



Florida Onsite Sewage Nitrogen Reduction Strategies Study

Task A.15

Passive Nitrogen Removal Study II Quality Assurance Project Plan

Final Report

November 2009

Revised and Amended February 2010

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HAZEN AND SAWYER
Environmental Engineers & Scientists

In association with



AET
Applied Environmental Technology

**OTIS
ENVIRONMENTAL
CONSULTANTS, LLC**

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TASK A.15 FINAL REPORT

Passive Nitrogen Removal Study II Quality Assurance Project Plan

Prepared for:

Florida Department of Health
Division of Environmental Health
Bureau of Onsite Sewage Programs
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FDOH Contract CORCL

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Section 1.0

Project Organization and Management

The Florida Department of Health has contracted to continue the study of passive nitrogen removal (PNRS II) under Task A of the Florida Onsite Sewage Nitrogen Reduction Strategies Study (FOSNRS). PNRS II is a follow up to the previous experimental evaluations of passive nitrogen removal technologies conducted under Contract CORY (Passive Nitrogen Removal Study I). The Passive Nitrogen Removal Study II (PNRS II) will be conducted by Hazen and Sawyer and Applied Environmental Technology, who will perform overall project management, establish and conduct the pilot studies, and who will deliver samples for water quality analyses to an approved analytical laboratory. The contractors will review and interpret the resulting data, adjust the pilot testing program as warranted, and generate a summary report and recommendations. Prudent project management will help minimize changes, ensure project continuity, and avoid delays in the project schedule. This type of project is highly specialized, requiring unusual equipment and services. Therefore it is crucial that adequate project management be used to ensure the success of the project. Figure 1-1 depicts the organization chart for PNRS II.

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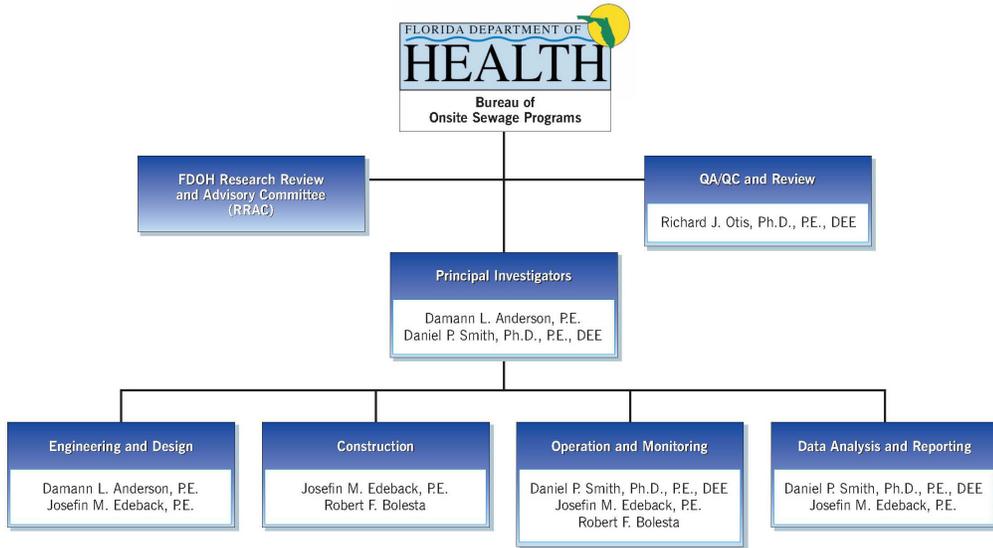


Figure 1-1: Organization Chart for PNRS II

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Section 2.0

Problem Definition and Background

2.1 Project Background

The Florida Department of Health (FDOH) has provided funding to evaluate methods that can be used to enhance nitrogen removal in onsite wastewater systems in a passive and cost effective manner. The Florida Onsite Sewage Nitrogen Reduction Strategies Study (FOSNRS) Passive Nitrogen Removal Study II (PNRS II) QAPP entails formulating a pilot testing plan to evaluate candidate technologies that can be used to remove nitrogen from septic tank effluent with more passive systems. The purpose of the PNRS II study is to extend and expand into field pilot testing the previous experimental studies of the two-stage biofiltration process that were conducted in PNRS I. PNRS II will perform field testing of passive nitrogen reduction treatment systems using a variety of candidate biofiltration media. Pilot test systems will consist of various configurations of in-tank biofilters and passive in-situ systems. The results of PNRS II may be used to develop and implement subsequent evaluations of full-scale systems that will be conducted under Task B of this project.

The *Florida Passive Nitrogen Removal Study Literature Review and Database* proposed the development of a two stage biofilter system for passive removal of total nitrogen from septic tank effluent (Smith et al., 2008). The two stage system consisted of an initial unsaturated media biofilter for ammonification and nitrification, followed in series by a saturated anoxic denitrification biofilter. The system would be deployed between the septic tank and the soil treatment unit (drainfield) or soil dispersal system of new or existing facilities. Nitrogen in septic tank effluent would be substantially removed before wastewater was directed to the soil for treatment or dispersal. Results from the previous experimental studies conducted in PNRS I provided the proof of concept of the two-stage passive nitrogen reduction system.

To perform PNRS II testing, it is desired to conduct studies in a manner that more closely resembles the functioning of actual onsite systems. Actual candidate media will be used, and placed in appropriate layers and depths distribution. Continuously operated biofilter operation will be employed, where microbial populations will establish their metabolic activities and perform desired biochemical transformations in response to conditions similar to an operating system. In addition, in-situ testing will be conducted using in-situ simulators consisting of subsurface drip irrigation application to the root zone of

surface vegetation, followed by downward transport through a layer of filter sand and engineered media. The use of actual septic tank effluent (STE) as feed source is deemed preferable to use of a synthetic analog STE. This Quality Assurance Project Plan (QAPP) describes the methods and procedures that will be used to conduct the passive nitrogen removal evaluations.

2.2 Candidate Study Site

A candidate site, the University of Florida Gulf Coast Research and Education Center (GCREC), has been identified and arrangements are being sought for its use. The acceptability of the site has been established. The chosen site has a source of actual septic tank effluent or primary effluent, a power supply to pump STE to test biofilters, and power for operation of equipment. The site location is isolated from public access and would cause minimal disruption to any activity, and it has reasonable security. The site is located in Hillsborough County, Florida.

Section 3.0

Project Description

3.1 Project Purpose

To evaluate candidate media and treatment processes for development of more passive nitrogen removal systems for onsite wastewater treatment.

3.2 Project Objectives

The objective is to establish pilot passive nitrogen removal systems to evaluate the effectiveness of various media and two-stage biofilter designs in removing total nitrogen from septic tank effluent. The pilot test systems will consist of various configurations of in-tank biofilters and passive in-situ systems. In-tank systems will primarily employ variants of the two-stage biofiltration concepts elucidated in PNRS I. In-situ technology evaluation will include a drip irrigation system for effluent dosing, with emitters located in shallow root zones.

In the two-stage biofilter process, a first stage unsaturated biofilter is followed in series by a second stage biofilter operated in a water saturated mode. Septic tank effluent will be applied to the top of the first stage media, resulting in a downward percolation of wastewater over and through the media biofilter bed. The unsaturated pore spaces in the first stage media will allow air to reach microorganisms attached to the media surfaces, enabling aerobic biochemical reactions to occur. The significant target reactions are aerobic heterotrophic oxidation (by microorganisms that oxidize organic material and reduce biochemical oxygen demand), hydrolysis and ammonification (releasing ammonia), and nitrification (biochemical conversion of ammonia to nitrate and nitrite). Of particular interest are the organic and ammonia nitrogen concentrations in first stage effluent, as well as nitrate and nitrite.

Effluent from the bottom of the first stage biofilter is passed through a saturated anoxic biofilter that contains a reactive media that supplies electron donor for denitrification (reduction of nitrate and nitrite to N_2 gas). The biofiltration systems will be operated over a twelve month period, if funding is available, and monitored for nitrogen species and other water quality parameters. Of particular interest are the concentrations of ammonia in first stage effluent and nitrate, nitrite and total nitrogen in the second stage effluent.

The interaction of media with applied wastewater governs the treatment process. Key features affecting nitrogen removal performance include:

1. The effects of hydraulic and nitrogen loading rates, on average daily and per dose basis, on first stage effluent nitrogen concentrations.
2. The effects of first stage media on effluent nitrogen levels.
3. Alkalinity consumption in the first stage and its possible effects on nitrification.
4. The effects of hydraulic and nitrogen loading rates, on average daily basis, on second stage effluent nitrogen concentrations.
5. The effects of second stage media on effluent nitrogen levels.
6. Second stage effluent total nitrogen concentrations and speciation into organic, ammonia, and oxidized nitrogen forms.
7. Alkalinity consumption or restoration in the second stage and its possible effects on denitrification.
8. Use of first stage recycle.

3.3 Project Tasks and Timeline

Project tasks and preliminary timeline are shown in Table 3.1. The start dates and tasks are contingent upon Recommendations for Process Forward (FOSNRS Task A.14). The task descriptions provide a template by which the project team will conduct the PNRS II project. The nature of technology demonstration projects will necessitate system and testing modifications during the course of the study. It is important to recognize that operational adaptation is a central feature of pilot testing and process optimization. A typical example is a modification in operation as a result of assessment of performance data, where a higher loading rate is applied to a well functioning system to evaluate performance over a wider loading envelope. The QAPP establishes initial loading rates for PNRS II systems that may be adjusted as the study progresses, based on ongoing results and the professional judgment of the project team. A degree of discretion must be afforded to the project team to make modifications as warranted. Additionally, longer term operation of successful onsite treatment systems is warranted but dependent on future funding. All substantive modifications will be fully communicated to FDOH.

**Table 3.1
Project Tasks and Timeline**

Task/Activity	Start	Projected Completion
Task 1 PNRS II infrastructure design	Week 1	Week 4
Task 2 Procurement of materials and media	Week 4	Week 8
Task 3 Construction of test facility and pilot systems	Week 6	Week 10
Task 4 Operation and monitoring of pilot systems	Week 12	Week 64
Task 5 Preparation of draft report	Week 68	Week 74
Task 6 Preparation of final report	Week 76	Week 80

Task 1: PNRS II Infrastructure Design

A final testing site will be established based on the acceptability of wastewater sources, use of the site for other FOSNRS work elements in Tasks B and C, and establishing site use arrangements. Once test facility infrastructure is designed (Tasks A.17 through A.19), the design of PNRS II infrastructure can begin and will be integrated into the test facility design. The design documents will define the needed materials and construction of the PNRS II testing component.

Task 2: Procurement of Materials and Media

Candidate media for evaluation in Stage 1 (unsaturated) biofilters and Stage 2 (saturated) biofilters are listed in Table 3.2, with physical properties and their sources. Included are media with high water retention and porosity, and the clinoptilolite additionally provides ion exchange capacity. Media will be procured from vendors for use (Table 3.2). Stage 1 media includes filter sand, expanded clay and clinoptilolite. The latter two exhibit greater than 45% porosity and high water retention. Characteristics of hydrous sodium aluminosilicates, clinoptilolites, include cation exchange capacities of 1.5 to 1.8 meq/g, high specific surface area generally 40 m²/gram, and will act to retain ammonia ions for enhanced ammonia removal under non-steady flows and higher loading rates. Livlite is an expanded clay with high water retention characteristics. Expanded polystyrene is a very lightweight, readily available and low cost material that appears to be quite suitable as a biofilter media for aerobic treatment.

The Stage 2 electron donor media are elemental sulfur, which will result in an autotrophic denitrification process in the anoxic biofilter; lignocellulosic materials, such as woodchips, which support heterotrophic denitrification, and glycerol, a readily available carbon source for heterotrophic denitrification. Crushed oyster shell and sodium sesquicarbonate will be used as alkalinity sources in sulfur-based denitrification biofilters, as auto-

trophic sulfur-based denitrification will consume alkalinity. Expanded shale may be included as a Stage 2 option for its anion exchange capacity to enhance nitrate removal performance. Stage 2 biofilters will be monitored for sulfate and CBOD which will characterize concentration and indicate the reduction achieved prior to soil infiltration following the systems.

Table 3.2
Biofilter Media

Material	Bulk density, lb/ft ³	Typical Particle Size Range as Supplied	Supplier
Zeo-Pure AMZ 8/20 Clinoptilolite	55	0.8 – 2.3 mm	Ash Meadows, Armagose, NV
Livlite (expanded clay)	41	3 to 5 mm	Big River, Alpharetta, GA
Expanded Polystyrene	0.34 – 1.5	2.2 – 3.6 mm	JSP
Elemental sulfur	77	2 – 4 mm	Georgia Sulfur, Valdosta, GA
Oyster shell	82	3 – 15 mm	Misc. Locations, FL
Sodium Sesquicarbonate T-50	69	1 – 3 mm	Solvay
Lignocellulosic material (woodchips, sawdust)	20 – 28	1 to 5 mm	Robbins Products, Tarrytown, FL
Glycerol	79	-	Greenhunter Energy
ACT-MS ESF-450 Utelite (expanded shale)	54	0.4 – 4.5 mm	ES Filter, Ogden, UT
Sand	100	0.8 - 1.2 mm 0.45 – 0.55 mm	National Suncoast Media, Gulfport, FL
Gravel	100	1 – 4 mm	National Suncoast Media, Gulfport, FL

Task 3: Construction of Test Facility and Pilot Systems

A test facility will be constructed that will provide a source of primary effluent (i.e. septic tank effluent) to the PNRS II systems, as well as dosing regimes, sampling ports, and effluent collection. Design of the test facility will be conducted under FOSNRS Tasks A.17 through A.19. Two types of testing systems will be constructed:

- A. Vertical/Horizontal Two-Stage Biological Filtration
- B. In-Situ Vegetation/Media Simulators

A. Vertical/Horizontal Two-Stage Biological Filtration

The two-stage biofiltration systems consist of a vertical unsaturated biofilter followed by a saturated denitrification biofilter. The general concept of a typical two-stage biofiltration process is illustrated in Figure 3-1. Primary effluent (i.e. septic tank effluent) is dosed to the upper surface of the Stage 1 biofilter, trickles through the unsaturated media, and then flows by gravity through the saturated horizontal denitrification biofilter. In PNRS II pilot testing, multiple Stage 1 biofilters will be operated in parallel on the same primary effluent, and multiple Stage 2 biofilters will be operated in parallel on Stage 1 effluent.

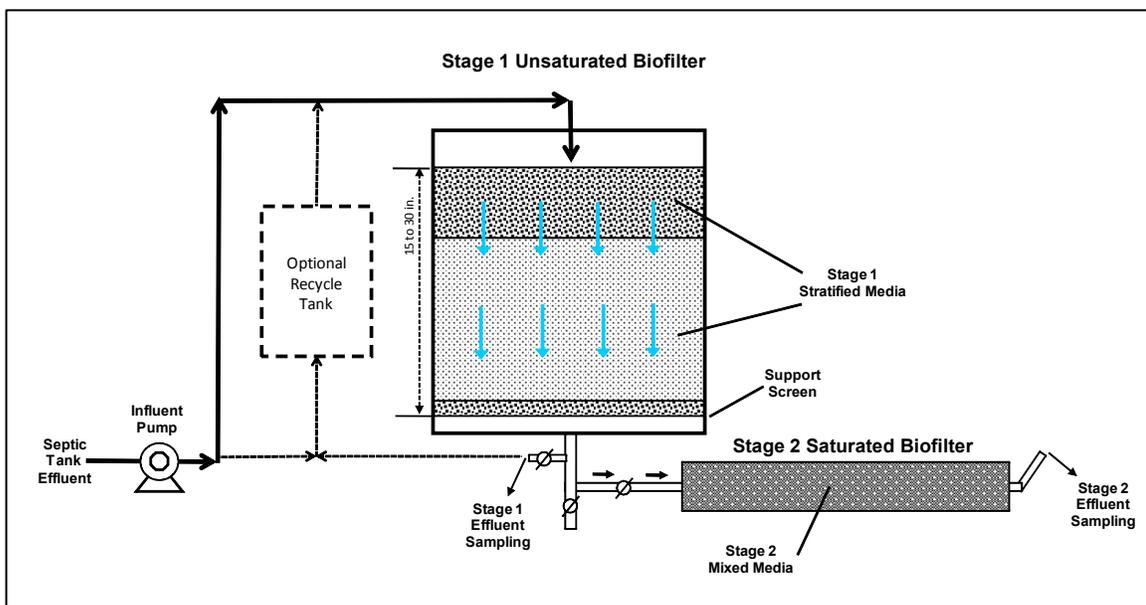


Figure 3-1: Schematic of Vertical/Horizontal Two-Stage Biofiltration Concept

An illustration of different configurations of the Stage 1 unsaturated biofilters is shown in Table 3.3. Four biofilter media will be examined in PNRS II pilot studies: expanded clay and clinoptilolite, both of which were evaluated in PNRS I, expanded polystyrene, a readily available low cost and light weight material, and filter sand, the most commonly used filter medium representative of a control system. Design of the expanded clay and clinoptilolite pilot biofilters will be guided by the results of PNRS I. The test matrix consists of two media depths (15 and 30 inch) and single pass and recycle operation (Table 3.3). All expanded clay, filter sand and clinoptilolite biofilters will employ a two layer stratified design for particle size (Table 3.4). The expanded polystyrene biofilter will be evaluated in single pass operation (Table 3.3). All pilot Stage 1 biofilters (Systems 1 through 11 in Table 3.3) will be dosed at a 30 to 60 minute interval (24 to 48 doses/day), which is simi-

lar to the dosing regime that was employed successfully in PNRS I. Systems 10 and 11 in Table 3.3 are in-situ simulators that consist of simulated dosing by drip irrigation tubing into mound sand media underlain by an engineered media with expanded clay, lignocellulosic and sulfur electron donor.

The initial hydraulic loading rate to Stage 1 biofilters 1 through 9 will be 3 gallon/ft²-day. As performance data is gathered over the course of the study, it is expected that this loading rate will be progressively increased. The PNRS II pilot studies will include recycle systems to delineate total nitrogen removal by pre-denitrification, and the use of two media size stratification and different media depths than were applied in PNRS I. These factors have direct technological and cost savings implications as they would affect the size of the treatment biofilters.

Stratification of media based on particle size is based on the expected progression of biochemical reactions within the biofilter. The processes in the upper coarse media layer include adsorption of wastewater particulates and colloids, hydrolysis and release of soluble organics, aerobic utilization of soluble organics, and biomass synthesis. In the upper layer, the biochemical processing of organic matter between doses must keep pace with the newly applied wastewater constituents from each dose. The greatest accumulation of organic and inorganic mass will occur in the upper layer, and the use of larger particle size media will provide greater space for accumulation of solids. Stratified media should enhance the potential for long term operation while maintaining treatment efficiency. The use of finer particle sizes in the lower media depths will provide greater surface area for microbial attachment and physical filtration, the later which could improve removal of pathogens and other wastewater constituents. The coarser sized particles in the upper layer will also filter out larger particulates and protect the underlying finer media. The two layer media size stratification (Table 3.4) is a simplification of the 3 layer design employed in PNRS I; the two layer design will simplify construction and reduce costs.

Table 3.3
Stage 1 Vertical Unsaturated Biofilter Configuration and Initial Operation

Unsaturated Biofilters (Stage 1)					
No.	Media	Biofilter	Media Depth (Inches)	Flow Regime	Recycle Ratio (α)
1		UNSAT-EC-1	15	Single Pass	-
2	Expanded Clay or Filter Sand	UNSAT-SAND-2		Recycle	3
3		UNSAT-EC-3	30	Single Pass	-
4		UNSAT-EC-4		Recycle	3
5	Clinoptilolite	UNSAT-CL-1	15	Single Pass	-
6		UNSAT-CL-2		Recycle	3
7		UNSAT-CL-3	30	Single Pass	-
8		UNSAT-CL-4		Recycle	3
9	Polystyrene	UNSAT-PS-1	30 (NS)	Single Pass	-
10	Upper: Mound Sand	UNSAT-IS-1	12	Single Pass	-
11	Lower: Expanded Clay, Lignocellulosic, Sulfur	UNSAT-IS-2	12	Single Pass	-

EC: expanded clay, CL: clinoptilolite, PS: polystyrene, SU: sulfur, α : recycle flowrate/forward flowrate, NS: non-stratified

Specification of pilot hydraulic loading rates was guided by the results of PNRS I. Unsaturated expanded clay and clinoptilolite biofilters both exhibited exceptional performance at 3 gallon/ft²-day. The PNRS I results suggest that the potential of these media was not fully utilized. The PNRS II pilot study will delineate treatment performance under real world conditions at the PNRS I loading rate of 3 gallon/ft²-day and at higher loading rates. Higher loading rates translate into a smaller footprint for Stage 2 biofilters and significantly lower construction costs. The general experimental progression will be to establish performance at the initial hydraulic loading rate of 3 gallon/ft²-day, characterize performance at that loading rate for approximately 3 months of operation, and if that operation is consistent, to modify operation to a fixed, higher hydraulic loading rate and characterize performance at that new operating condition.

Table 3.4
Stage 1 Vertical Unsaturated
Biofilter Media Depth and Stratification

Total media depth, inch	Layer	Media layer depth, inch	Particle diameter, mm
15	Upper	5	1.5 – 2.5
	Lower	10	0.3 – 0.6
30	Upper	10	1.5 – 2.5
	Lower	20	0.3 – 0.6

The Stage 1 biofilters will be supplied with septic tank effluent with a timed dosing of once per one half hour to one hour (24 to 48 doses/day), as was employed in PNRS I. A centrally located dosing system will be used to distribute primary effluent over the surface of the media of each Stage 1 biofilter. Water will percolate downward through the Stage 1 media, through the support screen, and into a line that conveys biofilter effluent to either the directly connected Stage 2 biofilter or the common Stage 1 effluent collection chamber. The water elevation in the line below the Stage 1 biofilter will provide hydraulic head for passive movement of water to the common collection chamber. A valve and sample port (with another valve) will be located in the line below the Stage 1 biofilter. In normal biofilter operation, the sample port valve will be closed and the valve leading to the effluent collection chamber will be open. The design of the biofilter system will minimize internal volumes within the connecting piping. At 48 doses per day and 3 gal/ft²-day, a single dose will add a volume that is approximately 6% of the water retained within the Stage 1 biofilter bed of a single pass system (Smith et al., 2008).

Unsaturated biofilter Systems 10 and 11 in Table 3.3 will be in-tank analogs of the in-situ simulators that will be placed in the ground as described in Section 3.3 Task 3B. The media configuration of Systems 10 and 11 is shown in Table 3.5. Biofilter Systems 10 and 11 have two significant media differences from the in-situ simulators that will be placed in the ground: they will not include plants at the upper surface, and will not include natural soil horizons. System 10 will receive septic tank effluent and System 11 will receive nitrified effluent supplied over a capillary seepage mat. Systems 10 and 11 will both be dosed at 0.8 gal/ft²-day and will not be subject to rainfall inputs at the surface. Sample ports will be provided at 4 inch increments along the depth of the biofilter, which will enable six point longitudinal profiling of nitrogen species and other water quality parameters. The design of the in-tank in-situ simulators will enable quantification of liquid volumes exiting the filter.

**Table 3.5
In-Situ Biofilter Media Depth and Stratification**

Total media depth, inch	Layer	Media layer depth, inch	Particle diameter, mm
24	Upper – Mound Sand	12	Slightly limited clean sand
	Lower - Engineered Media	12	0.5 – 1.0

Configuration of the Stage 2 saturated denitrification biofilters is shown in Table 3.6. The Stage 2 biofilters will be constructed with unstratified mixed media containing elemental sulfur, crushed oyster shell, sodium sesquicarbonate, lignocellulosic materials, expanded clay, and filter sand (Table 3.6). The use of elemental sulfur with oyster shell was successfully demonstrated in PNRS I. Sodium sesquicarbonate will provide alkalinity supply which will not release calcium and reduce the potential for calcium carbonate precipitation. The use of lignocellulosic materials as a source of organics in denitrification filters was reviewed in the PNRS I literature review. Expanded clay was also evaluated as microbial attachment medium in PNRS I. Glycerol is a low cost fermentable substrate which serves as a denitrification electron donor. Glycerol will be added by dosing pump or other methods.

Stage 2 biofilters will employ non-stratified mixed media of 1 to 2 mm particle size. The configuration of the Stage 2 biofilters that are supplied by the common Stage 1 STE effluent (i.e. Nos. 1, 2, 5, and 9 in Table 3.6) is as 6 inch diameter columns of 72 inch length. Sample ports will be provided at 12 inch increments along the length of the biofilter, which will enable six point longitudinal profiling of nitrogen species and other water quality parameters. The configuration of the Stage 2 biofilters that are directly connected to Stage 1 biofilters (i.e. Nos. 3, 4, 6, 7, and 8 in Table 3.6) is as 22 inch diameter circular upflow filters of 30 inch media depth. Sample ports will be provided at 6 inch increments along the depth of the biofilter, which will enable five point longitudinal profiling of nitrogen species and other water quality parameters. Detailed design will be conducted in Tasks A.16 through A.19.

Like PNRS I, the pilot PNRS II biofilter systems will be configured for simplicity of operation, minimal moving parts and passive gravity flow where possible. The same primary effluent (i.e. septic tank effluent) will be supplied to the surface of each of the Stage 1 vertical biofilters, which will be placed above ground to allow effluent to flow by gravity to either a directly connected Stage 2 denitrification filter or alternatively to a common Stage 1 effluent tank.

In the initial configuration, the single pass Stage 1 biofilters will be directly connected to Stage 2 denitrification filters, and effluent from the Stage 1 biofilters with recycle will be routed to a Stage 1 effluent collection tank that will produce a common effluent. The Stage 2 denitrification filters that are not directly connected to single pass Stage 1 biofilters will receive effluent from the Stage 1 collection tank by pumps that provide independent flowrate control to each. Stage 2 biofilters will be maintained in saturated mode by the Stage 2 overflow elevation pipe. Stage 2 effluent will be collected via gravity into a Stage 2 collection tank, for management or disposal. Details of design and fabrication of pilot biofilter systems will be addressed in Tasks A.16 through A.19.

Monitoring sample points are septic tank effluent, Stage 1 effluents, the common Stage 2 influent, and Stage 2 effluents (Table A.1). For each monitoring point, separate samples will be collected for field analyses and for laboratory analyses. Field analyses will be performed immediately upon sample collection. Samples for laboratory analyses will be collected by directing samples directly into sample collection containers that are located within iced coolers and that contain any required sample preservatives. Influent and effluent samples will not have contact with any intermediate sample devices. Effluent samples will be maintained in iced coolers and transported to the lab within 24 hours of collection.

**Table 3.6
Stage 2 Saturated Denitrification Biofilter
Configuration and Initial Operation**

No.	Electron Donor	Biofilter	Media Composition (by volume)	Initial Surface Loading Rate, gal/day-ft ²	Stage 1 Filter
1 ¹	Elemental sulfur	DENIT-SU-1	80% SU 20% OS	10.0	2,4,6,8
2 ¹		DENIT-SU-2	80% SU 20% NS	10.0	2,4,6,8
3 ²		DENIT-SU-3	80% SU 20% OS	4.7	1
4 ²		DENIT-SU-4	80% SU 20% NS	4.7	7
5 ¹	Lignocellulosic	DENIT-LS-1	50% LS 50% EC	10.0	2,4,6,8
6 ²		DENIT-LS-2	50% LS 50% EC	4.7	3
7 ²		DENIT-LS-3	50% LS 50% SA	4.7	5
8 ²		DENIT-LS-4	30% LS 70% EC	4.7	9
9 ¹	Glycerol	DENIT-GL-1	12" GR 60" EC	10	2,4,6,8

SU: elemental sulfur, LS: lignocellulosic, GL: glycerol, OS: oyster shell, NS: sodium sesquicarbonate, EC: expanded clay, SA: sand, GR: gravel

1. Fed from common Stage 1 effluent collection tank.
2. Directly connected to Stage 1 unsaturated biofilter

B. In-Situ Vegetative/Media Simulators

In-situ testing will be conducted using in-situ simulators as shown in Figure 3-2. The simulators will consist of subsurface drip irrigation application to the root zone of surface vegetation, followed by downward transport through a 12 in. layer of mound sand. Underlying the mound sand is a 12 in. layer of engineered media containing electron donor which is in turn underlain by natural soil.

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The configuration of the in-situ simulators is shown in Table 3.7. The 2¹ test matrix consists of subsurface drip irrigation emitter dosing of primary effluent (i.e. septic tank effluent) or nitrified effluent into the root zone of St. Augustine grass. The in-situ simulators will receive an average hydraulic application rate of 0.80 gallon/ft²-day on an aerial basis applied at 6 doses/day. Drip emitters will be placed at 12 inch spacings. Other than the pumping of effluent by subsurface irrigation, the in-situ simulators are completely passive systems.

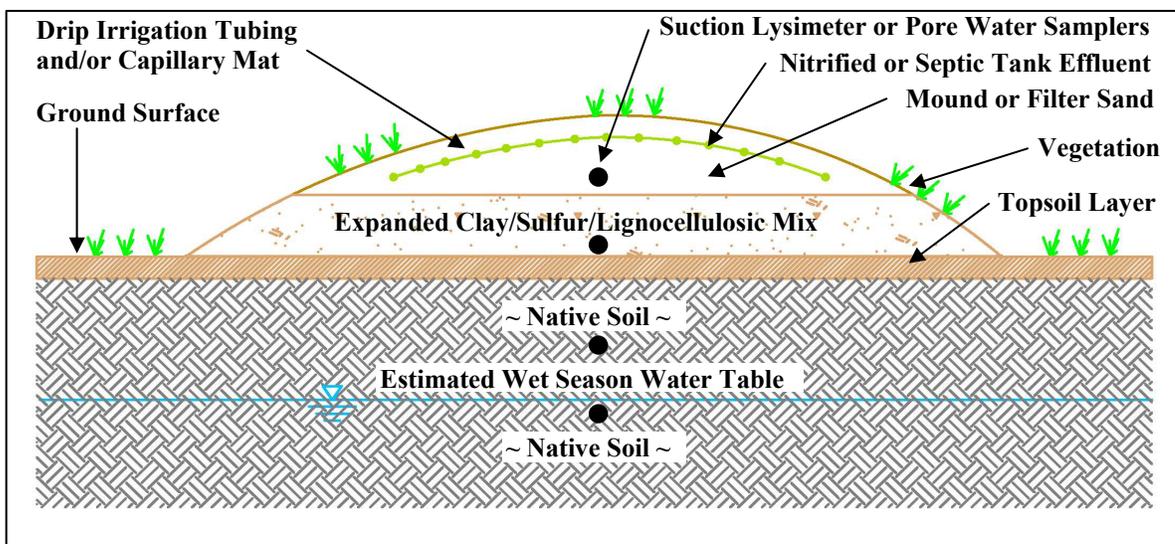


Figure 3-2: Cross-Section Schematic of In-Situ Vegetative Denitrification - Media Treatment System

In the INSITU-1 simulator, primary effluent (i.e. septic tank effluent) will be applied by subsurface drip irrigation to a near surface location, such that STE will interact with the active root zone of plantings, trickle downward through the sand layer and a 12 in. zone containing electron donor media, and then pass through an underlying zone of natural undisturbed soil (Figure 3-2).

In INSITU-2, primary effluent will first be nitrified and then applied to a near surface location by drip irrigation and using an innovative application of a capillary seepage mat that has been developed for irrigation of agricultural plants by scientists at the University of Florida Gulf Coast Research and Education Center (GCREC). Nitrified effluent will interact with the active root zone of plantings, trickle downward through the sand layer and a

12 in. zone of engineered media, and then pass through an underlying zone of natural undisturbed soil (Figure 3-2).

An innovative feature of the in-situ simulator design is the use of mixed media in unsaturated mode that contains both a high water retention media (expanded clay) and heterotrophic and autotrophic electron donor (Table 3.7). The media mix will provide three electron donor source options for denitrification: wastewater organics, lignocellulosics, and elemental sulfur. The use of solid electron donor media in an unsaturated operational mode will facilitate both aerobic processes (i.e. nitrification) and denitrification. This design will provide conditions for both nitrification and denitrification, with an additional supply of electron donor over that which would be available from with wastewater organics or endogenous carbon sources alone.

The goal of this testing is to quantify nitrogen reduction in systems where STE or nitrified effluent is applied with subsurface drip emitter tubing or capillary mat to shallow locations within the subsurface which contain plant root zones, unsaturated media, and electron donor media for enhanced denitrification. Timed dosing to shallow application points in the subsurface could be capable of affecting nitrogen reduction. This potential for in-situ treatment systems, including plant-assisted nitrogen transformations, has not been examined in Florida with innovative systems of this type but is of potentially high significance.

Issues that may affect nitrogen reduction are average daily hydraulic application rate, horizontal emitter spacing, doses per day, volume per dose, and the depth at which the bottom of emitter tubes is placed. Emitter tubing is available with spacings of as little as 12 in., which are preferred to typical 24 in. emitter spacings and will be used in this study. The lower emitter spacing results in lower effluent volume per dose at each emitter that are spread more uniformly over the plan area of the dosing zone, thereby increasing the effectiveness of utilization of the total plan area of the receiving surface. Hydraulic application rate affects volume per dose for any given dosing schedule, as interrelated to dosing frequency. As the average daily hydraulic application rate increases, the vegetative/media system will be increasingly challenged to assimilate nitrogen in the applied STE and limit downward nitrogen migration. The depth of emitters and the relationship of emitted effluent to surface vegetation root zones is an ostensibly significant factor affecting total nitrogen reduction. A dosing event can lead to water saturation in a temporally and spatially limited zone that creates oxygen limited conditions that favor denitrification. After saturated conditions end, microenvironments with limited DO can persist and provide continued denitrification. When bulk pore spaces are filled with air, conditions can favor nitrification. Plant roots can exude organic carbon and provide an electron donor rich region. The combination of the supply of organic carbon and reduced

nitrogen in the applied STE, the varying saturation and oxygen levels resulting from the dosing regime, and the characteristics of the plant root zone can affect sequential nitrification and denitrification reactions. Downward advective transport of organic carbon and nitrate can create a biologically active denitrification zone of some vertical extent. The interaction of all of these factors will determine the extent to which total nitrogen reduction can be affected by drip application of STE into plant/media systems and the significance of plant processes on overall nitrogen reduction. Another factor is downward migration of exudates from the in-situ treatment processes, including biochemical oxygen demand and sulfate that will result from the electron donor media in the engineered layer of the drainfield which will be monitored. Consideration of the additives rule per Florida Administrative Code (FAC) Chapter 64E-6 will be addressed under FOSNRS Task A.16 "Materials Testing for FDoH Additives Rule" further described in Appendix B. Detailed design of in-situ simulators will be conducted in Tasks A.17 through A.19.

For all PNRS II pilot units, system shakedown will proceed following fabrication and set up. System integrity and hydraulics will be fully evaluated with clean water. Basic features of system integrity and hydraulic conveyance will be examined, including system leaks, gravity flow conveyance where applicable, operation of pumps and valves, and sample access functionality. Media will be pre-screened where needed, washed at least three times to remove fines, and placed to appropriate depths in the biofilters. Denitrification biofilters will be initially filled with a clean water source which will be displaced upon commencement of operation. Operation on wastewater will proceed and flow monitoring will be commenced.

Table 3.7
In-Situ Vegetation/Media Simulator Configuration and Operation

No.	In-Situ Simulator	Influent	Flow Application	Unsaturated Media	Saturated Media	Hydraulic Loading Rate, Plan Area Basis, gallon/ft ² -day	Dosing Regime
1	INSITU-1	Primary effluent	Subsurface Drip Irrigation Tubing	12 in. mound sand 12 in. 0.5-1 mm 45% EC 35% LS 20% SU	Native soil	0.80	6/day
2	INSITU-2	Nitrified effluent	Subsurface Drip Irrigation Tubing	12 in. mound sand 12 in. 0.5-1 mm 45% EC 35% LS 20% SU			

SU: elemental sulfur LS: lignocellulosic EC: expanded clay

Task 4: Operation and Monitoring of Pilot Systems

The biofilter systems will be operated over a twelve month period, dependent on additional funding, during which six monitoring events will be conducted. The analytical template is shown in Table 3.8. A detailed analytical description is included in Appendix A. As outlined in Table A.1, there are up to 32 sampling points and a monitoring analyses structure that employs four analytical tiers. Tier 1 analytes include field and laboratory parameters that will be monitored at each sample point (up to 32) and at each sample event. Potential monitoring points are STE (1), Stage 1 effluents (11), Stage 2 influent (1), and horizontal Stage 2 effluents (9). In addition, the in-situ soil/vegetative simulator effluents will be monitored at 2 sampling points within each mound at up to 5 depths each (10). Tier 1 analytes include field parameters (temperature, pH, dissolved oxygen (DO), and oxidation reduction potential (ORP); the nitrogen series (laboratory parameters) of total kjeldahl nitrogen (TKN), ammonia (NH₃), and oxidized nitrogen (NO_x); five day carbonaceous biochemical oxygen demand (C-BOD₅) and total suspended solids (TSS). Tier 2 analytes are supporting parameters that will be monitored at much reduced frequency at the sample points. Tier 3 parameters will be conducted only on sulfur-based denitrification biofilter sample points. Tier 4 analyte is fecal coliform which will be monitored at a much reduced frequency at the sample points (Table 3.8).

Table 3.8
Analyses Template

Analysis Tier	Number of events	Sample points	Analytes	Total number of analyses
1	6	32	Temperature	192
			pH	192
			DO	192
			ORP	192
			Alkalinity	192
			TKN	192
			NH ₃ -N	192
			(NO ₃ +NO ₂)-N	192
			C-BOD ₅	192
			TSS	192
2	1 – 4	32	COD	68
			Total phosphorus	38
3	4 – 6	16 (sulfur systems)	Sulfate	108
			H ₂ S	72
4	3	32	Fecal Coliform	96

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Task 5: Preparation of Draft Report

A draft report will be prepared describing pilot testing methods and procedures, results of the research, discussion and conclusions, and all monitoring data. The draft report will be submitted to FDOH for review and comment.

Task 6: Preparation of Final Report

A final report will be prepared based on comments from reviewers of the draft report.

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Section 4.0

Quality Objectives and Criteria

The objective of this monitoring program is to evaluate media for passive nitrogen removal from septic tank effluent. The following summarizes the work to be performed:

- Two stage biofilters and passive in-situ systems will be constructed and operated on primary effluent over a twelve month period.
- The flowrates to each biofilter system provide a range of hydraulic loading rates.
- First stage recycle will be employed to evaluate pre-denitrification.
- Monitoring will be conducted for septic tank effluent, effluent from the Stage 1 (unsaturated) biofilters and effluent from the Stage 2 (saturated) biofilters.
- Field parameters will be monitored at the site. Samples will be collected and transported to the laboratory for analysis of nitrogen species, sulfate and other wet chemistry parameters.
- Operation or configuration of the biofilters will be modified based on analysis of results and adaptive management.
- In-situ soil/vegetative evaluations will be conducted using subsurface drip irrigation technology with emitters located in root zone and monitoring to develop nitrogen concentrations and vertical nitrogen flux.

The monitoring data will be used to calculate:

1. average concentrations and standard deviations of water parameters in septic tank effluent, Stage 1 effluent and Stage 2 effluents;
2. percent removal nitrogen and nitrogen species in Stage 1 biofilters, Stage 2 biofilters and two stage biofilter systems;
3. changes to dissolved oxygen, pH, oxidation reduction potential and alkalinity through biofiltration treatment stages;

4. average applied hydraulic loading rate, applied loading rates of total nitrogen and nitrogen species; and
5. vertical nitrogen flux in in-situ soil/vegetative systems.

4.1 Precision and Accuracy

Precision describes the reproducibility of results. Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy will be evaluated through the analysis of surrogate spikes, Laboratory Control Samples (LCS), Laboratory Control Sample Duplicates (LCSD), matrix spike samples (MS/MSD) and laboratory internal blind audit samples. Precision and accuracy information is tracked by the laboratory, with acceptable ranges updated periodically. NELAC requirements include the analysis of proficiency test samples to evaluate precision and accuracy. Analytical methods, precision and accuracy, method detection limits and practical quantification limits are shown in Table 4.1 for parameters which will be measured as part of the base monitoring program, as well as other potential parameters of interest. GCREC is not a NELAC certified laboratory; however, GCREC staff includes trained and qualified professionals with extensive experience in NELAC procedures and quality control who will insure that NELAC requirements are fully met.

Table 4.1
Aqueous Methodology, Precision and Accuracy, Detection Limits

Analyte	Method	Precision (%)	Accuracy (%)	MDL (ppm)	PQL (ppm)
pH	SM4500H+B	20	NA	0.1 pH units	0.1 pH units
Turbidity	180.1	20	90-110	0.2 NTU	0.2 NTU
Alkalinity	SM2320 B	20	90-110	5.0	5.0
C-BOD ₅	SM5210 B	20	85-115	2.0	2.0
COD	410.4	20	90-110	12.09452	25
TOC	SM5310 B	20	90-110	0.14778	1.0
TSS	SM2540 D	20	90-110	5.0	5.0
TKN	351.2	20	90-110	0.07121	0.5
NH ₃ -N	350.1	20	90-110	0.02	0.05
(NO ₃ +NO ₂)-N	353.2	20	90-110	0.02541	0.05
Total Phosphorus	365.1	20	90-110	0.0094	0.0376
Sulfate	300.0	20	90-110	0.05523	0.5
H ₂ S	SM4500S-E	20	80-120	1.0	1.0
Fecal coliforms	SM9222 B or SM9222 D	20	NA	1.0	1.0
Total coliforms	SM9222 B	20	NA	1.0	1.0
Escherichia coli	SM9222 B	20	NA	1.0	1.0

MDL = method detection limit

PQL = practical quantitation limit

4.2 Representativeness

Representativeness refers to the relationship of a sample taken from a site to be analyzed to the remainder of the sample matrix at the site. The samples will be taken directly from the influents and effluent of the biofilters and will provide representativeness.

4.3 Comparability

The use of NELAC approved procedures and consistent approved methodologies ensure the comparability of data sets generated by different laboratories.

4.4 Completeness

Completeness is defined as a measure of the extent to which the data fulfill the data quality objectives of the project. The completeness of the data will be determined during the data validation and verification process.

Section 5.0

Documentation and Records

All documentation archives will be kept for a minimum of 5 years after the date of project completion as outlined in Table 5.1. Reports and deliverables will be submitted in Word or Excel format.

Table 5.1
Documentation and Records Storage

Document/Record	Location	Retention Time	Format
QAPP and revisions	Hazen and Sawyer, AET	5 years after project completion	Paper, electronic
Field notes	Hazen and Sawyer	5 years after project completion	Paper
Chain of custody	Hazen and Sawyer, Lab	5 years after project completion	Paper
Laboratory QA manual	Lab	5 years after project completion	Paper, electronic
Laboratory SOPs	Lab	5 years after project completion	Paper, electronic
Laboratory data reports	Hazen and Sawyer, Lab	5 years after project completion	Paper, electronic
Laboratory equipment maintenance logs	Lab	5 years after project completion	Paper
Laboratory calibration records	Lab	5 years after project completion	Paper, electronic

5.1 Field Documentation

1. Field Notes
Field notes will be documented and maintained by field staff.

2. Field Parameters
Field staff will record specific sample point, date and time of sample collection, parameter name, result and units

Section 6.0

Sampling Process Methodology

6.1 Site Location

The project will be conducted at the Gulf Coast Research and Education Center in Hillsborough County as discussed in Section 2B.

6.2 Monitoring and Sampling Frequency and Duration

The biofilter systems will be monitored six times, dependent on future funding, over a twelve month period.

6.3 Number of Samples and Matrices

All sampling will be aqueous samples. On each monitoring date, samples will be collected for septic tank effluent, the effluents from Stage 1 biofilters, and the effluents from Stage 2 biofilters. Field analysis will be performed upon sample collection. Aqueous samples for laboratory analysis will be collected in sample containers prepared by the laboratories, maintained in an iced cooler during collection and transport, and transported to the laboratory. Samples will arrive at laboratories within twenty four hours after the completion of collection activities, or as needed for shorter sample hold times. Field analysis will be performed on the same date and for the sample locations taken for aqueous laboratory samples. Samples for field analyses will be collected in separate containers from laboratory samples. Stage 1 and 2 field parameter analyses will be measured in-situ by placing probes directly into collected samples or directly into effluent pipes. Shipping coolers will be supplied and decontaminated by the laboratories. Sample preservation and holding times are provided in Table 6.1 for parameters which will be measured as part of the base monitoring program, as well as other potential parameters of interest. The laboratories will follow all local, state and federal requirements pertaining to waste storage and disposal. No equipment except the sample container will be used to collect the samples, and the sampling equipment will be certified clean by the laboratory providing the equipment. A field blank will be collected for TKN, NH₃ and NO₃+NO₂ for a minimum of 5% of samples collected over the life of the project using distilled water supplied by the laboratories. As a part of its QC, laboratories will perform sample duplicates for a minimum of 5% of samples. Laboratory QC will also include matrix spikes, percent recovery on QC standards, and method blanks.

Table 6.1
Aqueous Matrix Containers, Preservation and Holding Times

Analyte	Method	Minimum Sample Volume	Holding Time	Container Type	Sample Preservation	Preservative Dosage
Physical and Inorganic Parameters						
Alkalinity as CaCO ₃	310.1/SM2320B	100 mL	14 days	250 mL	4° C	n/a
Ammonia	350.1	25 mL	28 days	250 mL	1:1 H ₂ SO ₄ to pH < 2	1 mL/ 250 mL
BOD / cBOD	SM5210B/405.1	1 L	48 hours	1 L Plastic	4° C	n/a
Chloride	300	50 mL	28 days	250 mL	4° C	n/a
COD	410.4	50 mL	28 days	250 mL	1:1 H ₂ SO ₄ to pH < 2	1 mL/ 250 mL
Hydrogen Sulfide	376.1	500 mL	7 days	500 mL Plastic	Zinc Acetate / NaOH	.1 / .5 gm/ 500 mL
Nitrate/Nitrite-N (NO _x)	SM4500	50 mL	28 days	250 mL	1:1 H ₂ SO ₄ / 4° C	1 mL/ 250 mL
Nitrate-N	SM4500	50 mL	48 hours	250 mL	4° C	n/a
Nitrite-N	SM4500	50 mL	48 hours	250 mL	4° C	n/a
Organic Nitrogen (calculation)	350.1/351.2	100 mL	28 days	500 mL	1:1 H ₂ SO ₄ to pH < 2	1 mL/ 250 mL
Ortho Phosphorus	365.4/9056/300.0	25 mL	48 hours	250 mL	4° C	n/a
pH	SM4500HB	50 mL	24 hours	250 mL	4° C	n/a
Sulfate	300	10 mL	28 days	250 mL	4° C	n/a
Sulfide	376.1/9030/9034	500 mL	7 days	500 ml	NaOH + Zn Acetate	1 mL/ 500 mL
TKN	351.2	100 mL	28 days	250 mL	1:1 H ₂ SO ₄ to pH < 2	1 mL/ 250 mL
Total Nitrogen (calculation)	300.0/351.2	100 mL	28 days	250 mL	1:1 H ₂ SO ₄ to pH < 2	1 mL/ 250 mL
Total Organic Carbon (TOC)	415.1/SM5310B	25 mL	28 days	125 mL Plastic	HCl to pH < 2 / 4° C	.5 mL/ 125 mL
Total Phosphorus	365.2/365.4	50 mL	28 days	250 mL	1:1 H ₂ SO ₄ to pH < 2	1 mL/ 250 mL
Total Suspended Solids	160.2	300 mL	7 days	1 L Plastic	4° C	n/a
Turbidity	180.1	30 mL	48 hours	125 mL Plastic	4° C	n/a

Table 6.1
Aqueous Matrix Containers, Preservation and Holding Times

Analyte	Method	Minimum Sample Volume	Holding Time	Container Type	Sample Preservation	Preservative Dosage
Microbiological Parameters						
Total Coliform (MMO-Mug)	SM9223	100 mL	30 hours	Micro-cup	4° C	n/a
Total Coliform (MF)	SM9222	100 mL	6 hours	Micro-cup	4° C	n/a
Fecal Coliform (MF)	SM9222	100 mL	6 hours	Micro-cup	4° C	n/a
Standard Plate Count	SM9222	100 mL	8 hours (DW)	Micro-cup	4° C	n/a
Standard Plate Count	SM9222	100 mL	6 hours (WW)	Micro-cup	4° C	n/a
Fecal Coliform (MPN)	SM9221	100 g.	24 hours	Micro-cup	4° C	n/a

Short hold times

Minimum volume does not include sample volume needed to perform required quality control parameters

6.4 Inspection/Acceptance of Supplies and Consumables

1. Sample Containers
To be provided by the laboratory prior to each sampling event.
2. Sample Coolers
To be provided by the laboratory prior to each sampling event.

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Section 7.0

Data Review, Verification and Validation

7.1 Data Verification

Data verification is the process for evaluating the completeness, correctness, and conformance of the data set against the methodology. This evaluation is integral to the final report. Verification will check that the data were complete, that sampling and analysis matched QAPP requirements, and that Standard Operating Procedures (SOPs) were followed. Verification of data compiled for a sampling event will be the responsibility of the Task Leader.

7.2 Data Validation

Data validation is an analyte and sample specific process that determines the quality of the data set relative to the end use. The entire set of data collected from individual biofilters and from the total set of biofilters operated during PNRS II will be entered into spreadsheets to enable global evaluation of individual parameters, trend analysis, quality of the overall data sets, and assessment of suitability for end use. In this process, outliers and data discrepancies will be identified. Any data deemed to be unusable for the stated objectives will be identified as such in the final report.



Section 8.0 References

References

Smith, D., R. Otis, and M. Flint (2008) Florida Passive Nitrogen Removal Study Final Report. Submitted to the Florida Department of Health, Tallahassee, Florida, June 26, 2008.

Smith, D. (2008) Florida Passive Nitrogen Removal Study Additional Monitoring. Submitted to the Florida Department of Health, Tallahassee, Florida, November 4, 2008.

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Appendix A

Analytical Schedule

Table A.1
Estimated Number of Analyses at each
Monitoring Point for each Sampling Event

Sample point	Influent (STE)	Vertical non-sulfur Stage 1 effluent	Vertical sulfur Stage 1 effluent	Stage 2 influent	Horizontal sulfur Stage 2 effluent	Horizontal non-sulfur Stage 2 effluent	In-situ vegetative/ media simulator SP#1	In-situ vegetative/ media simulator SP#2
No. of sample points	1	9	2	1	4	5	5	5
Analyses	No. of Sample Events							
Temp	6	6	6	6	6	6	6	6
pH	6	6	6	6	6	6	6	6
DO	6	6	6	6	6	6	6	6
ORP	6	6	6	6	6	6	6	6
Alkalinity	6	6	6	6	6	6	6	6
TKN	6	6	6	6	6	6	6	6
NH ₃	6	6	6	6	6	6	6	6
NO _x	6	6	6	6	6	6	6	6
C-BOD ₅	6	6	6	6	6	6	6	6
TSS	6	6	6	6	6	6	6	6
COD	4	2	2	4	2	2	2	2
Total P	4	1	1	4	1	1	1	1
SO ₄	6	0	6	6	6	0	6	6
H ₂ S	4	0	4	4	4	0	4	4
Fecal	3	3	3	3	3	3	3	3

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Table A.2
Estimated Total Number of Analyses
at each Monitoring Point over PNRS II Study

	Influent (STE)	Vertical non-sulfur Stage 1 effluent	Vertical sulfur Stage 1 effluent	Stage 2 influent	Horizontal sulfur Stage 2 effluent	Horizontal non-sulfur Stage 2 effluent	In-situ vegetative/ media simulators SP#1	In-situ vegetative/ media simulators SP#2	Total Samples
	1	9	2	1	4	5	5	5	
Analyses	No. of Samples								
Temp	6	54	12	6	24	30	30	30	192
pH	6	54	12	6	24	30	30	30	192
DO	6	54	12	6	24	30	30	30	192
ORP	6	54	12	6	24	30	30	30	192
Alkalinity	6	54	12	6	24	30	30	30	192
TKN	6	54	12	6	24	30	30	30	192
NH ₃	6	54	12	6	24	30	30	30	192
NO _x	6	54	12	6	24	30	30	30	192
C-BOD ₅	6	54	12	6	24	30	30	30	192
TSS	6	54	12	6	24	30	30	30	192
COD	4	18	4	4	8	10	10	10	68
Total P	4	9	2	4	4	5	5	5	38
SO ₄	6	0	12	6	24	0	30	30	108
H ₂ S	4	0	8	4	16	0	20	20	72
Fecal	3	27	6	3	12	15	15	15	96

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Appendix B

Amendments to QAPP

B.1 February 2010 Amendment for Additives Rule

At the request of FDOH materials testing to comply with *Florida's Additive Rule for Septic System Products* will be conducted as part of this QAPP. Initially, the testing will be for the four products described below. The department shall have the option of ordering (in writing) testing for additional products in future years as it determines necessary for the project. Four submittals will be prepared for the *Florida's Additive Rule for Septic System Products* based on the following products/applications:

- No. 1 **PNRS II Unsaturated Biofilter No. 10**, In-Situ Simulator: Single pass biofilter containing expanded clay, lignocellulosic and elemental sulfur media underlying filter sand and receiving primary effluent
- No. 2 **PNRS II Unsaturated Biofilter No. 11**, In-Situ Simulator: Single pass biofilter containing expanded clay, lignocellulosic and elemental sulfur media underlying filter sand and receiving nitrified primary effluent
- No. 3 **Oyster shell** as a general solid phase alkalinity source for use in Florida onsite wastewater systems, including as media component in saturated and unsaturated biofilters
- No. 4 **Sodium sesquicarbonate** as a general solid phase alkalinity source for use in Florida onsite wastewater systems, including as media component in saturated and unsaturated biofilters

Testing for each product will include collection, assembly, evaluation, and presentation of all required information to enable FDOH evaluation for compliance with *Florida's Additive Rule for Septic System Products*.

For the PNRS II Unsaturated Biofilters (Submittals No. 1 and 2), influent and effluent samples of steadily operating biofilters will be collected. Influent and effluent samples will each be evaluated by Acute Definitive Toxicity Testing (96 hour LC50) using *Bannerfin shiner* according to standard protocols included in *Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012). Laboratory water quality analyses will be conducted on influent and effluent samples, and will include Volatile Organic Compounds (EPA 8260) and possibly also sulfate, hydrogen sulfide and carbonaceous five day biochemical oxygen demand. Bioassays and water quality analyses will be conducted by certified laboratories.

Oyster shell and sodium sesquicarbonate (Submittals No. 3 and 4) will be evaluated as general alkalinity sources for Florida onsite wastewater treatment systems. The approval of these materials under *Florida's Additive Rule for Septic System Products* will enable these materials to be used in a wide variety of onsite wastewater systems throughout the State. These solid granular materials will be evaluated using a batch leaching test procedure that will be developed for the purpose of the *Additive Rule* evaluation. In the batch leaching test, a known mass of granular material will be introduced into a glass leaching chambers and mixed with a synthetic, moderately hard water that is compatible with an acute 96 hours *Bannerfin shiner* bioassay. Batch leaching tests will be conducted with continuous gentle mixing and under zero headspace conditions. The leaching test will be conducted for a 4 to 7 day period. At the end of the leaching period, mixing will be discontinued and the suspension settled for one hour. Supernatant samples will be withdrawn for chemical analyses, followed by supernatant withdrawal for toxicity bioassay. Starting water and leachate samples will each be evaluated by Acute Definitive Toxicity Testing (96 hour LC50) using *Bannerfin shiner* according to standard protocols included in *Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms* (EPA-821-R-02-012). Water quality analyses of influent and effluent samples will be conducted, including Volatile Organic Compounds (EPA 8260), and calcium and sodium if required. Bioassays and water quality analyses will be conducted by certified laboratories.

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