

INVESTIGATING THE EFFECT OF CEMENT STABILIZATION ON A SLATE  
WASTE ROCK

by

Matthew Walker

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## **Abstract**

Acid-producing waste rock is a by-product of urban construction, and its environmental impact necessitates proper disposal. Cement solidification and stabilization is a technique to limit the effect of acid drainage leaching from waste rock material and consists of mixing the waste rock with a cementitious binder, followed by placement in a containment cell. This technique aims to reduce water flow through the material and to increase the pH/decrease effluent contaminant concentrations. Many test methods are available to assess the performance of the stabilized material. Humidity cell testing weathers the treated material over time by repeatedly exposing the stabilized mixture to cycles of air and water. Based on humidity cell testing performed, the cement stabilization was effective in neutralizing the pH of the effluent leachate and slowing the release of most contaminants. Notable exceptions were sulfur and sulfate, which were detected in higher concentrations in the binder treated samples.

## List of Abbreviations

ABA	Acid Base Accounting
AMD	Acid Mine Drainage
ASTM	American Society for Testing and Materials
GU	General Use
HCl	Hydrochloric Acid
HRM	Halifax Regional Municipality
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectrometry
L/S	Liquid to Solid Ratio
RDL	Reported Detection Limit
SM	Silty Sand
SPLP	Synthetic Precipitation Leaching Procedure
S/S	Stabilization and Solidification
TDS	Total Dissolved Solids
UCS	Unconfined Compressive Strength
UCSC	United Soil Classification System
USEPA	United States Environmental Protection Agency
S	Slate
SB	Slate and Binder
SSB	Slate, Soil, and Binder
F	Fine
M	Medium
C	Coarse
CF	Cement and Fly Ash
CM	Cementing Materials
W	Water

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# Chapter 1 Introduction

## 1.1 Introduction

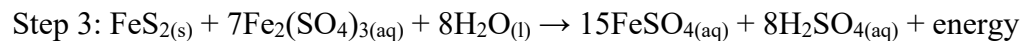
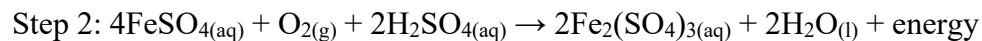
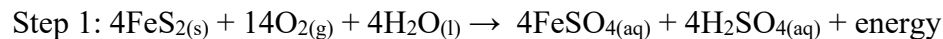
The development of urban areas continues to grow at a rapid pace. The construction of roads, buildings, and other infrastructure can often result in large-scale excavations of soil and rock. This excavated material can create issues of environmental concern if not properly characterized and managed. Some types of excavated rock have the potential to “react” with the environment and create acid producing leachate. Many studies have highlighted this environmental concern for mining projects in the form of acid mine drainage (AMD) (Benzaazoua et al., 2004b; Benzaazoua et al., 2008). However, studies have also highlighted a similar problem during subsurface construction in urban environments (Tabelin et al., 2018; Tatsuhara et al., 2012). In Nova Scotia, much of the developed land in urban areas of the Halifax Regional Municipality (HRM) is underlain by slate of the Meguma group (Waldron et al., 2009). When this rock is excavated or otherwise disturbed, it has the potential to produce an acidic chemical environment which can increase the risk of a negative environmental impact. In 1982 in Nova Scotia, approximately 224,000 m<sup>3</sup> of excavated rock from airport construction was disposed of within the watershed of the Salmon River. It is estimated that this rock caused upwards of 250,000 kg of CaCO<sub>3</sub> acid rock drainage production in the watershed. This acid rock drainage lowered the pH and increased metal concentrations, such as aluminum, in the river causing significant fish mortalities (Lund et al. 1987). These adverse effects on the environment are examples of urban development creating acid-generating waste rock outside the realm of mining applications. Because of this risk, the type of rock associated with such leachate characteristics requires special disposal consideration when excavated.

This introductory chapter provides a brief review of the chemical reactions associated with acid-producing rock, various techniques behind safely disposing of this rock, as well as the specific technique of cement-based treatment of the rock prior to disposal. Based on this literature review, research questions for this thesis are raised, and the structure of the thesis to answer these questions is presented.

## 1.2 Literature Review

### 1.2.1 Introduction to Acid Waste Rock

When acid producing rock is excavated through typical construction methods, the particle size is decreased, and the surface area of the rock is increased which allows it to more readily come into contact simultaneously with water and oxygen. This exposure to oxidizing conditions in the presence of water leads to chemical transformation of the sulfide minerals. As discussed by Lottermoser (2010), pyrite is one of the most common sulfide minerals present in rock formations and hence plays a large role in acid production in waste rock from mine sites. Lottermoser (2010) discusses how the most common form of pyrite degradation is indirect oxidation by oxygen and iron in the presence of bacteria through three main steps. Step 1 being the oxidation of pyrite by oxygen, step 2 being the oxidation of the ferrous iron to ferric iron, and step 3 being the oxidation of pyrite by ferric iron. The chemical reactions of these steps are summarized below:



In step 1, ferrous iron ( $\text{Fe}_2^+$ ) is produced along with sulfate and hydrogen ions. The dissolved iron sulfate ions are responsible for increased TDS (total dissolved solids) of the water. The release of hydrogen ions with the sulfate anions results in a decrease in pH. In step 2, at a lower pH and increased amounts of ferrous iron and in the presence of oxygen, ferric iron ( $\text{Fe}_3^+$ ) is produced. Step 3 utilizes ferric iron to also act as an oxidizing agent of pyrite. As noted in Step 3, the oxidation of pyrite by ferrous iron in turn generates more ferrous iron and a decrease in pH. As discussed by Lottermoser (2010), the byproducts of these three steps are occurring simultaneously and causes the reaction process to cycle until pyrite or ferric iron is exhausted. The low pH and increased amount of TDS is harmful to the environment and aquatic life (Lund et al. 1987). This acid rock drainage is a well-documented concern for mining projects and disposal of waste rock and tailings (Qureshi et al., 2016; Akcil and Koldas, 2006; Kefeni et al. 2017). Pyritic slates, such as those of the Meguma Group found in Nova Scotia (Glasmacher et al. 2003), are known to contain the sulfide-bearing mineral composition necessary to produce these conditions.

### **1.2.2 Acid Mine Drainage**

The safe disposal of acid waste rock generally involves reducing the contact of the rock with either water or oxygen to limit the reactions presented in the previous section. Large-scale mining projects that involve removing high volumes of potentially acid-producing rock, along with subsequent processing activities of this rock, can create acid mine drainage (AMD) (Qureshi et al., 2016). For many years, AMD has been an important consideration in the final disposition of mining waste once valuable ore or other materials are extracted. An established method of AMD treatment is through lime neutralization

which is used in many situations involving AMD (Skousen, 2014). This treatment produces a clean effluent and a fine-grained, alkaline sludge. It has been shown that this sludge, when mixed with a silty soil, can be used as a cover material to prevent the movement of oxygen into covered tailings, effectively preventing AMD generation (Demers et al., 2017). Though this is a use specifically directed towards the mining industry, there are no obvious limitations preventing its potential use in urban environments. Tailings ponds are a common technique for disposal of fine-grained mining waste. Tailing ponds are large, man-made ponds where mine waste and water are pumped and allowed to separate by sedimentation (Tuomela et al., 2021).

Dry waste piles are another method of storing mine waste rock. In this form of waste rock storage, the rock is placed in an unsaturated state but is susceptible to acid mine drainage due to its highly porous structure (Bao et al., 2021). These piles may use some form of soil cover to prevent the ingress of water into the waste pile.

Environmental desulfurization is a process by which sulfides are separated from mine tailings using a bulk sulfide flotation process. This results in two distinct forms of waste, desulfurized tailings and sulfide-rich tailings that may be disposed of using different processes based on the low and high acid-producing potentials, respectively (Benzaazoua et al., 2008).

Another method of tailings management involves using a combination of the fine-grained tailings (and water), a binding agent (such as cement), along with other additives to form a “paste” which is then used as backfill on location of the mine itself. The use of cement to “stabilize” or “solidify” soils is common for transportation applications, soil remediation, mine ground support (Benzaazoua et al., 2004a), etc (Tariq and Yanful, 2013).

After placement and curing, the paste forms a strong, concrete-like substance that can be used as structural backfill in the mine. While the resulting paste material is fit to be used as backfill, its structural compressive strength is generally less than 10% of low-strength, purpose build concrete. The addition of a cement material strengthens the tailings mixture making it suitable as structural backfill. The cement also significantly reduces the ability of water to move/percolate through the tailing material, preventing dangerous levels of heavy metals and other contaminants in the leachate released from the waste mixture (Mansour et al., 2014).

Thickened tailings are described as a combination of water and tailings with no cement present. This differs from traditional tailing slurries by a lower percentage of water and therefore a more viscous product. This helps save water, increase density (therefore decreasing the required volume of storage) and decrease the potential for acid mine drainage in the form of leachate caused by excess water movement through the material (Mansour et al., 2014).

### **1.2.3 Management of Acid Waste Rock in Urban Environments**

New development in urban areas creates waste rock, especially in cities that utilize significant underground infrastructure. Japan, with limited area and densely populated urban centers, has implemented underground construction (i.e. transportation, utilities, public-use spaces, etc.) for decades (Hanamura, 1990). Current management techniques of naturally contaminated waste rock primarily focus on the use of specially designed landfills which store the material and are designed to limit the detrimental impact on the surrounding environment.

Researchers have suggested that open quarries, mines, and underground tunnels no longer in use may be used for the disposal of potentially harmful waste rock (Tabelin et al., 2018). In Germany, an old limestone quarry was used to dispose of nearly 2.3 million cubic meters of waste rock, some of which was highly contaminated (Haid and Hammer, 2009). Waste rock that does not contain potentially harmful contaminants may be used in a variety of ways, such as aggregate in concrete or to construct embankments. It has been proposed that waste rock materials that do contain potentially harmful contaminants may similarly be used in embankment construction given some measures are put in place to reduce the likelihood of detrimental impacts to the environment (Tabelin et al., 2018). These measures include neutralization of the waste rock (with a neutralizing agent such as  $\text{CaCO}_3$ ), the use of a low permeability soil cover over the rock material to prevent water infiltration, and the addition of an adsorption layer at the base of the embankment. This combined neutralization-adsorption system has been shown to be more effective in neutralizing acid leachate and reducing effluent arsenic and heavy metal concentrations than neutralization-only methods (Tatsuhara et al., 2012).

In the Halifax Regional Municipality (HRM), there are currently two common methods to dispose of acid-generating waste rock materials that are excavated from construction sites; one is to dispose of it on land with some form of isolation from water and oxygen (e.g. cover systems), and second is to dispose of it in the ocean (i.e. Halifax Harbour) which limits its exposure to oxygen. Disposing of acid-producing rock material on-site can prove to be a practical solution in some situations where there is adequate space available. However, for many urban construction sites, this is not the case. Recently, a local

company, Envirosoil, has obtained environmental approval for disposal of sulfide-bearing waste rock in a geomembrane-lined disposal cell (i.e. off site).

Acid-producing rock disposal in the ocean is a common approach in HRM. Currently, a section of the Halifax Harbour is being infilled with acid-generating slate waste rock which will be eventually used to reclaim land. Unfortunately, this solution is likely not permanent as it depends on active construction projects that are accepting slate waste rock material. Another complication of this approach is that it is subject to several environmental restrictions (i.e. not accepting waste rock material contaminated with hydrocarbons or material with an excess fine grain-size fraction).

#### **1.2.4 Physical and Chemical Analyses of Waste Rock Materials to Assess Acid-Producing Potential**

There are several tests that can be used to assess the potential of a waste rock to be acid producing. Initial testing of a potential acid-producing waste rock might involve mineralogical testing such as x-ray diffraction, the use of scanning electron microscopes on thin sections of rock sample, or elemental analyses (Qureshi et al., 2016). Acid-base accounting (ABA) procedures are a common form of testing to assess the acid-producing potential of a rock and are used extensively to determine the acid-producing potential of mining waste rock (Downing, 2014a). Some of the most commonly performed tests are outlined briefly in the following paragraphs.

#### 1.2.4.1 BC Research Inc. Test

The BC Research Inc. Initial Test (Bruynesteyn and Duncan, 1979) is a preliminary test that can be quickly performed to determine if the acid-neutralization ability of a rock is higher than its acid-production ability. If this is found to be the case, the rock is regarded as non-acid producing. If the test shows that acid-production outweighs acid-neutralization, further testing is required. The general procedure is as follows. A representative sample of waste rock material is cone-crushed to a minus No. 10 sieve. A 250 g subsample of this material is then dried and pulverized to approximately a 60% minus No. 400 sieve. An assay is then performed on the material to determine the sulfide sulfur content, and this is expressed as acid-production potential (expressed as kg of H<sub>2</sub>SO<sub>4</sub> per ton). A titration is then performed on a remaining portion of the sample where acid consumption of the rock is determined (expressed as kg of H<sub>2</sub>SO<sub>4</sub> per ton). If the acid consumption exceeds acid-producing potential, the material will not produce acid drainage, and no further tests are required. If acid-producing potential exceeds acid consumption (or if the difference is small), acid drainage may be present, and an additional, more complicated test can provide further confirmation (Bruynesteyn and Duncan, 1979). This confirmation test uses biological oxidation to determine the acid-producing potential of the sample under field-like conditions. Advantages to this test include that it is relatively quick and inexpensive, it is widely used throughout Canada, and it provides information on the biochemical oxidation of the material. Potential disadvantages may be that it is difficult to find/use optimal bacteria cultures, it is hard to compare results between laboratories, and the scale-up effects are based on many factors that are difficult to assess (Bruynesteyn and Hackl, 1984; Downing, 2014a).

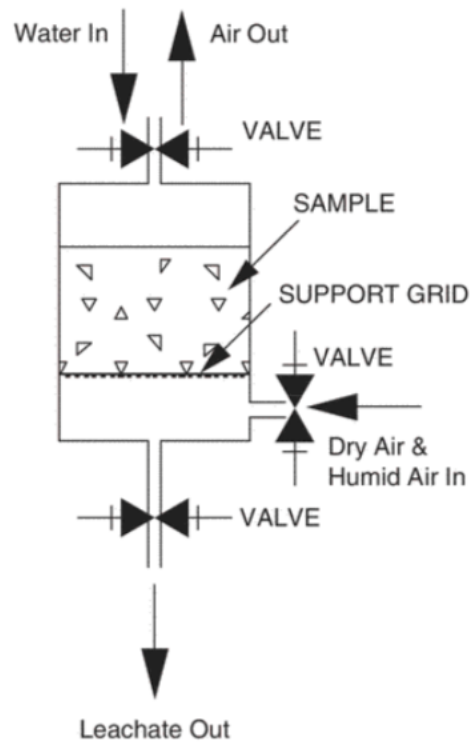
#### **1.2.4.2 Sobek Method – Neutralization Potential**

The Sobek method (Sobek et al., 1978; Downing, 2014a) is used to determine the neutralization potential of a mineral-based material. The method determines the amount of neutralizing bases present in a sample by treating the sample with a known quantity of standardized hydrochloric acid (HCl). The amount of HCl added to the 2 g test sample is determined by adding several drops of the HCl solution to a separate smaller sample (approximately 0.5 g) and qualitatively assessing the audible fizzing sound produced by the reaction. This qualitative assessment corresponds to a recommended volume of the HCl solution that will be added to the 2 g test sample. The test sample and the HCl solution are combined in a flask and heated to encourage the reaction to take place. Once the reaction is complete, the amount of unused acid (in CaCO<sub>3</sub> equivalent mass) is determined by titration with a standardized sodium hydroxide (NaOH) solution to a neutral pH of 7.0. If a small volume of the NaOH solution (generally less than 3 mL) is required to reach a pH of 7.0, it is likely that the amount of HCl solution added was not sufficient to completely neutralize the test sample. In this case, the test procedure should be repeated with a new sample and a higher volume of the HCl solution. Once the procedure is complete, the CaCO<sub>3</sub> equivalent mass per mass of sample material can be determined by the volumes and normalities of the solutions used. The Sobek method is implemented by the Dalhousie Mineral Engineering Centre to determine neutralization potential.

#### **1.2.4.3 Humidity Cell Testing**

A kinetic humidity cell test attempts to mimic real-world weathering of waste rock material to determine acid-generating potential over time. The current procedure used in Canada can be found within ASTM D5744-96 Standard Test Method for Accelerated

Weathering of Solid Material Using a Modified Humidity Cell (ASTM, 1996; Downing, 2014b). In this test, a small amount of waste rock material (around 1 kg) is placed in a humidity cell (Figure 1) and subjected to repeated seven day weathering cycles for upwards of 15-25 weeks. A cycle generally consists of exposure to ambient (relatively dry) and humidified air with leaching phases spaced intermittently. These leaching phases consist of adding deionized water to the sample, letting it sit for 2-3 hours, and then flushing the water out of the sample where it is then collected for testing. The pH of the effluent leachate is measured to help determine acidity, while other tests (such as metal concentrations) may be performed to further characterize the effluent leachate.



**Figure 1 – Humidity Cell Testing Apparatus (Downing, 2014b).**

### **1.2.5 Performance Testing of Cement-Based Solidification/Stabilization Materials**

In this thesis, the stabilization and solidification of a waste rock material with a cement-based binder will be considered. Several common industry testing methods that may be used to determine the effectiveness of such treatment are described in the following sections. With respect to waste material obtained from urban development, these tests are commonly used for contaminated soil applications but rarely for acid-producing waste rock applications.

#### **1.2.5.1 Mass Flux Testing**

Mass flux (also called mass transfer) leaching procedures may be used when evaluating the effectiveness of S/S implementations. One such testing procedure is described by the USEPA SW-846 Test Method 1315: Mass Transfer Rates of Constituents in Monolithic or Compacted Granular Materials Using a Semi-Dynamic Tank Leaching Procedure (USEPA, 2017). The procedure involves placing a solidified material in a series of water baths to determine rates of contaminant leaching over time. The ratio of the volume of water in each leachate bath to the surface area of the sample is recommended to be approximately 10 (in milliliters) to 1 (in squared centimeters). The solidified sample is placed into a water bath (called eluent) for a given time interval, after which the sample is removed, and the remaining water (eluate) may be tested for a variety of parameters. The length of the time interval generally starts at 2 hours and increases as the test progresses. The number of time intervals over which the test is performed will vary depending on the established eluate requirements.

### **1.2.5.2 Synthetic Precipitation Leaching Procedure (SPLP)**

The synthetic precipitation leaching procedure (SPLP) is a test method used to determine the mobility of contaminants in liquid and solid samples (USEPA, 1994). This method proves particularly useful for solid samples, as liquid is used to simulate contaminant leaching from a solid material. Samples containing both solid and liquid phases are separated, and the solid phase may be crushed or otherwise have its grain size reduced, if necessary. The solid phase is extracted with a mass of liquid approximately 20 times the mass of the solid sample portion. The liquid used for this extraction may vary depending on the application such as being formulated to have a pH near that of local rainwater. The results are generally presented as concentrations of various constituents present in the sample (USEPA, 1994). Due to the composition of the extracting liquid and the nature of the test, some constituents cannot be analyzed. These parameters included sulfate, sulfide, and sulfur, which are key metrics by which the leachate quality is being assessed in this thesis.

### **1.2.5.3 Hydraulic Conductivity and Unconfined Compressive Strength**

Hydraulic conductivity and unconfined compressive strength (UCS) testing are used to define key physical properties of a treated material. Hydraulic conductivity is a measurement of the rate at which liquid can flow through a porous material and hence is indicative of how water (or other liquids) will potentially flow through the solidified material. Compressive strength is often performed on cement-treated samples as an indication of the homogeneity of the mixing process and the effectiveness of the cement added to the material in providing “solidification” to the material.

### 1.3 Synthesis of Literature Review and Thesis Objectives

The overwhelming majority of research on the topic of cement stabilization and solidification for acid-generating waste rock is focused on the mining industry. Most cement-based S/S research related to acid-generating waste rock has focused primarily on fine-grained tailings and not the coarse-grained material often encountered in urban excavation projects. It is hypothesized that differences in surface area between fine-grained tailings and urban excavation waste rock will affect the acid-generation potential of the waste material, as well as the material's response to cement or other binding agents. It also appears that the commercial use of cement solidification and stabilization techniques for urban acid-generating rock is not widely used. Many of the common performance test techniques for cement-based S/S treatment of contaminated soils do not capture the necessary environmental exposure reactions of acid waste rock testing and hence it is questionable whether these two approaches to performance testing (i.e. acid waste rock versus cement-based S/S) are comparable.

Based on this literature review, there are several objectives to this research:

- Assess the acid producing potential of a waste rock material obtained from an urban construction site in HRM with a common testing technique (humidity cell testing was chosen based on the ability to extend testing over weeks, months, or years and the high potential to detect contaminants in the leachate) used in the mining industry,
- Assess the performance of a cement-based binder at reducing the potential for acid production and metal leaching using humidity cell testing,

- Compare the results of humidity cell testing (common mining industry performance test) to a more conventional mass flux test to assess the performance of the cement-based binder treatment.

## **Chapter 2 Evaluating the Performance of Cement-Based S/S in Reducing the Acid Producing Potential of an Urban Excavated Sulfide Bearing Rock Material**

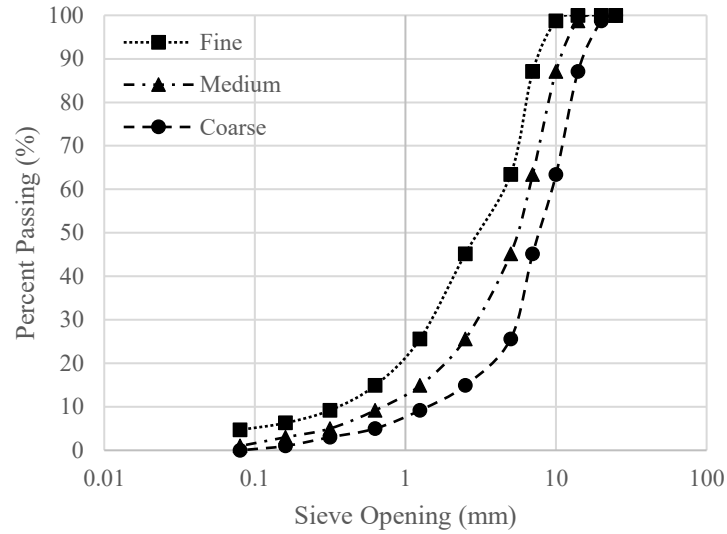
### **2.1 Introduction**

Acid producing waste rock is a common by-product of urban construction in Nova Scotia. When acid producing rock is disturbed through excavation, the exposed surfaces can readily react with water and oxygen in the environment to produce potentially harmful acidic leachate. Several techniques are currently used to reduce the potential for acid production, including cement stabilization and solidification (S/S). Cement S/S treatment consists of combining waste rock with cementing materials that have the ability to both neutralize acidity through chemical neutralization potential and reduce the interaction of oxygen and water with the waste rock through cement hardening. This chapter outlines the methods used to test the efficacy of a sulfide bearing slate waste rock treated with cement-based S/S materials. These methods include humidity cells, mass flux, hydraulic conductivity, and unconfined compressive strength.

## **2.2 Materials and Methods**

### **2.2.1 Material Characterization**

Slate pieces (generally between 150 mm and 300 mm in diameter) were retrieved from a waste rock stockpile comprised of material from various urban excavation projects throughout the Halifax Regional Municipality (HRM). Particle-size reduction for the research was performed by subsequent crushing (Whitelaw Machinery Company, Model 0) which reduced the slate's maximum particle size to a nominal 20 mm diameter. When a particle size less than 1 mm was required, the slate was placed in a chain impact mill for further size reduction. Slate particles were separated according to various sieve sizes and stored in a low humidity environment in plastic containers until the creation of the pre-determined grain-size profiles required for testing. Three different grain size distributions ("fine", "medium", and "coarse") were established for this research, as shown in Figure 2. To create these grain-size distributions, a 1000 g dry slate sample was created from the various size fractions of the crushed slate. These grain-size distributions were constructed based on the size of the humidity cell testing apparatus, and to produce samples with distinctly different slate surface area characteristics.



**Figure 2 – Fine, Medium, and Coarse Slate Grain-size Distributions Used for Testing.**

In addition to the slate, a cohesionless glacial till (referred to as “soil” hereafter) was obtained from a local construction site within HRM for blending purposes with the slate. This material was used to simulate co-disposing the slate with soil, as may be done in real-world applications. Approximately 60 kg of this soil was sampled and subsequently screened on a 9.5 mm sieve for further use; all material retained on the screen was discarded. A United Soil Classification System (USCS) descriptor of silty sand (SM) was determined for this screened material (ASTM, 2025). A standard proctor compaction test was performed on the screened soil according to ASTM D698-21 (ASTM, 2021a) and yielded a maximum dry density of approximately 2080 kg/m<sup>3</sup> at an 8.0% optimum water content.

The binder materials used in this thesis consisted of McInnis general use (GU) cement (i.e. “cement”) and Trenton #5 fly ash (i.e. “fly ash”). Further details on the

composition of these materials and their use in the research are described in the various following sections.

### **2.2.2 Mixture Preparation**

As outlined in Table 1, three different types of mixtures of the previously described materials were used for various testing in this research: slate (S), combined slate and binder (SB), and combined slate, soil, and binder (SSB). As noted in the previous section, three different slate grain sizes (fine (F), medium (M), and coarse (C)) were used when preparing mixtures. Table 1 presents the various proportions, as well as the notation used for these mixtures. When binder was used, the percentage was held constant at 6% (5% cement and 1% fly ash), expressed as a percentage of dry mass of the slate or combined soil/slate. These binder content values were selected to represent typical binder proportions used at Envirosoil's facility for other S/S projects. The binder and water/binder ratio used in Table 1 has been shown in previous projects to provide 7-day unconfined compressive strengths exceeding 350 kPa (a typical industry specification for S/S projects). An additional sample (CF) consisting of cement, fly ash, and water was prepared for humidity cell testing for as a baseline comparison. The medium grain-size slate was removed from binder-treated testing simply to reduce the total number of tests/samples submitted.

**Table 1 – Mixture Notation and Proportions Used.**

<b>Mixture Notation</b>	<b>Slate Grain Distribution</b>	<b>Dry Slate Mass (g)</b>	<b>Dry Soil Mass (g)</b>	<b>Dry Cement Mass (g)</b>	<b>Dry Fly Ash Mass (g)</b>	<b>W/CM* Ratio</b>
S-F	Fine	1000	0	0	0	N/A
S-M	Medium	1000	0	0	0	N/A
S-C	Coarse	1000	0	0	0	N/A
SB-F	Fine	1000	0	50	10	1.17
SB-C	Coarse	1000	0	50	10	1.17
SSB-F	Fine	500	500	50	10	1.33
SSB-C	Coarse	500	500	50	10	1.33
CF	N/A	N/A	N/A	50	10	0.42

\*Cementing Materials (CM) includes cement and fly ash. Water (W) denotes deionized water.

Table 2 outlines the various performance tests on the different mixtures of slate, binder, and soil. Details are provided in the following sections.

**Table 2 – Various Testing Performed on Material Mixtures (“Y” Denotes Test Performed).**

<b>Mixture Notation</b>	<b>Humidity Cell</b>	<b>Mass Flux</b>	<b>Unconfined Compressive Strength</b>	<b>Hydraulic Conductivity</b>
S-F	Y			
S-M	Y			
S-C	Y			
SB-F	Y	Y	Y	Y
SB-C	Y		Y	Y
SSB-F	Y	Y	Y	Y
SSB-C	Y		Y	Y
CF	Y			

Samples for unconfined compressive strength, hydraulic conductivity, and mass flux were compacted in single-use plastic molds (50 mm in diameter and 100 mm in height for mass flux samples and 75 mm in diameter and 150 mm in height for UCS and hydraulic

conductivity samples) using standard proctor compactive effort (600 kN-m/m<sup>3</sup>) (ASTM, 2021a).

### **2.2.3 Test Methods**

#### **2.2.3.1 Chemical Characterization of Slate, Soil, Cement and Fly Ash**

A series of chemical analyses were performed by the Materials Engineering Centre at Dalhousie University on individual samples of slate, soil, cement, and fly ash. The net neutralizing potential of each sample was determined using the EPA (Sobek) method (Sobek et al., 1978). An oxide analysis was performed by lithium borate fusion/inductively coupled plasma – optical emission spectrometry (ICP-OES). Acid producing potential was determined using total sulfur/sulfate/sulfide content.

#### **2.2.3.2 Unconfined Compressive Strength and Hydraulic Conductivity**

Preparation of samples for unconfined compressive strength (UCS) and hydraulic conductivity testing involved preparing the proportions outlined in Table 1 and thoroughly mixing them together by hand with a metal spoon. The mixtures were compacted in single-use plastic molds (75mm in diameter and 150mm in height) using approximately standard proctor compactive effort (ASTM, 2021a). Samples were allowed to cure for a minimum of 7 days prior to testing.

UCS testing was performed on cured samples in general accordance with ASTM D2166 (ASTM, 2017). Neoprene caps were used on either end of each sample to provide a smooth surface that was perpendicular to the direction of loading. A strain rate of 1.3 mm/min was used for all samples.

Hydraulic conductivity testing was performed using a flexible wall permeameter (ASTM, 2016) on cured samples. All samples were saturated incrementally to a maximum cell pressure and back pressure of 621 kPa and 455 kPa, respectively. Consolidation of the material was not performed due to the high stiffness of the samples. Permeation was performed with a hydraulic gradient of 19 and an effective confining stress of 151 kPa. Testing was terminated when inflow and outflow volumetric readings from the samples did not vary significantly for four consecutive readings.

### **2.2.3.3 Mass Flux Testing**

Mass flux testing involved preparing the proportions outlined in Table 1, thoroughly mixing them together by hand with a metal spoon and compacting them into single-use plastic molds (50 mm in diameter and 100 mm in height) using approximately standard Proctor compactive effort (ASTM, 2021a). Mass flux samples were allowed to cure for a minimum of 28 days, after which they were demolded, weighed, and measured for length and diameter.

A modified mass flux testing procedure used was based on ASTM C1308 (ASTM, 2021b) and USEPA 1315 (USEPA, 2017). Each sample was placed in separate non-reactive plastic containers each holding 2000 g of deionized water (at 0 hours). A harness apparatus was used to suspend the sample in the container, allowing the entire surface area of the sample to interact with the surrounding water. At an elapsed time of 2 hours, the samples were removed from their respective containers and immediately transferred to another container holding 2000 g of “fresh” deionized water. This process was repeated at elapsed times of 7 hours and 24 hours. At an elapsed time of 24 hours, samples were

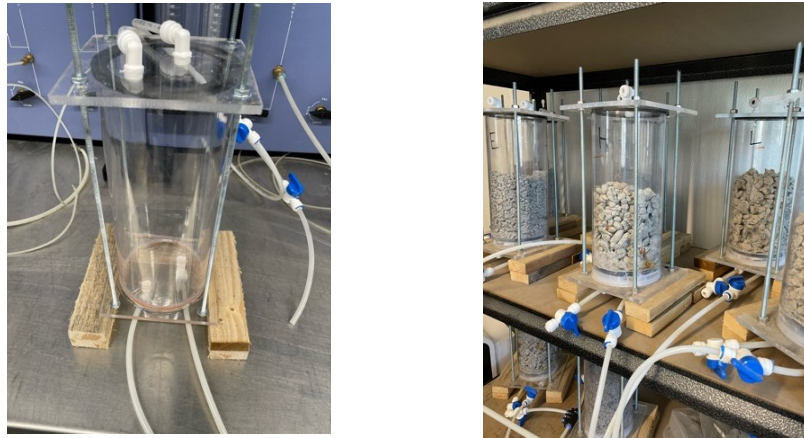
removed from the containers and set in open air for a further 24 hours to promote oxygen transfer to the samples. At an elapsed time of 48 hours, the samples were placed back into containers holding “fresh” deionized water for an additional 24 hours. This process of 24 hours in water and 24 hours in air was repeated. The total elapsed test time was 22 days. The leachate generated after each removal of the sample from the deionized water was tested for pH immediately after collection using an Apera PH400 handheld pH meter. The bulk of the leachate was processed by a third-party laboratory for the following tests: pH, total metals, mercury, cyanide, sulfur, sulfides, and anions using standard laboratory procedures (see Appendix C). A total of 13 leachate samples were generated and submitted for testing.

#### **2.3.3.4 Humidity Cell Testing**

As shown in Table 2, baseline humidity cell tests were performed on untreated, crushed slate using the three different grain-size distributions presented in Figure 2. Each sample had a mass of 1 kg. The slate samples used for humidity cell testing were placed directly in the humidity cell test cell by hand with no compactive effort applied.

Preparation of the binder-based samples for humidity cell testing involved preparing the proportions outlined in Table 1 and thoroughly mixing them together with a metal spoon. This mixture was then spread out into a thin layer to cure for a minimum of 28 days. The material was spread in a thin layer to prevent the formation of large aggregations, as required by the humidity cell testing procedure. Triplicate samples were prepared for humidity cell testing.

Humidity cell testing of the selected samples was performed according to a modified procedure based on ASTM D5744 (ASTM, 1996). The apparatus used in this procedure, as shown in Figure 3, consisted of three main components: the humidity cell, a pressurized air supply, and an air-humidifying cell. Each humidity cell consisted of a plexiglass cylinder (100 mm in diameter and 225 mm in height) which was sealed on the bottom with a fixed plate; the top was sealed with a removable, air-tight plate and gasket. Valves and plastic tubing were connected to the top and bottom plates to allow the flow of air and water in and out of both the top and bottom of the humidity cell. The cell interior housed a slotted plastic spacer onto which a 100 mm diameter porous stone and filter paper were placed. The plastic spacer ensured the porous stone did not impede the flow of air or water fluid out of the bottom valves.



**Figure 3 – Humidity Cell Testing Apparatus.**

For testing, the sample of interest was placed on top of the filter paper. The top of the cell was then secured, and tubing connected. The regulated air supply was then set to a maximum pressure of 69 kPa (10 psi), which was fed through a series of 5 mm diameter

plastic tubing sections, valves, and splitters to control airflow to the bottom of the appropriate cells.

Each humidity cell was run for a minimum of 16 weeks, with each week equating to one cycle. A cycle consisted of the following: a leaching phase with deionized water, a forced-air phase with dried air (less than 20% humidity), and a forced-air phase with humidified air (greater than 70% humidity). The leaching phase took place on day one of the seven-day cycle. During this phase, the humidity cell was disconnected from all air sources and the bottom valves closed. Approximately 1050 mL of deionized water (a volume based on the size of the sample and the amount of leachate required to complete necessary testing) was added to the sample and when necessary, mild agitation was applied to the sample to remove large air pockets. This water was allowed to sit in the humidity cell for  $120 \pm 5$  minutes. After this time, the bottom valve designated for leachate drainage was opened and the leachate was collected in non-reactive plastic specimen containers for further testing. Once all free-draining leachate was collected, the leachate drainage valve on the humidity cell was closed and the sample was allowed to dry for approximately 24 hours. On day two, the humidity cell was reconnected to the dry air supply, continuing for days three and four. On day five, the cell was switched to the humidified air supply for days six and seven. At the end of the seven-day cycle, the entire cycle was repeated (for a minimum of 16 weeks).

The pH of the leachate collected during each leaching phase was measured immediately after collection using an Apera PH400 handheld pH meter. The bulk of the leachate was processed by a third-party laboratory for the following tests: pH, total metals, mercury, cyanide, sulfur, sulfides, and anions using standard laboratory procedures (see

Appendix C). Although pH was measured on-site every week, third-party testing was only performed on samples every two weeks until week 8, and then every 4 weeks until the completion of the humidity cell testing. Each humidity cell was run until little change in pH and leachate constituent concentrations were seen, at which time the test was terminated.

The reported detection limit (RDL) is the minimum concentration of an individual parameter that may be detected using a given laboratory technique and varies by parameter. In cases where reported concentrations are below the RDL, a value equal to half of the RDL was used for calculations of average concentration.

## 2.3 Results

### 2.3.1 Chemical Characterization of Slate, Soil, Cement, and Fly Ash

Testing for total sulfur and sulfate sulfur is required as part of Nova Scotia's Environment Act related to sulfide bearing materials (Nova Scotia, 1994). According to the Act, a sulfide bearing material is one in which the acid producing potential (sulfide sulfur) content is equal to or greater than 0.4% (acid producing potential equal to or greater than 12.51 kg H<sub>2</sub>SO<sub>4</sub>/tonne). As is common for sulfide bearing rock in the HRM area, Table 3 shows that the slate used in this study exhibits an acid producing potential greater than provincial guidelines and hence requires special disposal procedures (Nova Scotia, 1994). The other materials used in this study are also shown in Table 3 and exhibited little to no acid-producing potential. The cement had the highest sulfur percentage by weight with most of that sulfur present as sulfate. No sulfur was detected in the soil sample.

**Table 3 – Total Sulfur, Sulfate, Sulfide Content and Acid Producing Potential (Sulfide Sulfur) of the Slate, Soil, Cement and Fly Ash.**

Sample	Weight Percentage (%)			kg/t H <sub>2</sub> SO <sub>4</sub>
	Total Sulfur	Sulfate	Sulfide	Acid Producing Potential
Slate	0.545	0.026	0.519	15.86
Soil	<0.001	<0.001	<0.001	<0.03
Cement	1.289	1.238	0.051	1.56
Fly Ash	0.480	0.401	0.079	2.42

Although acid producing potential can indicate the ability of material to produce acidic leachate, neutralization effects due to other minerals present may help to buffer this acid producing potential. Acid-base-accounting, also called neutralization potential (Sobek et al., 1978), was performed to establish the net acid generation and neutralization capacities of the individual sample components (see Table 4). As shown in Table 4, the

slate material has high acid producing potential with limited neutralization potential. The cement exhibited the highest value of net neutralizing potential, by an order of magnitude. The soil exhibited limited acid or neutralizing potential. In summary, the slate is a net producer of acid while the cement was the predominant net acid neutralizer.

**Table 4 – Acid-Base Accounting Results for the Slate, Soil, Cement, and Fly Ash.**

Sample	Kg CaCO <sub>3</sub> Eq./Ton		
	Acid Potential	Neutralizing Potential	Net Neutralizing Potential
Slate	16.22	2.5	-13.7
Soil	<0.03	3.3	3.3
Cement	1.59	914.0	913.0
Fly Ash	2.47	12.0	9.5

The results of an oxide analysis to establish the major elements present in the slate, soil, cement, and fly ash, are shown in Table 5 by weight percentage detected in each sample. A complete list of all detected oxides in the four materials is presented in Appendix A.

**Table 5 – Results of Major Oxide Analyses on Slate, Soil, Cement, and Fly Ash (rounded to nearest tenth of percent).**

Sample	Oxide (weight percentage)						
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MnO	Na <sub>2</sub> O	SrO
Slate	61.1	22.4	6.2	0.3	0.1	1.1	0.0
Soil	74.4	10.8	3.7	0.5	0.1	1.8	0.0
Cement	19.5	4.6	3.2	63.6	0.0	0.2	0.1
Fly Ash	51.4	20.5	10.7	2.1	0.0	0.8	0.1

### 2.3.2 Unconfined Compressive Strength and Hydraulic Conductivity

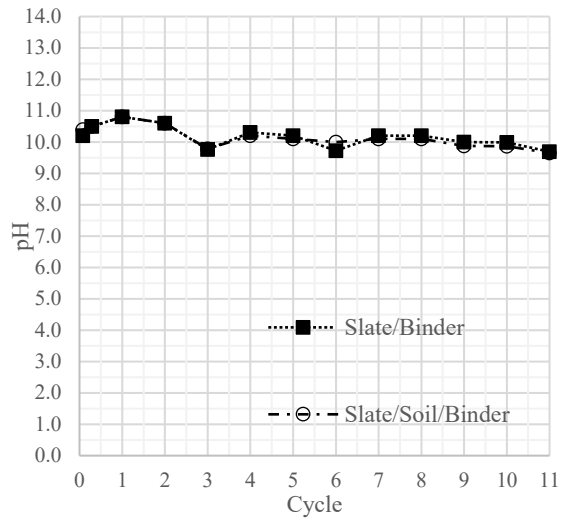
The hydraulic conductivity and unconfined compressive strength of the slate and slate/soil binder mixtures are presented in Table 6. The slate mixtures with the binder showed a relatively porous matrix compared to the slate/soil mixtures with binder. In cases where soil was added to the slate and binder mixture, the hydraulic conductivity of the produced sample was lowered significantly. The hydraulic conductivity results of the slate and soil binder-treated samples were in a similar order of magnitude to a recompacted local clayey glacial till. Additionally, the average compressive strength of these samples was higher than those containing only slate and binder materials. This can likely be attributed to the fact that the slate pieces are angular in shape and generally form a fairly open soil matrix when compacted. The addition of soil, which consists of more rounded particles, appears to create a more well-graded material with a lower void ratio.

**Table 6 – Hydraulic Conductivity and Unconfined Compressive Strength Results for Slate and Slate/Soil Binder Mixtures.**

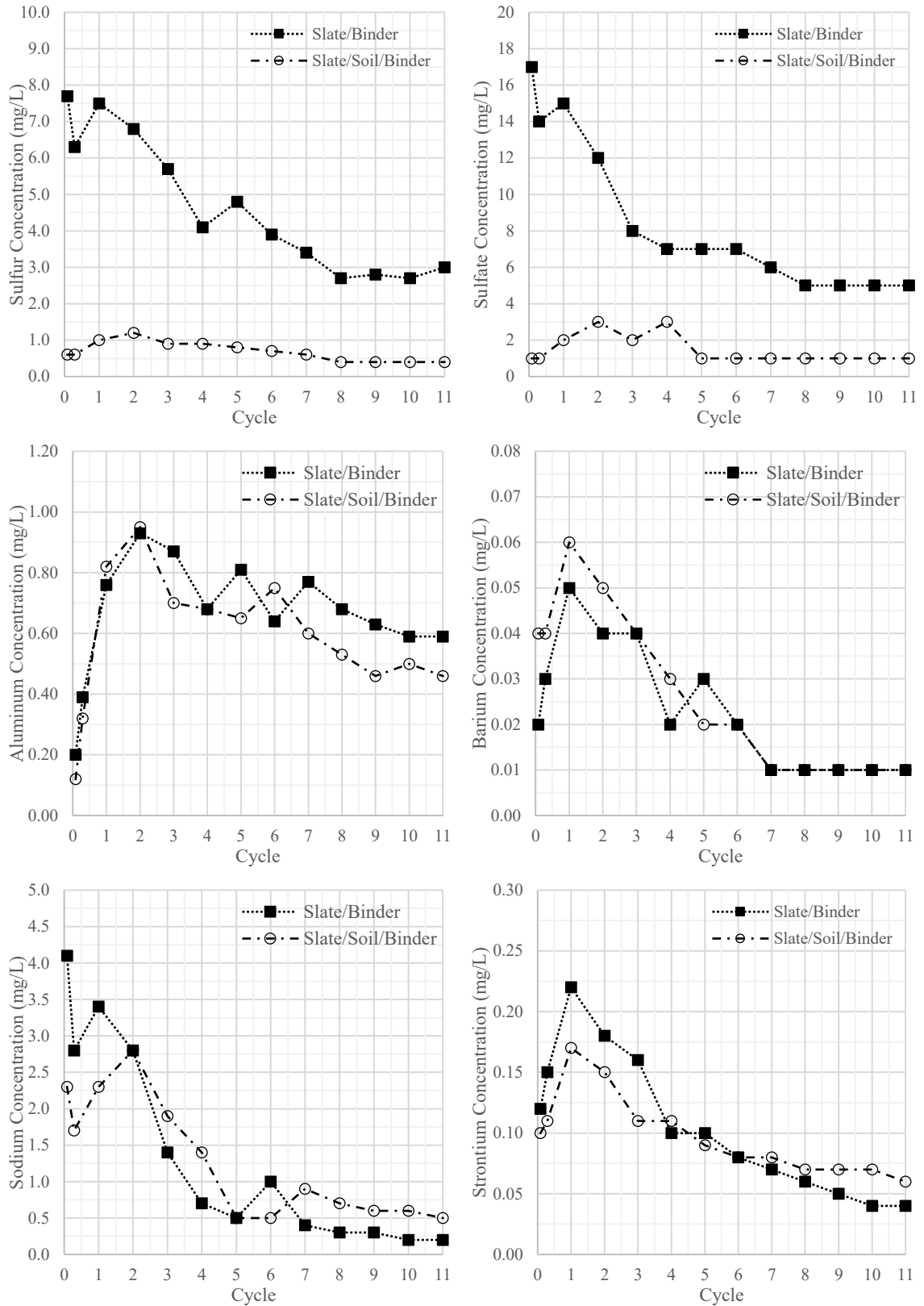
Sample	Hydraulic Conductivity (m/s)	Unconfined Compressive Strength (kPa)
SB-F	$1.9 \times 10^{-6}$	2900
SB-C	$1.3 \times 10^{-6}$	1720
SSB-F	$1.4 \times 10^{-8}$	4030
SSB-C	$1.9 \times 10^{-9}$	4200

### 2.3.3 Mass Flux Results

Mass flux results are shown on Figure 4 and Figure 5. Six chemical constituents were consistently detected over time in the mass flux tests. Sulfate, sulfur, aluminum, barium, sodium, and strontium were found to be consistently present in the eluate while all other chemical constituents tested for (see Appendix C) were not consistently present.



**Figure 4 – Mass Flux Results: pH.**



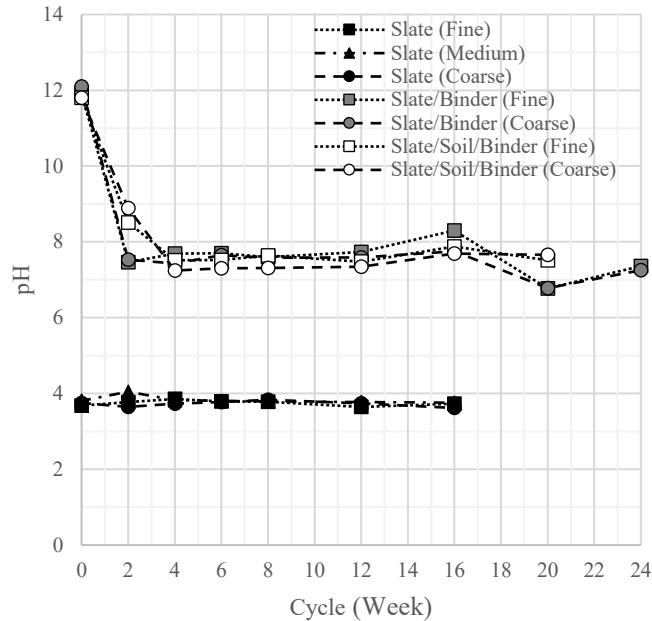
**Figure 5 – Mass Flux Results: Detectable Constituents in Eluate.**

When comparing the SB and SSB mixtures, difference in pH levels between these two samples were minor and ranged between 9.5 and 10.0 near the end of the test. When comparing the two mixtures with respect to the various constituents detected, aluminum, barium, sodium, and strontium showed similar concentrations throughout the test duration. However, sulfur and sulfate concentrations were generally higher in the SB sample compared to the SSB sample with concentrations beginning around 8 mg/L and 17 mg/L, respectively. Sulfur and sulfate concentrations started to show equilibrium trends at the end of the test period but with sulfur being consistently higher for the SB samples at the end of the test. This is not surprising when revisiting the sulfur and sulfate results in Table 3 where the slate had considerably more sulfur and sulfate than the soil. Since the SSB samples are 50% slate and 50% soil (ignoring binder), the slate is “diluted” within the mixed samples.

#### **2.3.4 Humidity Cell Results**

Figure 6 shows pH values obtained from humidity cell testing. As shown in Figure 6, the pH of the untreated slate samples remained consistently at around 4 over the entire testing period. This would confirm the potential acid-production potential of the slate in Table 4. It also confirms the effectiveness of the test method to generate acid producing conditions from the slate in the short and long term (up 16 to 24 weeks). As shown in Figure 6, treated samples exhibited high initial pH values (around 12) which decreased to between 7 and 8 over 2 to 4 cycles, after which values remained generally constant until the end of the testing procedure. These results are interesting in that it appears even a thin coating of binder (the samples are not in monolith form) appears to have eliminated acid production of the slate samples for the duration of testing. Given that this is a key measure

by which the effectiveness of the S/S treatment is assessed, this appears to be a successful treatment. Slate grain-size distribution and slate to soil ratio appeared to have little measurable influence over reported pH values both in untreated and treated samples.



**Figure 6 – Humidity Cell Results: pH.**

Leachate concentrations from the humidity cell tests are shown in Figures 7 and 8. The following five constituents were not detected in any leachate samples and hence are not shown on these figures: antimony, beryllium, bismuth, thallium, and tin. The following constituents were detected in one or more leachate samples, however, are also not shown on Figures 7 and 8: bromide, chloride, fluoride, nitrate, nitrite, mercury, arsenic, barium, chromium, lead, lithium, molybdenum, silver, vanadium, uranium, sulfide, and cyanide; these results are typically omitted as the concentrations were too low, inconsistent, or not detected over enough cycles to determine trends. All leachate test results from humidity cell testing are provided in Appendix B. Figures 7 and 8 show humidity cell leachate results for the following constituents: aluminum, cadmium, cobalt, copper, iron, manganese,

nickel, and zinc. For all these constituents, the untreated slate samples showed the highest initial concentrations, decaying in an approximately exponential manner over the following several weeks. Concentrations continued to decrease slightly each week throughout the duration of testing. Differences in measured concentrations between the slate grain-size distributions (fine, medium, and coarse) were most significant in the first several weeks; however, as the testing progressed, the concentrations became more similar between the different grain sizes. In general, highest initial concentrations were measured in sample S-M, followed by S-F, and S-C for the slate samples. In samples treated with binder (i.e. SB and SSB), concentrations remained low (in some cases below the RDL) throughout the testing procedure relative to levels reported in untreated slate samples. Grain-size distribution of the slate appeared to be an insignificant factor in binder treated samples for these constituents.

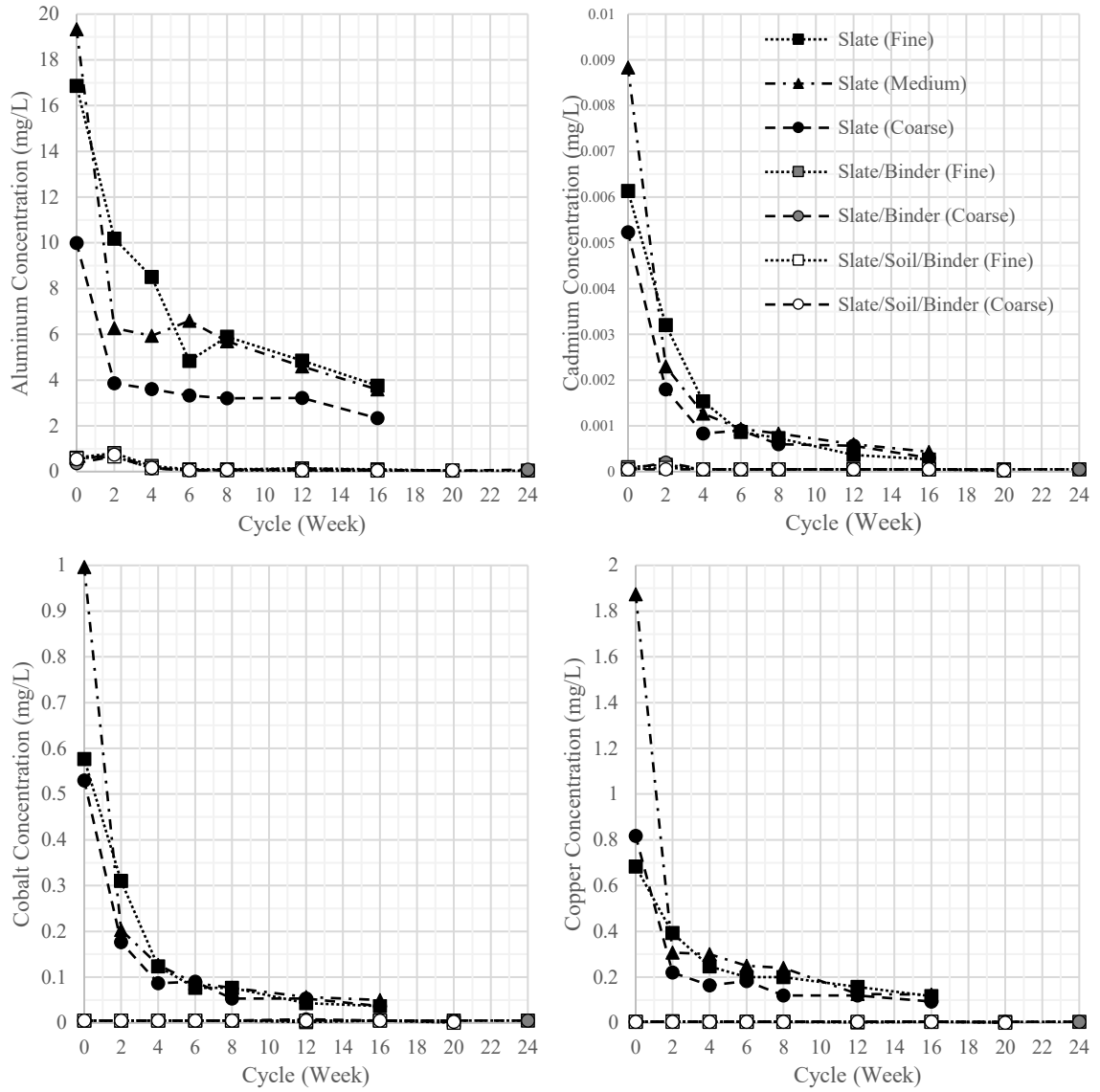
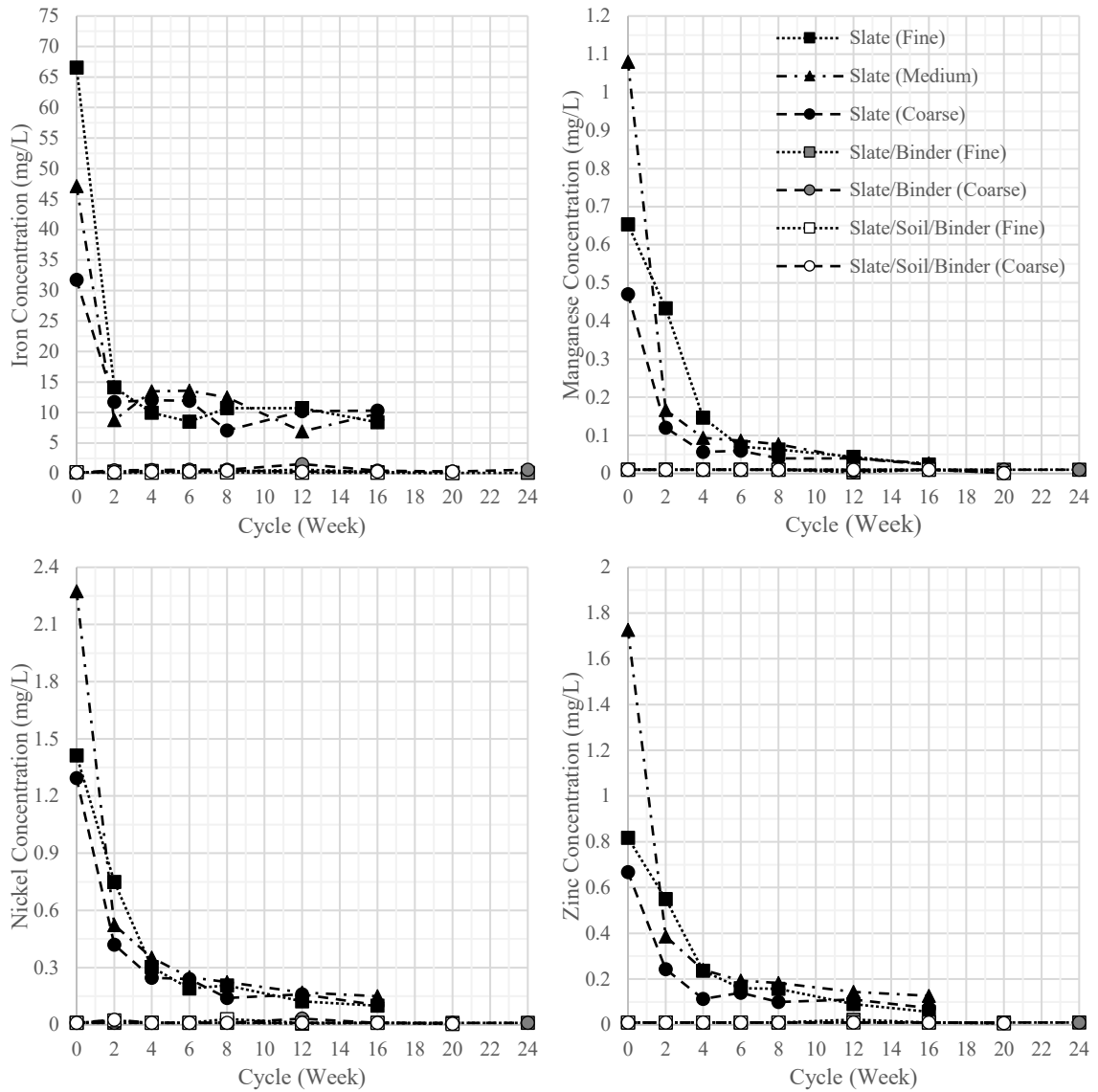


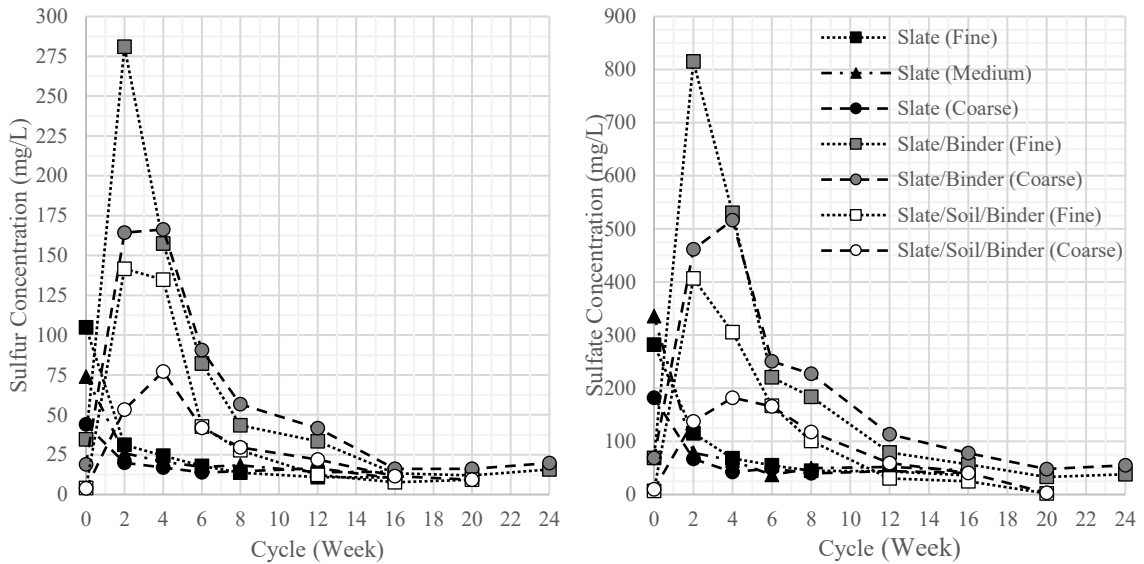
Figure 7 – Humidity Cell Results: Al, Cd, Co, and Cu.



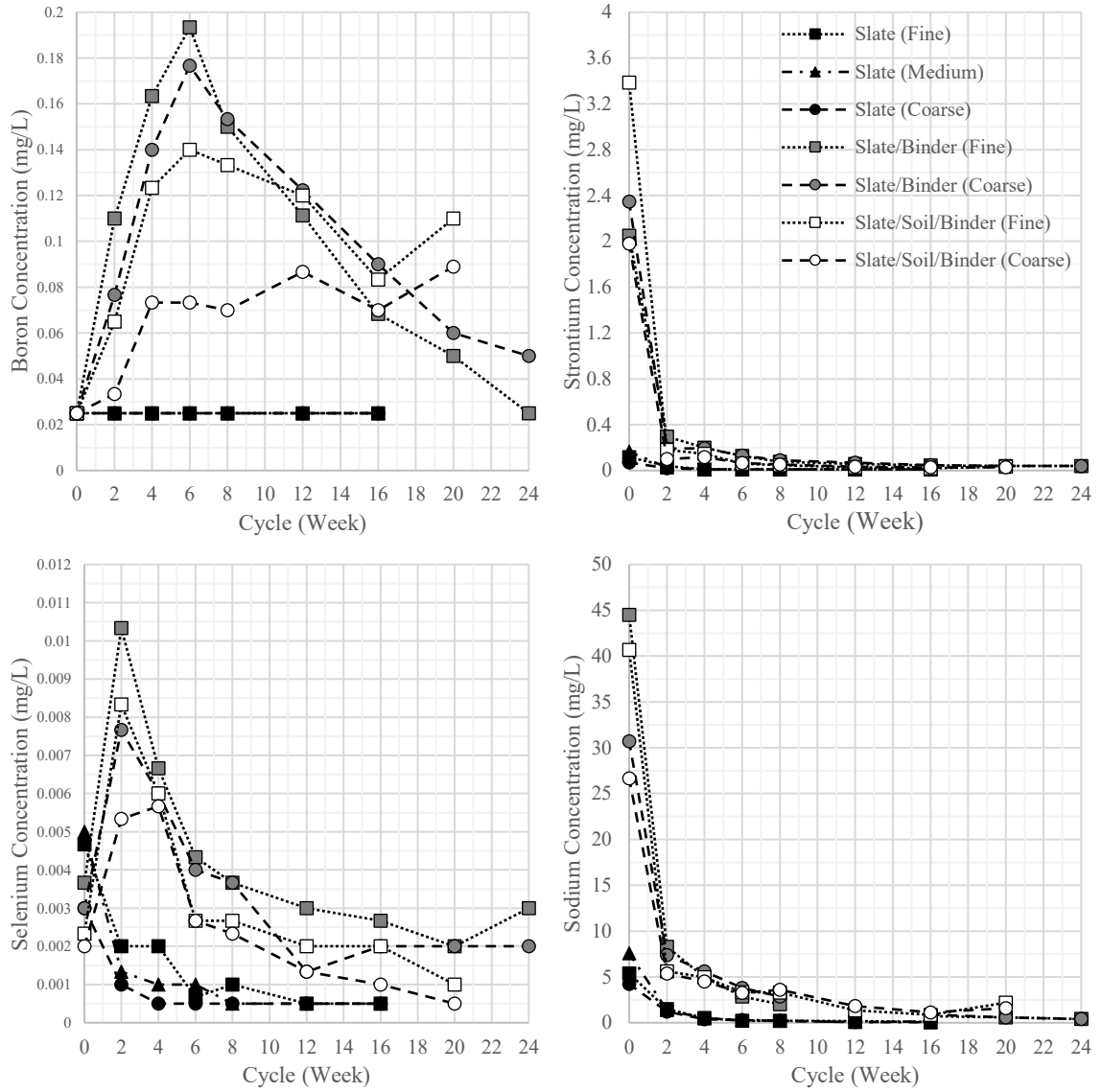
**Figure 8 – Humidity Cell Results: Fe, Mn, Ni, and Zn.**

Figures 9 and 10 show humidity cell results for the following constituents: sulfur, sulfate, boron, strontium, selenium, and sodium. For sulfur and sulfate (Figure 9), slate-only samples showed higher initial leachate concentrations (week 0) followed by a leveling-off over the duration of the test. For SB and SSB samples (Figure 10), the week 0 leachate level was lower than the slate but peaked in weeks 2-4 followed by a drop in

concentration for the remainder of the test. Given that the cement binder contains the most sulfate of any of the materials (see Table 3), it is not surprising to see these results and may be an artifact of surface wash off from the cement during initial leaching. Similar trends were noticed for selenium. Boron showed elevated leachate for the binder samples but below RDL leachate values for the slate without binder. Sodium and strontium showed initial elevated leachate levels in early weeks for the binder samples which may also be attributed to surface washoff of binder during initial leaching. Based on oxide analysis, sodium is present in the soil and strontium is present in the cement and fly ash binder (Table 5). The influence of slate grain-size distribution and slate to soil ratio varied for different parameters.



**Figure 9 – Humidity Cell Results: Sulfur and Sulfate.**



**Figure 10 – Humidity Cell Results: B, Sr, Se, and Na.**

## **2.4 Discussion**

### **2.4.1 Effect of Binder Materials on Controlling pH Levels from Rock Samples**

The use of the cement/fly ash binder material was very effective at controlling the long-term (several months) and short-term (hours in the case of mass flux testing) evolution of pH from the samples containing slate. Limited testing on the binder-only (CF) samples indicated an initial pH of over 11, dropping to 8.5 by week 4, a trend similar to the binder-treated slate and slate/soil (SB and SSB) samples (Figure 6). When comparing humidity cell testing on untreated slate samples to that of treated samples, the slate samples consistently had low pH values over the 16-week testing period (Figure 6). The addition of binder materials to the slate sample had a significant effect, keeping the pH to above 7.0 for the 16 weeks of testing. It should also be noted that the samples were prepared in aggregated form (i.e. non-monolithic), in an attempt to simulate potential long-term environmental stress cracking in monolithic samples (Jamshidi et al. 2016; Lake et al. 2017). The addition of soil (and thereby the reduction of slate by 50% by weight) appeared to have little impact on the pH over time. Grain size also did not appear to affect pH evolution as the introduction of the binder to the slate reduced the surface area of the samples exposed to the leachate (and oxygen). Higher pH values were observed to be maintained during the duration of the mass flux testing with both samples tested (Figure 4). Differences between humidity cell testing and mass flux testing is likely the result of differing surface areas in contact with the oxygen/water during the duration of the test, as described previously. One way to demonstrate this concept is the liquid to solid ratio (L/S) or the mass of water used to “leach” a specimen compared to the mass of the specimen tested. The L/S of each humidity cell and mass flux test were approximately 1 and 5,

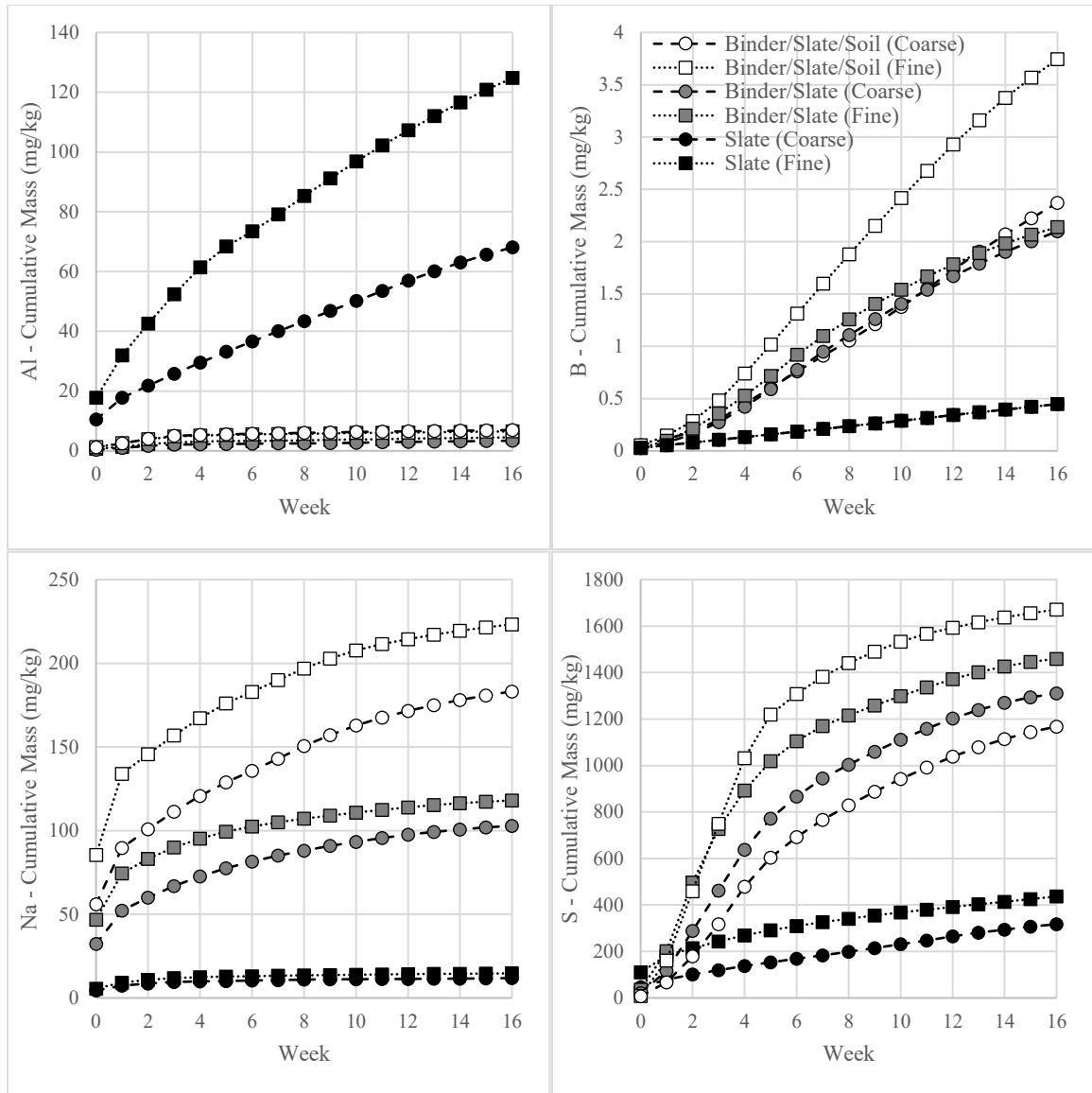
respectively. The higher L/S of the humidity cell tests resulted in a wider array of detected contaminants and higher concentrations of these contaminants when compared to mass flux testing. In essence, the negligible hydraulic conductivity, extended duration, and low L/S value allow the humidity cell testing to represent a worst-case scenario (which may be beneficial in determining the total array of possible contaminants) when compared to other testing procedures, such as the mass flux (which showed fewer detectable contaminants). If this was an actual project and these pH and leachate levels were not suitable, the binder design could be further optimized to produce better results as defined by the local regulations or specified performance targets. That being said, the humidity cell tests, as performed in this thesis are likely over-conservative in their L/S ratios for S/S projects. Since the time it would take to reach L/S ratios of that for the humidity test samples, it is likely that other processes such as mineral surface precipitation of iron would limit the rate of pH evolution before the high L/S ratios became a factor (Mahoney et al., 2019).

#### **2.4.2 Effect of Binder Materials on Cumulative Mass of Leaching Contaminants in Humidity Cell Testing**

Previous sections presented the temporal variation of average concentrations of contaminants detected in both humidity cell and mass flux leachate samples. In order to better compare the humidity cell testing results to mass flux results, the cumulative amount of mass of detectable contaminants from each test was calculated. This mass was then normalized per kilogram of slate in the test to account for differing slate amounts between slate and slate/soil specimens and mass flux specimens. Several assumptions and simplifications were made when processing and presenting the data. The concentrations

used were an average value obtained from the leachate samples. As humidity cell leachate samples were analyzed intermittently, linear interpolation was used to assign contaminant concentrations during weeks where no chemical analyses were performed. When contaminant concentrations were below the RDL, a value equal to half the typical RDL was used in its place. The data in Figure 11 compares the total mass of contaminants leached from untreated and binder-treated humidity cell specimens using this approach.

Four plots are presented in Figure 11 that each exemplify a different general contaminant release pattern. Aluminum is typical of contaminants that were seen in significantly higher concentrations in untreated versus treated specimens. In general, the mass of a given contaminant released per week decreases over time. Trends in the treated specimens are hard to detect due to the comparatively low concentrations. Boron exhibited cumulative concentrations in treated specimens that only slightly decreased towards the end of the testing period. Sodium and sulfur exhibited higher cumulative masses in treated specimens compared to the untreated slate itself.



**Figure 11 – Cumulative Mass of Contaminants Released for Select Contaminants in Humidity Cell Tests.**

### 2.4.3 Effect of Slate Grain-Size on Leachate Concentrations from Humidity Cell Testing

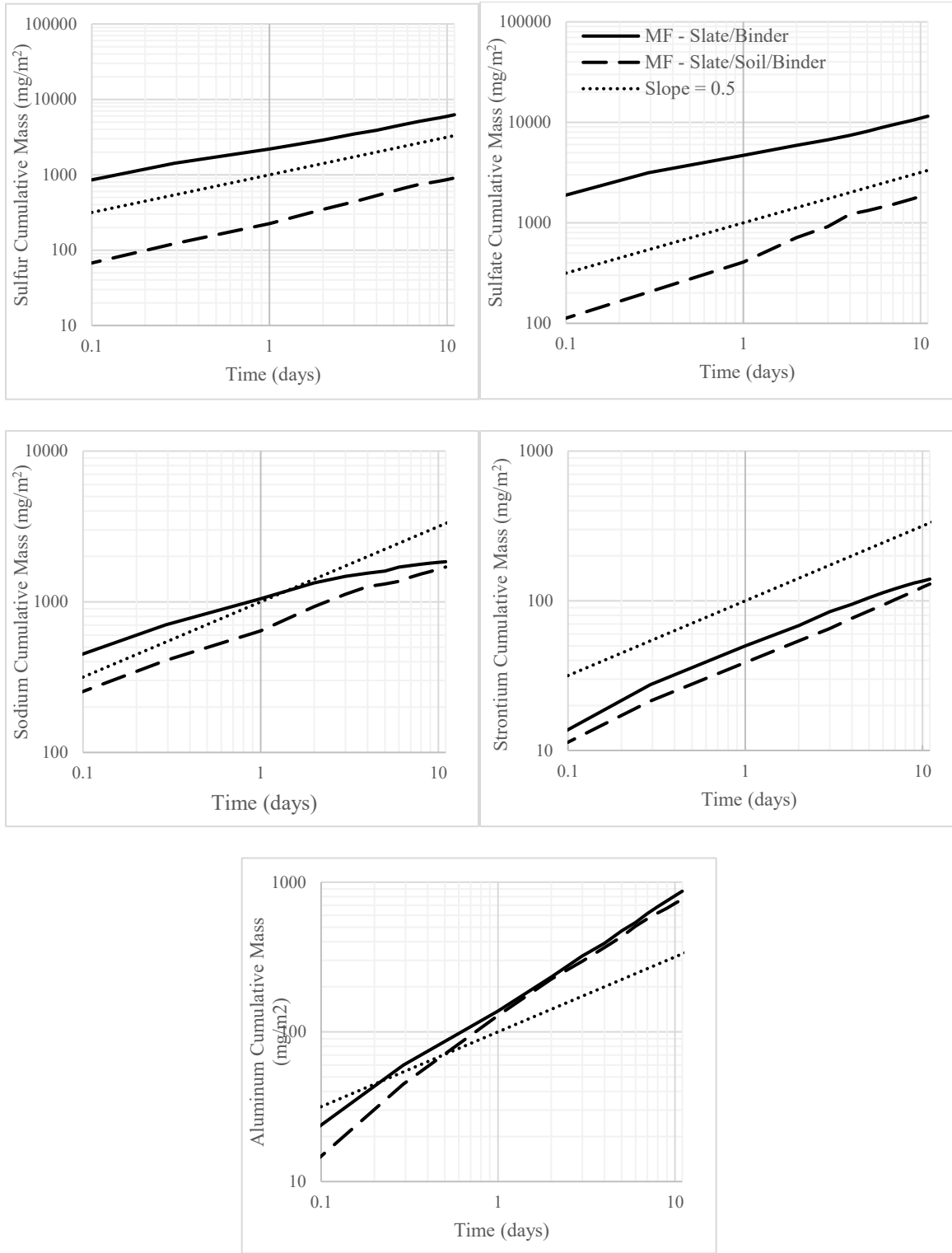
Grain-size distribution of the crushed slate was one of the key variables established in the initial testing plan. Increasing the proportion of fine grain-sized material increases the total amount of slate surface area with which air and water can react. It was expected

that this difference would be especially apparent in untreated slate samples. While pH levels of both untreated and treated specimens did not appear to be influenced by grain-size, the total mass of leached contaminants, in general, was consistently higher in specimens containing more finely graded slate fractions (see Figure 11). These trends are more easily identifiable in graphs depicting the cumulative mass released of a contaminant over the duration of the humidity cell testing procedure. When analyzing the cumulative mass released, small differences in week-to-week concentrations lead to notable differences between fine and coarse grain-size distributions.

#### **2.4.4 Determination of Contaminant Release Mechanism from Mass Flux Testing**

For mass flux testing, the shape of the cumulative mass released over time graph can give information on the release mechanism of contaminants (IRTC, 2011). When presented graphically on a log-log plot, a slope of approximately 0.5 is consistent with a diffusion-controlled release mechanism. Figure 12 shows the cumulative mass release of several contaminants over the test duration. The time presented only includes the duration the specimen was in contact with deionized water; the time spent exposed to air is not included. The release of sulfur, sulfate, and strontium appear to be consistent with a diffusion-controlled release mechanism. Similarly, the graph representing the release of sodium appears to have a slope near 0.5 initially, decreasing slightly as the test progresses which is indicative of the limited amount of sodium being reached. The release of aluminum is represented by a slope of approximately 1.0. This indicates that the release of aluminum is likely not diffusion-controlled, but is instead controlled by some other mechanism, such as solubility (IRTC, 2011). The shape of the cumulative mass released

graph alone is not enough to definitively determine the mechanism by which a given contaminant is released; such determinations are beyond the scope of this thesis.



**Figure 12 – Cumulative Mass of Leached Contaminants Over Time During Mass Flux Testing.**

#### **2.4.5 Sulfur and Sulfate Concentrations Observed in Humidity Cell Testing**

In humidity cell testing, sulfur and sulfate concentrations were notable contaminants that increased with the addition of binder materials. Untreated slate samples showed moderate concentrations at week 0, declining over several weeks to lower, steady state values. Binder-treated specimens showed low initial concentrations that increased rapidly, peaking at weeks 2 and 4 for fine and coarse grain sizes, respectively. Peak sulfur and sulfate concentrations detected in binder-treated specimens were, on average, 3 times higher than peak concentrations detected in the untreated slate specimens, when comparing equivalent grain-sizes.

As previously described, an additional humidity cell specimen consisting of only binder material was created in an attempt to isolate the source of increased sulfur and sulfate concentrations in the binder-treated slate specimens. The masses of cement, fly ash, and water in the test specimen were 50 g, 10 g, and 25 g, respectively. After mixing, the sample was poured into a cylindrical mold and allowed to cure in a temperature and humidity-controlled environment for a minimum of 28 days. After curing, the specimen was removed from the mold, crushed with a hammer (until a maximum grain size of approximately 20 mm was achieved, one fifth of the humidity cell diameter), and prepared in a humidity cell in the same manner as all other humidity cell tests. The humidity cell was run for four weeks and produced three samples (week 0, 2, and 4).

The mass of sulfur-based compounds released from each specimen does not appear to indicate that the addition of binder is the cause of the increase in sulfur/sulfate in the leachate of binder-treated slate specimens. The maximum sulfur and sulfate concentrations throughout the four-week monitoring period were 2.1 and 5 mg/L, respectively. However,

there are several factors that indicate this binder-only humidity cell analysis may not be sufficient to come to this conclusion. It is likely that the increased water content (relative to the total mass of the specimen) and more thorough mixing of the fine-grained binder-only specimen allowed a more completely hydrated and uniform material that would not readily release sulfur compounds into the leachate. Conversely, the aggregated humidity cell specimens contained both fine and coarse material, as well as less water relative to the total sample mass, which made thorough mixing and complete binder hydration more difficult. Incomplete hydration of the binder materials and increased surface area (as the binder material would remain on the surface of any larger slate particles) of the aggregated slate-based specimens may explain the increase in sulfur/sulfate due to cement and fly ash which could not be replicated in the binder-only humidity cell experiment. Further investigation would be required to fully explain, with experimental data, the increase in sulfur-based compounds in binder-treated specimens.

## **Chapter 3 Conclusions and Recommendations**

### **3.1 Conclusions**

The following objectives were outlined in Chapter 1 of this thesis:

- Assess the acid producing potential of a waste rock material obtained from an urban construction site in HRM using common testing techniques used in the mining industry (i.e. humidity cell testing),
- Assess the performance of a cement-based binder at reducing the potential for acid production and metal leaching using humidity cell testing,
- Compare the results of humidity cell testing (common mining industry performance test) to a more conventional mass flux test to assess the performance of the cement-based binder treatment.

To carry out these objectives, crushed slate material was combined with a binder mixture (consisting of cement and fly ash) to create a treated slate specimen material. Further testing incorporated soil into the slate and binder mixture to explore the possible co-disposal of slate and soil materials.

Humidity cell testing was used to initially determine the effectiveness of cement S/S treatment of acid producing slate waste rock. Humidity cells used water and air to accelerate the weathering of aggregated test specimens. Major contaminants and pH were tracked week-to-week to determine chemical release patterns and to compare untreated to binder-treated slate specimens. Mass flux testing, which fully submerges a monolithic test specimen, was used to compare to results of humidity cell testing. In general, the addition of a cement binder greatly reduced the concentration of contaminants in the tested leachate

and increased pH values above neutral. The notable exceptions to this observation were sulfur and sulfate, which were detected in significantly higher concentrations in binder-treated specimens when compared to untreated slate. Based on additional humidity cell testing completed on binder-only specimens, it is assumed this increase is likely due to excess non-hydrated cement remaining after mixing the aggerated specimens. The pH of the leachate did not appear to be impacted by the grain-size of the slate portion of the sample material. However, the cumulative mass released of individual contaminants was generally higher in specimens with finer-grained slate portions. This was true for both untreated and binder-treated specimens.

### **3.2 Recommendations**

Generally speaking, the humidity cell tests produced higher amounts of contaminants in the leachate due to the nature of the test (i.e. non-monolithic samples in humidity cell testing). Given that slate rock produced from urban excavation projects is not dealt with in the same way as mining waste, which is typically more controlled, there is some question as to whether the use of humidity cell testing for S/S treated samples from urban projects is valid. A modified version of the mass flux test performed in this thesis may provide a better prediction of performance than humidity cell testing, which is quite conservative (at least in the short term, i.e. several months) from a leaching performance perspective. Results for both testing methodologies may be compared to real-world leachate samples collected from active containment cells. It is important that the mass flux test be modified to incorporate water and oxygen exposure cycling to induce the pathway for acid water production. Mass flux testing in this thesis will likely be able to identify key contaminants that may be present in the leachate from this type of treatment. Given that

the objective of this work was not to optimize this treatment, the results presented herein should not be assessed in isolation.

Humidity cell testing duration for slate-based specimens lasted several months. In many cases it may be beneficial to extend the testing period to one year or longer. While these tests do accelerate weathering conditions beyond those likely to be encountered in the field, S/S treated waste materials are generally expected to last indefinitely without significant impacts on the surrounding environment. An extended testing period may reveal important long-term changes in the treated material such as pH fluctuations due to a limit in the cement's buffering capacity.

Further investigation into the elevated sulfur/sulfate concentrations in the leachate of binder-treated specimens would be required to make definitive conclusions on the source of the increase. While additional humidity cell testing was performed on a specimen consisting of only cement, fly ash, and water, this did not produce the results necessary to conclude that the increase was due to the addition of the binder materials. The results of the humidity cell and mass flux testing, including the elevated concentrations of sulfur/sulfate, could be compared to local environmental guidelines to further gauge the effectiveness of the cement-based binder treatment.

An uncontaminated silty sand was used to simulate the effect of co-disposing soil and acid rock material. The effect this addition had on leachate characteristics varied depending on the contaminant being examined, but in general did not significantly or unexpectedly change the results. It may be beneficial to use different types of soils, such as soils with increased buffering capacity (not typical of Nova Scotian soils) or those

contaminated with hydrocarbons to obtain a wider understanding of how differing soil characteristics affect the chemical and physical properties of S/S treated materials.

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## Appendix A – Major Elements/Oxides

**Table A1 – Major Elements (Expressed as Oxides) by Li-Borate Fusion, ICP-OES Finish.**

Oxide/Element	Units	Slate	Soil	Cement	Fly Ash
Al <sub>2</sub> O <sub>3</sub>	Wt. %	22.41	10.77	4.56	20.49
BaO	Wt. %	0.09	0.06	0.03	0.16
CaO	Wt. %	0.29	0.52	63.59	2.07
Cr <sub>2</sub> O <sub>3</sub>	Wt. %	0.02	0.02	0.02	0.03
Fe <sub>2</sub> O <sub>3</sub>	Wt. %	6.22	3.65	3.18	10.69
K <sub>2</sub> O	Wt. %	3.47	2.11	0.62	1.57
MgO	Wt. %	2.15	1.24	1.72	1.06
MnO	Wt. %	0.07	0.09	0.04	0.04
Na <sub>2</sub> O	Wt. %	1.07	1.78	0.20	0.78
P <sub>2</sub> O <sub>5</sub>	Wt. %	0.13	0.13	0.05	0.32
SiO <sub>2</sub>	Wt. %	61.07	74.42	19.51	51.41
SO <sub>3</sub>	Wt. %	0.07	<0.01	3.09	1.00
SrO	Wt. %	0.02	0.01	0.06	0.10
TiO <sub>2</sub>	Wt. %	0.93	0.66	0.58	0.95
V <sub>2</sub> O <sub>5</sub>	Wt. %	0.02	0.01	0.02	0.06
ZrO <sub>2</sub>	Wt. %	0.02	0.03	0.02	0.03
LOI (1000°C)	Wt. %	1.85	4.43	2.57	9.13
Oxide Total	Wt. %	99.88	99.93	99.86	99.87

## Appendix B – Humidity Cell Results (AGAT)

**Table B1 – Typical Reported Detection Limit (RDL) for Examined Parameters.**

Parameter	Typical RDL Concentration	Unit
Cyanide	0.002	mg/L
Bromine	0.3	mg/L
Chloride	1	mg/L
Fluoride	0.12	mg/L
Nitrate as N	0.05	mg/L
Nitrite as N	0.05	mg/L
Sulphate	2	mg/L
Aluminum	0.02	mg/L
Antimony	0.02	mg/L
Arsenic	0.02	mg/L
Barium	0.02	mg/L
Beryllium	0.04	mg/L
Bismuth	0.02	mg/L
Boron	0.05	mg/L
Cadmium	0.0001	mg/L
Chromium	0.02	mg/L
Cobalt	0.01	mg/L
Copper	0.01	mg/L
Iron	0.2	mg/L
Lead	0.005	mg/L
Lithium	0.02	mg/L
Manganese	0.02	mg/L
Molybdenum	0.02	mg/L
Nickel	0.02	mg/L
Selenium	0.001	mg/L
Silver	0.001	mg/L
Sodium	1	mg/L
Strontium	0.06	mg/L
Thallium	0.001	mg/L
Tin	0.02	mg/L
Uranium	0.0002	mg/L
Vanadium	0.01	mg/L
Zinc	0.02	mg/L
Sulfur	0.1	mg/L
Mercury	0.026	µg/L

**Table B2 – pH Triplicate Testing Results (AGAT).**

Week	pH					
	S-F			S-C		
	A	B	C	A	B	C
0	3.67	3.69	3.67			
2	3.79	3.79	3.75			
4	3.98	3.82	3.76			
6	3.87	3.77	3.72			
8	3.87	3.75	3.73			
12	3.73	3.62	3.58			
16	3.96	3.64	3.60			
	S-M			S-C		
Week	A	B	C	A	B	C
0	3.87	3.75	3.79	3.85	3.73	3.66
2	4.21	3.94	3.99	3.68	3.59	3.66
4	3.98	3.77	3.73	3.77	3.67	3.75
6	3.99	3.78	3.64	3.81	3.75	3.75
8	3.92	3.76	3.68	3.78	3.81	3.91
12	3.93	3.71	3.68	3.70	3.76	3.75
16	3.91	3.75	3.61	3.58	3.65	3.62
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	11.6	12.1	12.1	12.1	12.1	12.1
2	7.23	7.53	7.63	7.62	7.50	7.46
4	7.76	7.73	7.57	7.43	7.41	7.44
6	7.64	7.72	7.73	7.64	7.66	7.65
8	7.38	7.72	7.70	7.58	7.56	7.61
12	7.69	7.77	7.72	7.58	7.57	7.62
16	8.64	8.20	8.05	7.76	7.74	7.76
20	6.77			6.78		
24	7.36			7.25		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	11.7	11.9	11.8	11.8	11.8	11.8
2	7.39	8.82	9.33	8.50	8.92	9.24
4	7.63	7.51	7.40	7.35	7.15	7.23
6	7.60	7.56	7.40	7.32	7.24	7.35
8	7.63	7.71	7.59	7.31	7.17	7.44
12	7.41	7.54	7.48	7.29	7.34	7.39
16	7.82	7.92	7.88	7.66	7.67	7.75
20	7.51			7.66		

**Table B3 – Aluminum Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.02	ALUMINUM CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	13.3	15.4	21.9			
2	10.7	9.74	10.1			
4	8.67	8.30	8.57			
6	4.80	4.76	4.95			
8	5.63	6.25	5.79			
12	5.26	3.84	5.44			
16	4.24	2.73	4.31			
	S-M			S-C		
Week	A	B	C	A	B	C
0	18.9	18.6	20.5	9.70	8.96	11.3
2	6.35	5.52	6.93	3.79	3.83	3.97
4	4.56	6.84	6.42	3.69	3.50	3.62
6	6.61	5.62	7.56	3.53	2.85	3.58
8	5.62	4.97	6.48	3.09	3.04	3.47
12	4.80	4.67	4.32	3.41	3.08	3.17
16	4.33	3.14	3.30	2.71	2.19	2.11
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.93	0.39	0.52	0.36	0.33	0.42
2	0.72	0.84	0.87	0.75	0.59	0.67
4	0.35	0.14	0.25	0.10	0.15	0.17
6	0.13	0.07	0.08	0.06	0.07	0.06
8	0.16	0.05	0.08	0.07	0.06	0.06
12	0.227	0.084	0.105	0.143	0.137	0.139
16	0.23	0.03	0.04	0.12	0.04	0.05
20	0.04			0.04		
24	0.04			0.08		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.63	0.64	0.49	0.51	0.65	0.46
2	0.10	0.83	1.04	0.25	1.04	0.93
4	0.06	0.15	0.25	0.05	0.16	0.27
6	0.07	0.07	0.08	0.04	0.06	0.09
8	0.06	0.07	0.05	0.07	0.07	0.10
12	0.04	0.04	0.04	0.05	0.04	0.07
16	0.05	0.04	0.04	0.04	0.04	0.07
20	0.044			0.065		

**Table B4 – Boron Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.05	BORON CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A	B	C
0	0.025	0.025	0.025	0.025	0.025	0.025
2	0.025	0.025	0.025	0.025	0.025	0.025
4	0.025	0.025	0.025	0.025	0.025	0.025
6	0.025	0.025	0.025	0.025	0.025	0.025
8	0.025	0.025	0.025	0.025	0.025	0.025
12	0.025	0.025	0.025	0.025	0.025	0.025
16	0.025	0.025	0.025	0.025	0.025	0.025
	S-M			S-C		
Week	A	B	C	A	B	C
0	0.025	0.025	0.025	0.025	0.025	0.025
2	0.025	0.025	0.025	0.025	0.025	0.025
4	0.025	0.025	0.025	0.025	0.025	0.025
6	0.025	0.025	0.025	0.025	0.025	0.025
8	0.025	0.025	0.025	0.025	0.025	0.025
12	0.025	0.025	0.025	0.025	0.025	0.025
16	0.025	0.025	0.025	0.025	0.025	0.025
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.025	0.025	0.025	0.025	0.025	0.025
2	0.15	0.09	0.09	0.07	0.08	0.08
4	0.17	0.17	0.15	0.15	0.13	0.14
6	0.15	0.21	0.22	0.18	0.17	0.18
8	0.11	0.18	0.16	0.16	0.15	0.15
12	0.070	0.138	0.126	0.118	0.128	0.121
16	0.025	0.09	0.09	0.09	0.09	0.09
20	0.05			0.06		
24	0.025			0.05		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.025	0.025	0.025	0.025	0.025	0.025
2	0.11	0.06	0.025	0.025	0.025	0.05
4	0.15	0.12	0.10	0.07	0.07	0.08
6	0.15	0.14	0.13	0.07	0.07	0.08
8	0.13	0.15	0.12	0.06	0.07	0.08
12	0.11	0.12	0.13	0.08	0.08	0.10
16	0.08	0.08	0.09	0.07	0.07	0.07
20	0.110			0.089		

**Table B5 – Cadmium Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.0001	CADMIUM CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	0.0049	0.0066	0.0069			
2	0.0030	0.0033	0.0033			
4	0.0014	0.0016	0.0016			
6	0.0008	0.0009	0.0009			
8	0.0006	0.0009	0.0007			
12	0.0003	0.0004	0.0004			
16	0.0003	0.0003	0.0002			
	S-M			S-C		
Week	A	B	C	A	B	C
0	0.0098	0.0088	0.0079	0.0052	0.0051	0.0054
2	0.0022	0.0022	0.0025	0.0018	0.0014	0.0022
4	0.0015	0.0013	0.0010	0.0009	0.0008	0.0008
6	0.0010	0.0008	0.0010	0.0011	0.0007	0.0009
8	0.0009	0.0007	0.0009	0.0006	0.0006	0.0006
12	0.0006	0.0007	0.0005	0.0007	0.0005	0.0005
16	0.0005	0.0004	0.0004	0.0004	0.0002	0.0003
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.0002	0.00005	0.00005	0.00005	0.00005	0.00005
2	0.00005	0.0002	0.0002	0.0002	0.0002	0.0002
4	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
6	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
8	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
12	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
16	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
20	0.00005			0.00005		
24	0.00005			0.00005		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
2	0.00005	0.0001	0.0001	0.00005	0.00005	0.00005
4	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
6	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
8	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
12	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
16	0.00005	0.00005	0.00005	0.00005	0.00005	0.00005
20	0.000021			0.000029		

**Table B6 – Cobalt Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.01	COBALT CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	0.63	0.71	0.39			
2	0.30	0.31	0.32			
4	0.11	0.12	0.14			
6	0.07	0.08	0.08			
8	0.07	0.08	0.08			
12	0.04	0.04	0.05			
16	0.04	0.04	0.03			
	S-M			S-C		
Week	A	B	C	A	B	C
0	1.22	0.92	0.85	0.52	0.49	0.58
2	0.20	0.17	0.24	0.18	0.17	0.18
4	0.09	0.15	0.14	0.10	0.09	0.07
6	0.09	0.06	0.11	0.11	0.08	0.08
8	0.08	0.06	0.09	0.06	0.05	0.05
12	0.05	0.06	0.06	0.06	0.05	0.05
16	0.06	0.04	0.05	0.05	0.03	0.03
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.005	0.005	0.005	0.005	0.005	0.005
2	0.005	0.005	0.005	0.005	0.005	0.005
4	0.005	0.005	0.005	0.005	0.005	0.005
6	0.005	0.005	0.005	0.005	0.005	0.005
8	0.005	0.005	0.005	0.005	0.005	0.005
12	0.0025	0.0011	0.0013	0.0078	0.0067	0.0091
16	0.005	0.005	0.005	0.005	0.005	0.005
20	0.005			0.005		
24	0.005			0.005		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.005	0.005	0.005	0.005	0.005	0.005
2	0.005	0.005	0.005	0.005	0.005	0.005
4	0.005	0.005	0.005	0.005	0.005	0.005
6	0.005	0.005	0.005	0.005	0.005	0.005
8	0.005	0.005	0.005	0.005	0.005	0.005
12	0.005	0.005	0.005	0.005	0.005	0.005
16	0.005	0.005	0.005	0.005	0.005	0.005
20	0.0005			0.002		

**Table B7 – Copper Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.01	COPPER CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	0.71	0.88	0.46			
2	0.36	0.40	0.42			
4	0.22	0.25	0.27			
6	0.19	0.21	0.20			
8	0.18	0.22	0.20			
12	0.15	0.15	0.17			
16	0.12	0.12	0.11			
	S-M			S-C		
Week	A	B	C	A	B	C
0	2.26	1.78	1.58	0.72	0.76	0.97
2	0.40	0.28	0.24	0.21	0.24	0.21
4	0.25	0.37	0.28	0.16	0.18	0.15
6	0.30	0.19	0.26	0.17	0.18	0.20
8	0.28	0.20	0.24	0.11	0.12	0.13
12	0.15	0.12	0.11	0.11	0.12	0.13
16	0.17	0.11	0.09	0.09	0.09	0.10
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.005	0.005	0.005	0.005	0.005	0.005
2	0.01	0.005	0.005	0.005	0.005	0.005
4	0.005	0.005	0.005	0.005	0.005	0.005
6	0.005	0.005	0.005	0.005	0.005	0.005
8	0.005	0.005	0.005	0.005	0.005	0.005
12	0.002	0.002	0.002	0.005	0.005	0.004
16	0.005	0.005	0.005	0.005	0.005	0.005
20	0.005			0.005		
24	0.005			0.005		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.005	0.005	0.005	0.005	0.005	0.005
2	0.005	0.005	0.005	0.005	0.005	0.005
4	0.005	0.005	0.005	0.005	0.005	0.005
6	0.005	0.005	0.005	0.005	0.005	0.005
8	0.005	0.005	0.005	0.005	0.005	0.005
12	0.005	0.005	0.005	0.005	0.005	0.005
16	0.005	0.005	0.005	0.005	0.005	0.005
20	0.001			0.001		

**Table B8 – Iron Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.2	IRON CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A	B	C
0	54.1	55.3	90.2			
2	16.5	11.7	14.2			
4	10.5	9.0	10.4			
6	8.4	8.2	8.9			
8	9.2	10.0	13.0			
12	10.6	8.7	12.8			
16	8.6	7.5	9.1			
	S-M			S-C		
Week	A	B	C	A	B	C
0	47.0	41.9	52.4	31.6	28.6	35.0
2	11.0	5.8	9.4	14.4	11.0	9.7
4	8.9	13.3	18.3	14.8	11.9	9.4
6	13.8	12.1	14.8	13.5	11.4	10.9
8	12.7	10.8	13.9	6.3	7.9	7.0
12	6.9	6.5	7.3	11.3	10.8	8.6
16	10.5	9.0	10.0	12.9	9.4	8.7
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.1	0.1	0.3	0.1	0.1	0.2
2	0.4	0.3	0.3	0.5	0.6	0.4
4	0.6	0.2	0.4	0.5	0.6	0.6
6	0.4	0.3	0.4	0.6	0.7	0.6
8	0.4	0.3	0.3	0.6	0.6	0.6
12	0.759	0.545	0.618	1.58	1.69	1.39
16	0.5	0.1	0.1	0.5	0.4	0.5
20	0.1			0.3		
24	0.1			0.6		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.2	0.3	0.1	0.1	0.2	0.2
2	0.1	0.1	0.1	0.2	0.1	0.3
4	0.2	0.1	0.1	0.4	0.1	0.4
6	0.2	0.1	0.3	0.4	0.2	0.5
8	0.1	0.2	0.2	0.5	0.2	0.7
12	0.1	0.1	0.1	0.3	0.2	0.5
16	0.1	0.1	0.1	0.3	0.2	0.5
20	0.08			0.32		

**Table B9 – Manganese Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.02	MANGANESE CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A	B	C
0	0.71	0.79	0.46			
2	0.46	0.40	0.44			
4	0.15	0.14	0.15			
6	0.07	0.07	0.07			
8	0.06	0.07	0.06			
12	0.05	0.03	0.05			
16	0.03	0.01	0.03			
	S-M			S-C		
Week	A	B	C	A	B	C
0	1.24	0.99	1.01	0.45	0.46	0.50
2	0.15	0.13	0.22	0.11	0.11	0.14
4	0.09	0.09	0.10	0.05	0.06	0.06
6	0.08	0.09	0.09	0.06	0.06	0.06
8	0.07	0.07	0.09	0.04	0.04	0.04
12	0.04	0.04	0.04	0.04	0.04	0.04
16	0.03	0.02	0.03	0.03	0.02	0.02
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.01	0.01	0.01	0.01	0.01	0.01
2	0.01	0.01	0.01	0.01	0.01	0.01
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.01	0.01	0.01	0.01
12	0.005	0.001	0.003	0.007	0.006	0.006
16	0.01	0.01	0.01	0.01	0.01	0.01
20	0.01			0.01		
24	0.01			0.01		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.01	0.01	0.01	0.01	0.01	0.01
2	0.01	0.01	0.01	0.01	0.01	0.01
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.01	0.01	0.01	0.01
12	0.01	0.01	0.01	0.01	0.01	0.01
16	0.01	0.01	0.01	0.01	0.01	0.01
20	0.0015			0.0015		

**Table B10 – Nickel Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.02	NICKEL CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A	B	C
0	1.57	1.72	0.95			
2	0.74	0.72	0.79			
4	0.27	0.29	0.35			
6	0.18	0.19	0.20			
8	0.20	0.21	0.21			
12	0.12	0.11	0.14			
16	0.10	0.10	0.10			
	S-M			S-C		
Week	A	B	C	A	B	C
0	2.70	2.07	2.05	1.31	1.21	1.36
2	0.54	0.41	0.62	0.44	0.38	0.44
4	0.27	0.39	0.40	0.27	0.21	0.26
6	0.26	0.16	0.33	0.30	0.21	0.21
8	0.23	0.17	0.27	0.16	0.13	0.13
12	0.17	0.16	0.18	0.19	0.15	0.14
16	0.18	0.12	0.15	0.14	0.09	0.08
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.01	0.01	0.01	0.01	0.01	0.01
2	0.01	0.01	0.01	0.01	0.01	0.01
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.01	0.01	0.01	0.01
12	0.009	0.006	0.004	0.030	0.027	0.040
16	0.01	0.01	0.01	0.01	0.01	0.01
20	0.01			0.01		
24	0.01			0.01		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.01	0.01	0.01	0.01	0.01	0.01
2	0.03	0.01	0.03	0.01	0.01	0.06
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.07	0.01	0.01	0.01
12	0.01	0.01	0.01	0.01	0.01	0.01
16	0.01	0.01	0.01	0.01	0.01	0.01
20	0.004			0.006		

**Table B11 – Selenium Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.001	SELENIUM CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	0.004	0.005	0.005			
2	0.002	0.002	0.002			
4	0.002	0.002	0.002			
6	0.0005	0.0005	0.001			
8	0.001	0.001	0.001			
12	0.0005	0.0005	0.0005			
16	0.0005	0.0005	0.0005			
	S-M			S-C		
Week	A	B	C	A	B	C
0	0.005	0.005	0.005	0.003	0.003	0.003
2	0.001	0.001	0.002	0.001	0.001	0.001
4	0.001	0.001	0.001	0.0005	0.0005	0.0005
6	0.001	0.001	0.001	0.0005	0.0005	0.0005
8	0.0005	0.0005	0.0005	0.0005	0.0005	0.0005
12	0.0005	0.0005	0.0005	0.0005	0.0005	0.0005
16	0.0005	0.0005	0.0005	0.0005	0.0005	0.0005
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.005	0.003	0.003	0.003	0.003	0.003
2	0.011	0.010	0.010	0.008	0.008	0.007
4	0.006	0.007	0.007	0.006	0.006	0.006
6	0.005	0.004	0.004	0.004	0.004	0.004
8	0.005	0.003	0.003	0.004	0.004	0.003
12	0.003	0.002	0.004	0.001	0.001	0.002
16	0.004	0.002	0.002	0.002	0.002	0.002
20	0.002			0.002		
24	0.003			0.002		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.002	0.003	0.002	0.002	0.002	0.002
2	0.008	0.009	0.008	0.005	0.005	0.006
4	0.004	0.007	0.007	0.005	0.006	0.006
6	0.002	0.003	0.003	0.002	0.003	0.003
8	0.002	0.003	0.003	0.002	0.002	0.003
12	0.002	0.002	0.002	0.002	0.001	0.001
16	0.002	0.002	0.002	0.001	0.001	0.001
20	0.001			0.001		

**Table B12 – Sodium Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.1	SODIUM CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	5.0	5.5	5.6			
2	1.5	1.4	1.6			
4	0.5	0.5	0.6			
6	0.2	0.2	0.2			
8	0.2	0.2	0.2			
12	0.2	0.1	0.2			
16	0.1	0.05	0.1			
	S-M			S-C		
Week	A	B	C	A	B	C
0	8.4	6.9	7.4	4.2	4.2	4.3
2	1.3	1.2	1.7	1.1	1.1	1.5
4	0.5	0.5	0.4	0.4	0.4	0.3
6	0.3	0.3	0.3	0.3	0.3	0.2
8	0.2	0.2	0.2	0.2	0.2	0.2
12	0.1	0.05	0.05	0.1	0.05	0.05
16	0.05	0.05	0.05	0.3	0.1	0.05
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	43.8	45.0	44.7	31.5	30.6	30.0
2	9.8	7.2	7.8	7.0	7.5	7.6
4	3.7	5.5	5.7	7.6	4.7	4.5
6	2.1	3.2	3.3	3.5	4.0	3.9
8	1.6	2.4	2.1	2.7	3.0	2.9
12	Concentrations not reported by laboratory.					
16	0.4	0.9	0.9	0.9	0.9	0.9
20	0.6			0.6		
24	0.4			0.4		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	41	42	39	26	25	29
2	6.4	5.5	4.9	5.8	5.5	4.8
4	4.8	5.4	4.7	4.6	4.5	4.4
6	3.3	3.3	3.4	3.0	3.4	3.4
8	3.1	3.2	3.6	3.1	3.5	4.2
12	1.3	1.3	1.5	1.8	1.8	1.9
16	0.8	0.8	1.0	1.1	1.2	1.1
20	2.2			1.6		

**Table B13 – Strontium Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.02	STRONTIUM CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	0.10	0.12	0.13			
2	0.04	0.04	0.04			
4	0.01	0.01	0.01			
6	0.01	0.01	0.01			
8	0.01	0.01	0.01			
12	0.01	0.01	0.01			
16	0.01	0.01	0.01			
	S-M			S-C		
Week	A	B	C	A	B	C
0	0.18	0.15	0.16	0.07	0.07	0.07
2	0.03	0.02	0.03	0.02	0.02	0.02
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.01	0.01	0.01	0.01
12	0.01	0.01	0.01	0.01	0.01	0.01
16	0.01	0.01	0.01	0.01	0.01	0.01
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.91	2.67	2.57	2.38	2.30	2.36
2	0.35	0.27	0.27	0.18	0.19	0.20
4	0.17	0.21	0.22	0.21	0.19	0.18
6	0.12	0.12	0.14	0.12	0.14	0.13
8	0.09	0.07	0.07	0.09	0.09	0.09
12	0.058	0.056	0.057	0.073	0.064	0.065
16	0.05	0.04	0.04	0.06	0.04	0.04
20	0.04			0.04		
24	0.04			0.04		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	3.10	3.89	3.17	1.73	1.91	2.30
2	0.19	0.19	0.16	0.10	0.11	0.09
4	0.10	0.18	0.16	0.11	0.12	0.12
6	0.05	0.06	0.07	0.06	0.07	0.07
8	0.05	0.04	0.05	0.05	0.05	0.06
12	0.03	0.02	0.03	0.04	0.03	0.03
16	0.02	0.02	0.02	0.03	0.02	0.03
20	0.033			0.028		

**Table B14 – Zinc Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.005	ZINC CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A	B	C
0	0.87	1.04	0.54			
2	0.55	0.56	0.54			
4	0.23	0.23	0.25			
6	0.16	0.16	0.16			
8	0.14	0.18	0.15			
12	0.09	0.08	0.10			
16	0.07	0.05	0.05			
	S-M			S-C		
Week	A	B	C	A	B	C
0	1.85	1.97	1.36	0.68	0.62	0.70
2	0.33	0.35	0.48	0.23	0.18	0.32
4	0.25	0.24	0.23	0.13	0.11	0.10
6	0.19	0.17	0.22	0.16	0.12	0.14
8	0.18	0.16	0.21	0.11	0.09	0.10
12	0.12	0.15	0.16	0.13	0.09	0.11
16	0.14	0.12	0.12	0.12	0.05	0.05
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	0.01	0.01	0.01	0.01	0.01	0.01
2	0.01	0.01	0.01	0.01	0.01	0.01
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.01	0.01	0.01	0.01
12	0.048	0.01	0.01	0.01	0.01	0.01
16	0.01	0.01	0.01	0.01	0.01	0.01
20	0.01			0.01		
24	0.01			0.01		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	0.01	0.01	0.01	0.01	0.01	0.01
2	0.01	0.01	0.01	0.01	0.01	0.01
4	0.01	0.01	0.01	0.01	0.01	0.01
6	0.01	0.01	0.01	0.01	0.01	0.01
8	0.01	0.01	0.01	0.01	0.01	0.01
12	0.01	0.01	0.01	0.01	0.01	0.01
16	0.01	0.01	0.01	0.01	0.01	0.01
20	0.006			0.007		

**Table B15 – Sulfur Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 0.1	SULFUR CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	88.7	105	121			
2	30.8	30.4	32.4			
4	22.5	22.9	27.7			
6	16.7	17.1	19.8			
8	12.8	13.8	14.7			
12	10.3	10.0	12.5			
16	11.6	9.8	10.6			
	S-M			S-C		
Week	A	B	C	A	B	C
0	83.1	66.2	72.8	41.3	43.4	47.2
2	26.6	22.7	28.5	21.6	18.7	19.4
4	22.9	18.9	19.5	20.2	17.2	13.3
6	13.9	20.8	17.2	14.4	13.5	13.9
8	18.5	16.0	20.6	15.4	14.8	13.5
12	13.2	14.9	14.0	17.9	17.7	13.8
16	15.7	13.5	13.7	12.5	8.6	9.0
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	59.8	21.8	22.1	19.4	18.3	19.3
2	281	294	268	162	164	167
4	98.6	176	198	175	169	155
6	59.8	83.5	103	82.8	97.1	91.7
8	37.3	46.0	46.8	61.8	56.5	51.7
12	29	33	38	45	41	39
16	17.1	11.9	11.1	18.7	14.6	15.0
20	12.0			16.2		
24	15.8			19.8		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	4.7	3.9	3.7	4.8	3.1	3.4
2	185	141	99	53.2	52.2	54.5
4	79.4	160	165	71.0	75.0	85.6
6	33.5	39.6	54.6	38.3	43.3	43.9
8	26.4	23.9	32.9	29.6	26.8	32.6
12	11.7	11.6	13.8	24.1	20.2	21.6
16	8.8	6.7	7.3	12.9	10.6	11.1
20	9.2			9.3		

**Table B16 – Sulfate Concentration (mg/L) Triplicate Testing Results (AGAT).**

RDL = 2	SULFATE CONCENTRATION (mg/L)					
	S-F			S-C		
Week	A	B	C	A, B, and C represent different triplicate samples of the same specimen composition. Triplicate samples were submitted for the first 16 weeks, after which only single samples were submitted for testing. Where reported concentrations are below the RDL, half the RDL value is used in its place.		
0	248	261	338			
2	106	116	124			
4	60	67	78			
6	50	53	62			
8	47	55	33			
12	40	41	50			
16	37	34	37			
	S-M			S-C		
Week	A	B	C	A	B	C
0	371	308	329	184	179	184
2	78	71	86	73	62	66
4	70	59	63	49	44	34
6	37	27	46	54	45	47
8	49	42	58	41	39	39
12	48	55	53	48	44	40
16	50	41	45	51	36	35
	SB-F			SB-C		
Week	A	B	C	A	B	C
0	109	48	50	69	68	70
2	865	822	759	504	399	482
4	316	616	659	552	516	482
6	155	240	267	239	265	248
8	153	192	207	245	227	210
12	70	79	89	124	113	104
16	71	51	48	94	70	70
20	33			48		
24	38			55		
	SSB-F			SSB-C		
Week	A	B	C	A	B	C
0	9	5	6	13	7	9
2	556	387	278	140	131	141
4	170	361	385	161	175	211
6	124	163	214	140	165	192
8	94	88	121	118	103	131
12	28	29	34	65	53	59
16	29	22	23	46	38	38
20	2			3		

## Appendix C – Analytical Testing Methodologies

**Table C1 – Methodology Summary for Analytical Testing of Leachate (AGAT).**

Testing Parameter	Literature Reference	Analytical Technique
Bromide	SM 4110 B	Ion Chromatograph
Chloride	SM 4110 B	Ion Chromatograph
Fluoride	SM 4110 B	Ion Chromatograph
Nitrate as N	SM 4110 B	Ion Chromatograph
Nitrite as N	SM 4110 B	Ion Chromatograph
Sulfate	SM 4110 B	Ion Chromatograph
pH	SM 4110 H+B	PC Titrate
Total Mercury	SM 3112 B	CV/AA
Total Sodium	Modified from SM 3125/SM 3030 B/SM 3030D	ICP-MS
Total Sulfur	SM 3120B	ICP/OES
Total Aluminum	SM 3030 E; SM 3125 B	ICP-MS
Total Antimony	SM 3030 E; SM 3125 B	ICP-MS
Total Arsenic	SM 3030 E; SM 3125 B	ICP-MS
Total Barium	SM 3030 E; SM 3125 B	ICP-MS
Total Beryllium	SM 3030 E; SM 3125 B	ICP-MS
Total Bismuth	SM 3030 E; SM 3125 B	ICP-MS
Total Boron	SM 3030 E; SM 3125 B	ICP-MS
Total Cadmium	SM 3030 E; SM 3125 B	ICP-MS
Total Chromium	SM 3030 E; SM 3125 B	ICP-MS
Total Cobalt	SM 3030 E; SM 3125 B	ICP-MS
Total Copper	SM 3030 E; SM 3125 B	ICP-MS
Total Iron	SM 3030 E; SM 3120 B	ICP/OES
Total Lead	SM 3030 E; SM 3125 B	ICP-MS
Total Manganese	SM 3030 E; SM 3120 B	ICP/OES
Total Molybdenum	SM 3030 E; SM 3125 B	ICP-MS
Total Nickel	SM 3030 E; SM 3125 B	ICP-MS
Total Selenium	SM 3030 E; SM 3125 B	ICP-MS
Total Silver	SM 3030 E; SM 3125 B	ICP-MS
Total Strontium	SM 3030 E; SM 3120 B	ICP/OES
Total Thallium	SM 3030 E; SM 3125 B	ICP-MS
Total Tin	SM 3030 E; SM 3125 B	ICP-MS
Total Titanium	SM 3030 E; SM 3125 B	ICP-MS
Total Uranium	SM 3030 E; SM 3125 B	ICP-MS
Total Vanadium	SM 3030 E; SM 3125 B	ICP-MS
Total Zinc	SM 3030 E; SM 3125 B	ICP-MS
Sulfide	SM 4500-S2 D	Spectrophotometer
Cyanide, SAD	Modified from ON MOECC E3015, SM-4500-CN-A,B,C	Technicon Auto Analyzer