*Forward looking.* Lateral thinking.

# Minto Mine Constructed Wetland Treatment Research Program – Demonstration Scale 2015 Update

Document – 011_0316_03B

**Prepared for**
Minto Explorations Ltd.
Capstone Mining Corp.
#13 Calcite Business Centre
151 Industrial Road
Whitehorse, Yukon
Y1A2V3

**Prepared by:**
Contango Strategies Limited
15-410 Downey Road
Saskatoon, SK
S7N4N1

Date:
March 2016

**Table of Contents**

## 1. Introduction and Background

The Minto Mine, operated by Capstone Mining Corp., is located 240 km northwest of Whitehorse on the west side of the Yukon River. The Minto property lies within the eastern part of the Dawson Range, with elevations from 700 to 1,000 m; the landscape has rounded mountains intersected by broad valleys and drainages that are part of the Yukon River watershed.

The Minto Mine has been in commercial operation since October 2007 and the deposits being mined are copper sulphide mineralized zones. Surface and groundwater water quality is a key consideration in the evaluation of potential effects of mining and mineral development projects and changes to water quality parameters have the potential to affect aquatic and human use of water resources. A Reclamation and Closure Plan (RCP) is required under both the Water Licence and the Quartz Mining Licence. The RCP is intended to address the long-term physical and chemical stability of the site and closure of the proposed features and disturbances associated with the mine. As a part of the RCP, a Constructed Wetland Treatment System (CWTS) is being designed, evaluated, and optimized for water treatment at closure through a phased program (Minto Phase V/VI Expansion Project, YOR Project Number 2013-0100).

In order for CWTSs to be effective, they must be designed, piloted, optimized, implemented, and maintained in a site-specific manner. A scaled approach for CWTS implementation allows for improvement, optimization, and flexibility for modifications along each step. Phases include: 1) site assessment and information gathering, 2) technology selection and conceptual design, 3) pilot-scale testing and optimization (controlled environment), 4) on-site demonstration-scale confirmation and optimization, and 5) full-scale implementation. Phases 1-3 have been completed (reports 2013-0100-256 and 2013-0100-257 on YESAB registry, and Contango, March 2014; Contango, November 2014) and confirmed plant amenability to transplantation and the CWTS design for further on-site testing. During pilot-scale trials, the selected CWTS design achieved on average 92% removal of copper (mean influent 146 µg/L, outflow 11.3 µg/L) and 41% removal of selenium (mean influent 10.2 µg/L, outflow 6 µg/L) using synthetic influent designed to mimic the worst-case water chemistry of a long-term closure scenario, but tested under the controlled conditions (e.g., controlled flows, known temperature, etc) of an off-site treatability testing center (Contango, November 2014). It should be noted that lower influent concentrations will have a lower percent removal even when achieving the same final outflow concentrations.

Phase 4 of the project is underway, with the on-site demonstration scale CWTS constructed at the Minto Mine during fall 2014 (Contango, March 2015). This document reports on the on-site demonstration scale CWTS data from construction through 2015.

## 2. Construction

### 2.1. System layout and dimensions

The demonstration-scale CWTS includes 2 systems in parallel with 2 cells in each series and a final catchment basin that both systems flow into (Figure 1). Dimensions and construction details are available in the Minto Demonstration Scale Report Document 011_0315_01A (Contango, March 2015). The two parallel systems serve as a replicate for data analysis, and as testing has progressed, the two systems have also allowed for comparison of different management techniques. Dimensions of the systems are provided here in Table 1.

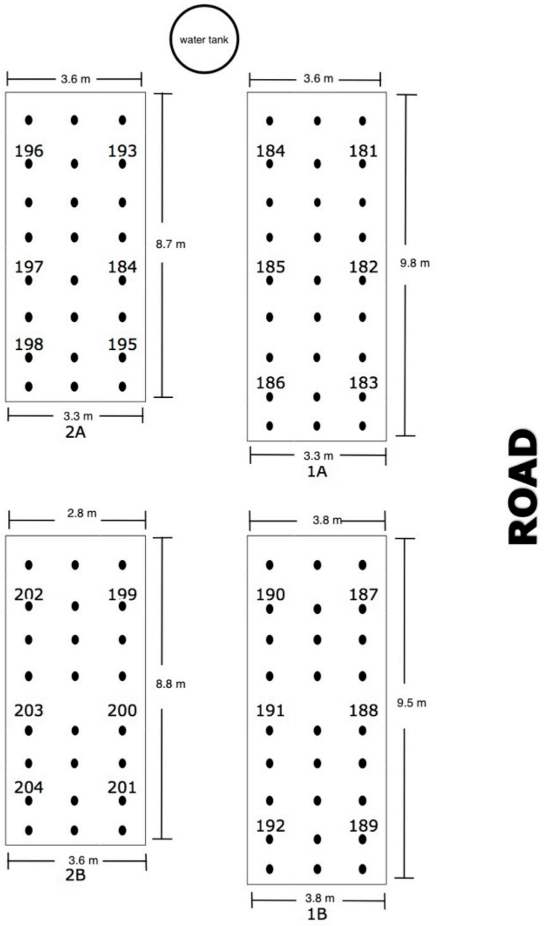

**Figure 1 – Diagram of demonstration-scale CWTS.**
Dimension measurements are indicated at soil surface. Black dots indicate initial construction grid marked by moss stakes, and locations of soil redox probes with identifying numbers.

**Table 1. Measurements of demonstration-scale CWTS cells at soil surface and resultant areas of treatment systems.**

| Measurement | | Series 1 | | Series 2 | |
|---|---|---|---|---|---|
| | | 1A | 1B | 2A | 2B |
| Width (m) | Inflow | 3.6 | 3.8 | 3.6 | 2.8 |
| | Outflow | 3.3 | 3.8 | 3.3 | 3.6 |
| Length (m) | | 9.8 | 9.5 | 8.7 | 8.8 |
| Approximate surface area at soil ($m^2$) | | 33.8 | 36.1 | 30.0 | 28.2 |
| Total area of system at soil ($m^2$) | | 69.9 | | 58.2 | |

### 2.2.Substrate Soils in Construction

Soils used to construct the CWTS are described in the initial report that outlines construction (Contango, March 2015). In brief, the recommended soil for the CWTS was sand, with 2-7% by volume as organic material (e.g., woodchips, peat). In the pilot-scale systems, this resulted in a total organic carbon (TOC) content by weight of 0.2-0.6% (the sand itself was at 0.1% TOC prior to adding amendment). Ideally, this number could be higher, approximately 2-10% by weight to stimulate the desired reducing conditions. For the demonstration-scale system, the soil added to each of the 4 cells during construction was from a local borrow site. As is expected in a mining area, the soils are likely mineralized. Although a potential borrow source was tested prior to construction ("Tested Soil"; Table 2), a different borrow source was available upon construction of the demonstration-scale wetland ("Soil Used"; Table 2). The material used in the construction of the demonstration-scale wetland was an organic peat, and analyses received after construction indicated an elevated concentration of leachable copper (Table 2). It should be noted that in a full-scale system, the variability in soil substrate would be normalized by the larger volume of soil used. The substrate in the wetland (composed of the organic peat soil, wood chips, and straw) had a TOC content of 1.8-3.1%. The implications of this are discussed in Section 4 if this report.

**Table 2. Comparison of Copper Concentrations in Soils of Pilot- and Demonstration-Scale CWTS**

| Test Method | Pilot-scale | Demonstration-scale | |
|---|---|---|---|
| | Initial Soil | Tested Soil (June, 2014) | Soil Used (August, 2014) |
| SPLP Copper (mg/L)[1] | - | 0.00546-0.0296[2] | 0.148-0.608 |
| Total Copper (mg/kg) | 5.3-5.5 | 210-1400[2] | 960-1400[3] |

[1] SPLP - Synthetic Precipitation Leaching Procedure

[2] For the June 2014 samples, the soil with the highest total copper concentration also had the lowest leachable copper concentration, and was therefore deemed acceptable for use.

[3] Total copper value for soils used was taken in June 2015 (no data for August 2014).

### 2.3. Vegetation Used in Planting

The demonstration-scale CWTS was planted with *Carex aquatilis* (aquatic sedge) and aquatic mosses from the W10 area of the Minto Site. The plant selection and borrow source was previously determined through the site assessment (reports 2013-0100-256 and 2013-0100-257 on the YESAB registry) and pilot-scale testing (Contango, November 2014). Five *C. aquatilis* were planted per square meter, with moss tied to stakes that outlined the 1 m x 1 m grid for planting (details provided in Contango, March 2015).

### 2.4. Water source

Water from the W36 area receiving seepage from the toe of the Mill Valley Fill Extension (MVFE) was selected for the demonstration-scale CWTS testing as the leachate is similar to that expected upon closure in the MVFE area. The chemistry of this water at the time of bringing the demonstration-scale CWTS online (September 18, 2014) is provided in Contango, March 2015.

After construction, the flow rates for the systems were planned to be set to have a 10 day nominal hydraulic retention time (HRT), meaning water entering the wetland takes 10 days to exit. This is referred to as a nominal HRT because it is a calculation based on the size of the wetland and the amount of water entering, and not confirmed empirically using tracing dyes. This HRT is much longer than what is necessary to achieve treatment based on the pilot-scale systems (~3 days), and was chosen to facilitate plant establishment and maturation. However, due to the leachable copper concentrations in the soils used for construction, it was instead decided to run the systems at a faster flow to wash as much leachable copper from the soils as possible. As such, a shorter nominal HRT of ~20 hrs was used as a starting point (HRT calculated using measured water depths of approximately 15 cm and negligible pore water involvement [30cm of soil at 10% pore volume] due to peat soils). The actual HRT will be confirmed by a tracer study in 2016 (Section 8). Despite the shorter HRT resulting from the faster flow of water, the systems have acclimated and matured as was expected for the longer HRT. Because the sizes of the CWTS systems are slightly different, Series 1 (closer to the road) and Series 2 (further from road) are set with flow rates to result in similar HRTs in each system. Flow rates were monitored and adjusted throughout 2015 based on CWTS establishment and maturation (Sections 3 and 4).

## 3. Commissioning

The time period between the construction of the CWTS and achieving the expected treatment performance is referred to as the commissioning period. This period is needed for operational adjustments to be made (e.g., raising water depth, modifying outflow patterns), and for plant and microbial populations to establish and mature. Based on pilot-scale testing (Contango, November 2014), the estimated commissioning period for the Minto demonstration-scale CWTS is 4 months of operation (i.e., period when water is flowing). In 2014, the demonstration-scale CWTS operated for less than one month prior to freezing, and was restarted along with spring thaw (freshet) in mid-May 2015. Based on these timelines, September 2015 was month 4 of commissioning, and the demonstration-scale system is trending towards performance as expected during the commissioning period.

Functionality similar to that of the matured pilot-scale systems is expected to occur by summer 2016.

Operational adjustments made in 2015 include:

- Increasing water depth
- Adding more aquatic moss
- Modifying inflow and outflow distribution
- Removing *Carex utriculata* (a small quantity was erroneously planted)
- Wrapping inflow tank to prevent algae growth

While full treatment functionality of the demonstration-scale system is not expected during the commissioning period, there is important data that should be collected during this period as it applies to the full-scale system in the following ways:

- evaluation of construction effectiveness and optimizations that could be made.
- assessment of timelines to reach targeted operational parameters to allow for effective phasing of implementation (e.g., soil redox, percentage and abundance of sulphate reducing bacteria).
- Effectiveness of *Carex aquatilis* and aquatic moss transplantation to assess planting density, time period to full density, and if plant propagation and/or a replanting schedule is necessary.

## 4. Timeline and Sampling Schedule

Major events and operational adjustments are listed in Table 3. The actual dates as well as relative days of operation are provided, which adjusts for time that the CWTS was not receiving water as it was frozen. The relative days of operation allows for comparison to expected timelines from the pilot-scale testing, and for planning and scheduling to be done for full-scale construction and commissioning in the context of mine closure planning.

The sampling schedule for 2015 was conceptually developed prior to beginning construction of the demonstration-scale CWTS (Appendix A). Actual dates of sampling were dependent on timing of spring thaw and winter freeze-up, and the associated ability to have the pumps operating at the W36 pond to supply water to the demonstration-scale CWTS.

**Table 3. Events and sampling activities since construction**

| Event | Key Activity | Flow Rate Setting m³/day (gal/min) | | Calendar Date | Day of Operation |
|---|---|---|---|---|---|
| | | CWTS Series 1 | CWTS Series 2 | | |
| CWTS constructed and planted | First sampling, water started. | - | - | August 27 – 31, 2014 | 0-4 |
| Freeze up for winter | Feed water pumps turned off. | - | - | September 19, 2014 | 23 |
| Start up for 2015 | Feed water pumps turned on. | 14.17 (2.60) | 11.61 (2.13) | May 16, 2015 | 24 |
| Contango Site Visit #1 | Microbiology, soils, water tested. *Carex* stem counts. Added more aquatic moss. Put black wrap on water tank to prevent algal growth. Water depth adjusted with sandbags. | | | June 18, 2015 | 57 |
| Flow rate increased | Flow rates increased. | 17.44 (3.20) | 15.81 (2.90) | July 13, 2015 | 82 |
| Contango Site Visit #2 | Microbiology, soils, water tested. Added more aquatic moss. | | | August 16, 2015 | 116 |
| Contango Site Visit #3 | Water tested. Started Fe-EDTA test on System 2. | | | September 17, 2015 | 148 |
| | Microbiology, soils, plants, water tested. | | | September 18, 2015 | 149 |
| Fe-EDTA Testing | Daily total and dissolved copper analysis conducted at Minto. | | | September 19 – 26, 2015 | 150-157 |
| Freeze up for Winter | Feed water pumps turned off. | - | - | September 29, 2015 | 160 |

## 5. Monitoring Explanatory Parameters

Explanatory parameters are quantifiable aspects of a CWTS environment that can be used to assess feasibility of treatment for a range of constituents, and therefore 'explain' the performance of a CWTS. These parameters, which often include acidity, alkalinity, conductivity, dissolved oxygen (DO), pH, oxidation reduction potential (ORP), ion balance, available electrons donors (e.g., organic carbon, reduced elements), and temperature, can be used to predict, promote, and/or optimize the ability of the system to treat different constituents (Haakensen et al., 2015). A YSI ProPlus meter was used in the field to test for water temperature, DO, conductivity (and specific conductivity; SPC), pH, and ORP.

Average water temperature of the demonstration-scale CWTS was 12.9°C, ranging from 0.5°C to 25°C through 2015. As would be expected, both the month of testing and time of day were found to affect temperature variation. DO concentrations of the CWTS feed and cells were on average 10 mg/L, which is higher than the pilot-scale systems (average 4.8 mg/L), which make it more difficult to carry out nitrate and selenium treatment, and sulphide production for copper and cadmium treatment. The conductivity, pH and ORP of the demonstration-scale CWTS cells are all very similar to the pilot-scale systems.

An additional explanatory parameter that can be used to monitor maturation of the wetland during the commissioning period is the soil redox potential, which is measured using platinum tip probes (in soil) and Calomel electrodes (in water). This measurement offers insight into the direction of electron flux between the sediment/soil/pore water and overlying water column (Faulkner et al., 1989; Huddleston & Rodgers, 2008), and can be used to confirm reducing conditions in the soil. Based on the information gathered in pilot-scale testing, the targeted soil redox for the demonstration-scale CWTS is between -100 and -250 mV. In these redox ranges, bacterial sulphide-production through reduction of sulphur compounds (e.g., sulphate) is expected. Sulphide production directly results in metals and metalloid treatment for elements such as cadmium, copper, molybdenum, nickel, lead, and zinc by precipitation as metal sulphides (Figure 2).

This maturation period is necessary for sufficient quantities of microbes to populate the wetland and become active in decomposing organic material. It is the electrons produced by the decomposition of organic material that is reported by the soil redox measurements. The decomposition of organic material then feeds the sulphate-reducing bacteria the type of energy they need to produce the sulphides that remove the copper from the water. The microbial activity of the system is discussed further in Section 6.5.

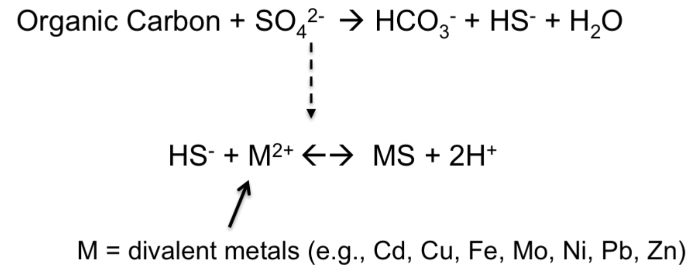

**Figure 2 – Example equation of sulphate reduction**
Equation is not balanced; demonstrates sulphate reduction with creation of alkalinity and sulphides, which can then form metal sulphide complexes.

As expected from the pilot-scale testing, the soil redox in all of the demonstration-scale CWTS cells decreased over time, indicating maturation of the system (Figure 2). At the end of 2015, only Series 1 had begun to achieve targeted soil redox values that are conducive to sulphide production (ahead of anticipated schedule). It is possible that Series 1 had more organic material in the soils than Series 2, because of how construction occurred and this wasn't reflected in the small sample size sent for analytical testing.

Some soil redox probes were reporting negative values in the targeted range by July 18, 2015 (87 days); however, in general the CWTS took approximately 4 months of operation to establish generally reducing conditions. By September 17, 2015 (day 148), most of the soil redox probes within Series 1 were reporting redox values within the targeted performance range, with Series 2 still trending downwards but not yet within the targeted range. In comparison, the pilot-scale systems were stable and reducing within approximately 4 months of construction (Figure 2).

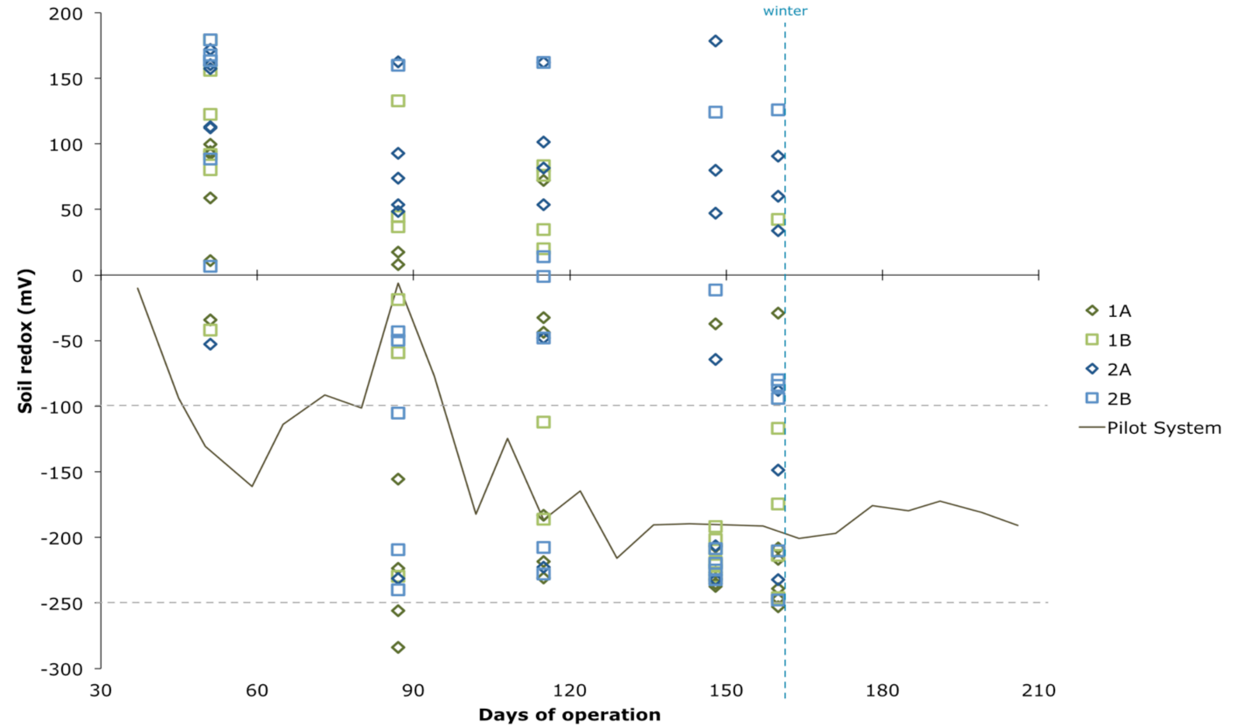

**Figure 3 – Soil redox potential of each CWTS cell over time**

All demonstration-scale CWTS soil redox measurements are plotted, along with the average soil redox of the pilot-scale systems. Targeted soil redox values based on pilot-scale testing are indicated with dotted lines. The blue dotted line indicates winter 2015.

## 6. Performance progress during commissioning period

### 6.1. Soils

To assess the effect of copper in the soils on CWTS functionality, three soil analytical test methods were used:

- total concentration of elements in the soils by ICP
- leachable concentrations of elements in the soils by Synthetic Precipitation Leaching Procedure (SPLP), which is a method to assess the mobility of elements in soils at the pH of rain water (i.e., if the wetland were to entirely dry out, then be subjected to leaching by rain water)
- concentration of elements in chemical extractions by sequential ICP-MS to assess the stability and form of elements in soils (Table 4)

**Table 4. Summary of extractable fractions from sequential ICP-MS analysis** [1]

| Fraction | Description | Elements Unstable when |
|---|---|---|
| **1** | Exchangeable fraction for adsorbed minerals | Readily released (i.e., soluble and exchangeable) |
| **2** | Mineral fraction bound to carbonates | Decreased pH |
| **3** | Mineral fraction bound to Fe-Mn oxides | Reducing conditions |
| **4** | Mineral fraction bound to organic matter and sulphides | Oxidizing conditions |
| **5** | Residual mineral fraction (primary and secondary minerals) | Not expected to be released in solution over time under conditions normally encountered in nature |

[1] Method based on Tessier *et al.,* 1979

Although unintentional, the use of soils in the CWTS with high initial leachable copper concentrations (Table 2) allowed for additional types of testing to be carried out on these systems. Because the soil substrates used for construction of the CWTS were from overburden sources, the copper was not in a mineral form that would typically be found in a reducing CWTS (i.e., negative relative soil redox). Therefore, there has been some initial leaching of copper (and other elements, such as aluminum) from the soils into the water (Figure 5 and Figure 7).

While the total copper concentrations have not changed significantly, the leachable copper (measured by SPLP) has decreased over time. By day 58 of operation, the top 5 cm of soil was decreased to only 10-20% of the initial leachable copper concentration, and the lower 10-20 cm layers reached similar concentrations by day 149 (Table 5). Analysis of the soils by ICP-MS with sequentially extracted acid analysis shows that despite elevated initial leachable copper concentrations, the soils have become more stable (less leachable) over time in the wetland setting as the soils have aged (Figure 4). This beneficial aging of soils to less soluble (sulphide) mineralized form is expected for this type of treatment wetland design. It should be noted that due to the starting soil substrate containing leachable copper and other metals such as aluminum (Appendices A and B) that these elements are leaching from the substrate into the water, putting additional treatment demands on the systems. It is recommended that for construction of the full-scale systems, soils with low total and leachable copper concentrations should be used.

**Table 5. Total and Leachable Soil Copper Concentrations in First Year of Operations.**

| Cell | Sampling Date | Days in Operation | Sample Depth (cm) | Total Cu (mg/kg) | SPLP Cu (mg/L) |
|---|---|---|---|---|---|
| **CWTS 1A** | 28-Aug-14 | 1 | Initial Composite | - | 0.298 (0.148-0.608) |
| | 19-Jun-15 | 58 | 0-5 | 960 | 0.055[1] |
| | 16-Aug-15 | 116 | 10-20 | 950 | 0.187 |
| | 18-Sep-15 | 149 | 10-20 | 1300 | 0.049 |
| | 29-Sep-15 | 160 | 10-20 | 910 | 0.069 |
| **CWTS 1B** | 28-Aug-14 | 1 | Initial Composite | - | 0.298 (0.148-0.608) |
| | 19-Jun-15 | 58 | 0-5 | 1400 | 0.033[1] |
| | 16-Aug-15 | 116 | 10-20 | 1400 | 0.209 |
| | 18-Sep-15 | 149 | 10-20 | 830 | 0.065 |
| | 29-Sep-15 | 160 | 10-20 | 880 | 0.059 |
| **CWTS 2A** | 28-Aug-14 | 1 | Initial Composite | - | 0.298 (0.148-0.608) |
| | 19-Jun-15 | 58 | 0-5 | 1175 | 0.037[1] |
| | 16-Aug-15 | 116 | 10-20 | 660 | 0.139 |
| | 18-Sep-15 | 149 | 10-20 | 880 | 0.081 |
| | 29-Sep-15 | 160 | 10-20 | 1000 | 0.073 |
| **CWTS 2B** | 28-Aug-14 | 1 | Initial Composite | - | 0.298 (0.148-0.608) |
| | 19-Jun-15 | 58 | 0-5 | 1100 | 0.039[1] |
| | 16-Aug-15 | 116 | 10-20 | 1000 | 0.201 |
| | 18-Sep-15 | 149 | 10-20 | 830 | 0.078 |
| | 29-Sep-15 | 160 | 10-20 | 540 | 0.059 |

[1] Samples collected in June 2015 were at a shallow depth (0-5 cm) and copper content had therefore likely already been removed by washing from the faster flows of the CWTS system. The aging of the deeper (10-20 cm) soil copper concentrations over time to a less soluble form is shown in Figure 4.

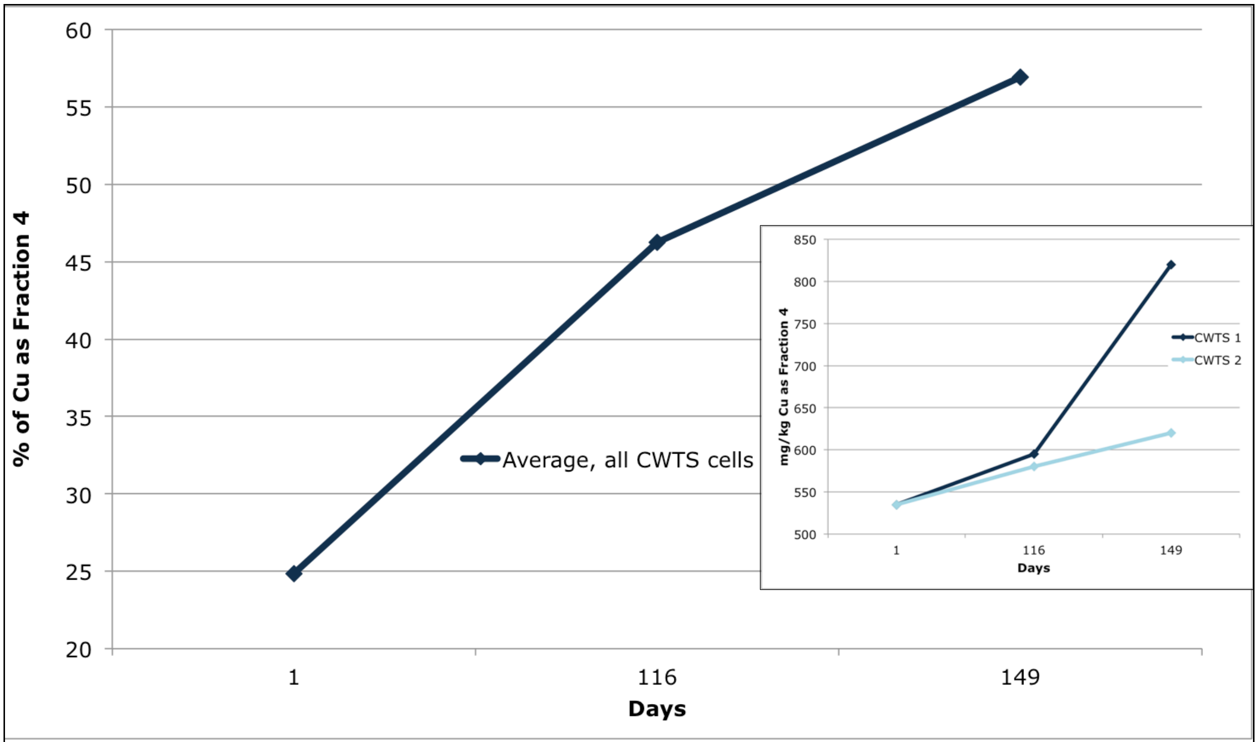

**Figure 4 – Copper as sequential leach fraction 4 over time.**
Copper in the form of the targeted Fraction 4 (sulphide mineral form and bound to organics), increased in the CWTS over time as the soils matured. Additional information in Appendix B.

### 6.2.Water

Pilot-scale testing was performed with water that mimicked the worst-case long-term post-closure water chemistry (mean influent concentrations of 146 µg/L copper and 10.2 µg/L selenium). In contrast, the on-site demonstration-scale system uses water from the W36 pond and therefore has inflow concentrations that are currently occurring during operations (mean influent concentrations of 58 µg/L copper and 6 µg/L selenium). Because of this, a lower percent removal will achieve the same extent of removal and associated outflow concentrations than was required in the pilot-scale systems. For example, the pilot-scale system achieved on average 92% removal of copper (mean outflow of 11.3 µg/L) and 41% removal of selenium (mean outflow of 6 µg/L). To achieve these same outflow concentrations with the demonstration-scale system, 80% removal of copper and minimal (or no) removal of selenium would be needed.

The treatment of copper has been improving through commissioning as the system has matured (Figure 7). However, as shown in Figure 5 and Appendix B, several elements are being released through the wetland by the soils, such as aluminum, cadmium, and copper (discussed further in Section 6.1). Because of this, negative percent removals of copper have been observed, especially in early operations (Figure 7). It was initially theorized that this was occurring when the May and June 2015 data reported outflow concentrations higher than the influent. This paired with the soils data (Section 6.1 and Appendix A) led to the design of a new sampling scheme to determine whether detectable levels of metals were being released from the soils within the wetland. For two timepoints (August 15, 2015 and

September 17, 2015), water was sampled not only at the feed and the outflow of each cell, but also through the cell. Beginning at the outflow of cell B and working upwards towards the feed, samples were taken at the B cell outflow, 1 m upstream of the B cell outflow, B cell mid point, 1 m downstream of the B cell inflow, and then the same 4 points for the A cell (Figure 6). In each case, the sample was taken from within a reaching distance of the side shore to ensure that sediments were not suspended in sampling.

It was found that significant metals concentrations were leaching from the wetlands soils of all cells (Figure 5). Moreover, metal types and concentrations (e.g., Aluminum, Appendix B) that could not be accounted for by the influent water chemistry were elevated at random points within the wetland. This suggested that because of metals leaching from the soils, the treatment occurring within the wetlands was far greater than what was being observed by simply measuring the inflow and outflow points (Figure 7). For example, in August of 2015, influent copper concentrations were measured as 50 µg/L, but within the first meter of cell 1A increased to 100 µg/L, and by the outflow of cell A, down to 42 µg/L (58 µg/L decrease), they then increased to 70 µg/L at the beginning of cell B, and again were treated to a final outflow concentration was 40 µg/L (30 µg/L) for a total of at least 88 µg/L removed by Series 1 of the treatment wetland. This suggests the wetland is actually achieving much greater copper treatment (88 µg/L removed), than would be suggested by only measuring the inflow and outflow of the system (suggests 10 µg/L removed). Similar leaching was observed through the wetland during testing in September 2015.

Copper, cadmium, zinc, and other metals and metalloids are being noticeably decreased in concentration from the water entering the wetlands (Figure 7 and Appendix B), while other metals/metalloids of potential concern are below background water quality for the area or below the detection limit in the influent of the wetland. Dissolved zinc had on average 65% removal in the wetland systems and by September 2015, the outflow concentrations were on average 20% and 50% lower than influent concentrations for dissolved copper and cadmium, respectively. This suggests that not only is the wetland maturing as expected, creating reducing sulphide-producing conditions, but that it is already performing far beyond what was anticipated from the design, sequestering orders of magnitude more copper than would be apparent by looking at the influent water alone (i.e., is also removing copper that has leached from the soils over time).

Removal rate coefficients (*k*) based on the HRT of each system would normally be calculated to compare treatment efficiency. However, because of the leaching of various elements from the soil, calculation of *k* for constituents of concern is not representative of what is ongoing in the system, as there is concurrent leaching and treatment occurring throughout a given cell (Figure 5). Furthermore, because the soil used for construction of the demonstration-scale system is different than what was used for pilot-scale systems, the hydrology and pore volume of the system is not known; a parameter that is needed for accurate calculation of the HRT and therefore *k*. As the same wetland substrate was used to construct both demonstration-scale systems, the removal rate coefficients can be estimated and compared between these two systems; however, comparison with removal rates achieved during pilot-scale testing is not possible until the HRT is defined. It is therefore recommended that a tracer study be performed in 2016 to assess the hydrology and pore volume of the CWTS (see Section 8).

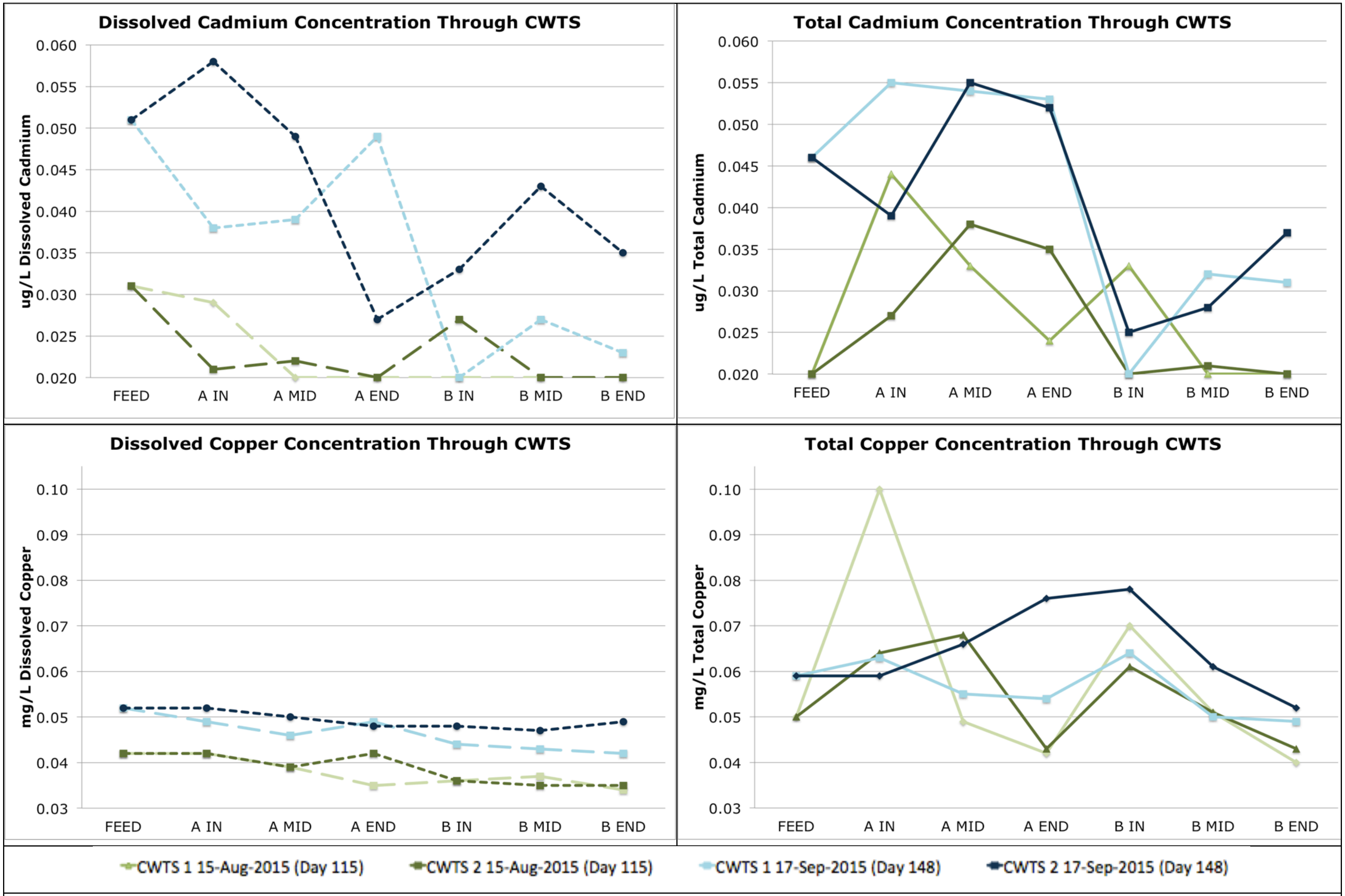

**Figure 5 – Concentrations of key elements through the CWTS.**

The y-axis values start at the detection limits.

Continued next page.

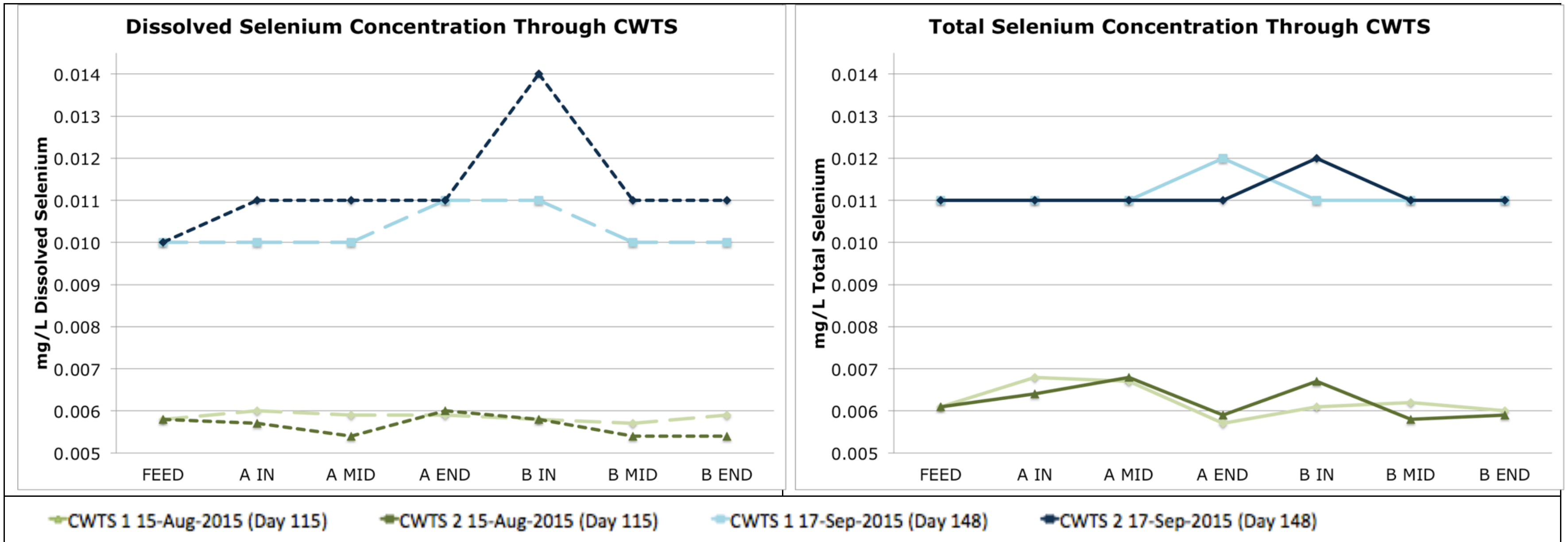

**Figure 5 – Continued**

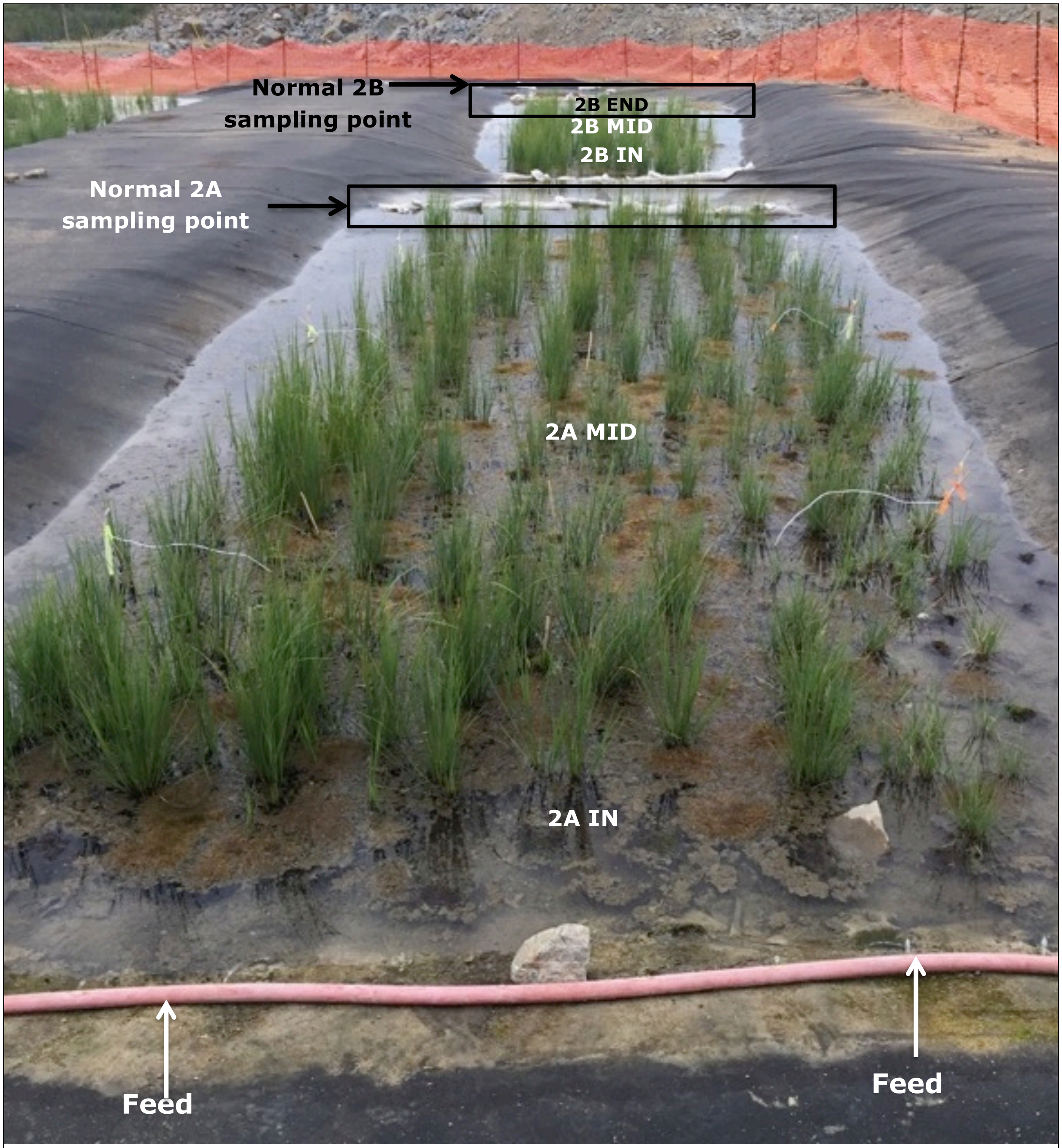

**Figure 6 – Sampling points through the CWTS.**

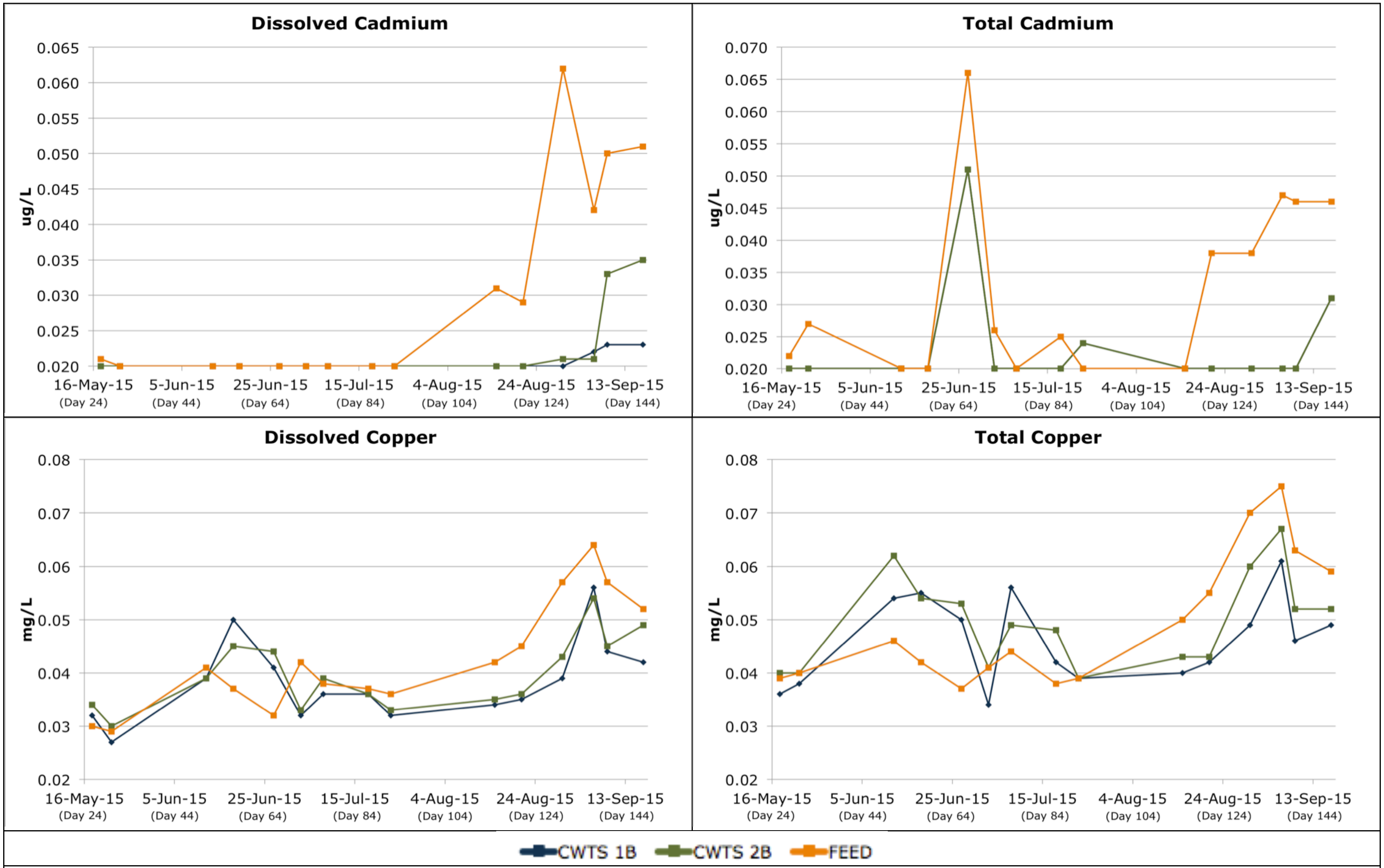

**Figure 7 – Element concentrations in the demonstration scale wetland from May 17, 2015 to Sept 17, 2015.**
The Measurement Uncertainty (MU) for cadmium is +/- 0.020 µg/L. The MU for copper and selenium is +/- 0.0002 mg/L. Timepoints where the dissolved cadmium concentration is higher than the total concentration are within the limit of uncertainty (Maxxam Analytics). Detection limits for cadmium, copper, and selenium are at or below y-axis values.

Continued next page.

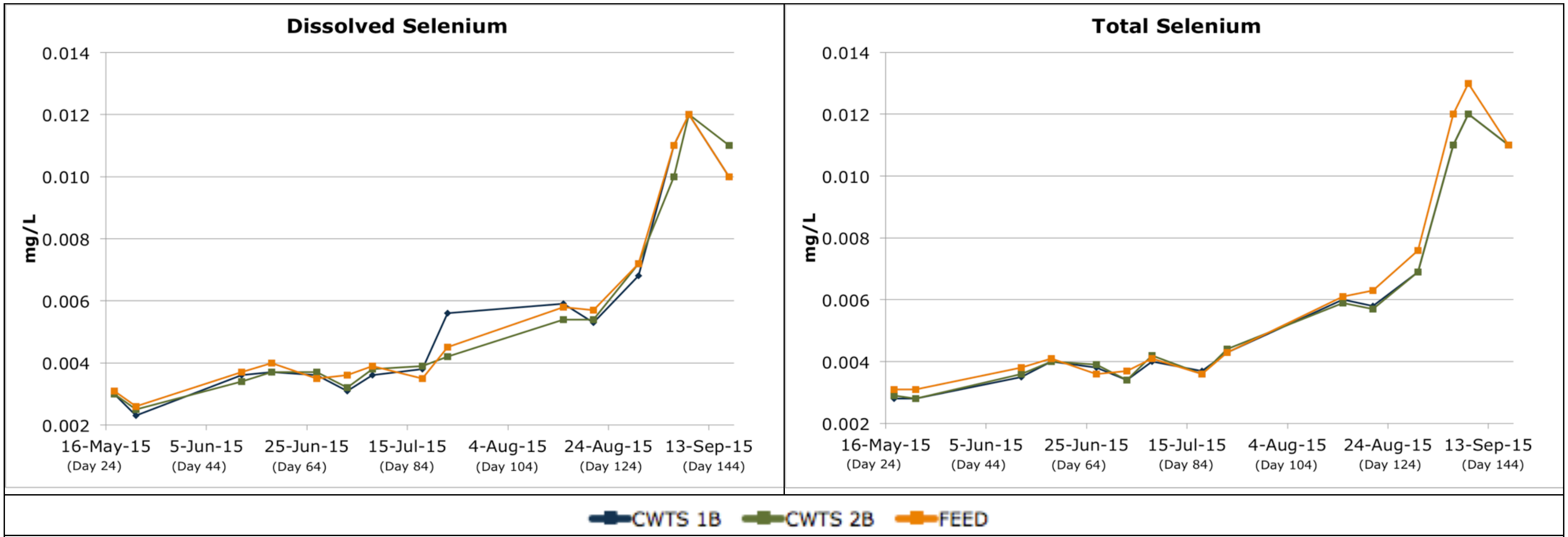

**Figure 7 -** Continued

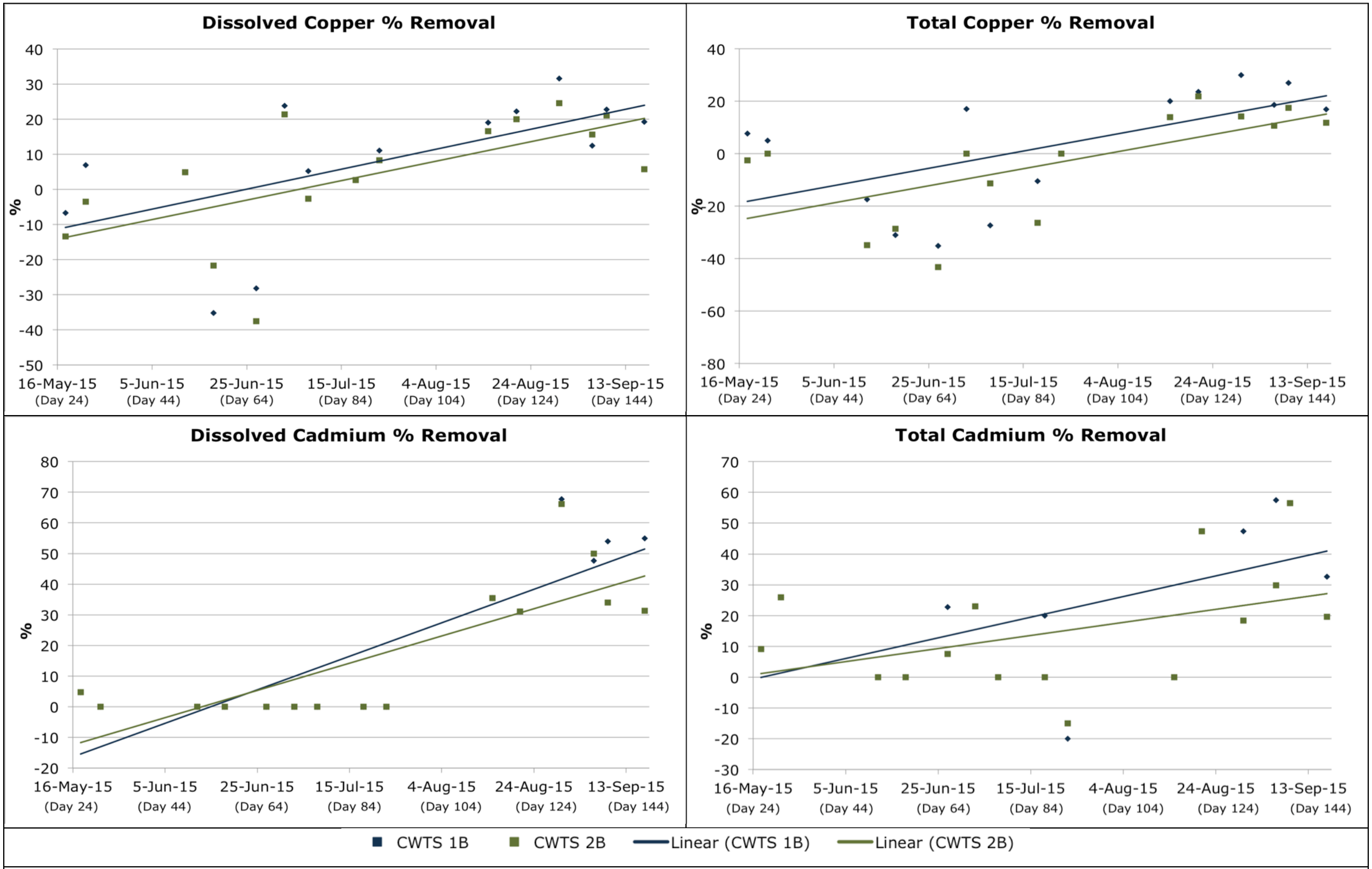

**Figure 8 – Percent removal of elements in the demonstration scale wetland from May 17, 2015 to Sept 17, 2015.** If the concentration was below the detection limit of the assay, the detection limit was used to calculate the percent removal. Note the Measurement Uncertainty (MU) for cadmium was +/- 0.020 µg/L. The MU for copper and selenium is +/- 0.0002 mg/L (Maxxam Analytics) (Appendix A). The percent removal calculations for cadmium are therefore estimations within this range of error.

Continued next page

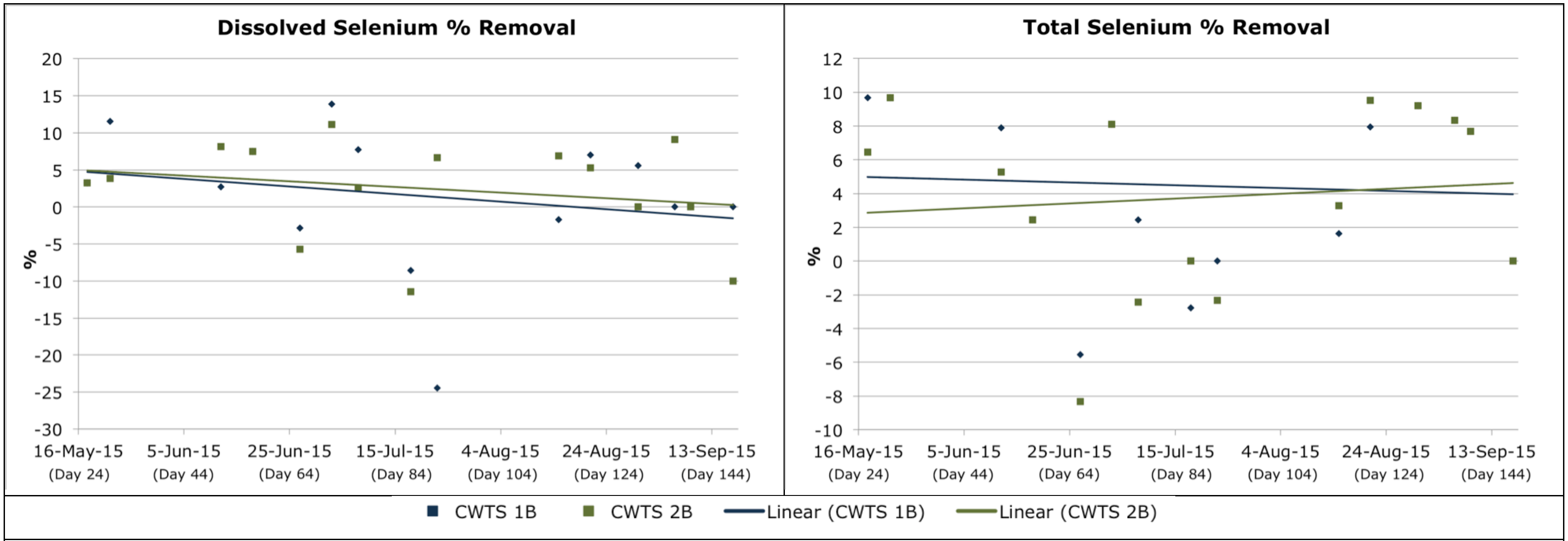

**Figure 8 – Continued**

## 6.3.Chelated iron (fertilizer micronutrient) treatment

Because elevated metals were present in the soils used for construction, there was an opportunity to assess whether non-invasive treatments could be used to further leach the metals and improve soil quality. A chelated iron (Fe-EDTA) product was added to the Series 2 wetland on September 17, 2015. The product selected is Fe-EDTA called Plant-Prod 13.2% Iron Chelate (containing 68% of the chelating agent Ethylenediaminetetraacetic acid (EDTA)), and is routinely used in the agricultural industry as a micronutrient supplement for iron-deficient soils. Fe-EDTA is also commonly used in remediation of metals contaminated soils (e.g., Thayalakumaran *et al.,* 2003).

The Fe-EDTA dissociates based on the pH of the water, and the free EDTA will be available to bind other elements such as copper and remove them from the soils, leaving the Fe in the wetland. Fe-EDTA was chosen instead of fertilizer with EDTA (which is also a commonly used agricultural product), as the fertilizer would provide additional nitrogen sources, which can interfere with selenium treatment, and phosphate which could cause an algal bloom that would be detrimental to the active water treatment facility at Minto that receives the CWTS outflow water. The Fe-EDTA treatment was performed on Series 2, with Series 1 remaining untreated for comparison purposes.

After addition of Fe-EDTA, copper concentrations in the wetland water for both CWTS Series 1 and 2 were monitored daily by Minto’s internal laboratory. Increases in copper concentrations were observed in the outflow from both cells in CWTS Series 2 after the addition of Fe-EDTA (Figure 9). Unfortunately, the CWTS feed copper concentration also increased, as the outflow from the demonstration-scale wetlands reports to the same area as the feed water draws from at the W36 area. As such, the leached copper (and presumably, also EDTA) is recirculating through the wetland (including Series 1).

In the 10 days following the addition of Fe-EDTA, approximately 75 g of copper was removed from the wetland Series 2. This is approximately 1.25 g of copper per m$^2$ of wetland. Testing of the soils at different depths in Spring 2016 will determine how much copper has been removed from the wetland as a result of this treatment.

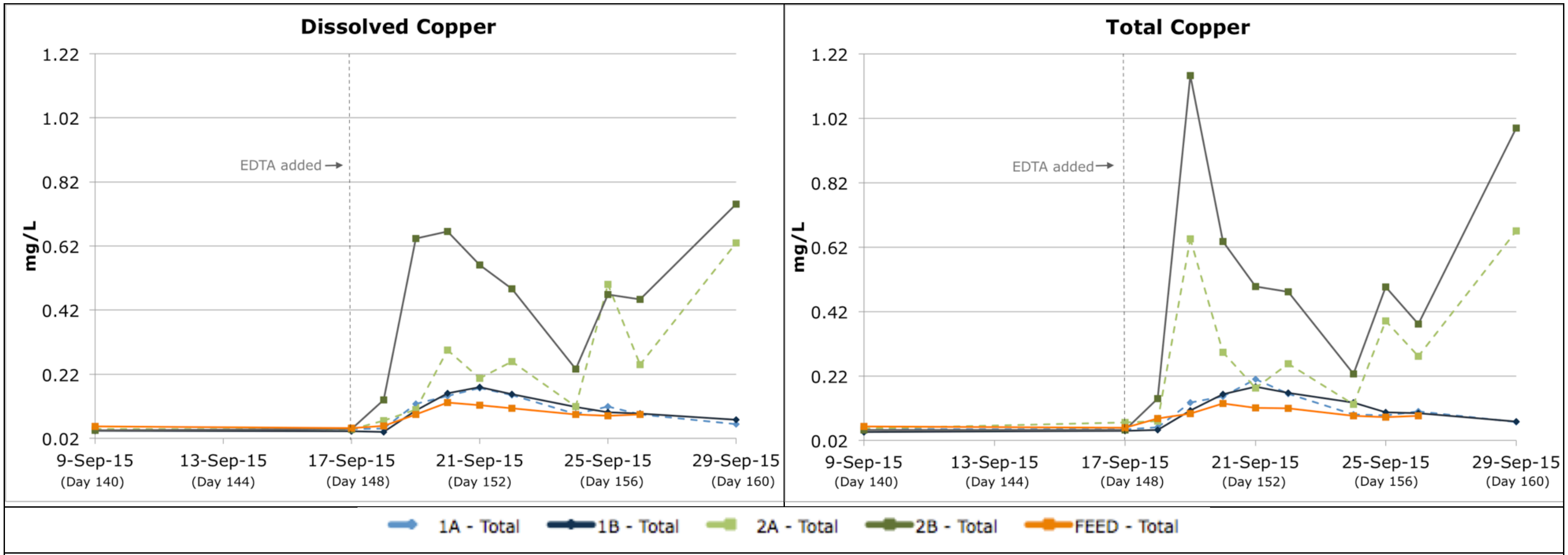

**Figure 9 – Copper concentrations in the wetland after the addition of Fe-EDTA to CWTS Series 2**
Detection limits are below y-axis values. No influent (feed) was tested September 29th as the system had frozen and was turned off for the winter.

### 6.4.Plants

During the commissioning period, plants establish and mature, with density expected to increase over time. When counted at the first site visit in June 2015, >95% of the plants were found to have survived their first winter. This survival rate is impressive considering the late fall planting of August 28, 2014 and suggests the *C. aquatilis* are very robust and reaffirms they are a good candidate for use in the full-scale CWTS at Minto. Additionally, there was a greater than 20% increase in total stems in the short growing season up to June 2015 (as counted by new shoots/runners). By the second site visit in August 2015, the plants were too dense to count stems, and therefore the survival and establishment was considered a success and no longer monitored by counting (Figure 6). Despite several mosses drying out during the winter after planting, there was 100% survival and establishment. However, their growth was much slower than the sedge and therefore they were supplemented with additional aquatic moss (harvested from the same W10 area as the original plants for the CWTS) in June and August 2015 to augment their general abundance and bring the CWTS through commissioning more quickly (Figure 10).

Pilot-scale testing suggested that *C. aquatilis* would increase more quickly the number of stems once the maximum plant height was achieved, and also that maximum plant height is dependent upon water depth (with deeper water resulting in taller plants). This was also seen in the demonstration-scale wetland on site, with the density of plants filling in greatly through July and August (Figure 11). It is anticipated that the *C. aquatilis* will have grown uniformly across the wetland by end of the 2016 or 2017 growing season.

Of note, it was observed that during the last site visit of the year by Contango (September 17-18th, 2015), the *C. aquatilis* in the demonstration-scale CWTS were still predominantly green, while other plants in the area had turned brown. This suggests that the CWTSs may have a longer range of activity than natural systems in the area, possibly due to liners and separation from colder ground water.

Concentrations of various elements in *C. aquatilis* and moss from the demonstration-scale systems were compared to those from pilot-scale testing. It is expected that the concentrations would be higher in the demonstration-scale system as the CWTS is not yet fully functional (i.e., elements are in greater bioavailability when in dissolved form) and also due to the composition of the soils used for construction. Accordingly, *C. aquatilis* and moss in the demonstration-scale system had higher copper concentrations than the pilot systems. However, concentration of copper in the *C. aquatilis* at the end of 2015 was not significantly different than that of the plants borrowed from the W10 area for planting of the systems in 2014 (Figure 12). The moss had higher copper and cadmium concentrations which is expected as it removes these elements in the wetland through sorption (and will eventually mineralize in subsequent years). As mosses are not a significant food source for any fauna, this is a safe mechanism to transfer elements from water eventually into stable mineral forms.

Cadmium concentrations were either below or close to the detection limit in *Carex* (Figure 12). Similarly, cadmium concentrations in moss and selenium concentrations in both plant types tested were lower in the demonstration-scale compared to pilot-scale. This result was presumably due to lower concentrations of both cadmium and selenium in the water of the demonstration-scale system than pilot scale. As mosses continue to grow and fill in the wetland, selenium treatment performance is expected to increase as mosses are a source of sorption, and also harbor the highest abundance of selenate-reducing microorganisms of all samples tested (see Section 6.5.2).

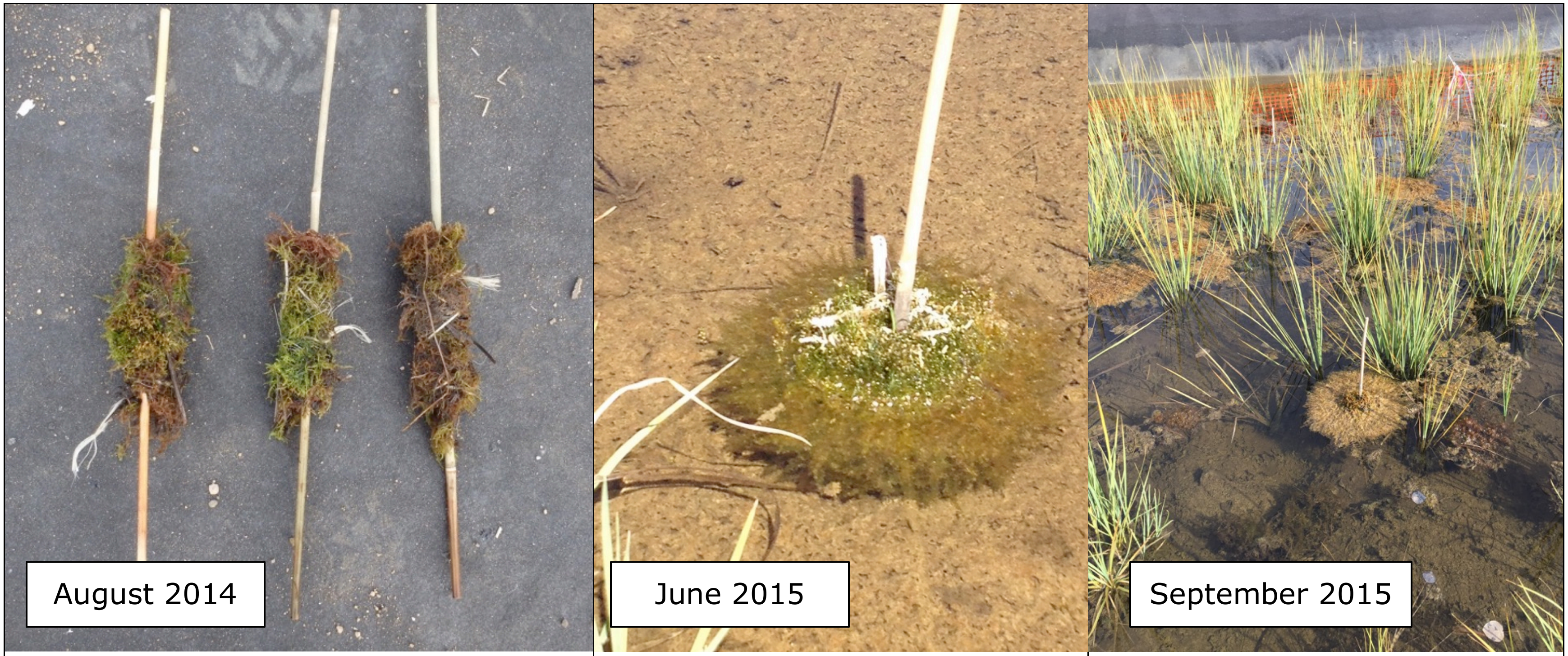

**Figure 10 – Moss maturation in CWTS.**

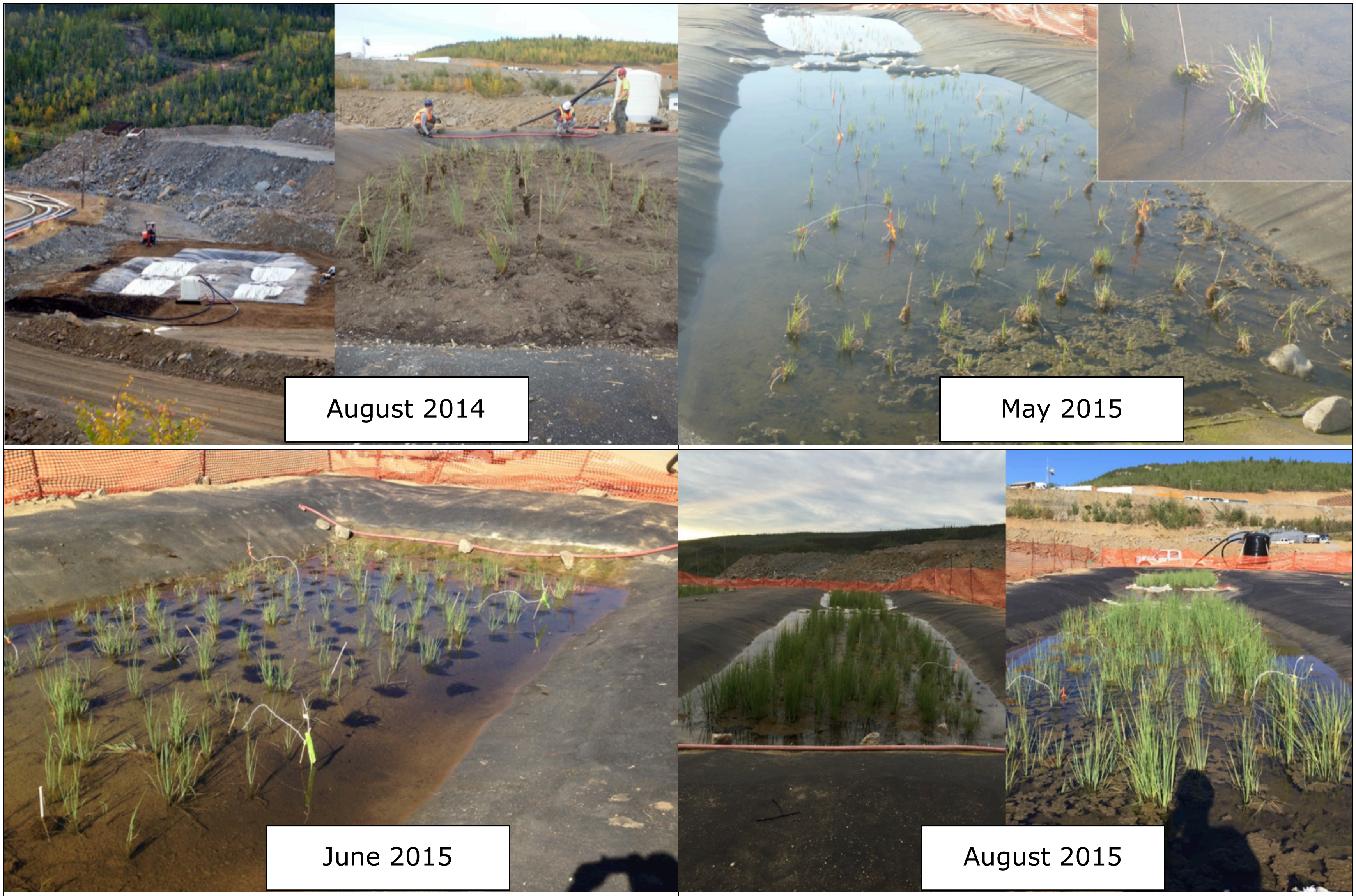

**Figure 11 – Maturation of the CWTS from construction to year-end 2015.**

Continued next page

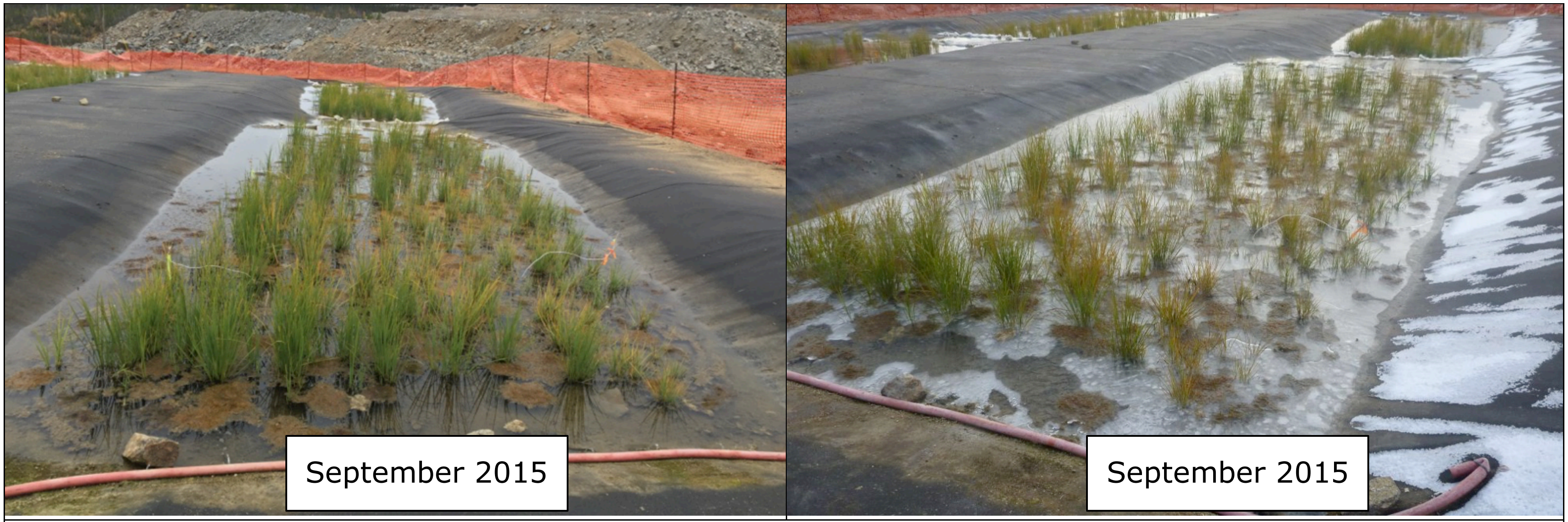

**Figure 11 - Continued**

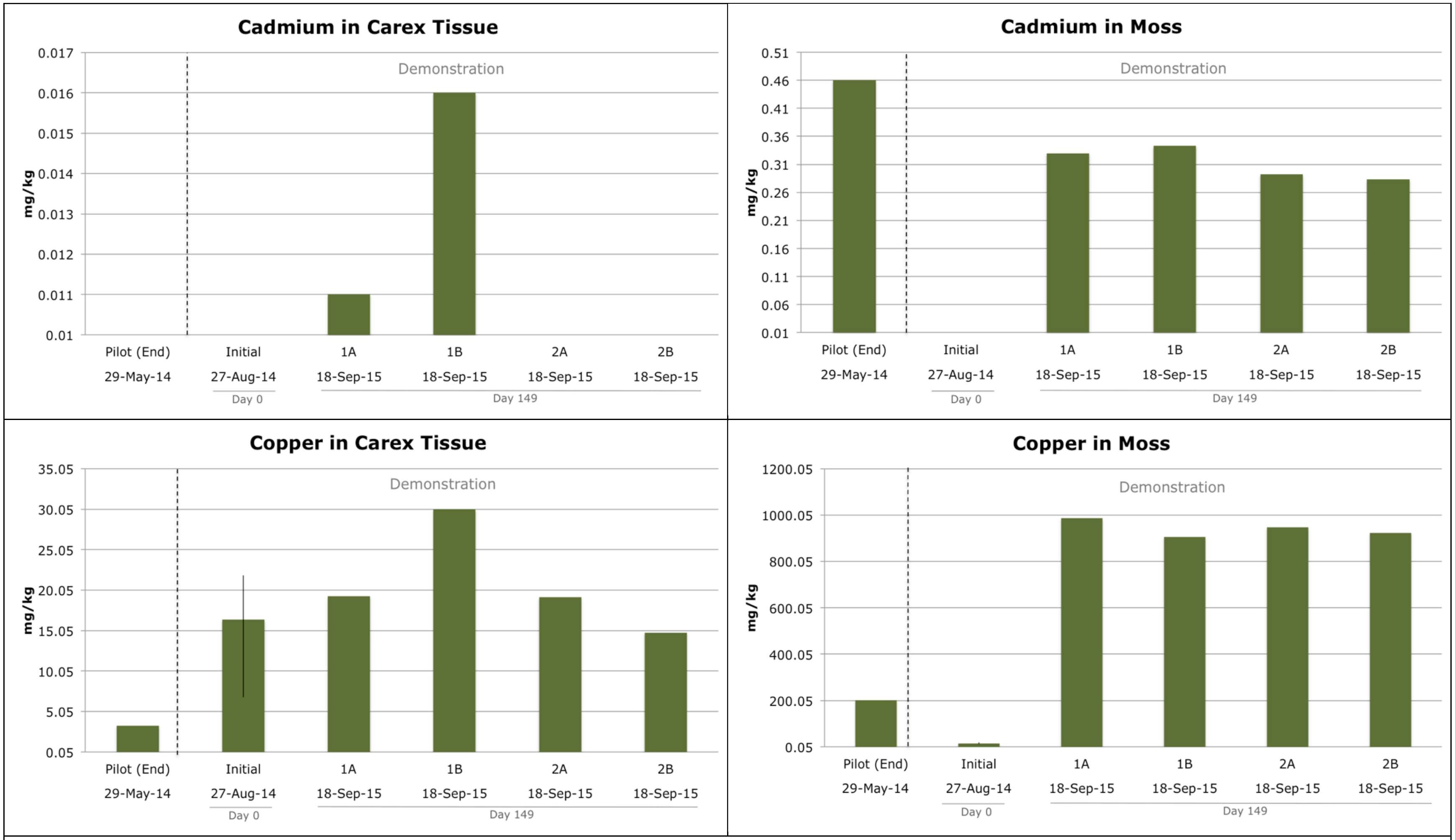

**Figure 12 – Element concentrations in plant tissue.**
Y-axis starts at the 2015 detection limit (DL). The 2014 DL for Cadmium is the same as 2015. The 2014 DL for Copper is 0.5 mg/kg, and Selenium is 0.2 mg/kg. The initial data set is the average value of CWTS 1A-2B. The error bars indicate the minimum and maximum values in that data set.
Continued next page.

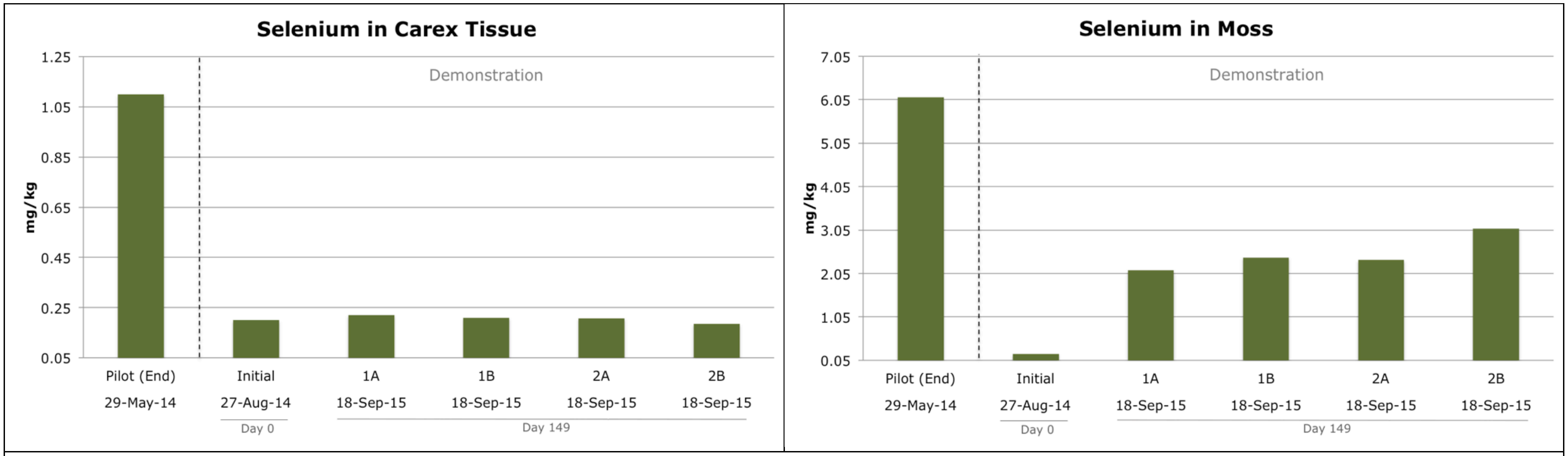

**Figure 12 - Continued**

### 6.5.Beneficial Microbes

Microbes are the driving force of many treatment pathways that are targeted in CWTSs. The beneficial microbes in these systems catalyze biogeochemical cycles for remediation of specific constituents of concern. Careful design of CWTSs can mimic the environmental conditions needed to enhance the abundance and metabolic activity of these beneficial microbes. Accordingly, complimentary methods of genetic and growth-based testing were used to characterize the microbial populations associated with a range of microbial habitats in the demonstration-scale CWTS (e.g., soils, sediment, biofilms, aquatic mosses, and plant roots).

In the context of the Minto Mine CWTSs, beneficial microbes include those that are involved in the reduction of selenium (selenate and selenite), nitrate, and sulphur compounds (which in turn treats copper and cadmium). Information on each of these mechanisms and the associated microbial populations in the demonstration-scale system is outlined in the following sections.

#### *6.5.1. Sulphide-producing bacteria*

Treatment is achieved by targeting the lithic biogeochemical sequestration of divalent metals through sulfide (i.e., $S^{2-}$, $HS^-$) precipitation as mineralized species (e.g., chalcocite [CuS], covellite [$Cu_2S$]). These sulfide bound species are relatively insoluble (CuS; $ksp=10^{-16}$; Stumm and Morgan 1996), and are transferred from the water column into the CWTS soil as non-bioavailable fractions (Murray-Gulde et al., 2003; Huddleston et al., 2008). Moreover, similar reactions occur with cadmium rending it non-bioavailable. As such, sulphide production is a key biogeochemical treatment mechanism for water treatment at Minto Mine. Sulphides can be created by beneficial microorganisms through the reduction of sulphur-containing compounds, such as sulphate, sulphite, thiosulphate, and elemental sulphur.

Based on the information gathered in pilot-scale testing, the targeted soil redox for the demonstration-scale CWTS is between -100 and -250 mV. This is in agreement with literature that indicates anaerobic conditions with relatively low ORP (-250 to -100 mV) are necessary for promoting anaerobic metabolisms in bacteria which oxidize organic matter, producing electrons which reduce sulfate to hydrogen sulfide ($H_2S$) and other reduced sulfide species (i.e. bisulfide ion ($HS^-$), sulfide ion [$S^{2-}$]; Mitsch and Gosselink 2007). In these redox ranges, bacterial sulphide-production through reduction of sulphur compounds is expected, alongside increases in the proportion (percentage) and abundance of these microbes.

The percentage of sulphate-reducing bacteria in the community increased over time in the demonstration-scale CWTS (Figure 13), as would be expected during the commissioning period. The percentage of sulphate-reducing bacteria is similar to what was present in pilot-scale systems at a comparable time point in operation (i.e., day 114 vs. day 80 in Figure 13), however, the types of microbes differ. Conversely, when considering all sulphide-producing bacteria (i.e., including those capable of sulphur, sulphate, sulphite, and thiosulphate reduction), the demonstration-scale systems have a higher percentage than the pilot-scale systems at a comparable time point (Figure 14), indicating the commissioning period is proceeding well. Sulphide-producing organisms identified during the site assessment at W10 and in low proportions at W15 (Contango, March 2014) were also present in both the pilot- and demonstration-scale systems, indicating that harvested plants were also a source of beneficial microbes.

As expected, the proportion of sulphide-producing bacteria increased as the soil redox decreased (Figure 14). Furthermore, the overall abundance of sulphide-producing bacteria has increased in the soil of the CWTSs over time as reducing conditions are achieved during the commissioning period (Figure 15). This is particularly notable in Series 1, which achieved targeted soil redox conditions quicker than Series 2, which is still establishing reducing conditions (see Section 5). The soils and *C. aquatilis* roots had the highest abundance of sulphide-producing organisms of all sample types tested, with the deeper soil (10-20 cm) having higher abundances than the shallow soils (0-5 cm) at day 149. As expected, algae and biofilm samples had very low abundances of sulphide-producing bacteria. Interestingly, the aquatic mosses had a large variability in the abundance of sulphide-producing bacteria (sometimes having as many or more than the soils). This variability may be related to general growth habits of the moss, with live, photosynthetic (oxygenating) parts of the moss having fewer sulphide-producing bacteria, while the older, decomposing regions have more. This fits with the targeted processes in the CWTS, where metals are first sorbed to moss, then eventually mineralized through sulphide-production.

The microbial analysis of various sample types in the CWTS has therefore confirmed that the commissioning period is proceeding as expected, with beneficial sulphide-producing bacterial populations establishing in the soils alongside reducing conditions.

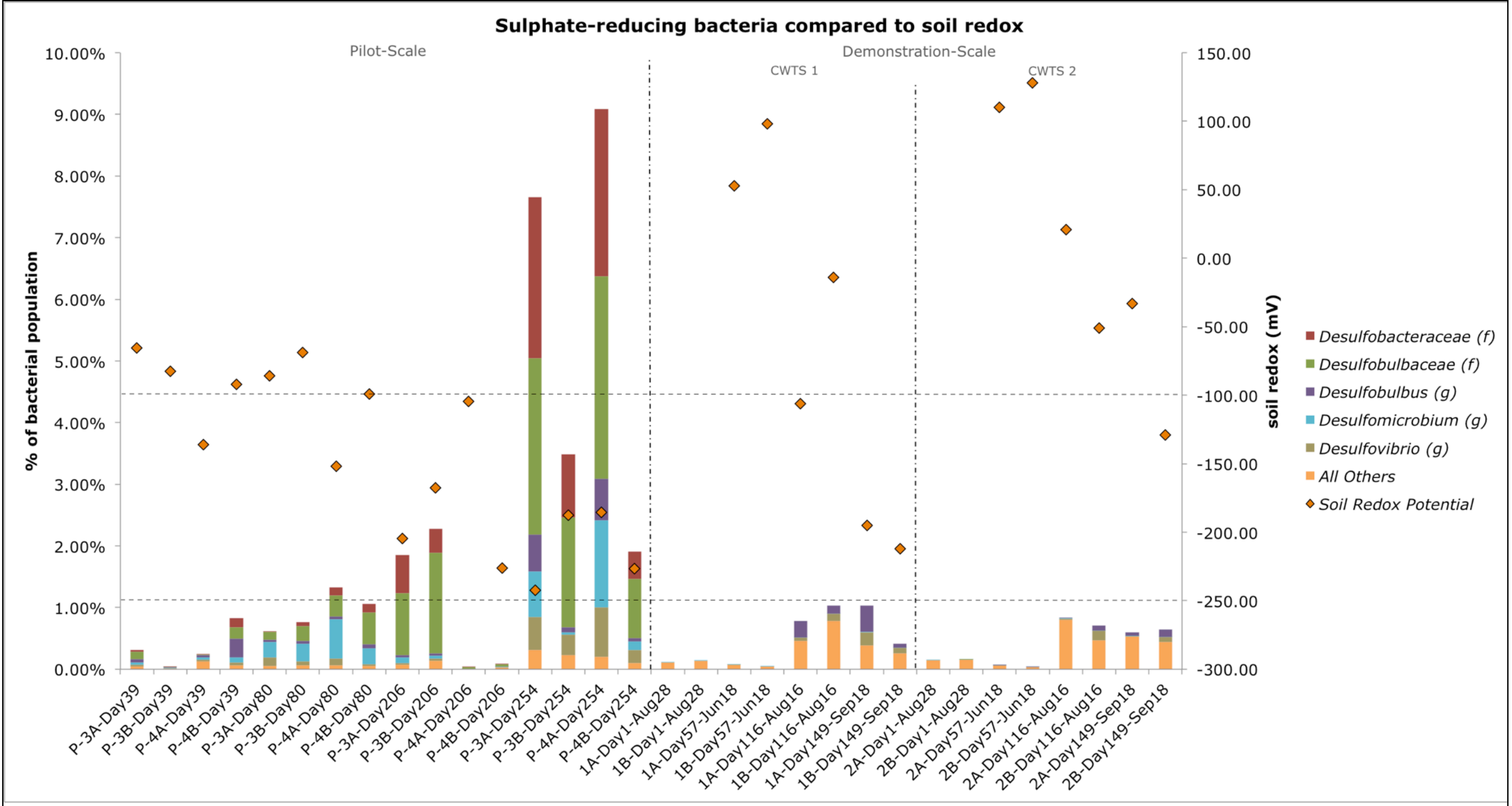

**Figure 13 – Percentage and identity of sulphate-reducing bacteria in soil of the pilot-scale and demonstration-scale CWTSs**

The vertical dashed lines separate the pilot-scale system data from the demonstration-scale system 1 and system 2 data. The horizontal dashed lines indicate the targeted soil redox range.

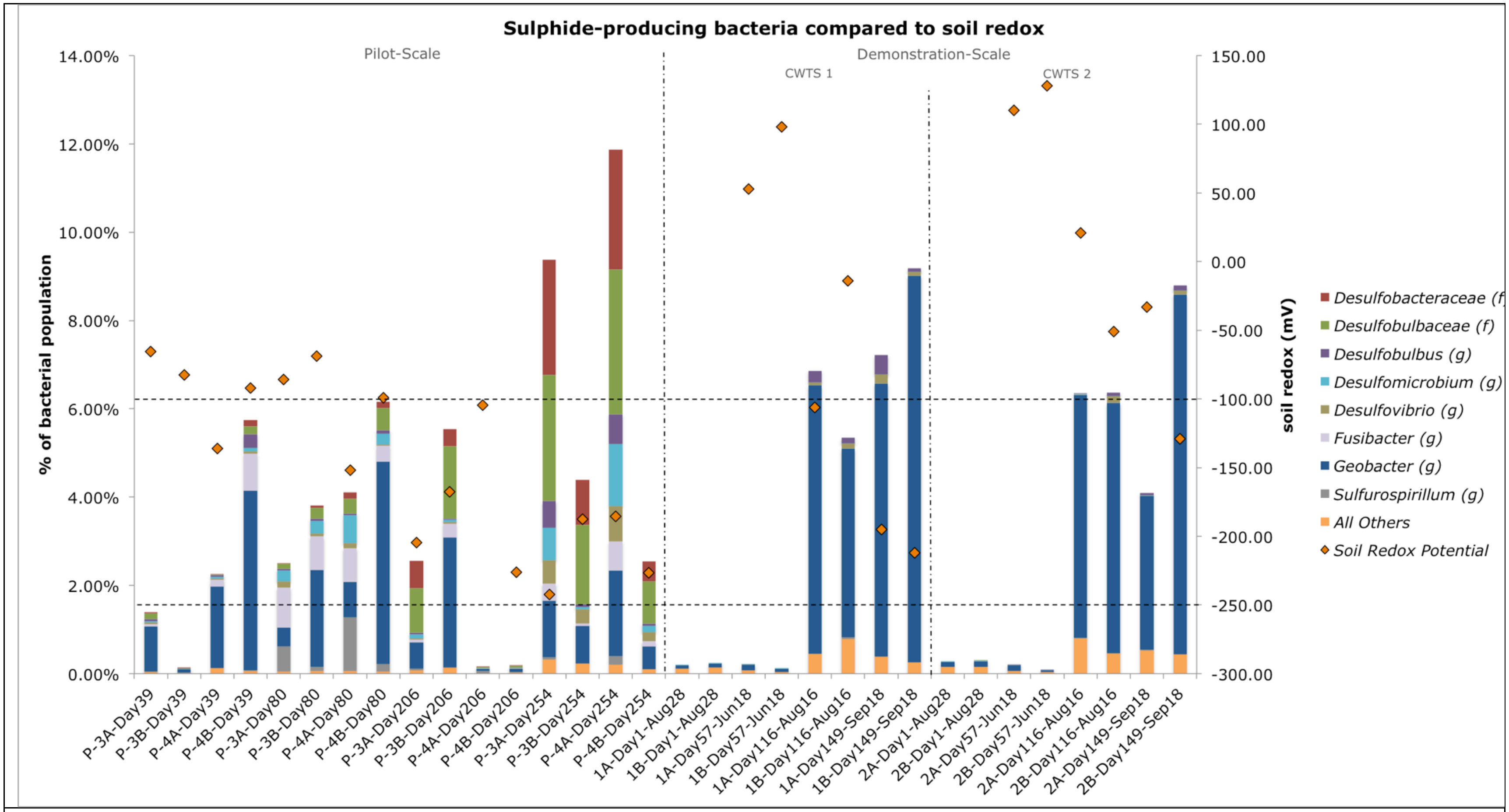

**Figure 14 – Percentage and identity of sulphide-producing bacteria in soil of the pilot-scale and demonstration-scale CWTSs**

The vertical dashed lines separate the pilot-scale system data from the demonstration-scale system 1 and system 2 data. The horizontal dashed lines indicate the targeted soil redox range.

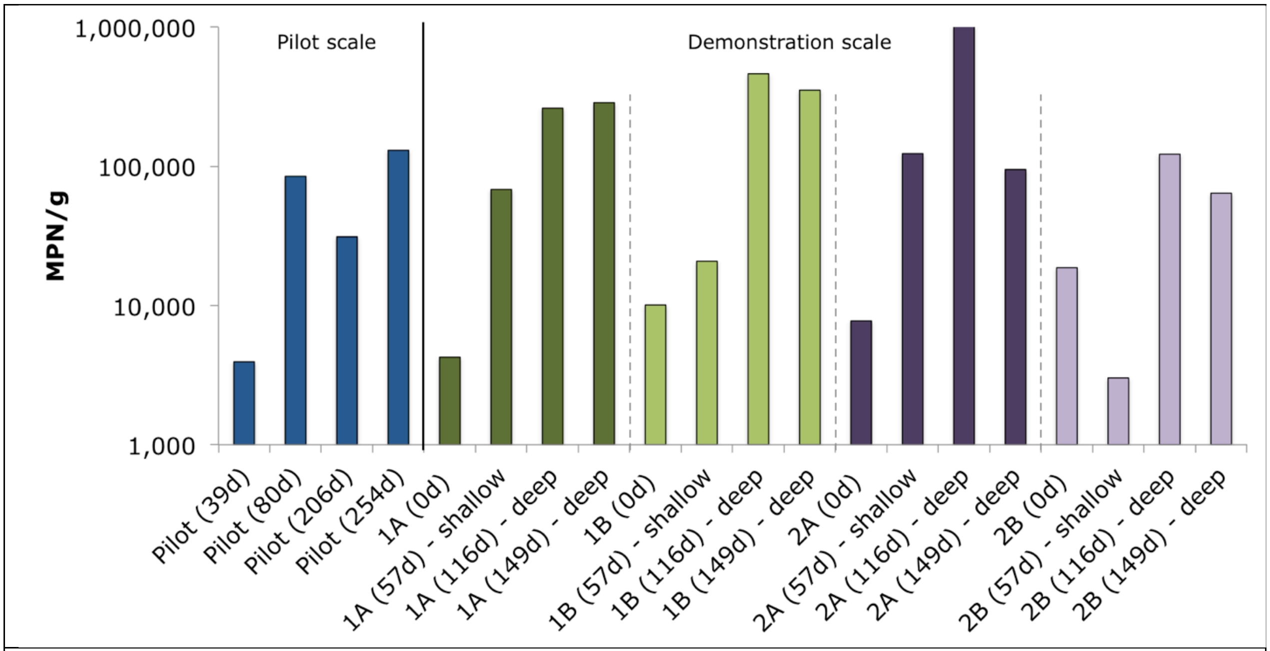

**Figure 15 – Inferred abundance of sulphide-producing bacteria in soil of the pilot- and demonstration-scale CWTSs**
Inferred abundance is calculated based on a combination of genetic and growth-based data.
- shallow soil (0-5 cm); deep soil (10-20 cm); soil at 0d was that used for construction of wetland

### *6.5.2. Selenium-reducing bacteria*

The targeted selenium treatment pathways in the Minto CWTS include sorption to moss and soils, and subsequent microbial reduction of soluble (sorbed) selenate (Se(VI)) and selenite (Se(IV)) to insoluble elemental selenium (Se(0)). This reductive process can also be achieved directly in the water column, but is more effective associated with mosses and biofilms due to their sorptive properties that bring the selenium in contact with beneficial selenium-reducing bacteria. This is achieved within the range of soil redox conditions targeted for sulphate-reduction as suggested by pilot-scale testing and literature (see Section 5).

Selenite-reducing organisms are ubiquitous in nature and as expected, were detected in all sample types, including algae, biofilm, moss, soil, sediment, and roots. Although selenate-reducing organisms are generally less abundant in the environment, they were found associated with all sample types, indicating that the conditions conducive to their proliferation have been created within the CWTS. Moreover, the abundance of selenite- and selenate-reducing organisms increased over time in the demonstration-scale system during the commissioning period (Figure 16). Aquatic mosses were found to host the highest abundances of both selenate- and selenite- reducing organisms, affirming the importance of the inclusion of moss in the CWTS. Additionally, shallow soils (i.e., top few centimeters, including associated biofilms) were found to host an abundance of selenite-reducing organisms, particularly in Series 1. The abundance of selenite-reducing organisms associated with the demonstration-scale moss and shallow soils is similar if not higher than that observed in the soil of the pilot-scale testing, indicating the commissioning period is proceeding as anticipated (selenate-reducing organisms were not tested for at pilot-scale).

### *6.5.3. Nitrate-reducing bacteria*

Nitrate is sometimes a constituent of concern during operations and early closure owing to residuals from blasting activities. Even if not in exceedance of water quality guidelines in terms of receiving environment objectives, nitrate often requires attention in order to achieve treatment of other constituents. The presence of nitrate can interfere with the treatment of certain elements in water (such as selenium). Nitrate can be removed from water by different types of microbes, including nitrate reducing bacteria which are capable of reducing nitrate ($NO_3$) to nitrite ($NO_2$), and also denitrifying organisms that are capable of fully reducing nitrate to nitric oxide (NO), nitrous oxide ($N_2O$), and dinitrogen gas ($N_2$ - which is the most abundant gas in air). MPN analysis was therefore used to quantify these organisms.

Nitrate-reducing and denitrifying organisms were found associated with all sample types in the demonstration-scale CWTS (Figure 17). Soil samples had an increase in nitrate reducers over time, with the highest abundance of all samples found in the deeper soil sample from day 149. In contrast, denitrifying organisms were found to be associated primarily with the shallower soil depths and also a high abundance associated with moss, which increased in abundance over the commissioning period.

Although the pilot-scale systems had higher nitrate concentrations (30 mg/L as N during High Nitrogen testing phase) than the demonstration-scale systems (average 10 mg/L as N), the abundance of nitrate-reducing and denitrifying organisms are both similar. As such, these results also confirm that the demonstration-scale system is establishing as expected during the commissioning period.

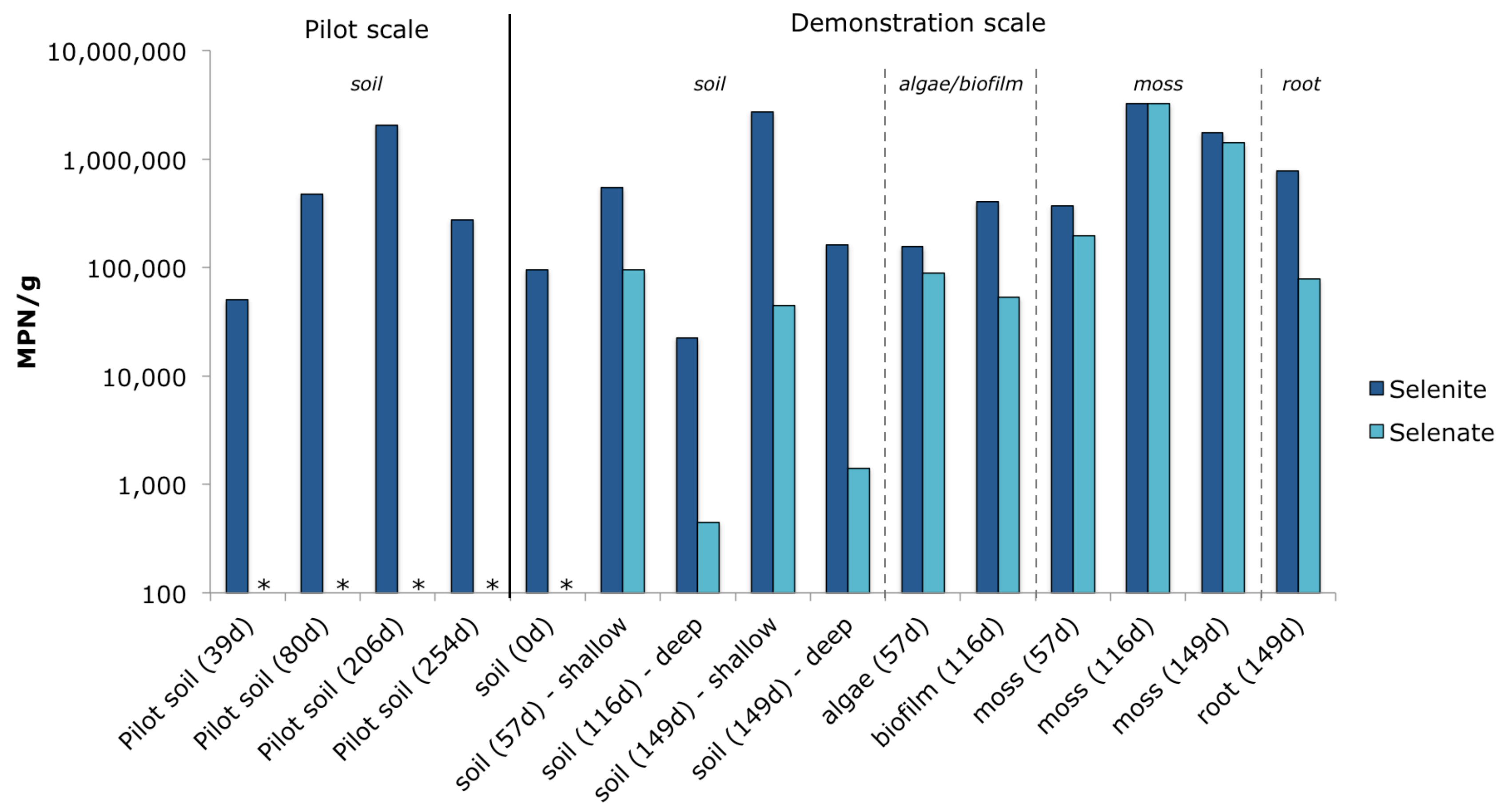

**Figure 16 – Abundance of selenite- and selenate-reducing organisms in various CWTS sample types over time**

* - pilot-scale system and initial timepoint for demonstration-scale system not tested for selenate-reduction

\- shallow soil (0-5 cm); deep soil (10-20 cm); soil at 0d was that used for construction of wetland

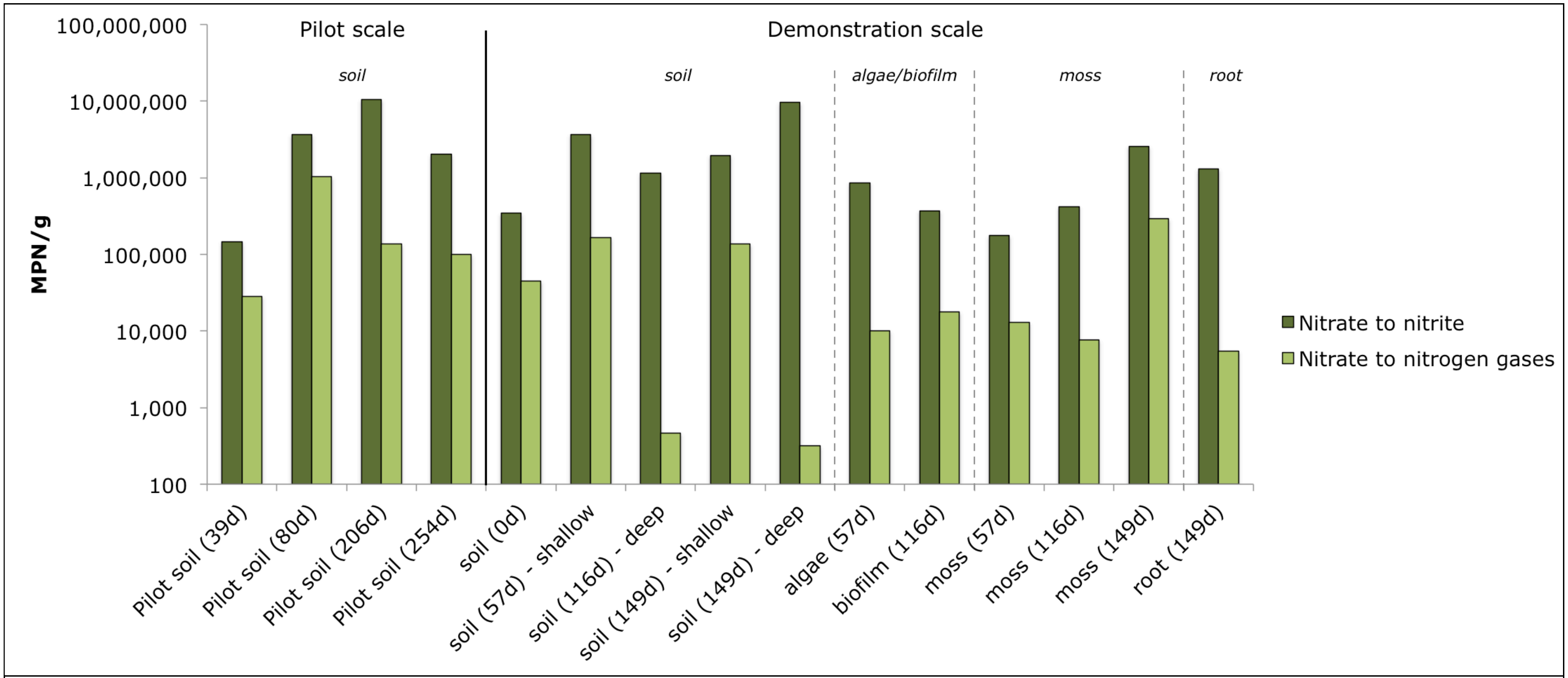

**Figure 17 – Abundance of nitrate-reducing and denitrifying organisms in various CWTS sample types over time**

Nitrate-reducing = reduction of nitrate to nitrite; denitrifying = reduction of nitrate to nitrogen gases. Shallow soil (0-5 cm); deep soil (10-20 cm); soil at 0d was that used for construction of wetland.

## 7. Summary of Results

Table 6 summarizes results and findings from 2015 commissioning of the demonstration-scale system at Minto.

**Table 6. Summary of Minto Demonstration-scale 2015 Testing**

| Objective | Purpose | Key Findings |
|---|---|---|
| Evaluate construction | Optimize construction and effectiveness of operation of full-scale systems | **Layout**<br>-Outflow collection pond should have outflow at base (not top), with shutoff valve<br>-Increased slope on sides, and riprap or sandbags added at shores would prevent water short circuiting and deter wildlife access |
| | | **Soils**<br>-Use substrate with less total and leachable metals and metalloids (especially copper)<br>-Higher sand content would improve hydrology, constructability (ability to level soils, ease of planting) and accessibility for sampling<br>-Organics should be mixed in bulk to soils prior to adding to cells |
| Assess commissioning timelines | Allow for proper phasing of implementing full-scale systems for closure | **Water**<br>-Copper treatment improving through commissioning period; wetland achieving better treatment than suggested by inflow and outflow concentrations of system, as soils are leaching copper<br>-Cadmium and selenium are also being removed from water<br>-Wetland is maturing as expected and is performing beyond anticipated from the design<br>-Tracer study recommended for 2016 to assess hydrology and pore volume of CWTS and determine HRT and removal rate coefficients for full-scale sizing |
| | | |

| Objective | Purpose | Key Findings |
|---|---|---|
| | | **Soils**<br>-Soil redox has decreased as expected, reaching targeted ranges in Series 1 by the end of 2015, while Series 2 continues to establish<br>-Significant amounts of metals are leaching from soil substrate into water, putting additional treatment demands on system |
| | | **Microbes**<br>-Sulphide-producing bacteria needed for copper and other metals removal have increased over time as soil redox achieved target ranges. Proportions are comparable to those in pilot system at similar point in commissioning<br>-Abundance of selenium- and nitrate-reducing organisms are similar to those in pilot testing, indicating maturation as expected<br>-Selenium treatment performance expected to increase as mosses continue to grow, as they can sorb dissolved selenium and harbour highest abundance of selenate-reducing microorganisms to render the selenium insoluble |
| *Carex aquatilis* transplantation effectiveness | Determine if plant propagation and/or replanting schedule will be needed for full-scale systems | ->95% survival from transplanting<br>-Within first 2 months a further increase of >20%<br>-Full-scale system could be planted more densely to bring online faster, or less densely if time is less of an issue than sourcing plants (the plants are vigorous and will fill in the wetland in due time) |
| Moss colonization/distribution | | -100% survival from transplanting<br>-Slower to spread, needs to be started more densely<br>-Staking helps maintain moss in 'upstream' parts of wetland, or could be transplanted multiple times through commissioning period |

## 8. Tentative Schedule and Action Items for 2016

Based on progress of commissioning and early performance results in 2015, an action plan for CWTS optimization and testing for 2016 has been developed (Table 7). An updated multi-year schedule as per the proposed scope of work in the Minto Demonstration Scale Report Document 011_0315_01A (Contango, March 2015) is provided in Appendix C.

**Table 7. Minto 2016 CWTS Demonstration-scale Action Items**

| Who/When | Task | Contango Action Required | Additional Information |
|---|---|---|---|
| Minto Staff, prior to first site visit | Add sandbags to perimeter of CWTS to minimize short circuiting | Provide diagram of sandbag location | Use sand that was confirmed to be good borrow source in 2016 (i.e., low copper) |
| | W15 creek monitoring | Develop research plan and schedule of testing | This is the creek(s) feeding the W15 area pond |
| Site visit 1 (May 2016) | Tracer study (salt) | Experimental design | Ensure salt tracer study will not affect other planned activities at the site (e.g., hydrogeology studies) |
| | Change AgCl probes | Replace old probes with new ones | These are the reference probes for soil-redox testing |
| | Develop flow rates for 2016 | Based on tracer study | Flow rates will be set (and varied) according to expected flows at water storage pond in closure, and theoretical hydraulic retention times needed to develop outflow water quality predictions |
| | Check on W15 creek monitoring | Confirm monitoring locations | Because at least 3 entry points exist from the creek to the W15 pond, check which creek is being monitored and how this compares to the W15 sampling point water quality |
| Site visit 2 (July 2016) | Add organic material to System 2 | Determine amount and type to add | This will only be performed if soil redox conditions have not consistently met targeted ranges by July 2016 |
| Site visit 3 (September 2016) | General follow up | Final sampling for year | |

## 9. Monitoring plan

A conceptual long-term testing plan was developed for the demonstration-scale CWTS and has been refined and adapted based on performance and scientific findings (Appendix A). The demonstration-scale wetlands are expected run until at least the end of 2016, and ideally longer in order to assess performance under a wider range of conditions. The conditions that could eventually be tested include both natural/environmental and selected influenced pressures, and can be imposed on the systems to mimic peak flow rates or droughts. In 2016 the systems are expected to continue to mature (i.e., complete commissioning), with plants becoming more established and abundant, and microbial communities accordingly acclimating to the targeted conditions. We expect to achieve targeted ranges of soil redox in 2016, which will indicate that the commissioning period has completed and allow for the monitoring program to shift focus to testing of operations and performance. A multi-year plan is provided in Appendix C, and includes work performed to date as well as a schedule for 2016 and potential activities for 2017.

## 10. Closure

We trust the information herein satisfies your present requirements. Should you have any questions, please contact the persons listed below. We appreciate the opportunity to provide the services detailed in this report, and look forward to discussing any comments you may have.

Respectfully submitted,

Contango Strategies Ltd

Monique Haakensen
PhD, RPBio, PBiol, EP

Vanessa Friesen (Pittet)
PhD, EPt

ALBERTA SOCIETY of Professional Biologists
Monique Haakensen
2131
PROFESSIONAL BIOLOGIST

COLLEGE OF APPLIED BIOLOGY
Monique Haakensen
R.P. Bio
2671
CAB

with contributions from: Rachel Martz, BSc and Jenny Liang, BSc

## 11. References

Contango Strategies. (March 24, 2014) Minto Mine Constructed Wetland Treatment Research Program – Site Assessment.

Contango Strategies. (November 3, 2014) Minto Mine Constructed Wetland Treatment Research Program – Pilot Scale Report.

Contango Strategies. (March 22, 2015) Minto Mine Constructed Wetland Treatment Research Program – Demonstration Scale. Document #011_0315_01A.

Faulkner, S.P., Patrick, W.H. Jr. and Gambrell, R.P. (1989) Field techniques for measuring wetland soil parameters. Soil Science Society of America Journal, Vol. 53, pp. 883-890.

Huddleston, G.M. III, Rodgers, J.H. Jr., (2008). Design of a constructed wetland for treatment of copper-contaminated wastewater. Environmental Geosciences 15, 9-19.

Mitsch, W.J. and J.G. Gosselink. (2007). Wetlands, 5th ed., John Wiley & Sons, Inc., New York

Murray-Gulde, C.L., Heatley, J.E., Karanfil, T., Rodgers, J.H. Jr., Myers, J.E., (2003). Performance of a hybrid reverse osmosis-constructed wetland treatment system for brackish water. Water Research 37, 705-713.

Stumm W & Morgan JJ. (1996). Aquatic chemistry, chemical equilibria and rates in natural waters. John Wiley & Sons, Inc. New York, NY, USA.

Tessier, A., Campbell, P.G.C., and Bisson, M. (1979) Sequential Extraction Procedure for the Speciation of Particulate Trace Metals. Analytical Chemistry, Vol. 51, pp. 844-851.

Thayalakumaran, T., Vogeler, I., Scotter, D.R., Percival, H.J., Robinson, B.H., and Clothier, B.E. (2003) Leaching of copper from contaminated soil following the application of EDTA. I. Repacked soil experiments and a model. Australian Journal of Soil Research, Vol 41, pp. 323-333.

# Appendix A

**Table 1. Summary of analytical sampling types, frequencies and locations**

| | |
|---|---|
| **Water** | |
| Dissolved and total metals | Weekly, outflow of each cell and feed |
| Flow rate | Weekly, feed |
| pH, DO, ORP, Conductivity (in situ) | Weekly, all cells and feed |
| Anion Sum | Monthly, outflow of each cell and feed |
| Cation Sum | |
| Hardness ($CaCO_3$) | |
| Ion Balance | |
| Total Dissolved Solids (TDS) | |
| Chloride (Cl) | |
| Sulphate ($SO_4$) | |
| Nitrate | |
| Nitrite | |
| Nitrogen (Total) | |
| Alkalinity | |
| pH | |
| Conductivity | |
| Chemical Oxygen Demand (COD) | |
| Total Organic Carbon (TOC) | |
| Ammonia | |
| Total Kjeldahl Nitrogen (TKN) | |
| Total Suspended Solids | |
| Biological Oxygen Demand (BOD) | |
| Bicarbonate ($HCO_3$) | |
| Carbonate ($CO_3$) | |
| Hydroxide (OH) | |
| **Soil** | |
| Relative soil redox (in situ) | Monthly, all probes (6 per cell) |
| Cation exchange capacity (CEC) | Seasonally (3x per year) |
| SAR, pH, EC, %sat, Ca, F, Mg, Na, K, Cl, SO4 | |
| Available NPK and sulphur | |
| Alkalinity | |
| Bicarbonate ($HCO_3$) | |
| Carbonate ($CO_3$) | |
| Hydroxide (OH) | |
| Total Organic Carbon (TOC) | |
| Dissolved Organic Carbon | |
| Metals Analysis (Total) | |
| Metals Leachable (SPLP) | |

| | |
|---|---|
| Ammonia | |
| Nitrate | |
| Nitrite | |
| Total Dissolved Solids | |
| Anion Sum | |
| Cation Sum | |
| Cation/EC ratio | |
| Ion Balance | |
| Sequential Leaching (5 Acid Test) | |
| Cation exchange capacity (CEC) | |
| **Plant tissue samples** | |
| Metals Analysis | *Carex aquatilis* and aquatic moss, each cell, year end |
| **Microbial samples** | |
| Growth-based most-probable number analysis (nitrate reduction, selenite and selenate reduction, total heterotrophs) | Seasonally (3x per year) |
| Genetic sequencing analysis for bacterial community composition and distribution | Seasonally (3x per year) |