Na)	7440-23-5	189	mg/kg	lx	16.6	25.0
Zinc (Zn)	7440-66-6	1.51	mg/kg	Ix	0.73	2.50
Sample ID:		102414-23		Calaria		
Sample Description/	Matrix:	St. Cloud	Harvest # 4	Grab solid		
Sample Date/Time:	ath a di	10/24/14	DA 7471	D		
Preparation Date/M	ethod:	10/24/14	EPA /4/11	В		
Mathod:		EPA 7471	14.10 R			
Batch No		102714-H	3005			
Initials:		SN	5093			
Analytes:	Cas No.	Results	Units	Dilution	MDL	PQL
Mercury	7439-97-6	0.020 I	mg/kg	1x	0.005	0.025

Note: These results are reported on a natural basis.

COC: 102414-009

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#### CERTIFICATE OF ANALYSIS

Analyte: Analysis Date/Time Analysis Method: Batch No. Initials:	2:	Ammonia 10/27/14 EPA350.1 141024(1) YP	as N (NH3-N) 9:59 3-38)			
Sample ID	Description	Results	Units	Dilution	MDL	PQL
102414-22	St. Cloud Harvest # 1	5.72	0,0	1 00¢	0.01	0.04
102414-23	St. Cloud Harvest # 4	5.43	0,6	1 00c	0.01	0.04
Analyte: Preparation Date / Analysis Date/Time Analysis Method: Batch No. Initials:	Method: ::	P <sub>2</sub> O <sub>5</sub> 10/24/14 10/27/14 EPA 6010 102714A- SN	EPA 3050B 10:10 B \$1332			
Sample ID	Description	Results	Units	Dilution	MDL	PQL
102414-22	St. Cloud Harvest # 1	29.5	0,6	500x	0.058	0.287
102414-23	St. Cloud Harvest # 4	29.7	%	500x	0.058	0.287

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### QUALITY CONTROL DATA EPA 6010B - Metals

27/14 10:10
714A-S1332
414-21
2

	LCS	LCS	LCS	Parent					MS	MSD
	Spike	Result	Recovery	Result	Spike @	Spike	Spike dup		Recovery	Recovery
Parameter	mg/L	mg/L	%	mg/kg	mg/kg	mg/kg	mg/kg	RPD	%	%
Aluminum (Al)	0.200	0.197	98.7	28.3	50.0	83.4	82.6	1.4	110	109
Arsenic (As)	0.200	0.212	106	0.19 U	50.0	52.7	51.5	2.4	105	103
Barium (Ba)	0.200	0.194	97.0	3.89	50.0	53.3	53.0	0.4	98.7	98.3
Cadmium (Cd)	0.200	0.218	109	0.07 U	50.0	51.2	49.8	2.8	102	99.6
Calcium (Ca)	0.200	0.183	91.3	474	250	721	696	10.9	99.1	88.8
Chromium (Cr)	0.200	0.207	103	0.04 U	50.0	47.3	49.8	5.0	94.7	99.5
Cobalt (Co)	0.200	0.203	102	0.06 U	50.0	49.4	47.9	3.1	98.8	95.8
Copper (Cu)	0.200	0.211	106	7.72	50.0	59.8	58.9	1.8	104	102
Iron (Fe)	1.00	1.10	110	206	50.0	259	256	5.9	105	98.8
Lead (Pb)	0.200	0.195	97.3	4.99	50.0	53.0	51.8	2.6	96.0	93.5
Magnesium (Mg)	0.200	0.184	91.8	34.4	50.0	81.5	79.6	4.2	94.2	90.4
Manganese (Mn)	1.00	1.05	105	0.35	50.0	51.5	50.1	2.6	102	99.6
Molybdenum (Mo)	0.200	0.208	104	30.0	50.0	78.5	76.5	4.3	97.0	92.9
Nickel (Ni)	0.200	0.214	107	1.13	50.0	54.2	52.6	2.9	106	103
Phosphorus (P)	0.200	0.189	94.4	270	250	504	495	3.8	93.6	90.1
Potassium (K)	2.00	1.77	88.3	66.1	500	576	582	1.2	102	103
Selenium (Se)	0.200	0.200	100	0.80 U	50.0	53.2	52.3	1.7	106	105
Silver (Ag)	1.00	1.03	103	0.06 U	50.0	50.2	49.1	2.3	100	98.2
Sodium (Na)	0.200	0.189	94.3	139	250	420	422	0.7	112	113
Zinc (Zn)	0.200	0.216	108	137	50.0	188	187	3.4	102	98.9



PES Phostab Environmental Services, Inc. 806 West Beacon Road • Lakeland, Fl 33803 • (863) 682-5897 • Fax: (863) 683-3279 **TOLL FREE 1-688-682-5897** FDOH ID: E84925

LAB BLANK		
Analysis Date/Time:	10/27/14	10:10
Batch:	102714A-S1332	
Initials:	SN	
Analytes:	Results	Units
Aluminum (Al)	3.85 U	mg/kg
Arsenic (As)	0.19 U	mg/kg
Barium (Ba)	0.04 U	mg/kg
Cadmium (Cd)	0.07 U	mg/kg
Calcium (Ca)	3.80 U	mg/kg
Chromium (Cr)	0.04 U	mg/kg
Cobalt (Co)	0.06 U	mg/kg
Copper (Cu)	0.21 U	mg/kg
Iron (Fe)	0.74 U	mg/kg
Lead (Pb)	0.16 U	mg/kg
Magnesium (Mg)	0.66 U	mg/kg
Manganese (Mn)	0.10 U	mg/kg
Molybdenum (Mo)	0.21 U	mg/kg
Nickel (Ni)	0.18 U	mg/kg
Phosphorus (P)	0.50 U	mg/kg
Potassium (K)	25.7 U	mg/kg
Selenium (Se)	0.80 U	mg/kg
Silver (Ag)	0.06 U	mg/kg
Sodium (Na)	16.6 U	mg/kg
Zinc (Zn)	0.73 U	mg/kg

MS = Matrix Spike

MS = Matrix Spike Duplicate LCS = Laboratory Control Standard U = Compound analyzed but not detected to the level shown RPD = Relative Percent Difference



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#### QUALITY CONTROL DATA EPA 7471B - Mercury

<b>SPIKE and LCS DAT</b>	<u>`A</u>									
Analysis Date/Time:		10/27/14	14:18							
Batch:		102714-Hg309S								
Initials:		SN								
Parent Sample ID:		102414-22								
Recovery Limits: (85°	%-115%)									
	LCS	LCS	LCS	Parent					MS	MSD
	Spike	Result	Recovery	Result	Spike @	Spike	Spike dup		Recovery	Recovery
Analyte:	μg/L	μg/L	%	mg/kg	mg/kg	mg/kg	mg/kg	RPD	%	%
Mercury (Hg)	2.50	2.83	113	0.0237	0.0625	0.0873	0.0900	4.2	102	106
LAB BLANK										
Analysis Date/Time:		10/27/14	14:18							
Batch:		102714-Hg309S								
Initials:		SN								
Analyte:		Results	Units							
Mercury (Hg)		0.005 U	mg/kg							

 $\label{eq:LCS} $=$ Laboratory Control Standard$$$ U = Compound analyzed but not detected to the level shown RPD = Relative Percent Difference$$$ MSD = Matrix Spike$$$ MSD = Matrix Spike Duplicate$$$$ 

Page 8 of 9



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### QUALITY CONTROL DATA EPA 350.1 - Ammonia, as N

<u>SPIKE and LCS DATA</u> Analysis Date/Time: Batch: Initials: Parent Sample ID:	10/24/14 141024(13-38) YP 32027-02 (LQC	9:59 RM)					
Recovery Limits; (90%-110%)	Spike @	Spike	Spike Dup		% Recovery		
	%	°/o	%	RPD	Spike	Spike Dup	Limits
Analyte:	11.16	10.81	10.68	1.2	96.9	95.7	90-110
NH3, as N							
LAB BLANK							
Analysis Date/Time:	10/24/14	9:59					
Batch No:	141024(13-38)						
Initials:	YP						
Analyte:	Results	Units					
NH3, as N	19.5 U	mg/kg					

MS = Matrix Spike MSD = Matrix Spike Duplicate LCS = Laboratory Control Standard J = Compound analyzed but not detected to the level shown

### DATA QUALIFIER CODES

### SYMBOL MEANING

- A Value reported is the arithmetic mean (average) of two or more determinations. This code shall be used if the reported value is the average of results for two or more discrete and separate samples. These samples shall have been processed and analyzed independently. Do not use this code if the data are the result of replicate analysis on the same sample aliquot, extract or digestate.
- H Value based on field kit determination; results may not be accurate. This code shall be used if a field screening test (i.e., field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
- 1 The reported value is between the laboratory method detection limit and the laboratory practical quantitation limit.
- J Estimated value. A "J" value shall be accompanied by a narrative justification for its use. Where possible, the organization shall report whether the actual value is less than or greater than the reported value. A "J" value shall not be used as a substitute for K, L, M, T, V or Y, however, if additional reasons exist for identifying the value as estimate (e.g. matrix spiked failed to meet acceptance criteria), the "J" code may be added to a K, L, M, T, V, or Y. The following are some examples of narrative descriptions that may accompany a "J" code:
  - No known quality control criteria exist for the component;
  - The reported value failed to meet the established quality control criteria for either precision or accuracy (the specific failure must be identified);
  - · The sample matrix interfered with the ability to make any accurate determination;
  - The data are questionable because of improper laboratory or field protocols (e.g., composite sample was collected instead of a grab sample).
  - The field calibration verification did not meet calibration acceptance criteria.
- K Off-scale low. Actual value is known to be less than the value given. This code shall be used if:
  - 1. The value is less than the lowest calibration standard and the calibration curve is known to be nonlinear; or
  - 2. The value is known to be less than the reported value based on sample size, dilution.
  - This code shall not be used to report values that are less than the laboratory practical quantitation limit or laboratory method detection limit.
- L Off-scale high. Actual value is known to be greater than value given. To be used when the concentration of the analyte is above the acceptable level for quantitation (exceeds the linear range or highest calibration standard) and the calibration curve is known to exhibit a negative deflection.
- M When reporting chemical analyses: presence of material is verified but not quantified; the actual value is less than the value given. The reported value shall be the laboratory practical quantitation limit. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is greater than the method detection limit. If the value is less than the method detection limit use "T" below.
- N Presumptive evidence of presence of material. This qualifier shall be used if:
  - 1. The component has been tentatively identified based on mass spectral library search; or
  - 2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternative procedures).
- O Sampled, but analysis lost or not performed.
- Q Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared or analyzed after the approved holding time restrictions for sample preparation or analysis.
- T Value reported is less than the laboratory method detection limit. The value is reported for informational purposes only and shall not be used in statistical analysis.
- U Indicates that the compound was analyzed for but not detected. This symbol shall be used to indicate that the specified component was not detected. The value associated with the qualifier shall be the laboratory method detection limit. Unless requested by the client, less than the method detection limit values shall not be reported (see "T" above).
- V Indicates that the analyte was detected in both the sample and the associate method blank. Note: the value in the blank shall not be subtracted from associate samples.
- Y The laboratory analysis was from an improperly preserved sample. The data may not be accurate.
- ? Data are rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
- Not currently accredited for this analyte.
- Not within scope of method.
- Sub-Contracted to a NELAC Certified Laboratory.

Chain of Custody Record # 102414 - 001

Compan					Project Name				•								-	-	-
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0	stara				Project #:										DEP	Form #: 62-770.900	2)		_
Addr 17	723 S Kings Ave	Brandon	33511		Project Mana	ger:									Form	Title: Chain of Cust	ory Record		
Email rp	rasad@ostara.com				Project Locat	ion:									Effec	tive Date: 8/2	004		
Phone:		Fax:			Evidence San	nple (s):	Yes	-	No						FDE	P Facility No.:			
Sampled	1 by [Print Name(s)] /	Affiliatic	u							Preservat	ives (se	se code	s)		Proj	ect Name:			-
															Sam	pling CompQAP	No.:		
Sampler	(s) Signature(s)								Ana	lyses Re	quested	_				Approval	Date:		_
							N-	d-0	E	sle						REQUESTE	D DUE DAT	ц	_
ltem		Samp	oled	Grab or	Matrix	Number of	EHN	hth	BM	steM						/ STI	_		- 1
No.	Field ID No.	Date	Lime	Composite	(see codes)	Containers		>								Remarks		Lab. No.	.1
1 St	. Cloud Harvest #1	9/11/14		Grab		1	×	×	×	×					9	22-1142			-
2 St	. Cloud Harvest #4	9/18/14		Grab		1	×	×	×	×	<u> </u>	$\vdash$				62 /			-
3									$\vdash$		$\vdash$								-
4									$\square$						$\vdash$				1
5															$\vdash$				r
9																			-
7																			
8																			1
6																			
	Shipment Met	hod					¥	Total N	Vumber	r of Cont	ainers								-
õ	at: / /	Via:		Item No.	Reli	nquished by /	Affilia	ttion		Date		Time		Accep	ted by /	Affiliation	Date	Time	1
Returne	d: / /	Via:				PhysL	ıb, Inc	./ Conta	iners					N					
Addition	nal Comments;				Jore	2. 2.	)			1064	Ke .	1155	2	4	-	15	10/2-6/14	55.6	
															$\langle \rangle$	A and	102414	10:44	1
					,	0							2	1		~			
					Cooler No.(s)	/ Temperatur	e(s) (	Û		0	-	San	npling K	it No.	$\setminus$	Equipm	ent ID No.		
				ð	200	3267		υ	Z	3	,						1997		_
MATRI	X CODES: A = A	ir GW	= Groun	dwater SE	= Sediment	SO = Soil	SW	= Surfa	ace Wa	ater W	/ = Wat	ter (Blar	nks)	O = Other	(specify	()			_
PRESE	RVATIVE CODES:	H = H	drochlori	ic acid + ice	I = Ice only	N = Nitri	c acid	+ ice	S=S	sulfuric a	cid + ic	e 0	= Other	(Specify)					

SMP-CHECK-A.orig

rev date: 10/25/13

Sample Log-in Checklist										
Sample L	og-in Cl	necklis	st				1	PhoslabEnvir	onmental	
Shipping Method	PE	5			Date / Tir	me of Rece	eip <u>t:</u>	102414	(0:45	
				Cooler C	heck					
	Ice in coo	ler			Custody	Seal				
Cooler #	Yes	No	Temp	Yes	.No	Intact	No	t Intact		
Thermometer ID: Note: If the tempe bottles in th	erature of a c	cooler is ab	ove 6 <sup>o</sup> C or a ote on <b>"Impr</b>	i custody seal oper Sample	is damaged, List".	then ident	tify the	9		
1)	Custody Se	al on Bottle	es present?	Yes		No	N	)(N		
2)	Condition o Headspace Bubble > 5 Loose Caps Broken Cor	f sample co (Volatiles) mm a ntainers	ontainers	Yes Yes Yes		No No	8	<u>A)/A</u>		
3)	Chain of Cu	istody inclu	ided?	Yes		No				
4)	Acid preser	ved: pH < 2	2	Yes		No	P	A pH strip Lot:		
5)	Base prese	rved: > 12	10	Yes		No	N	pH strip Lot:		
Cooler Unpacked	/ Checked b	y:	Alex	an		_				
Client:	USI	pero.				Date:	10	2414	-	
Project:	CG	00	<u> </u>			COC #:	10	2414-00	29	
			Impr	oper Sam	ple List					

iproper Samp

Bottle#	Out of Hold	Improper	Loose Cap	Damage Bottled	pH	Sample	Action
		Containers			preserve	volume	
	1						

# Appendix 4

#### Appendix 4. St. Cloud WWTF Lab Data. Pilot Testing 2014.

		Pre-dev	watered	Biosolids				Screw	Press - Set	tled Press	ate (Ostar	ra Feed)				Harv	rested Sup	ernate					Dewate	red Cake						WAS	STRIP			
																							Average				Batch In Us	e						
Date	TP as PO4	TS	NH3	TKN	рН	Ortho-P	Date	TP	TSS	NH3	TKN	рН	Ortho-P	Date	TP	TSS	NH3	TKN	рН	Ortho-P	Date	%TP	%TS	′ %NH3	%TKN	рН	WASSTRIP	Date	TP	TSS	NH3	TKN	рН	Ortho-P
8.21.14	1330	4.4	2370	3982	8.00	122	8.21.14							8.21.14							8/26-8/28	0.967	26.5	0.32	1.34	8.3		9/5/2014						
8.22.14							8.22.14							8.22.14							8/29-9/3	0.808	23.1	0.259	1.15	8.3		9/6/2014			210			67.5
8.23.14							8.23.14							8.23.14							9.9.14	3.36	21.1	1.24	5.31	8.1		9/7/2014			216			65
8.24.14							8.24.14							8.24.14			-				9.11.14	3.15	1 21	1.2	5.1	0.000	ļ	9/8/2014	210		45	122		74
0.25.14							0.25.14	22				0.22	26	0.25.14							-	2.07				0.233		9/9/2014	210		24	122		60
0.20.14					7.75	122	0.20.14	22				0.22	20	8.26.14			-											*******	09		34			00
0.27.14			1045	1	7.75	123	0.27.14	22	00	1200		0.14	24	0.27.14														*******		-				
9 20 14			1045		7.09	152.5	29.14 Start	Un	00	1590	1420	8.06	17 20	9.14 Start	17	62	1260		0.77	15.1														
8 30 14							8 30 14	1		1620	1450	8.00	19.45	8 30 14	17	02	1150		8.72	16.5	-						WASSTRIP	Date	TP	755	NH3	TKN	рH	Ortho_P
8.31.14							8.31.14			1575			20.3	8.31.14			1695		8.35	18.6	-						W/ASSTRIP	#########	68.9 65.4	133	112 27	22	рп	67.5
9 1 14							9114			1680			22.3	9114			1575		8 16	20.0									69.8		17	~~		62.3
9.2.14							3.1.14			1000				3.1.14			15/15		0.10	20.1									05.0					02.0
Direct																																		
Dig. 3	1151	4.4	2200		7.68		9.2.14	44		1270		7.57	33.2	9.2.14	16.5	42	1080 <b>1566</b>		8.22	21.2								****			19			69.7
9.3.14					7.61		9.3.14			1551			32.1	9.3.14			1440		8.21	12.6								*****	73.6		21			64.5
9.4.14		4.28		3882			9.4.14			1430			16.4	9.4.14			1510		8.3	12.3								*****	72.7					69.2
9.5.14							9.5.14			1510			23	9.5.14					8.32															
9.6.14							9.6.14						28	9.6.14					7.92															
9.7.14							9.7.14						29.1	9.7.14					7.88								WASSTRIP	Date	TP	TSS	NH3	TKN	рН	Ortho-P
9.8.14							9.8.14						32	9.8.14			351		7.88	22								*****	93.6		30			95.7
9.9.14	**706**	4.31/4.3	2245	3853	8.41/8.29	1	9.9.14	**107**		1490	1530		32	9.9.14	**11.6**		400 <b>403.5</b>	414	7.84	22.0								****	90		30			98
9.10.14	1075	4.2			8.41	743	9.10.14	47					44	9.10.14	14		438.5		7.9	10								****	86		27			96.2
9.11.14	**670**	4.46	2212	3831	7.7		9.11.14	**74.4**	136	1450	1500	7.49		9.11.14	16.7** 14	54	380 <b>419</b>	412	7.98	12								*****			26			94.6
9.11.14	1075	7.3			7.86	690	9.12.14	43					45.1	9.12.14	13.8		423.5		7.88	14														
							9.13.14	41.7					43.4	9.13.14	13.6		455		7.91	13.6														
							9.14.14						41.8	9.14.14			462		7.88	22.1														
							9.15.14	45.4					41	9.15.14	17.3		417		7.96	14														
					7.84	362.1	9.16.14	35 <b>41.5</b>				7.97	39.7	9.16.14	20 23.6	46	987		7.98	22.7														
							9.17.14	38.8					37.2	9.17.14	15.7		407		7.97	10.4														
							9.18.14	32	16	1440	1510	8.22	40	9.18.14	16	84	382 <b>387</b>	400	7.96	13.3														
							9.19.14	32		822	1520		37	9.19.14	24	86	369 <b>389</b>	383	7.62	23.3														
												7.95	32.1341						8.05	16.6211														

Appendix 4. St. Cloud WWTF Lab Data. WASSTRIP process during Pilot Testing 2014.

Sample #	Date	Sampler	ТР	NH3	TKN	
1	9/9/2014	SL	218	112	122	
2	9/12/2014	EM	68.9	11	22	MVTL

In Use

WASSTRIP TP Date

Date	TP	TSS	NH3	TKN	рΗ	Ortho-P
9/5/2014						
9/6/2014			210			67.5
9/7/2014			216			65
9/8/2014			45			74
9/9/2014	218		112 <b>67</b>	122		85
9/10/2014	69		34			68
9/11/2014						

WASSTRIP	Date	TP	TSS	NH3	TKN	рН	Ortho-P
	9/12/2014	<b>65.4</b> 68.9		112 <b>27</b>	22		67.5
	9/13/2014	69.8		17			62.3
	9/14/2014			19			69.7
	9/15/2014	73.6		21			64.5
	9/16/2014	72.7					69.2

WASSTRIP	Date	TP	TSS	NH3	TKN	рН	Ortho-P
	9/17/2014	93.6		30			95.7
	9/18/2014	90		30			<i>98</i>
	9/19/2014	86		27			96.2
	9/20/2014			26			94.6

### Appendix 4. St. Cloud WWTF Lab Data. Supplimental Struvite fFormation Data, Pilot Testing 2014.

### R2E2 Struvite Formation Sampling and Analysis

Date	Sampling Location	ρH	Soluble Phospate mg/L PO4-P	Phosphorus Water Extract mg/Kg	Ammonia mg/L	%тѕ	Particulate Calcium	Dissolved Calcium	Particulate Magnesium	Dissolved Magnesium
11.5.14	Influent (comp. 11.4.14)	7.39	2.02	0, 0	33.0		80.1		19.1	
-	Primary Effluent (comp. 11.4.14)	7.60	2.11		31.1		73.8		18.8	
	Digester Sludge Feed	6.00	2.19%		0.49%	8.70	2.32%		0.46%	
	Digester	7.59	220		2450	4.74	3.99%		0.80%	
	Storage Tank	7.79	169.4		2420	3.92	4.61%		0.71%	
	Holding Tank	7.79	166.1		2510	4.42	4.55%		0.80%	
11.12.14	Influent (comp. 11.11.14)	7.39	2.33		34.5		84.10	74.4	20.20	18.6
	Primary Effluent (comp. 11.11.14)	7.48	2.35		35.1		81.40	73.9	20.2	18.6
	Digester Sludge Feed	6.3	1.96%	1196	0.27%	9.94	1.80%		0.37%	
	Digester	7.59	262.2		2620	4.59	3.78%		0.67%	
	Storage Tank	7.82	182.4		2510	3.52	4.57%		0.77%	
	Holding Tank	7.82	179.2		2550	4.01	4.57%		0.78%	
11.18.14	Influent (comp. 11.17.14)	7.44	2.14		48		84.4	83.8	20.5	
	Primary Effluent (comp. 11.17.14)	7.5	2.25		45.6		77.00	83.4	18.9	
	Digester Sludge Feed	5.8	1.71%	8266	0.19%	9.79	1.51%		0.36%	
	Digester	7.72	244.3		2640	4.54	3.96%		0.97%	
	Storage Tank	7.91	236.2		2840	3.93	4.65%		0.82%	
	Holding Tank	7.97	175.9		2860	3.73	4.76%		0.76%	
11.24.14	Influent (comp. 11.17.14)	7.53	2.22		34.8		84.5	77	20.80	20
	Primary Effluent (comp. 11.17.14)	7.73	2.27		34.3		84.30	82.4	21.20	20.2
	Digester Sludge Feed	6.1	1.92%	8377	0.41%	8.33	1.94%		0.41%	
	Digester	7.7	224.8		1810	4.47	4.25%		0.86%	
	Storage Tank	7.89	177.5		1880	3.99	4.59%		0.94%	
	Holding Tank	7.87	158		2050	2.8	4.76%		0.73%	

\*\* Digester samples were centrifuged and supernate was filtered for Soluble Phosphate analysis

\*\* Influent and Primary effluent were filtered for soluble phosphate analysis

# St. Cloud response to MPCA comments 10-5-2015





DATE: OCTOBER 5, 2015

TO: BRUCE HENNINGSGAARD

FROM: EMMA LARSON, ENVIRONMENTAL COMPLIANCE SPECIALIST

RE: St. CLOUD WASTEWATER TREATMENT FACILITY, EMERGING CONTAMINANTS NUTRIENTS HARVESTING PILOT PROJECT 2015. MPCA COMMENTS RESPONSE, SEPTEMBER 2015.

This document is in response to comments received from the MPCA on September 24, 2015 relating to the Technical Reported submitted February 24, 2015.

# MPCA Comment # 1

Below are Goal 1 and the sub-goals from the work plan. Please provide a few specific sentences following Goal 1 and each additional sub-goal describing the outcome/results relating directly to each goal/sub-goal.

**Goal 1:** Maintain effluent phosphorus below 0.3 mg TP/L. Note that the 2013 annual average WWTF effluent phosphorous was 0.23 mg TP/L (achieved without biosolids dewatering and the resulting supernatant recycle). Additional sub-goals to achieve this goal include:

At the time of proposal submittal, it was expected that extrapolation would be used to predict the potential effluent concentration if a full scale project was installed and the goal of effluent total phosphorous was set at 0.3 mg/L. Dewatering scale and unforeseen operational issues did not allow for a return stream volume representative of a full scale project. The stored filtrate was discharged to the headworks of the WWTF on September 24, 2014 and samples were taken of the effluent were analyzed for Ortho P and Total P over the next 24 hours. These have been populated alongside the monthly average.





• Determine nutrient concentration in supernatant from dewatered biosolids.

Supernatant/filtrate from dewatered biosolids contained less than projected nutrients. The table below, (Table 8 in the Technical Report) lists nutrient concentrations for digested solids, biosolids in storage, biosolids in the pilot holding tank and the dewatered filtrate. These results are can also be found in Appendix 4, under Screw Press – settled pressate (Ostara Feed).

	DIGESTER	BIOSOLIDS STORAGE	BIOSOLIDS HOLDING	FILTRATE
MAGNESIUM (MG/L)	90	50	50	20
AMMONIUM (MG/L)	2500	2490	2460	2300
PHOSPHORUS (MG/L)	250	180	160	25
РН	7.6	7.8	7.9	8.1

- Determine nutrient concentrations in "nutrient reduced effluent" from struvite harvesting equipment and calculated removal efficiency. The harvesting effluent nutrient concentrations can be found in Appendix 4, and in the Ostara laboratory report.
- Determine the potential impact of biosolids dewatering and introduction of dewatering supernatant return flow on WWTF effluent concentrations. The projected impacts of biosolids dewatering and the introduction of recycled supernatant to the head of the facility was modelled using a BioWin wastewater treatment process simulator by Donohue and Associates. The Technical Report also states that "In addition to modelling, the WWTF completed supplemental sampling that follows soluble phosphorous and ammonia across treatment. These sampling events demonstrated that high levels of soluble phosphorous do exist after digestion, and under full scale implementation of dewatering after digestion, may, as originally hypothesized, create a filtrate from dewatering with an elevated soluble phosphorous concentration. Modelling and supplemental sampling are not sufficient to determine the fate of nutrients from liquid biosolids, dewatering and return to headworks, however this data, along with the Struvite Precipitation Potential graph (Figure 11) would suggest that nutrients are present in the applicable concentrations, forms and at a pH that would enable and even promote the precipitation of struvite. If un-controlled, this would cause significant operation and maintenance issues. Return stream phosphorous that is not consumed in controlled or uncontrolled precipitation may still result in elevated effluent total phosphorous from the WWTF thus exceeding the goal of 0.3 mg/L or even exceeding the regulated discharge limit of 1.0 mg/L."
- HARVEST #1 HARVEST #2 Produce high quality, nutrient rich fertilizer. PARAMETER UNITS 9/11/2014 9/18/2014 During this pilot, 80 lbs. of Ostara Pearl was 29.7 produced. The table below shows the P2O5 29.5 % nutrient concentrations of the fertilizer AMMONIA 5.72 5.43 % pellets produced during the pilot. (NH3-N) Additional parameters including metals, MAGNESIUM can be found in the Ostara Technical 10.1 9.72 % (MG) Report.



Ostara stated in their report that, "The data confirms that the struvite formed in the pilot and that none of the metals measured are present at levels that would inhibit the marketing of the formed product as a fertilizer. The analysis also shows that there are substantial trace components (0.5 to 2% Na, K, Fe and Ca). None of these impurities are of a concern in terms of fertilizer regulation, but some may interfere with the physical characteristics and/or yield recovery of a Pearl system. The WWTF pilot team relied on Ostara for their expertise in determining the viability of the product as a fertilizer.

• Prepare for the potential future implementation of phosphorous based agronomic application rates.

This study demonstrated the potential obstacles and operational issues involved in meeting potential phosphorous based application rates. This pilot indicated that significant phosphorus concentration would remain in the dewatered cake biosolids product and therefore reduce the application rates, if calculated using phosphorous agronomic rates. The pilot demonstrated that it is possible to divert phosphorous prior to digestion, if required in future projects or regulations.

 Determine the quantity of magnesium addition and pH adjustment necessary for effective struvite precipitation.
The Ostara laboratory report is being submitted with this response document. This report includes

the magnesium addition rates during this pilot. An average of 7.51 mg of magnesium was added per liter of feed to the Ostara reactor.

 Determine the polymer requirements to dewater biosolids to acceptable levels (indirect goal that will be necessary to determine the primary goals).
Polymer addition requirements to dewater to a desired total solids is instrumental in determining the cost effectiveness of any proposed dewatering project. During this pilot, 37lbs. of polymer were needed per dry ton of cake produced (Figure 6 in Technical Report).

# MPCA Comment # 2

The Measurable Outcomes from the work plan include the statement below regarding project success. Please provide a few specific sentences regarding if this project was successful and why.

Project success will be measured by the successful return of nutrient reduced supernatant to the head of the WWTF such that the quality of the liquid effluent discharge to the receiving waters would not be compromised.

The Summary Objectives of the submitted Technical Report states, "Using the soluble-phosphate results from Biosolids dewatering filtrate alone, even after consideration of the high volume of polymer dilution water, the concern for the quality of liquid stream effluent could be minimal, however modelling completed by Donohue for the purposes of an separate project, shows that the assumption of soluble phosphate precipitating prior to dewatering, and thus remaining with the solids in the cake product, may be unrepresentative of full scale operation. In addition to modelling, the WWTF completed supplemental sampling that follows soluble phosphorous and ammonia across treatment (Figures 16 & 17). These sampling events demonstrated that high levels of soluble phosphorous do exist after digestion, and under full scale implementation of dewatering after digestion, may, as originally hypothesized, create a filtrate from dewatering with an elevated soluble phosphorous concentration. Modelling and supplemental sampling are not sufficient to determine the fate of nutrients from liquid biosolids, dewatering and return to headworks, however



this data, along with the Struvite Precipitation Potential graph (Figure 11) would suggest that nutrients are present in the applicable concentrations, forms and at a pH that would enable and even promote the precipitation of struvite. If un-controlled, this would cause significant operation and maintenance issues. Return stream phosphorous that is not consumed in controlled or uncontrolled precipitation may still result in elevated effluent total phosphorous from the WWTF thus exceeding the goal of 0.3 mg/L or even exceeding the regulated discharge limit of 1.0 mg/L." The WWTF pilot team believe that this summarizes the opinion of our team when determining if the pilot project was a success.

### MPCA Comment # 3

The Measurable Outcomes from the work plan include the statements below regarding parameters to be analyzed. Were all these parameters analyzed for? In what waste streams were they analyzed? I cannot locate some of these parameters in the report.

Parameters to be analyzed:

- Total Phosphorous (TP)
- Total Suspended Solids (TSS)
- Total Solids (TS)
- Ammonia as N
- Total Kjeldahl Nitrogen (TKN)
- pH
- Orthophosphate

Appendix 4, WWTF laboratory results shows results for Pre-dewatered Biosolids, Stress-press – settled Pressate (Ostara feed), Harvested Supernatant, Dewatered Cake and WASSTRIP for all the parameters listed above.

# MPCA Comment # 4

The Measurable Outcomes from the work plan include the statements below regarding transferability. Please provide a few specific sentences regarding your thoughts on transferability of the dewatering and the struvite harvesting to St. Cloud at full scale but also to other facilities. It is hypothesized that data will be transferable to similar BNR treatment facilities with solids return streams.

The City of St. Cloud WWTF team has presented on this pilot numerous times to a variety of audiences.

- MWOA/CSWEA Innovative Conference, February 2015.
- MPCA Annual Conference, March 2015.
- Central States Annual Meeting, May 2015.
- MPCA Biosolids Refresher, May 2015.
- Mississippi River Forum, McKnight Foundation, June 2015.
- MWOA Annual Conference, July 2015.

While it is unknown, the exact nature of the audiences to which this pilot summary was presented, they include Wastewater Treatment Facility representatives, engineers and professionals from throughout the Midwest. The presentations also demonstrated the complexity of pilots at WWTFs which include several elements and pilots to work in series as well as maintaining general operations.



### MPCA Comment # 5

Page 11 of 13, Figure 16 - The modeled data in this figure shows no change between precipitation not simulated and precipitation simulated. Do you have any thoughts on why there was no change in the results between precipitation simulated and not simulated?

This question was forward to Donohue and Associates as it referred to modelling that they completed on behalf of the City of St. Cloud. Please see the response below. The information shown in the table provided reveals that "naturally-occurring" struvite precipitation in the biosolids

after dewatering is expected to have a minimal effect on the effluent P concentration. I believe a simple mass balance helps illustrate why.

The table shows simulated effluent phosphorus assuming dewatering and "naturally-occurring" struvite precipitation is on or off. The values reveal that struvite precipitation has little effect on the effluent phosphorus. Mass balance reveals that simulated sidestream phosphorus concentrations decrease when phosphorus precipitation is considered or included in the model run.

Sidestream Soluble P = 740 mg/L (no struvite formation) Sidestream Soluble P = 610 mg/L (with struvite formation) Difference = 740 - 610 = 130 mg/L (concentration removed by struvite formation in biosolids)

Mass balance calculations estimated the sidestream flow attributed to dewatering to be (in round numbers) 25,000 gpd or (0.025 mgd). The mass of phosphorus "precipitated" or retained in the biosolids is then 0.025 mgd x 130 mg/L x 8.34 = 27 ppd. This represents an estimate of the quantity of phosphorus retained in the biosolids rather than returned to the liquid train.

The forward flow is (in round numbers) 10 mgd. Assuming all the sidestream phosphorus attributed to biosolids dewatering passes through the liquid train to the effluent, because of the high ammonia loading and the inability of the bio-P process to remove it, then 27 ppd-P represents an effluent P concentration of 27 mg/L / 10 mgd / 8.34 = 0.3 mg/L.

The difference shown in Slide 17 is 0.1 mg/L. The above simple mass-balance calculation shows a difference of 0.3 mg/L. Although these values differ, they both represent relatively small fractions of the total predicted effluent concentrations and the general findings are the same:

- 1. Dewatering will increase sidestream ammonia and phosphorus loadings to the liquid train and consequently cause elevated effluent phosphorus concentrations (assuming, of course, no mitigating factors)
- 2. Phosphorus precipitation by "naturally occurring" struvite formation in the biosolids will reduce sidestream phosphorus loadings, but it is not expected to be enough to <u>significantly</u> reduce effluent phosphorus concentrations and, more to the point, provide a reliable singular means of consistent effluent phosphorus compliance.

Cursory data provided reveals that actual struvite precipitation in the biosolids may exceed what was predicted by the simple Biowin analysis. Nevertheless, "naturally occurring" struvite precipitation is not likely to provide a reliable mechanism of consistent effluent phosphorus compliance. Assuming a best-case dewatering sidestream phosphorus concentrations of 200 mg-P/L, the effective liquid train concentration associated with this "best-case" scenario would be 0.025 mgd / 10 mgd x 200 mg-P/L = 0.5 mg-P/L.

# MPCA Comment # 6

Appendix 3 - Ostara's report, page 14, table 1 - This table shows the NH3-N levels increasing from 396 mg/L in the Ostara influent to 446 mg/L in the Ostara effluent for a removal of 6.5%. Please provide additional clarification on why this increased and why the removal percent is positive.

# Please refer to Section 5, page 14, of the Ostara report, where they explain this concern. (Section copied below).

The results indicate the phosphorus and ammonia recovery rates were acceptable. The ammonia values seen in table 1 illustrate a compound error in feed flow and concentration. We had to calculate the combined feed concentration using a 3:1 blending ratio. Blend ratio is determined by the equimolar concentration for all three components in struvite. The 3:1 Average describes the flow into the reactor. It is three (3) parts WASSTRIP for one (1) part filtrate. The pilot needed 1/4 of the flow from the filtrate in order to get the correct ratio of Mg:P:N. The data



spreadsheet shows this to give a snapshot of what the concentrations of the flow going into the reactor were each day. In order to correctly adjust the dosing each day, we calculated the daily chemical averages to represent the total flow that was going into the reactor.

Feed Mg:P ratio is calculated based on the daily concentrations to determine how supersaturated the solution and understand the difference of Mg required. This provides information to understanding feed line scaling and potential feed formation issues.

Unfortunately, NH3-N Hach measurements can be unreliable due to a number of issues. Stoichiometric ally, ammonia removal is equimolar since we are making struvite. A calculation in the attached spreadsheet based on P removal yields approximately 6% NH3-N removal.

The amount of fertilizer produced during this study was lower than expected for the duration of the pilot due to the low concentration of phosphorus in the filtrate. Prior to the WASSTRIP feed, the struvite reaction was incomplete. Once corrected, we completed five harvest events. After subtracting the initial seed volume, the pilot produced 80 pounds (36.3 kg) of struvite. At full scale, performance design should be approximately 2 tons per day.