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Testing Large-Volume Water Treatment and Crude-Oil Decontamination Using the EPA Water Security Test Bed

Office of Research and Development
Homeland Security Research Program

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TESTING LARGE-VOLUME WATER TREATMENT AND CRUDE-OIL DECONTAMINATION USING THE EPA WATER SECURITY TEST BED

U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

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Disclaimer

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Abbreviations

AOP	Advanced Oxidation Process
AWWA	American Water Works Association
BTEX	benzene, toluene, ethylbenzene and xylene
BWS	bulk water sample
CB&I	CB&I Federal Services LLC
cfu	colony forming units
Cl_2	free chlorine
cm	centimeter
CP	Coupon
Ct	Concentration of disinfectant multiplied by contact time
DPD	N,N-diethyl-phenylenediamine
DRO	Diesel range organics
EPA	U.S. Environmental Protection Agency
ft	feet
gpm	gallons per minute
GRO	Gasoline range organics
HPC	Heterotrophic Plate Count
HSRP	Homeland Security Research Program
IA	Interagency Agreement
INL	Idaho National Laboratory
kg	kilogram
L	Liter
LCD	Liquid Crystal Display
m	meter
MCL	Maximum Contaminant Levels
µg/L	micrograms per liter
mJ/cm^2	milli-Joule per square centimeter area
mL	milliliter
MPN/mL	most probable number per milliliter
$mW\text{-}sec/cm^2$	milli-Watt-second per square centimeter area
NHSRC	National Homeland Security Research Center
NSF	National Sanitation Foundation
NTU	nephelometric turbidity units
OH	hydroxyl radicals
ORO	oil range organics
pH	numeric scale used to measure acidity or basicity of an aqueous solution
PVC	Polyvinyl Chloride
QAPP	Quality Assurance Project Plan
RF	radio frequency
T&E	Test and Evaluation
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbon
UV	Ultraviolet
VOC	Volatile Organic Carbon
WSTB	Water Security Test Bed

Executive Summary

The U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) partnered with the Idaho National Laboratory (INL) to build the Water Security Test Bed (WSTB) at the INL test site outside of Idaho Falls, Idaho. The WSTB was built using an 8-inch (20 cm) diameter cement-mortar lined drinking water pipe that was previously taken out of service. The pipe was exhumed from the INL grounds and oriented in the shape of a small drinking water distribution system. Effluent from the pipe is captured in a lagoon. The WSTB can support drinking water distribution system research on a variety of drinking water treatment topics including biofilms, water quality, sensors, and homeland security related contaminants. Because the WSTB is constructed of real drinking water distribution system pipes, research can be conducted under conditions similar to those in a real drinking water system.

In 2014, WSTB pipe was experimentally contaminated with *Bacillus globigii* spores, a non-pathogenic surrogate for the pathogenic *B. anthracis*, and then decontaminated using chlorine dioxide. In 2015, the WSTB was used to perform the following experiments:

- Four mobile disinfection technologies were tested for their ability to disinfect large volumes of biologically contaminated "dirty" water from the WSTB. *B. globigii* spores acted as the biological contaminant. The four technologies evaluated included: (1) Hayward Saline C™ 6.0 Chlorination System, (2) Advanced Oxidation Process (AOP) Ultraviolet (UV)-Ozone System, (3) Solstreme™ UV System, and (4) WaterStep Chlorinator.

- The WSTB pipe was contaminated with Bakken crude oil, and decontamination was performed by flushing with clean water with addition of a surfactant.

The following is a summary of conclusions based on the testing performed at the INL WSTB:

- Results from the water treatment experiments indicate that disinfection of large volumes of water contaminated with *B. globigii* spores is feasible. All treatment units achieved at least 4-log removal of spores from the lagoon water over the course of the experiments, with some units achieving 7-log reduction. Treated water volumes ranged from 1,250 to 5,000 gallons (4,732 to 18,927 L) with experiments ranging from 5.5 hours to 1 day. It is likely that larger volumes of water may need to be disinfected in a real world scenario, but all of the tested mobile treatment systems can be scaled up, or multiple units can be put into place. Data generated from this study does demonstrate that disinfection of contaminated water in the field is more challenging than disinfecting clean drinking water due to the disinfectant demand present in real world wash water, the potential for low temperature, and disinfectant dissipation due to sunlight.

- Data collected during the crude oil contamination experiment suggest that flushing the pipe with clean water was an effective decontamination method. Benzene detected in the WSTB pipe from the oil contamination dropped below the EPA prescribed Maximum Contaminant Levels (MCLs) with clean water flushing, and no other benzene, toluene, ethylbenzene and xylene (BTEX) components were detected in the water. No total

petroleum hydrocarbons or BTEX compounds were detected on the pipe infrastructure surface in contact with the water after flushing. Surfactant was injected because it was assumed that oily components could persist in the water phase or on the infrastructure surfaces. This was not the case, but online sensor data and visual observation of foaming in the water samples indicated that surfactant may have persisted in the dead-end portions of the WSTB pipe for weeks after the initial injection. This should be taken into consideration if a surfactant is used during decontamination of a drinking water distribution system.

1.0 Introduction

The U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) partnered with the Idaho National Laboratory (INL) to build the Water Security Test Bed (WSTB) at the INL test site 50 miles (80 km) west of Idaho Falls, Idaho. The WSTB was built using an 8-inch (20 cm) diameter cement-lined drinking water pipe that was previously taken out of service. The pipe was exhumed from the INL grounds and oriented in the shape of a small drinking water distribution system (see Section 1.1 for a detailed description). Effluent from the pipe is captured in a lagoon. The WSTB can support drinking water distribution system research on a variety of topics including biofilms, water quality, sensors, and homeland security related contaminants. Because the WSTB is made of previously used drinking water distribution system pipes, research can be conducted under conditions similar to those in a real drinking water system.

EPA led the experiments described in this study with technical support from CB&I Federal Services LLC (CB&I) under contract. Testing and analyses described in this report were conducted by CB&I in accordance with the Quality Assurance Project Plan (QAPP) (Appendix A). EPA and CB&I personnel conducted two experiments:

- August 2015: Four mobile disinfection technologies were tested for their ability to disinfect large volumes of biologically contaminated "dirty" water from the WSTB. *Bacillus globigii* spores, a non-pathogenic surrogate for pathogenic *B. anthracis*, acted as the biological contaminant. The four technologies evaluated included: (1) Hayward® Saline C™ 6.0 Chlorination System (Elizabeth, NJ), (2) Advanced Oxidation Process (AOP) Ultraviolet (UV)-Ozone System, (3) Solstreme™ UV System (Cincinnati, OH), and (4) WaterStep Chlorinator (Louisville, KY).

- September 2015: The WSTB pipe was contaminated with Bakken crude oil, and decontamination was performed using flushing with clean water and addition of a surfactant (SURFONIC® DOS-75PG, Huntsman Corporation, The Woodlands, TX).

1.1 WSTB Description and Setup

The WSTB consists primarily of an 8-inch (20 cm) diameter drinking water pipe oriented in the shape of a small drinking water distribution system. The WSTB contains ports for simulating water demands from service connections and a 15-foot (5 m) removable coupon section designed to sample the pipe interior. Figure 1 schematically depicts the main features of the WSTB.

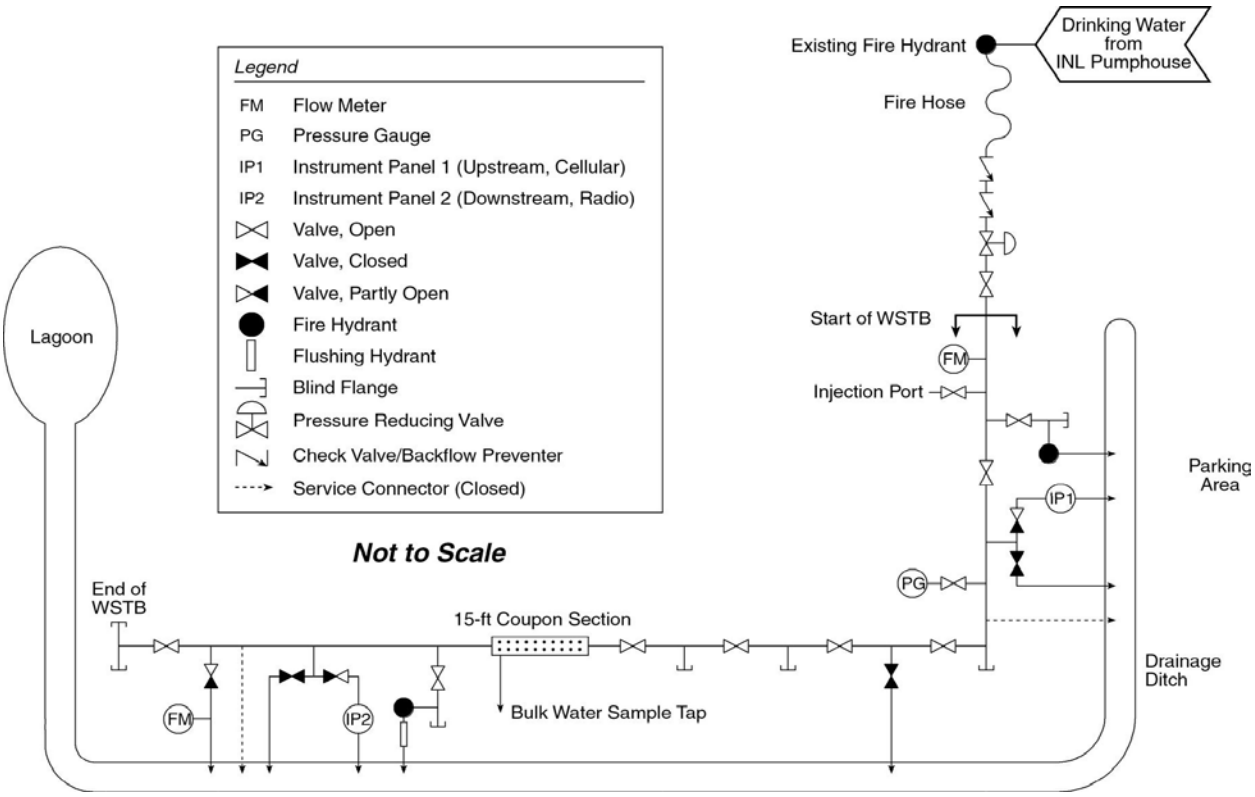

Figure 1. Schematic overview of Water Security Test Bed.

Figure 2 shows the aerial view of the WSTB. The lower right corner shows the upstream and system inlet; the upper left corner shows the lagoon.

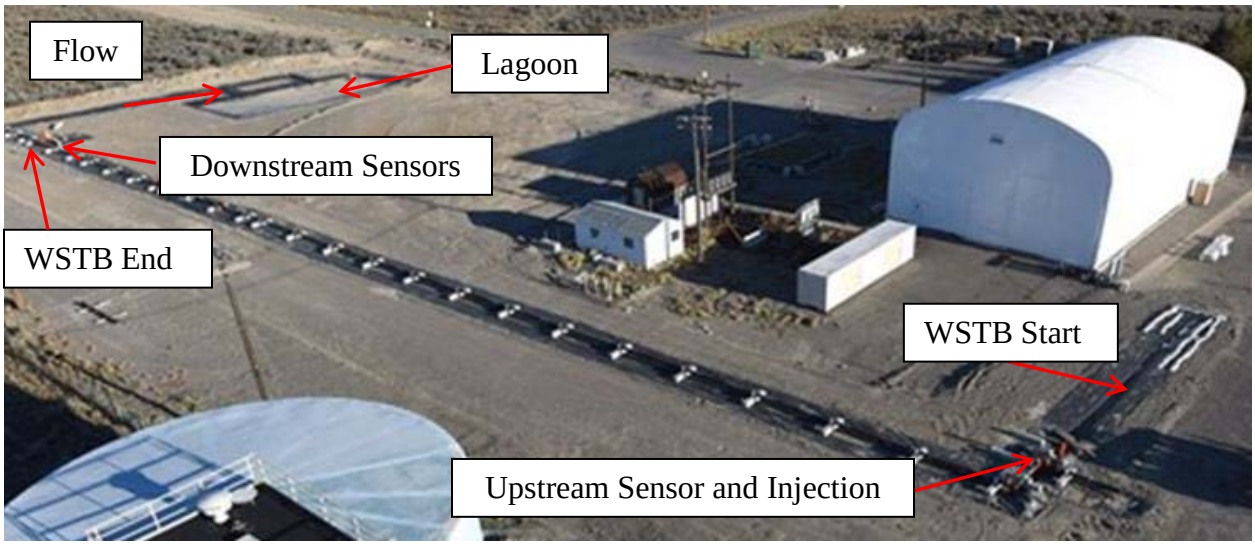

Figure 2. Aerial view of the Water Security Test Bed.

As depicted in Figure 1, drinking water was supplied to the WSTB through an existing fire hydrant.

The drinking water was chlorinated ground water that also supplied the surrounding INL facilities. The WSTB incorporates approximately 448 ft (137 m) of 8 inch (20 cm) diameter cement-lined pipe. The 8 inch (20 cm) pipe system is constructed directly over the lined drainage ditch for spill/leak containment (as shown in Figure 2). The total volume of the WSTB is estimated to be ~1,150 gallons (4,353 L). The valve near the end of WSTB along with the flow meter (shown in Figure 3) was used to regulate and maintain flow.

Figure 3. Water Security Test Bed system flow regulator.

The water from the WSTB system is discharged to a lagoon (Figure 4) which has a water storage capacity of 28,000 gallons (105,980 L).

Figure 4. Water Security Test Bed discharge lagoon.

Water from this lagoon was used for the studies on four disinfection technologies to determine their ability to treat large volumes of biologically contaminated water. Figure 5 shows a schematic layout (**not to scale**) of the test setup for the four large volume water treatment technologies. The four technologies used were EPA's Advanced Oxidation Process (AOP) trailer unit, the Solstreme UV system, the WaterStep chlorinator and the Hayward chlorinator. These devices and experimental protocols are described further in section 2.0.

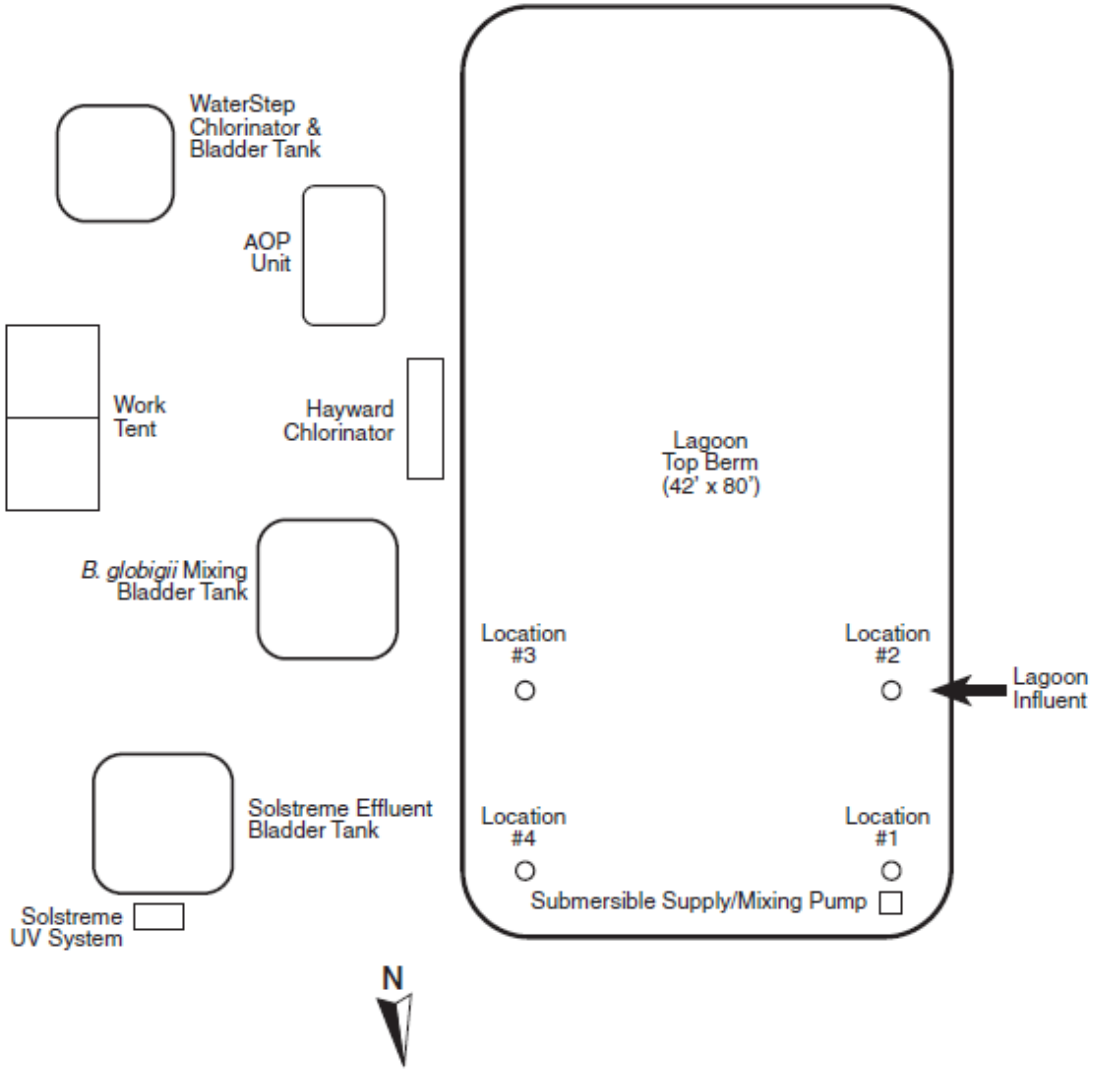

Figure 5. Schematic layout for large volume water treatment technologies testing.

The crude oil experiments used a positive displacement pump to inject the prepared stock contaminant (i.e., subnatant representing the miscible portion of the crude oil) at the beginning of the 448 ft (137 m) WSTB system. The stock was prepared in accordance to the procedure described in the QAPP (Appendix A). Additional information is also presented later in this report. Figure 6 shows the crude oil injection setup.

Figure 6. Prepared crude oil subnatant for Water Security Test Bed injection.

The bulk water samples (BWSs) and coupon samples were taken from the 15-foot (5 m) polyvinyl chloride (PVC) pipe-segment designed and fabricated to contain 10 sets of duplicate removable coupons (totaling 20 coupons) made from cement-lined pipe used to construct the rest of the WSTB. The coupons allow for the measurement any contaminant persistence on pipe material, and the effectiveness of decontamination. Figure 7 shows a portion of the 15-foot (5 m) PVC coupon section.

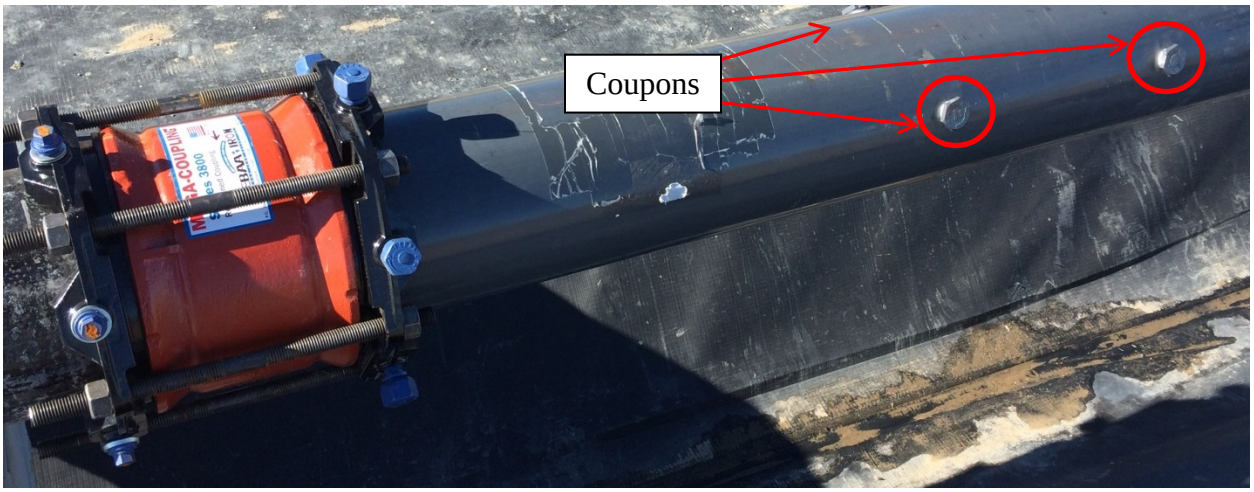

Figure 7. Removable 15-foot PVC coupon section.

The pipe material for the 20 small coupons (22/32 of an inch [1.8 cm] in diameter and 0.371 square inches [2.4 square centimeters] in area) were cut from the cement mortar-lined iron pipe obtained from INL and set into threaded plugs that were inserted into the PVC-coupon section of the pipe. Figure 8 shows a picture of the threaded coupon that was inserted into the pipe main. The twenty coupons were individually numbered CP-0/CP-0D through CP-9/CP-9D in duplicate (CP = coupon, D = duplicate).

Figure 8. Extracted pipe coupon.

2.0 Description of Experiments

2.1 Disinfection of Large Water Volumes

This experiment was designed to assess the ability of a portable disinfection unit to treat a large volume of water containing *B. globigii* spores. Water in the lagoon contained dirt and sediment from the surrounding area, as well as algae. The dirt and algal growth created disinfectant demand in the water and rendered the water "dirty." The following four treatment technologies were evaluated for their ability to treat dirty water from the lagoon: (1) Hayward Saline C™ 6.0 Chlorination System, (2) AOP UV-Ozone System, (3) Solstreme™ UV System, and (4) WaterStep Chlorinator. The test equipment was placed adjacent to the WSTB lagoon. A schematic layout of the tested systems was presented previously in Figure 5.

The effectiveness of individual treatment technologies was evaluated by sampling water containing *B. globigii* spores before it entered the individual treatment technology or before treatment began, and then after disinfection to determine the treatment effectiveness. The concentration of spores in the influent (or before treatment began) was then compared to the concentration in the effluent (after treatment). For experiments with the AOP trailer and Solstreme, water was pumped from the lagoon into a 2,000 gallon (7,571 L) bladder tank system that contained a mixing pump to provide a continuous stream of *B. globigii* spores in contaminated water (Figure 9).

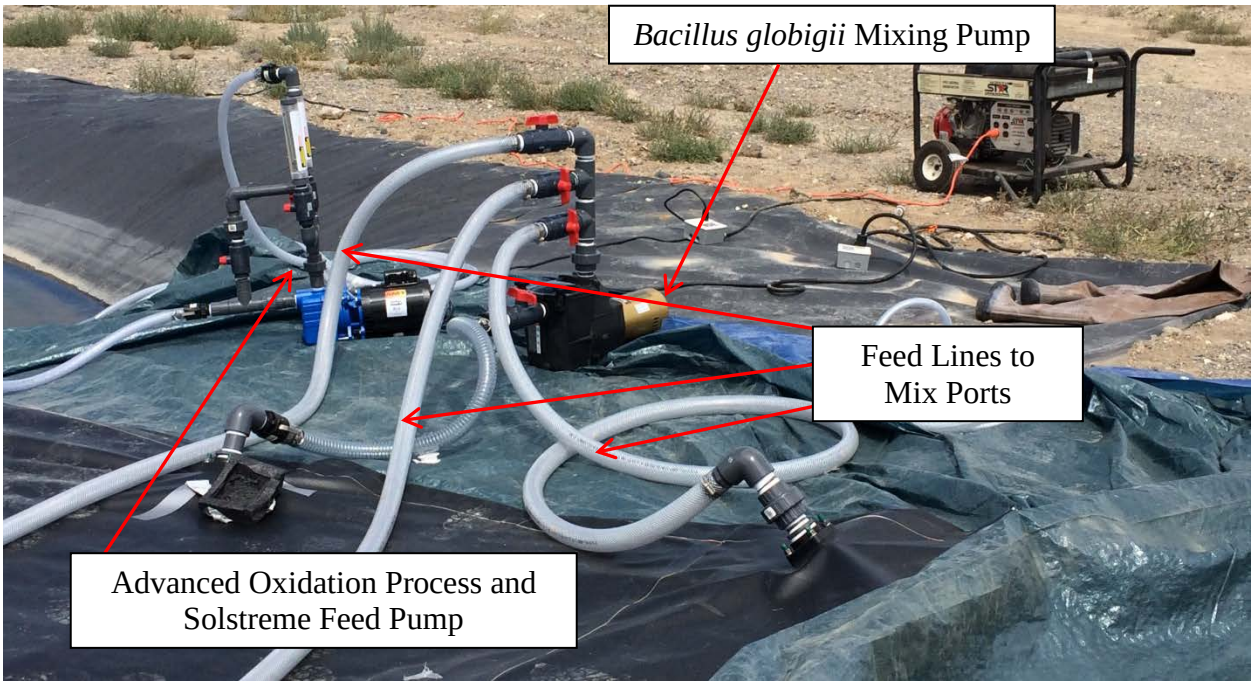

Figure 9. Inlet bladder tank and mixing.

Figures 10 shows a schematic depiction of how the mixing pump was connected to the bladder to perform mixing along with the inlet and outlet ports.

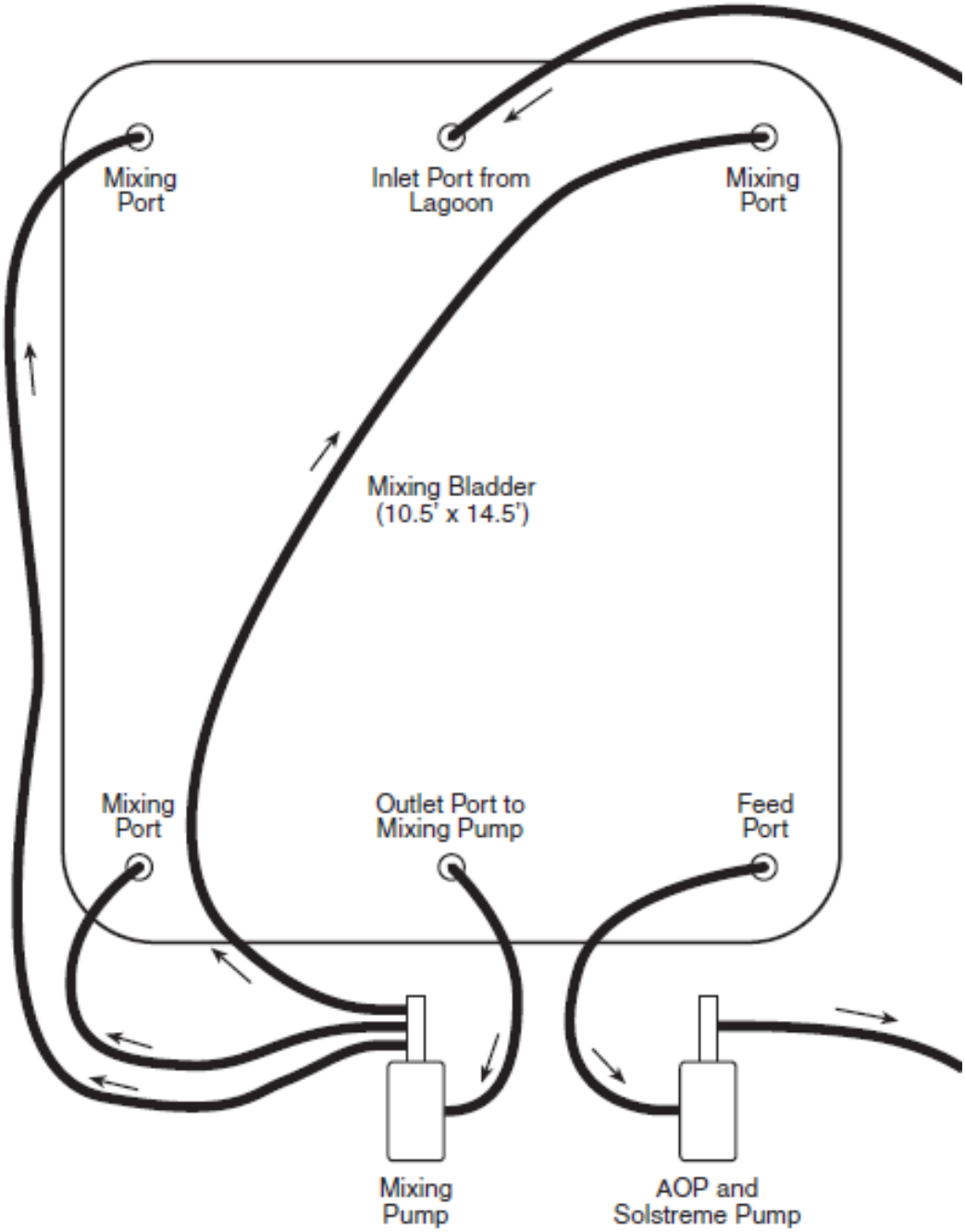

Figure 10. Schematic depiction of the inlet bladder tank mixing process.

For the AOP trailer and Solstreme unit, a target inlet concentration of greater than 10^6 spores/100 mL (or 10^4 spores/mL) was prepared using the inlet bladder tank and mixing pump shown in Figure 9. The water was then pumped through the selected treatment unit. Each unit was tested for 5.5 hours. Pre-treatment and post-treatment water samples for *B. globigii* analysis were collected at the same time.

For the WaterStep, a 1,250 gallon (4,732 L) vendor supplied bladder tank was spiked with *B. globigii spores* (10^6 spores/100 mL or 10^4 spores/mL), and then filled with lagoon water. The

bladder tank was manually agitated by pushing on its side to mix the spores. Manual agitation took place approximately every 15 minutes throughout the experiments. Before disinfection, the bladder tank was sampled to determine the initial spore density, and then the chlorination started. Subsequent samples were considered as treated, or disinfected, water samples.

As in the case of the WaterStep unit, the Hayward Saline C™ 6.0 Chlorination System also did not use the inlet/outlet bladder tank system for operation. It is an in-situ type of treatment technology where the salt used for generating the chlorine comes from the same contaminated "pool" or source of water. The testing protocol for this treatment device took place in the lagoon and is described in Section 2.1.4.

2.1.1 EPA AOP Trailer Testing

On August 17, 2015, the first large volume disinfection study using the EPA AOP system was performed. The system setup is depicted in Figure 11, where it has been removed from its transport trailer.

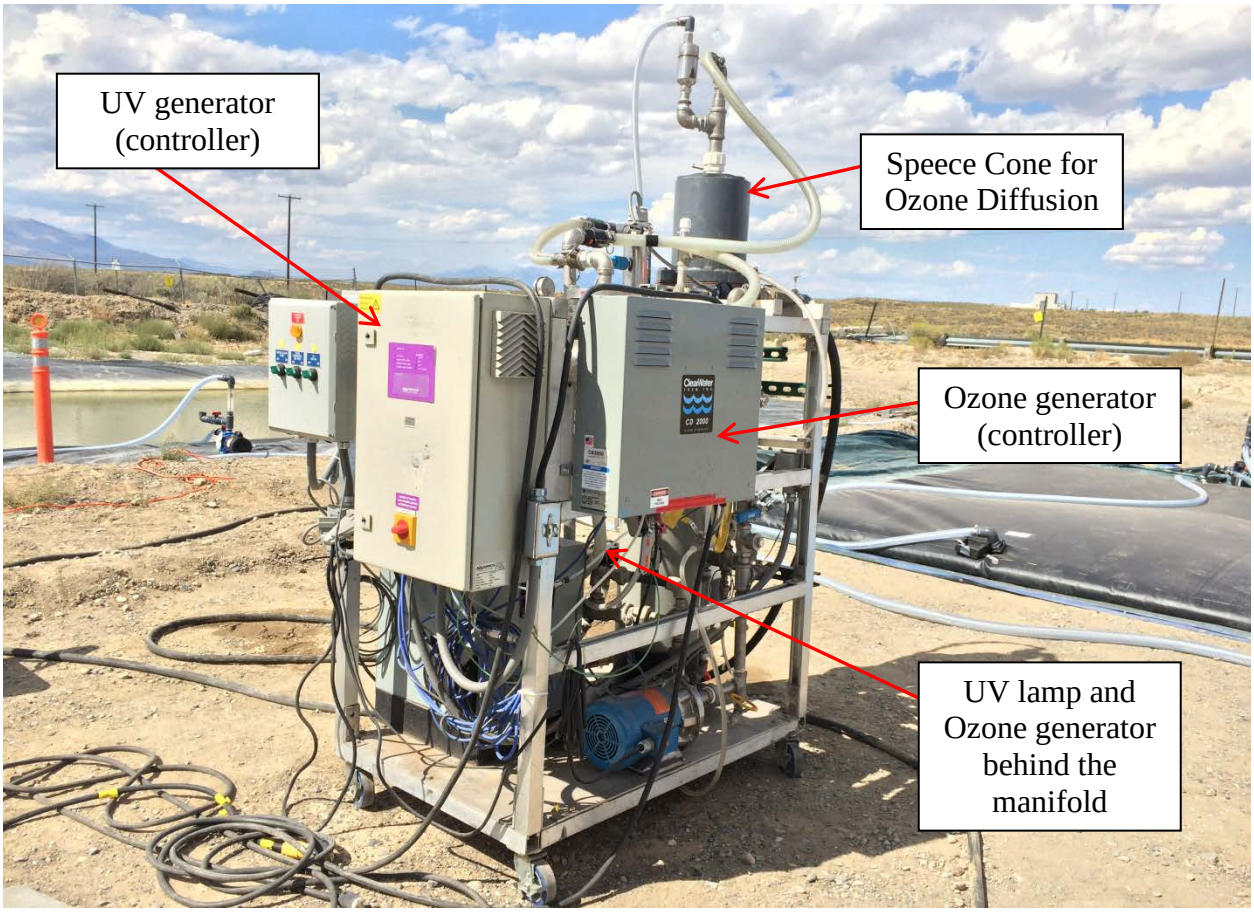

Figure 11. Advanced Oxidative Process System and influent/mixing bladder tank (black object to the right of the system).

The AOP system was custom-built at the EPA Test and Evaluation (T&E) Facility in Cincinnati, Ohio. The AOP system consists of four major components: the Power Prep 66 (air preparation

unit), CD2000 (ozone production unit), Trojan UVMax (UV generation unit), and the Aquionics UV (UV generation unit). During this study, the AOP system was operated with the CD2000 ozone generator and the Aquionics UV system operated in series. The Trojan UVMax unit was not used during this study. UV light and ozone act individually as disinfectants, but photolysis of ozone by UV light can lead to the formation of highly reactive hydroxyl radicals (•OH) through multiple mechanisms. The •OH is a short lived but potentially potent disinfectant.

The bulk water samples (BWSs) for *B. globigii* concentrations (BWS-0 through BWS-6) were collected from the inlet and outlet of the system simultaneously using the grab sampling technique in 100-mL sterile sample bottles with a 10 mg sodium thiosulfate tablet. The BWS sampling ports at both inlet and outlet of the system were opened and the water was drained for 15 seconds prior to collection of the sample. The AOP system was powered by a portable generator that had to be shut down for refueling twice during the 6 hour sampling period.

2.1.2 Solstreme™ UV System Testing

On August 18, 2015, the large volume disinfection study using both Solstreme™ UV system, was performed. The Solstreme™ UV system setup is depicted in Figure 12.

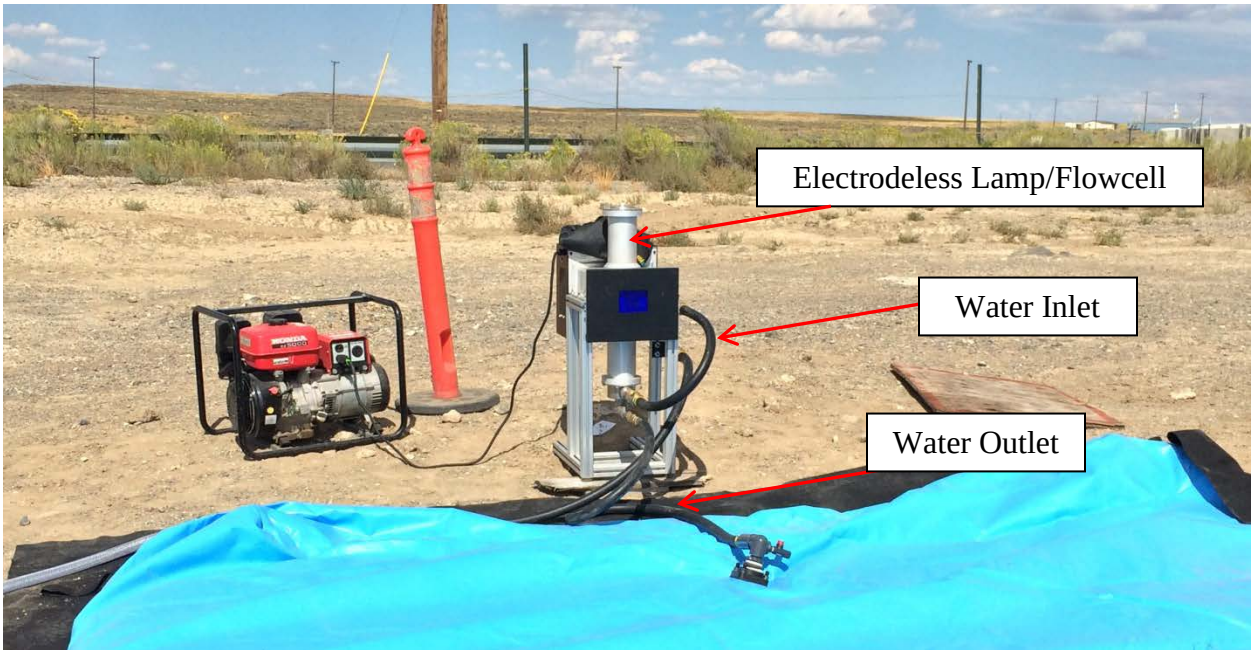

Figure 12. Solstreme™ UV System and effluent bladder tank (blue object in front of the UV system).

The Solstreme™ UV system uses a patented microwave-actuated electrodeless lamp technology to provide UV disinfection. The microwave is generated using a focused magnetron which activates UV energy inside the patented-electrodeless lamp. A typical UV lamp uses an electrical current passing through electrodes to excite the lamp to produce UV light; the Solstreme UV lamp uses radio frequency (RF) energy to induce the lamp to produce UV light through a quartz glass envelope. The electrodeless lamps can be run at higher power levels allowing it to produce greater

amounts of UV light than its counterpart electrode-based lamps. The National Sanitation Foundation (NSF) Standard 55 "Class A" Rated UV systems are required to operate at a minimum UV light dosage of 40 mJ/cm² (or 40 mW-sec/cm^2) (USEPA, 2003). The Solstreme system in comparison is expected to generate a higher level of UV dose compared to an equivalent electrode-based UV lamp. The manufacturer expects the Solstreme system operating under optimal conditions can deliver an equivalent total dosage of up to 1,700 mW-sec/cm^2 (NeCamp, 2008). However, the design of the instrument made it impossible to verify the dosage.

Similar to the AOP System, the BWS for *B. globigii* concentrations (BWS-0 through BWS-6) were collected from the inlet and outlet of the system simultaneously using the grab sampling technique in 100-mL sterile sample bottles with a 10 mg sodium thiosulfate tablet. The BWS sampling ports at both inlet and outlet of the system were opened, and the water was drained for 15 seconds prior to collection of the sample.

2.1.3 WaterStep Chlorinator Testing

On August 18, 2015, concurrent with the Solstreme™ UV System the WaterStep Chlorinator was tested. The system setup is depicted in Figure 13.

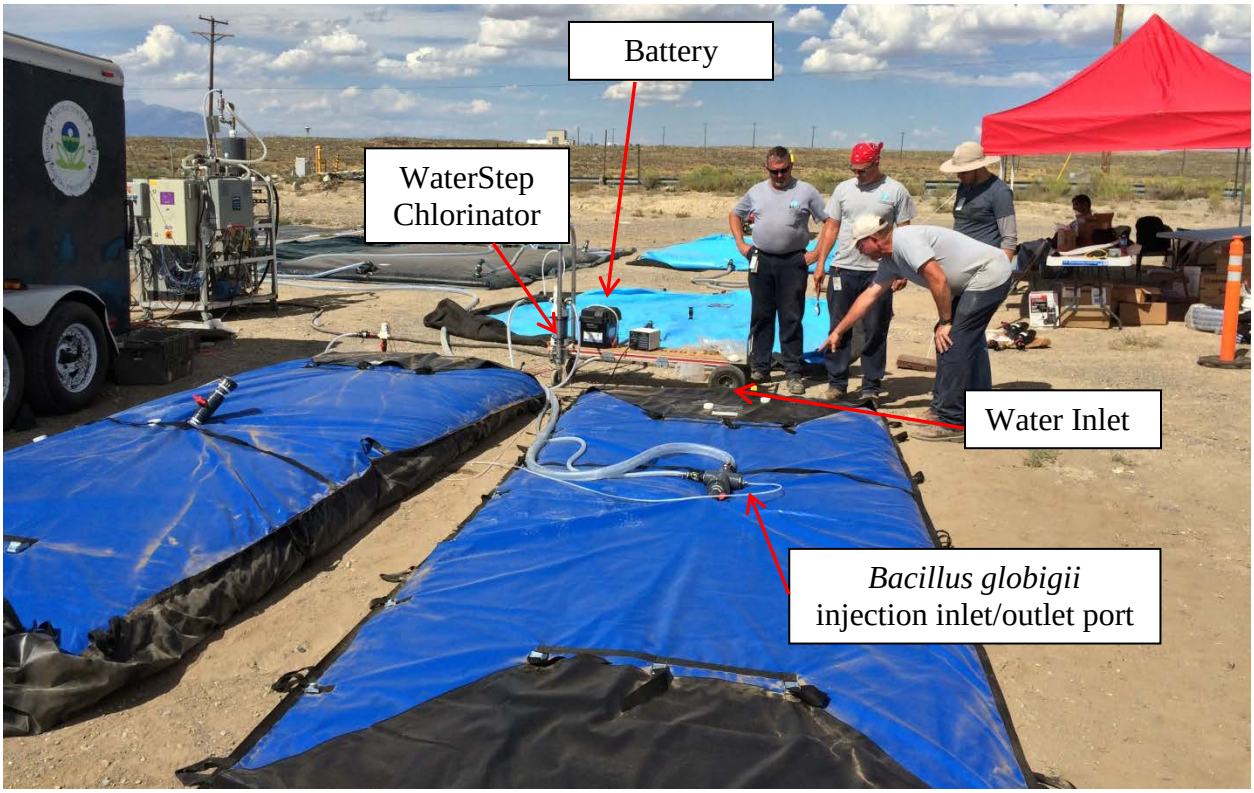

Figure 13. WaterStep Chlorinator System bladder tanks (dark blue).

The WaterStep (WaterStep, 2013) system uses electricity and sodium chloride (table salt) to generate chlorine to disinfect water. This occurs by applying a potential to a cell that contains electrolytic plates (an anode and cathode). Chlorine gas is formed at the anode, which forms free chlorine when dissolved in water ("free chlorine" is a mixture of hypochlorous acid and

hypochlorite ion, depending on pH). This free chlorine migrates into a 1,250 gallon (4,732 L) bladder tank where it can disinfect the contained water. The system was operated using a 12 volt DC battery on a cart (as shown in the middle of Figure 13). The battery was placed on a trickle charger to maintain full charge for operational stability during the testing. The WaterStep Chlorine generator setup is depicted on Figure 14.

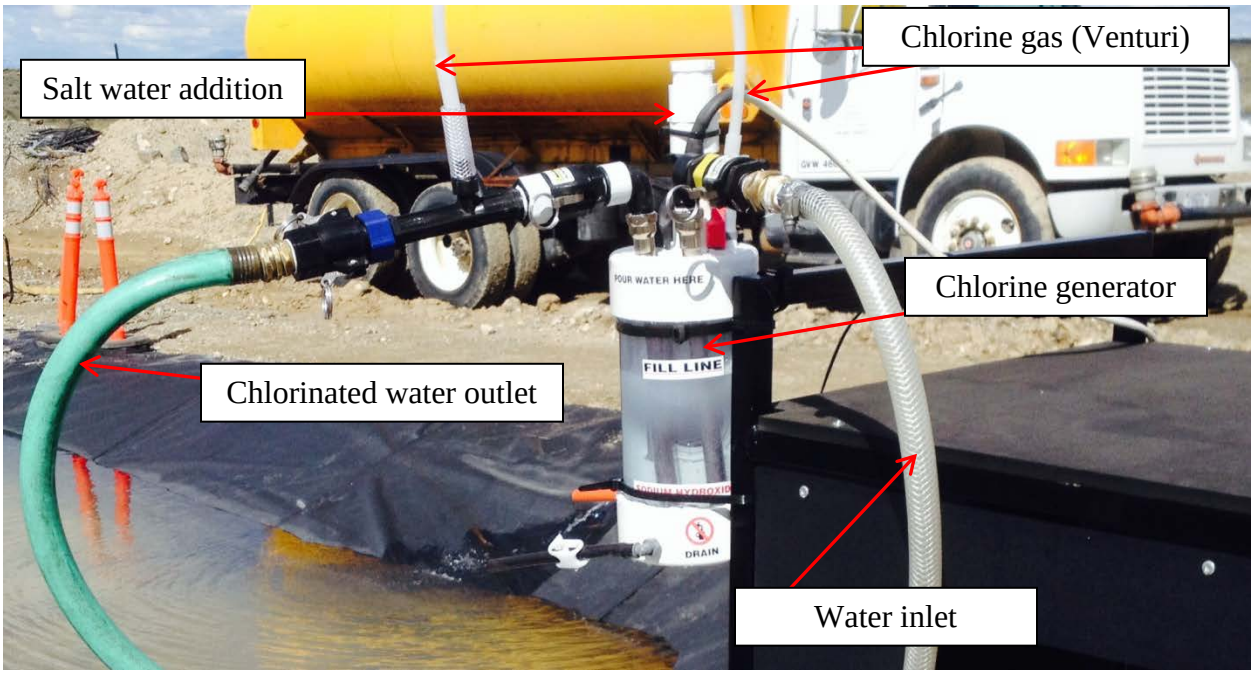

Figure 14. WaterStep Chlorine Generator components.
Note: image is from a previous experiment. It is presented here for illustration purposes only.

BWSs for *B. globigii* concentrations (BWS-0 through BWS-5) were collected from the same sampling port that served as both inlet/outlet of the system using the grab sampling technique in 100-mL sterile sample bottles with a 10 mg sodium thiosulfate tablet. The BWS sampling port was opened and the water was drained for 15 seconds prior to collection of the sample.

2.1.4 Hayward Saline C™ 6.0 Chlorination System Testing

The Hayward Saline C™ 6.0 Chlorination System is an *in-situ* type of treatment technology, and it was operated using the lagoon as the "pool" or source of water. The Hayward unit generates free chlorine using the same principle as the WaterStep, with free chlorine being generated from dissolved salt in water. A potential is applied to a cell that contains electrolytic plates (an anode and cathode). Chlorine gas is formed at the anode, which forms free chlorine when dissolved in water (a mixture of hypochlorous acid and hypochlorite ion, depending on pH). Flow moves through the chlorine generating cell, and dissolved free chlorine leaves the cell in the effluent (Hayward, 2013). The Hayward system as configured during the testing is shown in Figure 15.

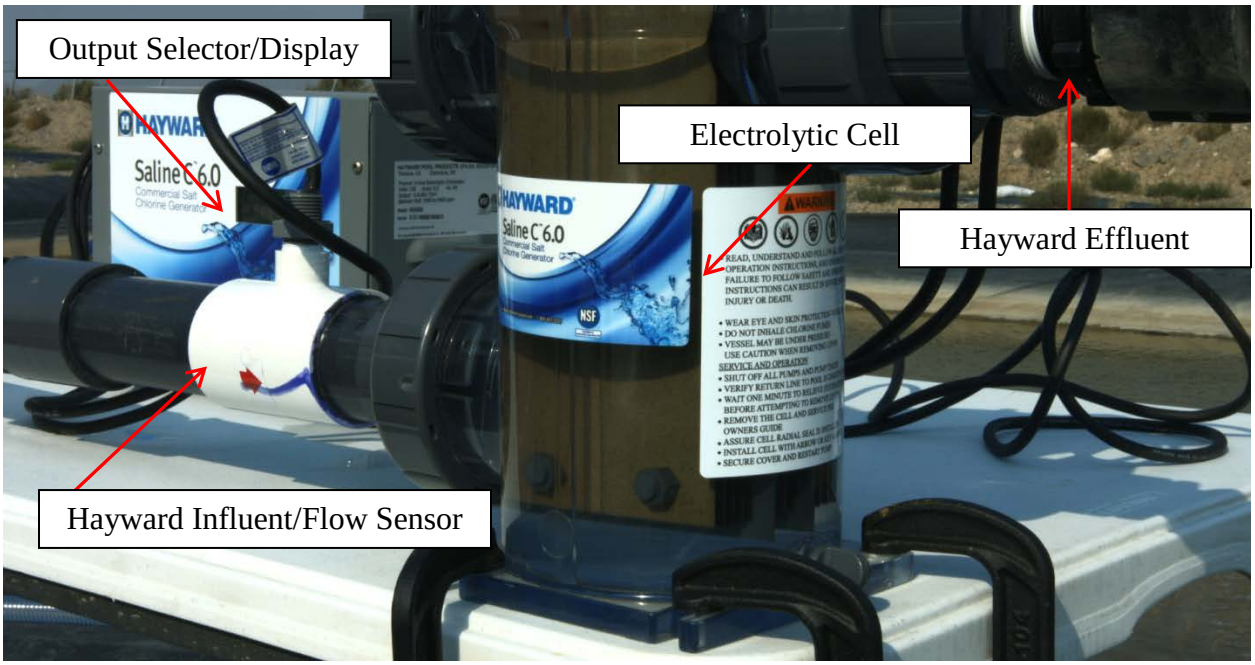

Figure 15. Hayward Chlorine Generator.

The manufacturer recommends 3,500 mg/L to 5,000 mg/L salt to be added to the pool for operations. On August 18, 2015 (the day before this system was tested), the lagoon was mostly drained and approximately 126 lbs (57 kg) of salt was added to the lagoon near the water inlet from the WSTB pipe. The water from the WSTB was then run at 5 gpm (19 L/min) for approximately 16 hours (releasing 4,800 gallons [18,170 L]) to mix the undrained water with and dissolve the salt in the lagoon. In total, it is estimated that approximately 5,000 gallons (18,927 L) of water was in the lagoon after filling. The overall Hayward system setup is depicted in Figure 16.

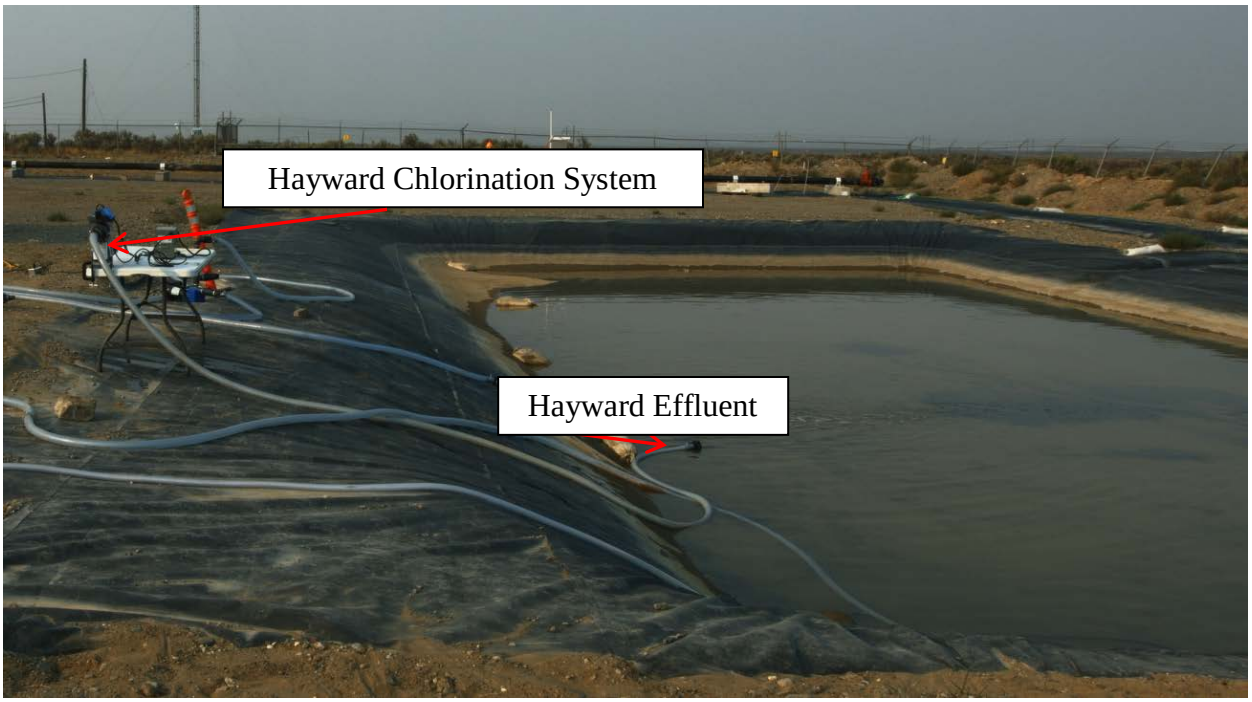

Figure 16. Hayward Saline C™ 6.0 Chlorination System setup on a table.

On August 19, 2015, the large volume disinfection study using the Hayward system was initiated. At 9:10 AM, 17 L of *B. globigii* stock solution were added to the lagoon to reach a target concentration of greater than 10^6 spores/100 mL (or 10^4 spores/mL) in the lagoon. Figure 17 shows the addition of *B. globigii* to the lagoon simultaneously at multiple locations.

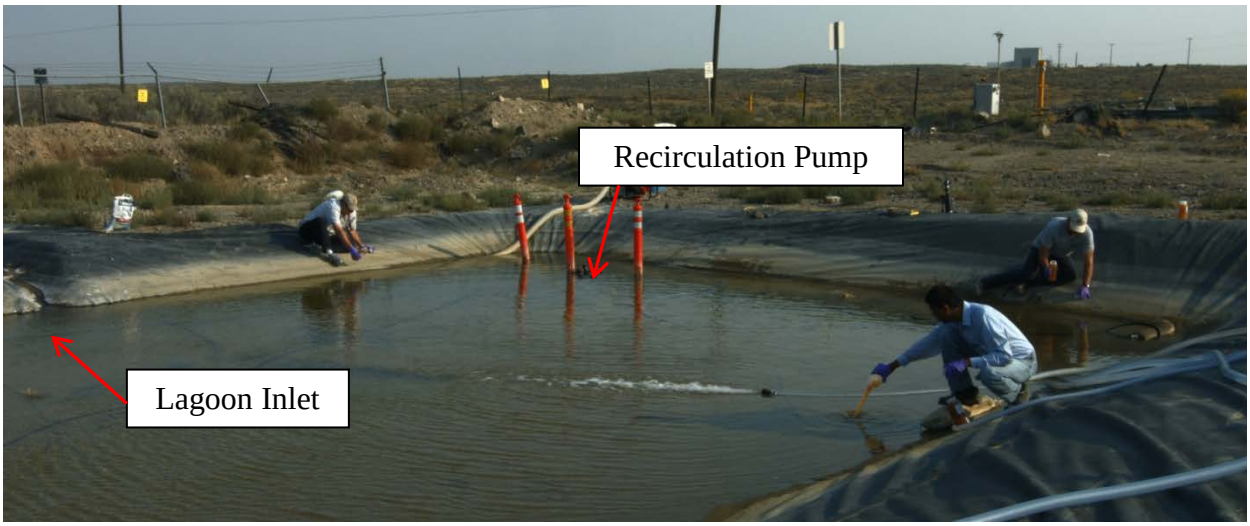

Figure 17. Contamination of the lagoon with *Bacillus globigii.*

A sump pump with a distribution manifold was used to recirculate the lagoon water and to provide mixing for the *B. globigii* stock in the lagoon (shown in Figure 18).

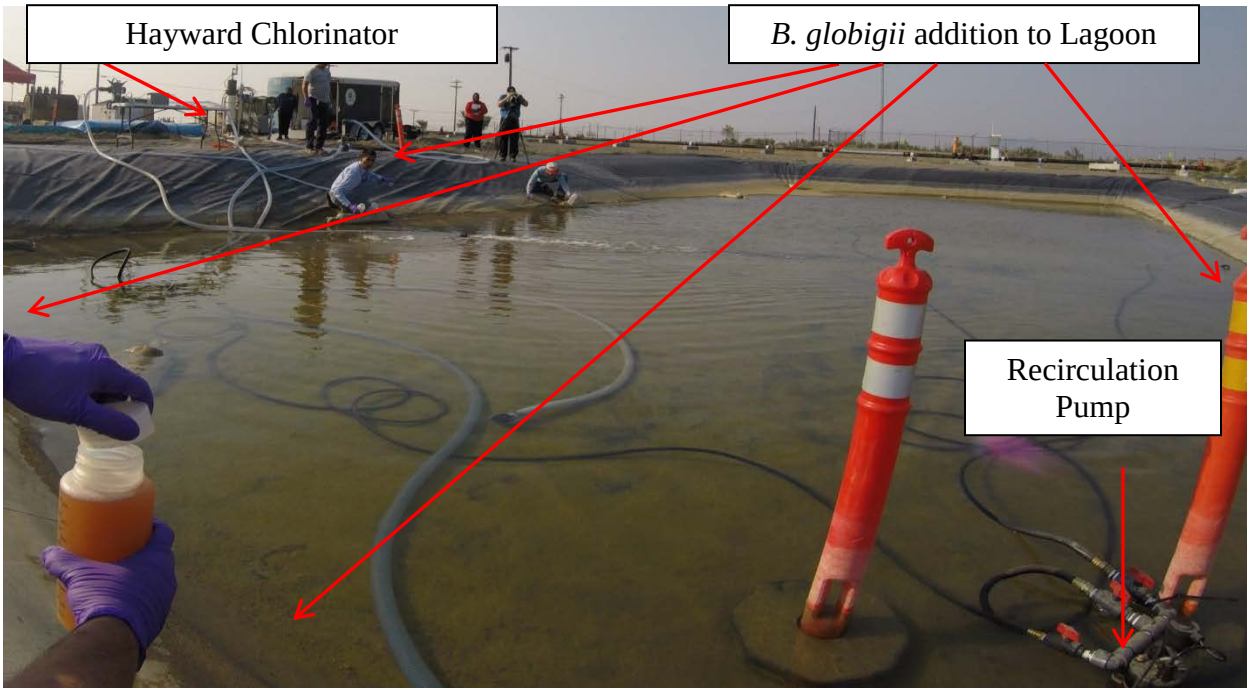

Figure 18. Lagoon *Bacillus globigii* recirculation/mixing pump.

At 10:00 AM, the initial, pre-disinfection *B. globigii* samples were collected from the four corner locations around the lagoon where water was pooled. Thereafter, the Hayward system was started. The amount of chlorine generated (i.e., output) of the system varies depending upon available salt in the water flowing through the system. The output is adjustable from 0 to 100% of the systems rated capacity and is displayed as % output on the display.

During chlorination of pool water under typical usage of the system, the amount of chlorine generated is automatically controlled based on the salt levels and automated measurement of chlorine levels in the water using a chemical controller feedback system. The tested field system was not equipped with a chemical feedback controller and was instead operated in manual mode. In manual mode, when the available salt falls below the level required for the set output level in %, the system stops generating chlorine and the LCD display flashes “LO SALT” (Hayward, 2013). A low salt alarm indicator came on as soon as the system was started at 100%. An additional bag of salt was added to bring the total salt added to ~154 lbs (70 kg) of salt. However, the low salt alarm remained. In accordance with the vendor manual (Hayward, 2013), the system was reset, and the output selector was lowered to 50% and stepped up in increments of to a final setting of 60% setting which was found to be stable for operation. This setting was used to run the system for the remainder of the test.

The system was operated at the manufacturer recommended rate of 40 gpm (18 L/min)) flow through the electrolytic cell that produced chlorine, and the chlorinated water was pumped back to the lagoon. Chlorine levels coming out of the Hayward chlorinator and the lagoon were monitored throughout the day. The free chlorine coming out of the Hayward chlorination cell was measured to be in the range of 4.3 mg/L. The chlorine level in the lagoon crept up slowly starting at 0.2 mg/L at noon, 0.61 at 1:00 PM, 1.07 at 3:00 PM and 1.19 at 3:30 PM. Based on the rate at which the

chlorine level was increasing, it was decided that the unit would be left to run unattended overnight.

The BWSs for *B. globigii* effluent concentrations (BWS-0 through BWS-5) were collected from the lagoon periodically throughout the day using the grab sampling technique in 100 mL sterile sample bottles with a 10 mg sodium thiosulfate tablet. The following day on August 20, 2015, at 8:30 AM, the final BWS sample was collected and the chlorine from the lagoon was measured to be 12.2 mg/L. At time of arrival at the site, it was noted that while the Hayward pump was still operating at 40 gpm (151.4 L/min), the low salt alarm was active. It is unknown when the salt activation would have stopped. However, the measured value of 12.2 mg/L of chlorine was sufficient to achieve inactivation *B. globigii* spores in the lagoon.

2.2 Crude Oil Contamination/Decontamination Tests

These experiments involved contamination of the WSTB using crude oil and the subsequent decontamination of WSTB using flushing at 15 gpm (56.8 L/min) followed by an injection of a surfactant. The contamination/decontamination experiment consisted of the following main steps:

- Step 1 – Pipe conditioning (cultivation of biofilm)
- Step 2 – Instrumentation panel setup, effluent oil capture treatment train, and background sampling
- Step 3 – Preparation of contaminant stock (subnatant, miscible portion of Crude Oil) and injection into the WSTB
- Step 4 – Preparation of decontaminant and decontamination using flushing along with a surfactant for crude oil removal,
- Step 5 – Post-decontamination flushing, reconditioning, and monitoring

Step 1 – Pipe conditioning (cultivation of biofilm)
Biofilm cultivation and pipe conditioning occurred by passing INL tap water through the WSTB continuously starting May 2015 until the late-September/early October 2015 contamination and decontamination testing. After initial flush to remove any debris at startup in May 2015, the flow rate was set at 2.5 gpm (9.5 L/min) during the conditioning period with a total discharge of 25,200 gallons (95,392 L) per week to the lagoon. This flow rate allowed for weekly trucking and disposal of the accumulated discharge.

Step 2 – Instrumentation panel setup, effluent oil capture treatment train, and background sampling
Instrument Panel Setup – The initial upstream/downstream instrument panel setup was completed in May 2015. In August 2015, a Turner Designs Hydrocarbon device (Model TD1000C), which measures oil in water was installed at the downstream sensor location (shown in Figure 19). The TD1000C is an online "hydrocarbon in water" monitor that detects aromatic hydrocarbons in water using fluorometry principles in combination with a proprietary flow cell (Turner, 2009).

The WSTB upstream/downstream instrumentation panels are also equipped with online sensors that continuously measure two basic water quality parameters: free chlorine and total organic carbon (TOC). Each of the instrumentation panels contains one Hach® CL-17 chlorine analyzer (Loveland, CO) and one RealTech M4000 TOC analyzer. The Hach CL-17 chlorine analyzer uses colorimetric N,N-diethyl-phenylenediamine (DPD) chemistry to monitor water continuously for

free chlorine (Hach, 2014). The RealTech M4000 uses the UV 254 nanometer wavelength (i.e., UV254) absorption measurement for determining the TOC content (RealTech, undated). UV254 instruments are often used as an inexpensive indicator of TOC in water. UV254 measurements are known to have some bias towards aromatic organics; however, they are relatively inexpensive to maintain and operate when compared to the traditional UV-persulfate based TOC analyzers.

Figure 19. Turner TD1000C Oil in Water Monitor.

Effluent Oil Capture Treatment Train – A carbon-based effluent oil capture treatment train was designed and implemented at the downstream location of the WSTB. The dual-drum treatment train capture (adsorbent) media contained a media mix of 30% TIGG oil removal media and 70% of TIGG 5DC 1240 NFS coconut-based activated carbon. Only one 55 gallon (208 L) drum of carbon was required to reduce the volatile organic compounds (VOC) of concern (benzene) to a concentration to below the targeted drinking water MCL values. Additional drums were added to the treatment train to accommodate operating flow rates, operating pressure, and increase the empty-bed contact time. In total, the effluent oil capture treatment train was comprised of four drums. Two drums were connected in series and the flow split evenly between each set of two-drums. During the surfactant decontamination step, the first drum in each set of drums was taken offline. This was done to prevent the potential release of the captured oil in the first drum. Figure 20 shows the overall oil capture treatment train with cam-lock connects to put individual drums in series and/or to take them offline as needed.

Figure 20. Effluent oil capture treatment train.

Background Sampling – Prior to initializing the contamination Step (Step 3), on September 21, 2015 at 8:30 AM, bulk water samples (BWS-0) and Coupon Samples (CP-0 and CP-0D) were collected to establish background levels. The BWS-0 sample was analyzed for background crude oil components such as VOCs, benzene, toluene, ethylbenzene, and xylene (BTEX), gasoline range organics (GRO), diesel range organics (DRO), and oil range organics (ORO). The coupon sample (CP-0D) was analyzed for biofilm density using heterotrophic plate count (HPC). And the CP-0 was analyzed for crude oil components along with the BWS-0 sample. Free chlorine (CL-F-#) was also measured periodically during the testing. All sampling activities related to crude oil testing are summarized in Table 1 and analytical methods are described in the QAPP (Appendix A).

Step 3 – Preparation of contaminant stock (miscible portion of Crude Oil) and injection into the WSTB

Preparation of Crude Oil Contaminant Stock – The crude oil for this study was obtained from Marathon Petroleum Corporation. The oil procured was from the Bakken shale in North Dakota. On September 20, 2015 at 16:05 PM, a measured amount of crude oil (2.5 liters) and Snake River water (22.5 liters) was mixed in a 25 liter carboy (shown in Figure 21). The detailed preparation methodology is documented in the QAPP (Appendix A).

Figure 21. Crude oil injection stock preparation.

On September 21, 2015, at 8:40 AM, 20 liters of the mixed water was drawn from the bottom of the carboy spigot into a 5 gallon (19 L) bucket for injection.

Prior to the contamination step, the Effluent Oil Capture Treatment Train was connected to the WSTB, and the flowrate was increased to 15 gpm (56.8 L/min). However, the increase in flow resulted in an increase in line pressure into the carbon drums above the levels recommended by the manufacturer. Therefore, the WSTB system flowrate was reduced to 13 gpm (49.2 L/min) during the contamination/decontamination step and evenly split between the two 2-drum effluent oil capture treatment trains.

Contamination Test Protocol – On September 21, 2015, at 9:03 AM, the crude oil suspension was introduced into the WSTB using a positive displacement pump. As indicated previously in Step 2, the oil capture system was designed to contain any crude oil component from entering the lagoon. Once flushing and decontamination activities were completed, the unit was disconnected.

During the injection, initially the WSTB was operated at 15 gpm (56.8 L/min) (adjusted to 13 gpm [49.2 L/min] as mentioned previously) for approximately 1 hour. In accordance with the QAPP (Appendix A), the injection duration was one hour so that there was a contact of one hour after the bolus of crude oil suspension reached the coupon section of the pipe. All sampling activities related to crude oil testing are summarized in Table 1.

Step 4 – Preparation of decontaminant and decontamination using flushing along with a

surfactant for crude oil removal

Preparation of Decontaminant Agent Stock – The surfactant Surfonic TDA-6 was identified as the decontaminant of choice based on pilot-scale decontamination experiments at EPA's Test and Evaluation (T&E) Facility (U.S. EPA, 2008). However, CB&I's technical discussions with the sales/technical representatives of the Huntsman Chemical Corporation (conducted over several weeks) led to the identification of SURFONIC® DOS-75PG as a better choice. Specifically, because this surfactant is derived from a naturally occurring material and is non-toxic to the marine environment when released. Additionally, SURFONIC® DOS-75PG surfactant has been shown to undergo 90% to 98% biodegradation in 11 to 17 days (Appendix C – Technical Bulletin SURFONIC® DOS-75PG Surfactant).

The previous flushing tests using the Surfonic TDA-6 were performed using a 5% Surfonic TDA-6 solution (U.S. EPA, 2008). The technical bulletin (Appendix C) property specifications also suggest using the product as a 5% solution. At the time of testing, Huntsman was only able to ship 3 gallons (11.3 L) of the surfactant. For the one-hour flushing event, a flow rate of 15 gpm (56.8 L/min) would generate 900 gallons (3,407 L) of water. If the entire available stock of the surfactant was used, it would only result in a 0.3% (3 gallon/900 gallons [11.3 L/3,407 L]) solution for cleansing. Therefore, for the purposes of the decontamination step, the surfactant was pumped through the pipes at the rate of 0.05 gpm (0.19 L/min) without dilution for one hour, which used up the available surfactant stock.

Decontamination Test Protocol – Once the crude oil injection was stopped, the WSTB was flushed for 2 hours at 13 gpm (49.2 L/min) between 10:00 AM and 12:00 PM. This flushing without surfactant was conducted to generate data on whether flushing alone removes crude oil subnatant from the water and pipe surfaces. Following the 2 hour flush at 12:00 PM, an attempt was made to pump the undiluted surfactant at the aforementioned rate of 0.05 gpm (0.19 L/min) using a positive displacement gear pump. This was not successful due to the high viscosity of the surfactant.

An alternate method of surfactant introduction was devised by isolating the valves close to the injection area and temporarily depressurizing the injection pipe section to manually introduce the surfactant in individual pulses every 15 minutes (three times). This pulsed introduction of surfactant was performed between 1:00 PM and 1:45 PM. The surfactant had to be diluted in 50 liters of water to allow for pouring through a funnel into the depressurized pipe section with the 2 inch connection. A total of 2 gallons (7.6 L) of the surfactant was introduced during this process. The plan to hold the surfactant stagnant in the pipe overnight was abandoned because of the pulsed injection. The water flow out of the WSTB was reduced to 5.0 gpm (19 L/min) at 4:15 PM to reduce the volume of water going to the lagoon overnight. All sampling activities related to crude oil testing are summarized in Table 1.

Step 5 – Post-decontamination flushing, reconditioning, and monitoring
Additional BWSs and CPs were collected. The sampling activities are described in Table 1.

Table 1. Crude Oil Contamination/Decontamination Related Sampling Activity

Sample ID	Sample Description	Estimated Timeline & System Flow
Step 2 – Background		
BWS-0 (Control)	• Collected sample at 8:30 AM prior to injection of crude oil.	September 21, 2015 Flow at 2.5 gpm (9.5 L/min)
CP-0 and CP-0D	• Collect at the same time as BWS-0 • After sampling the flow was increased. At 9:05 AM the flow was found to be at 11 gpm (4.16 L/min). The pressure drop across the Carbon Treatment system was too high. The pressure regulators in line with the Carbon system were adjusted and a flow upwards between 13 (49.2 L/min) and 15 gpm (56.9 L/min) was achieved.	September 21, 2015 Initial Flow at 2.5 gpm (9.5 L/min) then raised for injection scenario.
Step 3 – Injection (Start 9:03 AM – Stop 10:00 AM – Travel Time ~ 1 hour)		
BWS-1, BWS-1D, CP-1 and CP-1D	• Collected the post 15-minute sample later to accommodate for lower flow at 10:25 AM to ensure that the crude oil reached the coupon section.	September 21, 2015 Flow at 15 gpm (56.9 L/min)
BWS-2 and CP-2, CP-2D	• Collected the 45-minute at 10:55 AM.	September 21, 2015 Flow at 15 gpm (56.9 L/min)
Step 4 – Flushing (10:00 AM – 12:00 PM) / Surfactant Decontamination initial pumped injection attempt at 12:00 PM and then manual introduction between 1:00 and 1:45 PM		
BWS-3, CP-3 and CP-3D	• Collected within 15 minutes of the introduction of surfactant (i.e., 12:15 PM). • Allow surfactant to reach the end of the pipe after the manual introduction was completed – estimate 60 minutes.	September 21, 2015 Flow at 15 gpm (56.9 L/min)
BWS-4, CP-4 and CP-4D	• Collected the 1-hour sample after the surfactant injection was completed at 2:45 PM.	September 21, 2015 Flow at 15 gpm (56.9 L/min)
BWS-5, BWS-5D, CP-5, and CP-5D	• Collected at the 2-hour BWS sample at 3:30 PM and CP samples at 3:45 PM. The 3-hour sample had to be pushed to the next day because of FedEx® overnight shipping deadline. At 4:15 PM the flow was turned down to 5 gpm (19 L/min) to prevent lagoon overflow at night time.	September 21, 2015 Flow at 15 gpm (56.9 L/min) at the end of the day set to 5 gpm (19 L/min)
Step 5 – Post Decontamination Flushing and Monitoring		
BWS-6, CP-6 and CP-6D	• Collected sample at 9:00 AM.	September 22, 2015 Flow at 5 gpm (19 L/min)
BWS-7	• Collected sample at 12:00 PM.	September 22, 2015 Flow at 5 gpm (19 L/min)
BWS-8	• Collected sample at 3:00 PM.	September 22, 2015

Sample ID	Sample Description	Estimated Timeline & System Flow
		Flow at 5 gpm (19 L/min)
BWS-9, CP-7, and CP-7D	• Collected sample at 9:45 AM.	September 23, 2015 Flow 5 gpm (19 L/min)
BWS-10, CP-8, CP-8D	• Collected sample at 9:45 AM.	September 24, 2015 Flow at 5 gpm (19 L/min)
BWS-11, CP-9, CP-9D	• Collected sample at 9:45 AM.	September 25, 2015 Flow at 5 gpm (19 L/min)
BWS-12	• INL collected sample at 9:45 AM (7 days after the start of reconditioning).	September 30, 2015 Flow at 5 gpm (19 L/min)
BWS-13	• INL collected sample at 9:45 AM (14 days after the start of reconditioning).	October 7, 2015 Flow at 5 gpm (19 L/min)

BWS, bulk water sample; CP, coupon; D, duplicate; 1, 2, 3, etc., sequential sample number; gpm, gallons per minute; L/min, liters per minute; INL, Idaho National Laboratory

After completion, the WSTB blank coupons were left in place for shutdown and winter storage.

3.0 Analysis of Test Results

3.1 Disinfection of Large Water Volumes

The four mobile disinfection units were tested for their ability to disinfection *B. globigii* spores in water from the WSTB lagoon. Data analyses and results from the disinfection experiments are presented in the following sections. Summary descriptions of the disinfection units and experimental design are included for clarity and context.

3.1.1 EPA AOP Trailer Unit Testing

Water flowing through the EPA AOP trailer unit was subjected to treatment with UV light and ozone. Both UV light and ozone are disinfectants, but irradiation of ozone with UV light can lead to the formation of •OH (hydroxyl) radicals, which are short lived but potentially potent biocides. Before the treatment experiments began, two thousand gallons of water from the WSTB lagoon containing naturally occurring dirt and algae was pumped into the influent bladder tank. *B. globigii* spores were mixed into this volume and were kept well dispersed in the influent bladder tank using a recirculation pump (see section 2.1 for more detail). This served as the influent feed for the AOP trailer. Treated effluent from the AOP trailer was stored in another 2,000 gallon (7,571 L) bladder tank until experiments concluded. The trailer was operated for 5.5 hours at a flowrate of 5 gpm (19 L/min), with samples being taken every hour, except for the last sample.

Figure 22 shows the AOP trailer influent spore density (blue bars) and the density of spores in the treated effluent (orange bars). Influent and effluent samples were taken simultaneously, so the difference between the bars at each time point represents the amount of spore inactivation taking place, or log reduction (green line) at that point in time. The influent *B. globigii* density is stable throughout the treatment periods at approximately 2.3×10^5 colony forming units (cfu)/ml. This indicates that the recirculating pump attached to the 2,000 gallon (7,571 L) influent bladder tank kept the spores well mixed throughout the course of the experiment.

Effluent spore densities in the treated water varied over the course of the treatment experiment. On average, a 4-log reduction was observed between the AOP trailer influent and effluent. However, individual log reduction values fluctuated from a high of 5 at 120 and 240 minutes of operation, to a low of 1.5 at 300 minutes. This inconsistent disinfection may be related to a number of factors such as: 1) Changing lagoon temperature (from 15°C to 25°C) affecting ozone diffusion into the water and ozone generator output; 2) changing turbidity of the influent water with temperature and mixing in the tank; 3) inconsistent UV lamp output with temperature; 4) hours of operation; 5) unknown factors related to other site specific conditions and the unit being removed from the trailer. Also, the AOP system was powered by a portable generator that had to be shut down for refueling after the 240 minute sampling point, which may have negatively impacted the performance. However, a 5-log reduction in spores does appear possible. The factors mentioned above that could have influenced disinfection performance were not explored in-depth, and their impact requires further investigation.

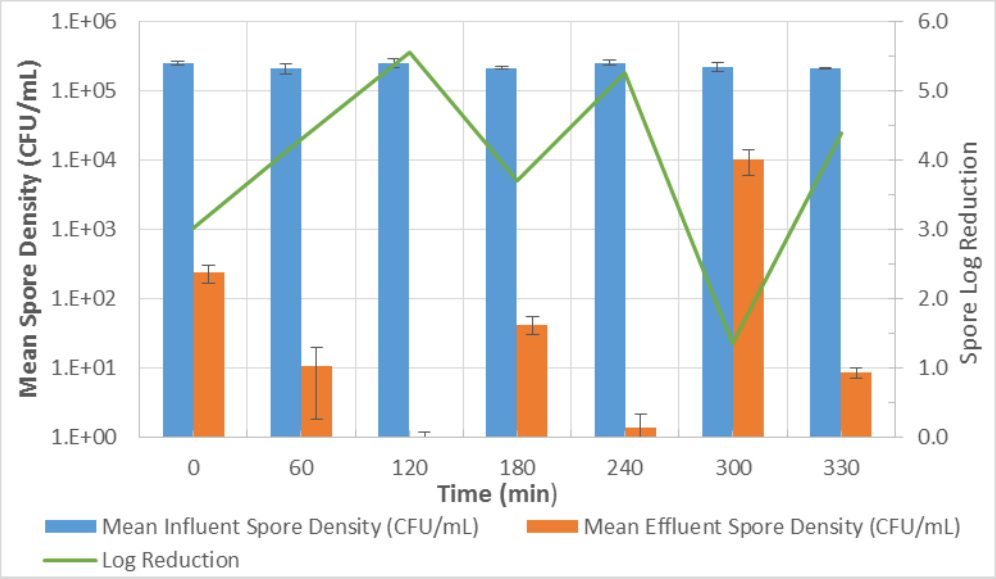

Figure 22. Treatment performance of the Advanced Oxidation Process trailer over the course of 5.5 hrs. Blue and orange bars represent the mean spore density in the AOP trailer influent and effluent, respectively. The green line represents log reduction, or the amount of inactivation occurring at each sampling point. Error bars represent the range between duplicate samples taken at each time point.

Table 2 contains a summary of AOP technology-specific equipment observations recorded during the treatment experiments and considerations for similar field deployments. The terms Low, Medium and High are the opinions of the authors of this study, and are based on their experience operating the equipment in the field. The text in the table is meant to support these opinions, and they are specific to this piece of equipment. Other equipment operators may come to different conclusions under different conditions.

Table 2. EPA Advanced Oxidation Process Trailer Technology-Specific Considerations and Observations*

Technology Considerations	Rating and Comments
Market Availability	Low. Originally custom designed by EPA for a remediation project to provide advanced oxidation with UV and Ozone. A trailer-mounted system that was re-purposed and tested for disinfection. One ozonation process component (Speece Cone diffuser) not commercially available. Other UV and ozonation process components commercially available.
Capital Cost	High (estimated > $40,000). Custom design, process components, plumbing, trailer, etc.
Shipment to Site	Medium. Requires a tow vehicle to pull the trailer to site. Trailer may require State inspection and driver that meets the training requirements for towing the vehicle.

Technology Considerations	Rating and Comments
Setup Considerations	Medium. Requires 110 and 220 Volt AC electric or generator, the plumbing connections to the process units need to be reassembled on site. The Ozonator cone setup requires 2-3 persons onsite to assemble.
Operational Considerations	Medium. Requires operation of valves to remove air from the process units, valve adjustment to meet pressure and flow requirements. Some of the vented air may contain contaminated droplets of water that need to be contained or recirculated back through the system. There is excess ozone emissions from process unit that needs to be destroyed or vented. The catalytic destruction unit was un-operable the unit had to be vented. Flow rate needs to be less than 5 gpm (19 L/min).
Maintenance and Consumables	Low. UV lamp replacement, pump repair when needed. Dual voltage electric supply (see setup consideration).
Result Summary	Under the tested conditions, an average of 4-log removal of *B. globigii* was observed in this flow through type operation (removal varied from 1.5 to 5 log). Improved understanding of the EPA AOP system performance may improve the consistency of disinfection.

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.

3.1.2 Solstreme™ UV System Testing

The Solstreme unit disinfects water through UV light only. Disinfection experiments were performed in the same manner as for the EPA AOP trailer discussed in Section 3.1.1. Figure 18 shows the Solstreme influent spore density (blue bars) and the density of spores in the treated effluent (orange bars). Influent and effluent samples were taken simultaneously, so the difference between the bars at each time point represents the amount of spore inactivation taking place, or log reduction (green line) at that point in time. Like the AOP trailer experiments, influent spore density was over the course of the experiment at approximately 1.6×10^5 cfu/ml. This was a positive finding since a consistent influent concentration was desired over the course of the experiment.

The effluent spore densities from the Solstreme consistently decrease as the experiment progressed. A corresponding increase in spore log reduction over the course of the experiment was also observed. After discussing this finding with the Solstreme manufacturer, a possible reason for this increase in disinfection performance emerged. The Solstreme UV output is higher at higher temperature. Over the course of the experimental period (from early morning to mid-afternoon), the air and lagoon water temperature at the test site increased from 12° to 28°C and 15° to 25°C, respectively. It should be noted that no free chlorine residual was detected in the water.

Figure 23 shows the log reduction data from Figure 24 plotted against the output intensity from the Solstreme device over the course of the experiment. The Solstreme output intensity is a proprietary, unitless measure of the UV output. Typically, for an electrode-based UV bulb, the intensity is measured in milli-watts per square centimeter (mW/cm^2). However, the electrodeless design of the Solstreme unit does not allow for direct conventional radiometer based UV intensity measurements. Figure 24 provides an indirect measure of the UV intensity based on achievement

of 3.5 to 4-log inactivation of *B. globigii* spores in lagoon water with ~11 to 13 NTU turbidity (pH of approximately 7.5). The increase in output intensity of the Solstreme in Figure 24 is perhaps due to the increase in water temperature over the course of the experiment. In the future, it may be beneficial to add a heating element to the Solstreme influent water line to bring water to a temperature between 25° to 30°C. Increased disinfection may be due to hydroxyl radical formation due to photolysis of the water with higher temperature. The influence of air and water temperature on disinfection performance merit further investigation.

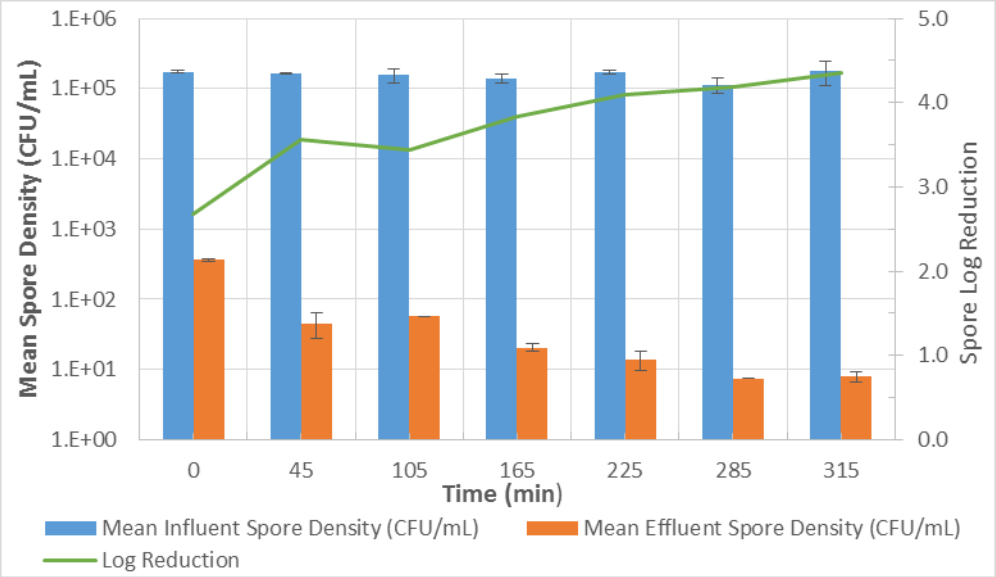

Figure 23. Treatment performance of the Solstreme over the course of 5.5 hrs. Blue and orange bars represent the mean spore density in the Solstreme influent and effluent, respectively. The green line represents log reduction, or the amount of inactivation occurring at each sampling point. Error bars represent the range between duplicate samples taken at each time point.

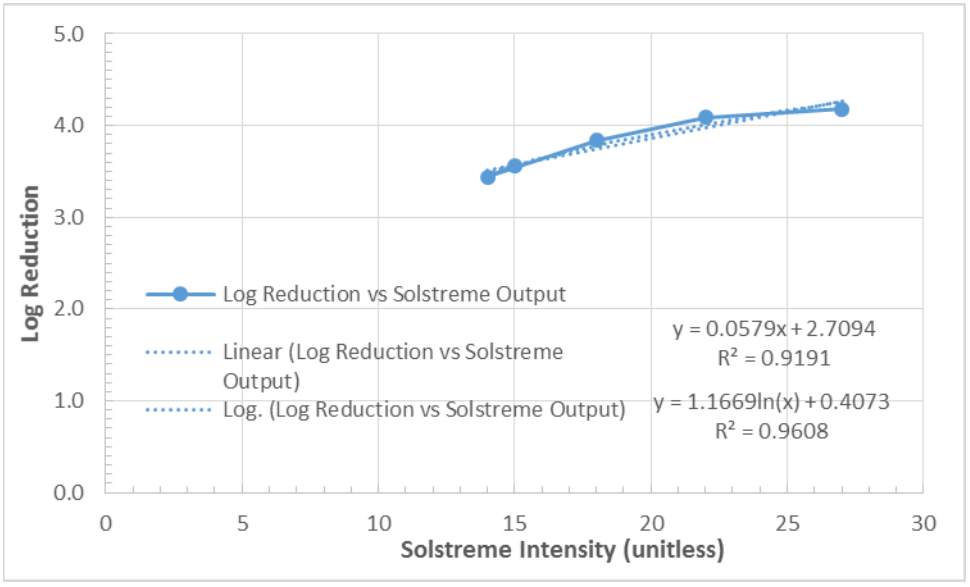

Figure 24. Spore log reduction for Solstreme UV treatment vs. UV output intensity. Solstreme output intensity is a proprietary, unitless measure of UV intensity. Both linear and logarithmic best fit lines are shown.

Table 3 contains a summary of Solstreme technology-specific equipment observations recorded during the treatment experiments and considerations for similar field deployments. The terms Low, Medium and High are the opinions of the authors of this study, and are based on their experience operating the equipment in the field. The text in the table is meant to support these opinions, and they are specific to this piece of equipment. Other equipment operators may come to different conclusions under different conditions.

Table 3. Solstreme Technology-Specific Considerations and Observations*

Technology Considerations	Rating and Comments
Market Availability	Medium. New startup company developed an innovative electrodeless UV lamp design. Made upon order (http://www.solstreme.com/)
Capital Cost	Medium (estimate $15,000).
Shipment to Site	Low. Requires a custom-box (wooden crate or cardboard box with contoured foam) and can be shipped via third party shipper to site. No chemicals or hazardous materials to ship. Can be carried in a truck or a personal vehicle to site.
Setup Considerations	Low. Plug and play, needs 110 Volt AC electric. If water is turbid, a pre-filter is recommended for optimal use. Temperature of the water (i.e., cold < 55^{o}F) impacts operations. Comes with cam lock type connectors. One person can set it up in the field.
Operational Considerations	Low. High turbidity and cold water adversely affect the disinfection process. It gets better results with water in the 70^{o}F to 90^{o}F temperature range and low turbidity. If high disinfection is desired, a heat exchanger may also be needed to regulate water temperature but this requires further investigation. The cost of this heat exchanger would depend on its size.
Maintenance and Consumables	Low. If the processed water is turbid or contains certain dissolved materials that stick to the quartz sleeve, the system (inside quartz sleeve) will need to be cleaned frequently. Other than regular commercially available cleaning agents, no other consumables are required. The UV lamp is electrodeless microwave technology, expected by the manufacturer to last more than 10 years. The quartz sleeve although robust needs to be handled carefully while cleaning. A plunger type device for cleaning the interior of the sleeve is recommended and gloves should be used to prevent smudging of the outside surface.
Result Summary	Under the tested conditions, a 3.5-to 4-log removal of *B. globigii* was observed in a flow through type operation during the experiment.

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.

3.1.3 WaterStep Chlorinator Testing

The WaterStep chlorination system disinfects through generation and application of free chlorine. Free chlorine was generated by electrolysis of sodium chloride (table salt). The WaterStep system operates by applying a potential to a cell that contains electrolytic plates (an anode and cathode). Chlorine gas is formed at the anode, which is channeled through a venturi tube to mix with process water forming free chlorine when dissolved in water (a mixture of hypochlorous acid and hypochlorite ion, depending on pH). This free chlorine migrates into a 1,250 gallon (4,732 L) bladder tank where it can disinfect the contained water. Disinfection experiments with the WaterStep were conducted by spiking the 1,250 gallon (4,732 L) tank with *B. globigii* spores, filling with lagoon water, and then chlorinating. These experiments differ from those conducted with the AOP trailer and Solstreme in that there is no continuous influent and effluent flow. There is one contained volume of contaminated water that is exposed to free chlorine, which can disinfect the *B. globigii* spores over time.

Figure 25 shows the increase in free chlorine concentration inside the WaterStep bladder tank over the course of the experiments, and the subsequent decrease in *B. globigii* spores. No free chlorine was detected in the water at the time the experiment began. During the first 60 minutes after the chlorinator was turned on, the free chlorine concentration in the bladder tank increased slowly due to the organic demand in the water (turbidity was measured as 11 to 13 NTU). However, after the first hour, the demand was overcome and free chlorin in the bladder tank increased at a faster rate. Free chlorine peaked at 210 minutes, at which time the chlorinator was turned off. The subsequent free chlorine samples reflect the decay due to demand and temperature in the bladder tank.

At the start of the experiment, *B. globigii* spores were mixed in the bladder tank volume by pushing on the outside of the tank to move the water around and promote mixing. The first three samples taken from the bladder tank show that the volume was well mixed. *B. globigii* spore density averaged 2.4×10^7 cfu/100 ml (2.4×10^5 cfu/ml) over the first three samples. Figure 21 shows that even as the free chlorine concentration rose from 0.14 to 3.30 mg/L from 60 to 120 minutes, spore density remained the same. This is due to a well-known phenomenon in the field of disinfection knowns as a “lag phase” or “shoulder”. *Bacillus* spores are well known to be resistant to inactivation via oxidative disinfectants, and their concentration will remain stable for a period time in the presence of disinfectants before decreasing (AWWA, 1999; Rice et al., 2005). Once free chlorine did inactivate the *B. globigii* spores, approximately 7-log reduction was achieved after 300 minutes of contact time.

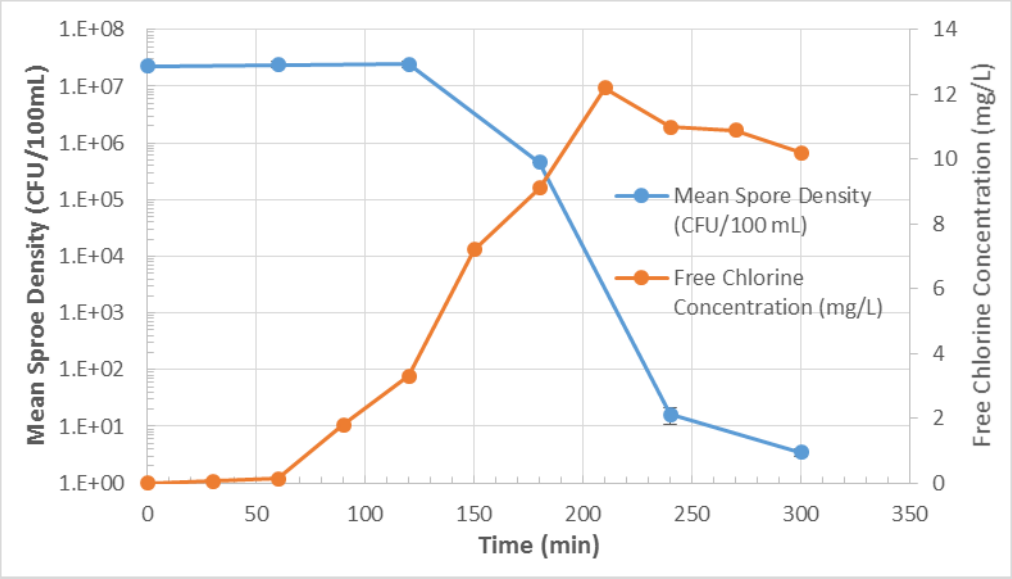

Figure 25. Free chlorine concentration (orange) and *Bacillus globigii* spore (blue line) density over time in the WaterStep bladder tank.

Figure 26 displays the log reduction of *B. globigii* spores plotted against disinfectant (free chlorine) concentration multiplied by the contact time with the disinfectant (Ct). The Ct concept is often used in the disinfection field to determine the combination of disinfectant concentration and contact time needed to achieve a log reduction for a microorganism at fixed pH and temperature conditions. If the disinfection kinetics are linear, different combinations of disinfectant concentration and contact time can yield the same Ct (AWWA, 1999). Often, disinfection kinetic curves for *Bacillus* spores developed using empirical data are not linear due to the "lag phase" or shouldering phenomenon mentioned earlier in this section. The disinfection kinetics displayed in Figure 25 and 26 are not linear, and this non-linearity is exacerbated by the presence of disinfectant demand in the lagoon water as well as varying temperature over the course of the experiment.

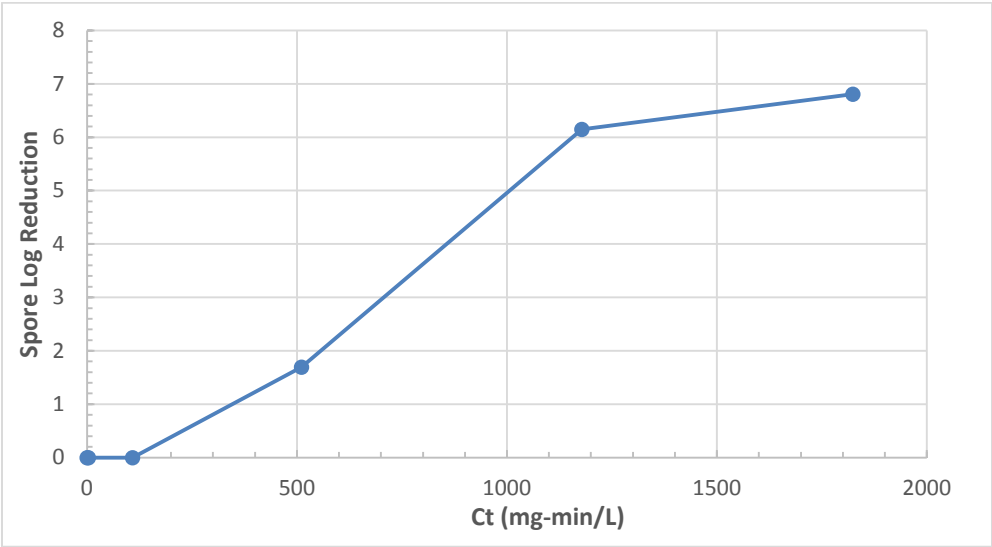

Figure 26. The log reduction in spores during the WaterStep experiment plotted against the Ct value (disinfectant concentration multiplied by time).

Ct values have been compiled in the literature for disinfection of pathogenic and non-pathogenic *Bacillus* spores. These Ct values were often collected in experiments focused on disinfection of drinking water, which generally has less disinfectant demand than the lagoon water used in these experiments. For example, a Ct of 106 mg-min/L was needed for a 3-log reduction of *B. anthracis* Ames at pH 7 and 25° C in the presence of 1 mg/L free chlorine. The 3-log reduction Ct value for *B. globigii* spores at similar conditions was 136 mg-min/L (US EPA, 2012). In the WaterStep experiments with lagoon water, the 3-log reduction Ct was 707 mg-min/L at pH 7 and temperature ranging from 20 to 25°C.

Some of the increase in the Ct values found in lagoon water comes from the fact that temperature started lower than in the drinking water Ct experiments (15°C to 25°C), where temperature was constant (25°C). Disinfectant concentration is generally fixed in lab Ct studies, where it had to increase from zero once the chlorinator was started. Furthermore, disinfectant demand is much less of a factor in lab studies, unlike the WaterStep field studies where disinfectant concentration had to build over time in the presence of an organic load. These factors resulted in a Ct value that is approximately 5 to 6 times higher than those found for the same or similar spores observed under drinking water treatment conditions.

Table 4 contains a summary of WaterStep technology-specific equipment observations recorded during the treatment experiments and considerations for similar field deployments. The terms Low, Medium and High are the opinions of the authors of this study, and are based on their experience operating the equipment in the field. The text in the table is meant to support these opinions, and they are specific to this piece of equipment. Other equipment operators may come to different conclusions under different conditions.

Table 4. WaterStep Technology-Specific Considerations and Observations*

Technology Considerations	Rating and Comments
Market Availability	High. Commercially available off-the-shelf product from a non-profit organization for producing drinking water in communities in developing countries. Self-contained kit, could be used in disaster zone to purify water if there was no power available from the electrical grid. Available from http://waterstep.org/
Capital Cost	Medium (estimate $8,000). Includes storage bladders, pump, battery, charger, solar cell, mounting/transportation rack, and salt based chlorine generator (Chlorinator).
Shipment to Site	Medium. Needs to go on a truck or commercial transportation. Could be transported in a smaller vehicle, if mounting and transportation rack are not used.
Setup Considerations	Medium. Need flat surface to spread out the bladder tanks. Need to recirculate chlorinated water to provide contact time for disinfection. Not a flow through system. Test kit (strips or colorimetric) required to

	periodically check chlorine generation. After disinfection, if chlorine is not consumed, the excess chlorine may need to be neutralized before discharging to the environment.
Operational Considerations	Low. Simple to operate on a short-term basis. If extended contact period is required greater than 3 hours, the salt solution needs to be replenished, electrolytic cell has to be drained, and if not on 110 volt AC power, the battery needs to be charged.
Maintenance and Consumables	Low. Table salt is the only consumable. For optimal chlorine generation, the electrolytic cell needs to be cleaned periodically. Pumps, hoses and O-rings need to be checked periodically for wear and cracking.
Result Summary	Under the tested conditions, a 7-log removal of *B. globigii* was observed in a batch type operation with 300-minutes of contact time.

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.

3.1.4 Hayward Saline C™ 6.0 Chlorination System Testing

The Hayward saline chlorinator was operated in a manner similar to WaterStep, except that disinfection took place in the lagoon instead of a bladder tank. The Hayward unit generates free chlorine using the same principle as the WaterStep, with free chlorine being generated from dissolved salt in water. A potential is applied to a cell that contains electrolytic plates (an anode and cathode). Chlorine gas is formed at the anode, which forms free chlorine when dissolved in water ("free chlorine" is a mixture of hypochlorous acid and hypochlorite ion, depending on pH). Flow moves through the chlorine generating cell, and dissolved free chlorine leaves the cell in the effluent.

The Hayward chlorination cell was set up on a table next to the lagoon. The night before the experiment, the lagoon was drained and 126 lbs (57 kg) of salt was poured into the lagoon. Flow into the lagoon was then set so that 5,000 gallons (18,927 L) would be in the lagoon at the start of the Hayward disinfection experiment. The morning of the experiment, *B. globigii* spores were poured into approximately 5,000 gallons (18,927 L) and mixed via a sump pump positioned in the lagoon. Pre-disinfection spore samples collected from the four corners of the lagoon (see section 2.1.4) resulted in a mean spore concentration of 1.7×10^7 cfu/100 ml with a standard deviation of 2.4×10^6 cfu/100 ml (14% relative standard deviation). This result suggested that the spores and salt, which was dissolved, were well mixed in the lagoon. The sump pump was operated continuously to ensure the salt and spores were well mixed in the lagoon. Flow with dissolved salt and spores was then pumped from the lagoon, though the Hayward chlorination cell and back into the lagoon.

Figure 27 shows the increase in free chlorine in the ~5,000 gallons (18,927 L) in the lagoon over the course of 22.5 hours (pH 7.5). Most samples were taken over the course of 5.5 hours on the first day of experimentation, with the final sample being taken the following morning. The data shows that free chlorine increased in the lagoon more slowly than in the WaterStep bladder tank (Section 3.1.3). For comparison, at 210 minutes after the start of the experiment, free chlorine was 12.2 mg/L in the WaterStep bladder tank, but only 0.65 mg/L in the lagoon. This could be due to more organic demand since there was sediment and algae on the bottom of the lagoon. Furthermore, sunlight could have contributed to degradation of the free chlorine in the lagoon during the day, and some of the free chlorine residual likely dissipated into the air from the surface

of the lagoon. After 22.5 hours, the free chlorine concentration in the lagoon had reached 12 mg/L. No sunlight and decreased temperature during the night could contribute to a rise in free chlorine levels.

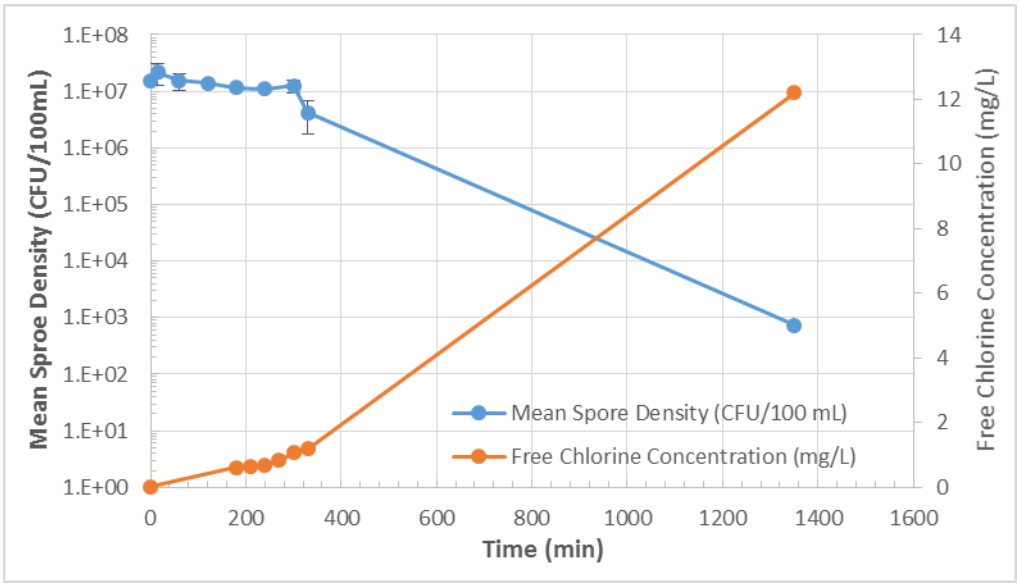

Figure 27. Free chlorine concentration (orange) and *Bacillus globigii* spore (blue line) density over time in lagoon water (~5,000 gal) during disinfection with the Hayward treatment unit.

Spore decrease within the first 5.5 hours was 0.5 log in the presence of 1.2 mg/L free chlorine, but this had increased to 4.3-log in the presence of 12 mg/L at 22.5 hours. For comparison, the WaterStep unit had achieved a 6-log reduction when 12 mg/L free chlorine was achieved. It is important to note that if the Hayward unit had continued to operate past 22.5 hours, a 6 to 7 log reduction might have occurred. If the log reduction continued as illustrated in the graph, then an estimated 7 log reduction would be achieved in the next 10 to 12 hours.

Figure 28 shows log reduction plotted against Ct. In the lagoon, the Hayward unit achieved 4.3 log reduction at a Ct of almost 7,000 mg-min/L (pH 7.5). For comparison, 4-log reduction Ct for *B. anthracis* Sterne at pH 7 and 2 mg/L free chlorine was 280 and 90 mg-min/L at 5° and 25° C, respectively (Rice et al., 2005). Interpolating between these values for 15°C, which was close to the lagoon temperature, yields a Ct value of 185 mg-min/L for drinking water disinfection. The Ct for 4-log removal using the Hayward unit was approximately 6,400 mg-min/L, or 35 times more than the Ct for drinking water.

These results highlight the challenges associated with disinfecting biological agents in a real world environment. The volume of water in the lagoon was larger and more spread out in the lagoon compared to the WaterStep bladder (Section 3.1.3). This kept the temperature in the 15° to 20° C range. Disinfection is slower at lower temperature. In addition, the aforementioned organic demand and free chlorine dissipation factors in the lagoon likely slowed disinfection. Despite these challenges, the results show that disinfection of a chlorine resistant microorganisms like *Bacillus* spores in real world dirty water is possible given time, planning, and the appropriate equipment.

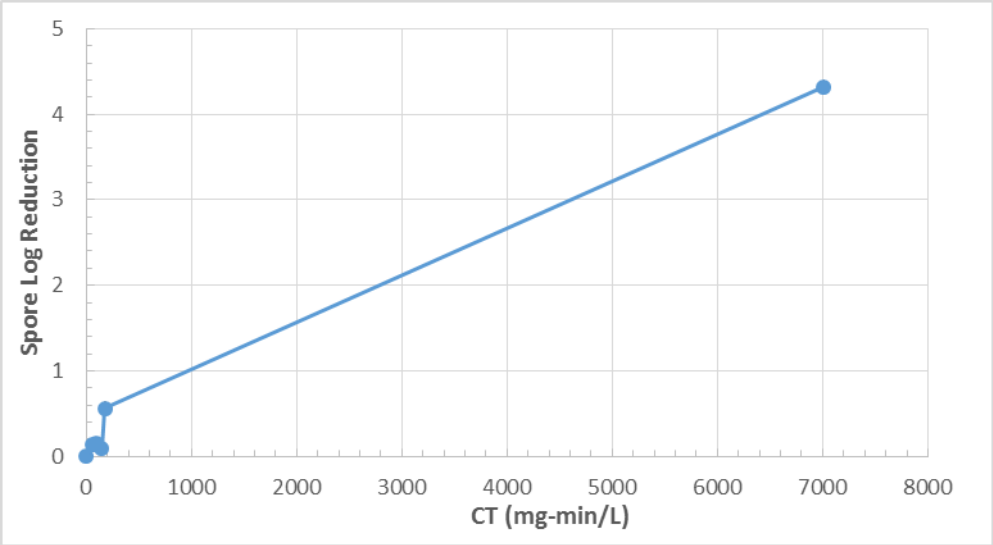

Figure 28. The log reduction in spores during the Hayward experiment plotted against the Ct value (disinfectant concentration multiplied by time).

Table 5 contains a summary of Hayward technology-specific equipment observations recorded during the treatment experiments and considerations for similar field deployments. The terms Low, Medium and High are the opinions of the authors of this study, and are based on their experience operating the equipment in the field. The text in the table is meant to support these opinions, and they are specific to this piece of equipment. Other equipment operators may come to different conclusions under different conditions.

Table 5. Hayward Technology-Specific Considerations and Observations*

Technology Considerations	Rating and Comments
Market Availability	High. Commercially available *in-situ* chlorine generator, off-the-shelf product from a pool product manufacturer. Commonly used for disinfecting swimming pools. Available from http://www.hayward-pool.com/
Capital Cost	Low. $4,000
Shipment to Site	Low. Small package easy to ship or carry in a car.
Setup Considerations	Medium. Can be setup on a table. Requires a 40 gpm (151.4 L/min) pump to run salted water through the system. Salt needs to be added to the source water in sufficient quantities (3,000 to 5,000 mg/L). Chlorine generation can be varied as needed. Need to recirculate chlorinated water to provide contact time for disinfection. Not a flow through system. Strip kit required to periodically check chlorine generation.
Operational Considerations	Low. Requires 110 volt AC power, high capacity (40 gpm [151.4 L/min]) pump. Initial setup requires the chlorine production of the system to be slowly ramped up by starting at ~50% production rate and increased incrementally. Salt may need to be added depending upon usage. It could be operated using bladder tanks, but also suited for open pools.

Technology Considerations	Rating and Comments
Maintenance and Consumables	Low. Table salt (NaCl ~98%). Pump and hoses need to be checked as needed.
Result Summary	Under the tested conditions, the unit achieved a 4.3 log reduction of *B. globigii* in 300 minutes at a Ct of almost 7,000 mg-min/L.

Ct, concentration of disinfectant multiplied by the contact time

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.

3.2 Crude Oil Contamination/Decontamination Tests

As described in detail in section 2.2, Bakken crude oil was mixed with water from the Snake River and allowed to mix overnight in a carboy. Before injection, the bottom oil layer containing dissolved or emulsified oil components was removed from the carboy. This subnatant water layer was injected into the flow in the WSTB pipe, and the slug of contaminated water contacted the inner surfaces of the WSTB pipe.

Tables 6 and 7 show the results for the suite of oil components analyzed for in the water samples and on the removable coupon surfaces, respectively. In the water phase (Table 6), the subnatant injection resulted in a spike in total petroleum hydrocarbons, gasoline-range organics and benzene. Total petroleum hydrocarbon is the summation which includes gasoline-, diesel-, and oil-range organics. Only gasoline range organics were detected. Other BTEX components and longer chain oil components such, diesel-, and oil-range organics were not detected.

Interestingly, some constituent in the background showed up or interfered with the gasoline-range organic/total petroleum hydrocarbon test. However, there was a spike in both parameters during injection of the subnatant and water mixture. Gasoline range organics and total petroleum hydrocarbons were 0.17 mg/L before injection, spiked up to 0.24 and 0.34 mg/L during injection (a 40 and 100% increase over baseline, respectively), and then settled back to 0.16 mg/L during flushing after the contaminant slug had passed. It should be noted that all personnel on site could detect an oil or gasoline smell in the water samples removed from the WSTB pipe when the oil slug was present.

The measured gasoline range organics and total petroleum hydrocarbons spiked again when surfactant was introduced. This is likely due to the methods for both parameters also detecting the surfactants in water, but this was not confirmed. The measured gasoline range organics and total petroleum hydrocarbons decreased once the main surfactant pulse had cleared the pipe, but background levels remained elevated above the initial baseline up to 16 days after the surfactant addition. This data suggest that some of the surfactant still persisted in the WSTB pipe for more than two weeks after its introduction. Upon shaking a water sample, it was noticed that some foaming did occur in the water samples that would suggest the presence of surfactant, or solubilized materials from the pipe walls.

Throughout the test, no components from the Bakken oil were detected on the pipe coupon surfaces. At two and three days after injection of the oil, ethylbenzene and toluene were detected on the coupons. It is possible that Bakken oil components were trapped some place in the

Table 6. Bulk Water Sampling Results

Date	Experimental Phase	Time	Elapsed Time hr	GRO (C6-C12) mg/L	DRO (C10-C20) mg/L	ORO (C20-C34) mg/L	TPH mg/L	Benzene ug/L	Ethylbenzene ug/L	m,p-Xylene ug/L	o-Xylene ug/L	Toluene ug/L	Total Xylenes ug/L
Method Detection Limit, ug/L				(1)	(2)	(2)		0.63	0.68	1.0	1.1	0.72	1.1
MCL, ug/L				NA	NA	NA	NA	5	700	10,000	10,000	1,000	10,000
9/21/2015	Background	8:30	0.0	0.17	0.0	0.0	0.17	0.0	0.0	0.0	0.0	0.0	0.0
	Crude Oil Inject, Start Flush	10:25	1.9	0.24	0.0	0.0	0.24	21.5	0.0	0.0	0.0	0.0	0.0
		10:55	2.4	0.34	0.0	0.0	0.34	32.0	0.0	0.0	0.0	0.0	0.0
	Flush End, Inject Surfactant	12:15	3.8	0.16	0.0	0.0	0.16	1.9	0.0	0.0	0.0	0.0	0.0
	Surfactant Injection Pulses	14:45	6.3	8.0	0.0	0.0	8.0	0.0	0.0	0.0	0.0	0.0	0.0
		15:30	7.0	6.9	0.0	0.0	6.9	0.0	0.0	0.0	0.0	0.0	0.0
9/22/2015	Residual Surfactant, Flowing Water	9:00	24.5	0.31	0.0	0.0	0.31	0.0	0.0	0.0	0.0	0.0	0.0
		12:00	27.5	0.31	0.0	0.0	0.31	0.0	0.0	0.0	0.0	0.0	0.0
		15:00	30.5	0.45	0.0	0.0	0.45	0.0	0.0	0.0	0.0	0.0	0.0
9/23/2015		9:45	49.3	0.28	0.0	0.0	0.28	0.0	0.0	0.0	0.0	0.0	0.0
9/24/2015		9:45	73.3	0.26	0.0	0.0	0.26	0.0	0.0	0.0	0.0	0.0	0.0
9/25/2015		9:45	97.3	0.29	0.0	0.0	0.29	0.0	0.0	0.0	0.0	0.0	0.0
9/30/2015		9:45	217.3	0.29	0.0	0.0	0.29	0.0	0.0	0.0	0.0	0.0	0.0
10/7/2015		9:45	385.3	0.31	0.0	0.0	0.31	0.0	0.0	0.0	0.0	0.0	0.0

DRO, diesel range organics; GRO, gasoline range organics; MCL, maximum contaminant levels; ORO, oil range organics; TPH, total petroleum hydrocarbon; NA, No applicable/no sample; MCL, maximum contaminant level.

Table 7. Coupon Sampling Results

Date	Experimental Phase	Time	Elapsed Time hr	Benzene ug/kg	Ethylbenzene ug/kg	m,p-Xylene ug/kg	o-Xylene ug/kg	Toluene ug/kg	Total Xylenes ug/kg
Method Detection Limit, ug/kg				3.0	3.0	5.8	3.8	6.2	5.8
9/21/2015	Background	8:30	0.0	0.0	0.0	0.0	0.0	0.0	0.0
	Crude Oil Inject, Start Flush	10:25	1.9	0.0	0.0	0.0	0.0	0.0	0.0
		10:55	2.4	0.0	0.0	0.0	0.0	0.0	0.0
	Flush End, Inject Surfactant	12:15	3.8	0.0	0.0	0.0	0.0	0.0	0.0
	Surfactant Injection Pulses	14:45	6.3	0.0	0.0	0.0	0.0	0.0	0.0
		15:30	7.0	0.0	0.0	0.0	0.0	0.0	0.0
9/22/2015	Residual Surfactant, Flowing Water	9:00	24.5	0.0	0.0	0.0	0.0	0.0	0.0
9/23/2015		9:45	49.3	0.0	12.0	0.0	0.0	11.0	0.0
9/24/2015		9:45	73.3	0.0	8.4	0.0	0.0	7.4	0.0
9/25/2015		9:45	97.3	0.0	0.0	0.0	0.0	0.0	0.0

MCL, maximum contaminant level.

WSTB and detached broke loose days after the contamination event and adhered to the coupons. However, no corresponding increase in toluene or ethylbenzene was detected in the bulk water samples. It is possible that some toluene and/or ethylbenzene were present on the coupons below the method limit of detection, and these coupons had higher amounts. It is also possible that the spike in toluene and ethylbenzene on the coupons on Sept. 23 and 24 was external contamination or some other unknown occurrence.

Figure 29 shows a more detailed picture of the spike in benzene that occurred during injection of the Bakken oil subnatant phase from the carboy. The figure shows the phase before injection of the Bakken crude oil-water mixture (Background), while the oil slug was travelling down the pipe (Crude Oil Subnatant Injection), during clean water flushing (flushing @ 15 gpm [56.8 L/min]), surfactant addition (Surfactant Injection-three pulses) and then during clean water flow (Flow@15 gpm [56.8 L/min] with residual surfactant). No benzene was detected in the background water sample before injection of the Bakken oil-water mixture. A spike in benzene was detected when the oil components were travelling down the pipe. However, after the oil slug exited the pipe and clean water was flushed through, the benzene level dropped precipitously. It appears that simple water flushing cleared the pipe of benzene. No benzene was detected during the addition of surfactant or thereafter.

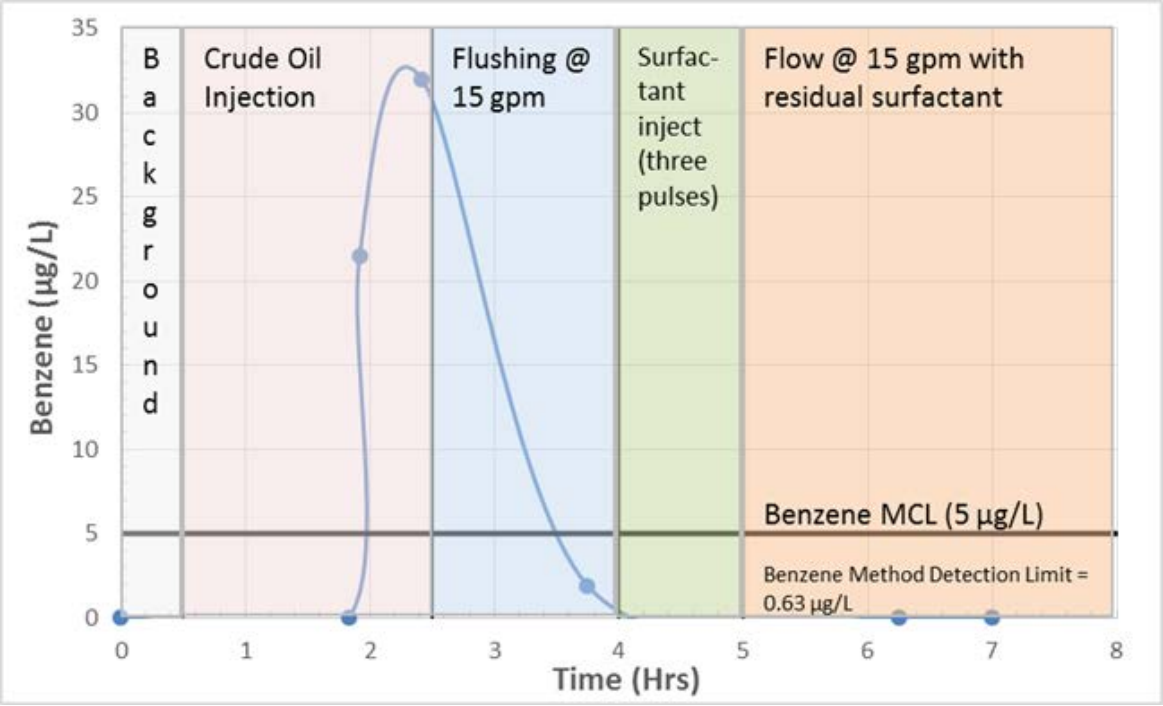

Figure 29. Benzene concentration in the Water Security Test Bed bulk water.

The results of the Bakken crude oil injection show that the oil components such as total petroleum hydrocarbons and BTEX do not persist in the bulk water phase. The inner pipe surface represented by the coupon samples in Table 7 show some presence of ethylbenzene and toluene on Days 3 and 4 (post injection on Day 1) at detectable concentrations. However, these results are outliers compared with the other coupon samples, and no corresponding spikes in ethylbenzene and toluene were observed in the bulk phase. Flushing clean tap water through the WSTB pipe after the oil

slug had exited was enough to drop the levels of benzene below the MCL in bulk water, and to undetectable levels thereafter both in bulk water and coupon samples. Surfactant was added to the pipe because it was anticipated that some oily components would persist in the water or on the infrastructure. However, it appears that flushing alone may have been sufficient to clear the pipe of Bakken oil subnatant, and surfactant addition may not have been necessary. Because no constituents from the Bakken oil were detected on the coupons prior to surfactant addition, additional experiments are merited to better understand the potential role of surfactants when contaminants are detectable.

3.2.1 Online Sensor Data

Figure 30 presents data from the downstream online M4000 TOC sensor from RealTech (RealTech, undated) and Turner oil in water monitor during the day the contaminant/decontaminant tests were conducted. It was expected that these sensors would have the best chance at detecting the Bakken oil subnatant components.

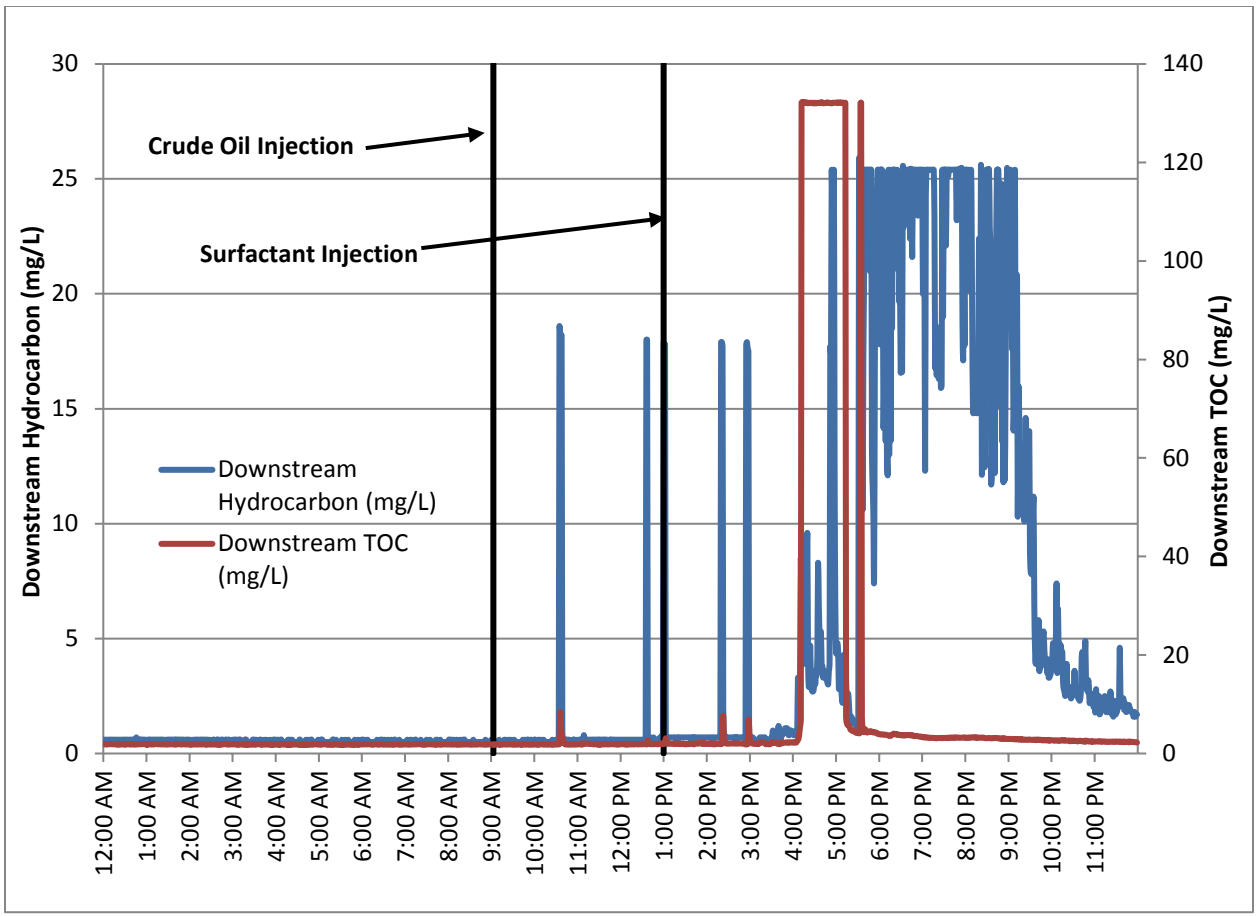

Figure 30. Online total organic carbon (TOC) and hydrocarbon sensor data during the crude oil contamination and surfactant decontamination (9/21/2015).

From Figure 30, it appears that the fluorescence-based hydrocarbon sensor is more sensitive to the surfactant than the subnatant. It should be noted that more of the surfactant was injected when

compared to the crude oil on a mass basis. The UV-based TOC sensor does see a brief spike in TOC value after the surfactant addition (between 4:00 and 5:00 pm) at the beginning of the phase when the corresponding hydrocarbon response is very high. The pulsed method of the surfactant injection is also reflected in the spiky nature of the online hydrocarbon instrument response. The sharp spikes in both sensors observed throughout the experiment are likely due to some undissolved globules of oil passing through the instruments.

Figure 31 shows data from the online Hach colorimetric chlorine sensors during the day the contaminant/decontaminant injection test was conducted. From Figure 31, it appears that the chlorine sensor does not respond to the Bakken oil subnatant components that comprised most of the crude oil injection, but they do respond to the surfactant injection. It is unknown if the chlorine sensor response to the surfactant was due to the interference in the colorimetric analysis or due to actual reduction in the chlorine values. Furthermore, spikes in the data similar to those observed in the TOC and hydrocarbon sensors are seen at the same points in time. Like the TOC and hydrocarbon sensors, these spikes are attributed to globules of oil interfering with the colorimetric analysis in the chlorine sensor. It should be noted that the discussions of the data from Figures 30 and 31 are preliminary.

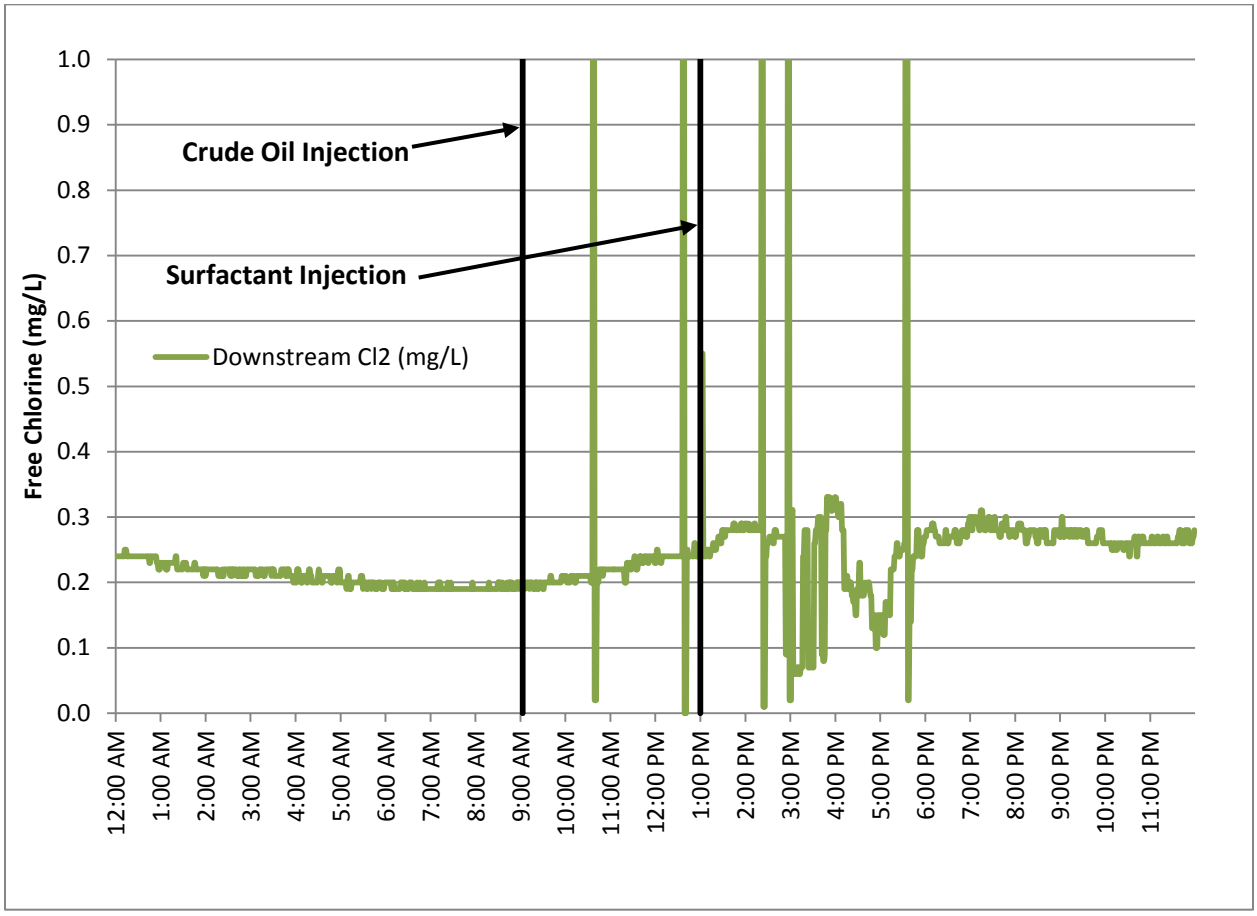

Figure 31. Online chlorine sensor data during the crude oil contamination and surfactant decontamination (9/21/2015).

4.0 Conclusions and Future Work

Experiments performed at the EPA WSTB in 2015 generated useful data on the ability of mobile water treatment devices to disinfect *Bacillus* spores in "dirty" water, or water with disinfectant demand resulting from naturally-occurring particles. Experiments also focused on contamination of the WSTB pipe with oil and effectiveness of decontamination with flushing and surfactants.

The following bullets are a summary of conclusions drawn from the testing performed at the WSTB with mobile water treatment devices:

- The experimental setup was able to provide a consistent source of *Bacillus* spores in lagoon water for treatment experiments. This was true whether the spores were added to a bladder tank or the lagoon.

- The EPA AOP (UV/ozone) trailer achieved an average of 4-log removal of *Bacillus* spores in the flow through the disinfection system. Log removal varied between 1.5 and 5.0 over the course of the 5.5 hour experiment and two thousand gallons of water were treated. Disinfection performance may be improved if temperature were constant, which can influence ozone diffusion into the water as well as UV lamp output. Equipment with less wear and usage may perform more consistently. Finally, shutting down and restarting the disinfection unit may have negatively impacted performance.

- The Solstreme UV disinfection unit achieved an average spore log reduction of 3.7, with log removal increasing from 3.0 to 4.0 over the course of the 5.5 hr experiment. This could have been due to the increase in temperature experienced during the daylight hours elevating the UV output/efficiency and leading to greater disinfection. Adding an inline heat exchanger could potentially help enhance the disinfection performance of the Solstreme since it would help provide a consistent influent temperature. Two thousand gallons of water were treated during the experimental run.

- The WaterStep unit achieved 6.8 log removal in a 1,250 gallon (4,732 L) bladder tank within 5 hours of the start of the experiment while achieving 12.2 mg/L free chlorine. However, this was the smallest volume disinfected during the overall testing period.

- The Hayward salt water chlorination unit achieved 4.3-log reduction of *Bacillus* spores after 22.5 hours of operation and achieved 12 mg/L free chlorine. Assuming the results can be extrapolated, it was estimated that an additional 10-12 hours of disinfection would have achieved 7-log removal. Treatment conditions were the most difficult in this experiment since disinfection occurred in 5,000 gallons (18,927 L) of water contained within the open lagoon. More sediments were present on the bottom of the lagoon, which were not present at those high levels in the other experiments. Furthermore, temperature was lower and exposure to sunlight likely degraded the free chlorine generated in the lagoon.

- Results from the four mobile water treatment units indicate that disinfection of large volumes of water contaminated with biological agents is possible. It is likely that larger

volumes of water may need to be disinfected in a real world scenario, but all of these systems can be scaled up, or multiple units can be put into place. Data generated from this study does demonstrate that disinfection of dirty water in the field is more challenging than disinfecting clean drinking water due to the disinfectant demand present in real world wash water, the impact of low temperature, pH and turbidity on disinfection, and disinfectant dissipation due to exposure to sunlight.

- Table 8 provides a combined performance summary of the mobile water treatment devices evaluated at the WSTB in August 2015. A more detailed summary table including technology specific considerations is included as Appendix B.

Table 8. Mobile Water Treatment Device Performance Summary

Water Treatment Technology Tested	Capital Cost	Average Log Reduction	Volume Treated (gal)	Flow (gpm)	Performance Summary
EPA AOP Trailer (UV and Ozone)	$40,000	4.0	2,000	5	Immediate disinfection, log reduction was unstable during this study due to experimental challenges
Solstreme (UV)	$15,000	3.5 to 4.0	2,000	5	Stable, immediate disinfection, easy to transport and set up.
Water Step (Chlorinator)	$8,000	7.0	1,250	N/A	6-log reduction in 300 min, lowest total treated volume.
Hayward (Chlorinator)	$4,000	4.3	5,000	40	4-log reduction in 1,350 min, under most difficult disinfection conditions.

The following bullets are a summary of conclusions based on the testing performed at the INL WSTB with crude oil:

- Bakken oil contaminated water (subnatant) was successfully injected into the WSTB pipe. This was confirmed by the increase in benzene and total petroleum hydrocarbons observed in the water when the contaminant was in the pipe. A detectable smell of oil or hydrocarbons was present in the sampled water.

- Data from water samples collected during the experiment suggest that flushing the pipe with clean water was an effective decontamination method. Benzene dropped below the MCL during flushing and no other BTEX components were detected in the water. No total petroleum hydrocarbons or BTEX compounds were detected on the pipe infrastructure coupons during or shortly after the crude oil injection. Toluene and ethylbenzene were detected at low levels on the coupons days after injection and decontamination, but it is unclear if these were outlier samples or if interference played a role. No Bakken oil components were detected on subsequent coupon samples.

- Surfactant was injected because it was assumed that oily components would persist in the water phase or on the infrastructure/pipe surfaces. This was not the case, but visual observation of foaming in the water samples might suggest that surfactant persisted in the WSTB pipe for days after the initial injection. Therefore, caution should be used when introducing a surfactant into a water system for the purposes of decontamination as it may not be easy to completely remove it in a timely manner.

Specific research needs that emerged during this study and should be addressed in the future are as follows:

- In water treatment experiments using the AOP trailer, disinfection performance could have been impacted by the following issues: 1) changing lagoon temperature (from 15°C to 25°C) affecting ozone diffusion into the water and ozone generator output; 2) changing turbidity of the influent water with temperature and mixing in the tank; 3) inconsistent UV lamp output with temperature; 4) hours of operation; 5) unknown factors related to other site specific conditions and the unit being removed from the trailer. All of these issues should be explored in-depth.

- The increase in output intensity of the Solstreme in Figure 24 is perhaps due to the increase in water temperature over the course of the experiment. In the future, it may be beneficial to add a heating element to the Solstreme influent water line to bring water to a temperature between 25° to 30°C. Increased disinfection performance may be due to hydroxyl radical formation due to photolysis of the water with higher temperature. The influence of air and water temperature on disinfection performance merit further investigation.

- The sharp spikes in on-line sensors signals observed throughout the experiment are likely due to some undissolved globules of oil passing through the instruments. However, this was not confirmed.

- The measured gasoline range organics and total petroleum hydrocarbons spiked when surfactant was introduced. This is likely due to the methods for both parameters also detecting the surfactants in water, but this was not confirmed.

Future research using the WSTB will focus on addressing other outstanding EPA National Homeland Security Research Center needs.

- *Bacillus* spores can be persistent on drinking water infrastructure, and decontamination of persistent spores is a challenge. Future work will examine physical removal of adhered *Bacillus* spores using pigging or ice pigging. Pigging involves insertion of a grinding mechanism into the pipe which physical scours the internal pipe wall. The principle behind ice pigging is similar, except that a slurry of ice is used to scour the pipe wall.

- If an oil spill occurs, the potential for a fire resulting from ignition of the spilled oil is increased. Fires from oil spills often burn at elevated temperatures, and polyfluoroalkyl firefighting foams are often used to control the flames. If the spill occurs near a water body

with a drinking water intake, it is possible that the foams could make their way into a water distribution system. Therefore, future work will focus on the persistence of polyfluoroalkyl firefighting foams on drinking water infrastructure and decontamination.

- Previous decontamination research has focused on drinking water distribution system materials. There is a need to examine the persistence of chemical and biological contaminants in home plumbing materials and in appliances. To further that goal, copper plumbing lines will be connected between the WSTB pipe and an adjacent vacant building. This plumbing system will have removable sections of copper, PVC and polyethylene with cross-links (PEX) piping so that pre-and post-decontamination contaminant persistence can be examined. Appliances such as refrigerators, washing machines, dishwashers and hot water heaters will also be installed. These appliances will be contaminated with chemical or biological agents, and the effort necessary to decontaminate them will be tested.

5.0 References

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Appendix A: Quality Assurance Project Plan

QUALITY ASSURANCE PROJECT PLAN

EPA QA ID No. - 61_2014_QAPP

EXPERIMENTS IN THE WATER SECURITY TEST BED AT IDAHO NATIONAL LABORATORY

EPA Contract No. EP-C-14-012
Work Assignment No. 1-08
CB&I DN: 500438-QA-PL-000145

Prepared for:
U.S. ENVIRONMENTAL PROTECTION AGENCY
National Homeland Security Research Center (NHSRC)
26 West Martin Luther King Drive
Cincinnati, Ohio 45268

Prepared by:

CB&I Federal Services LLC
5050 Section Avenue
Cincinnati, Ohio 45212

Ruth Corn, Contract-level Contracting Officer Representative
Jeff Szabo, Ph.D., P.E., Work Assignment Contracting Officer Representative
John Hall, Alternate Work Assignment Contracting Officer Representative

Revision No. 2
September 10, 2015

Summary of Work Assignment 1-08 Quality Assurance Project Plan Revision No. 1

The Quality Assurance Project Plan (QAPP) for Work Assignment (WA) 0-08, Revision 0, dated September 3, 2014 (EPA QA ID No. - 61_2014_QAPP), is being revised to include the following elements:

- Updated Work Assignment and CB&I Document Numbers.
- Replaced Mr. Steve Jones as CB&I QA Manager with Mr. Don Schupp.
- Added Ms. Jill Webster as a Project Chemist.
- Added evaluation of technologies to determine the ability to decontaminate large volumes of water to be conducted in mid-August, 2015.
- Added crude oil contamination/decontamination experiment to be conducted in mid-September 2015.
- Extended the project schedule through May 2016, with another *B. globigii* contamination/decontamination experiment.

Revision No. 2

The QAPP for WA 1-08, Revision 1, dated July 2, 2015 (EPA QA ID No. - 61_2014_QAPP), is being revised to address the observations from the EPA technical systems audit (TSA) conducted in August 2015.

- Page headers were revised on all pages to reflect the newer revision number to avoid potential confusion.

CB&I Federal Services LLC Concurrences:

1. E. Radha Krishnan, P.E.
Program Manager

Signature	**Date**

2. Paul C. Kefauver
Project Leader

Signature	**Date**

3. Donald A. Schupp, P.E.
Quality Assurance Manager

Signature	**Date**

U.S. Environmental Protection Agency Endorsement for Implementation:

4. Jeff Szabo, Ph.D., P.E.
Work Assignment Contracting Officer Representative

Signature	**Date**

5. John Hall
Alternate Work Assignment Contracting Officer Representative

Signature	**Date**

6. Ramona Sherman
NHSRC Quality Assurance Manager

Signature	**Date**

Quality Assurance Project Plan Distribution List

U.S. Environmental Protection Agency:

Jeff Szabo, Ph.D., P.E.	Work Assignment Contracting Officer Representative
John Hall	Alternate Work Assignment Contracting Officer Representative
Ramona Sherman	NHSRC Quality Assurance Manager

CB&I Federal Services LLC:

E. Radha Krishnan, P.E.	Program Manager
Paul C. Kefauver	Project Leader
Donald Schupp, P.E.	Quality Assurance Manager
Greg Meiners	Lead Project Scientist
Srinivas Panguluri, P.E.	Data acquisition and electronic communications networking specialist
Tim Kling	T&E Chief of Operations
Lee Heckman	Project Microbiologist
Jill Webster	Project Chemist
Sue Witt	Project Scientist

ALS Environmental

Mr. Rob Nieman	Analytical Project Manager

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1.0 PROJECT DESCRIPTION AND OBJECTIVES

1.1 Background and Project Description

EPA's National Homeland Security Research Center (NHSRC) has partnered with Idaho National Laboratory (INL) to build the Water Security Test Bed (WSTB) at INL in Idaho Falls, Idaho. The centerpiece of the WSTB is an 8-inch diameter drinking water pipe that was taken out of service. The pipe was exhumed from the INL grounds and oriented in the shape of a small drinking water distribution system. The WSTB has service connections to simulate water demands, fire hydrants, and removable coupons to collect samples from the pipe interiors. Experiments focused on contamination (Crude Oil), decontamination (Dispersant or Surfactant) and triggered flushing events will take place in the WSTB. Additional experiments will focus on treatment of large volumes of biologically contaminated water with mobile disinfection technologies; however, the WSTB pipe will not be used for these experiments. Instead the lagoon water will be pumped through a set of tanks and selected treatment systems.

Under contract to EPA (Contract No. EP-C-14-012), CB&I Federal Services LLC (CB&I) has been providing technical support in developing new technologies and evaluation of existing technologies at the EPA Test & Evaluation (T&E) Facility in Cincinnati, Ohio. CB&I will provide technical support for on-site setup and testing to EPA on an as-needed basis. This Quality Assurance Project Plan (QAPP) outlines the tests that will be performed in the WSTB. This QAPP follows the guidance for a Category B measurement project.

1.2 Project Objectives

The planned studies have the following goals:

1. Conduct decontamination tests on the WSTB with selected decontaminants (Dispersant orSurfactant) following intentional contamination (Crude Oil) of the WSTB, and evaluate the effectiveness of the selected decontaminants for removing contaminants from the WSTB.
2. Evaluate select online instrumentation installed on the WSTB to determine their efficacy in detecting anomalous contamination events in the WSTB and trigger flushing of the WSTB through the flushing hydrant.
3. Perform studies on disinfection technologies to determine their ability to treat large volumes of biologically contaminated water.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 Project Organization

The overall project management and distribution of responsibilities among the project personnel are described in this section. Figure 2-1 presents the organization chart for the project. Table 2-1 presents contact information for project personnel. Ms. Ruth E. Corn serves as the EPA T&E Contract-level Contracting Officer Representative (CLCOR). Dr. Jeff Szabo, the EPA Work Assignment Contracting Officer Representative (WACOR) for this study, is responsible for overall technical direction and adhering to the guidelines of the QAPP. Mr. John Hall is the EPA Alternate WACOR and will assist Dr. Szabo. Ms. Ramona Sherman, the EPA NHSRC Quality Assurance (QA) Manager, is responsible for approval of QA documents and QA project assessments.

Mr. Radha Krishnan, P.E., serves as the CB&I Program Manager for the EPA T&E Contract. The CB&I Program Manager will be responsible for the overall project management, program coordination, and management review of deliverables. Mr. Paul Kefauver, CB&I Project Leader, will be responsible for project planning, coordination of activities, and peer review of deliverables. Mr. Donald Schupp, P.E., is the CB&I QA Manager. Mr. Schupp will be responsible for the oversight of CB&I T&E quality program implementation, including QA review of documents and deliverables, and project assessments.

Mr. Greg Meiners, Mr. Srinivas Panguluri, Mr. Dave Elstun, and Mr. Gary Lubbers with CB&I will provide on-site support for setup and operation of the WSTB. Mr. Greg Meiners will serve as the Lead Project Scientist. Mr. Meiners will be responsible for experimental start-up, collection and analysis of the samples, interpretation of the data, and completion of the final report. Mr. Srinivas Panguluri, P.E. will serve as the data acquisition and electronic communications networking specialist. Mr. Dave Elstun and Mr. Gary Lubbers will assist with the on-site equipment setup and testing as needed (including injection pump(s), instrumentation, and data acquisition). Mr. Lee Heckman, CB&I Project Microbiologist, will provide sample analysis support. Mr. Timothy Kling, Ms. Sue Witt, and Ms. Jill Webster with CB&I will provide as needed remote support to Mr. Meiners on an as-needed basis.

Mr. Rob Nieman of ALS Environmental (ALS) will oversee chemical analyses performed at that laboratory.

2.2 Project Schedule

This revision presents anticipated activities expected to be performed between May 2015 and May 2016. A detailed timeline is presented later in the “Experimental Design and Test Conditions” (Section 3.2).

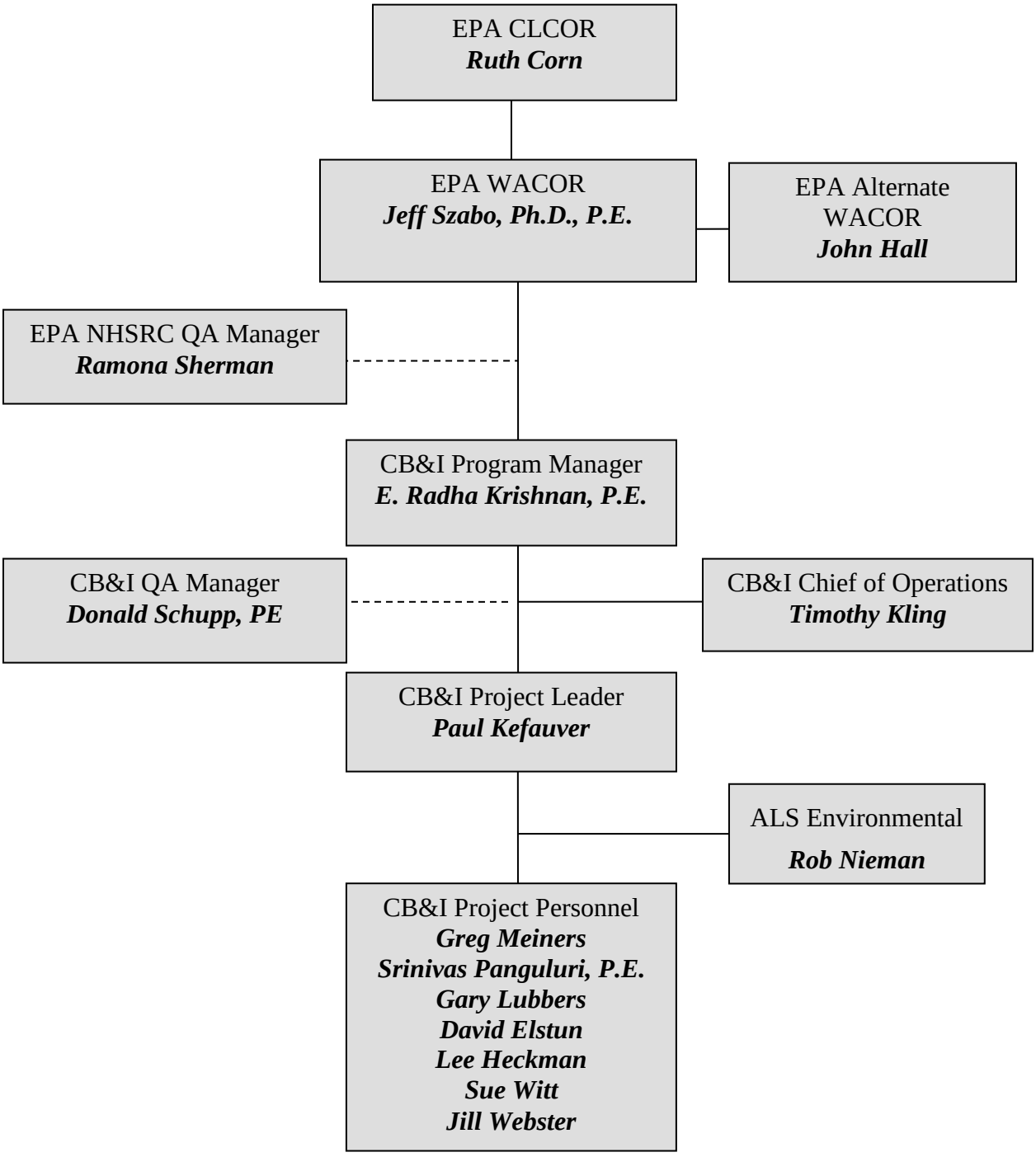

Figure 2-1: Project Organization

Table 2-1: Project Participant Contact Information

Name of Person/Affiliation	Project Role	Phone Number, email
Ruth Corn/EPA	T&E Contract CLCOR	513-569-7610, Corn.Ruth@epa.gov
Jeff Szabo/EPA	WACOR	513-487-2823 Szabo.Jeff@epa.gov
John Hall/EPA	Alternate WACOR	513-487-2814, Hall.John@epa.gov
Ramona Sherman/EPA	NHSRC QA Manager	513-569-7640, Sherman.Ramona@epa.gov
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Donald Schupp/CB&I	QA Manager	513-782-4974, Don.Schupp@cbifederalservices.com
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Greg Meiners/CB&I	Lead Project Scientist	513-487-2821, Greg.Meiners@cbifederalservices.com
Srinivas Panguluri/ CB&I	Data acquisition and electronic communications networking specialist	513-782-4893, Srinivas.Panguluri@cbifederalservices.com
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David Elstun/CB&I	Craftsman	513-569-7051, David.Elstun@cbifederalservices.com
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Sue Witt/CB&I	Project Scientist	513-782-4726, Sue.Witt@cbifederalservices.com
Jill Webster/CB&I	Project Chemist	513-487-2822, Jill.Webster@cbifederalservices.com
Rob Nieman/ALS	Analytical Project Manager	513-733-5336 Rob.Nieman@ALSGlobal.com

3.0 SCIENTIFIC APPROACH

3.1 System/Technology Overview

The WSTB system and the decontamination treatment systems are described in this section.

3.1.1 WSTB Description

The WSTB at INL is constructed from 8-inch diameter drinking water pipe that has been taken out of service. The pipe was exhumed from the INL grounds (by INL personnel) and oriented in the shape of a small drinking water distribution system. The WSTB has service connections to simulate water demands and removable coupons to sample pipe interiors. Experiments focused on contamination and decontamination will take place in the WSTB. Figure 3-1 depicts the main features of the WSTB.

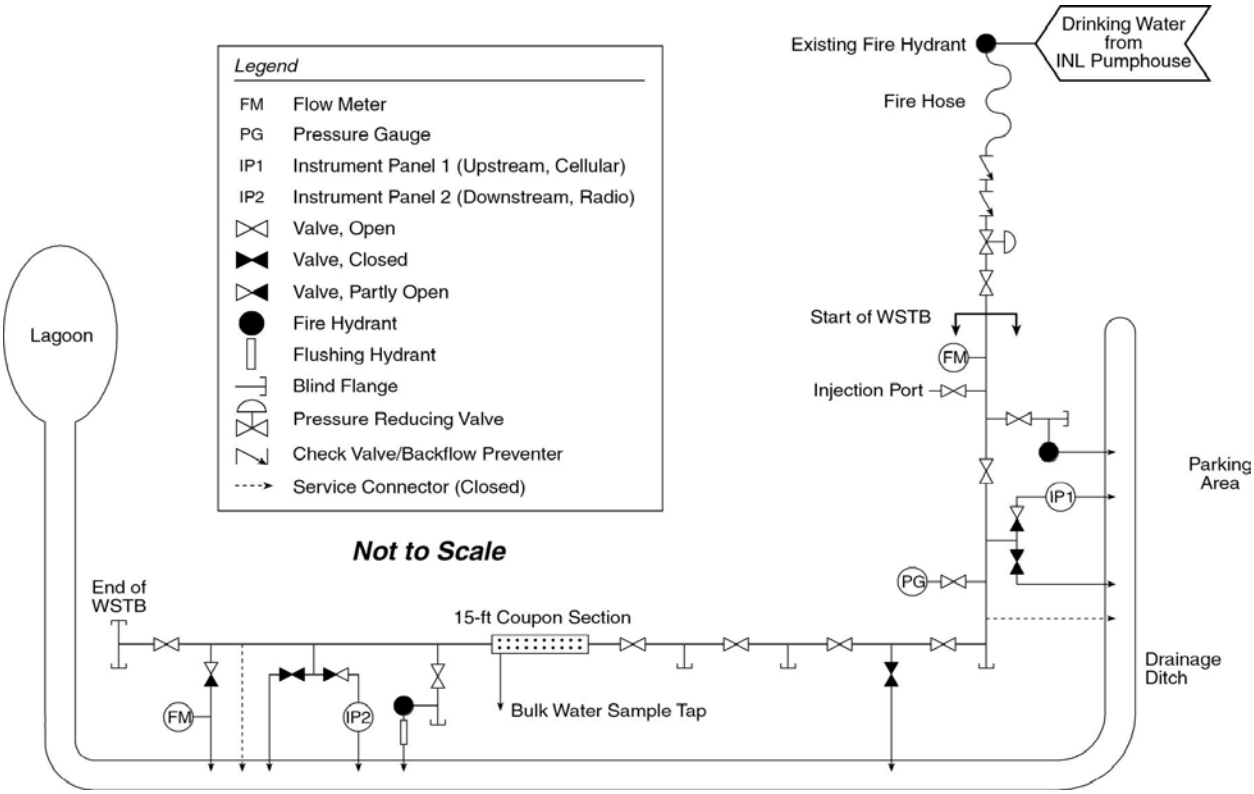

Figure 3-1: Schematic Overview of WSTB

As depicted in Figure 3-1, the source water at INL is connected to the WSTB through an existing fire hydrant. The WSTB consists of 400-feet of 8-inch diameter cement-lined iron pipe. The total volume of the WSTB is estimated to be 1,044 gallons. During infrastructure decontamination experiments, a positive displacement pump will be used to inject the target contaminant at the

beginning of the 400-foot pipe length (as shown in Figure 3-1). A 15-foot PVC pipe-segment is installed that contains 10 sets of duplicate removable coupons of specified pipe material to measure biofilm growth, contamination, and effectiveness of decontamination (a.k.a. coupon section in Figure 3-1). The pipe material for the 20 small coupons (7/10 of an inch in diameter) has been cut from cement mortar-lined iron pipe from INL and set into threaded plugs that will be inserted into the coupon section of the pipe. The twenty coupons are individually numbered CP-0/CP-0D through CP-9/CP-9D (D represents duplicate since duplicate coupons are removed during sampling).

The lagoon has a water storage capacity of 28,000 gallons. The water, contaminant, and decontaminant used during the pipe conditioning and experimental phases (described later in Section 3.2) will be conveyed via the drainage ditch and discharged to the lagoon. The discharged water will be trucked out for disposal on a weekly basis. During the conditioning phase, the system will be operated at 2.5 gallons per minute (gpm), resulting in a total of 25,200 gallons discharged per week (gpw). The partially closed valve near the end of WSTB (shown in Figure 3-1), along with the flow meter, will be used to regulate and maintain flow. During the contaminant/decontaminant injections, the system will be operated at a higher flow rate (~15 gpm) to reduce travel time and manage sampling activities. The higher system flow rate operations (~15 gpm) will be for short durations (1 to 2 hours at a time). Overall, even if the system was run at this high flow rate for a full day, the total discharge is 21,600 gallons, which is within the lagoon capacity. Suitable arrangements will be made by INL to empty the lagoon on a more frequent basis, as necessary, during the experimental phase.

As shown in Figure 3-1, the WSTB will be equipped with sensors in the instrumentation panels (IP1 and IP2) that continuously measure two basic water quality parameters: free chlorine and Total Organic Carbon (TOC). One Hach CL-17 chlorine analyzer and one RealTech M4000 TOC analyzer will be included in each of the instrumentation panels. The Hach CL-17 chlorine analyzer uses colorimetric DPD chemistry to monitor water continuously for free chlorine. The RealTech M4000 uses the ultraviolet (UV) 254 nanometer wavelength (i.e., UV254) for determining the TOC content. UV254 instruments are often used as an inexpensive indicator of TOC in water. UV254 measurements are known to have some bias towards aromatic organics; however, they are relatively inexpensive to maintain and operate when compared to the traditional UV-persulfate based TOC analyzers. The 8-inch pipe system is constructed directly over the lined drainage ditch for spill/ leak containment. Figure 3-1 depicts the drainage ditch offset in order to present the equipment more clearly.

Two fire hydrants will be installed in the pipe and one of the units (downstream location) is a flushing hydrant that will be used to automatically flush the pipe when anomalous water quality events are detected in the WSTB for the dechlorination/flushing test described in Section 3.2.2.

3.1.2 Treating Large Volumes of Contaminated Water

As mentioned previously, up to four large volume water treatment technologies are expected to be

tested to determine their ability to disinfect large volumes of biologically contaminated water. Figure 3.2 depicts a schematic layout of the proposed testing.

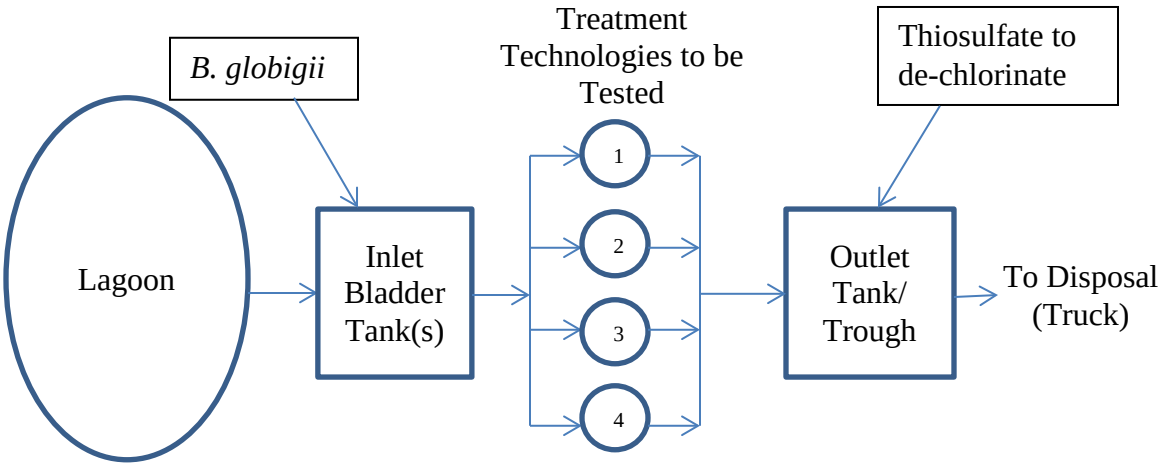

Figure 3-2: Large Water Volume Testing Schematic

The following four technologies will be studied to determine their effectiveness in decontaminating large volumes of water contaminated with *B. globigii*: 1) Hayward Saline C 6.0 Chlorination System, 2) Advanced Oxidation Process (AOP) Ultraviolet (UV)-Ozone System, 3) Solstreme UV System, and 4) WaterStep Chlorinator.

1. Hayward Saline C 6.0 Chlorination System – This is a commercial pool chlorination system that operates by electrolyzing sodium chloride (NaCl), salt that has been added to the pool to form free chlorine for disinfection. To operate the system salt is added directly to the pool at least 24 hours before the system is started. Roughly 28 pounds of salt is recommended for every 1,000 gallons of pool water to reach 3500 ppm.

2. AOP UV-Ozone System – The AOP system in a trailer was custom-built at the EPA T&E Facility in Cincinnati, Ohio. The AOP system is comprised of four major components – the Power Prep 66 (air preparation unit), CD2000 (ozone production unit), Trojan UVMax (UV generation unit), and the Aquionics UV (UV generation unit). This AOP system was designed for the treatment and destruction of organic compounds and microbes in water. The AOP trailer will be operated with both the CD2000 ozone generator and the Aquionics UV system operating in series.

3. The Solstreme™ UV System - Uses an electrode-less lamp technology to provide UV disinfection. The system is expected to provide much higher level of UV dose compared to an equivalent electrode based UV lamp.

4. WaterStep Mobile Water System (MWS) – The MWS uses sodium chloride (salt) to generate chlorine to disinfect water. The system operates on either 120V electricity, 12 volt DC battery, or available hand pumps and solar panels.

3.2 Experimental Design and Test Conditions

Overall, three different types of experiments or tests will be performed using the WSTB. They are presented in the order of the projected timeline for current contract year (June 1, 2015 through May 31, 2016). The disinfection of large volumes of water, not using the WSTB is summarized in Section 3.2.1 (August 2015), and the contamination/decontamination tests are summarized in Section 3.2.2 (September 2015). Dechlorination/flushing tests are described in Section 3.2.3 (previously completed and optional for the current year). The detailed projected timeline is summarized below for completing the equipment setup and performing the experiments:

- May 14, 2015 – The WSTB was drained and shut down over the winter (2014 to 2015). Return the WSTB to baseline activity in accordance to the previous version of this QAPP (Revision 0). Fill pipe and collect large volume samples to determine *B. globigii* residual from previous testing in October 2014. Set up and turn on upstream and downstream instrument panels.
- May 19, 2015 – Decontaminate pipe with ClO2 in accordance to the previous QAPP (Revision 0). Collect samples for *B. globigii*, ClO_2, and free and total chlorine.
- May 20, 2015 – Flush pipe with fresh water and begin conditioning in accordance to the previous QAPP (Revision 0). Purge flow is 2.5 gpm.
- August 12, 2015 through 21, 2015 – Personnel onsite to prepare and conduct testing using the following four large water volume decontamination technologies: 1) Hayward Saline C 6.0 Chlorination System, 2) Advanced Oxidation Process (AOP) Ultraviolet (UV)-Ozone System, 3) Solstreme UV System, and 4) WaterStep Chlorinator.
- September 14 – 18 or 21 - 25, 2015 – Conduct crude oil contamination/decontamination testing (in the WSTB)
- September 25, 2015 – October 30, 2015 – Collect large volume samples by INL, and analyze by CB&I.
- October 31, 2015 – Drain and winterize pipe by INL. Place instrument panels in storage.
- April/May, 2016 – Fill pipe and set the instrument panels in place. Begin conditioning of the pipe.

3.2.1 Disinfection of Large Water Volumes

This experiment will assess the ability of a portable disinfection unit to disinfect a large volume of water containing *Bacillus* spores. The following four treatment technologies will be evaluated to decontaminate the water from the lagoon: 1) Hayward Saline C 6.0 Chlorination System, 2) Advanced Oxidation Process (AOP) Ultraviolet (UV)-Ozone System, 3) Solstreme UV System, and 4) WaterStep Chlorinator. The effectiveness of individual treatment technology will simply be evaluated based on a mass balance approach where the water containing *B. globigii* spores drawn from the lagoon will be sampled before it enters the individual treatment technology and

then after to determine its effectiveness.

Water will be pumped from the lagoon into an inlet bladder tank system that contains a mixing pump to provide a continuous stream of *B. globigii* spores contaminated water as shown in Figure 3-2. A target inlet concentration of greater than 10^6 spores/100 mL (or 10^4 spores/mL) will be prepared using the inlet tanks and mixers. The water will then be pumped through one or more of the treatment units to be tested. Each unit will be tested for a minimum of 1 hour, up to a maximum of 6 hours. Pre-treatment and post-treatment water samples for *B. globigii* analysis will be collected at the same time. Two pre-treatment and two post-treatment samples will be collected for each system per hour. Each treatment system is expected to be operated at nominal rate of 5 gpm. If disinfectant such as free chlorine is used in the treatment unit, this will be measured once per hour.

The Hayward Saline C 6.0 Chlorination System is an in-situ type of treatment technology, therefore it will be operated using the lagoon as the "pool" or source of water. The day before this system is tested, the lagoon will be drained and approximately 126 lbs of salt will be added to the lagoon where the water flows in from the WSTB. The water from the WSTB will then be run at 5 gpm for approximately 15 hours (releasing ~4,500 gallons) and allowed to mix with the salt in the lagoon. Required amount of *B. globigii* will be added to reach a concentration of greater than 10^6 spores/100 mL (or 10^4 spores/mL) in the lagoon. For the purpose of evaluation, influent samples from four locations in the lagoon will be collected, then the system will be started and operated at the manufacturer recommended rate of 40 gpm for greater than 6 hours and periodic treated/effluent samples will be collected.

3.2.2 Contamination/Decontamination Tests

These experiments involve contamination of the WSTB using crude oil (September 2015), and the subsequent decontamination of WSTB using a flushing event at 15 gpm for 1 hour followed by an injection of Dispersant and/or Surfactant (for crude oil decontamination). Each contamination/decontamination experiment consists of the following main steps:

Step 1 - Pipe conditioning (cultivation of biofilm)
Step 2 - Instrumentation panel, injection equipment setup and background sampling
Step 3 – Preparation of contaminant stock and contaminant injection (addition of crude oil to the WSTB)
Step 4 – Preparation of decontaminant and decontamination using flushing along with a dispersant and/or surfactant for crude oil removal,
Step 5 - Post-decontamination flushing, reconditioning, and monitoring

The actual experiment/testing dates may vary depending upon CB&I/EPA/INL personnel availability and prevailing weather conditions or other unforeseen events. The dates presented in the subsequent section are dependent on the starting time-line presented earlier. Any changes in start date may shift the actual dates and times mentioned in this document.

Step 1 - Pipe conditioning (cultivation of biofilm)
To effectively study the adsorption of contaminants on pipe walls, it is essential to ensure that there is a viable biofilm. The biofilm could influence adsorption of the contaminant on the pipe wall in addition to metabolism, biodegradation, or detoxification of the contaminant.

Previously under EPA Contract EP-C-09-041, CB&I performed a literature review of biofilm cultivation and identified four primary techniques that could potentially be used for cultivating biofilm within the WSTB.

1) Sequential batch fermentation and introduction into the WSTB;
2) Using the WSTB as a reactor by passing water with low concentrations of carbon, nitrogen, and salts;
3) Use of an external annular reactor;
4) Natural biofilm cultivation by passing water through the WSTB.

The fourth option, natural cultivation of biofilm, has been chosen as the cultivation procedure for testing of the WSTB. This will be accomplished by passing INL tap water through the WSTB continuously over a period of time (estimated to be a minimum of 4 months – starting mid-May 2015 for the late-September 2015 Contamination/Decontamination testing). After initial flushing to remove any debris, the flow rate will be set at 2.5 gpm with a total discharge of 25,200 gallons per week to the lagoon, which allows for weekly trucking and disposal of the accumulated discharge.

Step 2 - Instrumentation panel, injection equipment setup and background sampling
In late-September 2014, a simple dye tracer study (using non-toxic biodegradable dye, such as Bright Dyes – www.brightdyes.com) was performed to visually confirm the theoretical calculations of travel times and system flows. This dye tracer study is not planned to be repeated during future tests.

Mid-September 2015 – Crude Oil Testing
Prior to the contamination Step (Step 3), bulk water samples (BWS-X) and Coupon Samples (CP-X) will be collected to establish background levels. The BWS samples will be analyzed for crude oil components such as volatile organic compounds (VOCs), benzene, toluene, ethylbenzene, and xylene (BTEX), gasoline range organics (GRO), diesel range organics (DRO), and oil range organics (ORO). Coupon samples will be analyzed for biofilm density using heterotrophic plate count (HPC), as well as crude oil components. It is expected that crude oil components will be non-detectable in the baseline samples. Free chlorine (CL-F-#) will also be measured periodically. All sampling activities related to crude oil testing are summarized in Table 3-1 and analytical methods are described in Table 4-2.

Step 3 - Preparation of contaminant stock and contaminant injection (addition of crude oil to the WSTB)

Mid-September 2015 – Crude Oil Testing

Preparation of Crude Oil (Contaminant Stock) – The crude oil for this study will be obtained from Marathon Petroleum Corporation. The oil procured will be from the Bakken shale in North Dakota. In general, the Bakken crude oil presents the same physical properties as gasoline or other fuels. It will float on water, as its specific gravity is less than 1, and it is considered moderately volatile. It is also known as "North Dakota Sweet," or "North Dakota Light" crude oil, due to its low sulfur content. In this respect, it is similar to traditional crude oil from West Texas, known as West Texas intermediate crude. This type of crude oil is very desirable, and out of each barrel produced, approximately 95% of it is refined into gasoline, diesel fuel, or jet fuel. (Appendix H - RR10, 2015).

A review of literature indicates that the maximum dissolution of gasoline/diesel (water soluble fraction) is achieved to 95% completion in 17.5 hours. In this referenced methodology (Guard, H.E. et al., 1983 – Appendix G), 210 mL of gasoline/diesel is added to 1,890 mL of water (a mix ratio of 1:9). The mixture is stirred slowly so the meniscus remains intact. The sample is drained from the bottom of the flask (Guard, H.E. et al., 1983). Since Bakken Crude is considered mostly gasoline, this methodology will be tested with tap water in Cincinnati. At Idaho this process will be repeated using water from the Snake River. One 25 liter Nalgene carboy with bottom spigot will be setup on a stir plate with gentle mixing. The carboy will contain 22.5 liters of water and 2.5 liters of crude. After a minimum of 17.5 hours of mixing, 20 liters of mixed water will be drawn from the bottom for injection. The drawing from the bottom of the carboy simulates a miscible crude drawn into the intake of a water treatment plant during a spill event. The 17.5 hour mixing process represents some weatherization that may occur during a spill event. Preliminary testing will be performed at the T&E Facility to determine confirm the crude mix ratio.

Contamination Test Protocol – The crude oil suspension as prepared above, will be introduced into the WSTB using a positive displacement pump. Prior to the introduction of the crude oil (and in conjunction with the INL) a commercially available appropriately-sized granular activated carbon (GAC) system will be connected to the outlet of the WSTB. The purpose of this system is simply to contain any crude oil component from exiting to the lagoon. Once flushing and decontamination activities are completed the unit will be disconnected. The WSTB will be operated at 15 gpm under this condition with a minimum contact time of approximately 1 hour (to accommodate for travel time). Injection duration is also estimated to be 1 hour so that there is a contact of 1-hour after the bolus of crude oil suspension reaches the coupon section of the pipe. All sampling activities related to crude oil testing are summarized in Table 3-1 at the end of main step descriptions.

Step 4 - Preparation of decontaminant and decontamination using flushing along with a dispersant and/or surfactant for crude oil removal

Mid-September 2015 – Crude Oil Testing

Preparation of Decontaminant Agent Stock – The surfactant, Surfonic TDA-6, was identified based on EPA pilot testing at the T&E Facility (EPA, 2008). The EPA study indicated that Surfonic

TDA-6 was effective in removing diesel from the drinking water pipe surfaces. Therefore, Surfonic TDA-6 (or equivalent decontaminant) will be applied during the current pilot-scale decontamination of crude oil from the WSTB pipe surface. Twenty five liters of Surfonic TDA-6 flushing mix will be prepared with water for injection.

Decontamination Test Protocol – Once the injected crude oil slug has cleared the pipe, the WSTB will be flushed for 2 hrs at 15 gpm. This will provide data on whether flushing along removes crude oil from the water and pipe surfaces. Following the 2 hr flush, the prepared Surfonic stock solution will be injected into the WSTB (see Table 3-1). Injection of the Surfonic stock solution will continue until it has reached the end of the pipe (estimated to be approximately 1 hour and 5 minutes based on theoretical calculations and the dye tracer travel time confirmation). Injection will be stopped, online instrumentation will be stopped, and water flow out of the WSTB will be stopped for 18-24 hours so that the water containing the surfactant will be stagnant in the pipe to perform crude oil removal. All sampling activities related to crude oil testing are summarized in Table 3-1 at the end of main step descriptions.

Step 5 - Post-decontamination flushing, reconditioning, and monitoring
Late-September 2015 – Crude Oil Testing
Following collection of the samples for Step 4 (shown in Table 3-1), the WSTB will be flushed with fresh water for approximately 1 hour at 15 gpm to clear the surfactant. The flow will then be reduced to 5 gpm. BWS and CP will be collected following the procedures described in Section 4.2. All sampling activities related to crude oil testing are summarized in Table 3-1.

Table 3-1. Crude Oil Contamination/Decontamination Related Sampling Activity

Sample ID	Sample Description	Estimated Timeline & System Flow
Step 2 - Background		
BWS-0 (Control)	• Collect a sample prior to injection of crude oil	September 21, 2015 Flow at 2.5 gpm
CP-0, CP-0D and Cl2-F-1	• Collect at the same time as BWS-0 • After sampling, turn up flow to 15 gpm	September 21, 2015 Flow at 2.5 gpm
Step 3 – Injection (Start 9:00 AM – Stop 10:00 AM – Travel Time ~ 1 hour)		
BWS-1, BWS-1D, CP-1 and CP-1D	• Collect after 15 minutes of the injection of crude oil reaches the coupon section (i.e., 10:15 AM).	September 21, 2015 Flow at 15 gpm
BWS-2, CP-2, CP-2D and Cl2-F-2	• Collect after 45 minutes of the injection of crude oil reaches the coupon section (i.e., 10:45 AM)	September 21, 2015 Flow at 15 gpm
Step 4 – Flushing (10:00 AM – 12:00 PM) / Surfactant Decon. (12:00 PM – 1:00 PM)		
BWS-3, CP-3 and CP-3D	• Collect within 5 minutes of the introduction of surfactant (i.e., 12:05 PM).	September 21, 2015 Flow at 15 gpm

Sample ID	Sample Description	Estimated Timeline & System Flow
	• Allow surfactant to reach the end of the pipe – estimate 60 minutes. Stop flow.	
BWS-4	• Collect after 1 hour of the surfactant reaches end of the pipe (i.e., 2:00 PM)	September 21, 2015 Flow at 0 gpm
BWS-5, BWS-5D and Cl2-F-3	• Collect after 2 hours (i.e., 3:00 PM)	September 21, 2015 Flow at 0 gpm
BWS-6, CP-4 and CP-4D	• Collect after 3 hours (i.e., 4:00 PM)	September 21, 2015 Flow at 0 gpm
BWS-7, CP-5, CP-5D and Cl2-F-4	• Collect after 1,200 – 1,440 minutes (20 – 24 hrs.) i.e., 9:00 AM. Restart flow flush (5 gpm)	September 22, 2015 Flow at 0 gpm/5gpm
Step 5 – Post Decon. Flushing and Monitoring		
BWS-8, CP-6, CP-6D and Cl2-F-5	• Collect after at 3 hours from the start of flow flush (12:00 PM)	September 22, 2015 Flow at 5 gpm
BWS-9, CP-7, CP-7D and Cl2-F-6	• Collect after at 1,200 – 1,440 minutes (20 – 24 hrs.) from the start of flow flushing and turn down flow to 2.5 gpm.	September 23, 2015 Flow reset at 2.5 gpm
BWS-10, CP-8, CP-8D	• Collect after 1,440 minutes of the start of reconditioning	September 24, 2015 Flow at 2.5 gpm
BWS-11, CP-9, CP-9D	• Collect after 1,440 minutes of the start of reconditioning	September 25, 2015 Flow at 2.5 gpm
BWS-12	• INL will collect 7 days after the start of reconditioning	September 30, 2015 Flow at 2.5 gpm
BWS-13	• INL will collect 14 days after the start of reconditioning	October 7, 2015 Flow at 2.5 gpm

After completion, leave the WSTB blank coupons in place for shutdown and winter storage.

3.2.3 Dechlorination/Flushing Experiments

The purpose of these experiments are to demonstrate the feasibility of using online water sensors in concert with flushing hydrants to intelligently divert and remove contaminants from water distribution systems. For these experiments, the Hach CL-17 and the Real Tech instruments will be used to signal a flushing hydrant to open and flush the injected contaminant from the WSTB. For this purpose, a test will be performed using sodium thiosulfate as the "injected contaminant" to de-chlorinate the system for approximately 30 minutes. A set point or trigger value of measured chlorine level (e.g., 0.05 mg/L or lower) using the Hach CL-17 at the upstream location will be used for opening the flushing hydrant valve. After the chlorine value at the upstream recovers (e.g., > 0.5 mg/L) to the background value, the valve will be automatically triggered to close. Grab

samples will be collected and analyzed for free chlorine residuals at a downstream location from the flushing hydrant. The purpose of this grab sample will be to determine if dechlorinated water is able to "jump" across the "tee" to the flushing hydrant and proceed downstream. Free chlorine levels for these grab samples will be measured using the Hach DR/890 Pocket Colorimeter. The triggered flushing experiment will be independent of the contamination/ decontamination experiment and will occur after the contamination/decontamination experiment if time and weather permits. These tests were completed in 2014 and may be repeated in 2015 or 2016.

For the second objective, the Hach CL-17 will be used to trigger a flushing event based on chlorine concentrations, as described above. The RealTech M4000 TOC instrument's ability to trigger events based on organic concentrations will be evaluated at a later date and the QAPP will be amended accordingly.

3.3 Measurements and Analytes

The samples generated during the studies described in Section 3.2, will be analyzed for the following parameters:

- *B. globigii* (BWS-B (background) and all BWS numbered samples)
- HPC (only sampled prior to start of test – CP-0/CP-0D, BWS-0)
- Free Chlorine (via Hach CL-17 – online upstream/downstream)
- TOC (via RealTech UV254 – online upstream/downstream)
- Free chlorine residuals (downstream grab samples – field measurement)
- Crude Oil components (VOC's, BTEX, GRO, DRO and ORO) BWS and CP numbered samples

4.0 SAMPLING PROCEDURES

4.1 Site-Specific Factors

Contamination/decontamination and flushing experiments will be conducted at INL. Samples will be shipped to the EPA T&E Facility for microbial analysis including HPC and *B. globigii* and to ALS Environmental in Cincinnati, Ohio for chemical analysis including VOC, BTEX, GRO, DRO, and ORO. A summary of the experimental sampling strategy (including the number of samples) is presented in Table 4-1.

Table 4-1. Summary of Experimental Sampling Strategy

Sample/ Sampling Location	Matrix	Measurement	Measurement Location	Sampling Frequency	Total No. of Samples
Contamination - Decontamination Tests/ WSTB	Biofilm	HPC	T&E Facility	1 sample in duplicate	2
	Biofilm/ Coupon	VOC/BTEX	ALS Laboratory	9 samples in duplicate	18
	Water	*B. globigii*	T&E Facility	38 100 mL samples (all in duplicate)	76 (100 ml)
	Water	VOC/BTEX	ALS Laboratory	10 samples in duplicate	20
	Water	GRO	ALS Laboratory	10 samples in duplicate	20
	Water	ORO+DRO	ALS Laboratory	10 samples in duplicate	20
	Water	Free Chlorine	Field Site	6 samples	6

4.2 Sampling Procedures

Extraction of Biofilm and Spores from Coupon Surface for HPC analyses

The coupons will be collected from the WSTB carefully without touching the surface that was exposed to WSTB water. The biofilm and spores will be scraped from the surface using a disposable sterile surgical scalpel. The extracted material will be collected in a sterile sample bottle with a sodium thiosulfate tablet and 100 mL of pre-filled carbon-filtered water. The extracted sample will be transferred to a cooler at $4^{\circ}\pm2^{\circ}$C. The samples will be shipped overnight to the EPA T&E Facility and analyzed upon receipt.

Samples for HPC Concentration Measurement

The BWS for HPC concentrations (BWS-0) will be collected using the grab sampling technique in 100-mL sterile sample bottles with a sodium thiosulfate tablet. The BWS sampling port will be opened and the water will be drained for 15 seconds prior to collection of 100 ml of water from the WSTB. The extraction of biofilm from the coupon surface (CP-0/CP-0D) will be conducted as described in the previous paragraph. The samples will be transferred to a cooler at $4^{\circ}\pm2^{\circ}$C. The samples will be shipped overnight to the EPA T&E Facility and analyzed upon receipt.

B. globigii spores during water treatment experiments

The samples for *B. globigii* concentrations will be collected using the grab sampling technique in 100-mL sterile sample bottles with a sodium thiosulfate tablet. The sampling port will be opened and the water will be drained for 15 seconds prior to collection of 100 ml of water from the WSTB. If needed, 20 L will be collected in 1-gallon flexible plastic bladders (cubitainers) with sodium thiosulfate tablets (0.01% w/v). A sample of water will be removed from the cubitainers to ensure that no free chlorine residual is present. The samples will be transferred to a cooler at $4^{\circ}\pm2^{\circ}$C. The samples will be shipped overnight to the EPA T&E Facility and analyzed upon receipt.

To estimate the BWS background (BWS-B), a sample bottle containing sterile buffer solution will be exposed to background air while the actual BWS is being collected to serve as the background control.

Samples for Free Chlorine – Field Measurement

During the dechlorination/flushing experiments, grab samples will be collected from a downstream location of the flushing hydrant using the grab sampling technique and a laboratory beaker and analyzed for free chlorine. The sample will be immediately processed for measurement using the Hach Method 10102 (pocket Colorimeter) in the field.

Water Sample Concentrator (if needed)

Once received at the EPA T&E Facility, the 20 L water samples (labeled WSC) will be subjected to concentration using the water sample concentrator. Vince Gallardo will operate the water sample concentrator according to EPA NHSRC's Water Sample Concentrator Standard Operating Procedure (SOP) 030 (Automated Concentrator Ultrafiltration Protocol – Appendix F). The resulting concentrated sample will be placed into sterile 100 mL sample bottles and analyzed in the same manner as all other *B. globigii* BWS.

Hayward Saline C 6.0 Chlorination System The BWS for *B. globigii* concentrations from the influent (i.e., the lagoon water mixed with *B. globigii*) prior to operating the system will be collected. After the system is started, periodic effluent/treated samples (BWS-0 through BWS-6) will be collected from the lagoon. Both influent and effluent samples will be collected using the grab sampling technique in 100-mL sterile sample bottles with a sodium thiosulfate tablet.

Advance Oxidation Process (AOP) Trailer

The BWS for *B. globigii* concentrations (BWS-0 through BWS-6) will be collected from the inlet and outlet of the system using the grab sampling technique in 100-mL sterile sample bottles with a sodium thiosulfate tablet. The BWS sampling ports at both inlet and outlet of the system will be opened and the water will be drained for 15 seconds prior to collection of 100 ml of water.

Solstreme, Water Treatment System

The BWS for *B. globigii* concentrations (BWS-0 through BWS-6) will be collected from the inlet and outlet of the system using the grab sampling technique in 100-mL sterile sample bottles with

a sodium thiosulfate tablet. The BWS sampling ports at both inlet and outlet of the system will be opened and the water will be drained for 15 seconds prior to collection of 100 ml of water.

WaterStep Mobile Water System

The BWS for *B. globigii* concentrations (BWS-0 through BWS-5) will be collected from the inlet to the WaterStep MWS sampling port using the grab sampling technique in 100-mL sterile sample bottles with a sodium thiosulfate tablet. The influent sample wil be collected from the sample port after the MWS system bladder tank is filled and prior to system operation). Because it is a closed system (where the chlorine generated is continuously mixed with the contents in the MWS system bladder tank), periodic effluent samples from the bladder tank will be collected to represent the various stages of treatment. The BWS sample port of the system will be opened and the water will be drained for 15 seconds prior to collection of 100 ml of water.

Extraction of Crude Oil from Coupon Surface for VOC/BTEX analyses

The coupons will be collected from the WSTB carefully without touching the surface that was exposed to WSTB contaminated/decontaminated water. The coupon surface will be scraped using a disposable sterile surgical scalpel. The extracted material will be collected in the sample bottle provided by ALS for this analysis. The extracted sample will be transferred to a cooler at $4^{\circ}\pm2^{\circ}$C and shipped to ALS Environmental (so that it arrives before the 48 hour hold-time) to be analyzed upon receipt.

BWSs for VOC/BTEX/GRO/DRO/ORO

The BWS for VOC and BTEX combined, DRO and ORO combined, and GRO will be collected using the grab sampling technique in the 40 mL Volatile Organic Analyte (VOA) vial 100-mL with preservative (hydrochloric acid) provided by ALS Environmental. The BWS sampling port will be opened and the water will be drained for 15 seconds prior to collection of the sample from the WSTB. The samples will be transferred to a cooler at $4^{\circ}\pm2^{\circ}$C. The samples will be shipped overnight to ALS Environmental for analysis.

4.3 Sampling Containers and Quantities

Sample containers and quantities are shown in Table 4-2.

4.4 Sample Preservation and Holding Times

Sample preservation and holding times are shown in Table 4-2.

4.5 Sample Labeling

Sample identification is discussed in Section 3 and summarized below.

Samples collected for analysis will be identified by type (BWS, CP, or Grab), collection interval (-0, -1, -2, etc.), analysis (*B. globigii*, HPC, free chlorine CL2-F, BTEX/VOC, GRO, ORO/DRO), and date collected. Duplicate coupons will be identified using a "D" after the collection interval.

Table 4-2: Grab Sampling and Analytical Procedures

Measurement	Sampling Method	Analysis Method	Sample Container/ Quantity of Sample	Preservation/ storage	Holding times
Free Chlorine	As specified in Section 4.2	Appendix B Hach Method 10102	Glass beaker (~50 mL)	None	Immediate
B. globigii spore	As specified in Section 4.2	Appendix C CB&I T&E SOP 309	100 mL sterile sample bottles 20 L cubitainers[1]	The bottles contains sodium thiosulfate tablet. Cool 4 ± 2°C	Analyze upon receipt at the EPA T&E Facility.
HPC	As specified in Section 4.2	Appendix A CB&I T&E SOP 304	100 mL sterile sample bottles	The bottles contain sodium thiosulfate tablets. Cool 4 ± 2°C	48 hours
VOC/BTEX (Bulk Water)	As specified in Section 4.2	EPA Method SW8260B	40 mL VOA vial	The bottles contain hydrochloric acid. Cool 4 ± 2°C	14 days
GRO (Bulk Water)	As specified in Section 4.2	EPA Method SW8015A	40 mL VOA vial	The bottles contain hydrochloric acid. Cool 4 ± 2°C	14 days
ORO/DRO (Bulk Water)	As specified in Section 4.2	EPA Method SW8015B	1 L amber bottle minimum 200 mL	Cool 4 ± 2°C	7 days
VOC/BTEX (Biofilm Coupons)	As specified in Section 4.2	EPA Method SW8260B/5035 sampling kit[2]	40 mL tared VOA vial with a stir bar	Cool 4 ± 2°C	48 hours

[1]The 20 L cubitainer samples will be concentrated via the water sample concentrator and placed into the 100 mL sterile sample bottles for analysis.

[2]Method 5035 - Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples

4.6 Sample Packaging and Shipping

The biofilm samples, BWSs, and WSC samples will be preserved in coolers with ice and shipped to the EPA T&E Facility overnight. Chain-of-custody forms will be completed and shipped with the samples. The Chain-of-Custody form is presented as Appendix E.

5.0 MEASUREMENT PROCEDURES

5.1 ANALYTICAL METHODS

The analysis methods are shown in Table 4-2. The microbiological methods are further discussed below for the *B. globigii* contamination/decontamination testing.

HPC determinations will follow T&E SOP 304, *Heterotrophic Plate Count (HPC) Analysis Using IDEXX SimPlate Method*. This method is based on multiple enzyme technology which detects viable bacteria in water by testing for the presence of key enzymes known to be present in these organisms. It uses multiple enzyme substrates that produce a blue fluorescence when metabolized by bacteria. The sample and media are added to a SimPlate plate, incubated, and then examined for fluorescent wells. The number of fluorescing wells corresponds to a Most Probable Number (MPN) of total bacteria in the original sample. This method is included as Appendix A in this document.

Preparation and analysis of *B. globigii* will follow T&E SOP 309, *Preparation and Enumeration of B. globigii Endospores*. *B. globigii* is an aerobic spore-forming bacteria used as a surrogate for evaluating the performance of water treatment systems for removal of bacterial endospores. In analyzing spores, the indigenous vegetative cells are inactivated by heat treatment. The surviving bacterial spores in the sample are analyzed by culturing that permits the spores to germinate and produce bacterial cells. Tryptic soy agar will be used for culturing *B. globigii*. This method is included as Appendix B in this document.

The samples are diluted, as necessary, depending on the expected concentration of cells/spores in the sample. For example, the expected initial concentration of spores in this study is 10^6 spores/mL. The initial samples will be diluted up to 10^5 fold. Duplicate plates using 0.1 mL of the 10^4 and 10^5 fold diluted samples will be analyzed using the spread plate method. If the number of colonies is too many to count in more than one plate, the sample will be diluted and re-analyzed. If the number of colonies is too many to count for one measurement, the remaining plates will be considered for enumeration of spore concentration for the sample.

For the crude oil injection test, Standard EPA Methods will be used for analyzing the chemical constituents. Specifically, EPA Method SW8260B will be used for VOC/BTEX in bulk water, EPA Method SW8015A will be used for GRO in bulk water and EPA Method SW8015B will be used for ORO/DRO in bulk water. Due to the limited coupon sample quantity, only VOC/BTEX contents of the coupon will be analyzed. Specifically, EPA Method SW8260B will be used for the coupon sample analysis and extraction will be performed using the EPA Method 5035 sampling kit.

5.2 CALIBRATION PROCEDURES

The calibration procedures, linearity checks, and continuing calibration checks are included in the T&E SOPs or the instrument manuals for the analysis methods referenced in Table 4-2.

6.0 QUALITY METRICS (QA/QC CHECKS)

6.1 QC Checks

Instruments/equipment will be maintained in accordance with the EPA ORD Policies and Procedures Manual, Section 13.4 *Minimum Quality Assurance (QA)/Quality Control (QC) Practices for ORD Laboratories Conducting Research* and in accordance with the SOPs and analysis methods listed in Table 4-2, and for field instruments, in accordance with the manufacturer's instructions. Table 6-1 presents the QA/QC checks to be implemented for the measurement of the specific parameters.

6.2 QA Objectives

The objectives of this study are described in Section 1.2. These objectives will be addressed by collecting data on contaminant reduction. Table 6-1 lists the QA/QC checks that will be used to verify the validity of the analyses conducted on grab samples conducted during this study. Table 6-2 summarizes the QA/QC requirements for the optical devices used in this study.

The RPD is calculated for duplicate analyses based on the following:

$$RPD = \frac{(C_1 - C_2)}{0.5(C_1 + C_2)} \times 100\%$$

where:
RPD = Relative Percent Difference
C1 = Larger of two values
C2 = Smaller of two values

If calculated from three or more replicates, the relative standard deviation (RSD) will be used according to the following equation:

$$RSD = 100\% \ \frac{s}{y_{ave}}$$

where:
RSD = relative standard deviation (%)
s = standard deviation
y_{ave} = mean of the replicate analyses

Standard deviation is defined as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - y_{ave})^2}{n-1}}$$

where:
s = standard deviation
y_i = measured value of the ith replicate
y_{ave} = mean of the replicate measurements
n = number of replicates

Table 6-1: QA/QC Checks for Grab Samples

Measurement	QA/QC Check	Frequency	Acceptance Criteria	Corrective Action
B. globigii	Positive control using stock	Once per experiment	±10 fold of the spiking suspension	Investigate laboratory technique. Change stock organisms and use new set of media plates. Re-analyze the spiking suspension and change it if necessary.
B. globigii	Negative Control using sterile buffer	Once per experiment	0 CFU[a]/plate	Investigate laboratory technique. Use a new lot. Re-analyze.
B. globigii	Negative control for heat shock	Once per experiment	0 CFU of vegetative cell/plate	Investigate the hot water bath. Heat samples for longer period.
B. globigii	Duplicate	Once per experiment	≤20% variation	Consider other dilutions. Reanalyze.
B. globigii	Field blank (an open bottle of sterile water in the vicinity of the BWS location)	Every 5 BWS	0 CFU/plate	Determine if background values impact results.
HPC	Negative Control	Before every set of measurements	No fluorescent wells	Re-analyze sterile buffer and change it if necessary.
HPC	Positive Control	Once per experiment	Fluorescent wells	Investigate laboratory technique. Re-analyze.
HPC	Duplicate	Once per experiment	Duplicate plates much agree within 5%	Investigate laboratory technique. Re-analyze.
Free Chlorine	Manufacturer DPD color standards kit	Once per experiment	As specified by the color standards kit	Clean the colorimeter measuring cell. Clean the DPD standards vials and recheck.

Measurement	QA/QC Check	Frequency	Acceptance Criteria	Corrective Action
VOC/BTEX (Bulk Water)	Initial calibration check	Once per batch of 20 samples	Pass	If fails repeat calibration
VOC/BTEX (Bulk Water)	laboratory control sample, matrix spike, and matrix spike duplicate	Once per batch of 20 samples	Method Criteria	If any of the QA/QC checks fail utilize the duplicate sample. Report with appropriate qualifier if necessary.
GRO (Bulk Water)	Initial calibration check	Once per batch of 20 samples	Pass	If fails repeat calibration
GRO (Bulk Water)	laboratory control sample, matrix spike, and matrix spike duplicate	Once per batch of 20 samples	Method Criteria	If any of the QA/QC checks fail utilize the duplicate sample. Report with appropriate qualifier if necessary.
ORO/DRO (Bulk Water)	Initial calibration check	Once per batch of 20 samples	Pass	If fails repeat calibration
ORO/DRO (Bulk Water)	laboratory control sample, matrix spike, and matrix spike duplicate	Once per batch of 20 samples	Method Criteria	If any of the QA/QC checks fail utilize the duplicate sample. Report with appropriate qualifier if necessary.
VOC/BTEX (Biofilm Coupons)	Initial calibration check	Once per batch of 20 samples	Pass	If fails repeat calibration
VOC/BTEX (Biofilm Coupons)	laboratory control sample, matrix spike, and matrix spike duplicate	Once per batch of 20 samples	Method Criteria	Report with appropriate qualifier if necessary.

a - Colony Forming Unit

Table 6-2. QA/QC Checks for Online Equipment

Instrument/ Measurement	Calibration/QC Alternative	Frequency	Acceptance Criteria	Corrective Action
RealTech UV254/ TOC	Custom Zero using deionized water	One time per quarter according to instrument O/M manual	N/A	Clean the quartz windows using 5% bleach solution.
Hach CL-17/ Free Chlorine	**Factory calibration – do not change**. Perform a one-point check against a DPD colorimetric method calibration based on DPD method	Quarterly	±10%	Clean colorimeter and check the instrument flow.
Modern Water/Multisensor 1200	**Factory calibration and setup performed by the vendor onsite**	Leased equipment for the crude oil test. Setup/calibration performed upon initiation by vendor	NA	NA

7.0 DATA ANALYSIS, INTERPRETATION, AND MANAGEMENT

7.1 Data Reporting Requirements

All data generated during the study will be presented in tabular/spreadsheet format. Table 7-1 identifies the reporting units for the various parameters.

Table 7-1. Reporting Units for Measurements

Measurement	Units
B. globigii	CFU/ mL
HPC	MPN/ mL
Free Chlorine	mg/L
TOC	mg/L
VOC/BTEX (Bulk Water/Biofilm Coupons)	µg/L
GRO (Bulk Water)	µg/L
ORO/DRO (Bulk Water)	µg/L

7.2 Data Validation Procedures

Calculations will be carried out on a computer and will be checked initially by the analyst for gross error and miscalculation. The calculations and data entered into computer spreadsheets will be checked by a peer reviewer for accuracy, and checking the calculation by hand or checking entries of data from the original. Detected errors will be corrected and other data in the same set investigated before it is released to the EPA WACOR.

7.3 Data Summary

All sample data will be presented by CB&I in tabular/spreadsheet format and submitted to the EPA WACOR for evaluation. Tabular data summaries will be included in the main discussion of the reports and raw data will be included as appendices.

7.4 Data Storage

Laboratory records will be maintained in accordance with Section 13.2, *Paper Laboratory Records*, of the Office of Research and Development (ORD) Policies and Procedures Manual. Controlled access facilities that provide a suitable environment to minimize deterioration, tampering, damage, and loss will be used for the storage of records. Whenever possible, electronic records will be maintained on a secure network server that is backed up on a routine basis. Electronic records that are not maintained on a secure network server will be periodically backed up to a secure second source storage media, transferred to an archive media (e.g., compact discs, optical discs, magnetic tape, or equivalent), or printed. Electronic records that are to be transferred for retention will be transferred to an archive media or printed, as directed by EPA.

8.0 REPORTING

8.1 Deliverables

Monthly progress reports will be prepared by the Project Leader and sent to the Program Manager, and submitted to EPA every month. CB&I will prepare data packages for each analysis to be placed into CB&I's project central file.

8.2 Final Report

CB&I will be responsible for preparing a data report that will include a description of the WSTB, how contaminant injections were performed, how decontamination was performed, analyses of data collected from experiments in the WSTB (including the degree of attachment crude oil), and the effectiveness of flushing and decontamination. Infrastructure samples (C_i) taken during crude oil injection will serve as the baseline or initial level of crude oil in the water or on the coupons. Samples taken during flushing and surfactant addition (C_d) will be compared to the samples taken during contamination as follows to determine percent decontamination (%D):

$$\%D = \frac{Ci - Cd}{Ci}$$

Coupons will be sampled in duplicate, and Ci and Cd will be the mean of these duplicates. The duplicate values and well as the range between these duplicate values will be reported in the tabulated data.

The effectiveness (%E) of individual large volume treatment technologies will be evaluated based on a mass balance approach where the water containing *B. globigii* spores will be sampled before it enters the individual treatment technology (Ci) and then after (Ce) to determine its effectiveness.

$$\%E = \frac{Ci - Ce}{Ci}$$

%E will be calculated for each time point sampled, but an overall mean %E will be calculated that using the mean Ci and mean Ce that includes all of the data collected over the course of the experiment. In this case, the variance of Ci and Ce (Sc_i and Sc_e, respectively) will be calculated and standard error (SE) will be calculated for the mean %E as follows (n is the number of samples).

$$SE = \sqrt{\frac{S^2ci}{\mathrm{n}} + \frac{S^2ce}{n}}$$

The analytical data will be presented in tabular form unless otherwise noted. Tabular data summaries will be included in the main discussion of the reports, and raw data will be included as appendices.

The report will include all data tabulated in Microsoft® Excel and Word formats, and will be provided in print and electronic formats. It will include narratives of the methods and results. Interpretive graphs will also be provided.

9.0 REFERENCES

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Appendix

 Appendix A_304 Heterotrophic Plate C	Appendix A	CB&I T&E SOP 304 - Heterotrophic Plate Count (HPC) Analysis Using IDEXX SimPlate Method
 Appendix B_DR 4000 Chlorine-Free Methoc	Appendix B	Hach Method 10102 for Measuring Free Chlorine in Water
 Appendix C_309 for Preparation of B. glob	Appendix C	CB&I T&E SOP 309: Preparation and Enumeration of B. globigii Endospores
 Appendix D-GO2_International.	Appendix D	Preparation of the GO2 4,000 ppm Solution
 Appendix E_EPA_COC_Form.pc	Appendix E	EPA Sample Chain of Custody Form
 Appendix F_Water Concentrator SOP.pd	Appendix F	EPA Water Concentrator SOP
 Gasoline_In_Water_ a270016.pdf	Appendix G	Characterization of gasolines, diesel fuels & their water soluble fractions
 BakkenCrude_Info_1 50213064220.pdf	Appendix H	Bakken Crude Oil

Appendix B: Summary of Technology Specific Considerations

Technology Considerations	EPA AOP trailer*	Solstreme*	WaterStep*	Hayward*
Market Availability	Low. Originally custom designed by EPA for a remediation project to provide advanced oxidation with UV and Ozone. A trailer-mounted system that was re-purposed and tested for disinfection. One ozonation process component (Speece Cone diffuser) not commercially available. Other UV and ozonation process components commercially available.	Medium. New startup company developed an innovative electrodeless UV lamp design. Made upon order (http://www.solstreme.com/)	High. Commercially available off-the-shelf product from a non-profit organization for producing drinking water in communities in developing countries. Self-contained kit, could be used in disaster zone to purify water even if there was no power. Available from http://waterstep.org/	High. Commercially available *in-situ* chlorine generator, off-the-shelf product from a pool product manufacturer. Commonly used for disinfecting swimming pools. Available from http://www.hayward-pool.com/
Capital Cost	High (estimated > $40,000). Custom design, process components, plumbing, trailer, etc.	Medium (est. $15,000).	Medium (est. $8,000). Includes storage bladders, pump, battery, charger, solar cell, mounting/transportation rack, and salt based chlorine generator (Chlorinator).	Low. $4,000
Shipment to Site	Medium. Requires a tow vehicle to pull the trailer to site. Trailer may require State inspection and driver that meets the training requirements for towing the vehicle.	Low. Requires a custom-box (wooden crate or cardboard box with contoured foam) and can be shipped via third party shipper to site. No chemicals or hazardous materials to ship. Can be carried in a truck or a personal car to site.	Medium. Needs to go on a truck or commercial transportation. Could be transported in a car, if mounting and transportation rack are not used.	Low. Small package easy to ship or carry in a car.
Setup Considerations	Medium. Requires 110 and 220 Volt AC electric or generator, the plumbing connections to the process units need to be reassembled on site. The Ozonator cone setup requires 2-3 persons onsite to assemble.	Low. Plug and play needs 110 Volt AC electric. If water is turbid, a pre-filter is recommended for optimal use. Temperature of the water (i.e., cold < 55ºF) impacts operations. Comes with cam lock type connectors. One person can set it up in the field.	Medium. Need flat surface to spread out the bladder tanks. Need to recirculate chlorinated water to provide contact time for disinfection. Not a flow through system. Strip kit required to periodically check chlorine generation. After disinfection, if chlorine is not consumed, the excess chlorine needs to be neutralized before discharging to the environment.	Medium. Can be setup on a table. Requires a 40 gpm (151.4 L/min) pump to run salted water through the system. Salt needs to be added to the source water in sufficient quantities (3,000 to 5,000 ppm). Chlorine generation can be varied as needed. Need to recirculate chlorinated water to provide contact time for disinfection. Not a flow through system. Strip kit required to periodically check chlorine generation.

Technology Considerations	EPA AOP trailer*	Solstreme*	WaterStep*	Hayward*
Operational Considerations	Medium. Requires operation of valves to remove air from the process units, valve adjustment to meet pressure and flow requirements. Some of the vented air may contain contaminated droplets of water that need to be contained or recirculated back through the system. There are excess ozone emissions from process unit that need to be destroyed or vented. The catalytic destruction unit was un-operable, the unit had to be vented. Flow rate needs to be less than 5 gpm (18.9 L/min).	Low. High turbidity and cold water adversely affect the disinfection process. It gets better results with water in the 70ºF to 90ºF temperature range and low turbidity. If high disinfection is desired a heat exchanger may also be needed to regulate water temperature. The cost of the heat exchanger will depend on the size of the unit.	Low. Simple to operate on a short-term basis. If extended contact period is required greater than 3 hours, the salt solution needs to be replenished, electrolytic cell has to be drained, and if not on 110 volt AC power, the battery needs to be charged.	Low. Requires 110 volt AC power, high capacity (40 gpm (151.4 L/min)) pump. Initial setup requires the chlorine production of the system to be slowly ramped up by starting at ~50% production rate and increased incrementally. Salt may need to be added depending upon usage. While it could be operated using bladder tanks, but suited for open pools.
Maintenance and Consumables	Low. UV lamp replacement, pump repair when needed. Dual voltage electric supply (see setup consideration).	Low. If the processed water is turbid, the system (inside quartz sleeve) will need to be cleaned frequently. Other than regular commercially available cleaning agents, no other consumables are required. The UV lamp is electrodeless microwave technology, expected to last more than 10 years. The quartz sleeve although robust needs to be handled carefully while cleaning. A plunger type device for cleaning the interior of the sleeve is recommended and gloves should be used to prevent smudging of the outside surface.	Low. Table salt is the only consumable. For optimal chlorine generation, the electrolytic cell needs to be cleaned periodically. Pumps, hoses and O-rings need to be checked as needed.	Low. High purity salt (NaCl ~98%). Pump and hoses need to be checked as needed.
Result Summary Under Tested Conditions	Under the tested conditions, an average of 4-log removal of B. globigii was observed in this flow through type operation (removal varied from 1.5 to 5 log). Improved understanding of the EPA AOP system performance may improve the consistency of disinfection.	A 3.5-to 4-log removal of *B. globigii* was observed in a flow through type operation.	A 7-log removal of *B. globigii* was observed in a batch type operation with 300-minutes of contact time.	The unit achieved a 4.3 log reduction of *B.* at a Ct of almost 7,000 mg-min/L.

* Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.

Appendix C: Technical Bulletin SURFONIC® DOS-75PG Surfactant

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Technical Bulletin

SURFONIC® DOS-75PG Surfactant

GENERAL NAME Dioctyl sodium sulfosuccinate

PRODUCT DESCRIPTION

SURFONIC® DOS-75PG surfactant is an anionic sulfosuccinate surface-active agent with excellent wetting and surface tension reducing properties. The solvent system is a mixture of propylene glycol and water. SURFONIC® DOS-75PG surfactant is compatible with other anionic surfactants and with nonionic surfactants.

APPLICATIONS

- wetting agents
- solubilizing agents
- emulsifiers
- detergents
- dispersants

SALES SPECIFICATIONS

Property	Specifications	Test Method*
Appearance, 25°C	Clear liquid	ST-30.1
Anionic Active, wt%	69 - 71	ST-31.145
Color, Pt-Co	60 max.	ST-30.12
pH, 5% in distilled water	5.0 - 7.0	ST-31.36,C

*Methods of Test are available from Huntsman Corporation upon request.

TYPICAL PROPERTIES

Chemical Properties	
Molecular Weight (theoretical)	444
Water Solubility	Soluble

Regulatory Information	
DOT/TDG Classification	Not Regulated
HMIS Code	1-1-0
CAS Number	577-11-7
TSCA Inventory	Yes
WHMIS Classification	D2B
Canadian DSL	Yes

Physical Properties	
Flash point, PMCC, °F	248
Flash point, PMCC, °C	120
Freeze point, °F	-4
Freeze point, °C	-20
Density, g/ml at 25°C (77°F)	1.120
Weight, lbs/US gal at 25°C (77°F)	9.33
Viscosity, Brookfield cps at 20°C (68°F)	700

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TOXICITY AND SAFETY

For information on the toxicity and safe handling of this product, please read the Material Safety Data Sheet prior to use of the product.

HANDLING AND STORAGE

SURFONIC® DOS-75PG surfactant may be satisfactorily stored in stainless steel tanks using stainless steel pipes and pumps. Carbon steel tanks are not recommended; storage in carbon steel for extended periods of time may cause discoloration of the product due to rusting. For this reason, lined, stainless steel or fiberglass tanks are recommended. An inert atmosphere such as nitrogen should be maintained in larger storage vessels.

Solid sediment may form upon standing. There should be circulation in the storage vessel to keep solids suspended.

Low pressure steam coils in storage tanks and steam tracing of transfer lines should be provided in cases where low environmental temperatures may make pumping of the product difficult.

SHIPPING DATA

Product is available in tank cars, tank trucks and drums of 485 pounds (220 kilograms) net weight. Small samples are available by contacting our sample department at 1-800-662-0924.

BIODEGRADABILITY AND ENVIRONMENTAL SAFETY

SURFONIC® DOS-75PG surfactant and related products have been shown to undergo 90% to 98% biodegradation in 11 to 17 days.

General References

Swisher, R. D., Surfactant Biodegradation, Marcel Dekker, 1987.

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