
**ONONDAGA LAKE PRE-DESIGN INVESTIGATION:
PHASE IV: ADDENDUM 7
CAP pH AMENDMENT EVALUATION**

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TABLE OF CONTENTS

	<u>Page</u>
1.0 INTRODUCTION.....	1
2.0 PROJECT OBJECTIVES	1
3.0 MOBILIZATION AND LOGISTICS.....	2
3.1 HEALTH AND SAFETY.....	2
3.2 SITE FACILITIES.....	2
3.3 DECONTAMINATION AND WASTE HANDLING	2
4.0 FIELD SAMPLING LOGISTICS AND HANDLING	2
5.0 BATCH TEST EXPERIMENTAL DESIGN	3
5.1 MATERIALS	4
5.2 SYNTHETIC POREWATER GENERATION	6
5.3 METHODS	6
5.4 ANALYSIS.....	7
5.4.1 pH and Electrical Conductivity	7
5.4.2 Porewater	7
5.4.3 Sediment	8
5.4.4 Amendments.....	8
6.0 ANC AND BNC TITRATIONS.....	8
7.0 QUALITY ASSURANCE/QUALITY CONTROL	9
8.0 GEOCHEMICAL MODELING.....	9
8.1 BATCH MODELING	9
8.2 CAP SIMULATIONS	9
9.0 REPORTING	10
10.0 REFERENCES.....	11

**PHASE IV PRE-DESIGN INVESTIGATION WORK PLAN
CAP pH AMENDMENT EVALUATION
ADDENDUM 7**

1.0 INTRODUCTION

The Onondaga Lake remedial design relies on biodegradation of organic contaminants occurring within a sediment cap; however, laboratory experiments conducted during Phase II of the Pre-Design Investigation (PDI) at the University of Texas, using sediment slurries from sediment management unit (SMU) 1, indicate there is a significant hindrance to microbial activity and biodegradation rates likely influenced by elevated pH.

Geochemical investigations undertaken by S.S. Papadopoulos & Associates (SSP&A), in collaboration with Portland State University (PSU), determined that the cause of hyperalkaline porewater (pH > 11) is the dissolution of cement-like calcium silicate minerals in the underlying sediment. These studies additionally demonstrated that chemical amendments consisting of sparingly soluble compounds of hydrolyzable metals (e.g. iron, aluminum) could potentially be added to a sediment cap to react with porewater and thereby neutralize pH. The addition of a solid phase amendment to a sand cap has design implications dictated by the rate of pH neutralization (how much to be added, and over what cap thickness) and capacity (i.e. how long will it need to last). The experiments in this Work Plan are designed to evaluate the long-term effectiveness of potential pH amendments.

The sample locations, data-gathering methods, and details of the analyses and testing to be performed at each location are described in this document. The core samples will be collected in accordance with the procedures outlined in the Phase I - IV PDI Work Plans and associated appendices (Parsons, 2005, 2006, 2007, and 2008).

2.0 PROJECT OBJECTIVES

This Work Plan provides the framework for evaluating both the performance and life cycle of potential cap amendments to reduce high pH in porewater passing through the isolation cap. Specific objectives include: 1) demonstrating that amendments are capable of buffering pH; 2) evaluating the life cycle of pH buffering amendments; and 3) determining the relationship between performance, life cycle, and the amount of amendment needed. Specific tasks that will be performed to meet these objectives include: 1) measuring amendment reaction rates; 2) determining the acid neutralizing capacity of the lake porewater; 3) determine the base neutralizing capacity of each amendment; and 4) using this data to calibrate a kinetic geochemical model that can be used to assess long-term performance of different design options (amendment type, application rate, and layer thickness) under field conditions.

This information will be combined with the existing data set for use during design.

3.0 MOBILIZATION AND LOGISTICS

This section covers the mobilization and logistics to support the field tasks outlined in this addendum.

3.1 Health and Safety

Parsons ranks health and safety as the highest priority. Parsons' Project Safety Plan (PSP) and our Subcontractor's Safety Plans (SSP) prepared for previous PDI activities will be used for this investigation and will be strictly followed by all personnel. Any task outside of the current scope defined in the PSP will have a new Job Safety Analysis (JSA) completed before the task begins. Copies of the PSP and SSPs will be maintained at the support zone and on each vessel.

3.2 Site Facilities

The support zone and facilities utilized for the previous phases of the PDI work will be relocated to the clearing off the upper road to Harbor Brook. The dock will be located west of the causeway, closer to the entry gate to the Onondaga Lake site. The support zone will be relocated due to field work for the installation of the Willis portion of the Willis/Semet IRM barrier wall. All decontamination and waste management activities will be conducted in accordance with the Phase I PDI Work Plan (Parsons, 2005)

3.3 Decontamination and Waste Handling

Decontamination will be conducted at the decontamination area established during Phase I and on the various barges and vessels. The decontamination and waste disposal procedures will be conducted in accordance with the Phase I PDI SAP (Parsons, 2005).

4.0 FIELD SAMPLING LOGISTICS AND HANDLING

Samples will be collected from a barge or a pontoon boat using a vibracore or push core in accordance with the procedures outlined in the Phase I PDI SAP (Parsons, 2005). Cores will be located in areas that are representative of the sediment that will be capped. Suitable sampling locations include the two sediment coring locations that exhibited high pH values during the initial materials characterization from samples collected as part of the Phase III PDI Addendum 3, Cores 10118 and 10119.

Initially, two, 3-inch diameter 10-ft long cores will be collected at each location. Three 14-inch sections from each core will be selected for shipment to the laboratory for testing. The selected segment from each core will be:

- representative of materials within the core;
- biased toward the top of the core, but excluding the top 3 to 6 inch nepheloid layer, which may be aerobic and not representative;

- exclude non ILWD; and
- exclude “crusty” layers that will be hard to handle in the laboratory.

Core segments will be capped and sealed immediately after collection and an effort will be made to minimize exposure to ambient air. A written description of each core will also be documented in the field. Photographs will be collected in the field of each core prior to shipment. Prior to shipment, the cores will be individually placed in double Ziploc bags which will be purged with air free nitrogen gas before sealing. The cores will be shipped vertically and will be maintained at 4°C and sealed with no headspace until tests are conducted.

Porewater will also be collected from an existing groundwater upwelling pump in SMU 1. Location TR03-A will be purged and then sampled over a two-day time period to obtain 5 to 6 gallons of porewater. Location TR03-A was selected as the most viable location given its proximity to the shoreline, the integrity of apparatus, and the ability to locate the apparatus tubing for sampling.

Once received by SSP&A personnel at the Geomicrobiology/Geochemistry Laboratory at Portland State University, sediments will be removed from their shipping materials and placed in a sealable jar or drum and stored in a refrigerator at 4°C until needed. One 14-inch section from each location will be set aside and used to generate porewater by Test America in accordance with standard porewater generating procedures used during the Phase IV PDI. This data will be used to verify test results as discussed in Section 5.2.

5.0 BATCH TEST EXPERIMENTAL DESIGN

A series of batch tests will be conducted to measure the rates at which chemical amendments neutralize hyperalkaline porewater. Porewater from SMU-1 will be obtained directly from existing piezometer TR03A. If this porewater is not representative of the larger ILWD alternate methods such as centrifugation, it will be considered for porewater generation as described below. Due to the large volume of sediment that would need to be processed to generate sufficient porewater volume for all batch tests, initial screening batch tests may be performed with synthetic porewater as described below and actual ILWD porewater generated from centrifugation would be used as a check on synthetic porewater solution makeup. Following these screening tests, batch test results for the best performing amendment will be confirmed using actual porewater.

Batch tests will be conducted in 500 mL HDPE jars, in which a pre-determined mass of amendment will be reacted with a measured volume of porewater solution. Batch tests will be carried out at initial liquid to solid amendment (L/S) ratios of approximately 2.5, 12.5, and 50 (40, 8 and 2 grams dry weight amendment per 100 mL solution). This range covers a potential range of amendment application rates of approximately 0.5% to 10% by dry weight of the cap material (assumed quartz sand with 40% porosity). These amendment application rates should

be sufficient to provide complete neutralization (defined as a final pH of 8 or less) of the hyperalkaline solution.

Prior to amendment application, the pH of the synthetic porewater will be adjusted to a value of 12 using NaOH, which is reflective of the highest pH values measured in Onondaga Lake porewater. Monitoring of each amendment/porewater batch test solution will occur over a period of approximately two months. Preliminary evaluations based on published dissolution rates (e.g. Pokrovsky and Schott, 2002) for candidate amendment materials indicate that much of the porewater pH neutralization in the batch tests will occur within the first week to month of reaction, and should be near completion by the end of the second month. Monitoring will continue on a monthly basis after the first two months if changes in pH continue to be observed. Monitoring will consist of periodic measurements of temperature, pH, and electrical conductivity. In addition, any visual changes in the appearance of each batch test jar (such as changes in solution color, formation of secondary precipitates), will be reported. At the end of the experiments, the final solution compositions will be measured for dissolved ions, and the solid phases collected and characterized for the occurrence of secondary minerals formed during the experiments.

All of the batch tests will be performed in a glove box under nitrogen atmosphere in order to simulate the oxygen-free porewater conditions at Onondaga Lake. Batch tests will be performed in duplicate for each amendment and each L/S ratio, and will also include duplicate control samples (synthetic porewater without amendments).

5.1 Materials

The following six amendments will be screened for this study:

- Ferrous sulfate;
- Siderite;
- Alum;
- Aluminum phosphate;
- Iron phosphate; and
- Peat.

As discussed in Section 1.0 and SSP&A (2008), the primary selection criteria for chemical amendments for batch testing included that they contain hydrolyzable aluminum or iron (which form insoluble hydroxides) and that they be sparingly to moderately soluble to provide long-term pH buffering *in situ*. A summary of amendment chemical and physical properties is provided below.

Amendment	Formula	Density ¹ (g/cm ³)	Solubility Product ² (log K _{sp})
Ferrous Sulfate	FeSO ₄ ·7H ₂ O	1.9	-2.35
Siderite	FeCO ₃	3.8	-10.52
Aluminum Sulfate	Al ₂ (SO ₄) ₃	2.71	1.685
Aluminum Phosphate	AlPO ₄	2.57	-19.53
Iron Phosphate	FePO ₄ ·2H ₂ O	2.74	-16.71

¹ from CRC Handbook of Chemistry and Physics (54th ed)

² calculated from Lawrence Livermore National Laboratory thermodynamic database file (*lml.dat*) supplied with PHREEQC. Values are cast in terms of the activity product of the dissolved ions in equilibrium with the solid (e.g. for siderite, [Fe⁺⁺][CO₃⁻] = 10^{-10.52})

Peat is being evaluated in parallel studies for its ability to retard the breakthrough of organic chemicals through the cap. It was also included in the batch testing to evaluate the potential effectiveness of acidic functional groups in peat for pH neutralization.

It is anticipated that for one or more of these amendments that are successful with respect to pH neutralization, the batch screening test results may identify certain undesirable side effects (e.g. elevated final dissolved concentrations of amendment constituents such as iron, sulfate and/or phosphate) that could be addressed through modification of the amendment form or addition of chemicals to counteract such effects. In such a case, the screening tests will be repeated for the modified amendment.

The technical grade chemical amendments used for this investigation will be obtained either from suppliers identified by SSP&A as part of previous investigations, or in the case of peat, from a suitable local source.

Candidate amendment materials will be used in the condition they are received unless their condition is not representative of how they would be added to the cap. For example, clumpy material will likely be disaggregated to produce a desired grain size similar to that of the sand cap.

5.2 Synthetic Porewater Generation

Actual SMU1 porewater for batch tests is preferred and will be used if a sufficient representative volume can be generated. If this is not possible, part of the testing may be carried out with synthetic porewater. Synthetic porewater will be generated by one of two possible procedures: 1) reacting DI water with ILWD material (and supplementing the resultant solution with background electrolyte to mimic the brine component); or 2) mixing stock solutions of various electrolytes to achieve a major salt composition similar to SMU 1 porewater.

The first method outlined above involves equilibrating homogenized sediment with DI water under a fume hood to achieve a liquid to solid (L/S) ratio of approximately 10 for at least 24 hours. In order to determine if the solution has also achieved equilibrium with other dissolved components, electrical conductivity and temperature will be measured twice daily for a maximum period of three days (samples may be agitated to shorten the equilibration time). Because it is anticipated that the brine component of Onondaga Lake porewater will not be simulated by this method, reagent grade solutions of background electrolyte will be added to account for this salt deficiency. Also, in the event that the resulting pH is less than 12, sodium hydroxide will be added until this value is achieved. For the initial comparison to Onondaga Lake porewater, approximately 100 mL of the synthetic porewater will be collected, filtered, and analyzed for major ions (sodium, calcium, magnesium, potassium, chloride, sulfate, nitrate, and alkalinity).

In the second porewater generation method, reagent grade electrolyte solutions will be mixed in a laboratory beaker to achieve a composition similar to reported values at Onondaga Lake. As with the first method described above, pH will be adjusted to a value of 12, and an aliquot of the final solution collected for chemical analysis as described above.

Final selection of the porewater (actual or synthetic) and, if applicable, generation method to be used for batch tests will be made in consultation with NYSDEC. Characterization of the porewater obtained from TR03-A will be provided. Selection will be based on which sample is more representative of ILWD porewater chemistry. Final analysis of the porewater used in the experiments will include major ions (as before), as well as, iron, phosphate, and aluminum (which are found in the chemical amendments and will be used to establish baseline conditions). Excess porewater, not initially required for the batch tests, will be placed in chemically inert containers with water-tight seals and stored in a refrigerator at 4°C.

After the initial screening batch test, the amendment that most effectively neutralizes the pH of the synthetic porewater will be tested with actual lake porewater to confirm the results.

5.3 Methods

Batch testing will provide the basic data needed to evaluate amendment performance and select an amendment for potential additional laboratory column tests. Batch testing will be conducted at three different amendment dosing rates: approximately 20, 80, and 400 grams per

liter of water (scaled to represent a potential range application of approximately 0.5 – 10 % by mass of amendment in the cap). All batch test preparations and monitoring will be carried out in a glove box under an oxygen-free, nitrogen atmosphere.

For each batch test, 200 mL of porewater solution will first be added to a 500 mL reaction bottle, degassed, and its temperature, pH, and conductivity recorded. Appropriate quantities of amendments will then be placed on plastic trays, weighed, and added to the beakers. Monitoring will consist of measuring and recording temperature, pH, and conductivity at 0, 1, 2, 4, 7, 14, 21, 28, 35, 42, 49, 56, and 63 days of reaction (or until pH drops below 8, whichever is shorter). As discussed in Section 5.0, observed physical changes in the sample and porewater solution will also be recorded at this time.

At the end of the batch tests, selected bottles will be sampled and analyzed for major and selected minor constituents (sodium, calcium, magnesium, potassium, chloride, sulfate, nitrate, alkalinity, iron, aluminum, and phosphate). In addition, the solid amendment and any secondary precipitates will be removed and characterized (as described in Section 5.4).

Batch testing will be conducted at Portland State University (PSU), under the direction of SSP&A, and all generated wastes will be disposed of following standard procedures of the PSU Geomicrobiology/Geochemistry Laboratory.

5.4 Analysis

5.4.1 pH and Electrical Conductivity

Monitoring will be performed directly in the reaction bottles using calibrated pH, conductivity, and temperature probes. Between sample measurements, the electrodes will be rinsed with distilled water and wiped dry. As discussed above, measurements will be made at 0, 1, 2, 4, 7, 14, 21, 28, 35, 42, 49, 56, and 63 days. Care will be taken to avoid agitating the beakers so that quiescent porewater-amendment interaction conditions are best simulated.

5.4.2 Porewater

Prior to batch testing, porewater will be sampled and analyzed for major ions, aluminum, iron, and phosphorus. Sampling will consist of the following: 1) removing 100 mL of the aqueous phase from the carboy using a plastic syringe; 2) attaching a 0.45 micrometer (μm) syringe filter to the syringe and pushing the contents through the filter into two clean polyethylene sample bottles; 3) recording any deviations; and 4) preserving one of the samples (for metals analysis) with nitric acid to pH <2. Analysis of the extract will include dissolved calcium, magnesium, sodium, potassium, chloride, sulfate, nitrate, alkalinity, iron, phosphate, aluminum, and total organic carbon. If analysis is not to be conducted immediately, the samples will be stored in a refrigerator at 4°C.

After batch testing is completed, the sampling and analysis procedure described above will be repeated for selected bottles.

5.4.3 Sediment

If synthetic porewater generated from sediment cores is to be generated, the mineralogy of the sediment will be compared to cores previously characterized by SSP&A by collecting two samples from random locations within the homogenized sediment and testing for mineralogy using standard XRD procedures. In the event that synthetic porewater for the batch tests is not generated from sediment, this analysis will not be performed.

5.4.4 Amendments

Prior to batch testing, samples of each chemical amendment will be collected and analyzed for grain size, bulk density, moisture content, and surface area. This information is important for calibration of the geochemical model that will be used to simulate long-term amendment performance. In preparation for physical characterization of the amendments, each sample will be oven dried at 60°C for a period of one week. Standard laboratory operating procedures will be followed for all analyses.

After batch testing is completed, samples of the solid materials remaining in each beaker will be collected for identification of mineral phases. It is anticipated that this will be predominantly composed of the amendments, themselves; however, secondary precipitates, which are important to interpreting batch test results (and understanding long-term effectiveness of the cap), may also be present. Solid materials will be collected from the selected reaction bottles, oven dried at 60°C for a period of one week and subsequently powdered in an agate or ceramic mortar and pestle (cleaned with acetone) and analyzed by X-Ray Diffraction (XRD). If surplus amendment material is available, it will be stored in sealed containers archived in a refrigerator at 4°C. These archived samples may be characterized by SEM/EDS at a later date.

6.0 ANC AND BNC TITRATIONS

Titration curves will be developed for acid neutralizing capacity (ANC) of the ILWD porewater and for purposes of characterizing base neutralizing capacity (BNC) requirements of amendments and for use in validating geochemical models of the pH neutralization process as discussed in Section 8 below.

ANC will be measured on four aliquots of porewater covering a range of porewater pH between 10 and 12 as observed in the ILWD porewater.

The proposed chemical amendments are selected for their expected slow reactivity (to ensure a reasonably long operating lifetime within the cap); therefore, measuring BNC of chemical amendments does not provide a useful estimate of total BNC, but instead represents a short-term value that is not related to total amendment BNC. Total theoretical BNC for the chemical amendments will be determined from their chemical formula. On the other hand, BNC of peat cannot be derived in this way and will be measured by titration to provide an estimate of the concentration of acidic functional groups in the organic matter that are involved in acid-base reactions.

The ANC titrations of porewater and BNC titration of peat will be conducted at PSU in a glove box under oxygen-free conditions.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

Analytical data will be collected in accordance with the Onondaga Lake Quality Assurance Project Plan as Level III data and will not undergo formal data validation. The data will be evaluated by the project manager as to consistency with site conditions and developed conceptual models to determine whether the analytical data meets the requirements for decision making.

8.0 GEOCHEMICAL MODELING

8.1 Batch Modeling

The geochemical models developed for amendment screening will be extended to include the rate of the pH neutralization process. This will be accomplished using the USGS-supported modeling software PHREEQC (Parkhurst and Appelo, 1999), which includes the processes of one-dimensional advection and diffusion, aqueous speciation, mineral dissolution and precipitation, surface complexation, ion exchange, redox transformations, and time-dependent biogeochemical and chemical reactions. PHREEQC was originally developed in the early 1980s and has been used to model sediment and soil systems in numerous peer-reviewed journal publications. Examples of these include the simulation of batch and column experiments (Delolme et al. 2004; Tiruta-Barna 2008), leaching of cementitious wastes (Pierrard et al. 2002; Halim et al. 2005), and porewater-surface water interactions (Balistrieri et al. 2003).

The objective of modeling the batch experiments using PHREEQC is to determine the rate constants for the dissolution of individual amendments in synthetic porewater. This is necessary for modeling the long-term performance of the amendments, as discussed in Section 8.2. Rate constants will be determined through a calibration process during which other primary variables affecting dissolution and pH neutralization will have been characterized in the laboratory, and the dissolution rate constant is the principal unknown. Rate constants will be adjusted during calibration until the model is able to replicate time-dependent changes in pH.

8.2 Cap Simulations

Cap simulations will also be performed using PHREEQC. These model runs will simulate a series of 1-D, *in situ* column experiments that will include a non-reactive quartz sediment cap comprised of between 0.5-10% chemical amendment and inflowing hyperalkaline groundwater. Implicit in this model will be the incorporation of the rate expressions derived above in order to predict the long-term effectiveness of amendments in maintaining an appropriate pH level (pH of 8). Model runs will be used to predict the performance of the cap over the course of several years, with simulation variables in individual runs which include amendment dose rate (quantity applied) and amendment distribution (single-layer reactive mat application vs. homogenization within the cap).

The cap simulations will predict the amount of amendment dissolved, porewater chemistry (pH and dissolved ions), and the formation of secondary precipitates as a function of time and depth. These model results will be used to evaluate the amount of each amendment required to provide long-term pH neutralization. They will additionally provide a basis for estimating the amount of each amendment and selecting the most suitable amendment(s) for potential design-level laboratory column testing. Criteria that will be employed in evaluating final performance for column testing will include dosage, long-term neutralization capacity, and cost.

9.0 REPORTING

Reported information for each batch test will include the following:

- sample ID and date of sampling;
- description of the amendment added;
- solids content;
- sample weight;
- date and time batch testing was started, monitoring performed, and samples extracted and analyzed;
- electrical conductivity, temperature, and pH for each monitoring event;
- observation of changes in test material or solution recorded during sampling;
- final synthetic porewater composition (selected samples); and
- final mineralogy (selected samples).

Summary information for chemical amendments (grain size and BET surface area), sediment (mineralogy), and synthetic porewater (initial and final chemical composition) will also be reported.

The report will provide the following information:

- summary of the batch test laboratory results;
- summary of the geochemical model calibration;
- description and summary of the cap simulation assessment, including source strength and duration of the various amendments simulated;
- ranking and screening of the amendments (relative to cost and the availability of high volume suppliers in the Syracuse area);
- short list of candidate amendments recommended for potential future bench-scale column testing; and

- discussion of cap design considerations (dosing and emplacement of amendment layer and any secondary issues such as elevated sulfate or phosphate).

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