

Pilot-Scale Treatment of Virginia Canyon Mine Drainage in Idaho Springs, Colorado, USA Using Octolig[®]

by

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Notice

This report is based on pilot scale testing conducted by Arcadis U.S., Inc. (contract EP-C-10-028), with sampling and analyses by EPA Region 8 and Arcadis. The research was funded under the Region 8 Regional Applied Research Effort (RARE) program. The RARE program is a mechanism used by the Office of Research and Development's (ORD) Regional Science Program to respond to high-priority, near-term research needs of EPA's regional offices. The Octolig[®] adsorption technology was proposed by Arcadis in response to a Request for Proposals (RFP). The information presented and the views expressed herein are strictly the opinion of the author and in no manner represent or reflect current or planned policy by the USEPA. Mention of trade names or commercial products does not constitute endorsement or recommendation of use. This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication.

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Cynthia Sonich-Mullin, Director
National Risk Management Research Laboratory

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1 Introduction

1.1 Research Need

Thousands of abandoned mines are scattered throughout the Rocky Mountains in EPA Region 8. The mining activities that disturbed the lands at these sites left exposed metals-laden rock and the interaction of water with these rocks has resulted in acidic rock drainage and other environmental problems. In particular, these waters contain high concentrations of metals and have low pH, each of which serves to damage the aquatic ecosystems and pose a threat to human health. Many of these sites are in remote locations having steep topography, limited access to power supplies, limited land on which to construct a treatment facility, and limited access for maintenance and monitoring.

To combat aquatic ecosystem damages and human health threats, water treatment facilities are in use or are planned at a dozen Superfund cleanup sites in Region 8. These treatment facilities extract heavy metals such that the water will not cause further problems downstream. Most of Region 8's treatment systems are expensive to operate, and they generate large quantities of sludge. For example, the Argo Treatment Facility, located in Idaho Springs, Colorado, generates 10 cubic yards of sludge per day from alkaline chemical treatment of the water. Non-alkaline chemical treatment technologies exist for removal of metals, but few have been implemented in the field specifically for the use of treating acid-mine drainage water. Additionally, few have examined reuse of the metal(s) recovered. To date, only one site has implemented a system to recover metals and reuse them: a sulfide precipitation system (Bioteq Environmental Technologies Inc., <http://bioteq.ca/operations/wellington-oro-co/>) is operated by Breckenridge Colorado's Water Division to treat drainage from the Wellington-Oro mine.

While the primary focus of water treatment is to remove metals from the environment to allow recovery of the affected ecosystem and to reduce the threat to human health, failing to reuse the metals and simply disposing of them represents a missed recycling opportunity, including the potential for recovery of some costs through income generated by recycling, and results in additional mining activities to acquire these metals. The additional mining activities then have the potential to exacerbate the environmental issues that EPA currently is addressing.

Knowledge of the chemical and physical parameters of the water is critical when considering treatment options in mining-influenced watersheds. Typically, pH and alkalinity are low; acidity, hardness and ionic strength are high; and metal (e.g., aluminum and iron) oxyhydroxides precipitate rapidly upon aerobic mixing of the mine drainage with natural

streams having near neutral pH. Water temperature, which typically is a function of both climate and altitude, also is an important variable for many treatment processes.

As well as removing contaminants from the water to meet water quality standards, other technical requirements of treatment systems for remote locations in EPA Region 8 include being compact (e.g., < 54' x 9', size of an 18-wheel trailer), requiring minimal maintenance/monitoring, the ability to function in low temperatures owed to high altitudes, and having a recyclable waste stream . Applicability of a given technology typically depends on the specific site's water quality characteristics (e.g. specific metals and concentrations present), and therefore it is desired to determine technologies that will both remove contaminants to meet water quality standards and produce waste residuals that may be recycled and reused.

Treatment technologies are needed for removing metals from mining-influenced water that would allow for reuse of the metals, while producing less sludge than traditional methods. These capabilities would reduce operation and maintenance (O&M) costs at mines by decreasing the volume of sludge requiring disposal, and potentially offset some costs through an income-generating waste stream. This Region 8 RARE project performed a small scale pilot field study (non-permanent structure) of the Octolig® adsorption technology that was proposed by Arcadis in response to a Request for Proposals (RFP) to address these technology treatment and reuse needs.

1.2 Purpose and Objectives

The purpose of the pilot study was to evaluate the utility of the Octolig® technology at a representative Superfund site in the Clear Creek Watershed, based on assessment of the project objectives. The project was a 6-week field pilot study to extract metals, including aluminum (Al), cadmium (Cd), copper (Cu), iron (Fe), lead (Pb), and zinc (Zn), from mining-impacted water into a form that makes them readily reusable, through either smelting or another recovery process.

Primary Objectives:

1. To meet or exceed (i.e., be better than) site-specific (watershed) water quality criteria for Al, Cd, Cu, Pb, and Zn (see Table 3)
2. To attain > 90% removal efficiencies for Al, Cd, Cu, Fe, Pb, and Zn
3. To produce a minimal waste stream, with attainment of a sludge volume of at least 30% less than traditional treatments, such as with lime
4. To evaluate reuse of the metals recovered via smelting or another process
5. To obtain capital and O&M costs associated with the technology's use at this representative site and a cost estimate for scaling up to 50 and 300 GPM

Secondary Objectives:

1. To determine removal of silver (Ag), arsenic (As), calcium (Ca), potassium (K), magnesium (Mg), manganese (Mn), sodium (Na), nickel (Ni), and selenium (Se)
2. To determine the effectiveness of the regeneration process for the Octolig[®] medium (i.e., evaluating removal over time to see if efficiency of the medium changes)

1.3 Study Site

The Argo Tunnel Water Treatment Plant (Argo WTP) in the Clear Creek Watershed in Idaho Springs, Colorado (Figure 1) treats mine drainage from three sources: the Argo Tunnel, the Big Five Tunnel, and Virginia Canyon.

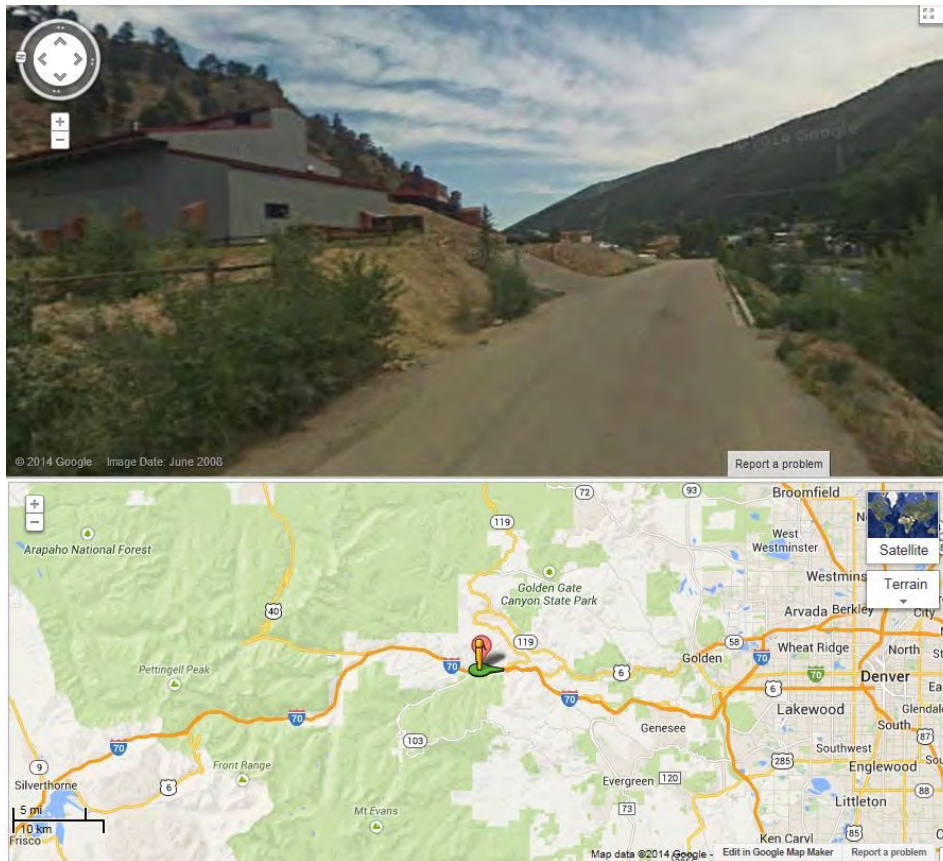


Figure 1. Argo Tunnel Water Treatment Plant location, 2300 Riverdale Drive, Idaho Springs, CO. Figure from <https://maps.google.com/> accessed September 2, 2014. Map data ©2014 Google; image ©2014 Google date: June 2008.

The average chemistries and flows of the three sources, approximated based on historical data, are presented in Table 1 (EPA, 2007). The water from the Argo Tunnel feeds directly into the

WTP equalization basins, and discharges from the Virginia Canyon groundwater and the Big Five Tunnel drain from pipes in front of the Argo Tunnel into the equalization basins (EPA, 2007). Flows from the Big Five Tunnel and Virginia Canyon can be controlled.

Table 1. Average chemistries of the drainages from the Argo Tunnel, the Big Five Tunnel, and Virginia Canyon (modified from U.S. EPA, 2007).

Parameter	Argo Tunnel	Big Five Tunnel	Virginia Canyon
Aluminum (mg/l)	20	5	80
Copper (mg/l)	4	1	9
Iron (mg/l)	120	65	3
Manganese (mg/l)	90	30	90
Zinc (mg/l)	40	8	92
pH	3	5.5	3
Average flow (GPM)	200 to 450	15 to 40	5 to 180

The photos in Figures 2 and 3 show the collection areas of these drainages prior to the water being treated at the Argo WTP.



Figure 2. Virginia Canyon (1st and 2nd from left) and Big Five Tunnel (far right) outfalls. Photo by Arcadis.



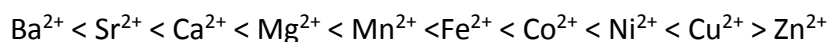
Figure 3. Argo Tunnel on right, sump region in lower left, and Big Five Tunnel and Virginia Canyon discharges in background left. Photo by Arcadis.

During the preliminary stages of the project (see Section 2.1), it was noted that precipitation began as soon as these three water sources mixed; therefore, it was decided to use water from a single source. The Virginia Canyon drainage was chosen primarily due to it having the lowest relative concentration of Fe and the highest relative concentration of Cu, which currently has the highest value of the metals present in the drainages.

1.4 Process Tested

Octolig[®] is a pH-responsive immobilized ligand that has a strong affinity for heavy metals. The technology is based on the chelation of heavy metals using a branched polyethyleneimine (BPEI) ligand that has been bonded to the surface of a silica gel with a silane linking group (Lindoy 1993; 1999). Figure 4 shows an example of the BPEI structure from Lindoy et al. (1999) and Figure 5 is a schematic diagram showing attachment of BPEI to silica gel to form Octolig[®]. Octolig[®] retains high amounts of metal ions from low concentration solutions in a pH range from 2 to 10. An acidic solution is used to release and concentrate the ions, which regenerates the Octolig[®] for further metals removal.

It is believed that the ligand selectivity of Octolig[®] follows the Hard Soft Acid Base (HSAB) principle – i.e., that a metal ion is a Lewis acid and the Octolig[®] ligand is a Lewis base. Octolig[®] is a soft base comprising primary, secondary, and tertiary amines (Figure 4). In the Irving-Williams series for select alkaline earths and divalent transition metals, the following trend in strength of complexation is observed:



The proton and metal-binding stability constants for Octolig[®] have been provided for a single deprotonation reaction only in the form of a conference presentation (Table 2), and thus have not been subjected to external technical peer review; however, the reported constants generally follow the Irving Williams series.

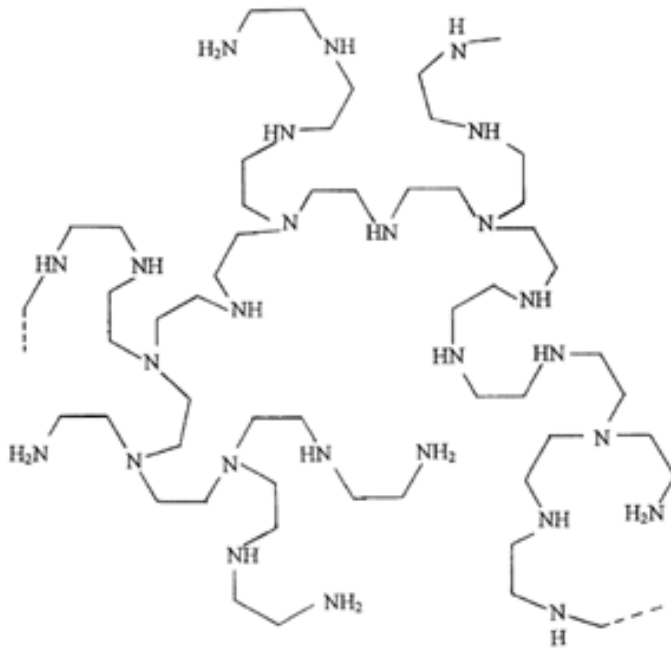


Figure 4. Branched polyethyleneimine (BPEI) structure (copied from Lindoy et al, 1999).

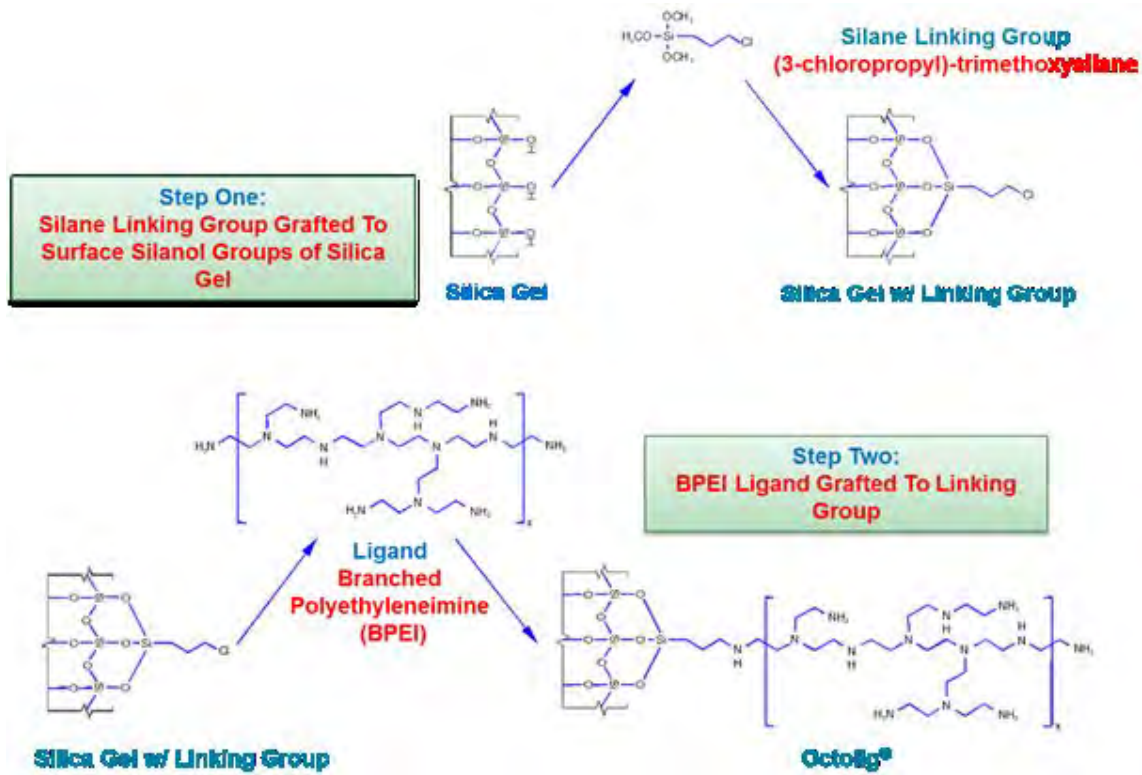


Figure 5. Schematic diagram of Octolig® showing attachment of BPEI to silica gel. Figure prepared by Jon Forbort, Arcadis, based on Lindoy et al., 1999.

Table 2. Stability constants for Octolig® (Martin, 2010)¹

Cation	Equilibrium Species	Log K
H ⁺	HL	10.2
Cd ²⁺	ML	None provided
Co ²⁺	ML	15.6
Cu ²⁺	ML	22.4 (24)
Fe ²⁺	ML	11.1
Mn ²⁺	ML	None provided
Ni ²⁺	ML	19.1
Zn ²⁺	ML	16.2

¹ ACS presentation not subjected to external technical peer review

Octolig® has been shown to take up arsenate, chromate, polymolybdate, selenious acid and fluoride, but not boric acid, in individual anion bench tests (Martin, 2010). Although nitrate and sulfate apparently do not interfere with the uptake of metals by Octolig®, since initial bench scale tests for patent development used metal salts having these forms (Lindoy et al., 1993; 1999), in a mixture study Stull and Martin (2009) observed removal of nitrate and sulfate from solution, as well as phosphate and nitrite. The mechanism proposed for uptake of anions by Octolig® is one of encapsulation (Stull and Martin, 2009; Martin et al., 2010), with a weak association between multiple protons on the nitrogen atoms of the ethyleneamine chain and the anion. This encapsulation likely is a weaker association than the metal ligand complexes by which the Octolig® is proposed to bind reversibly with metals (Lindoy, 1993).

2 Methods

2.1 Preliminary Studies

Preliminary testing of the Octolig® media was conducted by the supplier: EPS of Hebron, KY, subcontractor to Arcadis. Water was collected from the combined water source at the Argo Tunnel site in June 2010 and shipped to EPS's testing facility in Cincinnati, OH. The mixed water had a nominal pH of 2.5 and was saturated with Fe³⁺, which resulted in the precipitation of large amounts of ferric hydroxide when sodium hydroxide was added to raise the pH (ferric hydroxide precipitates at pH > 3.5). Because it was desired to evaluate the Octolig® technology for its ability to remove metals through chelation, rather than through precipitation and/or adsorption of ions onto precipitates in the pH modifying steps, this source water was deemed inappropriate to meet the project goals. Instead, the Virginia Canyon water source was chosen for use due to it having the lowest historical average Fe concentration (3 mg/l, Table 1) and the

highest historical average concentrations of both Cu and Zn (9 and 92 mg/l, respectively, Table 1).

Aluminum hydroxide precipitates at $\text{pH} > 4.5$. The Virginia Canyon water contains an average of 80 mg/l of Al (Table 1) and a white precipitate, assumed to be aluminum hydroxide mixed with some ferric hydroxide (solid phase was not tested for positive identification), was observed to begin to form when the pH was adjusted to 4.3. These results indicate that careful control of pH is necessary to maximize sequestration of metals by the media rather than by sorption onto any aluminum and/or iron hydroxides that might form during the pH adjustment stage of the process prior to the water being run through the Octolig[®] media.

To allow for pilot system sizing, time for regeneration, and pH set-points, tests were conducted to determine the mass transfer zone, determine the breakthrough for major ions in the water, and to determine the media capacity at anticipated field pilot conditions. These preliminary tests indicated that Fe and Cu were successfully removed at low pH (~ 4) and that Al, Cd, Cr, Co, Ni, and Zn were removed at higher pH (~ 6), which supported the use of a two-stage design for pilot testing.

2.2 Field Pilot Test

The desire was for the pilot treatment system to meet the objectives (see Section 1.2), including producing a product that had the potential to be processed to recover the metals sequestered. The approach used was to treat the water in two treatment beds having differing pH values to selectively remove the metals based on their affinity toward the Octolig[®]. The first bed of Octolig[®] was designed to operate at pH 3.5 – 4, followed by pH adjustment to pH 6.0 – 8.0, and then by an additional bed of Octolig[®] designed to operate at pH 6.0 – 8.0. These pH ranges were chosen due to the following:

- Minimum pH values at which metal sorption was expected with Octolig[®], based on bench-scale testing
- The magnitude of changes in pH values expected to result in differing metals sorption for divalent cations
- They were below pH values expected to lead to the formation of hydroxide precipitates (other than Fe and Al)
- pH values that would not require neutralization prior to being discharged

This section describes the system design, water treatment configuration, and water sampling locations and methods. Standard operating procedures (SOPs) for the system in both the water treatment and regeneration modes are provided in Appendix A; major equipment and parts are listed in Appendix B; figures showing the process flow diagram and spatial arrangement within the trailer are provided in Appendix C; and notes regarding system deployment and operations are provided in Appendix D.

2.2.1 System Design

In Appendix C, Figure C1 presents the process flow diagram (the water treatment configuration of this diagram is described in Section 2.2.2) and Figure C2 presents a visualization of the spatial arrangement of the equipment within an 18-wheel trailer, shown in 2 orientations. Equipment located outside the trailer included the water conveyance lines, a 2,300 gallon clarifier tank (T23), and bulk chemicals stored in a small shed (not shown in Figure C2). Supplies needed during testing were moved into the trailer in smaller portable containers.

2.2.2 Water Treatment Configuration

This paragraph describes the water treatment process flow diagramed in Figure C1 (the sampling ports and associated tanks are shown in Figure 6). The pilot began March 26, 2012 and ended May 7, 2012, with the first day being dedicated to filling the 1st tank with sufficient source water to begin running the treatment system. Raw influent water from Virginia Canyon was gravity fed into the 1st stage pH-adjustment tank (T10). Flow was controlled by a mechanically-actuated float valve. Using a dosing pump, 25% NaOH solution (on and after April 27, a 50% solution was used) was added to the water in Tank T10 mixed with a mixing pump) to adjust the pH with a set-point of pH 4.0, before being directed to Tank T13 (1st stage feed tank) and then fed into the 1st stage Octolig[®] treatment tank (T18), with flow rates ranging from 4.2 to 15.2 GPM over the testing time. The Octolig[®] treatment tank was operated in upflow mode. From Tank T18, the water was conveyed to the 2nd stage pH-adjustment tank (T20), with a set-point of pH 8.0 and mixed with a mixing pump. Overflow from Tank T20 was conveyed to the clarifier settling tank (T23). Overflow from Tank T23 flowed to the 2nd stage feed tank (T24). Water then was pumped (flow ranged from 4.2 to 15.3 GPM over the testing time) to the 2nd stage Octolig[®] treatment tank (T25), which was operated in upflow mode. Following treatment in Tank T25, the water was conveyed to the effluent clear-well tank (T11). Effluent from Tank T11 was pumped back to the headworks of the Argo WTP.

During the field pilot-scale test, the system was taken off line for media regeneration. In a larger scaled operation, parallel beds would be used to perform regeneration without causing downtime. Scheduling of regeneration was based on the results from the bench testing and

attempted to balance maximum loading onto Octolig® with maximum water treatment efficiency. Also considered was the desire to conduct this part of the process and restore the system to operation within the available time on site.

Field Zn testing suggested breakthrough from the 1st Octolig® bed on April 18, but testing on April 20 and 23 suggested resumed Zn removal. Due to both time constraints and field Zn results, the 1st Octolig® bed was regenerated April 24-26. Both the 1st and 2nd Octolig® beds were regenerated at the end of the demonstration (May 7-8). A pH 1.8-2.2 solution of sulfuric acid (H₂SO₄) was used to regenerate the 1st Octolig® bed (Tank T18) and a H₂SO₄ solution with pH 3.8-4.2 was used for the 2nd Octolig® bed (Tank T25). Sulfuric acid was used due to the potential for recycling of the regenerant via hydrometallurgical processing (see Section 3.5). After the first bed was regenerated in April, the pH of the bed was adjusted back to the operating pH (4.0) before water treatment resumed. Steps for regeneration are included in Appendix A.

2.2.3 System Control

Zn was monitored using a HACH colorimetric test (www.hach.com, pocket colorimeter II) to monitor real-time system performance. Zn was chosen because it showed moderately weak binding to Octolig® in preliminary work and was present at high concentration in the source water. Mn, Fe, and Cu concentrations also were monitored using field HACH kits (www.hach.com, pocket colorimeter II) to evaluate system performance, along with monitoring of pH.

2.2.4 Sampling and Analysis

Figure 6 is a simplified schematic of the tanks and water quality sampling ports from Figure C1 relative to the two stages of the system.

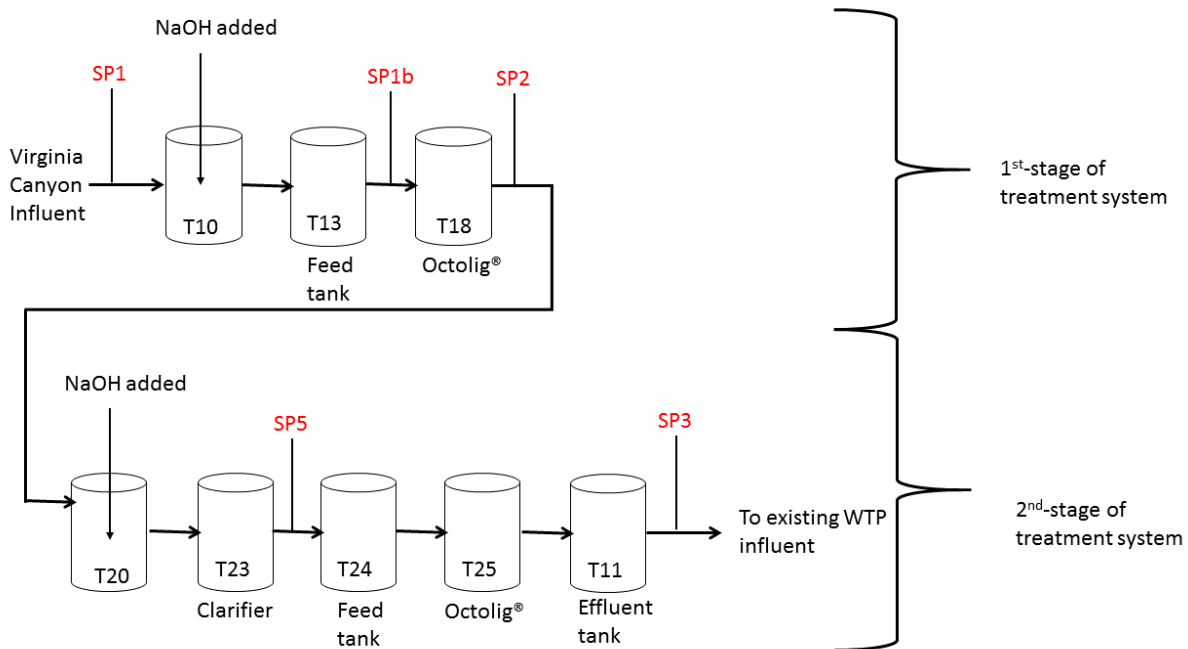


Figure 6. Schematic diagram showing tanks and sampling points (in red) through the treatment system.

Grab samples were collected by both EPA and Arcadis to attempt a greater temporal coverage with the limited funding for the project. EPA collected grab samples on 4/5, 4/9, 4/16, 4/20, 4/30, and 5/3 from sampling ports SP1, SP2, and SP3. QA duplicates were collected on 4/9 and 4/30. To evaluate any removal of metals through precipitation and/or adsorption to precipitates following the pH-modification steps, versus removal by Octolig[®], EPA collected additional samples at locations SP1b and SP5 on 4/5, 4/16, and 5/3.

Arcadis collected grab samples at SP1 and SP3 on 4/7, 4/9, 4/11, 4/16, 4/18, 4/27, 4/30, and 5/4; and at SP2 on the same dates, except 4/27 and 4/30. Regenerant solutions and sludge were sampled also by Arcadis. Composite regenerant samples were collected from three depths from Tanks T26 (1st stage regeneration tank) and T28 (second stage regeneration tank) on 5/8 and the remaining solution was blended back into the water stream to be treated by the Argo WTP. Tanks T26 and T28 are shown in Figure C1. A sludge sample was collected from Tank T23 on 5/4; sludge not needed for analyses was disposed of within the main Argo WTP's routine sludge management system by a manually controlled pump.

EPA-collected samples were analyzed at the EPA Region 8 laboratory in Golden, Colorado for chloride, fluoride, and sulfate (EPA 300.0), total recoverable and dissolved Al, Cu, Fe, Mn, Ni, and Zn and dissolved Ca, K, Mg, and Na by ICP-OES (EPA 200.7/6010); and total and dissolved

Ag, As, Cd, Pb, and Se by ICP-MS (EPA 200.8/6020). Dissolved ions were measured on samples that had been filtered in the field at 0.45 µm. Field parameters measured included pH, specific conductance (SC), and temperature. Alkalinity (EPA 310.1) and hardness (SM 2340B) were measured and calculated, respectively, in the laboratory. Hardness was calculated using the following equation: CaCO₃

$$\text{Hardness (mg/l as CaCO}_3\text{)} = [2.497 \times \text{Ca}^{2+} (\mu\text{g/l}) + 4.18 \times \text{Mg}^{2+} (\mu\text{g/l})]/1000$$

Arcadis-collected samples were analyzed by Test America in Denver, Colorado for total recoverable Ag, Al, As, Ca, Cd, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Se, and Zn (EPA 6010C). Parameters measured on grab samples collected by Arcadis included pH (field), specific conductance (SC), and total suspended solids (TSS). Sludge depth was measured in Tank T23 and samples collected were analyzed for TSS, and total metals analysis (EPA 3050/6010C).

2.2.5 Quality Assurance / Quality Control

EPA field duplicate samples (collected within 10 minutes of one another) were compared using relative percent difference (RPD). EPA and Arcadis each collected samples on April 9, 16, and 30; while these samples can't be considered as split samples, they were collected within two hours from one another and their RPDs were compared to determine the applicability of using total recoverable (dissolved analysis was not conducted on Arcadis-collected samples) data from both labs for analytes measured using similar analytical techniques (i.e., total Al, Ca, Cu, Fe, Mg, Mn, Ni, K, Na, and Zn measured by ICP-AES) to evaluate system removal efficiency over time.

Calculation of RPD: $\%RPD = 100 * (X_1 - X_2) / [(X_1 + X_2) / 2]$ where X₁ = value from replicate 1; X₂ = value from replicate 2.

Analytical methods included the use of laboratory method blanks, control standards and lab duplicates, matrix spikes and duplicates, and dilution series samples.

2.2.6 Data Analysis

Data were provided by EPA Region 8 laboratory in both Excel and Adobe PDF formats; data were transposed to another Excel spreadsheet for data analysis and presentation of results. Test America provided data in Adobe PDF format, which also was transposed into Excel spreadsheets for data analysis and presentation of results.

Observed effluent (SP3) concentrations of Al, Cd, Cu, Fe, Pb, Mn, and Zn (all but Fe and Mn were target metals for Primary Objective 1) were compared to available acute and chronic

water quality criteria (WQC) to determine if the concentrations in the effluent from the treatment process were equal to or less than the WQC values. State of Colorado criteria for the South Platte River Basin (Colorado State Regulation 38) and site-specific WQC for the Clear Creek Watershed segment where Argo is located and downstream (Segment 11 of the Clear Creek Basin in the Colorado State Regulation 38) are provided in Table 3. There is no state or site-specific criterion for Al; therefore, the National WQC is included in Table 3 and was used for comparison. Some metals have criteria based on average water hardness. Tim Steele of TDS Consulting [personal communication July 29, 2010] provided an average high-flow hardness value of 56.5 mg/l as CaCO₃ for calculations of criteria in this stream segment¹.

Influent (untreated mine-drainage: SP1) and effluent (following all treatment steps: SP3) samples were compared to determine the percentage removal (Primary Objective 2) for target metals of interest (Al, Cd, Cu, Fe, Pb, and Zn). The equation used was: $100 \times (\text{Effluent} - \text{Influent}) / (\text{Influent})$. This was done also for Ag, As, Ca, K, Na, Ni, Mg, Mn, and Se to evaluate Secondary Objective 1. Additionally, removal of ions was compared between SP1 and SP1b, SP1b and SP2, SP2 and SP5, and SP5 and SP3 for the three days the additional sites (SP1b and SP5) were sampled to evaluate the potential for removal by precipitation and/or adsorption processes resulting from pH-modification.

¹ When asked what hardness value was appropriate for use at this site, Mary Boardman of the Colorado Department of Public Health & Environment (CDPHE) suggested we contact Tim Steele, who has done work in this watershed for many years. Although a footnote to the equations states the hardness value should be “*based on the lower 95 per cent confidence limit of the mean hardness value at the periodic low flow criteria as determined from a regression analysis of site-specific data*” (Colorado State Regulation 38), Tim Steele stated that the average value should be used instead [July 29, 2010].

Table 3. Water quality criteria.

Metal	Water Quality Criterion (ug/l)		
	State of Colorado ¹	Colorado Site-specific ¹	National ²
Aluminum (ac) trec	none given	none given	750
Aluminum (ch) trec	none given	none given	87
Cadmium (ac)	1.7	none given	
Cadmium (ch)	0.3	1.42	
Copper (ac)	8	none given	
Copper (ch)	5	17	
Iron (ch) diss	none given	300	
Iron (ch) trec	1000	1000	
Lead (ac)	35	35	
Lead (ch)	1.3	1.3	
Manganese (ac)	2469	2469	
Manganese (ch)	1364	50	
Zinc (ac)	88	215	
Zinc (ch)	76	187	

ac = acute; ch = chronic; trec = total recoverable

Hardness-based values were calculated using the seasonal high-flow average hardness value (56.5 mg/l as CaCO₃, n=263); non-hardness based values are highlighted in gray

Site-specific Cd (ch) is a temporary modification to expire 7/1/15

For metals without a state or site-specific criterion, the national value is presented

¹ <http://www.colorado.gov/cs/Satellite?c=Page&childpagename=CDPHE-Main%2FCBONLayout&cid=1251595703337&pagename=CBONWrapper> (Site-specific is Segment 11 of the Clear Creek Basin)

² <http://water.epa.gov/scitech/swguidance/waterquality/standards/current/index.cfm>

3 Results and Discussion

3.1 Quality Assurance

3.1.1 Field Replicate Samples

Only flouride and silver in the replicated filtered samples for SP1 collected on April 9 exceeded 20% RPD, with values of 27% and 33%, respectively. The RPD values for the majority of analytes for all other sampling dates and all locations were less than 5%. The values for Ag in samples collected 4/9 were 0.5 and 0.7 µg/l, which are close to the reporting limit of 0.5 µg/l; therefore it is likely the high RPD for the field replicates is related to their values being low. The first replicate for each sampling date was used for subsequent analyses and evaluation of objectives.

3.1.2 Analytical QA

3.1.2.1 EPA data

Values for total Ag in SP1 samples for sampling dates 4/5 and 4/9 and SP3 collected 4/5 were not used because Ag was found in the blank at concentrations exceeding those in the samples; these are flagged with a “B” and data are not reported or used. Total Ag values for SP1 samples collected 4/16 and 4/20 and SP1b and SP5 collected on 4/16 were not used because control sample criteria were not met. The RPD for the laboratory duplicate sample for Ag was 23.3% for filtered SP1 samples collected 4/5 and 4/9; this slightly exceeded the 20% criterion and the samples are flagged as estimates. The RPD limit of 10% was slightly exceeded (10.4%) in the serial dilution for the total Cd concentration on the sample from SP1 collected on 4/16. Because this deviation was small, the sample result is reported, but flagged as an estimate. The matrix spike criterion was not met for fluoride (F) in SP1 collected on 4/20 and the sample result is flagged as an estimate. K, Mg, Ni, and Zn serial dilution results for SP2 collected on 4/16 exceeded the RPD criteria of 10% with values of 14.4, 11.6, 10.3, and 16.1%, respectively; these analyte results are flagged as estimates. No other QA issues were observed for the EPA data.

3.1.2.2 Test America data (Arcadis-collected)

Cu concentration in the laboratory blank for samples collected 4/16, 4/18, and 5/4 exceeded the method detection limit (MDL), but was below the reporting limit (RL) and was present at concentrations below 10% of the measured sample concentrations; therefore, samples were considered usable. Fe, Mn, and Na concentrations were identified in the laboratory blank for samples collected on 5/4, but concentrations were much less than 10% of the values measured in the samples and the samples were considered usable. As, Cd, Pb, and Ni failed the matrix spike and/or matrix spike duplicates for the SP1 sample collected 4/7. Batch matrix spikes conducted by Test America in subsequent analytical runs did not capture the Arcadis-collected samples from SP1, SP2, or SP3; therefore, it is not possible to determine if the observed negative matrix effect was evident in any other sample and/or sampling date. As, Cd, and Pb were measured by ICP-OES by Test America and by ICP-MS by EPA and therefore are not comparable; results from the more sensitive ICP-MS method were used for data analysis. No other QA issues were observed for the Test America data.

3.1.3 Comparisons of Data between Labs

Ca, Mg, Na, and K are conservative ions generally found in the dissolved phase in water samples. EPA measured these ions on filtered samples and Test America measured these ions

on un-filtered samples. Because they were expected to be predominantly in the dissolved phase, the results from the unfiltered and filtered samples were compared. The RPDs for Ca and Mg were < 5% for SP1, SP2, and SP3 samples from 4/9 and 4/16 and the RPDs for K and Na ranged from 6 to 21% for the same samples. All but Mg, Mn, and Zn exceeded 20% RPD in the comparison between analytes measured via ICP-OES (Al, Ca, Cu, Fe, Mg, Mn, Ni, K, Na, and Zn) on samples collected on 4/30 for SP1 and SP3; SP2 was not sampled by Arcadis on 4/30 so could not be compared. Except for K and Fe, analytes in SP1, SP2, and SP3 samples collected on 4/9 and 4/16 compared well, with RPD values < 20%; however, because the majority of analytes exceeded 20% RPD on 4/30 for both the SP1 and SP3 samples, the majority of analytes compared across 4/9, 4/16, and 4/30 appear biased low compared to concentrations in the EPA samples, and the reason for these deviations is not known, it was decided to exclude the Test America ICP-OES data for the water samples. Exclusion of these data did not alter the outcomes from assessment of project objectives or conclusions made.

3.2 Water Samples

Field parameters and water quality results for each sampling port are presented in Appendix E. Sampling ports and their corresponding locations are described below and shown in Figure 6; all but sampling point SP1b are shown also in Figure C1:

- SP1 = Virginia Canyon raw influent, pre-pH adjustment in Tank T10
- SP1b = effluent from the 1st-stage pH adjustment, prior to 1st-stage Octolig® treatment, port located on Tank T13
- SP2 = effluent from the 1st-stage Octolig® treatment, port located on Tank T18
- SP5 = effluent from 2nd-stage pH adjustment, prior to 2nd-stage Octolig® treatment, port located on Tank T23
- SP3 = effluent from the 2nd-stage Octolig® treatment, port located on Tank T11

3.2.1 Parameters

TSS was measured by Arcadis for a few of the sampling dates (Appendix E). TSS was below detection in SP1 samples analyzed and ranged from 2 to 8 mg/l at SP2 and from 8.8 to 18 mg/l at SP3. This increase through the system likely is due to the transport of unsettled precipitates formed from addition of NaOH.

Hardness remained essentially constant between about 1,050 to 1,100 mg/l as CaCO₃ through the 1st-stage of the treatment system (SP1 through SP2, Figure 7). At SP5, hardness was 947

mg/l as CaCO₃ on 4/5, but returned to ~ 1,000 mg/l as CaCO₃ for the other sampling events. Hardness at SP3 was ~ 900 mg/l as CaCO₃ for four of the six sampling events and was lower than the corresponding influent concentrations on all sampling dates. That hardness decreased below the influent concentrations only at SP5 and SP3 suggests that removal of Ca and Mg occurred predominantly in the 2nd-stage of the treatment system.

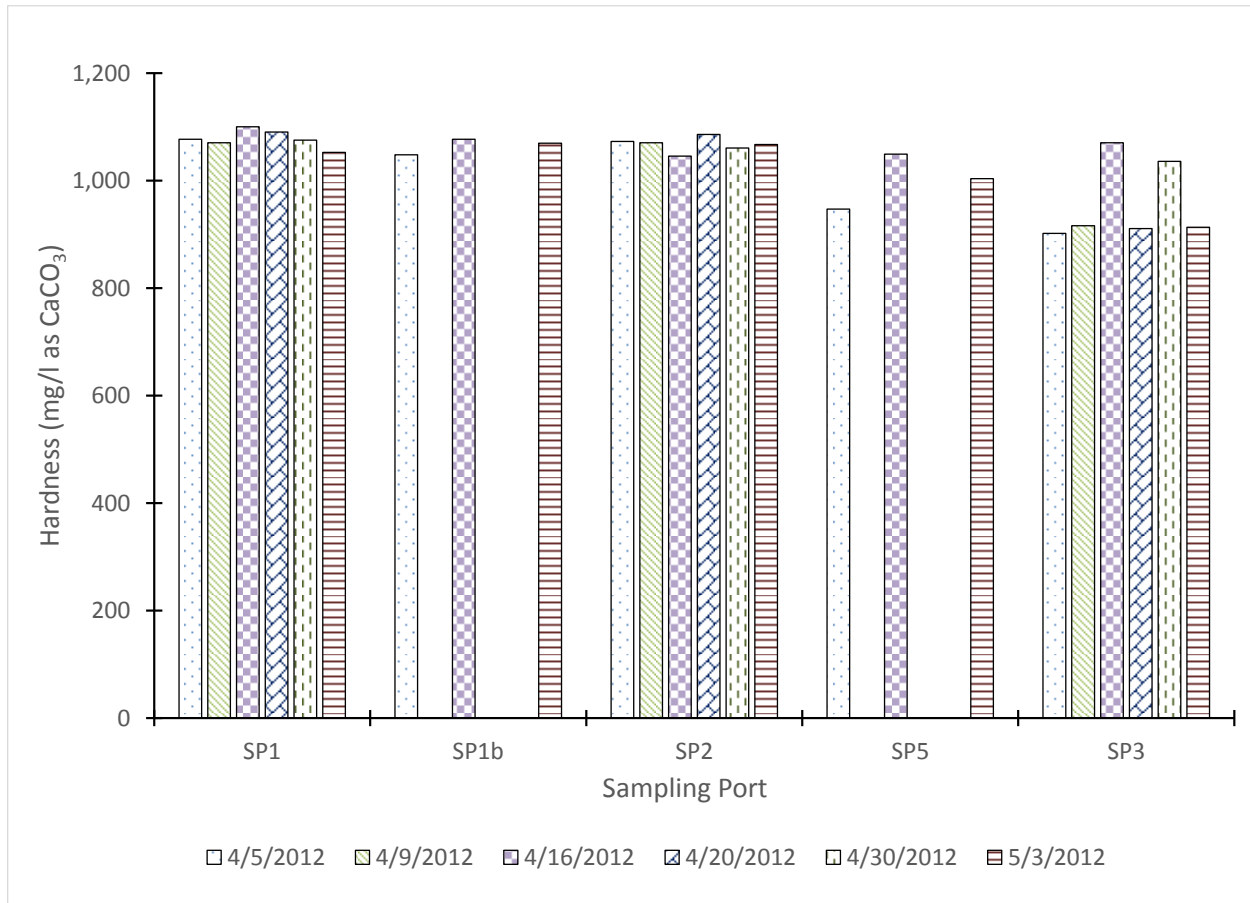


Figure 7. Hardness through the treatment system over time.

While total dissolved solids (TDS) is not a water quality parameter in the watershed where the pilot was conducted, and its assessment was not an objective in this work, whether the process removes or adds TDS is of interest for potential use of the treatment at other sites in EPA Region 8 where there might be a TDS limit. TDS was calculated from dissolved concentrations of ions measured (Appendix E) and concentrations are plotted in Figure 8. TDS concentration remained essentially constant through the 1st-stage of the system, with the exception of decreases observed at SP2 on 4/5 and 4/16, and a smaller (relatively) decrease observed on 4/9. Over the entire treatment system, TDS concentration was decreased on 4/5 and 4/9, but was similar to, or higher than, the influent concentration for the remainder of the study. The

higher TDS concentration observed at SP3 on 5/3 likely is due to the sulfate concentration being higher than was typically observed (see Section 3.2.7 and Appendix E).

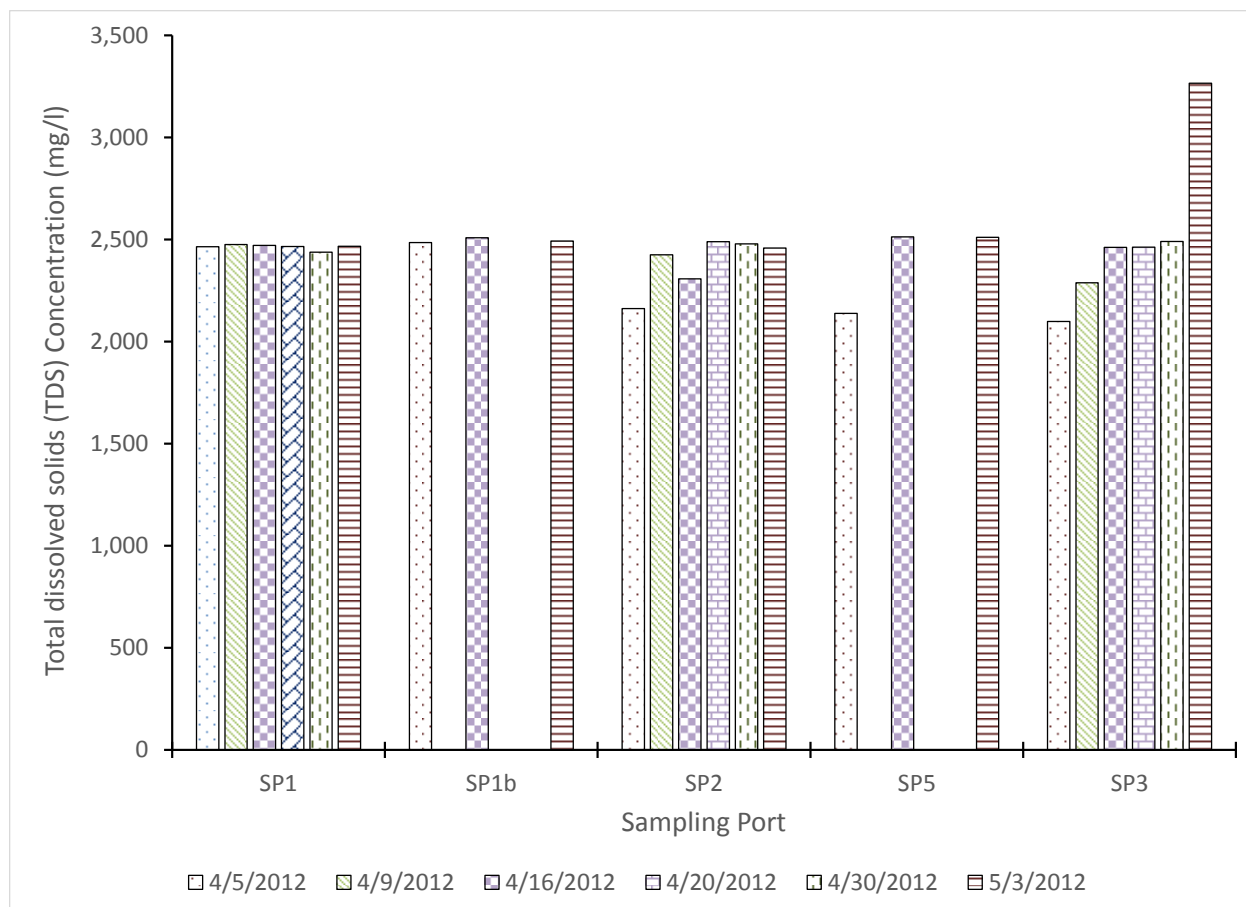


Figure 8. Calculated total dissolved solids (TDS) through the treatment system over time.

The pH was measured in the field by Arcadis when collecting samples for metals analysis and for monitoring system performance. Some pH data from system performance checks were provided to EPA, with other pH data discussed in operations notes (see spring 2012 notes in Appendix D). EPA measured pH on dates when water samples were collected. Results are plotted in Figure 9 for EPA-measured pH values on dates when EPA collected water samples and results from Arcadis are plotted for dates when EPA did not collect samples and for dates when pH was monitored (and provided) without any sample collection.

The higher pH observed at SP2 (following the 1st-stage Octolig[®] treatment) versus SP1b (pH set-point 4.0) for measurements obtained from 4/5 to 4/16 is believed to be due to pH buffering effects of the Octolig[®], which was experienced in bench scale testing by EPS (subcontractor for Arcadis), as well as during testing by MSE (1998) using Berkley Pit mine water. Being an intermediate sampling location, the pH at SP1b was not measured on all dates,

but this phenomenon was not observed in measurements on 4/27 or 5/3. This increase in pH was not seen in the data for the 2nd-stage of the treatment, however, except for the sample measured on 4/23 when the pH was 4.51 at SP5 and 4.85 at SP3.

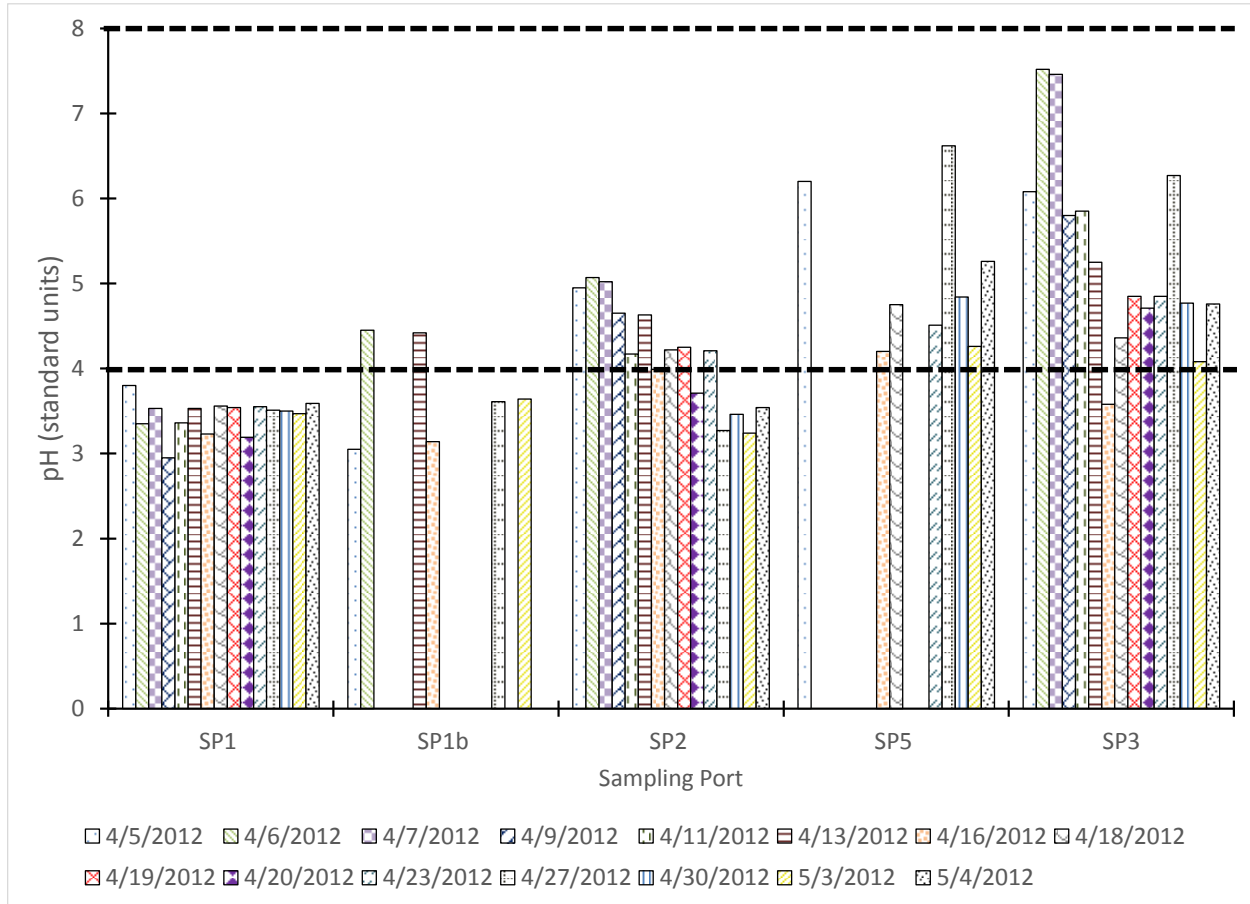


Figure 9. pH through the treatment system over time. EPA data are plotted for 4/5, 4/9, 4/16, 4/20, and 5/30; all other data were provided by Arcadis. The dashed lines represent the pH set-points (4.0 for the 1st-stage pH dosing and 8.0 for the 2nd-stage pH dosing).

The pH at SP2 was above the 1st-stage pH set-point for most of the study, except when it dropped to 3.7 on 4/20 and at the end of the study (4/27 and beyond). Based on the monitoring results at the intermediate sampling location SP1b, the pH in the 1st-stage feed tank (T13) did not reach the pH set-point of 4.0 after April 13th. On May 2nd, an overheating failure of the 1st-stage base pump was diagnosed, but field repair efforts did not succeed in returning the pump to service before the scheduled end of the demonstration (Appendix D).

The pH in the 2nd-stage of treatment never met the set-point of 8.0. The April 5 and 9 samples from SP3 and the April 5 sample from SP5 were the only samples having measurable alkalinity, which correspond to pH values above 6. The pH at SP3 was 7.52, 7.46, and 6.27 on April 6, 7,

and 27, as measured by Arcadis in the field; however, alkalinity was not measured on those dates.

3.2.2 Comparison to Water Quality Criteria (Primary Objective 1)

Observed effluent (SP3) concentrations of Al, Cd, Cu, Fe, Pb, Mn, and Zn (all but Fe and Mn were target metals for Primary Objective 1) were compared to available acute and chronic WQC to determine if the treatment process met or exceeded them (i.e., concentrations were less than the WQC values). The comparison is presented in Table 4. Unless otherwise noted, the comparison is based on dissolved concentrations.

Table 4. Comparison of effluent (SP3) concentrations with water quality criteria (WQC); gray highlighted cells indicate data meeting the WQC. Based on dissolved concentrations, except where noted for Al and Fe.

Metal	Criterion (µg/l)	Concentration in treatment system effluent (µg/l)					
		Date					
		4/5/2012	4/9/2012	4/16/2012	4/20/2012	4/30/2012	5/3/2012
Al (ac) trec	750 ^N	255	1460	70400	15600	39500	25800
Al (ch) trec	87 ^N	255	1460	70400	15600	39500	25800
Cd (ac)	1.7 ^{H, CO}	54.5	163	321	247	309	233
Cd (ch)	1.42 ^H	54.5	163	321	247	309	233
Cu (ac)	8.0 ^{H, CO}	524	109	2470	1150	2430	2470
Cu (ch)	5.0 ^{H, CO}	524	109	2470	1150	2430	2470
Cu (ch)	17	524	109	2470	1150	2430	2470
Fe (ch) diss	300	< 100	< 100	687	< 100	< 100	< 100
Fe (ch) trec	1000	< 100	< 100	744	116	379	286
Mn (ac)	2469 ^H	27600	33200	76700	53100	68600	52900
Mn (ch)	50	27600	33200	76700	53100	68600	52900
Pb (ac)	35 ^H	< 1	< 1	18.5	< 1	1.3	1.1
Pb (ch)	1.3 ^H	< 1	< 1	18.5	< 1	1.3	1.1
Zn (ac)	215 ^H	2880	14300	74500	46500	68000	49300
Zn (ch)	187 ^H	2880	14300	74500	46500	68000	49300

ac = acute; ch = chronic; trec = total recoverable; diss = dissolved
H = hardness-based; N = national standard; CO = state standard

Chronic criteria are lower than acute criteria because consideration is taken for the period over which an organism is exposed to a lower concentration, while acute toxicity occurs over a short time from exposure to a higher concentration. There is no site-specific acute criterion for Cu, only a numeric chronic criterion (17 µg/l), which is higher than the state's hardness-based acute and chronic criteria (8 and 5 µg/l, respectively; see also Table 3). Both the state and site-specific chronic criteria are compared to the treated effluent Cu concentrations in Table 4.

Concentrations of dissolved and total-recoverable Fe, and dissolved Pb, sometimes were below the reporting limit. In these cases, the reporting limit was compared to the water quality criteria values to determine if the effluent met the WQC.

Only the total-recoverable Fe chronic criterion (1 mg/l) and the acute Pb criterion were met on all dates sampled (note: the Pb concentration in the Virginia Canyon drainage was lower than the acute Pb criterion). Dissolved concentrations of Fe and Pb met the respective chronic criteria (300 and 1.3 µg/l, respectively) on all days sampled except April 16. The total-recoverable Al concentration met the acute criterion only on the first sampling date, April 5. No other WQC were met.

3.2.3 Removal efficiencies for target metals (Primary Objective 2)

To evaluate whether greater than 90% of the primary metals of interest (Al, Cd, Cu, Fe, Pb, and Zn) were removed in the treatment process, the effluent concentrations for each date were compared to the influent concentrations using the following formula:

Percentage Removal (%) = 100 * (Concentration in SP1 - Concentration in SP3) / Concentration in SP1.

Table 5. Comparison of effluent (SP3) concentrations with influent (SP1) concentrations for primary target metals. Gray highlighted cells indicate data meeting the desired 90% removal.

Analyte / Parameter	Date					
	4/5/12	4/9/12	4/16/12	4/20/12	4/30/12	5/3/12
Al - Total (µg/l)	99.6	97.8	3.2	77.8	48.3	62.7
Al - Dissolved (µg/l)	99.9 ^A	99.8	1.8	86.8	52.4	68.3
Cd - Total (µg/l)	81.9	45.9	-6 ^E	18.9	1.5	22.9
Cd - Dissolved (µg/l)	82.1	47.6	-4.9	18.5	5.2	25.3
Cu - Total (µg/l)	93.5	98.7	72.1	85.6	71.0	69.3
Cu - Dissolved (µg/l)	93.6	98.7	72.0	87.2	72.3	70.3
Fe - Total (µg/l)	95.7 ^A	95.9 ^A	70.4	95.5	88.0	90.8
Fe - Dissolved (µg/l)	95.6 ^A	96.0 ^A	73.3	96.3 ^A	96.6 ^A	94.0 ^A
Pb - Total (µg/l)	94.4 ^A	94.4	-1.6	90.7	81.9	83.3
Pb - Dissolved (µg/l)	94.1 ^A	94.2	0.5	94.9 ^A	93.3	92.5
Zn - Total (µg/l)	95.8	76.0	2.7	32.1	9.1	25.4
Zn - Dissolved (µg/l)	96.1	79.8	3.2	37.8	7.9	31.2

A = effluent sample below RL, value for RL used for calculations to indicate that removal is at least to the RL

E = QA criterion not met for either influent or effluent, value is an estimate

Percentage removal results are provided in Table 5; negative values indicate that the concentration in the effluent (SP3) was greater than the concentration in the influent (SP1).

Greater than 90% removal of all metals, except for Cd, was observed on the first sampling date. Cd removal was never greater than the 82% observed on the first sampling date. Zn removal exceeded 90% only during the first week of sampling. Only dissolved Fe and Pb removal remained above 90% for the duration of the study.

For most dates sampled, several ions were present at higher concentrations at downstream ports in the system relative to their concentrations in the immediate upstream samples (Appendix E). These increases generally were less than 10%, which is within the variability that could be expected in a continuously flowing system when times between sample collections might not match residence times, or could be due to impurities in the NaOH used for pH-adjustment. The concentration of Zn on 4/9/12, however, was 46% (total) and 52% (dissolved) higher at SP2 than at SP1. The reason for these larger differences is not known, but possible explanations include desorption from the 1st-stage Octolig[®] bed due to pH differences over time with preferential sequestration of Cu or another ion, or to leaching of Zn from some system part.

Removal efficiencies observed for April 16 show the system was not working well for ions other than Cu and Fe, although their removal was lower (~ 70%) than at the start of the study and lower than what was desired for project objectives (> 90%). At SP3, total and dissolved concentrations of Al, Cu, Fe, Mg, Mn, Ni, Pb, and Zn were higher on 4/16 than on any other sampling date, while Na concentration and pH (3.58) were lower than on any other date (Appendix E). Also on this date, total and dissolved concentrations of Al, Cu, Fe, Mg, Mn, Ni, Pb, and Zn were lower in the preceding tank sampled (Tank T23, SP5 – Figures 6 and 8) than in Tank T11 (SP3), while Na was 3.4 times higher, and pH was higher (4.2), but still acidic. Tank T23 is the settling tank (Figures 6 and 8), where basic water from Tank T20 traveled after being mixed with NaOH. Overflow from T23 traveled to the feed tank (T24) for the 2nd stage of Octolig[®] (Tank T25). The 2nd-stage pH dosing tank (T20) experienced repeated problems over time, including the dosing pump hose not remaining below the surface level of the tank of base and the pH not reaching the set-point of 8. The inconsistencies in dosing of NaOH in the 2nd-stage are evident in differences in sodium concentration over time at SP5 and SP3 in Figure 12; concentrations of Na are consistent over time within the 1st-stage of the treatment system (SP1b and SP2).

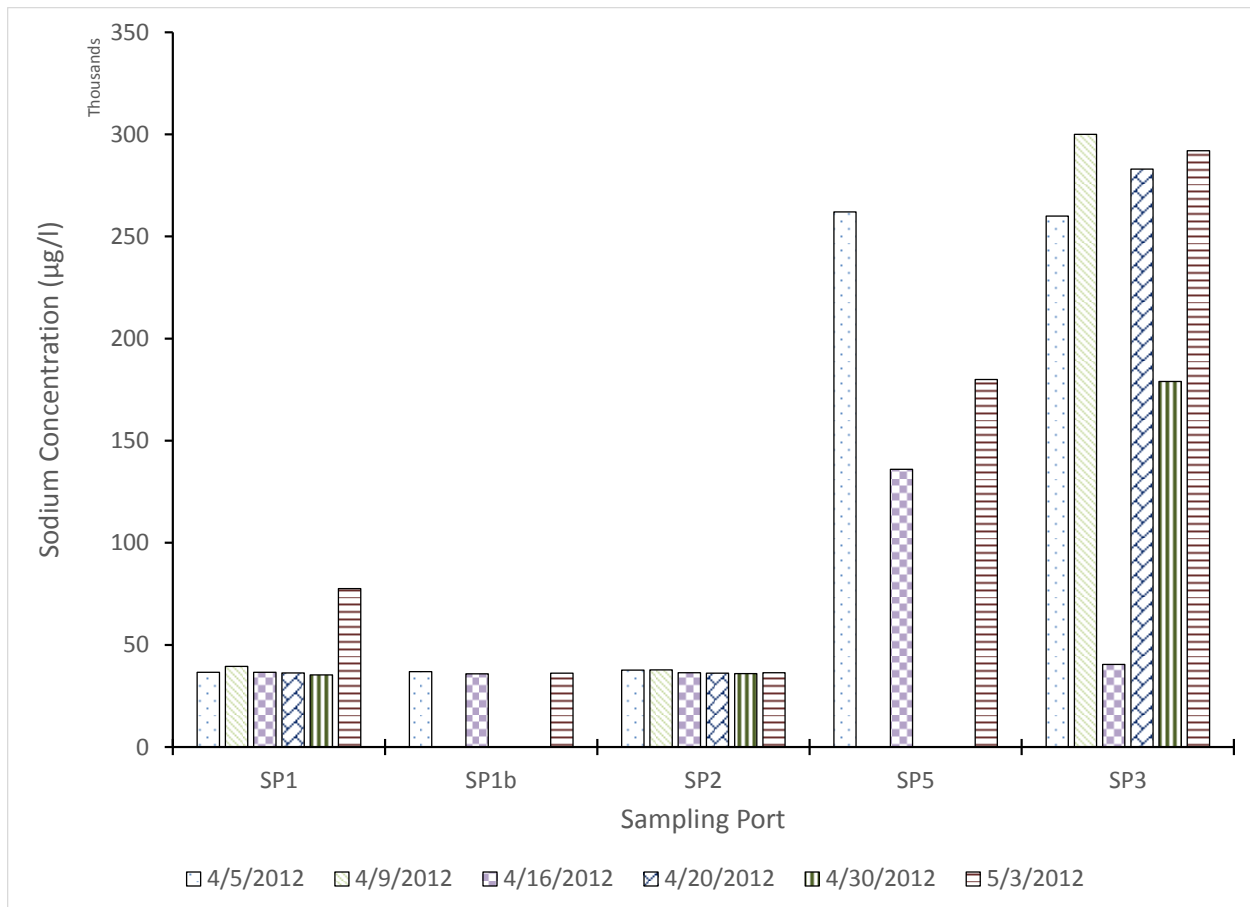


Figure 10. Sodium concentration at each sampling port over time.

When sampled on 4/5, 4/16, and 5/3, the pH of the water in samples from Tank T23 (SP5) was 6.20, 4.20, and 4.26, respectively, and the pH for samples from Tank T11 (SP3) was 6.08, 3.58, and 4.08, respectively (Appendix E and Section 3.2.1), indicating that the pH 8 set for the dosing tank was not sustained over subsequent tanks, including when it passed over the 2nd-stage Octolig® bed. Although it cannot be confirmed, it is possible that higher concentrations of ions observed in the samples from SP3 on 4/16, versus those in the SP5 samples, are due to the lower pH water causing release of previously sequestered ions from the Octolig® bed, especially considering that pH 3.8-4.2 was used for the regeneration of the 2nd-stage Octolig® bed (Section 2.2.2). The reason for the decreased Na concentration at SP3 relative to SP5 on 4/16 is not known. As a monovalent ion, Na would be expected to complex more weakly than would the divalent ions; however, at pH values similar to the regeneration pH values, perhaps Na was being sequestered by the 2nd-stage Octolig® bed as the metals were being released.

Because alkalinity originated from the base addition step in Tank T20 and it remained in the system through the clarifier (SP5) and the 2nd-stage Octolig® bed only on the dates when removal efficiencies were highest for metals other than Fe and Cu (removal of these metals was

predominantly in the 1st-stage Octolig[®], Section 3.2.6) is another indication that issues with base addition and pH control in the 2nd-stage of the process hindered overall effectiveness of the system. The decreased removal of Cu after April 20 could be due to the issues with the 1st-stage pH pump (Appendix D) and decreased pH values observed at SP2 (Section 3.2.1), or to sites in the 1st-stage Octolig[®] bed being unavailable for sequestration due to inefficient regeneration (see Section 3.3).

An additional potential cause for the lower than target (and sometimes acidic) pH evident in the SP3 and SP5 samples on all dates (Figure 9 and Appendix E) is a lack of sufficient residence time to achieve chemical equilibrium to neutralize acidity. The differences in Na concentrations between SP5 and SP3 (other than on 4/5) suggest that the 2nd-stage was not at equilibrium with the base added (Figure 10). If precipitation was still occurring after water flowed from the pH 8 adjustment tank (T20) into subsequent tanks (clarifier (T23 – SP5), feed (T24), Octolig[®] (T25) and effluent (T11 – SP3)), protons released during hydrolysis could have decreased the pH in any of these tanks (e.g., $2\text{Al}^{3+} + 4\text{H}_2\text{O} \rightarrow \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O} + 6\text{H}^+$). It is possible also that the residence time, or insufficient mixing, did not allow for complete physical exposure of the water to the added base, leading to only partially pH-treated water, with or without unreacted base, flowing to subsequent tanks. This potential issue might have been more important after April 27 when a higher concentration of NaOH (50%, versus 25% used prior to that date) would require a lower volume to be dosed into the large volume of water.

The tanks for pH adjustment were 330 gallon totes (Appendix A). Average influent flow from April 5 through April 10 was 5 GPM; average influent flow from April 11 to the end of the study was 10 GPM (Appendix D, Tables D1 and D2). The flow rate across the Octolig[®] beds ranged from 4.2 to 15.2 GPM (Appendix D), which originated from the feed tanks subsequent to each pH-adjustment tank. It was not documented what water level was maintained in the tanks preceding pH-adjustment, but if held constant with the influent rate, the residence times within the tanks can be estimated to have been a maximum of 66 minutes (330 gallon / 5 GPM) and 33 minutes (330 gallon / 10 GPM) for 4/5-4/10 and 4/11 – 5/7, respectively. Data indicate that better removal was achieved at influent flows of 5 GPM versus 10 GPM, which supports the potential lack of sufficient residence time under higher flow conditions to allow for equilibrium in the pH-adjustment tanks.

3.2.4 Removal efficiencies for ions of secondary interest (Secondary Objective 1)

To determine the removal efficiencies for Ag, As, Ca, K, Mg, Mn, Na, Ni, and Se, their concentrations in the effluent samples (SP3) were compared to their initial concentrations in the Virginia Canyon influent (SP1). Percentage removals for each element were calculated

using the equation provided in Section 3.2.3 and are presented in Table 6. Arsenic was not detected above the reporting limit (4 µg/l) in the influent or any system sample on any date and therefore is not included in Table 6.

Greater than 50% removal was observed for Mn, Ni, and Se on the first two sampling dates. Similar to the target metals, the best performance for all analytes presented in Table 6 generally was observed during the first week of the pilot test. The negative values observed for Na are due to the addition of sodium from the use of NaOH to adjust the pH, particularly from the 2nd-stage of the treatment process, as can be seen by the concentrations plotted in Figure 10. Negative values observed for potassium could be due to it being a contaminant in the NaOH used for pH adjustment in both the 1st and 2nd stages.

The high concentrations of Mg (92 – 120 mg/l) in the Virginia Canyon water were expected to result in some being removed by the Octolig[®]. Magnesium is the stronger of the two weakly bound divalent alkaline earth metals according to the Irving-Williams series (Section 1.4), and was observed to be removed by the treatment system at percentages greater than those for Ca.

Table 6. Comparison of effluent (SP3) concentrations with influent (SP1) concentrations for secondary interest elements.

Analyte / Parameter	Date					
	4/5/12	4/9/12	4/16/12	4/20/12	4/30/12	5/3/12
Ag - Total (µg/l)	n/a	n/a	n/a	n/a	44.4 ^A	37.5 ^A
Ag - Dissolved (µg/l)	28.6 ^E	n/a	30.0	44.4 ^A	37.5 ^A	28.6 ^A
Ca - Dissolved (µg/l)	5.2	1.6	2.7	8.3	0.4	5.6
K - Dissolved (µg/l)	-70.5	-16.9	-2.6	-2.5	-7.4	0.4
Mg - Dissolved (µg/l)	31.5	32.2	2.7	27.6	8.3	24.3
Mn - Total (µg/l)	65.4	51.1	3.3	26.6	10.0	25.8
Mn - Dissolved (µg/l)	63.8	55.9	2.4	30.8	10.8	29.6
Na - Dissolved (µg/l)	-610.4	-659.5	-10.4	-679.6	-407.1	-276.3
Ni - Total (µg/l)	73.6	69.3	0.2	37.6	12.2	31.8
Ni - Dissolved (µg/l)	74.6	69.5	-0.2	41.9	14.4	33.8
Se - Total (µg/l)	87.3	79.9	1.6	40.0	14.7	38.8
Se - Dissolved (µg/l)	84.0	76.1	-0.5	40.8	14.8	32.6

n/a = influent and effluent samples below RL or concentration in blank exceeded concentration in the influent sample

A = effluent sample below RL, value for RL used for calculations to indicate that removal is at least to the RL

E = QA criterion not met for either influent or effluent, value is an estimate

3.2.5 Regeneration process effectiveness (Secondary Objective 2)

The 1st-stage Octolig[®] bed was regenerated April 24-26 (Section 2.2.2). Preliminary bench testing had indicated that Fe and Cu were removed at low pH (~4) and Al, Cd, Cr, Co, Ni, and Zn were removed at higher pH (~6), which provided the basis for the 2-stage system and expectation of selective removal. The 2nd-stage Octolig[®] bed was regenerated only at the conclusion of the pilot; therefore, only Cu and Fe were used to evaluate regeneration effectiveness.

To compare effectiveness of the regenerated Octolig[®] media, the removal efficiencies shown in Table 6 for Cu and Fe collected on dates prior to regeneration were averaged and compared to the average removal efficiency for dates post regeneration; results are presented in Table 7. Because the anomalies in the percentage removals from 4/16 were believed to be due to issues with the 2nd-stage processes (see Section 3.2.3), and Cu and Fe were expected to be removed in the 1st-stage, 4/16 data were included in average for this comparison.

Table 7. Comparison between pre- and post-regeneration removal efficiencies for Cu and Fe in the 1st-stage of the treatment system.

	4/5/2012	4/9/2012	4/16/2012	4/20/2012	Pre-Regeneration Average	4/30/2012	5/3/2012	Post- Regeneration Average	Difference (post minus pre)
Cu - Total (µg/l)	98.1	96.8	61.1	43.0	74.7	48.9	25.5	37.2	-37.5
Cu - Dissolved (µg/l)	98.2	96.7	61.8	42.1	74.7	49.9	27.2	38.5	-36.2
Fe - Total (µg/l)	92.7	90.1	55.8	45.8	71.1	30.4	28.7	29.5	-41.5
Fe - Dissolved (µg/l)	91.7	91.3	59.9	47.2	72.5	34.2	-23.2	5.5	-67.0

Data in Table 7 indicate that removal of iron decreased over all dates and was not improved with regeneration. Percentage removal of copper also decreased with time, but a slight increase was seen immediately following regeneration (4/30 versus 4/20), although not to the level observed for fresh media (e.g., 4/5) and not sustained. On average, the regeneration process appears to have been ineffective. For both metals, percentage removal by the 1st-stage of the system was best during the first week of the pilot testing, similar to the overall system performance for these and other ions (compare with Tables 5 and 6).

3.2.6 Metal removal due to pH adjustment versus sequestration by Octolig[®]

To understand better the removal mechanisms of precipitation following NaOH addition and sequestration by Octolig[®] occurring in the system, the percent of target and secondary-interest ions removed by each step in the process were determined as follows:

f_x remaining = C_i/C_0 , where C_0 is the concentration of analyte in the Virginia Canyon water (SP1) and C_i is the concentration of the analyte at the specific sampling port.

- Percent removed by 1st-stage pH adjustment step (between SP1 and SP1b) = $100 * (1 - f_{SP1b})$; labeled SP1b in Table 8
- Percent removed by 1st-stage Octolig[®] step (between SP1b and SP2) = $100 * (f_{SP1b} \text{ remaining} - f_{SP2} \text{ remaining})$; labeled SP2 in Table 8
- Percent removed by 2nd-stage pH adjustment step (between SP2 and SP5) = $100 * (f_{SP2} \text{ remaining} - f_{SP5} \text{ remaining})$; labeled SP5 in Table 8
- Percent removed by 2nd-stage Octolig[®] step (between SP5 and SP3) = $100 * (f_{SP5} \text{ remaining} - f_{SP3} \text{ remaining})$; labeled SP3 in Table 8

The percent of each analyte removed at each step is presented in Table 8 for each of the dates when sampling was conducted at all five sampling ports, along with the overall treatment system removals (labeled SP1-SP3) for comparison. Alkalinity and pH also are shown for comparison.

Negative values indicate that the concentration increased over the particular step of the process. In some cases, the percentage removal at a given step was greater than the removal indicated for the system overall, due to water from some sampling locations having higher concentrations than at the prior sampling point (see Appendix E). Although the actual reason(s) for some sampling locations having concentrations of a given metal higher than that at an upstream sampling location is unknown, some potential reasons for observed higher concentrations between sampling ports are:

- Impurities in the NaOH used for pH-adjustment (e.g., K)
- Residence time within a given tank in the system being longer (or shorter) than the time required to collect samples from the tanks in question. In other words, the water collected might not have been the “same parcel” of water at the prior sampling point, even if well-mixed, due to inherent temporal differences in concentrations in the influent water and the volume of the water stored in tanks for treatment.
- Inherent error associated with measurements of very low concentrations (e.g., Se) and very high (e.g., Na) concentrations

Copper and iron were the only metals consistently removed by the 1st-stage Octolig[®], although efficiency decreased over time. Al, Ni, and Pb were removed by the 1st-stage Octolig[®] only at the start of the pilot with removal on later dates due to precipitation in the 2nd-stage (4/16/12) or a combination of precipitation and sequestration by the 2nd-stage Octolig[®] toward the end of the study (5/3/12). Only for the last date when all 5 ports were sampled (5/3/12) is there indication of the 2nd-stage Octolig[®] removing any metals at greater than 10% (except ~ 14% of Cd on 4/5/12), with the other sampling dates indicating removal predominantly through precipitation, indicated from the samples collected at SP5.

Table 8. Percentage of metals removed by each step in the overall pilot treatment system, percentage removed over the whole system, and pH and alkalinity at each step.

Analyte / Parameter	Date														
	4/5/2012	4/5/2012	4/5/2012	4/5/2012	4/5/2012	4/16/2012	4/16/2012	4/16/2012	4/16/2012	4/16/2012	5/3/2012	5/3/2012	5/3/2012	5/3/2012	5/3/2012
	SP1b	SP2	SP5	SP3	SP1-SP3	SP1b	SP2	SP5	SP3	SP1-SP3	SP1b	SP2	SP5	SP3	SP1-SP3
Al - Total (µg/l)	0.28	56.40	42.70	0.26	99.64	0.28	-11.55	39.75	-25.31	3.16	-5.93	-2.75	37.63	33.72	62.66
Al - Dissolved (µg/l)	-0.28	57.69	42.45 ^A (SP5 and SP3)	0 ^A (SP5 and SP3)	99.86 ^A (SP5 and SP3)	1.97	-12.24	38.82	-26.72	1.83	-6.61	-1.32	40.09	36.12	68.28
Ca - Dissolved (µg/l)	2.00	-2.00	1.20	4.00	5.20	2.34	2.34	-3.13	1.17	2.73	-0.80	0.40	1.60	4.40	5.60
Cd - Total (µg/l)	2.61	-4.89	70.16	14.01	81.89	-1.99 ^E (SP1)	-0.33 ^E (SP1)	14.62 ^E (SP1)	-18.27 ^E (SP1)	-5.98 ^E (SP1)	-6.35	1.90	12.38	14.92	22.86
Cd - Dissolved (µg/l)	-1.32	-0.99	70.59	13.78	82.07	0.65	3.59	6.86	-16.01	-4.90	-1.92	-2.56	14.42	15.38	25.32
Cu - Total (µg/l)	-0.47	98.57	-4.50	-0.07	93.54	-1.03	62.11	13.43	-2.41	72.10	-5.78	31.29	21.09	22.68	69.27
Cu - Dissolved (µg/l)	-1.58	99.73	-4.64	0.11	93.63	-0.45	62.24	12.36	-2.15	72.00	-5.17	32.37	21.42	21.66	70.28
Fe - Total (µg/l)	0.43	92.31	2.99 ^A (SP5 and SP3)	0 ^A (SP5 and SP3)	95.73 ^A (SP5 and SP3)	-2.39	58.17	20.52	-5.94	70.36	-6.45	35.16	38.71	23.35	90.77
Fe - Dissolved (µg/l)	-1.33	93.05	3.85 ^A (SP5 and SP3)	0 ^A (SP5 and SP3)	95.58 ^A (SP5 and SP3)	0.39	59.53	21.91	-8.56	73.27	-79.76	56.55	114.23	3.04 ^A (SP3)	94.05 ^A
K - Dissolved (µg/l)	0.20	-5.30	-33.79	-31.63	-70.53	0.20	-1.96	0.79	-1.57	-2.55	0.00	-0.37	-3.71	4.45	0.37
Mg - Dissolved (µg/l)	3.64	-2.73	26.18	4.45	31.55	1.79	3.57	3.57	-6.25	2.68	-2.88	0.00	12.50	14.71	24.33
Mn - Total (µg/l)	1.17	-10.51	65.50	9.21	65.37	-0.38	-1.15	18.21	-13.33	3.33	-3.94	1.14	16.52	12.07	25.79
Mn - Dissolved (µg/l)	1.70	-14.68	66.45	10.35	63.83	2.04	1.40	10.56	-11.58	2.42	-4.13	0.53	16.25	16.91	29.56
Na - Dissolved (µg/l)	-0.82	-2.19	-612.84	5.46	-610.38	2.19	-1.64	-272.13	261.20	-10.38	53.48	-0.39	-185.05	-144.33	-276.29
Ni - Total (µg/l)	0.49	29.66	38.25	5.19	73.58	0.00	-4.25	17.17	-12.76	0.16	-3.80	1.58	18.35	15.66	31.80
Ni - Dissolved (µg/l)	0.00	29.30	39.28	6.06	74.63	4.27	-2.37	9.97	-12.03	-0.16	-3.01	-0.33	19.73	17.39	33.78
Pb - Total (µg/l)	-11.67	31.11	75.00 ^A (SP5 and SP3)	0 ^A (SP5 and SP3)	94.44 ^A (SP5 and SP3)	-3.23	-8.06	43.01	-33.33	-1.61	-9.09	3.54	54.55	34.34	83.33
Pb - Dissolved (µg/l)	-12.35	31.76	74.70 ^A (SP5 and SP3)	0 ^A (SP5 and SP3)	94.12 ^A (SP5 and SP3)	-2.15	-2.69	41.40	-36.02	0.54	-36.05	-1.36	108.16	21.77	92.52
Se - Total (µg/l)	0.67	-11.33	98.00	0.00	87.33	0.54	0.54	22.70	-22.16	1.62	-0.59	4.71	18.24	16.47	38.82
Se - Dissolved (µg/l)	-4.17	-3.47	91.67	0.00	84.03	5.82	0.00	20.11	-26.46	-0.53	-11.81	1.39	26.39	16.67	32.64
Zn - Total (µg/l)	2.59	-1.50	90.44	4.26	95.78	-0.94	-0.40	21.79	-17.78	2.67	-6.73	5.33	15.48	11.29	25.38
Zn - Dissolved (µg/l)	4.04	-7.94	95.85	4.17	96.12	1.95	6.10	9.09	-13.90	3.25	-3.63	0.42	15.62	18.83	31.24
Alkalinity (mg/l as CaCO ₃)	<5 (RL)	<5 (RL)	33.7	47.9	-858 ^B	<5 (RL)	<5 (RL)	<5 (RL)	<5 (RL)	n/a	<5 (RL)	<5 (RL)	<5 (RL)	<5 (RL)	n/a
pH	3.05	4.95	6.20	6.08		3.14	3.99	4.20	3.58		3.64	3.24	4.26	4.08	

SP1b = removal from 1st-stage pH-adjustment; SP2 = removal from 1st-stage Octolig; SP5 = removal from 2nd-stage pH-adjustment; SP3 = removal from 2nd-stage Octolig; SP1-SP3 = overall system removal

n/a = influent and effluent samples below RL or concentration in blank exceeded concentration in the influent sample

A = effluent sample below RL, value for RL used for calculations to indicate that removal is at least to the RL

B = influent sample below RL, value for RL used for calculations to indicate the minimum increase in value

E = QA criterion not met for either influent or effluent, value is an estimate

To a greater degree than was observed for Cu and Fe, it appears that the majority of removal for each ion from the base addition in Tank T20 was negated by solubilization at the lower pH in Tank T11 (SP3) on 4/16/12 (Table 8). Without having more information about conditions within Tanks T20, T24, and T25 (see Figures 6 and 8), however, the mechanism for the increased concentrations at SP3 versus SP5 (negative removal percentages observed at SP3 in Table 8) cannot be determined.

Table 8 indicates removal of 53% of Na by the 1st-stage pH-adjustment on 5/3/12. Sodium concentrations in the Virginia Canyon influent were relatively constant over time – ranging from 36,000 to 39,500 mg/l, but the influent concentration reported for the 5/3 SP1 sample was 77,600 mg/l (Appendix E). The concentrations of Na on 5/3 at SP1b and SP2 were 36,100 and 36,400 mg/l, respectively, which are similar to concentrations at those sampling ports for the other dates in the study (range 36,200 to 37,800 mg/l) having the lower Virginia Canyon Na concentrations. Additionally, concentrations of all other analytes in SP1 on 5/3 were similar to previous sample dates (Appendix E). Therefore, although no error was noted in the laboratory analytical report or in the field notes, the Na data for SP1 on 5/3 is considered suspect and the removal observed at SP1b in Table 8 likely is not accurate.

Observed removal of Mg primarily was due to precipitation from addition of NaOH in the 2nd-stage (SP5) on 4/5 and to both precipitation and sequestration onto the 2nd-stage Octolig[®] on 5/3. Removal of Se was similar, with removal only observed at SP5 on 4/5 and 4/16, but at both SP5 and SP3 on 5/3. Nickel appears to interact with Octolig[®], having about half of the total removed on 4/5 being due to the 1st-stage Octolig[®] treatment and about half of the total removed on 5/3 being due to the 2nd-stage Octolig[®] treatment; removal on 4/16 appears to be solely due to base addition.

Coupled with results presented previously, it appears that the overall system was operating as expected only during the first week of the pilot and that issues with control of pH in the 2nd-stage, saturation of ligand sites in the 1st-stage, and later pH-adjustment pump issues in the 1st-stage hindered overall performance of the process. While sampling at the additional ports provided insight to the mechanisms for removal of the ions, results were not consistent across the three sampling dates. Therefore, it cannot be confirmed which mechanisms were predominating over the course of time when these intermediate sites were not sampled.

3.2.7 Anions

To understand how some anions interact with Octolig[®], F, Cl, and SO₄ were measured in water samples. Analytical results are provided in Appendix E and overall system removal efficiencies

are provided in Table 9. Table 10 presents percentages removed at each step in the treatment process using the same formulas provided in Section 3.2.6.

Table 9. Comparison of effluent (SP3) concentrations with influent (SP1) concentrations for F, Cl, and SO₄.

Analyte	Date					
	4/5/12	4/9/12	4/16/12	4/20/12	4/30/12	5/3/12
Chloride	-46.2	-4.8	-0.8	-5.8	-2.7	-0.6
Fluoride	14.8	42.9 ^A	0.0	0 ^E	8.7	-1050 ^B
Sulfate	22.9	13.9	-0.6	4.5	1.1	-40.7

A = effluent sample below RL, value for RL used for calculations to indicate that removal is at least to the RL

B = influent sample below RL, value for RL used for calculations to indicate the minimum increase in value

E = QA criterion not met for either influent or effluent, value is an estimate

Table 10. Percentage of Cl, F, and SO₄ removed and corresponding pH at each step and over all steps in the pilot system.

Date	Sample Port	Chloride	Fluoride	Sulfate	pH
4/5/2012	SP1b	-3.43	-62.96	-1.68	3.05
	SP2	-3.69	3.70	16.76	4.95
	SP5	-8.71	77.778	5.587	6.20
	SP3	-30.34	-3.704	2.235	6.08
	SP1-SP3	-46.2	14.8	22.9	
4/16/2012	SP1b	-2.39	-6.82	-2.81	3.14
	SP2	1.33	4.55	0.56	3.99
	SP5	0.00	15.91	1.69	4.20
	SP3	0.27	-13.64	0.00	3.58
	SP1-SP3	-0.8	0.0	-0.6	
5/3/2012	SP1b	-0.29	n/a	-2.82	3.64
	SP2	-0.29	n/a	1.69	3.24
	SP5	-0.29	n/a	1.13	4.26
	SP3	0.29	-1050.0 ^B	-40.68	4.08
	SP1-SP3	-0.6	-1050 ^B	-40.7	

B = influent sample below RL, value for RL used for calculations to indicate the minimum increase in value

n/a = influent and effluent samples below RL

Chloride removal percentages in Table 9 are negative (higher concentration in effluent sample than influent sample), indicating that it was not being sequestered by the Octolig[®] and suggesting that it likely was a contaminant in the sodium hydroxide added to the system.

Some fluoride was removed on different dates (Table 9), but it appears it may have been a contaminant as well; for example, removal is negative at the 1st pH-adjustment sampling point

(Table 10) on all dates when the site was sampled. Removal may have been through some association with Octolig[®], e.g., removal at SP2 on 4/5 and 4/16, or through the precipitation reactions, e.g., removal at SP5 on 4/5 and 4/16. The data do not provide a consistent trend to evaluate the potential removal mechanism(s) for F.

Sulfate was the predominant anion present in the Virginia Canyon influent water and concentrations were nearly constant over time at 1,760 to 1,800 mg/l. Results in Table 9 indicate that there was a trend of decreasing removal over the entire system, with some dates showing potential of an additional source (negative removal percentages). Table 10 indicates there was some removal via both Octolig[®] stages (SP2 and SP3) and the 2nd-stage pH-adjustment step (SP5), but that no consistent trend exists. On the last sampling date, the concentration of sulfate remained essentially constant through the system (Appendix E) at close to 1,800 mg/l; however, the concentration in the effluent from SP3 was 2,490 mg/l, which is ~ 41% higher than the influent concentration (1,770 mg/l). Potential causes for this difference are 1) an error in the sulfate measurement, although the lab did not note any issues; or 2) release of sulfate previously sequestered in the 2nd-stage Octolig[®] treatment bed. The concentration at SP5 was identical to the input, which indicates that the increase was not due to contamination from the NaOH added at that stage, as well as indicating that no sulfate was removed in previous steps on that date.

Martin et al. (2010) showed Octolig removed anions, including selenious acid and fluoride, in individual bench tests, but they did not specifically test removal of sulfate or chloride. As shown in Table 6, 80 to 87% of selenium was removed during the first few days of the study, but then removal ranged from 14 to 40%, excluding the anomalous data from 4/16. Martin et al. (2010) showed greater than 99% removal of the selenium, but at concentrations much higher than those in this study: 55 mg/l Se (as H₂SeO₃) versus 17 µg/l Se, with reported removal to below the detection limit (not provided) from DI water and to 173 and 229 µg/l from well and tap water, respectively. These results suggest competition with other ions present, which could explain lower removals observed in this study, especially at the comparatively lower starting concentrations in Virginia Canyon water. Fluoride removal seen by Martin et al. (2010), with a starting concentration of 190 mg/l, increased as the total dissolved concentration (TDS) increased, with 74% removal at TDS of 194 mg/l and 99.7 % removal at TDS of 602 mg/l (yielding 49 mg/l and 1 mg/l, respectively, remaining in solution). In a mixture study using sulfate, phosphate, nitrate, and nitrite, Stull and Martin (2009) obtained > 72% removal of sulfate with concentration decreasing from 30 mg/l to 8.5 mg/l. The pH of the mixture was 8.65, which is higher than the pH obtained in Octolig[®] beds in this study where sulfate was observed to be decreased on 4/5 by 17 and 2% at SP2 and SP3, respectively, with pH values of 4.95 and 6.08.

3.3 Regeneration Concentrate Samples

Composite samples were collected from three depths from Tanks T26 (1st-stage regeneration) and T28 (2nd-stage regeneration) and analyzed by Test America for total concentrations of target and secondary-interest ions. The 1st-stage Octolig[®] bed was regenerated April 24-26 and May 7-8, while the 2nd-stage Octolig[®] bed was regenerated only May 7-8. The 1st-stage Octolig[®] bed was regenerated with a solution of H₂SO₄ having a pH of 1.8-2.2, and the 2nd-stage Octolig[®] bed was regenerated with a solution of H₂SO₄ having a pH of 3.8-4.2; the final solutions contained approximately 1% sulfuric acid. The April and May regenerant solutions for the 1st-stage were combined in one tank during the pilot. Concentrations and masses of target and secondary-interest ions present in each of the final regenerant solutions is provided in Table 11.

The final volumes were 200 and 80 gallons for the 1st-stage and 2nd-stage regenerant solutions, respectively. Masses of analytes in the regenerant solutions were determined using the following equation:

$$\text{Mass lbs} = \text{volume of regenerant (gal)} * \text{concentration } (\mu\text{g/L}) * [(3.785411784 \text{ L/gal}) \div (453592.37 \text{ mg/lb}) / 1000 (\mu\text{g/mg})]$$

Table 11. Concentrations and masses of ions in regeneration concentrate samples.

Analyte	1st-Stage Regenerant (T28)		2nd-Stage Regenerant (T26)	
	Concentration (µg/l)	Mass (lb)	Concentration (µg/l)	Mass (lb)
Aluminum	270,000	4.5E-01	200,000	1.3E-01
Calcium	250,000	4.2E-01	250,000	1.7E-01
Cadmium	340	5.7E-04	310	2.1E-04
Coper	220,000	3.7E-01	110,000	7.3E-02
Iron	59,000	9.8E-02	19,000	1.3E-02
Lead	< 13 (MDL)	< 2.2E-05 (MDL)	37 (J)	2.5E-05 (J)
Magnesium	100,000	1.7E-01	110,000	7.3E-02
Managese	75,000	1.3E-01	81,000	5.4E-02
Nickel	680	1.1E-03	610	4.1E-04
Potassium	9900 (J)	< 1.7E-02 (J)	9000 (J)	< 6.0E-03 (J)
Sodium	310,000	5.2E-01	380,000	2.5E-01
Zinc	79,000	1.3E-01	85,000	5.7E-02

All values are total recoverable

J indicates the concentration was above the method detection limit (MDL), but below the reporting limit (RL)

Because the amounts of each ion removed via sequestration on the Octolig[®] bed and removal through precipitation varied over time, by amount and potentially by mechanism, it is not

possible to determine an accurate mass of metals believed to be sequestered on each Octolig[®] bed with which to compare the masses recovered in the regenerant solutions. A rough estimate can be calculated based on the average influent concentrations (totals), the average percentage removals onto Octolig[®] in the two stages using data from Table 8 (removals at SP2 for the first stage and SP3 for the second stage), and the total volume of Virginia Canyon water treated.

Metal removals by the 2nd-stage Octolig[®] bed on April 16, 2012 were negative (SP3 in Table 8), which indicates that mass was removed from the bed on that date. With the exceptions of Cu and Fe, which were associated primarily with the 1st-stage Octolig[®] bed (Table 8, SP2) as by design, overall system analyte removals were observed to be lower on 4/16 than on any other date (Tables 5 and 6), likely due to the behavior observed in the 2nd-stage Octolig[®] bed. Because the negative removals observed for the 2nd-stage Octolig[®] bed on 4/16 do not appear to be representative of the performance of the bed at other times, and the average removal represents the average mass of material expected to be present on the Octolig[®] bed from the entire time the pilot operated, the SP3 removal percentages in Table 8 on 4/16 were excluded from the calculation for the average removal by the 2nd-stage Octolig[®] step. The resulting masses of metals estimated to be sequestered (and subsequently expected to be removed from the media with regeneration) using the following equation are presented in Table 12.

Mass lbs = volume (gal) of Virginia Canyon water treated by stage * average concentration of analyte (mg/L) * [(3.785411784 L/gal) ÷ (453592.37 mg/lb)] * average percentage removal for Octolig[®] bed / 100

Table 12. Estimated masses of ions associated with each Octolig[®] bed.

Analyte / Parameter	Average Influent Concentration (ug/l) (n=6)	Average % Removed by 1st-stage Octolig [®] (n=3)	Average % Removed by 2nd-stage Octolig [®] (n=2)	Mass Removed by 1st-stage Octolig [®] (lb)	Mass Removed by 2nd-stage Octolig [®] (lb)
Aluminum	71,050	14.03	16.99	3.5E+01	4.1E+01
Calcium	251,333	0.25	4.20	2.2E+00	3.5E+01
Cadmium	311	NR	14.58	NR	1.5E-01
Copper	8,803	63.99	11.30	2.0E+01	3.3E+00
Iron	2,687	61.88	23.35	5.9E+00	2.1E+00
Lead	19	8.86	34.34	6.0E-03	2.2E-02
Magnesium	109,333	0.28	9.58	1.1E+00	3.5E+01
Manganese	77,117	NR	10.64	NR	2.8E+01
Nickel	634	9.00	10.43	2.0E-01	2.2E-01
Potassium	5,200	NR	NR	NR	NR
Sodium	36,860	NR	NR	NR	NR
Zinc	74,617	1.14	7.78	3.0E+00	1.9E+01

Volume treated in 1st-stage = 426,240 gallons; volume treated in 2nd-stage = 402,600 (Appendix D, Tables D1 and D2)

NR = negative removal calculated

A comparison between the estimated masses provided in Table 12 with the masses provided in Table 11 indicates very low recoveries of the mass of ions that were expected to be present on the media (Table 13), suggesting the regeneration process was inefficient.

Table 13. Percent recovered during regeneration of the Octolig[®] beds.

Analyte / Parameter	Al	Ca	Cd	Cu	Fe	K	Mg	Mn	Na	Ni	Pb	Zn
% Recovered in 1st-stage Regenerant	1.3	18.8	n/a	1.8	1.7	n/a	15.3	n/a	n/a	0.6	n/a	4.3
% Recovered in 2nd-stage Regenerant	0.3	0.5	0.1	2.2	0.6	n/a	0.2	0.2	n/a	0.2	n/a	0.3

n/a = either regenerant data was flagged or negative average percent removal was calculated for SP2 or SP3

It is possible also that the values in Table 12 are over-estimated and that the system did not sequester as much of the ions as was indicated using averages. A quick calculation (shown below) using the 4/5 data (Appendix E and Appendix D, Table D1), however, shows that removal of Cu onto the 1st-stage Octolig[®] bed over only the first few days was 2.3 lbs, which also is higher than the amount of Cu recovered in the 1st-stage regenerant solution (Table 11).

Mass lbs = (Cu concentration at SP1b – Cu concentration at SP2) µg/l * volume treated in 1st-stage from start to 4/5/12 (gal) * (3.785411784 L/gal ÷ 453592.37 mg/lb) ÷ 1000 µg/mg

$$\text{Mass lbs} = [299 - 162] \text{ mg/l} * 32,760 \text{ gal} * (3.785411784 \text{ L/gal} \div 453592.37 \text{ mg/lb}) = 2.3 \text{ lbs}$$

Therefore, it is probable that the regeneration process was not efficient at removing the ions from the Octolig[®] ligand, although some error likely also is due to the use of averages with the system having variability in efficiency over time. Additionally, it is possible that the pH of the solutions used for regeneration was not sufficiently low enough to allow release of the sequestered ions and for the sulfate not to interact with the Octolig[®] (see Section 3.2.7). For example, the pH of the solution used for regeneration (pH 3.8-4.2) of the second Octolig[®] bed was within a single pH unit or less to the pH measured on effluent from SP3 on a number of the sampling dates (Appendix E).

MSE (1998) used 4% nitric acid for regeneration of the Octolig[®] in their column tests and saw recoveries ranging from 28 to 60% for Al, 56 to 92% for Cu, 15 to 107% for Mn, 40 to 85% for Fe, and 31 to 114% for Zn. The authors noted that the “fluctuating and low recovery rates require further study”, but suggested that the regeneration time might not have been adequate (MSE, 1998). It is not known exactly why recovery was poor in this pilot study, but it is possible

that regeneration time was inadequate; however, the time for the regeneration process was not noted in Arcadis' report.

3.4 Sludge

Lime or other chemical treatments commonly are used to remove metals from mining-impacted water and result in the production of a sludge waste product. The sludge generally is dewatered and then trucked off-site for disposal, which adds to the cost of the treatment system. This project sought to evaluate a treatment system for its ability to remove metals from water into a form that was recoverable and to produce a sludge volume that was at least 30% lower than that produced by traditional treatment systems. Initially it was intended to make a direct comparison between the sludge produced in the pilot test and the sludge produced by the Argo WTP through their lime treatment process; however, there are several reasons a direct comparison was not possible:

- 1) Only one of the three water sources treated by the Argo WTP was used for the pilot (Section 1.3).
- 2) Approaches to dewatering may differ among operators, so a comparison on a mass of solids basis is more appropriate.
- 3) The Octolig[®] pilot suffered from a number of problems (e.g., maintaining desired pH across system steps) that affected performance over time, so the efficiency of a properly operating Octolig[®] system is not known
 - a. Some analytes showed a wide range of removal efficiencies over time (Tables 5 and 6)
- 4) Had pH been maintained at the higher value desired, sludge production likely would have been increased relative to actual results; therefore, the sludge reduction calculated in this section is not predictive of future performance.

3.4.1 Sludge comparison (Secondary Objective 3)

While the lime process is more efficient and consistent over time than was the Octolig[®] pilot, a rough comparison of sludge production can be made assuming a lime process having the same average removal efficiencies (based on unfiltered samples, except for Mg) as the pilot system, based on Tables 5 and 6. Because negative removal percentages by the 2nd-stage Octolig[®] bed were observed for most analytes on 4/16, which likely is the cause for much lower removals observed for the system overall (Tables 5 and 6), and this anomaly does not appear to represent the typical behavior observed at other times (see other dates in Table 8, and Tables 5

and 6), the removal percentages calculated for 4/16 in Tables 5 and 6 were not used to calculate the average removal percentages in Table 14.

Expected sludge generation from a traditional lime process on Virginia Canyon water was calculated stoichiometrically (Table 14). The approach was based on the average influent (SP1) concentrations of each metal over the study period; it assumed all metals were removed as hydroxide precipitates at pH 10.5, with iron as $Fe_2O_3 \cdot H_2O$ and aluminum as $Al_2O_3 \cdot H_2O$; and it did not include anion removal. The theoretical concentrations of solids that could be produced were multiplied by the observed removal efficiencies from the pilot system; the efficiency-adjusted concentrations of solids are shown in Table 14.

Table 14. Theoretical sludge composition expected from lime treatment of Virginia Canyon water with removal efficiency equivalent to that observed in the Octolig[®] pilot system.

	MW (mg/mmol)	Average Influent Concentration (mg/l) (n=6)	Formula	Formula Mass (mg/mmol)	Metal Oxide / Hydroxide Mass Expected (mg/l)	Total Solids (mg/l)	Average Pilot System Removal Efficiency (%) (n=5)	Efficiency- Adjusted Total Solids (mg/l)
Aluminum	27	71.05	$Al_2O_3 \cdot H_2O$	119.96	157.95	157.95	77%	122
Cadmium	112	0.31	$Cd(OH)_2$	146.40	0.41	0.41	34%	0.1
Copper	64	8.80	$Cu(OH)_2$	97.54	13.51	13.51	84%	11.3
Iron	56	2.69	$Fe_2O_3 \cdot H_2O$	177.69	4.27	4.27	89%	3.8
Lead	207	0.02	$Pb(OH)_2$	241.19	0.02	0.02	89%	0.0
Magnesium	24	109.33	$Mg(OH)_2$	58.31	262.24	262.24	25%	65.0
Manganese	55	77.12	MnO_2	86.94	122.04	122.04	36%	43.7
Nickel	59	0.63	$Ni(OH)_2$	92.71	1.00	1.00	45%	0.4
Selenium	79	0.02	$Se(OH)_4$	147.00	0.03	0.03	52%	0.0
Zinc	65	74.62	$Zn(OH)_2$	99.37	113.43	113.43	48%	54.1
Total						675		301

To obtain a mass of solids that could be produced, the following formula was used:

$$\text{Mass solids (lbs)} = \text{volume of water treated (gal)} * \text{total solids concentration (mg/L)} * [(3.785411784 \text{ L/gal}) \div (453592.37 \text{ mg/lb})]$$

The volume of water treated was 426,240 gallons. The calculated mass of solids expected from lime treatment is 1,069 lbs.

The Octolig[®] treatment process produced 5,500 gallons of sludge over the time of the pilot (chemical analysis of the sludge in Tank T23 at the end of the pilot is provided in Appendix E, data from Test America), with an average TSS concentration in the sludge of 8,950 mg/l. This equates to 411 lbs of sludge produced ($5500 \text{ gal} * 8,950 \text{ mg/l} * [(3.785411784 \text{ L/gal}) \div (453592.37 \text{ mg/lb})]$). The presence of solids was not assessed in any other tanks in the system

aside from the settling tank (T23). Therefore, the sludge mass may be a low estimate for sludge actually produced during the pilot due to the following:

- It does not take into account any solids that settled in the sludge sample prior to being analyzed by the laboratory (results were reported as TSS rather than total solids)
- It does not take into account any potential solids that might have formed in the 2nd pH-adjustment tank (Tank T20) that were not carried over into the settling tank (Tank T23)
- It does not take into account any suspended solids that might have been carried over into tanks subsequent to Tank T23 (e.g., 2nd stage feed Tank T24 or 2nd stage Octolig[®] Tank T25)
- It does not take into account any potential solids formed in the 1st pH-adjustment tank (Tank T10 – SP1b), although data in Table 8 indicate minimal loss of metals between SP1 and SP1b, or in Tanks T13 (1st stage feed tank) or T18 (1st stage Octolig[®] tank)
- It does not take into account any solids present in the clarifier underflow water (17,860 gal)

The reduction in sludge produced in the pilot compared to what could have been produced by treatment with lime is: $1,069 - 411 = 658$ lbs, which corresponds to 62% [$100 - 100 \cdot (411/1069)$] less solids mass produced by the Octolig[®] system and satisfies the third primary objective. For the reasons mentioned previously, however, this comparative reduction could be overestimated and is not predictive of future performance.

3.5 Recovery and Reuse (Primary Objective 4)

The fourth primary objective of the study was to evaluate reuse of the metals recovered into the regenerant from the Octolig[®] treatment process, via smelting or some other process. Some things that may influence the feasibility of recovering metals from a waste product include:

- Specific components present and their concentrations
- Physical form of the material
- Amount of material (mass or volume)
- Distance to the recycler from the point of material generation
- The number of sites supporting the recycler
- Whether or not the material would be a constant source and of constant consistency (composition and concentration)
- Market value of the given metal to be recovered

Various methods are used to remove metals from acidic solutions. For example, electrowinning of sulfuric acid leach solutions is a process that is done at a mine site prior to smelting of some metals, such as Zn. There are a number of centralized facilities in the U.S. that provide off-site recycling for the plating industry, some of whom use hydrometallurgical processes, which should be a sufficient method for the regenerant solution from Octolig® treatment.

The National Metal Finishing Resource Center (NMFRC) has an online reference book for metals recycling (www.nmfrc.org/bluebook). The 'Blue Book' provides a listing of a number of companies that recycle materials to recover the metals and discusses the source materials accepted and the methods used. Some of the companies discussed accept aqueous solutions. Of the companies surveyed by NMFRC and provided in the NMFRC Blue Book, the three most likely candidates for recycling small volumes of treatment residual similar to that from the Octolig® treatment process are 1) Horsehead Resource Development Company, 2) CP Chemicals, and 3) Encycle/Texas Inc. The following paragraph provides a summary of the capabilities of these three companies from the Blue Book (www.nmfrc.org/bluebook).

Horsehead Resource Development Company operates six facilities located in IL, PA, TN, OK, and TX. They accept metal-bearing sludges, filter cakes, bag house dusts and soils, and process them using two kiln technologies. Only the Chicago, IL and Rookwood, TN facilities accept electroplating wastes. CP Chemical is a major US producer of inorganic metallic salts and accepts metal bearing wastes from over 1,000 clients through their Environmental Recovery Services Division, at six plant locations. Their process includes hydrometallurgical steps, but the details of their method were not provided. Materials accepted and processed include mostly segregated metal bearing wastes from plating baths, etchants, pickling solutions, and strippers containing brass, cobalt, copper, nickel, tin, solder, or zinc. Encycle/Texas Inc. operates in Corpus Christi, TX for approximately 150 electroplating shops. They accept liquid and solid wastes containing copper, lead, zinc, nickel, and other metals to a lesser extent. Processing is via chemical and hydrometallurgical methods with their products being used by primary smelters and others.

It was hoped that the regenerant solutions produced in this pilot study could be transported and assessed by operators at smelters or other metals recycling facilities for the potential to recover the metals present. The low masses of metals recovered (Table 11), however, did not warrant paying the shipping and processing costs required for such an assessment. Instead, Arcadis provided details on the regenerant solutions to several companies through discussions. The following paragraphs provide the feedback on the potential for recycling of the regenerant solutions obtained in the Octolig® process.

PM Recovery, which acquired CP Chemical, and said they could not accept the regenerant or the sludge wastes from the Octolig[®] treatment process for recycling. A subsidiary of Horsehead Resource Development Company, INMETCO in Pittsburg, PA, is set up to receive aqueous plating wastes, but they require the following: 1) payment of a fee for processing; 2) a sample is needed prior to agreement or quoted price; 3) feed specifications are Ni > 100 mg/l, Cr > 10 mg/l, Cu < 1800 mg/l, P < 860 mg/l, and Sn < 400 mg/l. Based on these requirements, the regenerant solution from the Octolig[®] process cannot be accepted because it is not high enough in either Ni or Cr.

Environmental Quality Company (EQ), which was a company not included in the NMFRC survey, has the capabilities to recover metal residues from acidic solutions, but they require a higher purity of copper (or copper and nickel together, which is their desired combination) than what was present in the regenerant solutions. The company stated that a 2% Cu solution in 1% sulfuric acid would be acceptable, if it were relatively pure. The concentration of Cu recovered in the first regenerant solution was 220 ppm (which was 1.8% of the expected mass to be recovered). If there had been 100% recovery of the mass of Cu expected to be associated with the 1st-stage Octolig bed, this would have resulted in the regenerant having a concentration of 12,222 ppm Cu [$200/x = 1.8/100$; $x = (220 \text{ ppm} \times 100 / 1.8)$], which equates to a 1.2% solution of Cu. EQ stated they would accept solutions of low concentrations for recycling, however, they would charge a fee for processing that may exceed any value obtained from the recovered metal(s).

Intec Ltd, an Australian hydrometallurgical firm, stated that a key issue for recovery of any value is that the waste is either of high grade (i.e., high concentration) or high volume, and preferably both. The company suggested that revenue from a waste containing 20% Cu would cover some processing costs, if there was sufficient volume of material, but that the value recovered from a 0.5% Cu solution would not cover any processing costs, or even the costs to move the material to the recycler.

Given the response of the recyclers surveyed, both by Arcadis and through the NMFRC survey (www.nmfrc.org/bluebook), it presently appears that it would be quite difficult to economically recycle the waste product from the Octolig[®] treatment system with influent water similar to the Virginia Canyon mine drainage. To do so would require that 1) the Octolig[®] treatment system, including the regeneration process, be much more efficient over time than was evident in this pilot test; 2) concentrations of influent water treated would need to have higher concentrations of economically-important metals or a larger volume would need to be treated; and/or 3) that the regenerant solutions be concentrated further (e.g., by evaporation) to allow for a greater mass of metals per unit volume prior to being sent for recycling.

3.6 Capital and O&M Costs (Primary Objective 5)

It was desired in this project to obtain capital and O&M costs associated with the technology's use at this representative site and cost estimates for scaling up to 50 and 300 GPM for evaluation of potential use at other sites. Partial costs for this pilot were provided by Arcadis and are listed below:

- Bench-testing:
 - Labor = \$20,000
 - Analytical = \$3,317
- Start-up, including labor to the start of monitoring: \$14,660
- Monitoring, maintenance, sampling, and operations post start-up: \$11,879
 - This cost does not include sampling conducted by EPA, for which there was no cost to the project
- Analytical costs during monitoring: \$3,870
 - These costs do not include testing conducted by EPA, for which there was no cost to the project
- Materials:
 - Equipment costs were not provided
 - Reagent costs were estimated at \$100 per week
 - Power costs were estimated at \$46 per week

Spreadsheets for estimated capital and annual costs were provided by Arcadis for an assumed 50 GPM treatment having water chemistry similar to that of the Virginia Canyon water. Two estimates were provided: one assuming the use of the Octolig[®] system and another assuming the use of a conventional chemical/physical treatment system using lime. Both estimates were based on removal of Al, Cd, Cu, Fe, Pb, Mg, Mn, Ni, Se, and Zn, and included bi-weekly sampling and analytical costs. For the Octolig[®] system, cost was based on the average overall removal percentages observed in the pilot for each analyte (Table 14) and 75% recovery of sequestered analytes from the Octolig[®] media into the regenerant solution (which is higher than what was observed in the pilot). Because efficiency was observed to be varied over time and variable within each of the stages of the system, the cost estimate may not be representative of an actual 50 GPM system. Costs for the Octolig[®] system also assume there is no cost for disposal of the regenerant solution, but don't assume there is a payment for regenerated metals; i.e., costs are assumed to be net zero for regenerant. For the lime system, 100% removal of analytes as hydroxide precipitates was assumed. Details are provided as Appendix F.

In summary, the total capital investment for the 50 GPM Octolig[®] system was estimated to be ~ \$4.4 million and for the lime treatment system it was estimated to be \$3.3 million. Total annual costs were estimated to be ~ \$578,000 and ~ \$412,000 for the Octolig[®] treatment system and the lime treatment system, respectively, including sludge disposal costs. Therefore, for the Octolig[®] system to have an economical advantage over a traditional lime system, costs recovered from recycling of metals from the regenerant would need to be ~ \$166,000 per year, or higher, and another ~ \$1.1 million would need to be obtained over the course of time to make up for the capital investment cost differences. Because removal efficiency was observed to be varied over time and variable within each of the stages of the system, the cost estimate may not be representative of an actual 50 GPM Octolig[®] system.

A cost estimate for a 300 GPM system was not provided by Arcadis; however, treatment of so high a flow with Octolig[®] as the sole treatment likely would not be possible in a remote location due to the size of the system that would be required.

4 Concluding Remarks

The first primary objective – to meet or exceed (i.e., be better than) site-specific (watershed) water quality criteria for Al, Cd, Cu, Pb, and Zn – was not met for Cd, Cu, or Zn, and not met consistently for Al or Pb. The second primary objective of greater than 90% removal of Al, Cd, Cu, Fe, Pb, and Zn was never met for Cd, but was met for the other ions on at least one sampling date, with the best system performance (values of percentage removed and numbers of ions having > 90% removed) occurring during the first week of sampling. Overall, removal efficiency was not consistent. At the influent concentrations in the Virginia Canyon mine drainage, with the exceptions of Fe and Pb, removal of metals to the levels of the water quality criteria in Table 4 would require greater than 95% removal, with most sampling dates having influent concentrations requiring greater than 99% removal. The site at which to perform the pilot study was not decided upon prior to award of the contract; therefore, unlike the Virginia Canyon drainage, treatment at a different site to > 90% removal might result in concentrations that would meet the WQC.

The pilot test seemed to operate best during the first part of the study, approximately during the first week. The lack of the system's efficiency over time appears due to a combination of things: 1) problems with pH pumps and control during operations; 2) potentially insufficient retention times or incomplete mixing to reach equilibrium within the pH-control tanks and within the Octolig[®] treatment tanks; and 3) inefficient regeneration of the Octolig[®] media,

perhaps due to retention time being too short and/or that the pH was not low enough to allow complete removal of the sequestered ions.

As well as removing contaminants from the water to meet water quality standards, other technical requirements of treatment systems for remote locations include being compact (e.g., < 54' x 9', size of an 18-wheel trailer), requiring minimal maintenance/monitoring, and the ability to function in low temperatures owed to high altitudes. The Octolig® pilot demonstrated that such a system can fit into relatively small locations, with all components, except the water conveyance lines, clarifier tank, and bulk chemicals, fitting within an 18-wheel trailer, for the flow treated during the pilot (Figure C2). Larger flows, however, would require a larger system that may or may not fit onto a small remote site (Table E1, a 50ft x 50ft pre-engineered building was suggested by Arcadis to house a system to treat 50 GPM flows). Low temperatures would require an insulated trailer, and perhaps a heating source. The pilot initially was planned to start during the summer/fall, but our hosts at the Argo site were unable to accommodate us at that time. Instead, the system was started in November 2011, but low temperatures were encountered and testing was cancelled and postponed to spring 2012 due to freezing issues resulting in leaks in water and chemical lines. The trailer used was not insulated because pilot testing during the cooler season was not anticipated; however, these colder temperatures would be anticipated in any full-scale treatment system operating year-round at remote sites in Region 8. The pilot test required daily monitoring and maintenance, and it isn't clear that such frequent monitoring and maintenance wouldn't be required even with a more efficiently operating system, due to the careful control of pH that is necessary to maximize sequestration of metals. Based on responses of recyclers surveyed, it appears that it would be difficult to economically recycle the waste product from the Octolig® treatment system, unless concentrations of economical metals in the regenerant were higher than those observed in this pilot study.

Based on the results from this pilot study, Octolig® does not appear to be an appropriate sole treatment system for remote sites where the desire is to consistently meet water quality criteria, consistently remove > 90% of the metals present, have minimal monitoring requirements, and recover metals from the waste stream(s) to offset costs of treatment. It is possible that further refinement of the process could allow for it to be used as a secondary treatment; however, it may not be cost-effective without the ability to recycle the metals at a profit.

5 References

Gao, Baojiao, An, Fuqiang, and Liu, Kangkai. 2006. Studies on chelating adsorption properties of novel composite material polyethyleneimine/silica gel for heavy metal ions. *Applied Surface Science*, **253**:1946-1952.

Lindoy, Leonard F. and Eaglen, Peter L. 1993. Ion complexation by silica-immobilized polyethyleneimines. US Patent # 5,190,660, March 2, 1993. Available online: <http://www.freepatentsonline.com/5914044.pdf>. Last accessed May 23, 2014.

Lindoy, Leonard F., Eaglen, Peter L., and Alldredge, Robert L. 1999. Immobilized branched polyalkyleneimines, US Patent #5,914,044, June 22, 1999. Available online: <http://www.freepatentsonline.com/5914044.pdf>. Last accessed May 23, 2014.

Martin, Dean F. 2010. Robert Alldredge, chemical engineer, inventor, entrepreneur, developer of Octolig®, American Chemical Society Division of the History of Chemistry, Presentation. 240th ACS National Meeting, Boston, MA, August 22-26, 2010. Abstract available online: <http://www.scs.illinois.edu/~mainzv/HIST/meetings/2010-fall/HIST%20Fall%202010%20Abstracts%20Program%20Only.pdf>.

Martin, Dean F., Lizardi, Christopher L., Schulman, Eileen, Vo, Bryan, and Wynn, Darius. 2010. Removal of selected nuisance anions by Octolig. *Journal of Environmental Science and Health Part A*, **45**:1144-1149.

MSE Technology Applications Inc. and Montana Tech of the University of Montana. 1998. Final report – Berkley Pit innovative technologies project phase II: Metre General Inc., demonstration. Prepared for US EPA and US DOE. Final Report December 1998. Available online: <http://www.epa.gov/ordntrnt/ORD/NRMRL/std/mwt/a4/a4113.pdf>.

Smith, Robert M. and Martell, Arthur E. 1975. Critical Stability Constants. Volume 2: Amines. Plenum Press, New York.

Stull, Frederick W. and Martin, Dean F. 2009. Comparative ease of separation of mixtures of selected nuisance anions (nitrate, nitrite, sulfate, phosphate) using Octolig®. *Journal of Environmental Science and Health Part A: Toxic/Hazardous Substances and Environmental Engineering*, **44(14)**:1545-1550.

U.S. Environmental Protection Agency (U.S. EPA). 2007. Remediation system evaluation (RSE) Central City / Clear Creek Superfund site Argo Tunnel water treatment plant Idaho Springs, Colorado, Report of the remediation system evaluation site visit conducted May 16, 2007. Final Report Sept. 2007. EPA-542-R-07-019.

6 Appendices

6.1 Appendix A: Arcadis (EPS) Operational SOPs for Treatment System Operation and Regeneration



Environmental Products & Solutions

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Octolig® Pilot System Standard Operation Procedure

The objective of this Operation Procedure (SOP) is to provide a brief description of the Octolig® pilot unit systems employed for the Recovery and Reuse of Metals from Mining Influenced Water field technology demonstration at the ARGO Treatment Plan in Colorado Springs, Colorado. This SOP assumes that the end user has, at a minimum, reviewed the site specific Health and Safety Plan (HSP) and has general knowledge and understanding of the pilot system operation.

Pilot System Operational Description

Acid Mine Drainage (AMD) waters will be collected from the Virginia Canyon pipe outlet and conveyed via gravity to 1st Stage pH Neutralization Tank T10. A flexible coupling will connect a SCH 80 PVC Tee and the flow through branch will be connected to a hose that will be routed to the pilot system. The branch connection will be left open to facilitate a siphon break as well as provide a means of overflowing raw influent during periods of variable flow. A control valve (V-1) is provided to set the flow rate to 15 gpm. A float valve (V-2) is provided within tank T10 to provide a mechanical shutoff to prevent over filling (the Virginia Canyon stream will overflow into the Argo Tunnel outfall through the Tee connected to the Virginia Canyon outlet). ***You cannot count on V2 to prevent overfilling with the setup. V1 must be set to provide less than adequate flow so pump P14 shuts off occasionally.***

Tank T10 will be mixed with Mixing Pump P11 with an integral float switch to control operation (e.g., if the tank level is drawn because the influent flow rate from Virginia Canyon drops below the established operational flow rate of the pilot unit ***Based on current drawing, pump P11 is only stopped in the event of a catastrophic Tank failure or a failure in the drain pipe integrity***) to facilitate neutralization of pH. Sodium hydroxide will be dosed to the Tank T10 via 1st Stage Base Pump P12 and adjust the pH to the proper set point (the set point will be established at 4.0 at the startup of the pilot unit). The pH of Tank T10 will be monitored with a pH sensor, pH11. The sensor is connected to a pH monitor/controller that provides a visual display of the pH within the tank and also controls the operation of Pump P12. The

pump controller provides on/off control, the operator will have to adjust the stroke and frequency settings of the pump manually to optimize delivery of the sodium hydroxide reagent.

The pH neutralized influent will overflow from Tank T10 into 1st Stage Feed Tank T13. The 1st Stage Feed Pump P14 will convey the pH neutralized influent into 1st Stage Octolig[®] Unit T18. Note that 1st Stage Filter F17 is provided to remove total suspended solids (TSS) prior to entering T18. **The valve immediately after the filter (V8) will be used to choke back flow of P14 to the desired feedrate (15 gpm until further notice) as indicated at the flow meter.** The influent flow will enter and be distributed at the bottom of the 1st Stage Octolig[®] Unit (T18) and uniformly flow upward through the Octolig[®] bed. The 1st Stage Octolig[®] Unit (T18) effluent will then overflow into 2nd Stage pH neutralization Tank T20.

Tank T20 will be mixed with Mixing Pump P21 with an integral float switch to control operation (e.g., if the tank level is drawn because the influent flow rate from Virginia Canyon drops below the established operational flow rate of the pilot unit. **Again, the drawing indicates this is only possible in the event of tank or pipe failure, not problems with flow.**) to facilitate neutralization of pH. Sodium hydroxide will be dosed to the Tank T20 via 2nd Stage Base Pump P22 and adjust the pH to the proper set point (the set point will be established at 8.0 at the startup of the pilot unit). The pH of Tank T20 will be monitored with a pH sensor, pH21. The sensor is connected to a pH monitor/controller that provides a visual display of the pH within the tank and also controls the operation of Pump P22 (as well as 2nd Stage Acid Pump **{No planned service for this pump}** – see description for regeneration cycles). The pump controller provides on/off control, the operator will have to adjust the stroke and frequency settings of the pump manually to optimize delivery of the sodium hydroxide reagent.

The pH neutralized process water will overflow from Tank T20 into the Clarifier T23, where aluminum hydroxide precipitates will settle out and form a sludge blanket at the bottom of the vessel. Clarified effluent will overflow to 2nd Stage Feed Tank T24. The 2nd Stage Feed Pump P25 will convey the clarified process water into 2nd Stage Octolig[®] Unit T25. **The valve (V20) after the P25 MUST be set to feed faster than the overflow. T24 must empty and be shut off by the level control periodically to ensure it does not overflow!** The process water flow will enter and be distributed at the bottom of the 2nd Stage Octolig[®] Unit (T25) and uniformly flow upward through the Octolig[®] bed. The 2nd Stage Octolig[®] Unit (T25) effluent will then overflow into Effluent Clearwell Tank T11. Overflow from Effluent Clearwell Tank T11 will flow via gravity to a water collection sump within the Argo Treatment Plant. Note that a portion of this treated effluent will be used in the Regen/Rinse Feed Tank T12 to make up the acid regeneration solution. Tank T12 should be filled after start-up and lineout but well before regeneration (fill when you confirm Zn treatment at the outfall) and then left isolated.

Operations sampling must take place before routine shutdown for sludge management and regeneration. Sludge measurement, sampling and discharge is integrated into routine shut down.

Routine Shutdown

The Influent control valve(V1) from Virginia Canyon is closed. pH adjusted water from T13 should be drawn down until level control shuts off P14. Valve V10 feeding T18 should be closed. The liquid remaining in the 1st stage Octolig[®] tank (T18) will be pumped out through the bottom and valve V9 using

pump P16, to T20 where it will be neutralized and flow to the clarifier and continue treatment. When T18 is empty, P16 should be turned off and V9 closed.

Sludge in T23 may now be measured and managed.

Regeneration Description

When detection of a metal breakthrough, after an Octolig® stage is found, the system will be shut down for regeneration of the Octolig® beds. The 1st Stage Octolig (T18) is expected to require regeneration weekly or semi-weekly. The 2nd Stage Octolig (T25) will likely be regenerated once in the demonstration.

1st Stage Octolig (T18) will always be regenerated first. Liquid in tank T12 will be made acidic to pH 2 by adding sulfuric acid with the controller fed by P15 Regen Acid Pump while the P16 Regen Feed Pump is recirculating water in T12. Acid from T12 will then be pumped with P16 into T18. P16 will be turned off and T18 will be drained back through V9 to T12 where the pH can be re-adjusted to pH 2. This process is repeated until pH in Tank T12 is \leq pH2.2 prior to adjustment. Acid in tank T12 is pumped with P16 to tank T28. Tank T28 is then isolated pending sampling and disposal.

2nd Stage Octolig (T25) is drained (this should be possible without pumping) into T12 **with special attention to stopping flow from T25 before the T12 overflows**. Water from T12 will be pumped into 1st Stage Octolig (T18). Water will be drained from T18 into T12. The pH of T12 will be adjusted up to 4.0 while recirculating though P16. Water will be pumped with P16 into T18. And the process repeated until pH is \geq 3.8 and \leq 4.5 prior to adjustment in T12.

If no 2nd Stage Regeneration:

- Water will then be pumped through P16 from T12 into pH neutralization tank T20. Begin Startup Procedure.

With 2nd Stage Regeneration:

- Water will then be pumped through P16 from T12 into 2nd Stage Octolig (T25). T25 is drained (this should be possible without pumping) into T12 **with special attention to stopping flow from T25 before the T12 overflows**. The pH of T12 will be adjusted to 4.0 while recirculating though P16. Water will be pumped with P16 into T25. The process will be repeated until pH is \geq 3.8 and \leq 4.5 prior to adjustment in T12. Water in tank T12 is pumped with P16 to tank T26. Tank T26 is then isolated pending sampling and disposal.
- Water will then be drained from T25 (this should be possible without pumping) into T12 **with special attention to stopping flow from T25 before the T12 overflows**. If the level in T12 is below the fill pipe from the effluent clearwell tank (T11), T11 should be allowed to drain into T12 **with special attention to stopping flow from T11 before the T12 overflows**. The pH of T12 will be adjusted to 8.0 while recirculating though P16. Water will be pumped with P16 into T25. Water will then be drained from T25 into T12. The process will be repeated until pH is \geq 7.8 and \leq 8.5 prior to adjustment in T12. Water will then be pumped from T12 into T25. Begin Startup Procedure.

ARCADIS Regeneration SOP for Field Pilot Plant

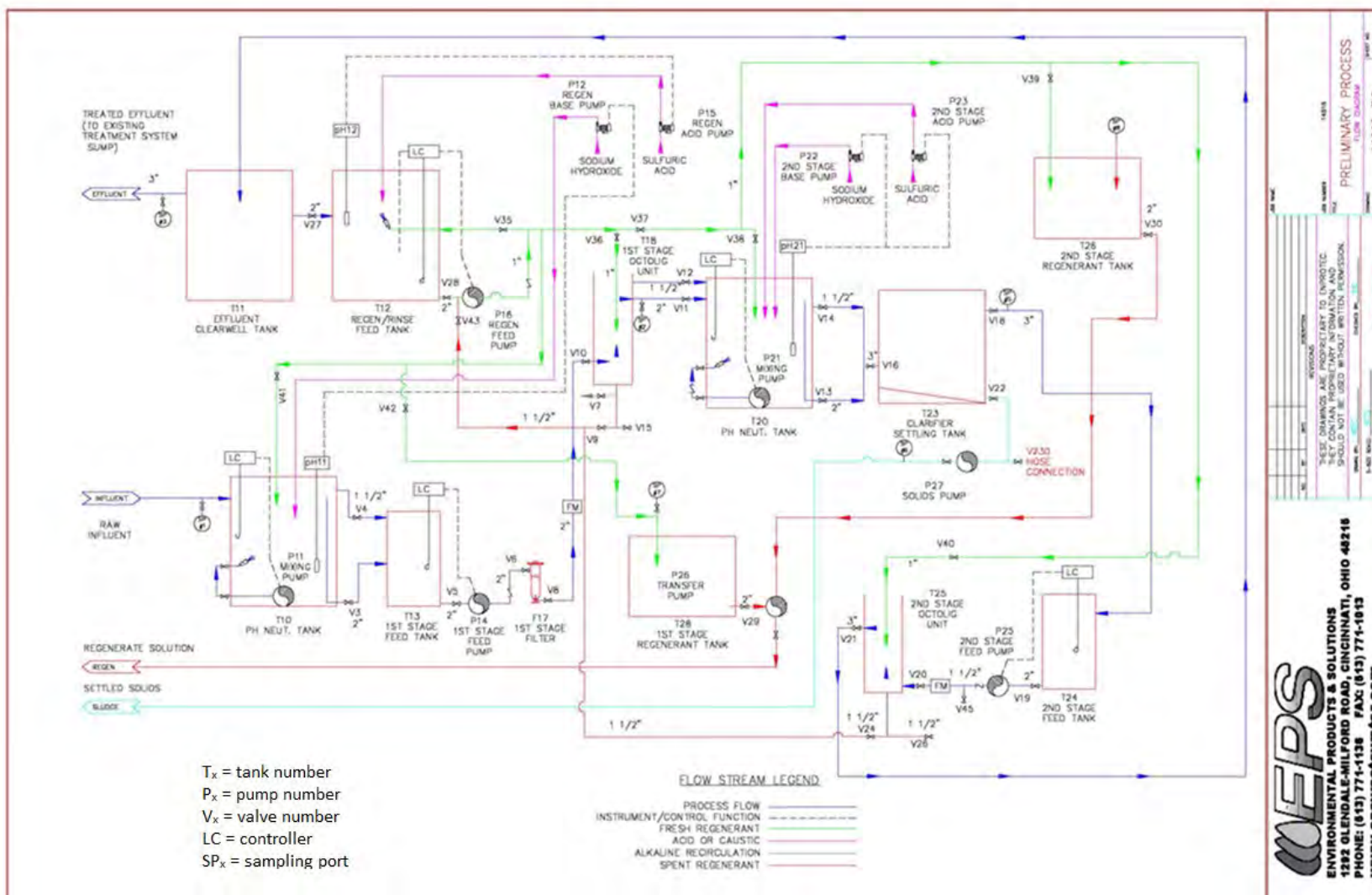
Start from normal operation treating AMD. T12 should be full of treated effluent. Operations samples in this mode of operation

1. Move to Step 1 – Drain T18. Stop AMD influent. Empty T18 into T20 and continue treating
 - a. Measure and Manage sludge in T23 after Step 1 and before Step 7.
2. Step 2 – pH adjust T12. Bring pH to 1.8-2.2 while recirculating adding H₂SO₄ as necessary
3. Step 3 – fill T18 with solution from T12.
4. Step 4 – purge T18 into T12.
5. Repeat 2., 3., and 4. until pH does not require acid adjustment after Step 4. T12 is full of pH 1.8-2.2 water and T18 is empty.
6. Step 5 – Transfer T12 contents (used regenerant) to T28
7. Step 6 – Purge T25 to fill T12. CAREFUL not to overfill T12. Leave remaining water in T25!
8. Repeat Step 3 – fill T18 with solution from T12. This solution should be ~pH 8. This is to rinse acid from T18.
9. Repeat Step 4 – purge T18 into T12.
10. Repeat Step 2 – pH adjust T12. Bring pH to 3.8-4.2 while recirculating adding NaOH as necessary.
11. Repeat Step 3 – fill T18 with solution from T12.
12. Repeat Step 4 – purge T18 into T12.
13. Repeat 10., 11. And 12. Until pH does not require base adjustment after step 4. T12 is full of pH 3.8-4.2 water and T18 is empty.
14. IF NO 2nd Stage REGENERATION proceed to 15. If 2nd Stage REGENERATION skip to 18.
15. Step 7 – Purge T12 to T20. Continue treatment (Neutralization etc) but there will be insufficient volume to discharge effluent.
16. Restart system Treating AMD. Open V1 allowing pH control to operate as tanks fill.
17. Step 10 - Fill T12 with Effluent from T11. CAREFULL not to overfill T12.
18. Step 8 – Fill T25 with solution from T12. Solution should be pH 3.8-4.2.
19. Repeat Step 6 – Purge T25 to fill T12. CAREFUL not to overfill T12. Leave remaining water in T25!
20. Repeat Step 2 – pH ad adjust T12. Bring pH to 3.8-4.2 while recirculating adding H₂SO₄ as necessary.
21. Repeat 18, 19, and 20. Until pH does not require base adjustment after step 6. T12 is full of pH 3.8-4.2 water and T25 contains residual 3.8-4.2 water.
22. Step 9 - T12 contents (used regenerant) to T26.
23. Repeat Step 6. Be careful not to overfill T12. There may not be enough water in T25 to fill T12.
24. Step 10 - Fill T12 with Effluent from T11. CAREFULL not to overfill T12.
25. Step 7 - Purge T12 to T20. Continue treatment (Neutralization etc) but there will be insufficient volume to discharge effluent.
26. Repeat Step 6.
27. Repeat 25. and 26. Until pH in T12 is >7.5.
28. Repeat Step 7 – purge T12 to T20.
29. Restart system Treating AMD. Open V1 allowing pH control to operate as tanks fill.
30. Step 10 - Fill T12 with Effluent from T11. CAREFULL not to overfill T12.

6.2 Appendix B: Equipment/Parts List from Arcadis

Item #	Item Description	Quantity
T10	pH Neutralization tank - 330 gallon tote	1.00
P14	mixing pump for T10 - Dayton Pump # 3YV65 POPR 32 GPM	1.00
LC10	Level Control	2.00
T12	Firststage feed tank - 330 gallon tote	1.00
LC12	Level Control	2.00
P14	Firststage feed pump - Dayton # 2MRP POPR 1.0 HP, 1ph	1.00
P17	Firststage bag filter	1.00
T18	Firststage Clarify media filter - Hercules 64" x72" vertical filtration tank, w bottom collection piping, PVC, S40182	1.00
FM	Flow rate paddle wheel, Signal #S-2027-00-PO, 4-20 mA Output	1.00
FM	PVC Installation Filling, 2" 6M P487E20F	1.00
T20	pH Neutralization tank - 330 gallon tote	1.00
P21	mixing pump for T20 - Dayton 3YV65	1.00
LC20	Level Control	2.00
pH 20	Wetspan pH analyzer (M Popen per T P16 S-432014)	1.00
P22	Second stage feed pump - Walchem chemical dosing pump	1.00
P23	Second stage acid pump - Walchem chemical dosing pump	1.00
T20	Clarifier settling tank - supplied by/owner	1.00
T24	Second stage feed tank - 330 gallon tote	1.00
LC24	Level Control	2.00
P25	Second stage feed pump - Dayton # 6P70R 1 HP	1.00
FM	Flow rate paddle wheel, Signal #S-2027-00-PO, 4-20 mA Output	1.00
FM	PVC Installation Filling, 1-1/2", 6M P487E18F	1.00
T25	Second stage Clarify media filter, Hercules 48" x72" Vertical Storage Tank, S40148	1.00
T11	Effluent clarifier tank - 330 gallon tote	1.00
T26	Firststage regan tank - 330 gallon tote	1.00
P26	Firststage regan transfer pump	1.00
P27	Regan transfer pump from clarifier, #20-23832-1, 1.0 HP	1.00
T28	Second stage regan tank - 330 gallon tote	1.00
T12	Regan/Reflow tank tank - 330 gallon tote	1.00
pH 11	pH analyzer, included with P12	1.00
pH 11	Regan beam pump pH sensor, 608U 04	1.00
pH 12	pH analyzer, included with P10	1.00
pH 12	Regan acid pump pH sensor, #648U 04	1.00
LC12	Level Control	2.00
P28	Regan feed pump - Dayton #12MVB	1.00
P22	Regan beam pump - direct tank at starting pump Regan pH monitor w/ pump, # 648U 04	1.00
P23	Regan acid pump - chemical dosing pump same as P22	1.00

6.3 Appendix C: Process Flow and Equipment Placement



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14818-010
 PRELIMINARY PROCESS
 1 OF 1

Figure C1. Process flow diagram. Figure prepared by EPS, subcontractor to Arcadis and modified by EPA.

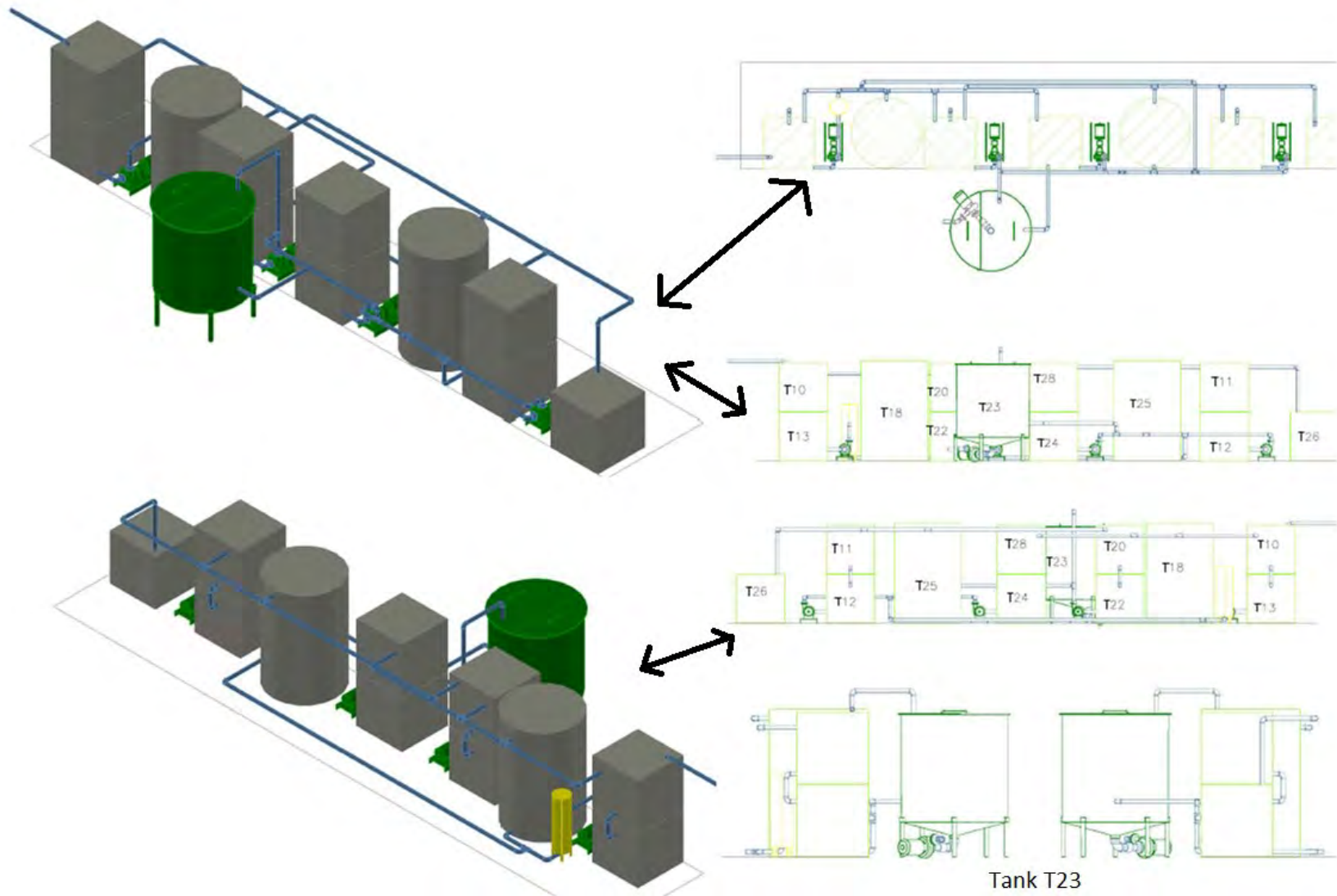


Figure C2. Visualization of spatial arrangement of equipment in the trailer, in two orientations. The large green tank is T23, which was located outside the trailer. Figure prepared by EPS, subcontractor to Arcadis and modified by EPA.

6.4 Appendix D: Equipment Installation and Operation Notes from Arcadis

6.4.1 Installation and Fall 2011 Operations

The pilot test equipment within the trailer was assembled at the EPS (subcontractor) facility in Cincinnati Ohio. It arrived at the Argo site September 24th, 2011 but was unable to be maneuvered into the narrow space behind the large scale treatment building allotted for this demonstration until September 27th. Work in October 2011 included installing the sample collection piping, interconnecting the trailer to the exterior settling tank, and the Argo plant for discharge, connecting electrical power for the system, loading the Octolig media into the appropriate tankage. Collection piping was designed and installed for approximately 10 gpm, based on the sustained fall flow reported for Virginia Canyon.

The system was used for limited initial operations in November of 2011. During this commissioning period, numerous problems in the system were diagnosed and addressed. For example, efforts were required to balance the flows of pumps within the system to maintain proper hydraulic levels in tanks, despite the presence of a PLC control system. An air release point was installed in the system discharge piping to improve flow performance. However, freezing weather conditions led to multiple freezeups and some leakage from piping, despite efforts to provide insulated and heat traced exterior lines. It was determined to be impractical to heat the trailer with electrical resistance heaters during extended periods of cold weather, since it had very poor insulating properties. A decision was made in early December to delay further demonstration operations until Spring 2012. The system was drained to the extent feasible, detached from the water supply, turned off and winterized to the extent practicable. Vulnerable parts such as pH meter parts were removed for warm storage in an ARCADIS facility. Total flow in November 2011 was minimal – approximately 5,700 gallons to the 1st Stage Octolig Unit (T18) and 1,100 gallons to the 2nd Stage Octolig Unit (T25). Therefore, a relatively small percentage of the bed capacity was consumed and no regeneration cycles were performed.

6.4.2 Spring 2012 Operations Notes

Field work to restart the system began March 26, 2012. Pipes and fittings were reinstalled/repared. The water supply line to the Virginia Canyon discharge began rapidly on March 27, since water was needed to commission the system. With an influent flow rate of 6

gpm and numerous tanks to fill, an extended period was required for startup². That time was also extended because initially operations proceeded only during normal working hours to ensure that any leaks that developed were rapidly addressed. Monitoring with field test kits began on March 30th and the first laboratory samples were collected on April 5th. Operational periods and flow rates are compiled in Tables C1 and C2. Flow through the system and volume treated is presented graphically in Figures C1 through C3.

On April 13, 2012, it was observed that the second stage pH dosing pump hose had risen above the level of the base in the supply container. The container was refilled and the hose secured at a lower position and the system ran well until April 16th. On April 18th it was noted that the increased flow rate had markedly increased base consumption and so the size of the ready use base reservoir for Tank T20 was increased. Early in the day, the T20 tank pH was 4.12, which was well below the set-point. But, after the larger tank was installed and refilled, the T20 pH returned to near the set-point at 8.05. Because the observed pH at SP5 and in a special sample drawn directly from T20, were lower than their target range, the pH set-point at Tank T20 was adjusted from pH 8 to pH 9. Data suggested that pH was dropping across the clarifier, perhaps due to proton release resulting from precipitation of manganese within the clarifier (rather than in Tank T20) due to slow oxidation kinetics (greater than one hour) at pH less than 9 and in the presence of high sulfur (Hemm, 1963). Another possible cause of these pH drops considered was the buildup of lower than target pH water in the Clarifier Settling Tank (T23), perhaps due to the temporary base feed outages that had been experienced (discussed above). The set-point was adjusted to pH 8.7 on April 19th, to attempt balance between the need for pH control and available reagent vendor delivery schedules. On Friday morning, April 20th, the operator again noted that the intake hose had moved above the level of the base, despite efforts to secure it, the flexible tube coiled and pulled away from the bottom of the container. A piece of rigid PVC tube was then installed to maintain the flexible base intake tube in a straightened, vertical configuration.

On Monday morning, April 23rd, the ARCADIS operator noted that the entire contents of the tank of base, about 30 gallons of 25% NaOH, had been consumed over the weekend. It was also noted that sludge generation had increased, and that the sludge level had risen from approximately 1.0 to 1.5' depth in the clarifier to 4' to 4.5', which caused some sludge to overflow the clarifier into succeeding tanks. The sludge that had overflowed into the succeeding tanks and was cleaned out and added back into the clarifier that day. After

² There were five 330 gallon tanks in the system, a 1,000 gallon 1st Stage Octolig® Unit, 2,300 gallon Clarifier Settling Tank and 560 gallon 2nd Stage Octolig® Unit for a total system hydraulic capacity (not counting lines in the main system or tanks used for regeneration only) of approximately 5,500 gallons.

collecting field parameters, the system was taken off line later in the afternoon of April 23rd to allow the first stage media to be regenerated. As a diagnostic step, the pH read by the dosing pump controller was compared to the field pH meter and found to agree within 0.02 pH units. On April 23rd, a drum of 50% NaOH solution arrived. This had been ordered after exhaustion of the initial supply 25% NaOH solution, since the stronger solution was more quickly available, and warmer temperatures allowed use of the stronger solution, which is more vulnerable to crystallization at lower temperatures.

In scheduling the regeneration, ARCADIS wanted to balance the objectives of maximum water treatment efficiency with maximizing the loading onto the Octolig. High Octolig loading was desirable both for treatment economics and to maximize the potential to recover a valuable product. Through April 16th, the field zinc data suggested that treatment was efficient. While data on April 18th suggested zinc breakthrough, data on April 20th and 23rd suggested resumption of zinc removal. The decision to go into regeneration on the 23rd was thus guided in part by field data and by the desire to complete regeneration and restore the system to operation within the available time on site.

Since this was the first time this unit had been regenerated, and the first time these staff members had performed a regeneration with strong acid, the work was undertaken slowly for added caution. Regeneration was completed by the evening of April 26th and the system restarted early in the morning on April 27th. On April 27th, using the stronger 50% NaOH solution for pH adjustment, ARCADIS observed some substantial “overshooting” above the pH set-point in Tank T20. Due to the higher strength of the 50% NaOH solution, the feedback control loop lagged behind the response in pH at the pumping rate previous used for the 25% NaOH solution. As a result, the pH in Tank T20 tank rose as high as pH 11 before gradually drifting down to the set-point. To address this, ARCADIS reduced the pumping rate of the 2nd Stage Base Pump (P22). This reduced the base consumption over the weekend (April 28 and 29) to 15 gallons of 50% NaOH, which was within the capacity of the ready use reservoir. However, at this lower pump setting, Pump P22 was not able to achieve the pH set-point. On April 30th, the pH was observed to range between 7.50 and 7.63 at Tank T20 before the base pump was adjusted to an intermediate pumping rate.

On Tuesday May 1st, it was noted that after 11 straight measurements of pH at SP 2 (after the 1st Stage Octolig® Unit [T18]) in the expected range (greater than pH 4) two sequential measurements well below pH 4 had been recorded (on Friday the 27th and Monday the 30th). This led to efforts to diagnose problems with the 1st Stage pH Neutralization Tank (T10), 1st Stage Base Pump (P12), and appurtenant controls. On May 2nd it was determined that the Pump P12 was overheating. It was determined that the repair could not be rapidly completed in the field. There were project schedule constraints (driven in part by the need of the host facility

to be freed of the extra burden of balancing effluent flows from pilot test before their busiest season – spring snow melt). Therefore, in consultation with EPA, the decision was made to proceed with sampling by EPA on Thursday May 3rd even with the pH at Tank T10 at approximately 3.5, below the pH 4 target, because of the malfunctioning pump. ARCADIS collected its final round of samples on Friday May 4th. The system was shut off on Monday May 7th after the completion of the planned operational period. Regeneration of both Octolig® beds was completed on May 7th and May 8th.

The total volume of acid used for all of the regeneration was 3 gallons of Sulfuric Acid (66 Degrees Baume 93.2%). The total volume of base used in water treatment was 110 gallons of 25% NaOH solution and 35 gallons of 50% NaOH. The total volume of first stage regenerant produced was 200 gallons (Tank T28) and 80 gallons of second stage regenerant solution (Tank T25).

Careful coordination was needed with the Argo Treatment Plant staff, who received our discharge from the pilot scale unit, to ensure that they were prepared for changes in flow rate. During this initial operational period, problems with the pH dosing pumps and flow meters were diagnosed and resolved. Since temperatures in early spring can still dip down below freezing overnight, foam insulation, heat tape, and electrical space heaters were used to maintain system temperatures.

Attempts were made to begin overnight operation on the evening of April 5th, but this was discontinued because constrictions in the system discharging water back to the main Argo treatment plant prevented proper flow balancing. Overnight operations were successfully begun as of April 6th. The system was shut down on April 11th for maintenance including leak repair, pH meter recalibration and installation of a larger discharge line to the Argo treatment plant sump. The system was restarted the same day. The influent flow rate was increased on April 13th, taking advantage of the larger discharge line.

Table D1. First Stage flow rates and treatment volumes

Date / Time	Period Time (min)	Period Down-Time (min)	Period Run-Time (min)	Cumulative Run-Time (min)	Average Influent Flow Rate (GPM)	Total Influent Volume (gal)	Total Volume Treated (gal)	Bed Volumes	Average Period Flow Monitor Flow Rate (GPM)	Average Influent Flow Rate (GPM)
3/26/12 9:00	--	--		0			0		0.0	--
3/27/12 9:00	1,440	960	480	480	6	2,880	2,880	19	14.5	0.0
3/28/12 9:00	1,440	960	480	960	6	5,760	5,760	39	13.5	0.0
3/29/12 9:00	1,440	960	480	1,440	6	8,640	8,640	58	13.4	0.0
3/30/12 9:00	1,440	960	480	1,920	10	13,440	13,440	90	14.0	0.0
4/2/12 9:00	4,320	3,300	1,020	2,940	10	23,640	23,640	159	14.0	0.0
4/3/12 9:00	1,440	960	480	3,420	10	28,440	28,440	191	14.0	3.0
4/4/12 9:00	1,440	960	480	3,900	10	33,240	30,600	205	4.5	4.0
4/5/12 9:00	1,440	960	480	4,380	5	35,640	32,760	220	4.5	5.0
4/6/12 9:00	1,440	0	1,440	5,820	5	42,840	39,096	262	4.4	5.0
4/7/12 9:00	1,440	0	1,440	7,260	5	50,040	45,144	303	4.2	5.0
4/9/12 9:00	2,880	0	2,880	10,140	5	64,440	58,104	390	4.5	5.0
4/11/12 9:00	2,880	0	2,880	13,020	10	93,240	93,240	626	14.5	11.0
4/13/12 9:00	2,880	0	2,880	15,900	10	122,040	122,040	819	15.2	11.0
4/16/12 9:00	4,320	0	4,320	20,220	10	165,240	165,240	1,109	15.0	11.0
4/18/12 9:00	2,880	0	2,880	23,100	10	194,040	194,040	1,302	15.0	11.0
4/19/12 11:00	1,560	0	1,560	24,660	10	209,640	209,640	1,407	15.0	10.0
4/23/12 9:00	5,640	0	5,640	30,300	10	266,040	266,040	1,786	15.0	10.0
4/24/12 9:00	1,440	1,440	0	30,300	0	266,040	266,040	1,786	0.0	10.0
4/25/12 9:00	1,440	1,440	0	30,300	0	266,040	266,040	1,786	0.0	10.0
4/26/12 9:00	1,440	1,440	0	30,300	0	266,040	266,040	1,786	0.0	10.0
4/30/12 9:00	5,760	0	5,760	36,060	10	323,640	323,640	2,172	14.8	10.0
5/2/12 9:00	2,880	0	2,880	38,940	10	352,440	352,440	2,365	15.2	10.0
5/4/12 12:00	3,060	0	3,060	42,000	10	383,040	383,040	2,571	15.0	10.0
5/7/12 12:00	4,320	0	4,320	46,320	10	426,240	426,240	2,861	15.1	10.0
5/8/12 0:00	System Shutdown - regeneration of T18 and T25 tanks; draining of water tanks									

T18 regenerated April 24-26

Table D2. Second Stage flow rates and treatment volumes

Date / Time	Period Time (min)	Period Down-Time (min)	Period Run-Time (min)	Cumulative Run-Time (min)	Average Influent Flow Rate (GPM)	Total Influent Volume (gal)	Total Volume Treated (gal)	Bed Volumes	Average Period FM2 Flow Rate (GPM)	Average Influent Flow Rate (GPM)
3/26/12 9:00	--	--		0			0		0.0	--
3/27/12 9:00	1,440	960	0	0	6	0	0	0	14.5	0.0
3/28/12 9:00	1,440	960	0	0	6	0	0	0	13.5	0.0
3/29/12 9:00	1,440	960	0	0	6	0	0	0	13.4	0.0
3/30/12 9:00	1,440	960	0	0	10	0	0	0	14.0	0.0
4/2/12 9:00	4,320	3,300	0	0	10	0	0	0	14.0	0.0
4/3/12 9:00	1,440	960	480	480	10	4,800	4,800	32	14.0	3.0
4/4/12 9:00	1,440	960	480	960	10	9,600	6,912	46	4.4	4.0
4/5/12 9:00	1,440	960	480	1,440	5	12,000	8,976	60	4.3	5.0
4/6/12 9:00	1,440	0	1,440	2,880	5	19,200	15,024	101	4.2	5.0
4/7/12 9:00	1,440	0	1,440	4,320	5	26,400	21,072	141	4.2	5.0
4/9/12 9:00	2,880	0	2,880	7,200	5	40,800	34,032	228	4.5	5.0
4/11/12 9:00	2,880	0	2,880	10,080	10	69,600	69,600	467	13.8	11.0
4/13/12 9:00	2,880	0	2,880	12,960	10	98,400	98,400	660	14.0	11.0
4/16/12 9:00	4,320	0	4,320	17,280	10	141,600	141,600	950	14.8	11.0
4/18/12 9:00	2,880	0	2,880	20,160	10	170,400	170,400	1,144	15.3	11.0
4/19/12 11:00	1,560	0	1,560	21,720	10	186,000	186,000	1,248	14.8	10.0
4/23/12 9:00	5,640	0	5,640	27,360	10	242,400	242,400	1,627	14.7	10.0
4/24/12 9:00	1,440	1,440	0	27,360	0	242,400	242,400	1,627	0.0	10.0
4/25/12 9:00	1,440	1,440	0	27,360	0	242,400	242,400	1,627	0.0	10.0
4/26/12 9:00	1,440	1,440	0	27,360	0	242,400	242,400	1,627	0.0	10.0
4/30/12 9:00	5,760	0	5,760	33,120	10	300,000	300,000	2,013	14.8	10.0
5/2/12 9:00	2,880	0	2,880	36,000	10	328,800	328,800	2,207	14.7	10.0
5/4/12 12:00	3,060	0	3,060	39,060	10	359,400	359,400	2,412	14.8	10.0
5/7/12 12:00	4,320	0	4,320	43,380	10	402,600	402,600	2,702	14.9	10.0
5/8/12 0:00	System Shutdown - regeneration of T18 and T25 tanks; draining of water tanks									

T18 regenerated April 24-26

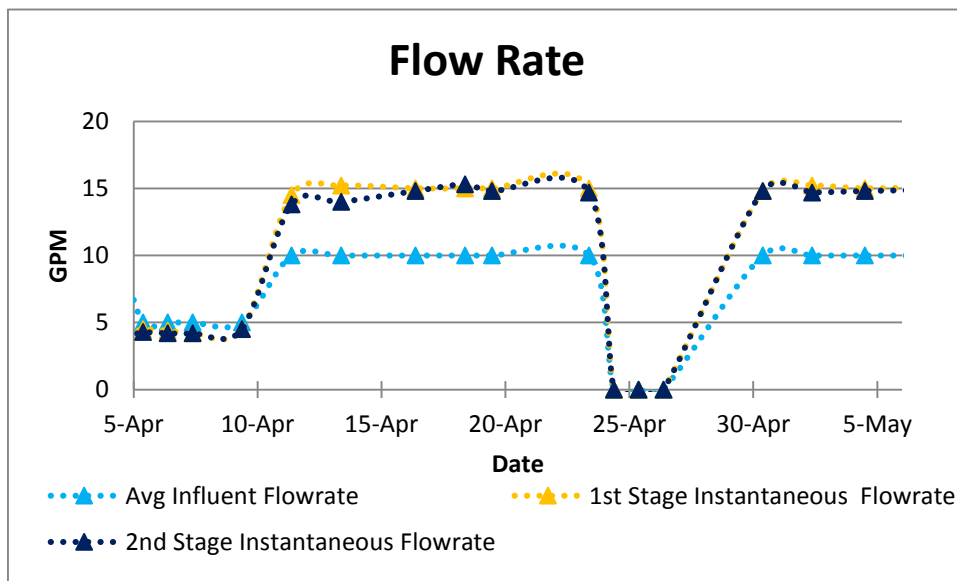


Figure D1. Flow rate versus time. Flow is zero gpm for regeneration April 24-26.

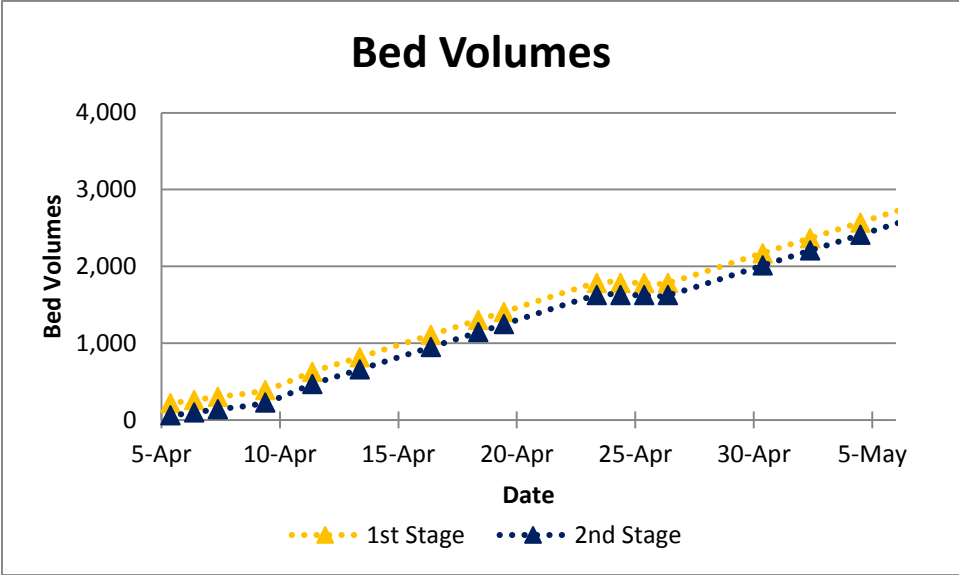


Figure D2. Bed volume versus time. Bed volume was constant during regeneration April 24-26.

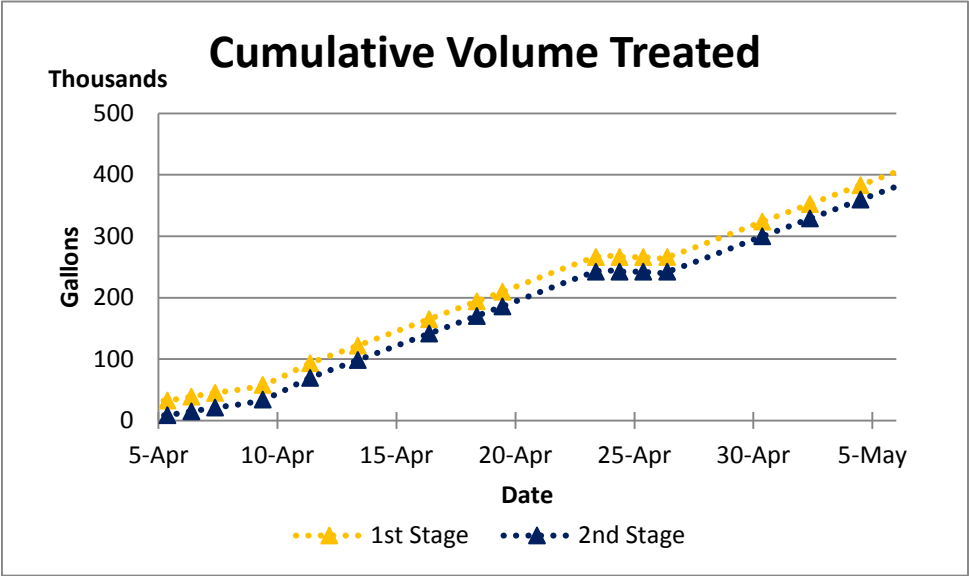


Figure D3. Cumulative volume treated over time. Cumulative volume was constant during regeneration April 24-26.

6.5 Appendix E: Field Parameters and Analytical Data for Water and Sludge Samples

Sampling Point SP-1: Virginia Canyon Influent Water

Analyte / Parameter	Sampling Date										
	4/5/2012	4/7/2012*	4/9/2012	4/11/2012*	4/16/2012	4/18/2012*	4/20/2012	4/27/2012*	4/30/2012	5/3/2012	5/4/2012*
Ag - Total (µg/l)	B		B		B		B		0.9	0.8	
Ag - Dissolved (µg/l)	0.7 (E)		0.5 (E)		1		0.9		0.8	0.7	
Al - Total (µg/l)	71,100		66,800		72,700		70,200		76,400	69,100	
Al - Dissolved (µg/l)	70,200		71,200		71,100		69,500		72,000	68,100	
As - Total (µg/l)	< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)	< 4 (RL)	
As - Dissolved (µg/l)	< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)	< 4 (RL)	
Ca - Dissolved (µg/l)	250,000		249,000		256,000		252,000		251,000	250,000	
Cd - Total (µg/l)	307		307		301		301		333	315	
Cd - Dissolved (µg/l)	304		311		306		303		326	312	
Cu - Total (µg/l)	8,540		8,620		8,710		8,840		9,290	8,820	
Cu - Dissolved (µg/l)	8,220		8,160		8,820		8,970		8,760	8,310	
Fe - Total (µg/l)	2,340		2,410		2,510		2,600		3,160	3,100	
Fe - Dissolved (µg/l)	2,260		2,470		2,570		2,690		2,920	1,680	
K - Dissolved (µg/l)	5,090		5,330		5,090		5,280		5,020	5,390	
Mg - Dissolved (µg/l)	110,000		109,000		112,000		112,000		109,000	104,000	
Mn - Total (µg/l)	77,100		71,100		78,000		75,200		82,600	78,700	
Mn - Dissolved (µg/l)	76,300		75,300		78,600		76,700		76,900	75,100	
Na - Dissolved (µg/l)	36,600		39,500		36,600		36,300		35,300	77,600	
Ni - Total (µg/l)	617		642		635		644		631	632	
Ni - Dissolved (µg/l)	611		614		632		645		616	598	
Pb - Total (µg/l)	18.0		17.8		18.6		19.4		21.0	19.8	
Pb - Dissolved (µg/l)	17.0		17.2		18.6		19.5		19.3	14.7	
Se - Total (µg/l)	15.0		15.4		18.5		18.0		17.0	17.0	
Se - Dissolved (µg/l)	14.4		14.2		18.9		17.9		14.9	14.4	
Zn - Total (µg/l)	73,300		68,000		74,800		72,500		80,300	78,800	
Zn - Dissolved (µg/l)	74,300		70,900		77,000		74,800		73,800	71,700	
Alkalinity (mg/l as CaCO ₃)	< 5 (RL)		< 5 (RL)		< 5 (RL)		< 5 (RL)		< 5 (RL)	< 5 (RL)	
Hardness (mg/l as CaCO ₃)	1,077		1,071		1,100		1,090		1,076	1,053	
Chloride (mg/l)	37.9		39.9		37.6		36.4		37.2	34.1	
Fluoride (mg/l)	2.7		3.5		4.4		3.1		4.6	< 0.2 (RL)	
Sulfate (mg/l)	1,790		1,800		1,780		1,790		1,760	1,770	
TSS (mg/l)		< 1.1 (MDL)		< 1.1 (MDL)		not measured		not measured			< 1.1 (MDL)
Acidity (test america)		650	650	660	590	520		600	610		630
Resistivity (test america)		3.7	3.6	3.6	3.8	3.8		3.5	3.6		
Specific Conductance (µS/cm)	2,663	2,700	2,729	2,800	2,736	2,700	2,787	2,800	not measured	2,838	2,900
Temp (°C)	7.08		14.12		7.47		3.63		not measured	12.39	
pH	3.80	3.53	2.95	3.36	3.23	3.56	3.19	3.51	not measured	3.47	3.59

* analyzed by Test America on unfiltered sample; pH and conductivity measured in the field by Arcadis

RL = reporting limit

MDL = method detection limit

E = QA criterion not met, value is an estimate

B = concentration in blank higher than sample value, value not reported

Sampling Point SP-1b: Post 1st Base Addition, Pre 1st Octolig® Bed

Analyte / Parameter	Sampling Date		
	4/5/2012	4/16/2012	5/3/2012
Ag - Total (µg/l)	B	1 (E)	0.8
Ag - Dissolved (µg/l)	< 0.5 (RL)	0.6	0.5
Al - Total (µg/l)	70,900	72,500	73,200
Al - Dissolved (µg/l)	70,400	69,700	72,600
As - Total (µg/l)	< 4 (RL)	< 4 (RL)	< 4 (RL)
As - Dissolved (µg/l)	< 4 (RL)	< 4 (RL)	< 4 (RL)
Ca - Dissolved (µg/l)	245,000	250,000	252,000
Cd - Total (µg/l)	299	307	335
Cd - Dissolved (µg/l)	308	304	318
Cu - Total (µg/l)	8,580	8,800	9,330
Cu - Dissolved (µg/l)	8,350	8,860	8,740
Fe - Total (µg/l)	2,330	2,570	3,300
Fe - Dissolved (µg/l)	2,290	2,560	3,020
K - Dissolved (µg/l)	5,080	5,080	5,390
Mg - Dissolved (µg/l)	106,000	110,000	107,000
Mn - Total (µg/l)	76,200	78,300	81,800
Mn - Dissolved (µg/l)	75,000	77,000	78,200
Na - Dissolved (µg/l)	36,900	35,800	36,100
Ni - Total (µg/l)	614	635	656
Ni - Dissolved (µg/l)	611	605	616
Pb - Total (µg/l)	20.1	19.2	21.6
Pb - Dissolved (µg/l)	19.1	19.0	20.0
Se - Total (µg/l)	14.9	18.4	17.1
Se - Dissolved (µg/l)	15.0	17.8	16.1
Zn - Total (µg/l)	71,400	75,500	84,100
Zn - Dissolved (µg/l)	71,300	75,500	74,300
Alkalinity (mg/l as CaCO ₃)	<5 (RL)	<5 (RL)	<5 (RL)
Hardness (mg/l as CaCO ₃)	1,048	1,077	1,070
Chloride (mg/l)	39.2	38.5	34.2
Fluoride (mg/l)	4.4	4.7	< 0.2 (RL)
Sulfate (mg/l)	1,820	1,830	1,820
Specific Conductance (uS/cm)	2,661	2,735	2,786
Temp (°C)	6.58	7.11	11.79
pH	3.05	3.14	3.64

RL = reporting limit

E = QA criterion not met, value is an estimate

B = concentration in blank higher than sample value

Sampling Point SP-2: Post 1st Octolig® Bed

Analyte / Parameter	Sampling Date									
	4/5/2012	4/7/2012*	4/9/2012	4/11/2012*	4/16/2012	4/18/2012*	4/20/2012	4/30/2012	5/3/2012	5/4/2012*
Ag - Total (µg/l)	< 0.5 (RL)		< 0.5 (RL)		< 0.5 (RL)		0.5 (E)	< 0.5 (RL)	< 0.5 (RL)	
Ag - Dissolved (µg/l)	< 0.5 (RL)		< 0.5 (RL)		< 0.5 (RL)		< 0.5 (RL)	< 0.5 (RL)	< 0.5 (RL)	
Al - Total (µg/l)	30,800		50,200		80,900		77,000	78,500	75,100	
Al - Dissolved (µg/l)	29,900		50,600		78,400		73,500	72,700	73,500	
As - Total (µg/l)	< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)	< 4 (RL)	< 4 (RL)	
As - Dissolved (µg/l)	< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)	< 4 (RL)	< 4 (RL)	
Ca - Dissolved (µg/l)	250,000		249,000		244,000		252,000	250,000	251,000	
Cd - Total (µg/l)	314		465		308		304	339	329	
Cd - Dissolved (µg/l)	311		464		293		307	316	326	
Cu - Total (µg/l)	162		280		3,390		5,040	4,750	6,570	
Cu - Dissolved (µg/l)	152		268		3,370		5,190	4,390	6,050	
Fe - Total (µg/l)	170		239		1,110		1,410	2,200	2,210	
Fe - Dissolved (µg/l)	187		215		1,030		1,420	1,920	2,070	
K - Dissolved (µg/l)	5,350		5,310		5,180		5,240	5,180	5,410	
Mg - Dissolved (µg/l)	109,000		109,000		106,000 (E)		111,000	106,000	107,000	
Mn - Total (µg/l)	84,300		73,000		79,200		76,300	83,800	80,900	
Mn - Dissolved (µg/l)	86,200		76,600		75,900		76,400	76,600	77,800	
Na - Dissolved (µg/l)	37,700		37,800		36,400		36,200	35,900	36,400	
Ni - Total (µg/l)	431		762		662		646	640	646	
Ni - Dissolved (µg/l)	432		770		620 (E)		625	616	618	
Pb - Total (µg/l)	14.5		21.6		20.7		20.2	21.7	20.9	
Pb - Dissolved (µg/l)	13.7		19.9		19.5		20.1	19.3	20.2	
Se - Total (µg/l)	16.6		18.7		18.3		19.2	16.9	16.3	
Se - Dissolved (µg/l)	15.5		17.2		17.8		18.5	14.7	15.9	
Zn - Total (µg/l)	72,500		99,200		75,800		71,700	81,000	79,900	
Zn - Dissolved (µg/l)	77,200		108,000		70,800 (E)		75,000	72,900	74,000	
Alkalinity (mg/l as CaCO ₃)	< 5 (RL)		< 5 (RL)		< 5 (RL)		< 5 (RL)	< 5 (RL)	< 5 (RL)	
Hardness (mg/l as CaCO ₃)	1,073		1,071		1,046		1,086	1,061	1,067	
Chloride (mg/l)	40.6		42.3		38		38.1	37.8	34.3	
Fluoride (mg/l)	4.3		5.2		4.5		4.6	4.6	< 0.2 (RL)	
Sulfate (mg/l)	1,520		1,740		1,820		1,810	1,810	1,790	
TSS (mg/l)		8		2.4 (J)		not measured				2
Specific Conductance (µS/cm)	2,307	2,500	2,457	2,600	2,566	2,500	2,641	not measured	2,850	2,900
Temp (°C)	5.85		11.51		6.54		8.19	not measured	12.15	
pH	4.95	5.02	4.65	4.17	3.99	4.22	3.71	not measured	3.24	3.54

* analyzed by Test America on unfiltered sample; pH and conductivity measured in the field by Arcadis

RL = reporting limit

E = QA criterion not met, value is an estimate

J = above MDL, but below RL

Sampling Point SP-5: Post 2nd Base Addition, Pre 2nd Octol

Analyte / Parameter	Sampling Date		
	4/5/2012	4/16/2012	5/3/2012
Ag - Total (µg/l)	< 0.5 (RL)	0.6 (E)	< 0.5 (RL)
Ag - Dissolved (µg/l)	< 0.5 (RL)	< 0.5 (RL)	< 0.5 (RL)
Al - Total (µg/l)	440	52,000	49,100
Al - Dissolved (µg/l)	< 100 (RL)	50,800	46,200
As - Total (µg/l)	< 4 (RL)	< 4 (RL)	< 4 (RL)
As - Dissolved (µg/l)	< 4 (RL)	< 4 (RL)	< 4 (RL)
Ca - Dissolved (µg/l)	247,000	252,000	247,000
Cd - Total (µg/l)	98.6	264	290
Cd - Dissolved (µg/l)	96.4	272	281
Cu - Total (µg/l)	546	2,220	4,710
Cu - Dissolved (µg/l)	533	2,280	4,270
Fe - Total (µg/l)	< 100 (RL)	595	1,010
Fe - Dissolved (µg/l)	< 100 (RL)	467	151
K - Dissolved (µg/l)	7,070	5,140	5,610
Mg - Dissolved (µg/l)	80,200	102,000	94,000
Mn - Total (µg/l)	33,800	65,000	67,900
Mn - Dissolved (µg/l)	35,500	67,600	65,600
Na - Dissolved (µg/l)	262,000	136,000	180,000
Ni - Total (µg/l)	195	553	530
Ni - Dissolved (µg/l)	192	557	500
Pb - Total (µg/l)	< 1 (RL)	12.7	10.1
Pb - Dissolved (µg/l)	< 1 (RL)	11.8	4.3
Se - Total (µg/l)	1.9	14.1	13.2
Se - Dissolved (µg/l)	2.3	14.0	12.1
Zn - Total (µg/l)	6,210	59,500	67,700
Zn - Dissolved (µg/l)	5,980	63,800	62,800
Alkalinity (mg/l as CaCO ₃)	33.7	< 5 (RL)	< 5 (RL)
Hardness (mg/l as CaCO ₃)	947	1,049	1,004
Chloride (mg/l)	43.9	38	34.4
Fluoride (mg/l)	2.2	3.8	< 0.2 (RL)
Sulfate (mg/l)	1,420	1,790	1,770
Specific Conductance (uS/cm)	2,599	2,687	2,902
Temp (°c)	7.12	6.3	13.05
pH	6.20	4.20	4.26

RL = reporting limit

E = QA criterion not met, value is an estimate

Sampling Point SP-3: Post 2nd Octolig® Bed

Analyte / Parameter	Sampling Date										
	4/5/2012	4/7/2012*	4/9/2012	4/11/2012*	4/16/2012	4/18/2012*	4/20/2012	4/27/2012*	4/30/2012	5/3/2012	5/4/2012*
Ag - Total (µg/l)	B		< 0.5 (RL)		< 0.5 (RL)		< 0.5 (RL)		< 0.5 (RL)	< 0.5 (RL)	
Ag - Dissolved (µg/l)	< 0.5 (RL)		< 0.5 (RL)		0.7		< 0.5 (RL)		< 0.5 (RL)	< 0.5 (RL)	
Al - Total (µg/l)	255		1,460		70,400		15,600		39,500	25,800	
Al - Dissolved (µg/l)	< 100 (RL)		141		69,800		9,160		34,300	21,600	
As - Total (µg/l)	< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)	< 4 (RL)	
As - Dissolved (µg/l)	< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)		< 4 (RL)	< 4 (RL)	
Ca - Dissolved (µg/l)	237,000		245,000		249,000		231,000		250,000	236,000	
Cd - Total (µg/l)	55.6		166		319		244		328	243	
Cd - Dissolved (µg/l)	54.5		163		321		247		309	233	
Cu - Total (µg/l)	552		109		2430		1270		2690	2710	
Cu - Dissolved (µg/l)	524		109		2470		1150		2430	2470	
Fe - Total (µg/l)	< 100 (RL)		< 100 (RL)		744		116		379	286	
Fe - Dissolved (µg/l)	< 100 (RL)		< 100 (RL)		687		< 100 (RL)		< 100 (RL)	< 100 (RL)	
K - Dissolved (µg/l)	8,680		6,230		5,220		5,410		5,390	5,370	
Mg - Dissolved (µg/l)	75,300		73,900		109,000		81,100		100,000	78,700	
Mn - Total (µg/l)	26,700		34,800		75,400		55,200		74,300	58,400	
Mn - Dissolved (µg/l)	27,600		33,200		76,700		53,100		68,600	52,900	
Na - Dissolved (µg/l)	260,000		300,000		40,400		283,000		179,000	292,000	
Ni - Total (µg/l)	163		197		634		402		554	431	
Ni - Dissolved (µg/l)	155		187		633		375		527	396	
Pb - Total (µg/l)	< 1 (RL)		< 1 (RL)		18.9		1.8		3.8	3.3	
Pb - Dissolved (µg/l)	< 1 (RL)		< 1 (RL)		18.5		< 1 (RL)		1.3	1.1	
Se - Total (µg/l)	1.9		3.1		18.2		10.8		14.5	10.4	
Se - Dissolved (µg/l)	2.3		3.4		19.0		10.6		12.7	9.7	
Zn - Total (µg/l)	3,090		16,300		72,800		49,200		73,000	58,800	
Zn - Dissolved (µg/l)	2,880		14,300		74,500		46,500		68,000	49,300	
Alkalinity (mg/l as CaCO ₃)	47.9		22.5		< 5 (RL)		< 5 (RL)		< 5 (RL)	< 5 (RL)	
Hardness (mg/l as CaCO ₃)	902		916		1,071		911		1,036	913	
Chloride (mg/l)	55.4		41.8		37.9		38.5		38.2	34.3	
Fluoride (mg/l)	2.3		< 2 (RL)		4.4		3.1		4.2	2.3	
Sulfate (mg/l)	1,380		1,550		1,790		1,710		1,740	2,490	
TSS (mg/l)		18		8.8		not measured		not measured			17
Specific Conductance (µS/cm)	2,564	2,700	2,774	2,900	2,515	2,500	2,962	3,300	not collected	3,030	3,000
Temp (°c)	7.7		12.03		7.1		8.66		not collected	13.28	
pH	6.08	7.46	5.8	5.85	3.58	4.36	4.71	6.27	not collected	4.08	4.76

* analyzed by Test America on unfiltered sample; pH and conductivity measured in the field by Arcadis

RL = reporting limit

B = concentration in blank higher than sample value, value not reported

Sludge Chemistry

Analyte / Parameter	Sampling Date		
	4/26/2012	5/4/2012	5/8/2012
Ag - Total (µg/l)			55 (J)
Al - Total (µg/l)			490,000
As - Total (µg/l)			< 44 (MDL)
Ca - Total (µg/l)			480,000
Cd - Total (µg/l)			1400
Cu - Total (µg/l)			35,000
Fe - Total (µg/l)			19,000
K - Total (µg/l)			13,000 (J)
Mg - Total (µg/l)			460,000
Mn - Total (µg/l)			380,000
Na - Total (µg/l)			800,000
Ni - Total (µg/l)			3000
Pb - Total (µg/l)			180.0
Zn - Total (µg/l)			330,000
TSS (mg/l)	9,100	8,800	8,950

MDL = method detection limit

J = above MDL, but below reporting limit (RL); RL = 100 for Ag and 30,000 for K

Average TSS reported for 5/8/12

6.6 Appendix F: Estimates of Costs for 50 GPM Octolig® and Conventional Lime Treatment Systems

The following assumptions apply to the cost estimates for the 50 GPM Octolig® treatment system (Tables F1 and F2):

- Costs are based on removal of Al, Cd, Cu, Fe, Pb, Mg, Mn, Ni, Se, and Zn at influent concentrations observed during the pilot study.
- Costs associated with regulatory negotiations are excluded.
- Capital costs are based on a treatment system with 50 GPM capacity employing two stages of Octolig® media each with clarification.
- Costs are for the treatment facility and conveyance piping only – collection systems at the source are excluded.
- Plant water is assumed to be available for washdown, polymer makedown, etc.
- Octolig® media replacement is expected to occur every 5 years, O&M schedule includes amortized cost.

The following design calculations were used to develop the costs estimated for the 50 GPM Octolig® treatment system (Tables F1 and F2):

Design Parameter	Design Value	Notes
Raw Influent Flowrate (gpm):	50.0	
Raw Influent Flowrate (MGD):	0.072	
Raw Wastewater flowrate (gpd):	72,000	
Solids Thickener Decant (gpd):	1,541	decant water = solids loading, wet minus thickened solids, wet
Filter Press Filtrate Return (gpd):	1,996	filter press filtrate = solids loading, wet minus pressed sludge, wet
Design Influent Flowrate (gpm):	52	raw influent, thickener decant, and filtrate return
Current Nominal Flowrate (gpd):	75,537	raw influent, thickener decant, and filtrate return
(MGD):	0.0755	
Inlet pH:	3.4	based on observed influent pH in pilot
Inlet Acidity (mg/L as CaCO ₃):	613.8	Buffering capacity of raw influent to a pH of approximately 8.3
CaCO ₃ Equivalanece (mg/L as CaCO ₃):	1,122	Reagent demand based on equivalent weight of metals and sulfate
Inlet Aluminum Concentration (mg/L):	71	based on average influent concentrations from pilot
Inlet Cadmium Concentration (mg/L):	0.311	based on average influent concentrations from pilot
Inlet Copper Concentration (mg/L):	9	based on average influent concentrations from pilot
Inlet Iron Concentration (mg/L):	2.7	based on average influent concentrations from pilot
Inlet Lead Concentration (mg/L):	0.0	based on average influent concentrations from pilot
Inlet Magnesium Concentration (mg/L):	109	based on average influent concentrations from pilot
Inlet Manganese Concentration (mg/L):	77.1	based on average influent concentrations from pilot
Inlet Nickel Concentration (mg/L):	0.634	based on average influent concentrations from pilot
Inlet Selenium Concentration (mg/L):	0.017	based on average influent concentrations from pilot
Inlet Zinc Concentration (mg/L):	74.617	based on average influent concentrations from pilot
Inlet Calcium Concentration (mg/L):	251	based on average influent concentrations from pilot
Inlet Potassium Concentration (mg/L):	5	based on average influent concentrations from pilot
Inlet Sodium Concentration (mg/L):	44	based on average influent concentrations from pilot
Inlet Chloride Concentration (mg/L):	37	based on average influent concentrations from pilot
Inlet Flouride Concentration (mg/L):	3.7	based on average influent concentrations from pilot
Inlet Sulfate Concentration (mg/L):	1,782	based on average influent concentrations from pilot

Influent Equalization

Detention Time (days):	1.0	assumed time for equalization of influent sources (raw influent, decant water, filtrate water)
EQ Tank Volume (gal):	80,000	based on flow rate and detention time
EQ Tank Diameter (ft):	20.0	size of tank necessary for volume
EQ Tank Sidewater Depth (ft):	34.0	actual depth of water expected
EQ Tank Sidewall Height (ft):	36.0	height of tank necessary for volume

1st Stage pH Neutralization Vessel

Reactor Detention Time (hr):	0.2	Based on nominal design flowrate
Target Reactor Tank Volume (gal):	525	flow x detention time
Reactor Tank Sidewater Depth (ft):	5.0	
Reactor Tank Length (ft):	5.0	
Reactor Tank Width (ft):	5.0	
Nominal Reactor Tank Volume (ft ³):	125.0	calculated from tank dimensions
Nominal Reactor Tank Volume (gal):	935	

1st Stage Sodium Hydroxide Dose

Target pH:	4.0	based on pH to remove copper and iron onto the Octolig without
Target Alkalinity Addition (mg/L as CaCO ₃):	100	Estimated
Target Sodium Hydroxide Dosage (mg/L):	80.0	Estimated
Sodium Hydroxide Strength (wt% NaOH):	50%	
Sodium Hydroxide Density (lbs/gal):	12.8	
Target Dosage (gals/day):	7.53	Based on raw influent flowrate
Estimated TSS (mg/L):	67	Based on raw influent flowrate assuming 10% of solids precipitate out in this step

1st Stage Octolig

No. of Vessels:	3	Series Operation
Vessel Diameter (in):	72	
Vessel Diameter (ft):	6	
Vessel Height (in):	96	
Vessel Height (ft):	8	
Vessel Volume (gal):	1,692	calculated from vessel dimensions

	<u>Each Vessel</u>	<u>Total</u>	
Octolig Media Bed (ft):	4		
Octolig Media Volume (ft ³):	113	339	
Octolig Media Volume (gal):	846	2,538	
Octolig Media SG:	0.6		
Media Bed (lbs):	4,233	12,700	
Empty Bed Contact Time (EBCT) (min):	16.1		media volume ÷ nominal flow rate
Hydraulic Loading (gpm/ft ²):	0.62		
Vessel Velocity (ft/min):	0.083		
Apsorptive Capacity (mol/kg Octolig):	0.2		
Apsorptive Capacity (lbmol/lb Octolig):	0.0004		
System Adsorptive Capacity (lbmol):	5.5946		Value for entire amount of Octolig within the 3 vessels
Influent Copper Loading (lbs/day):	4.4193		based on pilot removal efficiency, assumes all Cu in 1st stage
Influent Copper Loading (lbmols/day):	0.0696		
Influent Iron Loading (lbs/day):	1.4423		based on pilot removal efficiency, assumes all Fe in 1st stage
Influent Iron Loading (lbmols/day):	0.0258		
Total Molar Mass of Metals (lbmols/day):	0.0954		
Operating Days Until Regeneration (days):	59		based on capacity and molar mass of metals
Bed Volumes Until Regeneration (days):	1,756		flow x operating days ÷ total volume of Octolig
Copper Mass in Media at Regen (lbs):	261		
Iron Mass in Media at Regen (lbs):	85		
Percent Recovery at Reneration (wt%):	75%		Assumed recovery from the media into regenerant solution

1st Stage Regeneration/Rinse Vessel

Target Reactor Tank Volume (gal):	2,500
Reactor Tank Sidewater Depth (ft):	6.6
Reactor Diameter (ft):	8.0
Reactor Tank Sidewall Depth (ft):	9.0
Nominal Reactor Tank Volume (ft ³):	452.4
Nominal Reactor Tank Volume (gal):	3,384

Regeneration Cycle

Sulfuric Acid Strength (wt% H ₂ SO ₄):	93%
Sulfuric Acid Density (lbs/gal):	15.3

Volume of Regenerant per Cycle (gal):	5,076	3 Vessels x 1,692 gal Vessel
Acid Strength (vol%):	3%	
Acid added per batch (gal):	152	
Annual Acid Consumption (gal/yr):	942	based on number of days per year of regeneration
Annual Regenerant Volume (gal):	31,401	based on number of days per year of regeneration
Copper (mg/L):	4,619	75% recovery from Octolig assumed
Iron (mg/L):	1,508	75% recovery from Octolig assumed

Rinse Cycle

Sodium Hydroxide Strength (wt% NaOH):	0.5
Sodium Hydroxide Density (lbs/gal):	12.8

Volume of Rinsate per Cycle (gal):	7,614	Assumed 1.5 x volume of Regenerant
Base Strength (vol%):	3%	
Base added per batch (gal):	228	
Annual Base Consumption (gal/yr):	1,413	based on number of days per year of regeneration

2nd Stage pH Neutralization Vessel

Reactor Detention Time (hr):	0.2
Target Reactor Tank Volume (gal):	525
Reactor Tank Sidewater Depth (ft):	5.0
Reactor Tank Length (ft):	5.0
Reactor Tank Width (ft):	5.0
Nominal Reactor Tank Volume (ft ³):	125.0
Nominal Reactor Tank Volume (gal):	935

Based on nominal design flowrate
Based on nominal design flowrate

2nd Stage Sodium Hydroxide Dose

Target pH:	8.0
Target Alkalinity Addition (mg/L as CaCO ₃):	395
Target Sodium Hydroxide Dosage (mg/L):	316.0
Sodium Hydroxide Strength (wt% NaOH):	50%
Sodium Hydroxide Density (lbs/gal):	12.8
Target Dosage (gals/day):	29.7
Estimated TSS (mg/L):	285

Estimated based on aluminum concentration
80% of target alkalinity
Based on raw influent flowrate
Based on raw influent flowrate, concentrations and pilot observed

Flocculating Aid - Polymer Dosage

Target Concentration (mg/L):	3
Target Dosage (lbs/day):	1.89
As Delivered Polymer Density (lb/gal):	8.340
As Delivered Polymer Feed (gpd):	0.227
Neat Polymer (lbs/day):	0.009

As Delivered, made down solution, 0.5% strength
Based on nominal design flowrate

Clarifier

Influent TSS (mg/L):	285
Solids Load, dry basis (lbs/day):	171
Aluminum, dry basis (lbs/day):	32.9
Cadmium, dry basis (lbs/day):	0.0638
Lead, dry basis (lbs/day):	0.01020
Magnesium, dry basis (lbs/day):	16.28
Manganese, dry basis (lbs/day):	16.58
Nickel, dry basis (lbs/day):	0.171
Selenium, dry basis (lbs/day):	0.00526
Zinc, dry basis (lbs/day):	21.4

Based on raw influent flowrate, concentrations and pilot observed
Based on raw influent flowrate, concentrations and pilot observed
Based on pilot removal efficiency
Based on pilot removal efficiency
Based on pilot removal efficiency
Based on pilot removal efficiency
Based on pilot removal efficiency
Based on pilot removal efficiency
Based on pilot removal efficiency
Based on pilot removal efficiency

Hydraulic Loading (gpm/ft ²):	0.400	
Surface Area Required (ft ²):	131	Inclined plate clarifier
Clarifier underflow solids content (%):	1.000%	Assumed
Underflow density (lbs/gal):	8.34	
Underflow wastage, wet basis (lbs/day):	17,137	
Underflow wastage (gal/day):	2,055	
Clarifier Effluent (gpd):	73,482	Nominal flow rate minus underflow wastage
Clarifier Effluent (gpm):	51	

2nd Stage Octolig

No. of Vessels:	3	Series Operation
Vessel Diameter (in):	72	
Vessel Diameter (ft):	8	
Vessel Height (in):	96	
Vessel Height (ft):	8	
Vessel Volume (gal):	3,008	

	<u>Each Vessel</u>	<u>Total</u>	
Octolig Media Bed (ft):	6		
Octolig Media Volume (ft ³):	302	904.8	
Octolig Media Volume (gal):	2,256	6,767.7	
Octolig Media SG:	0.6		
Media Bed (lbs):	11,289	33,865.8	
EBCT (min):	43.0		media volume ÷ nominal flow rate
Hydraulic Loading (gpm/ft ²):	0.35		
Vessel Velocity (ft/min):	0.047		
Apsorptive Capacity (mol/kg Octolig):	0.2		
Apsorptive Capacity (lbmol/lb Octolig):	0.0004		
System Adsorptive Capacity (lbmol):	14.9189		Value for entire amount of Octolig within the 3 vessels
Influent Cadmium Loading (lbs/day):	0.18655		
Influent Cadmium Loading (lbmols/day):	0.0016597		
Influent Lead Loading (lbs/day):	0.0115		
Influent Lead Loading (lbmols/day):	0.0001		
Influent Manganese Loading (lbs/day):	46.3070		
Influent Manganese Loading (lbmols/day):	0.8429		
Influent Nickel Loading (lbs/day):	0.3804		
Influent Nickel Loading (lbmols/day):	0.0065		
Influent Selenium Loading (lbs/day):	0.0101		
Influent Selenium Loading (lbmols/day):	0.000128		
Influent Zinc Loading (lbs/day):	44.8058		
Influent Zinc Loading (lbmols/day):	0.685419		
Total Molar Mass of Metals (lbmols/day):	1.536637		
Operating Days Until Regeneration (days):	9.7		
Bed Volumes Until Regeneration (days):	108		nominal flow x operating days to regeneration ÷ total volume Octolig
Cadmium Mass in Media at Regen (lbs):	1.810		
Lead Mass in Media at Regen (lbs):	0.1113		
Manganese Mass in Media at Regen (lbs):	449.18		
Nickel Mass in Media at Regen (lbs):	3.69		
Selenium Mass in Media at Regen (lbs):	0.10		
Zinc Mass in Media at Regen (lbs):	434.62		
Percent Recovery at Reneneration (wt%):	75%		Assumed

2nd Stage Regeneration/Rinse Vessel

Target Reactor Tank Volume (gal):	4,000
Reactor Tank Sidewater Depth (ft):	10.6
Reactor Diameter (ft):	8.0
Reactor Tank Sidewall Depth (ft):	13.0
Nominal Reactor Tank Volume (ft ³):	653.5
Nominal Reactor Tank Volume (gal):	4,888

Regeneration Cycle

Sulfuric Acid Strength (wt% H ₂ SO ₄):	93%	
Sulfuric Acid Density (lbs/gal):	15.3	
Volume of Renerant per Cycle (gal):	9,024	3 Vessels x 1,692 gal Vessel
Acid Strength (vol%):	3%	
Acid added per batch (gal):	271	
Annual Acid Consumption (gal/yr):	10,187	
Annual Regenerant Volume (gal):	339,550	
Cadmium (mg/L):	18	
Lead (mg/L):	1.1	
Manganese (mg/L):	4,476	
Nickel (mg/L):	37	
Selenium (mg/L):	1.0	
Zinc (mg/L):	4,331	

Rinse Cycle

Sodium Hydroxide Strength (wt% NaOH):	50.0%	
Sodium Hydroxide Density (lbs/gal):	12.8	
Volume of Rinsate per Cycle (gal):	13,535	Volume of Regenerant x 1.5
Base Strength (vol%):	3%	
Base added per batch (gal):	406	
Annual Base Consumption (gal/yr):	15,280	

Solids Thickener Tank

Solids Storage Tank Volume (gal):	12,000	
Sludge density (lbs/gal):	9.0	
Sludge solids content (%):	4.0%	
Solids Loading, dry basis (lbs/day):	171	flow x influent TSS
Solids Loading, wet basis (lbs/day):	17,137	underflow wastage
Solids Loading (gpd)	2,055	
No. Days Storage (days):	5.8	
Thickened Solids, wet basis (lbs/day):	4,284.3	
Decant Water (gpd):	1,541	

Filter Press

Cake solids, dry basis (%):	35%	
Cake density (lb/ft ³):	70	
Minimum Filter Press Size (ft ³):	7	Based on solids loading, dry
Pressed sludge, wet (lbs/day):	490	solids loading, dry ÷ by cake solids, %
(ton/day):	0.24	
(ton/yr):	89	
Aluminum (mg/kg):	67,268	
Cadmium (mg/kg):	130.3	
Lead (mg/kg):	20.8	
Magnesium (mg/kg):	33,253	
Manganese (mg/kg):	33,858	
Nickel (mg/kg):	349	
Selenium (mg/kg):	10.74	
Zinc (mg/kg):	43,649	
Filtrate (gpd):	1,996	solids loading, wet minus pressed sludge, wet

Coagulant (Sludge Conditioning)

Target Coagulant Concentration (mg/L):	200	
Target Coagulant Concentration (lbs/day):	7.1	based on target concentration and thickened solids, wet
Coagulant Feed (gpd):	0.86	

Polymer (Sludge Conditioning)

Target Polymer Concentration (lbs/day):	1.7	1% per dry lb solids
Polymer Feed (gpd):	0.21	

Effluent Equalization

Detention Time (days):	1.0	
EQ Tank Volume (gal):	80,000	
EQ Tank Diameter (ft):	20.0	
EQ Tank Sidewater Depth (ft):	34.0	
EQ Tank Sidewall Height (ft):	36.0	

Table F1. Octolig® with conventional coagulation / flocculation treatment: Total capital investment cost.

Purchased Equipment Costs	QTY	Unit	Unit Cost	Extension	Description
Inclined Plate Clarifier	1	Lump	\$75,000.00	\$75,000.00	Includes flash mix tank, flocculation tank, catwalk and appurtenances 20-ft diameter, 36-ft high, bolted glass lined steel tank with roof, ladders, safety platform, and appurtenances
Equalization Tanks	2	Lump	\$120,000.00	\$240,000.00	
1st Stage Octolig Skid	1	Lump	\$175,000.00	\$175,000.00	Three (3) 6-ft Diameter Reactor Vessels with 4-ft of Octolig, Neutralization Vessel, Regenerant/Rinse Vessel and appurtenances, chemical day tanks
2nd Stage Octolig Skid	1	Lump	\$225,000.00	\$225,000.00	Three (3) 8-ft Diameter Reactor Vessels with 6-ft of Octolig, Neutralization Vessel, Regenerant/Rinse Vessel and appurtenances, chemical day tanks
Bulk Acid Storage Tank	1	Lump	\$30,000.00	\$30,000.00	
Bulk Caustic Storage Tank	1	Lump	\$30,000.00	\$30,000.00	
Octolig Media	1,244	ft ³	\$300.00	\$373,221.21	
Chemical Feed System	1	Lump	\$12,500.00	\$12,500.00	Flocculant
Sludge Pumps	2	Lump	\$5,000.00	\$10,000.00	Progressive cavity pumps
Filter Press	1	Lump	\$65,000.00	\$65,000.00	
Filter Press Feed Pump	1	Lump	\$4,000.00	\$4,000.00	
Filter Press Polymer and DE systems	1	Lump	\$12,500.00	\$12,500.00	
Polymer Delivery System (Dynablend or equivalent)	1	Each	\$12,000.00	\$12,000.00	Nominal 14,000 gallon tank with catwalk, sludge rake, and appurtenances
Sludge Storage Tank	1	Each	\$35,000.00	\$35,000.00	
Piping, valves, and appurtenances	1	Lump	\$75,000.00	\$75,000.00	
Outfall	1	Lump	\$25,000.00	\$25,000.00	
Instrumentation	1	Lump	\$25,000.00	\$25,000.00	
Power drop	1	Each	\$15,000.00	\$15,000.00	
Motor Control Center	1	Lump	\$50,000.00	\$50,000.00	
Control Panels / SCADA System	1	Each	\$150,000.00	\$150,000.00	
Equipment Subtotal (EQ):				\$1,639,221.21	
Taxes (5% of EQ)	1	Lump	\$81,961.06	\$81,961.06	
Freight (2% of EQ)	1	Lump	\$32,784.42	\$32,784.42	
Total Purchased Equipment Cost (PEC):				\$1,753,966.69	
Direct Installation Costs	QTY	Unit	Unit Cost	Extension	Description
Mobilization, Demobilization, Permits, and Temporary Controls	1	Lump	\$40,000.00	\$40,000.00	
Treatment Building Foundation and Building Erection	2,500	sf	\$200.00	\$500,000.00	50 ft x 50 ft Pre-Engineered Building
Conveyance Force mains to Distribution Build.	5,280	ft	\$50.00	\$264,000.00	
Imported Backfill	750	ton	\$22.00	\$16,500.00	Pipe Bedding as Necessary
Set Process Equipment (8% of EQ)	1	Lump	\$131,137.70	\$131,137.70	
Process Piping (15% of EQ)	1	Lump	\$163,922.12	\$163,922.12	
Heat trace and insulation (2% of EQ)	1	Lump	\$32,784.42	\$32,784.42	
Painting (1.5% of EQ)	1	Lump	\$24,588.32	\$24,588.32	
Heating and Lighting (8% of EQ)	1	Lump	\$131,137.70	\$131,137.70	
Site Security (4.5% of EQ)	1	Lump	\$73,764.95	\$73,764.95	
Electrical (15% of EQ)	1	Lump	\$245,883.18	\$245,883.18	
Total Direct Installation Cost (DI):				\$1,623,718.39	
TOTAL DIRECT COST (DC) [PEC + DI]:				\$3,377,685.08	
Indirect Costs	QTY	Unit	Unit Cost	Extension	Description
Engineering (6% of DC)	1	Lump	\$202,661.10	\$202,661.10	
Administration/PM (5% of DC)	1	Lump	\$168,884.25	\$168,884.25	
Geotechnical	1	Lump	\$15,000.00	\$15,000.00	
Bonds (1.5% DC)	1	Lump	\$50,665.28	\$50,665.28	
Construction Oversight (6% of DC)	1	Lump	\$202,661.10	\$202,661.10	
Start Up	1	Lump	\$25,000.00	\$25,000.00	
Contractor Profit (10% of PEC)	1	Lump	\$175,396.67	\$175,396.67	
Contingencies (5% of DC)	1	Lump	\$168,884.25	\$168,884.25	
Total Indirect Cost (IC):				\$1,009,152.66	
TOTAL CAPITAL INVESTMENT (TCI) [DC + IC]:				\$4,386,837.75	

Table F2. Octolig[®] with conventional coagulation / flocculation treatment: Total annual operating costs.

Direct Annual Operating Costs	QTY	Unit	Unit Cost	Extension	Description
Maintenance and Replacement Parts (4% EQ)	1	Lump	\$65,568.85	\$65,568.85	
Laboratory Analytical	26	Biweekly	\$500.00	\$13,000.00	pilot
Sampling/Operator & Maintenance Labor (OL)	2,080	Staff Hours	\$85.00	\$176,800.00	40 hours per week, 52 weeks per year; loaded cost
Octolig Media Replacment	1	Lump	\$75,000.00	\$75,000.00	
Polymer	690	lb	\$3.50	\$2,414.40	based on target dose of flocculant
Coagulant	2,608	lb	\$3.50	\$9,129.32	coagulant
Sodium Hydroxide (50%)	30,297	gal	\$3.50	\$106,038.06	based on target doses in 2 stages and regeneration rinsing steps
Sulfuric Acid	11,129	gal	\$3.50	\$38,949.88	Assumed based on 600 ppm dosage, 90% strength (~0.2 TPD).
1st Stage Regnerant	31,401	gal	\$0.00	\$0.00	dispose, but no value assumed for recycling
2nd Stage Regenerant	339,550	gal	\$0.00	\$0.00	dispose, but no value assumed for recycling
Sludge Disposal	89	ton	\$75	\$6,701.90	Assumed 35% solids filter cake
Sludge Transportation & Demurrage	8.9	Load	\$700	\$6,255.10	
Electricity (1,780 kW-hr/day assumed)	649,700	kW-hr	\$0.12	\$77,964.00	Equivalent to 100 brake horsepower
Total Direct Annual Operating Cost (DAC):				\$577,821.51	
Indirect Annual Operating Costs	QTY	Unit	Unit Cost	Extension	Description
Overhead & Administrative Charges (1.0% TC)	1	Lump	---	Excluded ---	
Property Taxes (1.0% TC)	1	Lump	---	Excluded ---	
Insurance (1.0% TC)	1	Lump	---	Excluded ---	
Total Indirect Annual Operating Cost (IAC):				\$0.00	
TOTAL ANNUAL COST (TAC) [DAC + IAC]:				\$577,821.51	

The following assumptions apply to the comparative cost estimates for a 50 GPM conventional lime treatment system (Tables F3 and F4):

- Treatment train consists of a 20-ft diameter clarifier/thickener; quicklime (CaO) is dosed to precipitate metals and gypsum (CaSO₄) if/when sulfate concentrations exceed 2,000 mg/l.
- Stoichiometric lime dose to yield a pH of 10-11; recarbonation step to be employed to reduce pH to < 9 to facilitate discharge to meet NPDES permit.
- Costs are based on removal of Al, Cd, Cu, Fe, Pb, Mg, Mn, Ni, Se, and Zn at influent concentrations observed during the pilot study.
- Costs associated with regulatory negotiations are excluded.
- Capital costs are based on a treatment system with 50 GPM capacity employing a solids contact, upflow clarifier.
- Costs are for the treatment facility and conveyance piping only – collection systems at the source are excluded.
- Plant water is assumed to be available for washdown, polymer makedown, etc.

The following design calculations were used to develop the costs estimated for the 50 GPM lime treatment system (Tables F3 and F4):

Design Parameter	Design Value	Notes
Raw Influent Flowrate (gpm):	50.0	
Raw Influent Flowrate (MGD):	0.072	
Raw Wastewater flowrate (gpd):	72,000	
Solids Thickener Decant (gpd):	1,012	decant water = solids loading, wet minus thickened
Filter Press Filtrate Return (gpd):	1,481	filter press filtrate = solids loading, wet minus pressed
Design Influent Flowrate (gpm):	52	raw influent, thickener decant, and filtrate return
Current Nominal Flowrate (gpd):	74,493	raw influent, thickener decant, and filtrate return
(MGD):	0.0745	
Inlet pH:	3.4	based on observed influent pH in pilot
Inlet Acidity (mg/L as CaCO ₃):	613.8	Buffering capacity of raw influent to a pH of approximately 8.3
Stoichiometric Equivalance (mg/L as CaCO ₃):	1,122	Reagent demand based on equivalent weight of metals and sulfate
Inlet Aluminum Concentration (mg/L):	71	based on average influent concentrations from pilot
Inlet Cadmium Concentration (mg/L):	0.311	based on average influent concentrations from pilot
Inlet Copper Concentration (mg/L):	9	based on average influent concentrations from pilot
Inlet Iron Concentration (mg/L):	2.7	based on average influent concentrations from pilot
Inlet Lead Concentration (mg/L):	0.0	based on average influent concentrations from pilot
Inlet Magnesium Concentration (mg/L):	109	based on average influent concentrations from pilot
Inlet Manganese Concentration (mg/L):	77.1	based on average influent concentrations from pilot
Inlet Nickel Concentration (mg/L):	0.634	based on average influent concentrations from pilot
Inlet Selenium Concentration (mg/L):	0.017	based on average influent concentrations from pilot
Inlet Zinc Concentration (mg/L):	74.617	based on average influent concentrations from pilot
Inlet Calcium Concentration (mg/L):	251	based on average influent concentrations from pilot
Inlet Potassium Concentration (mg/L):	5	based on average influent concentrations from pilot
Inlet Sodium Concentration (mg/L):	44	based on average influent concentrations from pilot
Inlet Chloride Concentration (mg/L):	37	based on average influent concentrations from pilot
Inlet Flouride Concentration (mg/L):	3.7	based on average influent concentrations from pilot
Inlet Sulfate Concentration (mg/L):	1,782	based on average influent concentrations from pilot

Influent Equalization

	assumed time for equalization of influent sources (raw
Detention Time (days):	1.0 influent, decant water, filtrate water)
EQ Tank Volume (gal):	70,000 based on flow rate and detention time
EQ Tank Diameter (ft):	20.0 size of tank necessary for volume
EQ Tank Sidewater Depth (ft):	29.8 actual depth of water expected
EQ Tank Sidewall Height (ft):	36.0 height of tank necessary for volume

Reactor Vessel

Reactor Detention Time (hr):	0.3 Based on nominal design flowrate
Target Reactor Tank Volume (gal):	776 flow x detention time
Reactor Tank Sidewater Depth (ft):	5.0
Reactor Tank Length (ft):	5.0
Reactor Tank Width (ft):	5.0
Nominal Reactor Tank Volume (ft ³):	125.0 calculated from tank dimensions
Nominal Reactor Tank Volume (gal):	935

Quicklime Dose

Target pH:	10.5
Target Alkalinity Addition (mg/L as CaCO ₃):	1,122 lime precipitation
Target Quicklime Dosage (mg/L):	628
Quicklime Strength (wt% Ca):	90%
Target Dosage (tons/day):	0.2
Estimated TSS (mg/L):	Based on raw influent flowrate assuming all ions 675 precipitate as hydroxides

Flocculating Aid - Polymer Dosage

Target Concentration (mg/L):	3 As Delivered, made down solution, 0.5% strength
Target Dosage (lbs/day):	1.86 Based on nominal design flowrate
As Delivered Polymer Density (lb/gal):	8.340
As Delivered Polymer Feed (gpd):	0.223
Neat Polymer (lbs/day):	0.009

Clarifier

Influent TSS (mg/L):	Based on raw influent flowrate and concentrations 675 assuming all ions precipitate as hydroxides
Solids Load, dry basis (lbs/day):	Based on raw influent flowrate and concentrations 405 assuming all ions precipitate as hydroxides
Aluminum, dry basis (lbs/day):	42.7 Based on pilot removal efficiency
Cadmium, dry basis (lbs/day):	0.187 Based on pilot removal efficiency
Copper, dry basis (lbs/day):	5.29 Based on pilot removal efficiency
Iron, dry basis (lbs/day):	1.61 Based on pilot removal efficiency
Lead, dry basis (lbs/day):	0.0115 Based on pilot removal efficiency
Magnesium, dry basis (lbs/day):	65.7 Based on pilot removal efficiency
Manganese, dry basis (lbs/day):	46.3 Based on pilot removal efficiency
Nickel, dry basis (lbs/day):	0.380 Based on pilot removal efficiency
Selenium, dry basis (lbs/day):	0.0101 Based on pilot removal efficiency
Zinc, dry basis (lbs/day):	44.8 Based on pilot removal efficiency
Clarifier Diameter (ft):	18
Clarifier Sidewater Depth (ft):	15.0
No. of Clarifiers:	1.0
Hydraulic Loading (gpd/ft ²):	293 Target <400 gpd/ft ²
Hydraulic Loading (gpm/ft ²):	0.203
Solids Loading (lbs/day-ft ²):	1.6 Target less than 20 lbs/day-ft ²
Weir Overflow (gal/day-ft):	1,317 Target less than 10,000 gal/day-ft

Clarifier Volume (MG)	0.029	
Design basin solids inventory, dry basis (lbs)	200	Clarifier volume x TSS (mg/L)
Design sludge blanket (ft):	2	
Design clarifier solids (gal):	3,807	Based on volume of sludge blanket
Design clarifier solids (lbs):	971	Based on % solids and density of sludge
Clarifier underflow solids content (%):	3.0%	
Underflow density (lbs/gal):	8.5	
Underflow Wastage required, dry basis (lbs/day)	405	
Underflow wastage (vol% of Influent Flow):	2.1%	
Underflow wastage, wet basis (lbs/day):	13,509	
Underflow wastage, wet basis (tons/day):	6.8	
Underflow wastage (gpm):	1.1	
Underflow wastage (gpd):	1,589	
Underflow wastage (MGD):	0.002	
Underflow wastage (MG/yr):	0.6	
Upflow Recycle (Recycle to Influent TSS Ratio):	33.1	20:1 to 30:1 recycle to influent solids ratio, dry basis
Upflow Recycle (Recycle to Influent Flow Ratio):	6	4:1 to 6:1 recycle to influent flow
Upflow Recycle, dry basis (lbs/day):	13,409	
Upflow Recycle, wet basis (tons/day):	1,900	
Upflow Recycle (gpm):	310.4	
Upflow Recycle per clarifier (gpm)	310.4	
Upflow Recycle (gpd):	446,960	
Upflow Recycle (MGD):	0.4	
Clarifier Effluent (gpd):	72,904	
Clarifier Effluent (gpm):	51	

Solids Thickener Tank

Solids Storage Tank Volume (gal):	12,000
Sludge density (lbs/gal):	9.0
Sludge solids content (%):	8.0%
Solids Loading, dry basis (lbs/day):	405
Solids Loading, wet basis (lbs/day):	13,509
Solids Loading (gpd)	1,589
No. Days Storage (days):	7.6
Thickened Solids, wet basis (lbs/day):	5,065.9
Decant Water (gpd):	1,012

Filter Press

Cake solids, dry basis (%):	35%	
Cake density (lb/ft ³):	70	
Minimum Filter Press Size (ft ³):	17	Based on underflow wastage required, dry
Pressed sludge, wet (lbs/day):	1,158	solids loading, dry ÷ by cake solids, %
(ton/day):	0.58	
(ton/yr):	211	
Aluminum (mg/kg):	36,846	
Cadmium (mg/kg):	161	
Copper (mg/kg):	4,565	
Iron (mg/kg):	1,393	
Lead (mg/kg):	9.91	
Magnesium (mg/kg):	56,699	
Manganese (mg/kg):	39,992	
Nickel (mg/kg):	329	
Selenium (mg/kg):	8.72	
Zinc (mg/kg):	38,695	
Filtrate (gpd):	1,481	solids loading, wet minus pressed sludge, wet

Coagulant (Sludge Conditioning)

Target Coagulant Concentration (mg/L):	200	
Target Coagulant Concentration (lbs/day):	8.4 wet	based on target concentration and thickened solids,
Coagulant Feed (gpd):	1.01	

Polymer (Sludge Conditioning)

Target Polymer Concentration (lbs/day):	4.1 [1% per dry lb solids]
Polymer Feed (gpd):	0.49

Effluent Equalization

Detention Time (days):	1.0
EQ Tank Volume (gal):	70,000
EQ Tank Diameter (ft):	20.0
EQ Tank Sidewater Depth (ft):	29.8
EQ Tank Sidewall Height (ft):	36.0

Table F3. Conventional lime treatment: Total capital investment cost.

Purchased Equipment Costs	QTY	Unit	Unit Cost	Extension	Description
Clarifier (Bridge and Internals)	1	Lump	\$50,000.00	\$50,000.00	20-ft diameter
Clarifier Tank	1	Lump	\$75,000.00	\$75,000.00	20-ft diameter, 15-ft sidewall height, ring foundation to be installed on a concrete base
Equalization Tanks	2	Lump	\$105,000.00	\$210,000.00	20-ft diameter, 36-ft high, bolted glass lined steel tank with roof, ladders, safety platform, and appurtenances
Paste Slaker/Slurry Tank/Slurry Pumps/Super Sacks	1	Lump	\$115,000.00	\$115,000.00	2,000-lb supersacks will be employed to deliver bulk pebbled quicklime
Chemical Feed System	1	Lump	\$12,500.00	\$12,500.00	Flocculant
Carbonic Acid System	1	Lump	\$250,000.00	\$250,000.00	Packaged Pressurized CO2 Delivery System
Sludge Pumps	2	Lump	\$5,000.00	\$10,000.00	Progressive cavity pumps
Filter Press	1	Lump	\$112,500.00	\$112,500.00	Based on sludge characteristics and amount
Filter Press Feed Pump	1	Lump	\$4,000.00	\$4,000.00	
Filter Press Polymer and DE systems	1	Lump	\$12,500.00	\$12,500.00	
Polymer Delivery System (Dynablend or equivalent)	1	Each	\$12,000.00	\$12,000.00	Nominal 14,000 gallon tank with catwalk, sludge rake, and appurtenances
Sludge Storage Tank	1	Each	\$35,000.00	\$35,000.00	
Piping, valves, and appurtenances	1	Lump	\$75,000.00	\$75,000.00	
Outfall	1	Lump	\$25,000.00	\$25,000.00	
Instrumentation	1	Lump	\$25,000.00	\$25,000.00	
Power drop	1	Each	\$15,000.00	\$15,000.00	
Motor Control Center	1	Lump	\$50,000.00	\$50,000.00	
Control Panels / SCADA System	1	Each	\$75,000.00	\$75,000.00	
Equipment Subtotal (EQ): \$1,163,500.00					
Taxes (5% of EQ)	1	Lump	\$58,175.00	\$58,175.00	
Freight (2% of EQ)	1	Lump	\$23,270.00	\$23,270.00	
Total Purchased Equipment Cost (PEC): \$1,244,945.00					
Direct Installation Costs	QTY	Unit	Unit Cost	Extension	Description
Mobilization, Demobilization, Permits, and Temporary Controls	1	Lump	\$40,000.00	\$40,000.00	
Treatment Building Foundation and Building Erection	1,250	sf	\$200.00	\$250,000.00	25 ft x 50 ft Pre-Engineered Building
Clarifier Foundation and Erection	1	Lump	\$75,000.00	\$75,000.00	
Clarifier Roof	314	sq. ft.	\$45.00	\$14,137.17	Fiberglass
Equalization Tank Foundation and Erection	2	Lump	\$35,000.00	\$70,000.00	
Conveyance Forcemaains to Distribution Build.	5,280	ft	\$50.00	\$264,000.00	
Imported Backfill	750	ton	\$22.00	\$16,500.00	Pipe Bedding as Necessary
Set Process Equipment (8% of EQ)	1	Lump	\$93,080.00	\$93,080.00	
Process Piping (15% of EQ)	1	Lump	\$116,350.00	\$116,350.00	
Heat trace and insulation (2% of EQ)	1	Lump	\$23,270.00	\$23,270.00	
Painting (1.5% of EQ)	1	Lump	\$17,452.50	\$17,452.50	
Heating and Lighting (8% of EQ)	1	Lump	\$93,080.00	\$93,080.00	
Site Security (4.5% of EQ)	1	Lump	\$52,357.50	\$52,357.50	
Electrical (15% of EQ)	1	Lump	\$174,525.00	\$174,525.00	
Total Direct Installation Cost (DI): \$1,299,752.17					
TOTAL DIRECT COST (DC) [PEC + DI]: \$2,544,697.17					
Indirect Costs	QTY	Unit	Unit Cost	Extension	Description
Engineering (6% of DC)	1	Lump	\$152,681.83	\$152,681.83	
Administration/PM (5% of DC)	1	Lump	\$127,234.86	\$127,234.86	
Geotechnical	1	Lump	\$15,000.00	\$15,000.00	
Bonds (1.5% DC)	1	Lump	\$38,170.46	\$38,170.46	
Construction Oversight (6% of DC)	1	Lump	\$152,681.83	\$152,681.83	
Start Up	1	Lump	\$25,000.00	\$25,000.00	
Contractor Profit (10% of PEC)	1	Lump	\$124,494.50	\$124,494.50	
Contingencies (5% of DC)	1	Lump	\$127,234.86	\$127,234.86	
Total Indirect Cost (IC): \$762,498.33					
TOTAL CAPITAL INVESTMENT (TCI) [DC + IC]: \$3,307,195.50					

Table F4. Conventional lime treatment: Total annual operating costs.

Direct Annual Operating Costs	QTY	Unit	Unit Cost	Extension	Description
Maintenance and Replacement Parts (4% EQ)	1	Lump	\$46,540.00	\$46,540.00	
Laboratory Analytical	26	Biweekly	\$500.00	\$13,000.00	
Sampling/Operator & Maintenance Labor (OL)	2,080	Staff Hours	\$85.00	\$176,800.00	40 hours per week, 52 weeks per year
Polymer	3,764	lb	\$3.50	\$13,175.71	
Coagulant	3,084	lb	\$3.50	\$10,794.68	
CO2	55,482	lb	\$0.50	\$27,740.89	Assumed based on 250 ppm dosage
Quicklime (CaO)	76	ton	\$200.00	\$15,297.70	Assumed based on 600 ppm dosage, 90% strength (~0.2 TPD).
Sludge Disposal	211	ton	\$75	\$15,848.89	Assumed 35% solids filter cake.
Sludge Transportation & Demurrage	21.1	Load	\$700	\$14,792.29	
Electricity (1,780 kW-hr/day assumed)	649,700	kW-hr	\$0.12	\$77,964.00	Equivalent to 100 brake horsepower.
Total Direct Annual Operating Cost (DAC):				\$411,954.15	
Indirect Annual Operating Costs	QTY	Unit	Unit Cost	Extension	Description
Overhead & Administrative Charges (1.0% TC)	1	Lump	---	Excluded ---	
Property Taxes (1.0% TC)	1	Lump	---	Excluded ---	
Insurance (1.0% TC)	1	Lump	---	Excluded ---	
Total Indirect Annual Operating Cost (IAC):				\$0.00	
TOTAL ANNUAL COST (TAC) [DAC + IAC]:				\$411,954.15	